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Appendices

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Volume 2 of 2

NASSAU UNIFORM SERVICES
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APPENDICES

APPENDIX A

INSTRUCTION MANUAL
PERMAC BOWE DRY-CLEANING
AND DEGREASING MACHINE

Büse

Böhler & Weber KG Maschinenfabrik Augsbur

BÖHLER & WEBER KG
Augsburg

INSTRUCTION MANUAL

for the PERMAC BÖWE Dry Cleaning and Degreasing Machine

SE 120 "maxima export"

BA 1.105.0002-0, 15e.365

Applicable up to No. 316 (inclusive)

Dear Customer,

first of all may we congratulate you on your new PERMAC BÖWE degreasing machine and hope that it will come up to your expectations.

PERMAC BÖWE SE machines of the "maxima" series are based on the latest developments in dry cleaning machinery engineering and are the result of experiences gained with more than 8.000 PERMAC BÖWE dry cleaning plants all over the world.

When designing this model, our engineers tried to satisfy all your requirements and to provide the best possible conditions for a smooth, reliable dry cleaning service with excellent cleaning results.

Only a thorough study of this operating manual ensures that your dry cleaning machine will produce the results you expect. All the experiences of many years have been evaluated in this manual for you. Please, do not try to learn from mistakes, but learn from the manual!

We, therefore, ask you to study it prior to installing the machine and to keep it always handy for continual use by your operating personnel. Your new dry cleaning machine will reward your efforts by better cleaning results and trouble-free operation.

Yours very truly,

BÖHLER & WEBER KG
MASCHINENFABRIK

C O N T E N T S

D r a w i n g s 1 - 7

1. Foundation for the PERMAC BÖWE 3oo me, SK 12o me, SE 12o me and W 12o me

   Drawing No. 11o5.13-o8

2. Reinforcing Ironwork for the Foundation of the PERMAC BÖWE 3oo me, SK 12o me, SE 12o me and W 12o me

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3. Service and Maintenance Area for the PERMAC BÖWE 1oo m, SK 4o m, W 4o m as well as 3oo me, SK 12o me, SE 12o me, W 12o me

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4. Front View of the PERMAC BÖWE SE 12o me

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6. Piping Diagram of the PERMAC BÖWE SE 12o me

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7. Pneumatic Controls for the PERMAC BÖWE SE 12o me Three Bath Automatic Control and Activa

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A) GENERAL DESIGN

PERMAC BÖWE degreasing machines are used for the removal of oil and grease based soiling from industrial overalls, machinery cleaning cloth as well as for the degreasing of leather and skins.

The PERMAC BÖWE degreasing machines for perchlorethylene or trichlorethylene are identified by the following special features:

1) Well-proportioned, space-saving packaged construction, partly panelled and finished with a chip and solvent proof special enamel in a range of attractive colours.

2) Cleaning, resp. degreasing, extracting, drying and deodorizing in one complete cycle.

3) Additional facilities for special aftertreatments.

4) Favourable diameter and loading factor of the stainless steel cleaning cage.

5) Two-way air guide with contra-flow principle for the drying.

6) Solvent recovery by means of corrosion-resistant heat exchangers and - if required - by means of an adjustable spray jet.

7) Independent distillation and drying owing to separate cooling systems with thermostatically controlled cooling water regulation.

8) Removal of sludge and grease residues from the still without interruption to the working process.

9) Facility for the additional installation of a fully automatic PERMAC BÖWE Expander Filter with spring stainless steel elements.

10) Separate motors for cleaning and extracting.

Deodorizing solvent losses are eliminated, when installing a PERMAC BÖWE ACTIVA Active carbon recovery plant. Total solvent losses will be reduced considerably this way, generally 50 %.

The PERMAC BÖWE ACTIVA units are supplied in two distinct basic models: space-saving individual small units for top-mounting on the cleaning machine, and allowing for the connection of two machines operating alternatively; the large size industrial units for larger groups of machines.

Whenever the ceiling height does not allow the top-mounting of an individual small unit, this can be placed on wall brackets.

PERMAC BÖWE degreasing machines correspond to the latest developments in dry cleaning technology. They guarantee exceptional reliability, maximum versatility in processing methods and are extremely easy to operate.

B Ö W E also offers:

Thorough instruction in the use of the machine through experienced dry cleaning technicians and by means of detailed printed information;

An advisory service which covers dry cleaning in general and plant organisation as well as cleaning technology;

A world-wide service organisation with spare parts depots in many countries.

B) TECHNICAL DATA

Output per load	12o	lbs.
Cage diameter	4' 2"	
Cage depth	2' 6"	
R.p.m. wash	28	rpm.
R.p.m. extract	41o	rpm.
Shipping dimensions Machine		
Length	11' 6"	
Width	6' 3"	
Height	9' 6"	
Button trap, industrial type (not standard):		
Length	4' 3"	
Width	3' 6"	
Height	2' 4"	
Filter (not standard):		
Length	5' 9"	
Width	3' 4"	
Height	2' 8"	
Installation dimensions:		
Length	1o' 9"	
Width	5' 5"	
Height	9' 7"	
Height with top-mounted ACITVA	11' 6"	
Width with button trap, industrial type (not standard):	8' 1"	
Length with side-mounted filter (not standard):	12' 1"	
Required entry opening (minimum)		
Width	5' 7"	
Height	9' 7"	
Floor space occupied	1o' 9" x 4' 9"	
Weight without solvent	9o65	lbs.
Weight with solvent	15235	lbs.

Contents	Imp.Gals.	U.S.Gals.
Tank I	92	111
Tank II	62	74
Tank III	61	73
Tank IV	128	153
Still I	128	153
Still II	33	4o
Filter (not standard)	4o	48

Electrical connections

Wash motor	5.o	HP
Extractor motor	1o.o	HP
Pump motor	3.o	HP
Fan motor	6.7	HP
Pump motor II	2.o	HP
	26.7	HP

Electrical service connection (maximum)

22o V, 3 phase	71	amps.
415 V, 3 phase	39	amps.
Filter throughput	6ooo	U.S.gals./h
(filter not standard)	5ooo	Imp.gals./h
Deodorizing duct	6"	Ø
Compressed air connection	1/4"	
Required air pressure	6o - 85	PSI
Required water pressure	3o - 45	PSI
Required steam pressure		
with perchlorethylene	3o	PSI
with trichlorethylene	35	PSI
Rate of steam consumption (maximum)	8.8	lbs./min.
Steam supply	1.1/4"	
Steam connection	3/4"	
Condensate drain	3/4"	
Water connection: Supply	1.1/4"	
Drain	2"	

Normal consumption values:

Perchlorethylene or trichlorethylene	5 - 1o +)	% on the weight of work
Electricity	3.5	kWh/load
Filter powder (only where filter fitted)	4oo	g/load
Compressed aif	2.5	cu.ft./load
S t e a m		
Air heater	5o	lbs./load
Still (based on one bath per load distilled)	165 ++)	lbs./load
or	3	lbs./Imp.gal. of solvent
	2.5	lbs./U.S.gal. of solvent
W a t e r +++)		
(based on 55° F inlet temperature)		
Air cooler	62	Imp.gals./load
	74	U.S.gals./load
Condenser	22o	Imp.gals./load
	26o	U.S.gals./load
or	3.7	gals./gal. solven

+) varies with the type of work

++) with an average solvent quantity of 79 U.S.gals./load (66 Imp.gals./load)

+++) These quantities depend on the initial temperature of the cooling water.

C) DESCRIPTION

1) INSTALLATION AND STARTING UP

Mounting of the machine see foundation plans

Diagrams are provided which show various methods of installation and the arrangement of the foundation for the machine. It is always advisable to keep the cleaning machine away from a wall by a distance equal to its width, particularly on the motor end side of the machine and at the rear.

The foundation bolt sizes are listed in the foundation plan, and when the machine is mounted above a cellar or on an upper floor, one should note that it is advantageous to mount the machine directly above supporting walls or in the immediate proximity of such walls. If in doubt, always consult an architect.

With uneven floors one should raise the machine by approximately 1/2" about the floor level by means of wedges, while the frame is filled in with thin concrete, and the rag bolts are set in position.

The foundation bolts should not be fully tightened until the concrete has completely set.

The machine should be placed in a horizontal position.

For ceiling mounting the following weights have to be considered:

Net weight of the total plant	9080 lbs.
Additional solvent weight	6180 lbs.
Additional out-of-balance forces	7700 lbs.
Total weight	22960 lbs.
Load per sq.ft. of floor area	460 lbs./sq.ft.

When tested on full load, no resonance shoule be apparent. In case of resonance, however, additional supports must be provided.

C o n n e c t i o n s :

Electricity, steam, compressed air, condensate, water, water drain and deodorizing duct according to diagram and technical data.

The electricity supply to the cleaning machine should incorporate a separate wall-mounted fused isolator and the air, steam and water lines should always include stop valves adjacent to the machine.

A T T E N T I O N :

The electricity supply must be checked to insure that it is suitable for the machine.

Pay particular attention to the rotation of the motors. The cage must - viewed from the front of the machine - turn clockwise, all other motors will run in the correct direction if washer turns clockwise. The fan motor which is dismantled for transport must turn anti-clockwise.

Tighten up all bolts and hose pipe connections which may have become loose in transport.

The water pressure should not drop below 3o P.S.I. and should be as constant as possible.

Avoid long feed lines of small diameter, if the water supply varies, choose the next larger pipe diameter to the machine connections.

Where cooling water is used from an overhead tank, and no mains pressure is available, separate water connections of suitable size should be made, using a pressure pump. The heat exchangers of the machine are pressure tested, and the water can therefore return by pump pressure for re-chilling. When connecting the water supplies of the cleaning machine to an evaporative cooling tower, then the syphon which is built into the water outlet of the cleaning machine should be separated by inserting a steel plate below the second water outlet which is normally closed with a cap. The lower outlet of the water drain still has to be used to discharge the water which drains from the water separator. This water should not be returned to the cooling tower. The top outlet connection is connected back to the cooling tower circuit.

If an ACTIVA carbon recovery is mounted on the cleaning machine and the cooling water is provided from a cooling tower, the steam condensate drain of the ACTIVA which is normally fed into the water drain, must be separated and the condensate returned to waste.

If cooling water conditions are particularly critical, please, refer to our technical sales service department.

The compressed air should be adjusted to 6o - 85 PSI,

the steam pressure should not exceed 35 PSI with trichlorethylene, and 6o PSI with perchlorethylene.

II) FILLING WITH SOLVENT

All valves are normally closed. Main switch on "HAND".

The solvent is either entered to the loading door, or via the button trap. Alternatively the free suction connection in front of the pump can be connected to a barrel in order to allow the solvent to be pumped directly into the machine.

a) Filling of the tanks

For this purpose valve no. 5 "from button trap to pump" and either valve 3 "from/to tank II" or valve 2 "from/to tank IV" are opened, until tank II or tank IV are filled.

After tank IV is filled, valve 5 "from button trap to pump" is closed, valve 4 "from tank I" is opened, and the pump switched on, until tank I is filled.

Correspondingly tank III is filled by openeing of valve 16 "to clean solvent tank". The tank IV must for this purpose be refilled in between time.

b) Filling the filter

After the individual tank now the filter is filled.

1)	Main switch on	HAND
2)	Valve 8 "to filter"	OPEN
3)	Valve 7 "Precoat circuit"	OPEN
4)	Valve 5 "from button trap to pump"	OPEN
5)	Valve 15 "from clean solvent tank III"	OPEN
6)	Pump	ON

The filter is filled when the filter outlet sight glass shows solvent flowing through it.

Now the plant is fully filled.

7)	Valve 15 "from clean solvent tank III"	CLOSED
8)	Valve 16 "to clean solvent tank III"	OPEN
9)	Valve 8 "to filter"	CLOSED

The solvent which is left over in the washer is returned to tank III.

As soon as the button trap is empty,

1o)	Valve 16 "to clean solvent tank III"	CLOSED
11)	Pump	OFF

The quantity of solvent which is needed to fill the filter should be replenished immediately to the clean solvent tank.

c) Filling the water separator

The water separator must always be filled with solvent up to a height of approximately 12" prior to cleaning. After this water is added until it overflows.

A T T E N T I O N :

Before filling of the water separator with solvent, it must be completely empty and dry.

III) THE OPERATING CYCLE OF THE MACHINE

The positions of the individual valves and switches are seen from the machine front and rear viewes, whereby the numbers of the valves also correspond to the valves indicated in the piping diagram of the SE 12o "me".

The cleaning process according to the two mostly used methods takes place as follows:

1) Two bath method without filter

The goods are loaded into the cleaning machine, and the automatic control is started.

Cleaning

The goods are cleaned in a precleaning bath, then a rinsing bath generally with an additon of dry cleaning aid follows, and an aftertreatment can also be applied. The solvent is continuously circulated from the washer via the pump back to the washer in a so-called pump circuit; between the individual bath the solvent is extracted.

Pumping back

The precleaning bath is pumped to distillation. The rinse bath of distilled solvent is returned to tank I, where it is used during the next load as precleaning bath.

Distillation

Tank IV fills automatically by means of continuous distillation. It is connected via an overflow with tank III.

Aftertreatment (tank II)

Solvent from tank II which may have been used for an aftertreatment after cleaning and rinsing is always returned back to tank II.

Sludge distillation

The distillation residue which is left behind in still I, is automatically pumped with a special sludge pump(pumpII) to still II, in which the residues are continuously steam-distilled.

Distillate

The distillate drains from the corresponding condenser and flows through the water separator back to the clean solvent tank III.

The cooling water is automatically controlled.

Drying

The fan produdes a continuous air stream which is heated in the air heater and then directed on to the tumbling goods.

The air which causes the evaporation of the solvent, becomes enriched with solvent vapours and leaves the washer housing via the lint filter and is pushed though the air cooler via the fan, where the solvent vapour is condensed out on the air cooler.

The condensed solvent and water return to the water separator, where solvent is separated from water on account of the different specific gravities (perchlorethylene 1.62, trichlorethylene 1.47, water 1.o). The solvent returns to the clean solvent tank, the water goes to waste. The chilled air is now rehumidified by means of a humidifying jet and the air is then reheated on its way back to the washer.

The drying temperature can be regulated automatically by the thermostat "before cooler" or optionally by a second thermostat which is regulating the air inlet temperature.

At the end of the drying process the work is deodorized and now the goods can be removed from the machine cleaned and dried.

2) Two bath method with filter

After loading the solvent drains from tank I into the washer until a low solvent level is reached, which is circulated through the pump circuit. After 3 - 4 minutes precleaning this solvent is pumped into the still and extracted into the still.

Now the washer is refilled from tank I and IV, until the normal dip is reached. The pump delivers this solvent from the washer via the button trap to the filter, and from there via the precoat circuit back to the button trap. This precoats the filter with the necessary filter powder; after approximately 1 minute the precoat circuit closes and the solvent circulates through the work.

Cleaning continues in this filter circuit until all insoluble substances have been removed by the filter and the filter inlet and outlet sight glasses are equally clear.

At the end of the cleaning process the valve "to filter" is closed, and the pumping-back to tank I takes place.

Now the residual solvent is extracted back to tank I; during the breaking process at the end of the extraction the filter residues are automatically shaken off the filter elements.

D) O P E R A T I N G I N S T R U C T I O N S

Consult the machine diagrams in conjunction with the following instructions.

1) H and operation

1) Two bath method

a) Preparation and loading		Operation
Main switch to	HAND	
Loading door	OPEN	The loading door switch causes the starting up of the fan and the opening of the deodorizing and fresh air inlet damper. The fan draws room air into the machine, so that no vapours can enter the cleaning room.
Loading - door	CLOSED	Lever "Circulation/ Deodorizing" goes to "Circulation" and fan switches off.

b) Filter shocking

Applies to machines with additonal filter only.

Operate the press button "brake" 2 - 3 times.

The filter cake is thrown off the filter elements, the filter let-down valve remains closed.

c) Cleaning or degreasing (precleaning)

1) Toggle switch "Heating/signal" up	ON	
2) Valve 1 "from tank I"	OPEN	Solvent drains into the washer.
3) Wash motor	ON	
4) Hand timer adjust precleaning time (f.i. 5 minutes)		
5) Valve 1 "from tank I"	CLOSED	As soon as required solvent level in the washer is reached (see solvent level control).

d) Intermediate extraction

1) Valve 1o "to still"	OPEN	after the adjusted time has expired.
2) Pump I	ON	The level of the solvent must not rise above the lower edge of the still sight glass on still I.
3) When solvent has been pumped away, extraction	ON	
4) Extraction	OFF	after 5 - 7 seconds pre-extraction, then 2o seconds coasting. Total extraction time 2 - 3 minutes.

5) Press button "brake" on, until washer has returned to wash speed.		
6) Valve 1o "to still"	CLOSED	
7) Pump I	OFF	
8) Only for machines <u>with fitted filter:</u> (toggle switch "solvent level" stands in the "high" position.		
a) Valve 1 "from tank I"	OPEN	Remaining solvent drains into the washer. If necessary, replenishment from tank IV. (Refer to level control.)
b) Valve 8 "to filter"	OPEN	Filter must be prepared as explained in section F. Hand timer to be set on filtration time. (F.i. 6 mins.)
c) Pump I	ON	
d) Press button "Precoat circuit"	OPEN	Filter powder is coated onto the filter elements.
e) Press button "Precoat circuit"	CLOSED	as soon as after approximately 1 minute clear solvent leaves the sight glass from the filter.
f) Valve 8 "to filter"	CLOSED	at the end of the filtration time.
g) Valve 4 "to tank I"	OPEN	Solvent is returned.
h) Wash motor	OFF	after 3o seconds pumping-off time.

i) Wash motor	ON	As soon as returning solvent to tank I drops off in quantity as observed in the tank sight glass.
k) Extraction	ON	5 - 7 seconds pre-extraction, then 2o seconds coasting, total extraction time 2 - 3 minutes.
l) Press button "brake"	ON	until washer returns to wash speed.
m) Valve 4 "to tank I"	CLOSED	
n) Pump I	OFF	

e) Rinsing

(Toggle switch "solvent level" stands on "medium")

1) Valve 2 "from/to tank IV"	OPEN	
a n d valve 5 "from button trap to pump"	CLOSED	
2) Pump I	ON	Rinse solvent is pumped from tank IV into the washer
3) Valve 9 "from pump to washer"	OPEN	
4) Set hand timer to rinsing time (f.i. 5 minutes)		Valve 2 closes automatically as soon as the required solvent level is reached. (See level control). With insufficient solvent quantities in tank IV, additional solvent must be let down from tank III and valve 2 "from/to tank IV" must be closed.

5) Valve 5 "from button trap to pump"	OPEN	
If necessary, dry cleaning aid and water can be added through the soap addition funnel.		
6) Valve 9 "from pump to washer"	CLOSED	at the end of the adjusted time.

f) Extraction

1) Valve 4 "to tank I"	OPEN	Solvent is pumped into tank I.
2) As soon as washer is empty, extracter	ON	5 - 7 seconds extraction, then 2o seconds coasting; total extraction time 2 - 3 minutes.
3) Extraction	OFF	
4) Press button "brake"	ON	until washer returns to wash speed.
5) Valve 4 "to tank I"	CLOSED	
6) Pump I	OFF	

g) With three bath methods, as f.i. subsequent water-proofing, the following operations have to precede the final drying process:

1) Valve 3 "from/to tank II"	OPEN	Proofing.
2) Valve 9 "from pump to washer"	OPEN	

3) Pump I	ON	
4) Wash motor	ON	
5) Valve 5 "from button trap to pump"	CLOSED	Tank II is drained through the pump circuit directly into the washer.
Valve 3 "from/to tank II"	CLOSED	after the normal solvent level has been reached.
Valve 5	OPEN	
6) Valve 9 "from pump to washer"	CLOSED	at the end of the proofing time (f.i. 3 minutes.)
7) Pump	OFF	
8) Valve 3 "from/to tank II"	OPEN	Solvent draingge begins as soon as valve 3 is operated.
9) Extractor motor	ON	5 - 7 seconds extraction, and 2o seconds coasting; extraction time 1 - 2 minutes.
1o) Extractor motor	OFF	
11) Press button "brake"	ON	until the machine has returned to wash speed.
12) Valve 3	CLOSED	

h) Drying

1) Spray jet - if required -	ON	The spray jet is fed with condensate. Cooling water regulator to be adjusted to a cooling water outlet temperature of 3o - 32°C. After-cooler temperature not to exceed 35° C.

2)Fan	ON	Adjust hand timer to desired drying time, f.i. 1o minutes.
3) Thermostat "before cooler" adjust to the desired maximum drying temperature o r thermostat II for low temperature drying (operate toggle switch on control panel).		By means of the air heater the air is brought to the temperatured adjusted on the thermostat. About the use of the thermostats see "Drying methods".

i) Deodorizing

1) Toggle switch "Heating/signal" down also toggle switch "spray jet" down	OFF	
2) After 1 minute lever "circulation/ deodorizing" to	DEODORIZING	Deodorize for 2 - 3 minutes.
3) Loading door	OPEN	Wash motor stops, fan switches on.

k) Unloading and loading

2) Desludging still I

With the described two bath methods the first bath is always distilled out in still I. At the end of this distillation the residue is pumped into still II and stripped by means of further steam distillation from the residual solvent. Since the pumping from one still to the other is incorporated into the automatic control, the following systematic method of operation should be adhered to:

During deodorizing the small still should be drained by opening of the drain valve. The drained liquid must not contain any more solvent. If necessary make adjustment to live steam.

After the load is finished, the machine is unloaded and loaded.

At the beginning of the next load the pump II operates automatically during the precleaning period. Its running time is adjustable and normally 3o seconds. The hot sludge is transferred into the small still. The distillation in still I should be almost complete at this time, which can be checked at the sight glass from the large condenser to the water separator.

The hot residue is now further distilled in still II by means of indirect heating and live steaming. This distillation must be complete by the time the machine deodorizes, i.e. in the sight glass from the small condenser to the water separator only water should be draining, and globules of solvent should no longer be visible in this sight glass.

3) Solvent level control

The machine is equipped with three level control switches. Their functions are:

a) Medium solvent level

This level control switch can be controlled by the program card as well as by the aid of a toggle switch at the front of the machine. The medium level is regulated through tank IV and is generally used in the two bath method with tank I and tank IV without filter.

b) High solvent level

This level control switch works in the same way as the medium level, but it is generally used only when cleaning from tank I via the filter.

c) Low solvent level

This level control switch can only be used via the program card. The switch controls the let-down valve from tank I, so that precleaning with or without filter at low dip is possible through this method of control.

A T T E N T I O N :

With insufficient solvent quantity in the tanks the missing solvent is always replenished from tank IV. It is important that this tank is always sufficiently filled.

It is replenished via the overflow from tank III. Tank III should, therefore, always be full.

Should the level control fail to operate properly, in particular should the solvent level rise too high in the machine, then it is certain that air is getting into the connecting pipe between the measuring points of the level control switch at the button trap and the switch itself. All connections should be stripped and remade with the use of suitable jointing materials.

4) SINGLE BATH method

Less soiled and less fatty goods can be processed according to the single bath method.

The sequence of operations is as under 1 a) to 1 c).

After the intermediate extraction as described under 1 d) the drying process follows immediately.

If the contamination of the solvent is only very slight, it is possible to return the solvent back to tank I and only distill the solvent which is extracted out of the material. For this purpose valve 1o "to still" is opened at the beginning of the extraction, and valve 4 "to tank I" closed.

After extraction valve 1o (to still) is closed again, and the drying process can follow as described under 1 h).

If tank IV is used for single bath cleaning, the solvent should not be returned to tank IV, otherwise soiling may settle in this base tank, which is difficult to remove.

Tank IV solvent should, therefore, always be returned to tank I or to still.

II) AUTOMATIC OPERATION

1) Loading -
close loading door.

2) Togglw switch "heating/signal" up.

Insert program card, start program card automatic control by pressing of the start button.
If any additives are used, these should now be poured into the soap additon funnel.

3) During the deodorizing approximately 2 - 3 minutes before the end of the cycle still II should be drained by opening of drain valve 3o.

This valve is preferably left open until the signal sounds; this insures that the sludge drains out as fully as possible.

Now valve 3o must be securely closed again.

All other functions are controlled by the automatic card control.

4) As soon as the signal sounds, switch toggle switch "heating/signal" down.

This silences the signal.

5) Open loading door -
unload machine.

Fan automatically starts and deodorizing damper opens.

6) Load -
close loading door.

7) According to type of work processed clean air filter bag and button trap.

8) Machines which have a filter fitted:

Every 8 - 1o loads the filter should be precoated. (See section H "filter").

E) O P E R A T I N G M E T H O D S

1) MACHINE EQUIPMENT

The machine is fitted with the following solvent tanks:

Cleaning tank	I	as side tank
Clean solvent tank	III	as side tank
Rinse tank	IV	as base tank
Aftertreatment tank	II	as base tank

The clean solvent tank is connected through an overflow pipe to the rinse tank for the automatic solvent addition. The solvent pump is arranged in such a way, that solvent can be delivered to and from all the tanks without the danger of intermixing.

The standard two bath equipment of the machine permits fully automatic operation of tank I and IV.

Machines which are equipped with three bath control can also be automatically operated on tank II.

In cases of extreme lint and soil accumulation it is recommended to have the special industrial button trap with vibrator fitted to the machine, which is an extra. The industrial button trap is a square container with a special sloping button trap insert. During the pumping off of the solvent from the machine, the whole button trap is vibrated which causes the deposites which cling on the sides of the button trap to peel off and collect in the bottom of the strainer.

On account of this self-cleaning effect all problems due to filter blockage are avoided.

When operating the machine by hand, the vibrator can be energized with the press button on the front of the machine. It should be operated as soon as the solvent level in the button trap begins to sink.

About filter additon see section H"filter".

If an additional filter is fitted to the machine, the valves "to filter" and "precoat circuit" are automated.

II) DESCRIPTION OF THE METHODS OF OPERATION

The following well-tried working methods can be performed with or without program card control.

1) Single bath method

Cleaning or degreasing from tank IV with medium level

Pumping off to still

Extraction

Drying

2) Two bath method

a) Cleaning or degreasing from tank I with medium level

Pumping off to still

Extraction

Rinsing from tank IV with medium level

Pumping off to tank I

Extraction

Drying

b) If instead of a rinse bath an aftertreatment bath is used:

Cleaning or degreasing from tank IV with medium level

Pumping off to still

Extraction

Aftertreatment from tank II (only with three bath automatic control)

Pumping off to tank II

Extraction

Drying

With the two bath automatic control one can also aftertreat from tank I.

3) Three bath method

Only for three bath machines

a) Cleaning or degreasing from tank I with medium level

Pumping off to still

Extraction

Rinsing from tank IV with medium level

Pumping off to tank I

Extraction

Aftertreatment from tank II

Pumping off to tank II

Extraction

Drying

b) It is possible to dry between extraction after rinsing prior to the aftertreatment, otherwise as 3 a).

4) Methods with filter

(only for machines with fitted filter)

Methods according to 1 and 2, with three bath automatic control also according to 3.

a) Single stage cleaning

Precleaning from tank I with low level

Pumping off to still

Extraction

Cleaning via filter from tank I with high level, solvent addition from tank IV automatically

Pumping off to tank I

Extraction

Drying

b) Three stage cleaning

Precleaning from tank I with low level

Pumping off to still,

Extraction

Cleaning via filter from tank I with high level, (prefiltration), solvent additon from tank IV automatically,

Cleaning without filter with dry cleaning aid and water addition

Clarification via the filter

Pumping off to tank I
Extraction
Drying

c) With three bath automatic control methods according to 4 a) or 4 b), however before drying:

Aftertreatment from tank II
Drain to tank II
Extraction
Drying

Note :

It is possible to stop the wash motor intermittently during the cleaning, degreasing, rinsing or aftertreatment. This is important when processing delicate work and for the pumping off prior to extraction.

III) SELECTION OF THE RIGHT PROCESSING METHOD

The following explanations are designed to help in the selection of the correct processing method. These suggestions can of course be varied according to special conditions.

1) Single bath method

For the cleaning and degreasing of not so heavily soiled or not so greasy goods this is the most rational method.

The additon of dry cleaning aid and water to the bath is possible. The whole bath is pumped to still and is available fully cleaned up for the next load. Of course with each load only a proportion of the solvent can be distilled; in this case one returns only the drainings to the tank and pumps the extraction liquid to the still.

2) Two bath method

More seriously soiled and very greasy goods should always be processed according to the two bath method. This is the mostly used process.

The precleaning bath is stilled, and additions of dry cleaning aid water increase the cleaning effect.

Since the rinsing bath becomes the precleaning bath for the next load, one uses the dry cleaning aid twice, if it is added to the rinse.

3) Two bath method with filter

If in respect to the quality of the cleaned goods, particularly industrial overalls extremely high demands are made, to the complete absence of fibre dust, then the use of a filter is unavoidable.

The precleaning bath is sent to the still, and the second bath is then filtered in a one stage or three stage method.

The single stage method employs the filter during the total cleaning process, in the three stage method prefiltration is followed by a stationary dip with water and detergent addition, which again is followed by clarification over the filter in the third stage.

4) Three bath method

This is used if an aftertreatment is required, f.i. for the lickering, sizing or proofing of various kinds of material.

5) Drying methods

a) Normal drying with thermostat I "before cooler"

This thermostat is set to 6o° C. The normal drying time with textiles is approximately 1o minutes.

b) Low temperature drying with thermostat II, expecially for leather and skins

In this case thermostat II is set for the most suitable temperature for the particular type of load.

A T T E N T I O N :

With hand operation one should note, that when employing thermostat II the drying time must be prolonged by about 4 minutes.

With hand operation the thermostats are selected by means of the toggle switch "thermostat I/II".

6) Drying controller

This is an optional extra which can be supplied with the cleaning machine, in order to automatically control the length of the drying time.

This drying time controller ensures that drying is continued until the solvent has been removed from the goods down to a level which is predetermined and which ensures most economic operation of the machine.

IV) PROGRAM CARDS

	Order No.	SE 1/18C	SE 2/18C	SE 3/18C	SE 4/18C	SE 5/18C	SE 6/18C	R 1/18C	R 14/18C	R 15/18C	R 16/18C	R 17/18C	Machine equipment	Process
First bath	from tank I		3	5	5		3	8x)	4	3	4	4	2 bath automatic	Cleaning and/or degreasing
	from tank IV	6				6								
Extract	to still	3	2	2	2	2	2		2	2	2	2	2 bath automatic	Cleaning and/or degreasing, rinsing
Second bath	from tank I												2 bath automatic	Cleaning and/or degreasing, rinsing
	from tank IV		5	5	8	5	5		6x)	5x)	6x)	10xx)	3 bath automatic	Cleaning and/or degreasing, aftertreating
Extract	to tank I		3	3	3		2	0,5	3	3	2	2	2 bath automatic with filter	cleaning over filter
	TO Still							2						
After-treat	from tank II					3	3				3	3	3 bath automatic	Cleaning and/or degreasing, aftertreating
Extract	to tank II					2	2				2	2	3 bath automatic with filter	Precleaning, cleaning over filter, aftertreatment

x) During this time the solvent is filtered.

xx) 3 mins. prefiltration,
4 mins. cleaning (rinse circuit)
3 mins. filtering clear

V) SUPPLIED PROGRAM CARDS

The program cards are identified by a code and an ordering number. Please, see program card table.

For the most generally used methods prepared program cards are supplied with the machine. Should you, however, request a special program card, would you please pass this on to the local PERMAC service organisation, together with the number of the machine and the exact timing which you prefer.

A corresponding program card will be cut for you with pleasure.

We would also ask you not to make any changes to the program card yourself; this work will be done for you by us.

1) Two bath automatic control - standard equipment

SE 1/18 C, SE 2/18 C, SE 3/18 C SE 4/18 C

2) Two bath automatic control with filter

SE 1/18 C, SE 2/18 C, SE 3/18 C, SE 4/18 C,
R 14/18 C, R 15/18 C

3) Three bath automatic control

SE 1/18 C, SE 2/18 C, SE 3/18 C, SE 4/18 C
SE 6/18 C

4) Three bath automatic control with filter

SE 1/18 C, SE 2/18 C, SE 3/18 C, SE 4/18 C,
SE 6/18 C, R 14/18 C, R 15/18 C, R 16/18 C

VI) PROGRAM CARDS IN STOCK

SE 5/18 C, R 1/18 C, R 17/18 C

F) OPERATING INSTRUCTIONS FOR PERMAC BÖWE COMPUTOMATIC PROGRAM CARD CONTROLLERS - FOR THE MODEL "PERMAC BÖWE SE 120 me"

1) PURPOSE

The program card control on the PERMAC BÖWE dry cleaning machines automatically operates motors and valves either electrically or electro-pneumatically, while operating sequence and timing are still individually adjustable.

2) DESCRIPTION

The controller is mounted with its timing gear into the front of the cleaning machine and can be easily removed for service. For its operation it has a large red illuminated push bar on the left hand side of the card which acts as a clutch and serves for the entering and removal of the program card.

Beside this red bar is a slot for inserting the program card; underneath the controller is a start button for starting the automatic program.

3) OPERATION

The COMPUTOMATIC program card controller controls the automatic functions of the cleaning machine when the main switch has been turned to "AUTO".

1) The program card is pushed into the slot of the controller; the ribs pointing to the left, while the push bar is simultaneously depressed.

   Before operating the start button the card should be slightly pulled outwards in order to determine whether the gear wheel for the transport of the card has engaged.

   Do not overlook this as it can lead to a faulty operation of the process at the beginning of the cycle.

2) The automatic cycle starts immediately after the start button has been pressed. The red push bar is illuminated and indicates the functioning of the COMPUTOMATIC program card controller. Alternately the start button is illuminated.

   The program card travels slowly outwards from the controller and the elapsed processing time can be read off on the timing scale of the card. These times are, however, only reliable up to the beginning of the drying process, since the synchronous motor of the control is intermittently interrupted during drying.

3) At the end of the atomatic cycle a signal sounds and then the program card stops. The card can only be pushed back into the controller or removed from the controller and exchanged for a new card while pressing the push bar.

A T T E N T I O N :

Neger try to move the card by hand without pressing the red push bar, otherwise the controller will be damaged!

G) GENERAL TECHNICAL DESCRIPTION OF THE AUTOMATIC PROGRAM CARD CONTROL SYSTEM

The reliability of the planned program of work as laid down by the individual program cards guarantees a uniform quality of cleaning with the most economical use of labour and materials. With individual cleaning processes evaluated by experience, separate program cards provide for their automatic control. Special cleaning processes not used very frequently are usually controlled by hand.

1) DRYING CONTROL

The wash, extraction and aftertreatment times are always constant and fixed by the program card.

The drying time, however, is controlled fully automatically, i.e. it may sometimes be longer or shorter.

The automatic drying control operates as follows:

The program card travels from the start of the automatic operation until the drying time begins with the start of the fan. At this very moment the card stops. The automatic functions, however, which have been initiated by the card, continue during this standstill period.

During the drying process the air temperature in the machine rises and is indicated on the thermostat "before cooler".

Normally the thermostat "before cooler" will be set to 60° C (140° F) and the fixed after-drying time will be 2 minutes. Therefore, the time required to reach the adjusted maximum drying temperature is fully variable because it is independent of hot air inlet temperatures (steam pressure), cooling conditions and weight of work loaded.

As soon as the adjusted maximum "before cooler" temperature is reached, the synchronous motor of the program card control is re-started by the temperature control gauge. The controller now continues to operate for the full length of the cut-out after-drying time and switches the machine off as usual.

The hot air inlet thermostat serves for low-temperature drying and will normally be set to an air inlet temperature of 60° C (140° F). Thereby a maximum temperature of 40 - 45° C (104 - 113° F) will be maintained for the "before cooler" thermostat and the articles being cleaned. With the machine operating by automatic control, the program card selects the suitable thermostat and thus controls the drying temperature. The drying time control is provided by the program card exclusively. In case of manual control of the machine and low-temperature drying with thermostat II, the toggle switch on the front control panel has to be switched from thermostat I to thermostat II.

Machines equipped with automatic drying controller: The drying time is regulated by this controller together with the thermostats to avoid shutting down of the machine before the end of the cycle. The drying time controller is set in such a way that it transmits an impulse to the program card controller by closing its microswitch two minutes prior to the desired end of drying. The remaining two minutes of drying time are controlled by the program card.

2) DISTRIBUTION OF CONTROL FUNCTIONS ON THE PROGRAM CARD

The program card has eleven ribs which are numbered from 1 to 6 and 8 to 12. Rib 7 is serrated and serves for the transport of the card through the program controller. The individual ribs or tracks run along a timing scale and by cutting-out of a portion of the tracks, microswitches which ride along the individual tracks are operated and the desired electrical functions initiated.

With several functions it is necessary to use not only one track, but simultaneously with it a second or third one. Through this combination alternative functions are initiated. Details are indicated in the wiring diagram.

The following list gives detailed information about the individual functions of the control tracks. The supplied wiring diagram enables an electrician to follow up the processes electrically.

Track 1: Valve 8 "to filter"
track 1 and track 11: extractor

Track 2: Pump

Track 3: Valve 1 "from tank I"
track 3 and track 11: valve 4 "to tank I"
track 3 and track 12: signal

Track 4: Valve 2 "from/to tank IV"

Track 5: Valve 3 "from/to tank II"
track 5 and track 12: deodorizing damper
(= lever "Circulation/Deodorizing")
track 5 and track 3: "low dip level"
(from tank I)

Track 6: Automatic control

Track 7: Card transport track

Track 8: Wash motor
track 8 and track 12: heating

Track 9: Track 9 and track 12: spray jet
track 9 and track 1: "precoat circuit"

Track 1o: Valve 11 "soap funnel"
track 1o and track 11: valve 1o "to still"

Track 11: Change-over relay

Track 12: Fan
track 12 and track 3 or track 4 or track 5:
valve 15 "from tank III"

3) CONTROL BOX

The electrical controls are mounted in a central control box on the left hand side panel of the cleaning machine, while the necessary switches are mounted on the front of the machine and are easily accessible.

A T T E N T I O N :

Do not open the control box before switching off the main isolating switch.

H) THE PERMAC BÖWE EXPANDER FILTER

1) GENERAL NOTES

For certain goods an additional clarification of the solvent by filtration is necessary during the cleaning process. For this reason PERMAC BÖWE S^{L} machines are prepared from the work for the additional installation of a properly dimensioned filter.

When a filter is fitted, the following installation work has to be done:

Besides the pipe work from the pump to the filter the pipes for the filter solvent to the washer and to the button trap as well as the connection between the filter outlet and the still have to be made.

In this method of installation the principle of normal dry cleaning has fully preserved, however the machine then offers the possibility of processing excessively soiled solvent through distillation at a much higher rate than usual in standard dry cleaning.

The filter with its automatic valves is incorporated in the lay-out of the automatic control, and its operation is therefore fully automatic.

2) DESCRIPTION OF THE FILTER

The PERMAC BÖWE Expander Filter consists of

the filter housing which in its conical part has a drain valve through which the filter is connected to the still;

The filter lid with pneumatic ram;

The filter element supporting plate which is mounted between the filter housing and the filter lid;

The Expander Filter elements which are screwed into the filter element supporting plate.

The pneumatic ram which is mounted on the lid is connected to a steel plate which presses on to the extension rods of the filter elements when operated. The filter elements themselves consist of a brass tube with a threaded head piece and the stainless steel wire element. The wire element can be stretched by the depression of the extension rod so that by means of this movement the filter cake which normally clings to the wire element is dislodged. Dual solvent entry is provided so that one part of the entering solvent is directed into the cone and the other just underneath the filter element supporting plate.

This solvent distribution ensures the agitation of the filter powder and prevents the deposition of lint under the filter element supporting plate. A filter pressure gauge mounted on the front panel serves to indicate the condition of the filter.

3) PRECOATING

The PERMAC BÖWE Expander Filter is a precoat filter which can be used with all good filter powders which are used in dry cleaning. It works according to the single charge/multi charge principle which means that a new precoat is formed on each new load, whereas the quantity of filter powder which is sufficient for several loads, is deposited in the filter in advance.

The clean filter is precoated via the button trap with the following quantities of filter powder after the filter has been filled with clean solvent:

Approximately 9 - 11 lbs. of filter powder.

This filter aid is moved to the filter by the pump via the precoat circuit within 1 - 2 minutes.

Now the pump can be stopped and the machine is prepared for cleaning. The precoat quantity for filter aid is sufficient with normal soiling of the garments for a maximum of 8 - 1o loads. If the soiling of the garments is less, the filter may be used even longer.

4) METHOD OF OPERATION

At the beginning of the cleaning process the filter powder which is already in the filter, is deposited on the elements and the solvent is, therefore, continuously filtered.

During the cleaning time the filter pressure increases from its initial value which, with a clean filter, should vary between 9 and 1o PSI, to 15 or even 2o PSI according to the degree of soiling and to the amount of moisture added to the load.

When the cleaning cycle is completed and the machine has finished extraction, the pneumatic ram on the Expander Filter is actuated during the slowing-down period which causes the dislodging of the filter cake from the elements.

For the hand operation of the filter shocking mechanism, the "filter shocking" button on the panel should be pressed. This, however, need not be done during the load, but preferably prior to starting the next load. In any case the button should be operated 2 - 3 times at the beginning of each new cleaning load.

With the cleaning of further loads the ratio of soil to filter powder in the precoat varies and the initial pressure on the filter pressure gauge will gradually rise. With normal work, however, this pressure will not exceed approximately 15 PSI if 8 - 1o loads have been processed with the above mentioned precoat, whereas, the final pressure at the end of filtration may quite easily reach 26 PSI.

If an initial pressure of 15 PSI is reached before the tenth load, then the filter must be let down completely, refilled and the precoat renewed.

In practice the following method of working is, therefore, recommended:

Precoating with the recommended quantity for the first load;

cleaning 8 - 1o loads without intermediate filter powder additions;

completely draining the filter;

refilling the filter with fresh solvent;

renew precoat.

If it can be estimated that only a small quantity of loads has to be processed in the second half of the working da, then the filter powder quantity for those loads can be reduced by approximately half.

In practice it has proved advantageous to use only the smaller quantity of filter powder of 9 lbs. for precoating, and dump the filter every 8 loads. It is important that the filter is dropped fully at least every 15 - 2o loads, so that all insoluble particles which are lighter than the solvent and which tend to float in the top of the filter are thoroughly removed.

A T T E N T I O N :

Never leave the dirty filter standing over night with the candles shocked. If you do not want to dump the filter, the filter should be recoated for at least two or three minutes, then the pump can be switched off. If these instructions are not observed, filter blockages will arise sooner or later.

5) DESLUDGING THE FILTER

Make sure that the still is empty. Having finished the last load, let down solvent from tank I to the cage, and then open valve 8 "to filter", valves 5 and 7 "precoat circuit" by pressing the buttons; valve 6 "machine circuit" then remains closed. Then put main switch to "HAND" position and start pump, thereby precoating the filter. 2 - 3 minutes later, after having switched off the pump, press push button "brake/filter shocking" several times.

Now open filter let-down valve completely for about one minute.

It is recommended to refill the filter immediately, and if the filter has been drained off during the day, it should also be precoated again.

FILLING AND PRECOATING

a)	Main switch to	HAND	
b)	Valve 15 "from clean solvent Tank III"	OPEN	Solvent flows into cage.
c)	Valve 5 "from button trap to pump"	OPEN	
d)	Valve 8 "to filter"	OPEN	
e)	Press push button "precoat circuit open"		
f)	Pump	ON	Filter is filled. Add filter powder to button trap.

As soon as filter inlet and outlet sight glass is clear, the precoat circuit is ended.

g)	"Precoat circuit"	CLOSED
h)	Pump	OFF

If the filter is only filled with solvent, no filter powder addition will be necessary.

R e m a r k :

The filter may also be filled with solvent from rinse tank IV. In this case valve 2 "from/to tank IV" should be opened.

As soon as the filter is filled, valve 2 is closed again, and operation continues as above.

6) REMOVAL OF FILTER ELEMENTS

Should it be noticed that the filter cannot be properly desludged despite of having been carefully complied with the operation instructions - shown by an early filter pressure rise, the filter must be opened for inspection and cleaning.

For this purpose the individual filter elements must be unscrewed with a special spanner after the removal of the filter lid, and the filter element plate taken off for cleaning out the filter vessel.

Lightly brush the elements as soon as they are dry, with the nylon brush provided.

Should it prove to be impossible to remove the filter elements because of excessive build-up of residues inside the filter, they should be unscrewed just enough to free the circlip on the neck of the filter elements which can then be taken off.

Screw the filter elements clockwise into the filter element plate until they drop off inside. If necessary, the filter element plate can first be lifted with little wedges by about 2". Before the filter elements are replaced, the circlip must be refitted. The elements should be screwed into the filter element plate only hand-tight.

The filter can only be heavily blocked by incorrect operation or through wrong methods of water addition to the solvent. If in doubt, please contact our service department

7) THE USE OF ACTIVE CARBON POWDERS

The regular use of active carbon powders is recommended in order to avoid colourizing of the solvent. The addition should be made with the first precoat. An intermediate addition of active carbon powder is, therefore, not necessary. Should, however, active carbon powder be intermediately added, it must be mixed with equal portions of filter powder.

With hand operation it must be observed that at the beginning of the load the precoat is made via the precoat circuit, since otherwise active carbon may contact the garments.

I) DISTILLATION AND COOLING WATER REGULATION

1) GENERAL

The PERMAC BÖWE SE machines have two stills, the lower still I is so dimensioned that it can accommodate a complete bath of solvent. Still I must be heated all day long with indirect steam, and in order to ensure a constantly high rate of distillation at the end of each load the soil residues are drawn off to still II which is situated above still I. This enables the operator to remove the residues arising from the cleaning process without interruption from load to load.

In the following notes particular points are discussed which deserve careful attention.

ATTENTION :

The stills should never be filled above the lower edge of the still sight glass. If necessary, the drain cock on the front of the still should be used to remove some solvent from the still. Overfilling leads to boiling-over and the vapour pipe and the condenser will become soiled.

2) OPERATION OF STILL I

The still is fitted with three steam connections of which the two lower ones go to the immersion heaters, the middle one for the jacket heating and the top one for the direct or live steam.

For distillation the steam valve for the jacket heating and for the immersion heaters are opened. The factory side water valve should be opened to allow the automatic cooling water regulating valves to adjust themselves to the right water flow.

Should the solvent in the still present foaming problems and boil over at time, then this can be due to insufficient pumping off of residues from the still, or irregular stripping of still II. If foaming still persists, then suitable anti-foaming agents should be added. Addresses of suppliers will be furnished on request.

A T T E N T I O N :

The live steam valve must under no circumstances be opened as long as liquid perchlorethylene is contained in the still.

Only when with indirect heating no more solvent flows through the sight glass to the clean solvent tank, the live steam valve may be opened gradually. At the same time open the by-pass water valve fully. When distilling wet solvent or when using live steam, the distillate becomes cloudy which is due to minute quantities of water. In course of time those minute quantities of water settle in the clean solvent tank. This tank is equipped with w ater separating device preventing the accumulated water from leaving the tank and from getting into the washer. The accumulated water must be drawn off at least once a week by the hand valve in the inspection plate of the clean solvent tank III, after this tank has been normally discharged to the washer. The solvent/water mixture is separated and the solvent put back into the still.

If this instruction is not observed, textile damage due to water may arise!

The built-in safety valve of the still opens at a pressure of approximately 7 PSI, whenever wrong operation of the still or insufficient maintenance should lead to distillation difficulties.

The safety valve must not be altered or opened. In case of leakage it must be exchanged.

For checking and cleaning the safety valve seat the valve piston should be lifted at least once a week by means of the lever on top of the valve.

The still is also fitted with a vacuum gauge with the operating range of -7 to +21 PSI. This gauge is marked with a red line at +o.5 kg/cm^2 (7PSI) indicating the point at which the safety valve will begin to open.

Particularly when starting the still the pressure should be checked on the pressure gauge. If the pressure rises above +7 PSI, the vapour pipe to the condenser and the condenser itself must be dismantled and cleaned. Any further use of the still at a registered pressure of +7 PSI and above is not permissible.

"STRIPPING" SOLVENT OUT OF SLUDGE RESIDUES

As soon as distillation ceases (see sight glass to clean solvent tank), the manual cooling water regulating valve (below the automatic cooling water regulating valves) should be completely opened and the live steam valve should be gradually opened while observing the still pressure gauge.

The live steam is used until solvent flow in the sight glass to the clean solvent tank ceases. This is normally the case after approximately 1o minutes of steaming. Under no circumstances may the live steam valve be used for normal distillation or even for faster heating up of the solvent in the still.

This would lead to a violent evaporation of solvent, to a boil-over and subsequent blockage of vapour pipe and condenser!

The sludge agitator should be put backward and forward several times in order to break up the residues. At the end of the distillation it should be left in such a position that the marking on the agitator shaft is in a horizontal position, otherwise it may be difficult the following morning to turn the arms of the agitator through the stiff residues.

At the end of distillation the live steam valve should be thoroughly closed. A partially open live steam valve will continuously produce cloudy distillate. The distillation residues should be removed through the cleaning-out door of the still by means of the supplied too

3) OPERATION OF STILL II

Just before the first cleaning bath is pumped into the still, sludge pump II starts and transfers the oily residues from still I to still II, where it is subjected to a continuous distillation with indirect heating and direct steam.

The adjustment of free steam and indirect heating should be such that the liquid residues are fully stripped off solvent at the end of each load. The liquid residue is then run off from the still during the deodorizing perod, and the drain cock should be left open for the whole two to three minutes.

The outlet pipe can be arranged to suit local requirements.

4) COOLING WATER ADJUSTMENTS AND OPERATION OF THE CONDENSER

The PERMAC BÖWE machines are equipped with a fully automatic cooling water system which guarantees minimum water usage and constant drying results with the lowest possible solvent consumption.

The distributing header for the cooling water control is fitted with three automatic cooling water regulating valves (air cooler and distillation condenser) and a manual control valve. The upper cooling water regulator has its sensing element in the water outlet of the air cooler, the lower one in the cooling water outlet of the condenser. Both regulators ensure that the outlet temperature of the cooling water of the two coolers is kept constant under normal conditions. The hand valve is used for an independent additional cooling of the solvent, when normal cooling of the solvent is insufficient (with live steam distillation and also for filter jacket cooling).

Make particularly sure that sufficient cooling water is available when using the immersion heaters or boosters. If necessary throttle steam valve to booster!

/cont

Adjustment of the valves:

a) Cooling water regulator of air cooler

Measure the water outlet temperature during drying. Adjust the valve until the outlet water has a temperature of 30 - 32° C (86 - 90° F). The valves are already adjusted at our works.

In the cooling water regulating valve of the air cooler a little by-pass is provided in the body of the valve permitting a very small quantity of water to pass through it continuously. This ensures quick valve opening and also continuous solvent temperature control.

The factory-side water isolating valve should be closed at the end of the working day.

b) Cooling water regulator for condenser

Measure the water outlet temperature during full distillation and adjust regulating valve until the prescribed temperature is maintained (also already adjusted at our works).

Maximum temperature at condenser I 45° C (113° F)
Maximum temperature at condenser II 35° C (95° F)

This value must be maintained by the valve even if the machine is simultaneously drying.

K) TIPS FOR THE CLEANER

1) DRY CLEANING AID

For the achievement of first-class cleaning results the use of good cleaning aids and water is advisable. Besides producing a good cleaning effect the dry cleaning aid should possess good filtration, good soil suspension and water carrying properties, it should not cause any corrosion and/or foam during distillation or interfere with the clarity of the distill ate.

The dry cleaning aid addition of 0.5 - 1 % on the weight of work in combination with twice to fourfold the quantity of water when cleaning of industrial overalls, machinery cleaning cloth and sheep skins has produced very good results.

With the degreasing of undied, white sheep skins the addition of a quantity of hydrogene peroxide together with the water has also proofed very beneficial.

The bleaching effect is very noticeable and very acceptable. In a two bath method these additions should be made during the rinse.

2) AFTERTREATMENT CHEMICALS

Waterproofing agents, retexturing agents, mothproofing agents etc. can be applied fully automatically in the cleaning machine and they should be used according to the instructions of their manufacturers. If necessary, one additional tank can be fitted to the cleaning machine for aftertreatment.

With goods which are to be waterproofed after cleaning, a rinse bath with distilled solvent from tank IV should follow the cleaning bath, since otherwise excessive amounts of cleaning aif, which act as a wetting agent, will remain in the textiles and reduce the efficiency of the proof.

3) PRETREATMENT OF GARMENTS

a) Cleaning of overalls

Prior to cleaning, pockets, trouser turnups etc. should be brushed out. This generally removes dust and dirt which on account of its position is sometimes not fully removed during the treatment in the cleaning machine.

The pockets of industrial overalls should be searched carefully for metallic objects like tools, bolts and nuts etc.

b) Machinery cleaning cloth

These can only be cleaned satisfactorily in solvent if they are not water wet. Wet machinery cleaning cloth must be predried in the machine. The cloth must not be contaminated with inflammable solvent.

There is objection to processing cloth which contain mineral spirits with boiling points below 150° C.

c) Fur and leather cleaning

The furs and leathers should be air-dried. Pickeled materials and such which contain acids must be neutralized prior to degreasing and dried, since otherwise corrosion will take place in the machine.

PERMAC BÖWE SE machines are corrosion protected as far as this is necessary. This protection is, however, based on the used of chemically neutral cleaning materials (ph value of a 1o % aqueous extract should be approximately 7).

The manufacturers will not beheld responsible if corrosion due to such materials arises, despite the fact that everything has been done in order to protect the most effected components as much as possible.

T E X T B O O K S

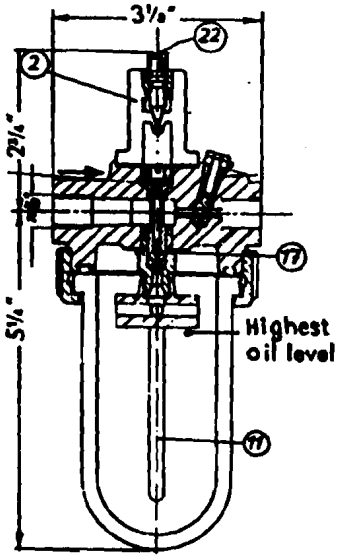
a) SILK FINISHING	by Jennie W. Maher, published by NATIONAL INSTITUTE OF DRY CLEANING, Silver Spring, Maryland, 1945
b) SPOTTING	by Judson C. Randlett and William J. Nicklaw, published by NATIONAL INSTITUTR OF DRY CLEANING, Silver Spring, Maryland, 1956
c) DRY CLEANING MANUAL OF CLEANING ROOM PRACTICE	by E. Roland Philipps, jr., published by NATIONAL INSTITUTE OF DRY CLEANING, Silver Spring, Maryland, 1961
d) APPLIED SCIENCE FOR DRY CLEANERS	by George P. Fulton, published by NATIONAL INSTITUTE OF DRY CLEANING, Silver Spring, Maryland, 1951
e) THE TECHNOLOGY OF DRY CLEANING	Published for the Guild of Dyers and Cleaners by DRY CLEANING AND DYEING JOURNAL, 8-1o, Temple Ave., London E.C. 4, 1962
f) DRY CLEANING TECHNOLOGY AND THEORY	by Albert R. Martin and George P. Fulton, NATIONAL INSTITUTE OF DRY CLEANING, Silver Spring, Maryland

I) MAINTENANCE

Daily according to type of work processed every 3 - 6 loads	Cleaning Lint filter in recovery section. Button trap insert. In order to remove the solvent from the residues in the button trap, these should be transferred into the provided bag, where they are allowed to accumulate. As soon as the bag is full, it is closed, removed and dried together with the textiles during the drying process. A special device is fitted to the industrial button trap for holding such a bag in position.	Lubrication All greasing points should be turned: Trunnion bearing 2 turns, pump and fan 1/2 turn, filter 1 turn. Do not forget to replenish the greasing points in good time. Only greases based on lithium shall be used, as f.i. "Beacon M 285" (Esso) "Alvania 3" (Shell) "Retinax A" (Shell)
Every 8 - 10 loads	For machines with fitted filter only: Drain filter and precoat.	
Weekly	Drain water separator , rinse with water, and refill at first with fresh solvent and then with water. Empty clean solvent tank to washer and drain remaining solvent/water mixture through hand valve at the inspection cover. Pour into funnel on the back of the still, check water accumulation in air water separator and fill micro oiler, if necessary.	

Cleaning	
	Lint at the entrance to the air filter ducting should be removed with the brush provided.
	Change air filter bag and clean in the machine.
	If soiling is excessive, wet clean.
Monthly	Open inspection cover to spray jet, check spray jet area for lint collection, clean if necessary. Check drain to water separator for good passage.
	Open inspection cover to air cooler and check for lint accumulation; if necessary, clean finns with steam gun and brush.
	Check seating of air filter. The driving belt to the washer should be tight enough, so that in the middle between driving and machine pulley the belt can be pushed in only by the thickness of one belt. Tighten up motor, if necessary.
	Check valves and connectors for leakages. Tighten valve glands and hose pipe connections if necessary.

	Cleaning	Lubrication
Half-yearly	Remove hand inspection covers to tanks and clean tanks. Remove lid to condensers and clean both condensers with water. Check air cooler and remove if necessary.	
Annually	Vapour pipe from still to condensers must be removed and cleaned. If the pressure gauge on the still shows a positive pressure approaching o.5 atm. then this vapour pipe must be cleaned immediately. With machines fitted with filter the elements should be removed and inspected. Also check filter vessel for deposits.	Oil change in wash motor gear box. Use SHELL gear oil SAE 14o or any other good quality motor gear oil of the same viscosity. Also observe lubricating instructions on motors. For the greasing of bearings only lithium based greases should be used.


3⅛"
22
2
2¼"
77
5⅛"
Highest
oil level
77

MAINTENANCE INSTRUCTIONS FOR COMPRESSED AIR WATER SEPARATOR AND MICRO OILER ÖNP-50

The PERMAC BÖWE dry cleaning machines are fitted in the compressed air supply line with a water separator and a micro oiler in order to protect the pneumatic rams from wear and in order to ensure their faultless operation. The collection of condensate is easily visible in the compressed air water separator and the vessel should be drained with the compressed air turned off when need arises.

The oiler supplies an oil mist through all the air lines so that oiling of individual compressed air components is no longer necessary.

Both, the water separator and the oiler require no additional maintenance apart from draining the water and refilling the oiler.

A T T E N T I O N :

Do not attend to either instrument with the compressed air still turned on!

Do not bring solvent into contact with the plastic components of the equipment, since this material will not withstand it.

Clean only with white spirit or petrol. Both, the water separator and the oiler are designed for a maximum pressure of 150 PSI and a maximum temperature of 160° F.

TROUBLE SHOOTING

1) Leaks: They influence the efficient formation of a reduced pressure in the oiler and prevent the supply of oil mist.
   Check all screws and threaded connections.
2) Oil does not drip: Clean outlet in the oil orifice.
3) Oil is not being drawn up into the ventury. Ventury or ventury housing clogged.
4) Leaks: Check gaskets.
5) Oil supply pipe loose: Screw in and seal with sealing compound. For the filling of the oiler any good quality machinery oil of low viscosity is suited.

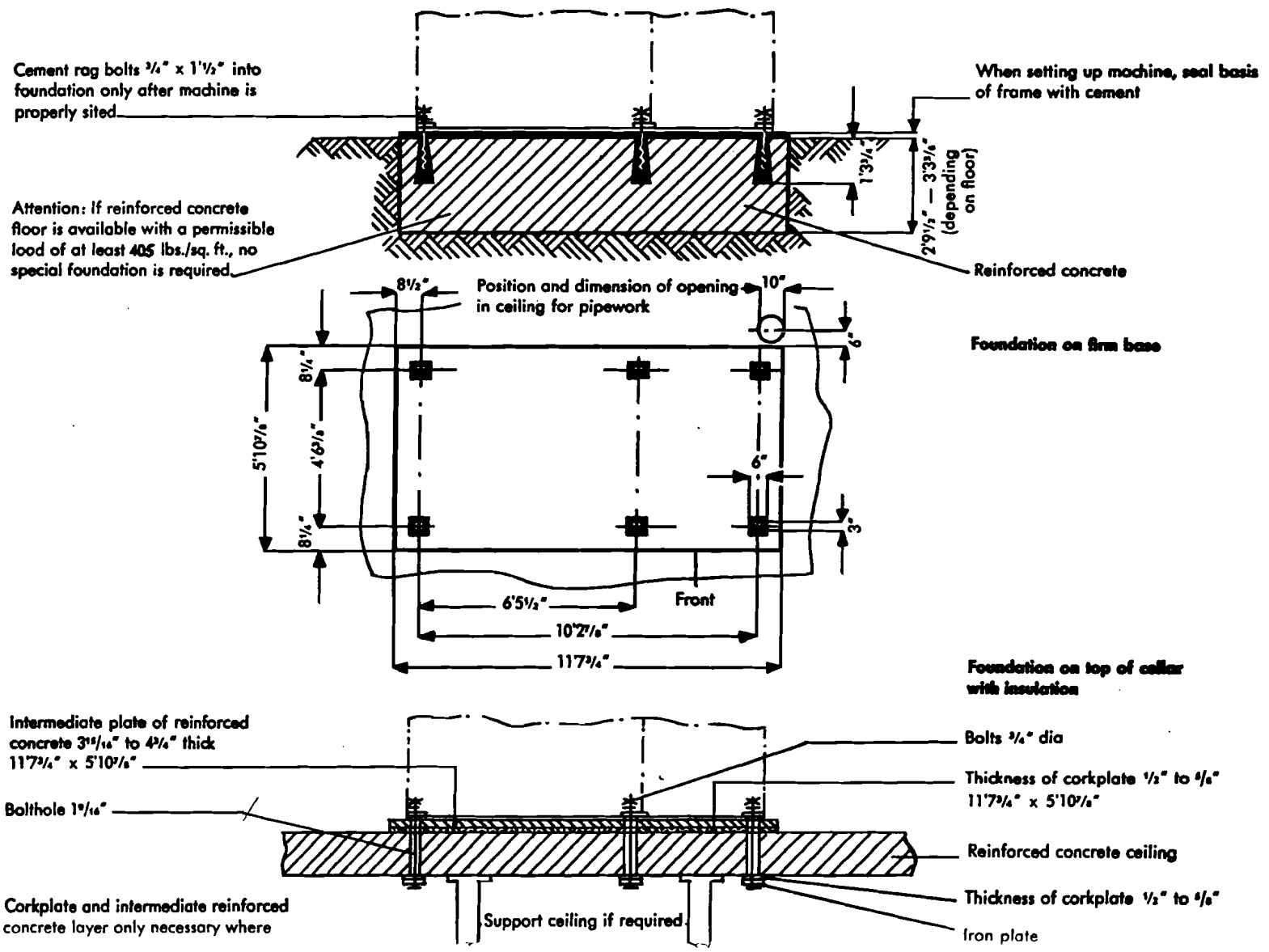
The oiler takes approximately 5 fl.ozs. of oil which should have a viscosity of 4 - 6° E at 5o° C (3o - 45 cSt at 12oo F). e.g. Tellus 33 (Shell), Esstic 5o (Esso).

Screw which serves to control the quantity of oil used, should be opened 3/4 - 1 turn with fully closed air by-pass.

This instruction manual is based on our practical experience and is meant to help and advise you in operating your machine, however, we cannot be held responsible for any possible faults that may occur dur to local operating conditions.

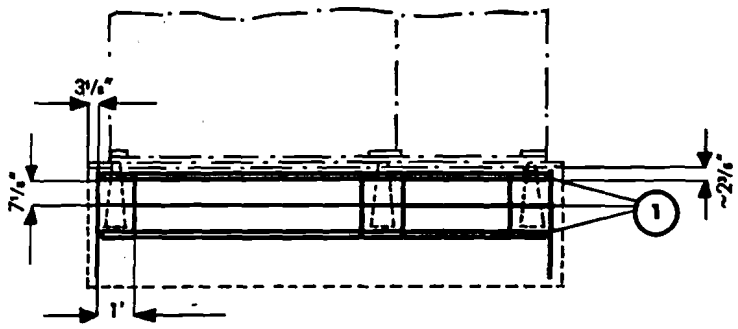
March 1965,
JHS/gr

Foundation for the Permac Böwe 300 me, SK 120 me, SE 120 me and W 120 me

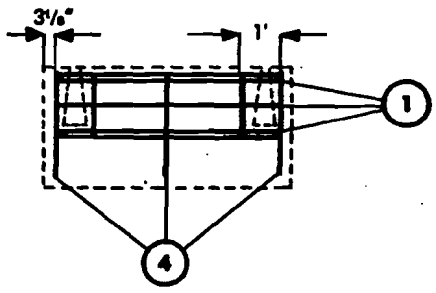


Scale 1:50

Reinforcing Ironwork for the Foundation of the Permac Böwe 300 me, SK 120 me, SE 120 me and W 120 me



Foundation on firm base

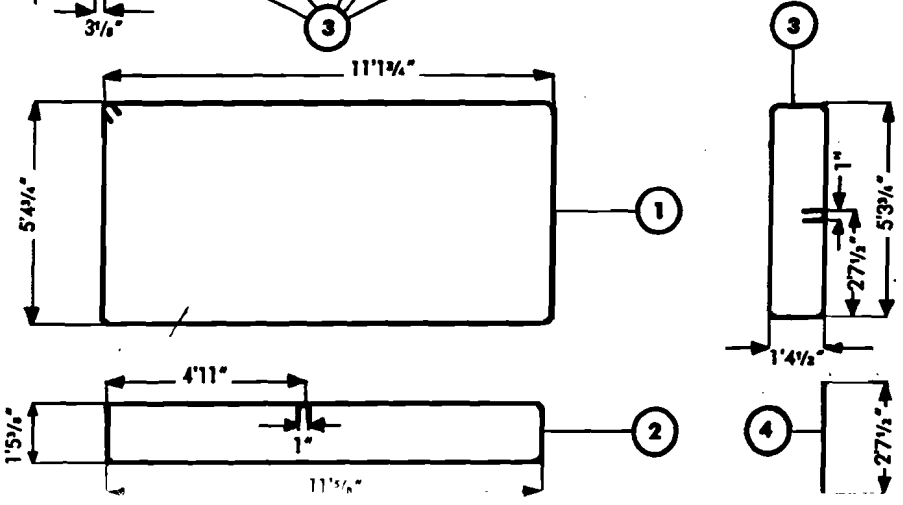


1 — 3 Round iron bars ½" dia, bent (concrete reinforcing bars)

2 — 3 Round iron bars ⅜" dia, bent (concrete reinforcing bars)

3 — 5 Round iron bars ⅜" dia, bent (concrete reinforcing bars)

4 — 6 Straight iron bars ½" dia (concrete reinforcing bars)

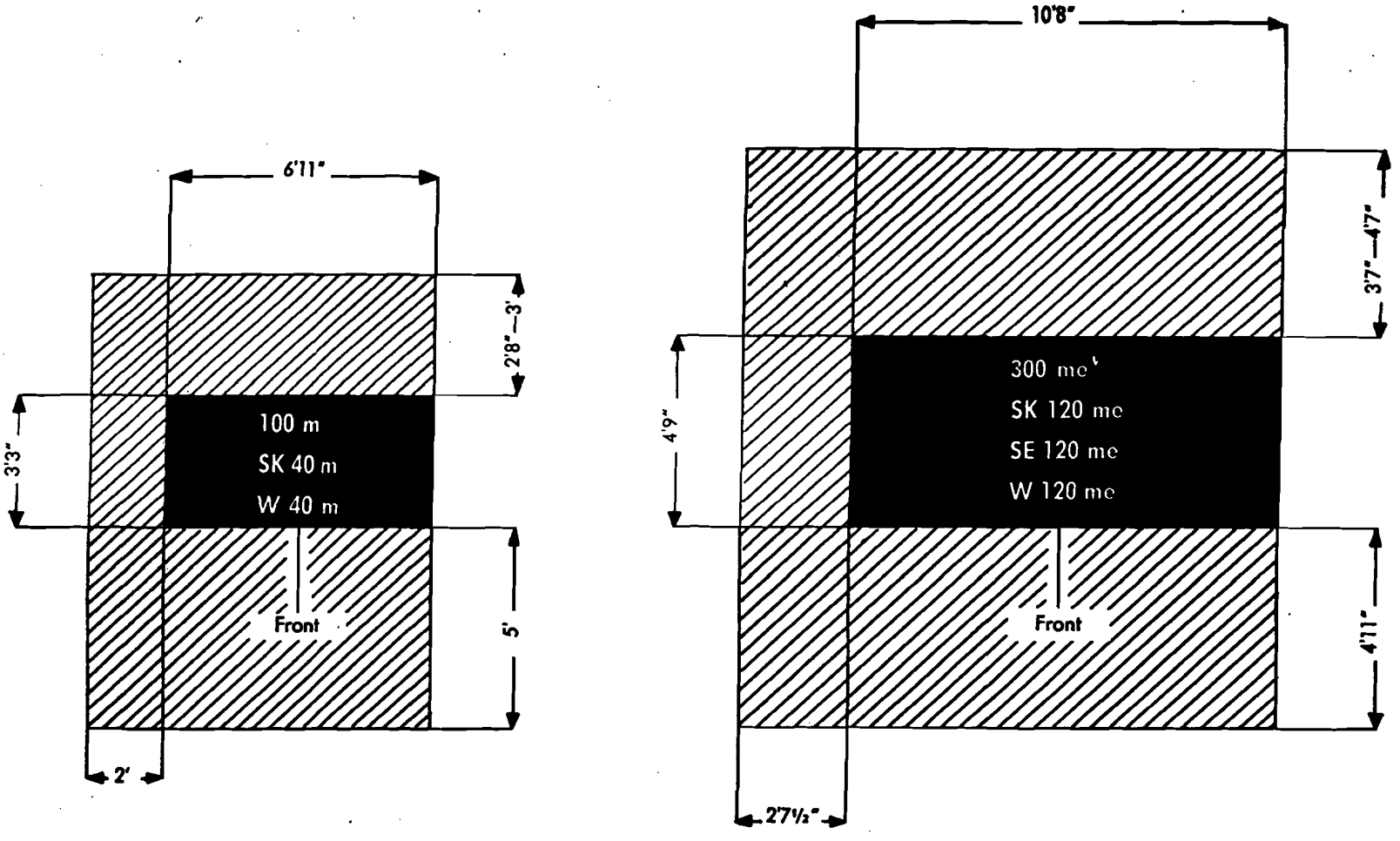


Adjust length of vertical rods according to depth of foundation

Scale 1:50

1.105.13 27

Service and Maintenance Area



Scale 1:50

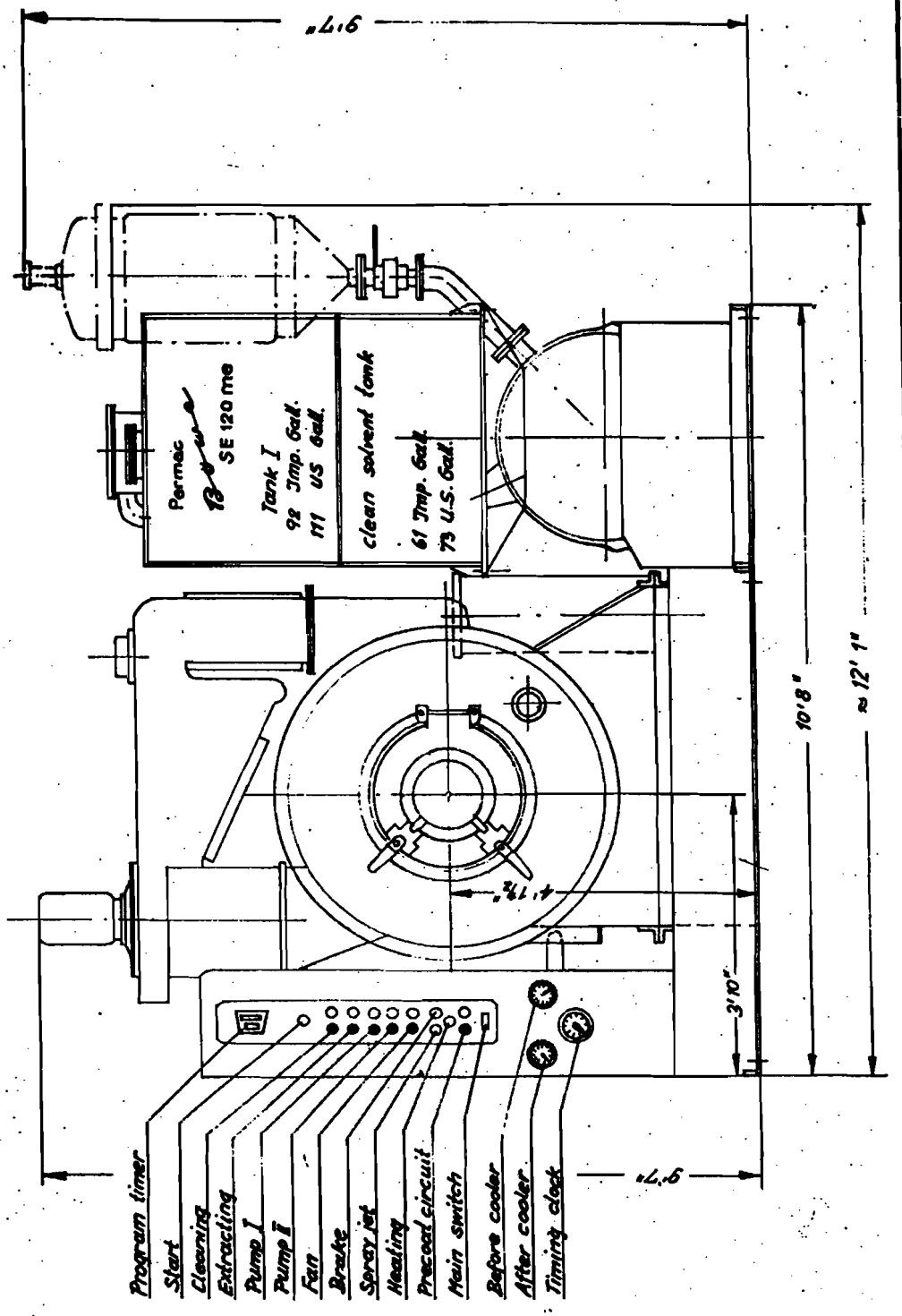
1 101.13—09

BÖHLER & WEBER KG
MASCHINENFABRIK
AUGSBURG

Front View of the Permac Böwe SE 120 me

1105.24-06

Maschinen-Nr. | Blatt
Ersatz für



	Datum	Gezeichnet	Geprüft	Werkstoff	Maßstab
	25.3.65	Wis			1 : 25

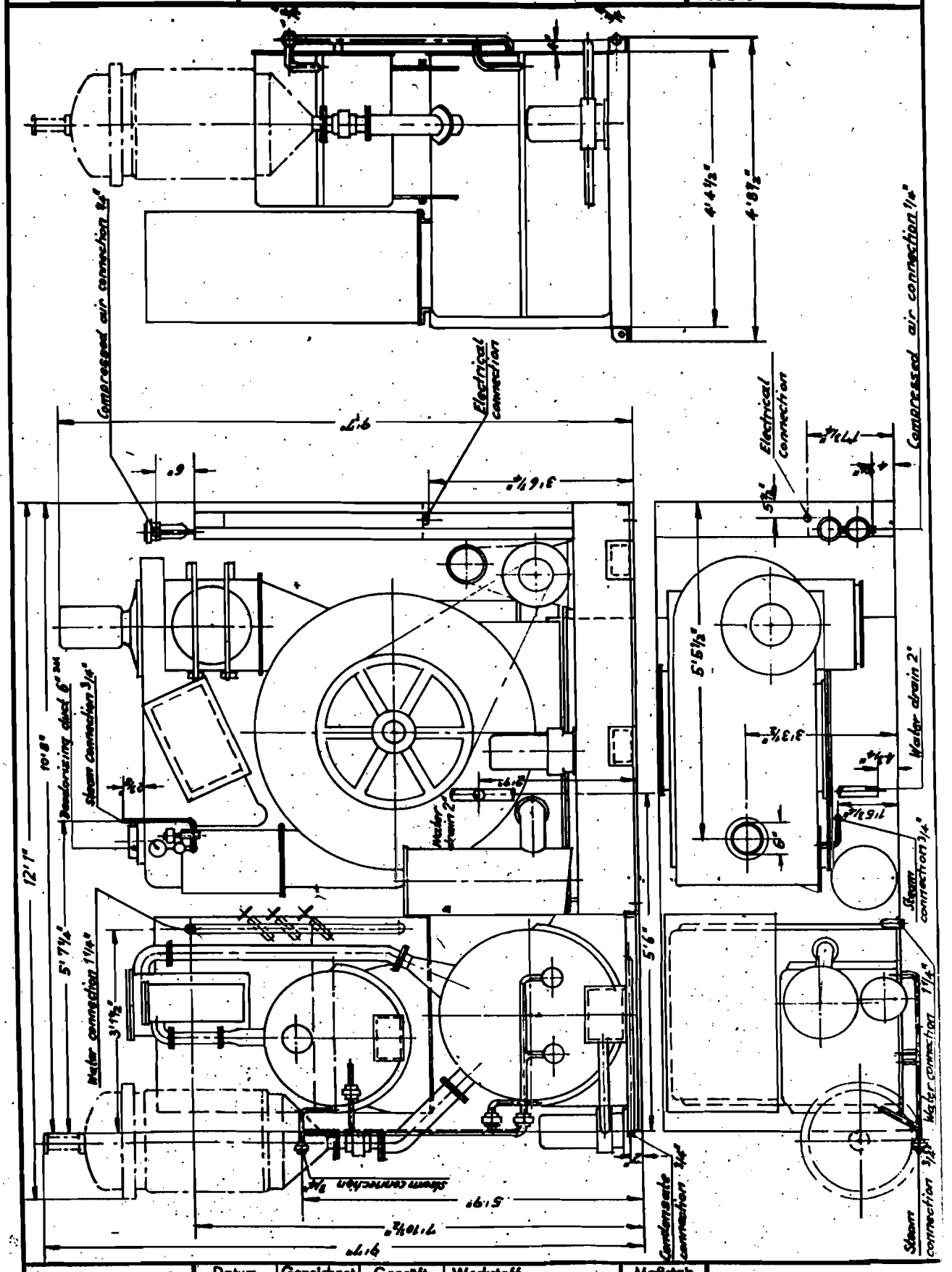
WN 004 401

BÖHLER & WEBER KG
MASCHINENFABRIK
AUGSBURG

Table of Dimensions of the Permac Böwe SE 120 me

1105,24-07

besteht aus 1 Blatt 1
Ersatz für



Datum	Gezeichnet	Geprüft	Werkstoff	Maßstab
25.2.65	Wi			

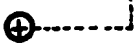
WN 004 401

SYMBOLS


Diaphragm valve


Valve (hand operated)


Reducing valve




Line strainer


Check valve


Steam trap


Sight glass

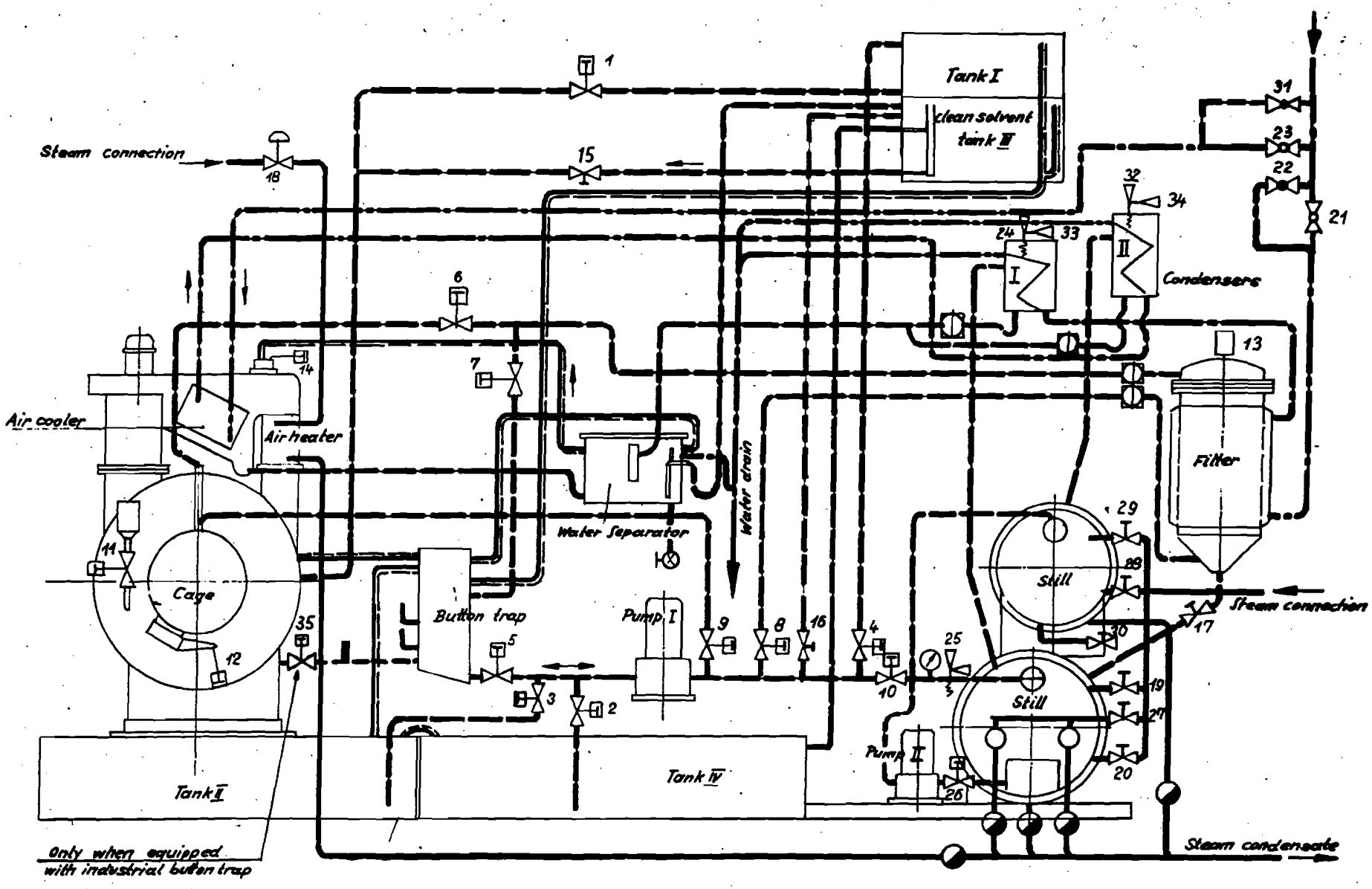

Pump


Valve (hand operated)


Valve (automated)


Pressure gauge

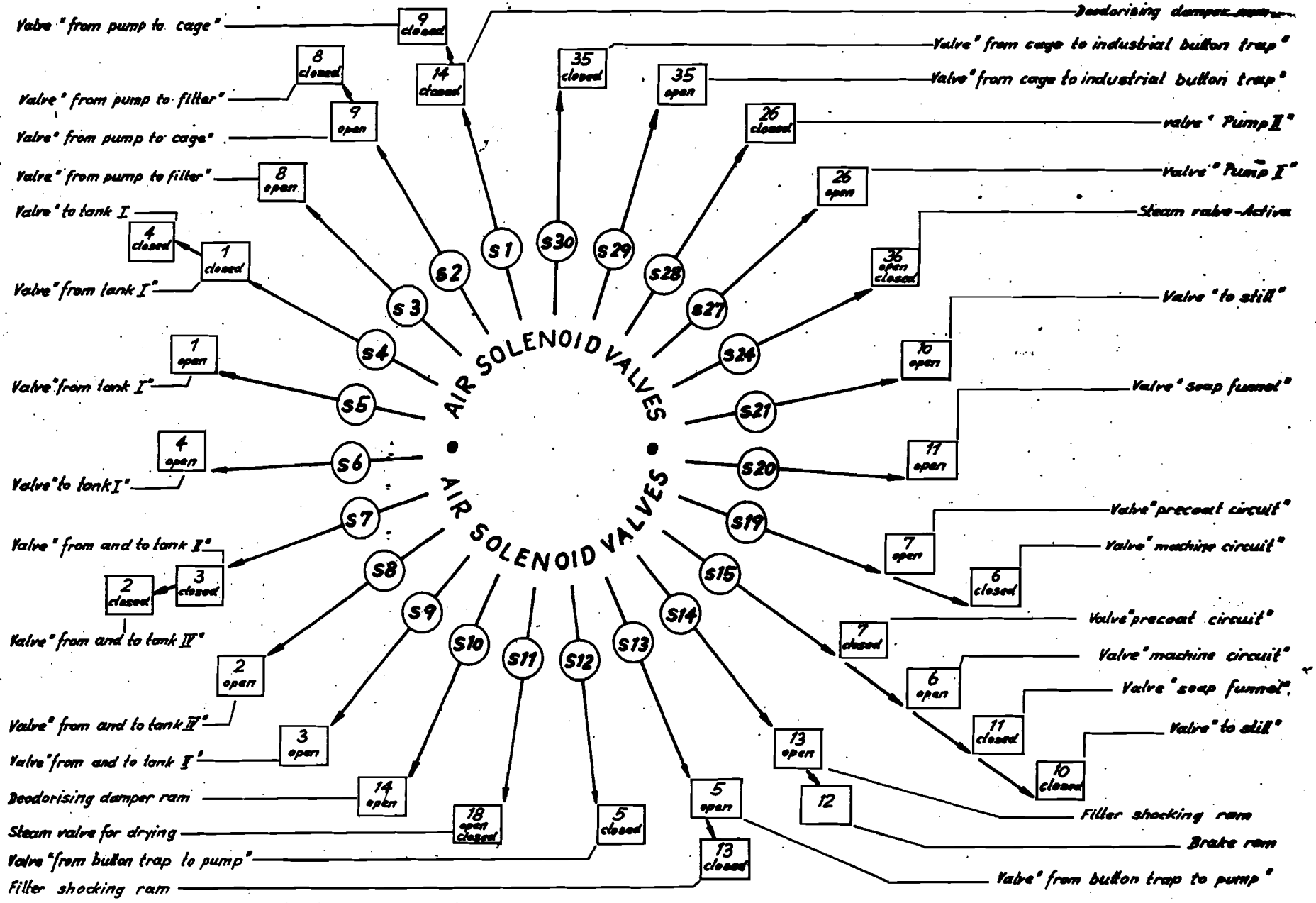

Spring-loaded angle relief valve



Piping Diagram of the Permac Böwe SE 120 me

Solvent Cooling Water Vent Steam Condensate

1 Valve "from tank I" (automatic)
2 Valve "from and to tank IV" (automatic)
3 Valve "from and to tank II" (automatic) - with three-bath automatic control only
4 Valve "to tank I" (automatic)
5 Valve "between button trap and pump" (automatic)
6 Valve "machine circuit" (automatic)
7 Valve "precoat circuit" (automatic) 6 and 7 only when equipped with filter
8 Valve "to filter" (automatic) only when equipped with filter
9 Valve "from pump to cage" (automatic)
10 Valve "to still" (automatic)
11 Valve "soap funnel"(automatic)
12 Brake (automatic)
13 "Filter shocking" (automatic)
14 "Deodorising damper" (automatic)
15 Valve "from clean solvent tank III"
16 Valve "to clean solvent tank III"
17 Filter drain valve
18 Steam valve for drying (automatic)
19 Hand operated valve for jacket heating from still I
20 Hand operated valve for live-steam to still I
21 Hand operated valve for cooling water
22 Cooling water regulating valve for condenser I
23 Cooling water regulating valve for air cooler
24 Angle relief valve for condenser I
25 Angle relief valve for still I
26 Valve "between still I and sludge pump" (automatic)
27 Steam valve for heater elements of still I
28 Hand operated valve for jacket heating from still II
29 Hand operated valve for live-steam to still II
30 Hand operated valve for draining still II
31 Cooling water regulating valve for condenser II
32 Angle relief valve for condenser II
33 Check valve for condenser I
34 Check valve for condenser II
35 Valve "between button trap and cage" (automatic) only when equipped with industrial button trap



Pneumatic Controls for the Permac Böwe SE 120me Three Bath Automatic control and Active

APPENDIX B

GROUNDWATER MONITORING WELL INSTALLATION PLAN

Appendix B - Groundwater Monitoring Well Installation Plan

One new monitoring well (MW3) will be installed at the SITE (Figure 4). This well will be screened in the Upper Glacial aquifer at approximately 30 - 40 feet below land surface.

As per USEPA guidance, the screened intervals of a shallow well will not be across the water table. The depth selected will be based on photoionization readings, or, if all readings for the particular well are the same, the screen will be set at 10 feet below the water table. During well installation, an assessment will be made to determine if floating product exists at the saturated/unsaturated zone interface. The method for accomplishing this is described below.

The monitoring wells will be drilled using a truck mounted hollow stem auger rig. Upon completion of the borehole, a 4-inch diameter PVC casing with a 10-foot long screen will be installed through the auger. When the screen and casing are in place, a clean, graded silica sand will be used to pack the annular space around the screen. The sand pack will be installed. A photoionization meter will be used to scan the split spoon samples for each well. MW3 will be logged continuously (24 inch intervals).

When the well screen has been properly sand packed, two feet of fine sand will be placed immediately over the filter pack and a five foot thick layer of clean, certified 100% bentonite high solids grout will be tremied onto the top of the fine sand to seal the annular space. The remainder of the annular space will then be grouted with a cement/bentonite slurry to two feet below grade. Well MW3 will be finished flush with grade, have a locking cap installed, and protective meter box cemented in place over the well. USEPA guidelines will be followed for all steps of well drilling and construction; drill cuttings will be stored on site and samples analyzed for storage and disposal

requirements prior to disposal. RCRA regulations will be followed (E.g., closed dumpster).

Upon completion, MW3 will be developed by surging and pumping to remove any fine sediment from around the screen zone and to establish a good hydraulic connection between the aquifer and well. Development will continue until the water is less than 50 nephelometric turbidity units (NTUs) or for one half hour , which ever comes first, as required by the NYSDEC.

Monitoring well (MW1, MW2 and MW3) locations and elevations will be surveyed by a New York State Licensed Land Surveyor to the nearest 0.01 feet with a closure of $\pm$ 0.05 feet for the SITE. The elevation measuring point will be marked on each well casing and all water level measurements will be referenced to this point. All elevations and depths, including well casings, will be referenced to mean sea level.

Water levels in all the wells will be measured using a steel tape at least three times; once after the development and prior to each of the ground-water sampling events. The initial water-level measurements will be taken at least two days after development. Information on the vertical hydraulic gradient will be provided from the three monitoring wells. The responsiveness of each well to water-level fluctuations in the aquifer will be tested by measuring recovery rates after pumping.

An attempt will be made to redevelop the two presently existing wells (MW1 and MW2). If these two wells can be redeveloped, they will be fitted with locking caps, and sampled during the first round of groundwater sampling.

Two weeks after the monitoring well has been installed and developed, groundwater will be sampled and analyzed following USEPA approved protocols to determine the quality of the

groundwater at the Site. The groundwater samples will be analyzed for the compounds on the Target Compound List (TCL). Analyses for Target Analyte List (TAL) parameters will be performed on unfiltered samples.

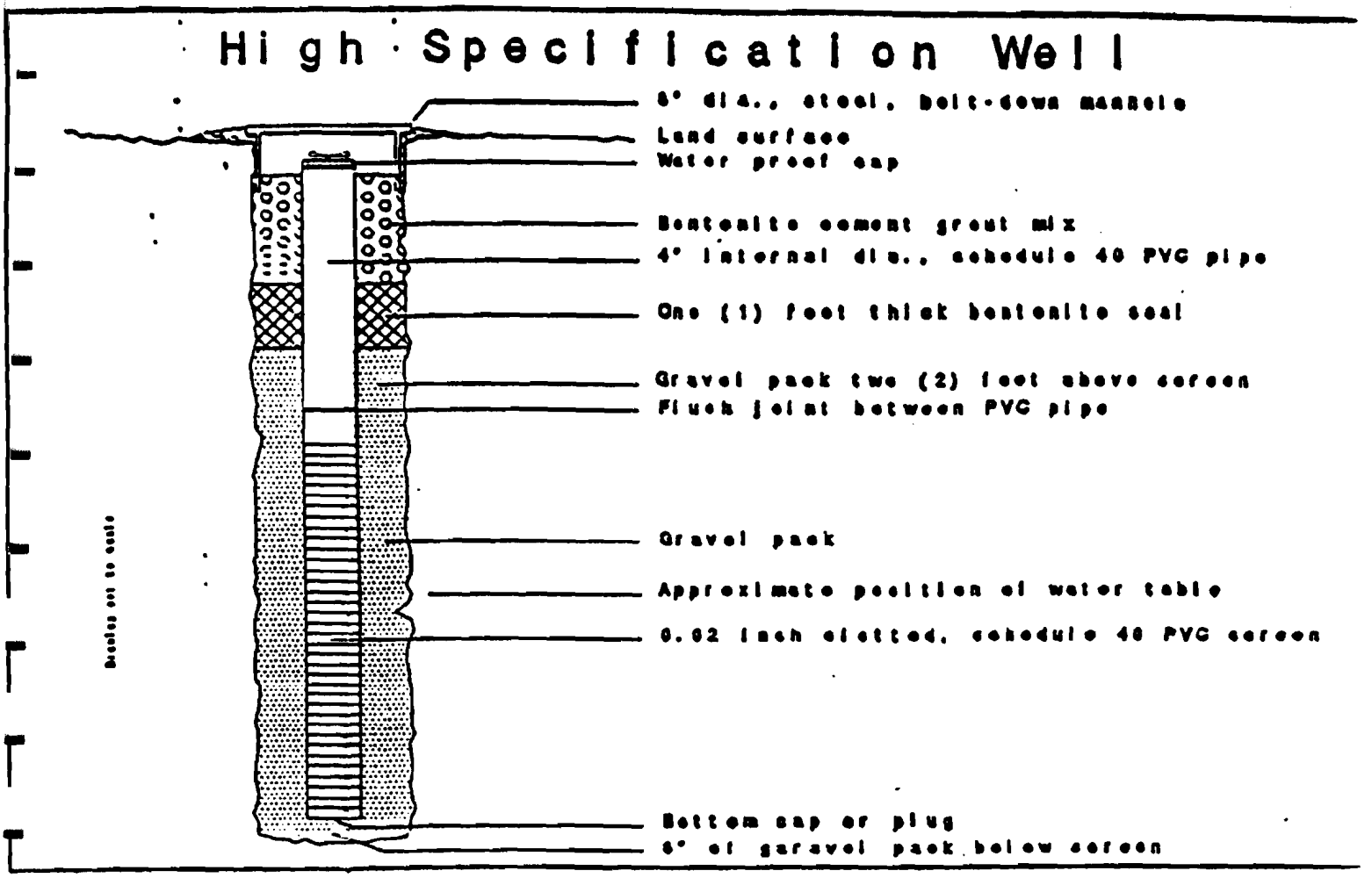
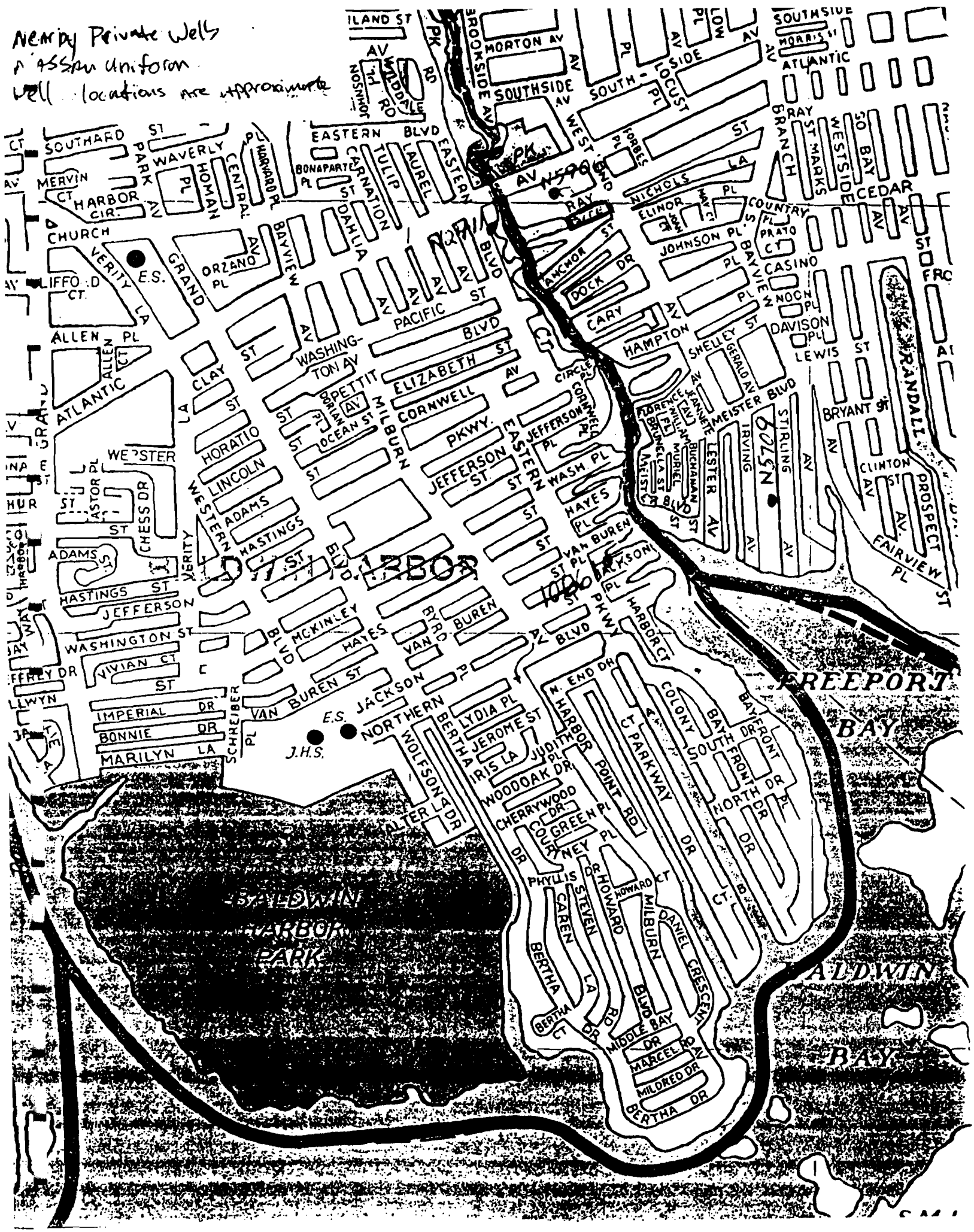

High Specification Well
8" dia., steel, bolt-down manhole
Land surface
Water proof cap
Bentonite cement grout mix
4" internal dia., schedule 40 PVC pipe
One (1) foot thick bentonite seal
Gravel pack two (2) feet above screen
Flush joint between PVC pipe
Gravel pack
Approximate position of water table
0.02 inch slotted, schedule 40 PVC screen
Bottom cap or plug
6" of garavel pack below screen

FIGURE 2

APPENDIX C

NASSAU UNIFORM SERVICES FOIL DOCUMENTS

Nearby Private Wells
N 455m Uniform
Well locations are approximate



W-1561 DUPLICATE—RETAIN

N-39570
N-0617

Well No. 5006
(on preliminary papers)

State of New York
Department of Conservation
Division of Water Power and Control

LOG

COMPLETION REPORT—LONG ISLAND WELL

Ground Surf., El.....ft. above sea ...

X .....ft.

Y

Top of Well

Owner Aerodynamics Lab., Polytech Inst of Blyn

Address 99 Livingston St., Brooklyn

Location of well Bayport

Depth of well below surface 85'3" feet

Depth to ground water from surface 2 feet

CASINGS:

Diameter	6 in.	in.	in.	in.
Length	73 ft.	ft.	ft.	ft.
Sealing	Lead Packer			
Casings removed	none			

SCREENS: Make Everdur Openings 18

Diameter	6 in.	in.	in.	in.
Length	13 ft.	ft.	ft.	ft.
Depth to top from top of casing	71'9" ft.			

PUMPING TEST: Date 6-25-56 Test or permanent pump? T

Duration of Test days 8 hours

Maximum Discharge 30 gallons per minute

Static level prior to test 2 ft. in. below top of casing

Level during Max. Pumping 5 ft. in. below top of casing

Maximum Drawdown 3 ft.

Approx. time of return to normal level after cessation of pumping hours minutes

PUMP INSTALLED:

Type DWT Make Byron Jackson Model No.

Motive power Elec. Make U.S. Motor H.P. 5

Awd Capacity 25 g.p.m. against ft. of discharge head

No. bowls or stages ft. of total head

DROP LINE: SUCTION LINE:

Diameter in. in.

Length ft. ft.

Use of water Cooling

Work started June 6, 1956 Completed June 27, 1956

Date June 29, 1956 Driller C. W. Lauman & Co.

License No. 13

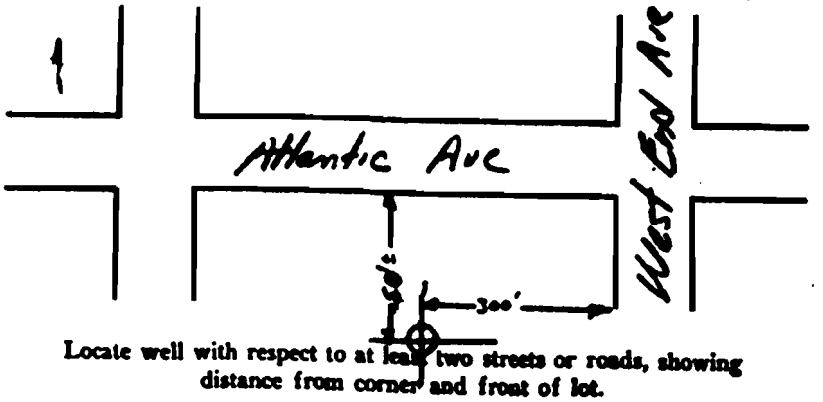
STATE OF NEW YORK
WATER POWER AND
JUL 2 - 1956
CONTROL COMMISSION
RECEIVED

NOTE: Show log of well—materials encountered, with depth below ground surface, water bearing beds and water levels in each, casings, screens, pump; additional pumping tests and other matters of interest. Describe repair jobs.

See Instructions as to Well Drillers' Licenses and Reports—pp. 5-7.

N5906

SKETCH OF LOCATION



Locate well with respect to at least two streets or roads, showing distance from corner and front of lot.

Show North Point

0-4 Bl Muck
4-19 Dirty cse brn SnG
19-25 Cln brn cse. sd, some grvl
25-27 Fi whitish-gry sd f small grvl
27-38 VF gry beach sd
38-40 Fi gry beach sd, humps cl
40-50 Fi gry sd, cl, some grvl
50-52 Cse gry sd f cl
52-54 Fi dk gry beach sd
54-58 Fi gry beach sd
58-62 Fi whitish-gry beach sd
62-65 Fi gry beach sd, humps cl
65-72 Fi dk gry beach sd
72-75 Fi gry beach sd
75-80 Fi dk gry beach sd
80-84 Cse gry sd
84-90 Cse sd w/ lig.
90-100 VF gry beach sd
100-105 Fi gry beach sd
105-110 Lig f pyr
110-111 DK gry sd

County NASSAU

ORIGINAL—TO COMMISSION

State of New York
Department of Conservation
Division of Water Power and Control

COMPLETION REPORT—LONG ISLAND WELLS

Well No. N-2411 (on preliminary report)

LOG

Ground Surf., El. ....ft. above sea

0' ft.

Top of Well

S.W.L. FLOWS

Log
SAND & FILL
1'
HARD BRN CLAY
3'
STINKY BOG
13'
WHITE SAND GRAVEL
24'
VERY FINE GRAY SAND
37'
FINE GRAY SAND GRITS GRAVEL
44'-6"
COARSE GRITS GRAVEL LITTLE SAND
46'
FINE WHITE SAND
48'
DARK GRAY CLAY
50'
DIRTY GRAY SAND, GRITS FEW GRAVEL
55'
HARD BLK. CLAY
59'
FINE DIRTY GRAY SAND
73'
DIRTY WHITE SAND
76'
MEDIUM COARSE SAND GRITS
83'
MED. FINE WHITE SAND
85'

Owner BALDWIN BOAT BASIN

Address 651 ATLANTIC AVE BALDWIN L.I.

Location of well SAME

Depth below surface 84'-9" feet

Depth to water: Ground water FLOWS ft.; Finished well 84'-9" ft.

CASINGS:

Diameter 4" in. ... in. ... in. ... in.

Length 80' ft. ... ft. ... ft. ... ft.

Sealing

Casings removed

SCREENS: Make COOK RED BRASS Openings 020"

Diameter 4" in. ... in. ... in. ... in.

Length 5' ft. ... ft. ... ft. ... ft.

Depth to top from top of casing 79'-7" ft.

PUMPING TEST: Date JULY 14 Test or ~~permanent pump~~?

Duration of Test 1 days 6 hours

Maximum Discharge 15 gallons per minute

Static Level Prior to Test FLOW ft. ... in. below top of casing

Level during Max. Pumping 8' ft. ... in. below top of casing

Maximum Drawdown 8' + ft.

Approx. time of return to normal level after cessation of pumping ... hours 5 minutes

PUMP INSTALLED: NONE (REPORTED BY OWNER)

Type ROTARY Make WESTCO Model No. —

Motive power ELEC. Make DIEHL H.P. 1

Capacity 600 g.p.h. against ... ft. of discharge head

No. bowls or stages 1 ... ft. of total head

DROP LINE: **SUCTION LINE:**

Diameter ... in. ... in.

Length ... ft. ... ft.

Use of water DOMESTIC

TOP SCR. 79'-7"

Work started June 24 Completed July 12

Date OCT. 30 - 1947 Driller EASTERN WELL & PUMP CO.

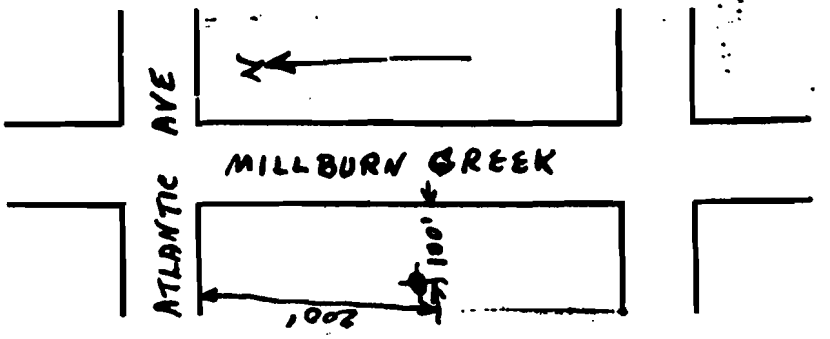
License No. 251

NOTE: Show log of well—materials encountered, with depth below ground surface, water-bearing beds and water levels in each, casings, screens, pump, additional pumping tests and other matters of interest. Describe repair job.

See Instructions as to Well Drillers' Licenses and Reports—pp. 5-7.

N 2411

SKETCH OF LOCATION



Locate well with respect to at least two streets or roads, showing distance from corner and front of lot.

Show North Point

County ..... Well No. ..... (on preliminary report)

ORIGINAL—TO COMMISSION

State of New York
Department of Conservation
Division of Water Power and Control

LOG

Ground Surf., El. .....ft. above sea

COMPLETION REPORT—LONG ISLAND WELL

A .....ft.

V Top of Well

Owner .....

Address .....

Location of well .....

Depth of well below surface .....feet

Depth to ground water from surface .....feet

LOG ON REVERSE SIDE.

REF. P. SCALE 1" = 1'

CASINGS:

Diameter	6" in.	in.	in.	in.
Length	ft.	ft.	ft.	ft.
Sealing	PACKER			
Casings removed				

SCREENS: Make JOHNSON STAINLESS Openings .....

Diameter	4" in.	in.	in.	in.
Length	ft.	ft.	ft.	ft.
Depth to top from top of casing	39'-2" ft.			

PUMPING TEST: Date ..... Test or permanent pump? PERM.

Duration of Test .....days .....hours

Maximum Discharge .....gallons per minute

Static level prior to test .....ft. .....in. below top of casing

Level during Max. Pumping .....ft. .....in. below top of casing

Maximum Drawdown .....ft.

Approx. time of return to normal level after cessation of pumping .....hours .....minutes

PUMP INSTALLED:

Type D.W.T. Make JOHNSTON Model No. 12A-4E-4

Motive power ELEC. Make U.S. MOTORS H.P. 5

Capacity ..... g.p.m. against ..... ft. of discharge head

No. bowls or stages 13 ..... 200 ft. of total head

STATE OF NEW YORK
WATER POWER AND
JUL 13 1956
CONTROL COMMISSION
RECEIVED

DROP LINE: SUCTION LINE:

Diameter 2" in. — in.

Length 20' ft. — ft.

Use of water COMM. SYSTEM SPRINKLING.

Work started 1-25-1956 Completed 7-11-1956

Date 7-12-1956 Driller .....

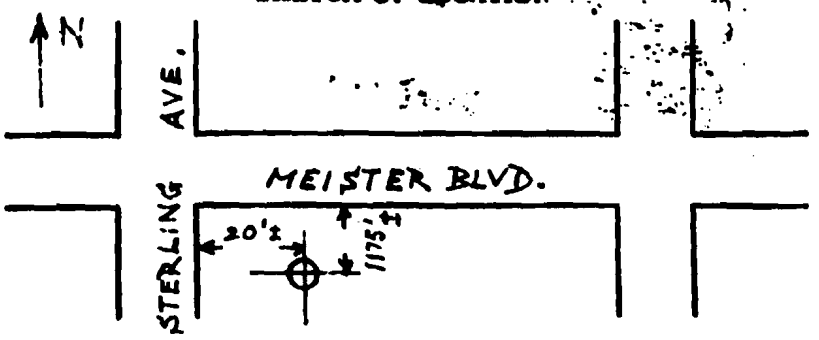
License No. 13

NOTE: Show log of well—materials encountered, with depth below ground surface, water bearing beds and water levels in each, casings, screens, pump, additional pumping tests and other matters of interest. Describe repair job.

See Instructions as to Well Drillers' Licenses and Reports—pp. 5-7.

N5709

SKETCH OF LOCATION



Locate well with respect to at least two streets or roads, showing distance from corner and front of lot.

Show North Point

LOG:

0-10' — BLACK MUCK.
10'-15' — FINE SAND AND MUCK.
15'-20' — SAND AND GRAVEL.
20'-30' — COARSE BROWN SAND AND GRAVEL.
30'-38' — FINE GRAY SAND.
38'-40' — FINE GRAY SAND MIXED WITH PIECES OF GRAY CLAY.
40'-45' — SOFT BLACK CLAY.
45'-50' — COARSE SAND AND GRAVEL.
50'-65' — SOFT GRAY CLAY.
65'-70' — FINE MUDDY SAND.
70'-89'9" — FINE GRAY SAND.
89'9"-95' — FINE AND COARSE CLEAN SAND.
95'-100' — GRAY FINE SAND, LITTLE DIRTY.

County Nassau — Well Number N-10661

COMPLETION REPORT—LONG ISLAND WELL

OWNER: JoAnn Taormina

ADDRESS: 1109 Jackson Pl. Baldwin

LOCATION OF WELL: N/S Jackson Pl 200' E/ Eastern Pkwy Baldwin

*LOG
Ground Surface
EL. _____ ft. above sea

A/V _____ ft.

DEPTH OF WELL BELOW SURFACE	DEPTH TO GROUNDWATER FROM SURFACE
78'	6'

CASINGS

DIAMETER	4 in.	in.	in.	in.
LENGTH	ft.	ft.	ft.	ft.

SEALING

CASINGS REMOVED

SCREENS

MAKE	JOHNSON SS Steel	OPENINGS	10 SLOT
DIAMETER	4 in.	in.	in.	in.
LENGTH	4 ft.	ft.	ft.	ft.

DEPTH TO TOP FROM TOP OF CASING

PUMPING TEST

DATE

TEST OR PERMANENT PUMP?

DURATION OF TEST: days | hours

MAXIMUM DISCHARGE: gallons per min.

STATIC LEVEL PRIOR TO TEST: ft. | in. below top of casing

LEVEL DURING MAXIMUM PUMPING: | in. below top of casing

MAXIMUM DRAWDOWN: ft.

Approximate time of return to normal level after cessation of pumping: hours | min.

PUMP INSTALLED

TYPE	MAKE	MODEL NUMBER
SUB	GOULD	10EJ05422
MOTIVE POWER	MAKE	H.P.
E	FRANKLIN	1/2

CAPACITY: 15 g.p.m. against | ft. of discharge head

NUMBER OF BOWLS OR STAGES | ft. of total head

DROP LINE	SUCTION LINE
DIAMETER in.	DIAMETER in.
LENGTH 21 ft.	LENGTH ft.

METHOD OF DRILLING: ☐ rotary ☒ cable tool ☐ other _____

USE OF WATER: Heat Pump

WORK STARTED: Sept 86

COMPLETED: Sept 86

DATE	DRILLER	LICENSE NUMBER
5-18-88	Louis H. Jackson	586

* NOTE: Show log of well materials encountered, with depth below ground surface, water bearing beds and water levels in each, casings, screens, pump, additional pumping tests and other matters of interest. Describe repair job. See instructions as to Well Driller's License and Reports. Page 5-7.

TOP OF WELL

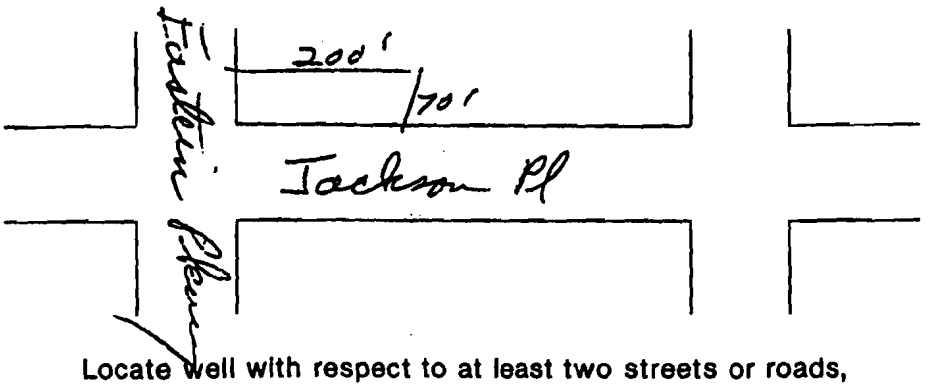
	SAND & GRAVEL
40'	
70'	CLAY
78'	COURSE SAND

ORIGINAL—Environmental Conservation Copy

N-10661

N

SKETCH OF LOCATION



Locate well with respect to at least two streets or roads, showing distance from corner and front of lot.

Show North Point

CHECK THE TOWN IN WHICH THE PROJECT IS LOCATED:

Nassau County:

☐ Hempstead ☐ North Hempstead ☐ Oyster Bay

Suffolk County:

☐ Babylon ☐ Brookhaven ☐ East Hampton
☐ Huntington ☐ Islip ☐ Riverhead
☐ Shelter Island ☐ Smithtown ☐ Southampton
☐ Southold

PRELIMINARY INFORMATION (__) SUFFOLK (X) NASSAU NYSDOT # 84-0959

1. DATE OF SPILL: 7/5/84 APPROX. TIME: UNK.
2. INFO. SOURCE: AMY LIBOLT. TEL. NO. _____ TIME 9:57A DATE: 7/5/84
FROM REPORT BY: BETTY MAUSER. TEL. NO. _____ TIME 3:55P DATE: 7/5/84
FROM REPORT BY: PAUL FENLEY * TEL. NO. 586-9900 TIME _____ DATE: _____
* F&N.
3. SPILL LOCATION: RAY ST., FREEPORT.
4. MATERIAL SPILLED: GASOLINE QUANTITY: UNK. (GALS.)
5. SOURCE: U.G. TANK.
6. CAUSE OF SPILL: FAILED HYDROSTATIC
7. ENVIRONMENTAL IMPACTS: (X) Possible Groundwater
( ) Surface Water
8. SPILLER: NISSAN UNIFORM SERVICE.
9. INITIAL ACTIONS TAKEN:

(X) NYSDOT INVESTIGATING
10. NOTIFICATIONS (NAME/TEL. NO./AGENCY/TIME)

(X) NCDH 7/9/84 L. Hoffman 11AM
( ) NCFM
( ) SCDHS

11. FOLLOWUP ASSIGNMENTS & ACTIONS REQUIRED

COMMENTS:

REPORT PREPARED BY: _____ DATE 7/9/84.

PP 6.28.84 - 7.11.84.

SPILL NO. [redacted]

NEW YORK STATE
DEPARTMENT OF TRANSPORTATION

REGION 10

REGIONAL OIL/HAZARDOUS SUBSTANCES SPILL REPORT

Time of Spill [redacted] AM PM Time Region Notified 10:00 AM PM

LER'S NAME [redacted] Notifier's Name Paul Feeley

LLER'S AGENCY [redacted] Notifier's Agency F Bailey & Nassl

LER'S PHONE [redacted] Notifier's Phone 586-4900

TERIAL SPILLED Gas

UNT SPILLED Unk

CATION (Town & County) Freeport, Nassau

t, Road, or Route) Ray St.

ATERBODY AFFECTED [redacted] Or ☐ Groundwater [redacted]

E OF SPILL possible leaking tank Name of Spiller Nassau Uniform Service

USE OF SPILL Hydrostatic test failed.

TION TAKEN Removed tank had holes

DOT Investigating

Name of Person Contacted
L. Peters

TO BE COMPLETED BY DOT REPRESENTATIVE AT SCENE OF SPILL

ate	Time	Spiller		Investigated by
ca n of Spill		Material Spilled	Amount Spilled Recovered	Resources Affected

☐ First Report ☐ Follow-up Report

Cleanup by
- ☐ SPILLER
- ☐ STATE GOVERNMENT PERSONNEL
- ☐ LOCAL GOVERNMENT PERSONNEL
- ☐ Contractor hired by spiller
- ☐ Contractor hired by Coast Guard or EPA
- ☐ Contractor hired by DOT

NAME OF CONTRACTOR [redacted]

ESTIMATED STARTING DATE OF CLEANUP [redacted]

ESTIMATED COMPLETION DATE OF CLEANUP [redacted]

ESTIMATED COST OF CLEANUP [redacted]

MARKS: Sent pot. well letter

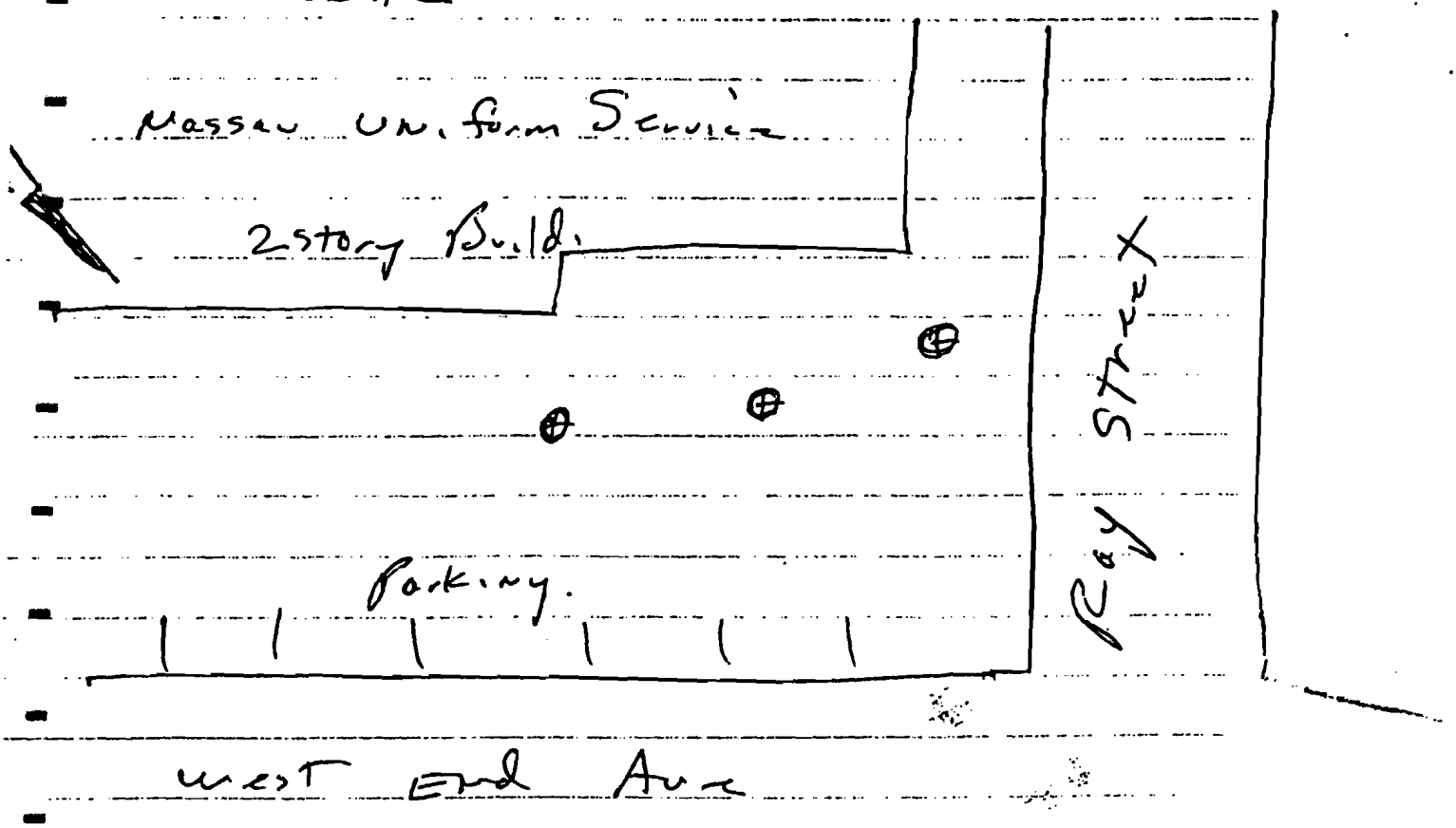
Signature of Person Preparing Report

1:37

Freeport

7-3-87

Ray Street



July 10, 1984

CERTIFIED MAIL-RETURN RECEIPT REQUESTED

Nassau Uniform Service, Inc.
525 Ray Street
Freeport, N Y 11520

Gentlemen:

Re Spill #84-0959

On July 5, 1984, Fenley and Nicol Inc. removed a 2000 gal. underground storage tank that failed a hydrostatic test. This tank had numerous holes indicating the existence of a gasoline spill.

It will be necessary for you to install a site well in the same spot as where the tank was located, in accordance with the attached sketch.

This work is requested in accordance with Article 12 of the N.Y.S. Navigational Law and should be done before July 24, 1984. If work is not started by this date, the State of New York will have no recourse but to hire a contractor to do the work and to seek reimbursement from you at a later date.

If you have any questions, I can be reached at 360-6139.

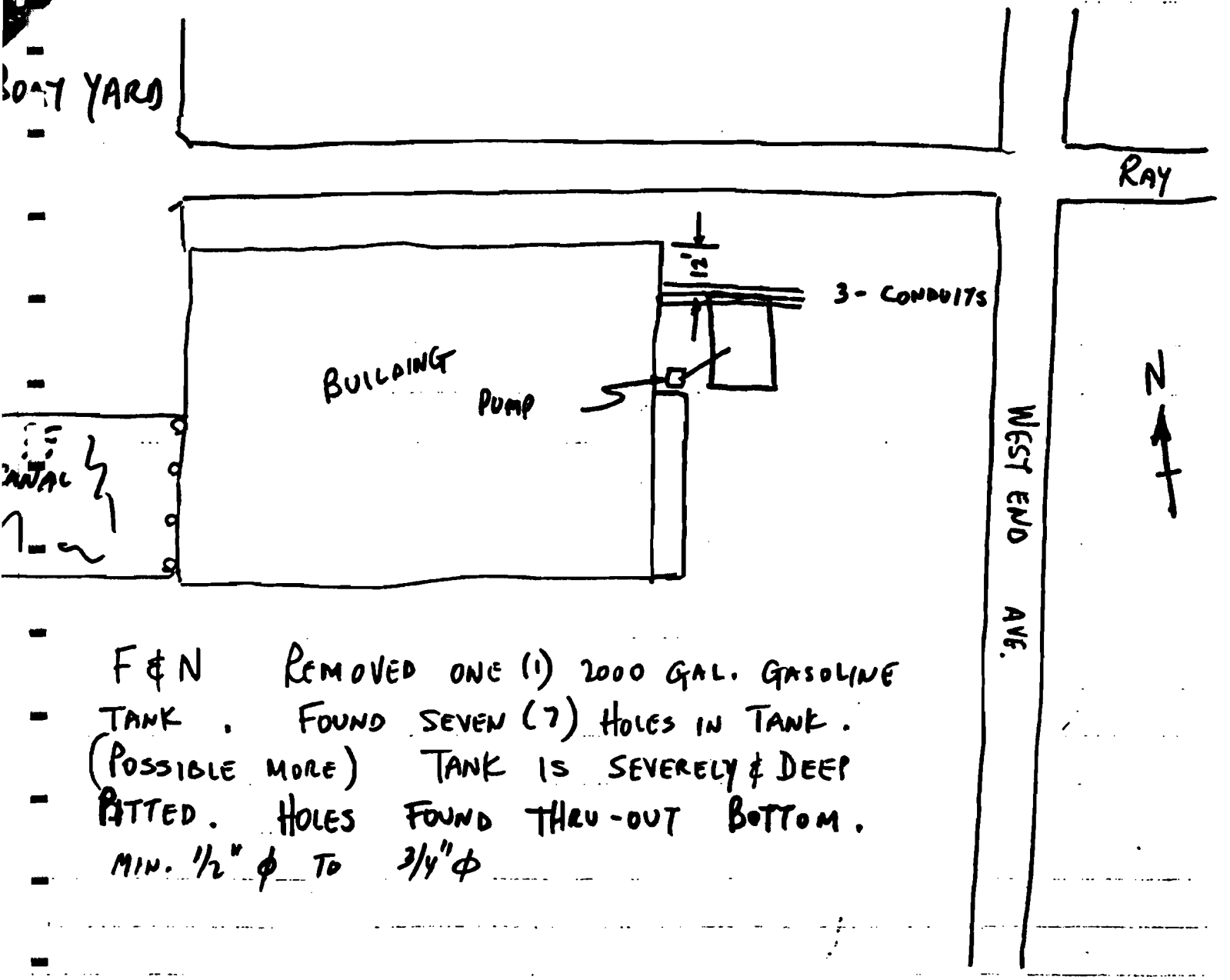
Very truly yours,

L. J. PETEREC, P.E.
Regional Oil Spill Engineer

Attachment

LP:ADM
bcc W. Parish, NYS D. E. C.
S. Silvers, N.C. Health Dept.
N. C. Fire Marshal Bartow, w/a
ROSE File

NASSAU UNIFORM SERICE
RAY STREET, FREEPORT



F & N REMOVED ONE (1) 2000 GAL. GASOLINE TANK. FOUND SEVEN (7) HOLES IN TANK. (POSSIBLE MORE) TANK IS SEVERELY & DEEP PITTED. HOLES FOUND THRU-OUT BOTTOM. MIN. 1/2" ϕ TO 3/4" ϕ

July 10, 1984 CERTIFIED MAIL-RETURN RECEIPT REQUESTED

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525 Ray Street
Freeport, N Y 11520

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Regional Oil Spill Engineer

Attachment

LP:ADM
bcc W. Parish, NYS D. E. C. w/a
S. Silvers, N.C. Health Dept. w/a
N. C. Fire Marshal Bartow, w/a
ROSE File

7e (7/80)

SPILL NO. [illegible]

NEW YORK STATE
DEPARTMENT OF TRANSPORTATION

REGION 10

REGIONAL OIL/HAZARDOUS SUBSTANCES SPILL REPORT

DATE 7-5-84 Time of Spill ____ AM PM Time Region Notified 10:00 AM ~~PM~~

CALLER'S NAME ____ Notifier's Name Paul Fenley

CALLER'S AGENCY ____ Notifier's Agency ____

CALLER'S PHONE ____ Notifier's Phone ____

MATERIAL SPILLED gas

AMOUNT SPILLED U.

LOCATION (Town & County) Freeport, N.

(Street, Road, or Route) Ray St.

WATERBODY AFFECTED ____ Or ☐ Groundwater ____

SOURCE OF SPILL possible leaking tank Name of Spiller Nassau Uniform Servic

CAUSE OF SPILL hydrostatic test failed

ACTION TAKEN removed tank had holes

DOT —

Name of Person Contacted
L. Peterec

TO BE COMPLETED BY DOT REPRESENTATIVE AT SCENE OF SPILL

Date	Time	Spiller		Investigated by
Location of Spill		Material Spilled	Amount Spilled Recovered	Resources Affected

☐ First Report ☐ Follow-up Report

Cleanup by
☐ SPILLER
☐ STATE GOVERNMENT PERSONNEL
☐ LOCAL GOVERNMENT PERSONNEL
☐ Contractor hired by spiller
☐ Contractor hired by Coast Guard or EPA
☐ Contractor hired by DOT

NAME OF CONTRACTOR ____

ESTIMATED STARTING DATE OF CLEANUP ____

ESTIMATED COMPLETION DATE OF CLEANUP ____

ESTIMATED COST OF CLEANUP ____

REMARKS:

Signature of Person Preparing Report

7/19/84 (P.J)

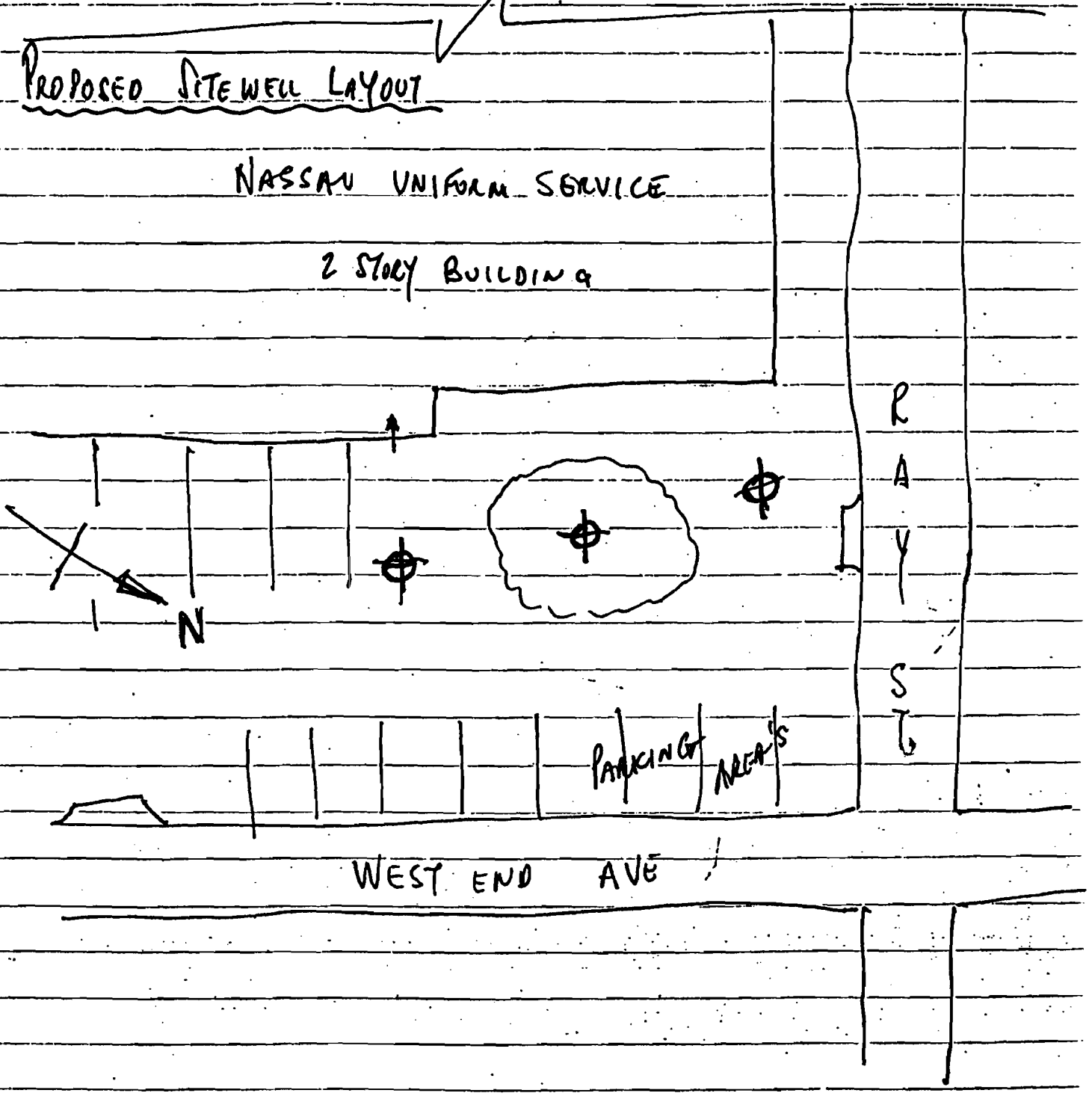
NASSAU UNIFORM SERVICE
FREEPORT - RAY STREET
84-0959

CANAL

PROPOSED SITE WELL LAYOUT

NASSAU UNIFORM SERVICE

2 STORY BUILDING



7/10/85 (PJ)

84-0959

NASSAU UNIFORM - FREEPORT

WITH SONIC TAPE MEASURED

THREE (3) SITEWELLS

AND FOUND THEM TO BE CLEAN.

3/26/85 (PJ)

84-0959

NASSAU UNIFORM

FREEPORT

WITH SONIC TAPE MEASURED

THREE (3) SITE WELL'S

ALL CLEAN,

19/85 (PT) 84-0959

NASSAU UNIFORMS - FREEPORT

WITH SONIC TAPE MEASURED
THREE (3) SITE WELL'S
ALL CLEAN

A-0859 Nassau Uniform Service 1-3-85
Freeport

3 site wells clean used Sonic tape

B J Bradley

8/25/84 RD

84-0959

NASSAU - UNIFORMS

RAY ST. FREEPORT

WITH SONIC TAPE MEASURED

THREE (3) SITE WELLS

ALL CLEAN.

10/20/84 (RT) 84-0959

FREEPORT - UNIFORM

WITH SOME TAPE MEASURED
THREE (3) SITE WELL'S

BILL CLOAN

8/3/84 84-0959

Nassau Uniforms
Freeport

MR ZINN (FR-8-0018) advises that 3 Sets Wells have been installed.

Would like somebody to check them so he can pave area.

9/7/84 (PJ)

84-0959
NASSAU UNIFORM
RAY ST. FREEPORT

WITH SONIC TAPE MEASURED
THREE (3) SITE WELLS
ALL "CLEAN"

DEPTH OF SITE WELL 15'
DEPTH TO GROUND WATER 4.51

SPOKE TO MR. MARTY ZINN AND TOLD HIM HE MAY
REPAVE EXCATION, KEEP SITE WELLS LEVEL WITH
PAVEMENT.

RECORD PREPARED BY: [illegible] DATE 11/14/84.

PP 6,28,80 - 7.51 01.

APPENDIX D

GROUNDWATER TECHNOLOGIES, INC.
1994 SAMPLING DATA

DRAFT

GROUNDWATER TECHNOLOGY ®

Groundwater Technology, Inc.
101-1 Colin Drive, Holbrook, NY 11741 USA
Tel: (516) 472-4000 Fax: (516) 472-4077

October 14, 1994

Mr. Michael E. White
Jaspan, Ginsberg, Schlesinger, et al
300 Garden City Plaza
Garden City, New York 11530-3324

Re: Summary Report for Additional Soil and Groundwater Investigation
Nassau Uniform Service, Inc.

Dear Michael:

On September 23, 1994, Groundwater Technology, Inc. supervised the installation of six geoprobe points for the collection of soil and groundwater samples. A site map illustrating the locations of the points is presented as Figure 1.

Initially, a core drill was utilized to drill through the concrete floor in the building warehouse. The concrete ranged from approximately 4" - 6" in thickness. A quad mounted geoprobe unit then set up at each location and sampling procedures began. A total of three samples, each consisting of a 4-foot length core of soil, were extracted from each location. The samples were collected from depths of 0-4', 4-8', and 8-12' below grade. An open borehole to 12 feet below grade was thus created. Each core sample was field screened with a flame ionization detector (FID) for the detection of volatile organic compounds (VOCs). A summary of the FID results and lithologic descriptions is presented as Table 1. One soil sample with the highest FID results from each boring was submitted to EcoTest Laboratories of North Babylon, New York for analysis of VOCs by EPA Method 8010. A summary of the soil analytical results is presented as Table 2.

Upon coring to a depth of 12 feet below grade, groundwater samples were then collected. A drill rod fitted with a two foot length of stainless steel screen was fitted within each borehole from a depth of 10'-12' below grade. New polyethylene tubing fitted with a ball check valve was installed inside the drilling rods and screen and oscillated up and down to push a column of water to the top of the tubing. Approximately one standing water well volume was purged from each location prior to collecting the groundwater sample. The samples were submitted to EcoTest Laboratories for analysis of VOCs by EPA Method 601, chloride and sodium. A summary of the groundwater analytical results is presented in Table 3.

Conclusions

The soil deposits at the site consists mainly of fine to medium sands with some clay and gravel, and marsh deposits located from five to eight feet below grade. Fill material was noted in GP-2 from grade to four feet. Groundwater was encountered at approximately seven feet below grade.

As specified in the Division Technical and Administrative Guidance Memorandum (TAGM: #HWR-94-4046) Determination of Soil Cleanup Objectives and Cleanup Levels dated January 24, 1994, a total of two locations (GP-2 and GP-3) exceeded the cleanup objective for 1,2-Dichloroethene, three locations (GP-2, GP-3, and GP-5) exceeded the cleanup objectives for trichloroethylene, two locations (GP-2 and GP-3) exceeded the cleanup objective for tetrachloroethene, and two locations (GP-2 and GP-3) exceeded the cleanup objective for total volatile organic compounds.

Groundwater samples GP-2 through GP-6 exceeded the Class GA standards for those volatile organic compounds listed in Table 3. Specifically, GP-2 exceeded the class GA standard for tetrachloroethene, GP-3 exceeded the Class GA standard for vinyl chloride, 1,2-Dichloroethene, trichloroethylene, and tetrachloroethene, GP-4 exceeded the Class GA standard for vinyl chloride, 1,2-Dichloroethene, trichloroethylene, tetrachloroethene, and chlorobenzene, GP-5 exceeded the Class GA standard for 1,1-Dichloroethane, 1,2-Dichloroethene, trichloroethylene, and tetrachloroethene, and GP-6 exceeded the Class GA standard for 1,2-Dichloroethene, 1,1,1-Trichloroethane, trichloroethylene, and tetrachloroethene. GP-1 did not exceed any Class GA standard for volatile organic compounds. The Class GA standard for chloride was exceeded in GP-5 and sodium was exceeded in GP-1 through GP-6.

The results of the laboratory analysis indicates that VOC contamination in groundwater extends to the perimeter of the garage area. There were no VOCs detected in the apparent upgradient groundwater sample. Results of the sodium analysis were above the Class GA standards in all water samples and chloride concentrations were above Class GA standards in one sample, GP-5. These results could be used to argue with the NYSDEC for a different classification, and therefore less stringent cleanup requirements.

Please contact this office if you have any questions or comments regarding this report.

Sincerely,

GROUNDWATER TECHNOLOGY, INC.

Albert M. Tonn
Project Manager

Enclosures

NassauUniform/sumrpt1.094

TABLE 1
SUMMARY OF SOIL BORING LOGS
NASSAU UNIFORM SERVICES
525 RAY STREET
FREEPORT, NEW YORK

SEPTEMBER 23, 1994

DRAFT

GEOPROBE POINT	DEPTH (ft)	PID READING (ppm)	LITHOLOGIC DESCRIPTION
GP-1	0-4	0	Brown sand, fine grained, poorly sorted, some gravel, clayey sand at 2-4'.
	4-8	0	Orange brown sand, fine to medium grain, wet at 7', fairly well sorted.
	8-12	3	Same as above, trace coarse sand.
GP-2	0-4	90	Brown sand, poorly sorted, some concrete and brick fill.
	4-8	400	Black marsh deposits, wet.
	8-12	400	Black sand, then gray, then brown, fine to medium grained, fairly well sorted.
GP-3	0-4	15	Brown sand, fine to medium grained, trace small gravel, fairly well sorted.
	4-8	100	Brown clayey sand to 4.5', then black marsh deposits, then grey sand, fine grained, fairly well sorted.
	8-12	>1000	Grey sand, fine grained, fairly well sorted, then sand becomes brown at 10', sheen detected on water.
GP-4	0-4	0	Brown sand, fine to medium grain, fairly well sorted, trace gravel, dark brown clayey sand at 3.7'.
	4-8	4	Brown sand with black marsh deposits and some brown and grey clay, wet.
	8-12	400	Grey sand, fine to medium grained, trace coarse sand and gravel, trace black marsh deposits.
GP-5	0-4	0	Tan, brown and dark brown sand, fine to medium grained, fairly well sorted, trace gravel.
	4-8	90	Brown sand to 5', then black organic marsh, then brown sand, fine to medium grained, fairly well sorted, trace soarse sand, wet.
	8-12	90	Brown sand, fine to medium grained, fairly well sorted, trace coarse sand and gravel, becomes orange at 11'.
GP-6	0-4	30	Brown sand, fine to medium grained, fairly well sorted, then black clayey sand deposits, marsh.
	4-8	>1000	Black organic marsh deposits to 7', then grey sand, fine to medium grain, fairly well sorted, trace gravel.
	8-12	200	Grey sand, fine to medium grained, fairly well sorted to 10', then sand becomes orange.

TABLE 2
SOIL ANALYTICAL RESULTS
NASSAU UNIFORM SERVICES
525 RAY STREET
FREEPORT, NEW YORK

DRAFT

SEPTEMBER 23, 1994

LOCATION					
GP-1 (0'-4')	ND	ND	160	ND	160
GP-2 (0'-4')	7,400	12,000	11,000	280	30,680
GP-3 (0'-4')	800	7,400	2,200	70	10,470
GP-4 (0'-4')	55	400	1,000	ND	1,455
GP-5 (0'-4')	230	1,400	1,400	ND	3,030
GP-6 (4'-8')	5	5	ND	ND	10
RECOMMENDED CLEANUP OBJECTIVE*	300	700	1,400	7,900	10,000

Results reported in ug/kg (ppb)
Samples analyzed by EPA Method 8010

* – Based upon NYSDEC TAGM#HWR-94-4046, January 24, 1994, Determination of Soil Cleanup Objectives and Cleanup Levels

Nassau Uniform/soilana.wk3

GROUNDWATER TECHNOLOGY

DRAFT

TABLE 3
GROUNDWATER ANALYTICAL RESULTS
NASSAU UNIFORM SERVICES
525 RAY STREET
FREEPORT, NEW YORK

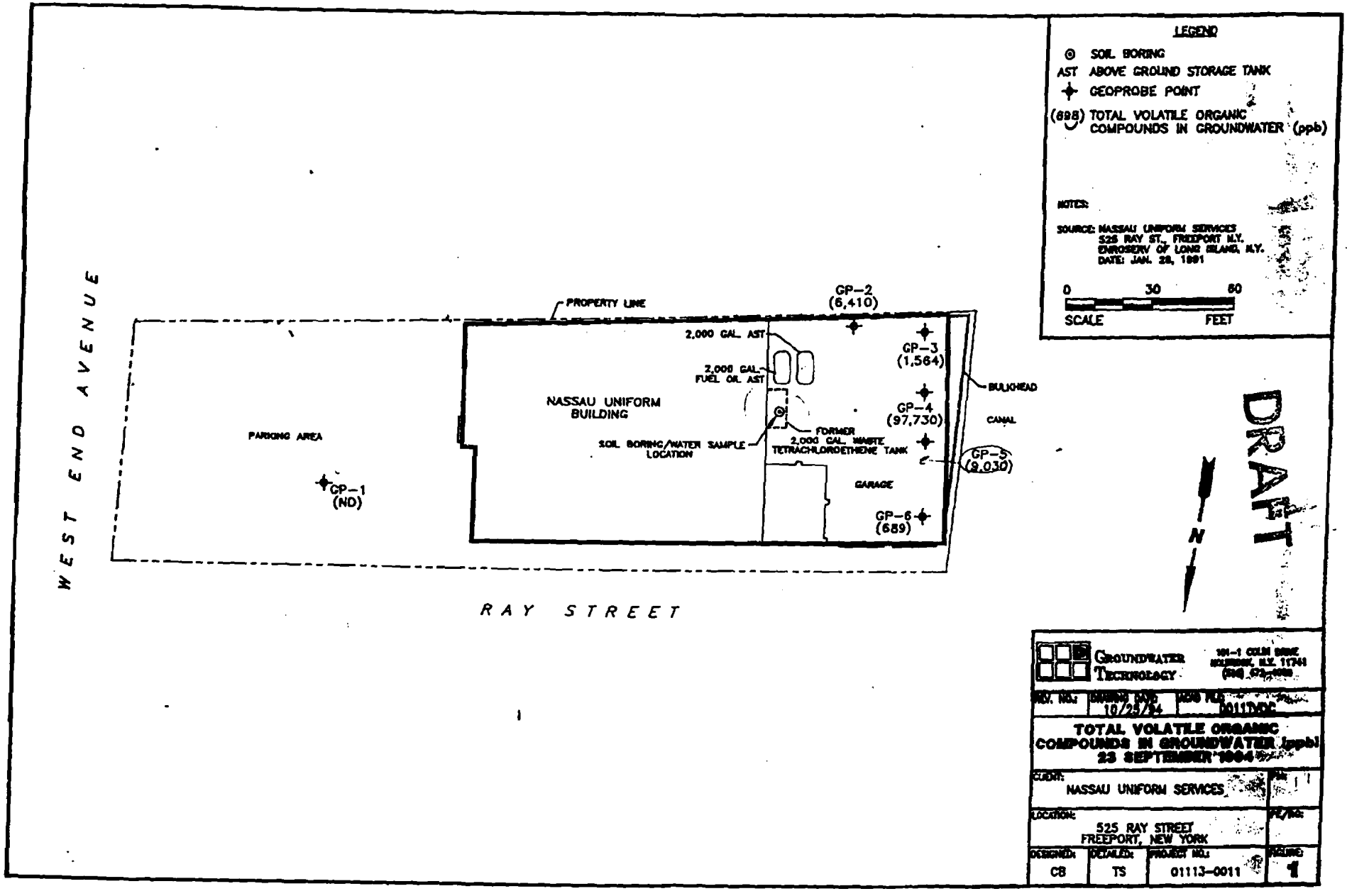
SEPTEMBER 23, 1994

COMPOUND	LOCATION						CLASS GA STANDARDS
	GP-2	GP-3	GP-4	GP-5	GP-6	GP-8	
Vinyl Chloride	ND	ND	280	180	ND	ND	2
1,1 Dichloroethane	ND	ND	ND	ND	10	ND	5
1,2 Dichloroethene	ND	ND	150	4,100	5,600	24	5
1,1,1 Trichloroethane	ND	ND	ND	ND	ND	26	5
Trichloroethylene	ND	10	34	2,300	630	39	5
Tetrachloroethene	ND	6,400	1,100	91,000	2,800	600	5
Chlorobenzene	ND	ND	ND	150	ND	ND	5
Total VOCs	ND	6,410	1,564	97,730	9,030	689	NA
Chloride	77,000	250,000	210,000	140,000	610,000	190,000	250,000
Sodium	120,000	190,000	150,000	140,000	380,000	140,000	20,000

All results reported in ug/l (ppb)
Samples analyzed for Volatile Organic Compounds by EPA Method 601, Chlorides by 4500CL-B and Sodium by EPA Method 3500 NA-C.
ND -- Not Detected
NA -- Not Applicable

nassau uniform/gwana

GROUNDWATER TECHNOLOGY


LEGEND
SOIL BORING
AST ABOVE GROUND STORAGE TANK
GEOPROBE POINT
(698) TOTAL VOLATILE ORGANIC COMPOUNDS IN GROUNDWATER (ppb)
NOTES:
SOURCE: NASSAU UNIFORM SERVICES
525 RAY ST. FREEPORT N.Y.
ENVROSERV OF LONG ISLAND, N.Y.
DATE: JAN. 28, 1991
0 30 60
SCALE FEET
WEST END AVENUE
PROPERTY LINE
GP-2 (6,410)
2,000 GAL. AST
2,000 GAL. FUEL OIL AST
GP-3 (1,564)
NASSAU UNIFORM BUILDING
GP-4 (97,730)
BULKHEAD
CANAL
PARKING AREA
SOIL BORING/WATER SAMPLE LOCATION
FORMER 2,000 GAL. WASTE TETRACHLOROETHENE TANK
GP-5 (9,030)
GARAGE
GP-1 (ND)
GP-6 (689)
RAY STREET
N
DRAFT
GROUNDWATER TECHNOLOGY
101-1 COLIN DRIVE
HOLBROOK, N.Y. 11741
(516) 472-4900
REV. NO.:
DRAWING DATE: 10/25/94
WORK FILE: 0011DWG
TOTAL VOLATILE ORGANIC COMPOUNDS IN GROUNDWATER (ppb)
23 SEPTEMBER 1994
CLIENT: NASSAU UNIFORM SERVICES
LOCATION: 525 RAY STREET FREEPORT, NEW YORK
PZ/No.:
DESIGNED: CB
DETAILED: TS
PROJECT NO.: 01113-0011
DRAWING: 1

APPENDIX E

RESUMES
OF KEY PERSONNEL

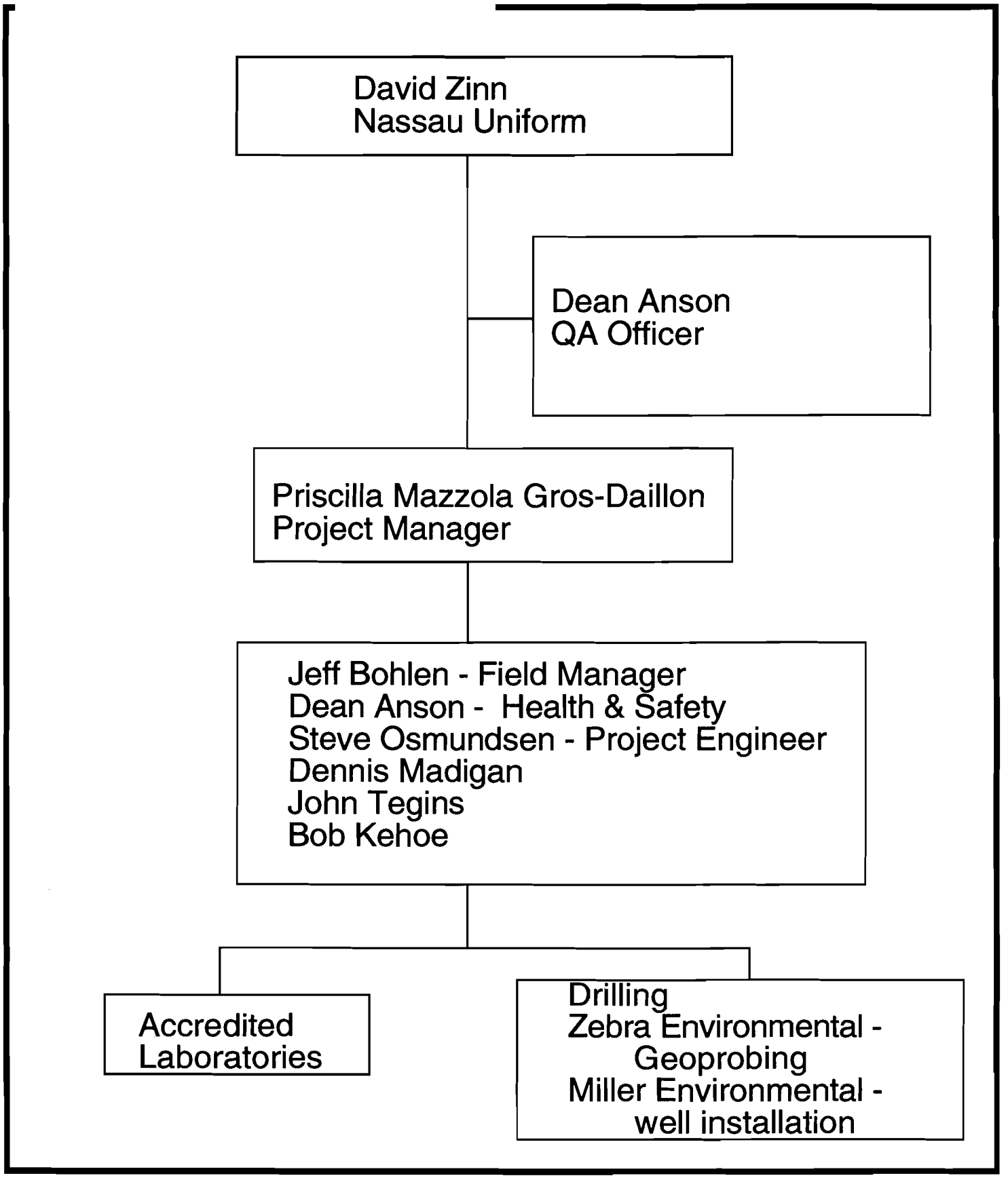


Figure 1 Nassau Uniform Project Organization rev. 3/18/97

Dean Anson II
President and Supervising Scientist

Experience Summary:
Over 20 years of experience: President of Anson Environmental; Associate and Supervising Environmental Scientist at Storch Associates; Project Manager and Supervising Environmental Scientist at Gibbs & Hill, Inc. Directed and participated in RI/FS studies, site remediation, environmental impact statements, groundwater assessments, technical support for legal counsel, expert witness, negotiations with federal, state, and local regulatory agencies.

Education:
B.A. Zoology, Ohio Wesleyan University, 1969
M.S. Biology, New York University, 1976
M.B.A. Marketing and Finance, New York University, 1981
USEPA Bioremediation Symposium, 1993
OSHA - 40 Hour Health & Safety Operations at Hazardous Materials Sites, 1991
Appointed to Suffolk County Pine Barrens Review Commission
ASCE Modeling Groundwater Quantity and Quality Using Microcomputers, Seminar Participant
ENR Hazardous Waste Management & Cleanup, Seminar Participant
AHERA Building Inspector
AHERA Management Planner
NYS Air Sampling Technician

Key Projects:
• Facility Coordinator and Health and Safety Officer for Anchor Chemical Superfund Site in Hicksville, NY. Implementing Remedial Investigation Project Operations Plan including the new installation of indoor borings, drywell sampling, and installation of groundwater monitoring wells. Developed site specific Health and Safety Plan.

• Principal-in-Charge of soils and groundwater investigations for thirty-five properties (approximately 40 acres) in the New Cassel Industrial Area. Purpose of investigation is to demonstrate that properties did not contribute to groundwater contamination.

• Principal-in-Charge of groundwater investigation at former drycleaning site where remediation included the removal of over 440 tons of contaminated soil and installation of groundwater remediation system to filter tetrachloroethylene from groundwater. Currently performing six-month long engineering study to identify other sources of contamination by volatile organic compounds.

• Principal-in-Charge of RI/FS for groundwater remediation project in Great Neck, Long Island. Site was contaminated by leaking underground storage tanks which discharged volatile organic compounds. Approximately 400 tons of contaminated soil were removed and groundwater sampling program is ongoing.

• Developed Health and Safety Plan for Katonah Municipal Well site in Bedford, New York included soils investigation at the municipal pump house. Plan was accepted by USEPA, Region 2.

• Site Coordinator for Tronic Plating Superfund site in Farmingdale, Long Island. Negotiated work plan with USEPA, Region 2 and developed Project Operation Plan which was implemented by others.

• Principal-in-Charge of several studies to define plumes of groundwater contamination in Nassau and Suffolk Counties. Negotiated groundwater and hazardous waste matters with USEPA and NYSDEC regarding scope of groundwater studies.

Dean Anson II
President and Supervising Scientist (cont)

- Lectured and provided expert witness comments at New York University, Nassau County Bar Association, Suffolk County Pine Barrens Review Commission, and numerous Town Board meetings on Long Island.

- Project Manager for asbestos surveys and asbestos awareness training for Suffolk County Department of Public Works project for 60 County-owned buildings.

- Evaluated environmental conditions at over 100 industrial plants and commercial sites along the East Coast from Connecticut to Virginia.

- Managed removal of over 100 underground storage tanks and installation of new above and underground storage tanks.

- Member of Suffolk County Bar Association Environmental Committee, Hauppauge Industrial Area Environmental Committee, and Huntington Chamber of Commerce Environmental Committee

Fritzl Mazzola Gros-Daillon
Vice President/Senior Environmental Scientist

Experience Summary:
Over 20 years of experience: Manager of Quality Assurance at Anson Environmental; Independent Management Consultant; Vice President at Bankers Trust Company, District Manager for Consolidated Edison Company. Project management, quality control and assurance, contractor coordination and administrative management.

Education:
B.S. Business Administration, Bloomfield College, 1979
M.S. Business Policy, Columbia University, 1981
Certified Environmental Inspector (CEI), 1991
In Situ and On-Site Bioreclamation Symposium, 1993
NYS Air Sampling Technician, 1993
Indoor Air Quality Seminar, Hazmat Conference, 1991
Hazardous Waste in New York Seminar, 1991
Asbestos Awareness Course, 1990

Key Projects:
• Performed environmental assessments in over fifty industrial and commercial buildings on Long Island. Assessments included identification of operations out of compliance with environmental regulations, searches of regulatory databases and files, site reconnaissance, and report preparation.

• Quality Assurance Officer for asbestos surveys in 60 Suffolk County-owned buildings where over 1,500 samples were collected for laboratory analysis. Sample data were entered into a computerized database for the prioritization of abatement activities.

• Quality Assurance/Quality Control officer, providing technical oversight and guidance on variety of environmental projects including resource allocation, hazardous material contamination, proposal, and work plan preparation.

• In the past year, coordinated the removal of over 50 underground storage tanks in Nassau and Suffolk Counties.

• Project Manager for the groundwater investigation and subsequent delisting petitions for forty-two properties within the State Superfund New Cassel Industrial Area. Delisting petitions have been successful in thirty-seven cases with the balance still under review by the NYSDEC.

• Project Manager of groundwater investigation at former drycleaning site. Investigation includes the sampling of eleven groundwater monitoring wells located in the Upper Glacial Aquifer and delineation of contaminant plume. Alternative remediation technologies under consideration by client.

• Evaluated laboratory data for groundwater remediation project in Great Neck, Long Island. Site was contaminated by leaking underground storage tanks which discharged volatile organic compounds that have contaminated the Upper Glacial Aquifer.

• Responsible for oversight of all data validation, field schedules, laboratory analyses, and report production at Anson Environmental.

• Site Manager for Federal Superfund site during installation of monitoring wells and soil borings. Coordinated sample shipment to laboratories and correspondence with USEPA. Conducted data evaluation and assisted in preparation of Remedial Investigation Report.

• Coordinated several underground storage tank removal projects for local clients in small commercial and residential properties, including coordination with appropriate County officials.

Jeffrey Bohlen
Environmental Geologist

Experience Summary:
Over 2 years experience: Field Technician, Anson Environmental Ltd. Summer positions in construction and marine industries.

Education:
B.S. Environmental Geology, Long Island University, Southampton, 1995.

Summer Hydrogeology Field Course at Western Michigan University, 1995.

OSHA 40-Hour Hazardous Waste Course (HAZWOPER), 1995.

Key Projects:

• Designed and installed soil vapor extraction system (SVES) at Dry Cleaners where tetracholorethrene contamination was identified in drywells, leaching pools and floor drains. Remediation was started by using a vactor truck to excavate the most grossly contaminated soils. The horizontal and vertical extent of contamination was identified by installing borings and correction continuous split spoons to define soil conditions onsite.

• Participated in Phase I and II investigations at a former jewelry plating facility. This work included the collection of samples from an onsite drywell and from beneath the concrete flooring of the facility. The facility was successfully closed to the satisfaction of the NYSDEC.

• Field manager in charge of collecting groundwater and soil samples at a manufacturing facility in the New Cassel Industrial Area. This work involved determining the complex geological subsurface conditions and evaluating the impact on groundwater beneath the site.

• Participated in several Phase I investigations for properties across Long Island. This work included site inspections, research of site history, and data base searches of government documents.

• Provide ongoing bioremediation system support for remediation of large underground fuel oil spill.

STEPHEN J. OSMUNDSEN, P.E.

PROFILE

Mr. Osmundsen is President of S. J. Osmundsen Engineering. He has over 20 years experience in the design, construction, and management of major environmental projects in the industrial arena. He has completed complex projects in many process sectors including: pulp and paper, petroleum refining, cosmetic, pharmaceutical, mining, dairy, food processing, plating, and printing. He is an experienced specification and construction contract writer, value-engineer, and project manager.

EXPERIENCE

Mr. Osmundsen's experience includes the development of plans, specifications and the design for: industrial water supply and fire protection systems; wastewater treatment and pretreatment plants; hazardous waste treatment, storage, and disposal facilities; industrial and RCRA landfills; air pollution control devices; bulk chemical storage and tank farms; and chemical recovery systems.

EDUCATION

Master of Engineering
Cornell University (1975)

Bachelor of Science
Clarkson College of Technology (1974)

PROFESSIONAL

Engineering licenses: New York, Georgia, Kansas, Wisconsin and Michigan

Memberships: American Society of Civil Engineers, New York Water Pollution Control Federation, Technical Association for the Pulp and Paper Industry, Water Environment Federation, Consulting Engineers Council, and Empire State TAPPI

SELECTED PROJECTS

Supervised and led the design team for a $12 million tertiary wastewater treatment system for Murphy USA's 30,000 Bbl refinery in Superior Wisconsin. The design included provisions to meet the stringent requirements of the Great Lakes Initiative. The successful design required the coordination of refinery process changes, waste minimization, process wastewater flow reduction, and stormwater diversion and control.

Developed the design and supervised the detailed engineering and construction of a $6 million wastewater treatment facility for the Bay West 400 ton deinked towel and tissue mill in Middletown, Ohio.

Supervised the design of the $2.5 million expansion of the General Electric Schenectady Works industrial wastewater treatment plant form a 40 million gallon a day capacity to 60 million gallons per day. The design included the up-grading the industrial based control system to an on-line computer system with interactive control and data acquisition.

Supervised and prepared designs and specifications for the closure, clean-up and remediation activities at Fairchild Industries', Farmingdale, New York Facility. Activities at the site included hazardous waste treatment unit closure, underground tank removal, soil removal, asbestos removal, building demolition and soil vapor extraction.

Designed a $4 million industrial wastewater pretreatment plant and individual double contained waste collection system for concentrated and dilute waste streams for Clairol's Stamford, Connecticut plant. The eight vertical aboveground collection and batch treatment tanks are provided with concrete berming for secondary containment.[1]

RELEVANT PROJECTS

Designed the secondary induststrial wastewater treatment system for the pretreatment of Bristol me

Developed the process design for the cooling water treatment for the condensing water at Fort Orange Paper Company's 60 megawatt LM 5000 combined cycle cogeneration plant. The process evaluation included surface water and stand-by well water treatment, cooling tower materials of construction, and cooling tower blowdown treatment.

The process design for cooling tower to temper the wastewater form Pentec Papers 600 ton per day pulp mill as the preparatory stop for biological treatment. The design had to consider some unusual conditions including the relatively high temperature of the six million gallon a day, the extremely corrosive nature of the pulp mill effluent, and the high fiber content of the wastewater. The design of the distribution system and tower configuration were especially important due the plugging potential and the materials of construction critical because of the high temperature and corrosivity of the waste stream.

1

APPENDIX F

QUALIFICATIONS
OF
ANSON ENVIRONMENTAL LTD.

Environmental Qualifications of Anson Environmental Ltd.

1. Introduction

Anson Environmental Ltd. (AEL), a full-service environmental consulting firm has had extensive experience performing environmental investigations throughout the northeastern United States. These investigations have included:

-soil and groundwater studies and remediations
-storage tank programs
-environmental site assessments
-asbestos management plans
-wetland investigations and delineations

Remedial techniques employed by AEL include the traditional soil excavation and disposal, pump and treat groundwater, as well as, state-of-the-art soil vapor extraction, air sparging and bioremediation.

AEL's staff is composed of environmental scientist and engineers who have current working knowledge of local, state and federal regulations. AEL's engineers are licensed to perform work in New York, New Jersey and Connecticut.

The attached matrix illustrates AEL's personnel's 1996 experience working on Inactive Hazardous Waste Disposal Sites on Long Island.

2. Soil and Groundwater Investigations

Since its founding in 1988, AEL has performed soil and groundwater investigations at over one hundred site on Long Island where our role has included:

-preparing site specific work plans and health and safety plans
-directing the removal of contaminated soils
-installing wells and monitoring groundwater contamination
-identifying the extent of groundwater contamination
-developing engineering reports
-developing and implementing groundwater and soil remediation programs.

These investigations were the result of contaminations resulting from leaking underground storage tanks, illegal discharges or spills. Soils or sediments contaminated with volatile and/or semi-volatile organic compounds, metals and PCBs have been excavated and disposed of in approved landfills. Volatile organic compounds have been removed from the soil using soil vapor extraction systems.

3. Soil and Groundwater Remediation

The following are examples of selected remediation projects performed by Anson Environmental Ltd. (AEL). Each of these recent projects included a soil vapor extraction system (SVES) to remediate volatile organic compounds in the soil. The vertical and horizontal extent of contamination was defined and pilot test performed to identify the areas of influence.

Busy Bee Dry Cleaners
Merrick, NY

Soil samples were collected from twenty soil boring locations and from leaching pools to define the extent of contamination with tetrachloroethene and its breakdown products. Once the horizontal extent of contamination was defined, a soil vapor extraction system was designed. The extraction well was installed horizontally because groundwater was eight feet below the ground surface.

A two horsepower Carbonnaire system was connected to the four-inch diameter horizontal extraction wells. The skid-mounted remediation system included two carbon canisters that each contained one-thousand pounds of carbon which were used to filter the air before it was emitted to the atmosphere. A moisture separator removed the moisture in the extracted air. This liquid was disposed according to federal, state and county guidelines.

Project oversight was provided by Nassau County Department of Health.

Following operation for thirteen months, the soil contamination was eliminated and the endpoint samples were below the NYS TAGMs. The concentration of volatile organic compounds in the groundwater were reduced to acceptable levels.

Village of East Hills
East Hills, NY

A leaking underground gasoline storage tank contaminated soil in an area 20 feet by 40 feet by 30 feet deep. A four-inch diameter extraction well was installed and a pilot test was performed to determine the radius influence and data needed to prepare an air permit application for the Nassau County Department of Health.

The two horsepower blower operated for approximately twelve months before the soil contamination was remediated to the satisfaction of Region 1 of the NYS Department of Environmental Conservation.

System monitoring included the removal and proper disposal of liquids accumulated during the operation of the SVES and monthly monitoring of the emissions from the system. The emission monitoring was critical not only to satisfy the air permit but, because the system was located in a residential area.

Baclays Bank
Lake Success, NY

Following the performance of a pilot test, a two horsepower blower is attached to three four-inch diameter extraction wells that are manifolded. These wells are screened at 30-40, 40-50 and 50-60 feet depth below grade where fuel oil contaminated soils are located. These wells are part of the bio venting system (bioremediation and soil vapor extraction) that is being used to remediated a spill of 6,000 gallons of fuel oil.

The extent of contamination was determined by installing ten groundwater monitoring wells and sixty soil borings to establish the vertical and horizontal extent of contamination in the soil and groundwater.

The performance of the bioventing is monitored by collecting soil samples from different depths and analyzing the soil for BTEX and bacterial colonies. The exhaust from the SVES is samples to both monitor the concentration of BTEX at each depth as well as to make sure the system is in compliance with the federal Clean Air Act Amendments. The system has continued to operate in compliance with the permit requirements stipulated by the Nassau County Department of Health.

Monthly status reports are submitted to Region 1 NYS Department of Environmental Spills Group.

Lee Myles Transmissions
Huntington, NY

Approximately 600 cubic yards of soil were contaminated by discharges of BTEX and halogentated solvents. These chemical compounds were discharged into a cesspool on site. The horizontal (1,000 feet by 750 feet) and vertical (9 to 11 feet depth below grade) extent of contamination was defined by installing soil borings. The soil types were determined to be coarse to medium sands in the contamination zone with layers of clay both above and below this contamination.

The pilot test of the two horsepower blower determined a fifteen foot diameter of area of influence. The eight extraction wells were screened at 9 to 11 feet below grade and were manifolded to the blower such that the contamination could be prevented from migrating off site.

The application for an air permit is currently being prepared. Suffolk County Department of Health Services is overseeing the project.

Imperial Cleaners
Lake Success, NY

A drycleaner's discharges of tetrachloroethene to floor drains, drywells and cesspools resulted in soil and perched water contamination. Gross contamination was remediated using a vactor truck to excavate contaminated soil from the two floor drain areas, two drywells and three cesspools.

Because the soil conditions in the vicinity of the site are known to vary significantly, soil conditions were defined by collecting continuous soil samples using a truck-mounted drill rig, hollow stem augers and split spoons.

Five extraction wells were installed. The two wells installed in the floor drains were screened at 5 to 10 feet below grade, while the three deeper extraction wells installed in the vicinity of the drywells and cesspools were screened at 10 to 30 feet below grade.

Perched water was encountered at thirty feet below grade and extends to fifty feet where a significant clay layer was present which functions as an aquiclude.

BP Gasoline Service Station
Freeport, NY

Leaking gasoline tank piping and surface spills of gasoline have contaminated a 100 foot by 75 foot area of soil and the Upper Glacial Aquifer which is approximately 9 feet below grade. The active gasoline/service station has soils that are sand and gravel and the entire surface area is paved.

A Corrective Action Plan was prepared as part of the Stipulation Agreement that client signed with NYS Department of Environmental Conservation's Region 1.

SVES is being used to remediate the soils in the vadose zone and includes two extraction wells located on site that are screened from 2 to 7 feet below surface.

The groundwater will be remediated using an air sparging system which is currently being designed.

4. Inactive Hazardous Waste Disposal Site Experience

AEL has worked over fifteen New York State designated Inactive Hazardous Waste Disposal sites on Long Island. Also, AEL has worked on four Federal Superfund sites.

These Inactive Hazardous Waste Disposal sites include:

-New Cassel Industrial Area
-Simkins Industries
-Columbia Cement
-Nassau Uniform
-Flower Fashion
-IMC Magnetics
-I. W. Industries
-Arkwin Industries
-Atlas Graphics
-Utility Manufacturing

Our Federal Superfund site work includes:

-Anchor Chemical
-Fairchild Republic
-Tronic Plating

APPENDIX G

DRILLING LOGS FOR NASSAU COUNTY WELLS N5906 AND N2411

Nassau Uniform

Figure 1
Location of Nassau County wells in the vicinity of Nassau Uniforms

Anson Environmental Ltd.
Not to Scale

w-1561 **DUPLICATE—RETAIN**

N-59570
N-4517

State of New York
Department of Conservation
Division of Water Power and Control
COMPLETION REPORT—LONG ISLAND WELL.

Well No. N 5928
(on preliminary report)

LOG

Ground Surf., El. .... ft. above sea

.... ft.

Top of Well

Owner Aerodynamics Lab., Polytech Inst of B'lyn

Address 35 Livingston St., Brooklyn

Location of well Freeport

Depth of well below surface 85'6" feet

Depth to ground water from surface 2 feet

CASINGS:

Diameter 6 in. .... in. .... in. .... in.
Length 73 ft. .... ft. .... ft. .... ft.
Sealing Lead Packer
Casings removed none

SCREENS: Make Everdur Openings 18

Diameter 6 in. .... in. .... in. .... in.
Length 13 ft. .... ft. .... ft. .... ft.
Depth to top from top of casing 71'9" ft.

PUMPING TEST: Date 6-25-56 Test or permanent pump? T

Duration of Test .... days 8 hours
Maximum Discharge 30 gallons per minute
Static level prior to test 2 ft. .... in. below top of casing
Level during Max. Pumping 5 ft. .... in. below top of casing
Maximum Drawdown 3 ft.
Approx. time of return to normal level after cessation of pumping .... hours .... minutes

PUMP INSTALLED:

Type DWT Make Byron Jackson Model No. ....
Motive power Elec. Make U.S. Motor H.P. 5
Auth Capacity 25 g.p.m. against .... ft. of discharge head
No. bowls or stages .... ft. of total head

DROP LINE: **SUCTION LINE:**

Diameter .... in. .... in.
Length .... ft. .... ft.

Use of water Cooling

Work started June 6, 1956 Completed June 27, 1956

Date June 29, 1956 Driller C. W. Lauman & Co.

License No. 13

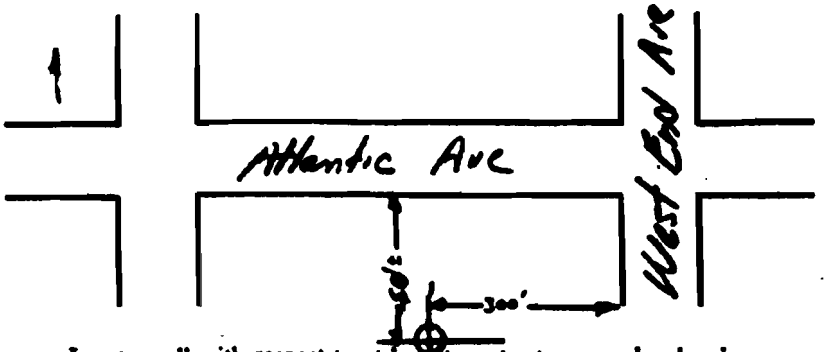
STATE OF NEW YORK
WATER POWER AND
CONTROL COMMISSION
RECEIVED
JUL 2 - 1956

NOTE: Show log of well—materials encountered, with depth below ground surface, water bearing beds and water levels in each, casings, screens, pump, additional pumping tests and other matters of interest. Describe repair job.

See Instructions as to Well Drillers' Licenses and Reports—pp. 5-7.

N5906

SKETCH OF LOCATION



Locate well with respect to at least two streets or roads, showing distance from corner and front of lot.

Show North Point

0-4 Bl Muck
4-9 Dirty cse brn SvG
9-25 Cln brn cse. sd, some grvl
25-27 Fi whitish-gry sd & small grvl
27-38 VF gry beach sd
38-40 Fi gry beach sd, lumps cl
40-50 Fi gry sd, cl, some grvl
50-52 Cse gry sd & cl
52-54 Fi dk gry beach sd
54-58 Fi gry beach sd
58-62 Fi whitish-gry beach sd
62-65 Fi gry beach sd, lumps cl
65-72 Fi dk gry beach sd
72-75 Fi gry beach sd
75-80 Fi dk gry beach sd
80-84 Cse gry sd
84-90 Cse sd w/ lig.
90-100 VF gry beach sd
100-105 Fi gry beach sd
105-110 Lig & pyr
110-111 DK gry sd

County NASSAU

ORIGINAL—TO COMMISSION

Well No. N-2411 (on preliminary report)

State of New York
Department of Conservation
Division of Water Power and Control

LOG

Ground Surf., El.....ft. above sea

COMPLETION REPORT—LONG ISLAND WELLS

A 0' ft.
V 0'
Top of Well

S.W.L. FLOWS

Owner BALDWIN BOAT BASIN

Address 651 ATLANTIC AVE BALDWIN L.I.

Location of well SAME

Depth below surface 84'-9" feet

Depth to water: Ground water FLOWS ft.; Finished well 84'-9" ft.

CASINGS:

Diameter	4"	in.	in.	in.
Length	50'	ft.	ft.	ft.
Sealing				
Casings removed				

SCREENS: Make COOK RED BRASS Openings .020"

Diameter	4"	in.	in.	in.
Length	5'	ft.	ft.	ft.
Depth to top from top of casing	79'-7"			ft.

PUMPING TEST: Date JULY 14 Test or ~~permanent pump~~?

Duration of Test 1 days 6 hours

Maximum Discharge 15 gallons per minute

Static Level Prior to Test FLOW ft. in. below top of casing

Level during Max. Pumping 8' ft. in. below top of casing

Maximum Drawdown 8' + ft.

Approx. time of return to normal level after cessation of pumping hours 5 minutes

PUMP INSTALLED: NONE (REPORTED BY OWNER)

Type ROTARY Make WESTCO Model No. —

Motive power ELEC. Make DIESEL H.P. /

Capacity 600 g.p.m. against } ft. of discharge head

No. bowls or stages / } ft. of total head

DROP LINE:		SUCTION LINE:	
Diameter	in.		in.
Length	ft.		ft.

Use of water DOMESTIC

Work started June 29 Completed July 17

Date OCT. 30 - 1947 Driller EASTERN WELL & PUMP CO.

License No. 251

JAP SCR 79'-7"

NOTE: Show log of well—materials encountered, with depth below ground surface, water-bearing beds and water levels in each, casings, screens, pump, additional pumping tests and other matters of interest. Describe repair job.

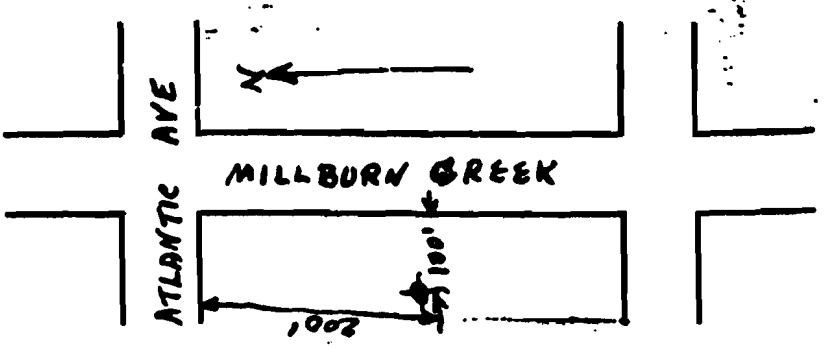
See Instructions as to Well Drillers' Licenses and Reports—p. 5-7.

SAND & FILL	1'
HARD BRN CLAY	3'
STINKY BOG	13'
WHITE SAND GRAVEL	24'
VERY FINE GRAY SAND	37'
FINE GRAY SAND GRITS GRAVEL	44'-6"
COARSE GRITS GRAVEL LITTLE SAND	46'
FINE WHITE SAND	48'
DARK GRAY CLAY	50'
DIRTY GRAY SAND, GRITS FEW GRAVEL	55'
HARD BLK. CLAY	59'
FINE DIRTY GRAY SAND	73'
DIRTY WHITE SAND	76'
MEDIUM COARSE SAND GRITS	83'
MED. FINE WHITE SAND	95'

STATE OF NEW YORK
WATER POWER &
NOV 1 - 1947
CONTROL COMMISSION
RECEIVED

N2411

SKETCH OF LOCATION



Locate well with respect to at least two streets or roads, showing distance from corner and front of lot.

Show North Point

APPENDIX H

EQUIPMENT PURCHASES
BY
NASSAU UNIFORM SERVICES

TELEPHONE: AC 516 MAyfair 3-6655
CABLE: AMPERCO · TWX 516-868-9042

AMERICAN PERMAC, INC.
EXECUTIVE OFFICES: 1569 MERRICK ROAD, MERRICK, N. Y.

PURCHASE AGREEMENT

December 21, 1964

1. American Permac, Inc. agrees to sell to Nassau Industrial Uniform Service, Inc., 525 Ray Street, Freeport, New York; subject to the terms herein provided:

   a. Two (2) 120 lb. SE Industrial Drycleaning machines 3 bath operation for complete automation of entire unit $45,000.00

   b. Titan 700 Industrial Dyrcleaning Machine $48,000.00

   c. Model 200 Activated Carbon Recovery Unit ..... $ 6,500.00

2. Nassau Industrial Uniform Service agrees to buy said machines subject to the terms herein provided, said selling price to include the above.

3. The prices mentioned above do not include freight or rigging, nor do they include the installation of the mentioned equipment.

4. Nassau Industrial Uniform Service agrees upon signing of this agreement to pay over to American Permac, Inc., a deposit of $10,000.00. Five thousand dollars of this deposit is to be applied to the SE 120 lb. machines plus the A-200 Activa. The balance due on these three machines is to be paid on delivery to Nassau Industrial Uniform Service. The remaining $5,000.00 of this deposit is to be applied to the remaining Titan 700 machine, which is to be delivered at a later date. The balance due on this unit is to be paid on delivery of the machine to Nassau Industrial Uniform Service.

PURCHASE AGREEMENT

December 21, 1964

5. American Permac, Inc. agrees to give to Nassau Industrial Uniform Service the warrantee of one year on defective parts and workmanship as it is passed on to American Permac, Inc. by the manufacturer, Boehler & Weber, K.G. American Permac, Inc. has agreed to furnish 90 days free service. American Permac, Inc. agrees to train operating personnel on the operating and maintaining of the machines.

Buyer _____ Seller _____

12/21/64
TS:awd

TELEPHONE: AC 516 MAyfair 3-6655
CABLE: AMPERCO

AMERICAN PERMAC, INC.
EXECUTIVE OFFICES: 1569 MERRICK ROAD, MERRICK, N.Y. 11566

August 16, 1965

Mr. Sol Zinn
Nassau Industrial Uniform Service
525 Ray Street
Freeport, L.I., New York

Dear Mr. Zinn:

Mr. Tony Spampinato has requested this letter be written to you covering certain guarantees and warrantees that are to be included in the purchase agreement of the Permac Industrial Cleaning Machine (330 SE).

1. The Permac 330 SE will carry the same guarantee on parts and defective workmanship as it is extended by Boehler & Weber KG on all equipment sold by American Permac, Inc. being:

a. Replacement of defective parts for a period of one year.

b. 5 year pro-rated guarantee on all stills.

c. 90 day free service from date of installation completion.

d. The same quality and standards are built into this unit, as shown on equipment demonstrated to you.

2. American Permac, Inc. warrantees, the model SE 330 Dry Cleaning Machine, will dry clean cotton pants, shirts, coveralls, jackets, wipers. Using water additives to leave garments with a moisture content for the purpose of finishing. Any additional moisture necessary for proper finishing, is built into

the finishing units supplied in conjunction with this order. The unit SE 330 will Dry Clean almost as clean as a washed cotton.

The above warrantees and guarantees are extended with the following exception. The unit will be operated and maintained in the manner outlined in the Installation, Operating Manual. Trichlorethylene and other additives recommended by American Permac, Inc. are to be used according to our directions.

Yours very truly,

AMERICAN PERMAC, INC.

Joseph Lascari
Sales Manager

JL/mm

TELEPHONE: AC 516 MAyfair 3-6655
CABLE: AMPERCO · TWX 516-868-9042

AMERICAN PERMAC, INC.
EXECUTIVE OFFICES: 1569 MERRICK ROAD, MERRICK, N. Y.

December 3, 1964

Mr. Martin H. Zinn
Nassau Industrial Uniform Service, Inc.
525 Ray Street
Freeport, New York

Dear Zinn:

We are pleased to quote the following prices of our Industrial Drycleaning machines:

2	120-SE Industrial Drycleaning machines 3 bath operation for complete automation of entire unit.	$45,000.00
1	Titan 700 Industrial Drycleaning Mach.	$48,000.00
1	Model A-200 Carbon Recovery Unit	$ 6,500.00

The latter unit, being our largest carbon recovery unit, will accomodate the Titan 700 Drycleaner when it is installed, as well as the two 120-pound machines.

As we are anxious to sell one of our larger activated carbon units, we are selling this unit to you at our cost. The price of this unit is $8,500.00. If you were to buy carbon units for each 120-pound unit at $2,500 per unit for the 120-pound machines, plus the carbon recovery unit for the Titan 700 at $5,200.00, the combined cost for carbon recovery units would by $10,200.00. The savings here would be $3,700.00 for carbon recovery units only.

The Model A-200 carbon recovery unit will perform the following functions automatically:

1. Absorb solvent fumes from any machine without being concerned with the cycle the machine is in.
2. Cook itself down automatically when the carbon bed is filled with solvent vaport.
3. Having two carbon beds, it switches from one bed to the other when the bed becomes saturated and strips itself of solvent.
4. The A-200 has its own solvent tank and pump. Solvent can be pumped to the desired machine when ever necessary.

Very truly yours,
AMERICAN PERMAC, INC.

Anthony Spampinato

AS:awd
A. Spampinato, Regional Sales Mgr.

October 6, 1975

Mr. Martin Zinn
Nassau Uniform Service
525 Ray Street
Freeport, New York 11520

Dear Mr. Zinn:

May I take this opportunity to thank you for your order for one (1) Brill Model X-40 oil skimmer. I know you will be more than pleased with its performance under heavy oil and grease conditions.

The Brill X-40 oil skimmer will be shipped via Yellow Freight from Cleveland, Ohio within the next few days. It will consist of one (1) crate whieghing approximately 100 Lbs. The unit is shipped dry, necessitating your people filling the gear box with enclosed oil. The motor voltage is connected forthe highest voltage in this case 230V-single phase, 60 cycle, and may be easily conmerted to 115V if desired.

The following spare parts are included in purchase price:

1- 14' oil collector tube
1- Ceramic drive wheel
4- Ceramic wear pieces

If you will notify me upon arrival I will make arrangements with Margaret Pritchard to be present during installation.

Sincerely,
Western Environmental Engineering

Gordon Fleisher:
President

GF:pg

1747 HANCOCK STREET • SAN DIEGO, CALIFORNIA 92101 • TELEPHONE: (714) 296-6524

September 12, 1975

Mr. Martin Zinn
Nassau Uniform Service
Freeport, New York 11520

Dear Mr. Zinn:

Enclosed is information on the oil and grease skimmer we would recommend for your particular oil and grease problem.

This unit should do the job and is the least expensive unit available today. After you have installed it and if for any reason it should not work, we will give you full credit on the cost of this unit (less collector belt cost) towards the belt skimmer. The Brill X-4U cost is $1495.00 F.O.B. Cleveland, Ohio. Terms 1%-10 days.

If you should favor us with your order, shipment will be made at once from Cleveland and Margaret Pritchard will be available to help in start-up.

Sincerely,
Western Environmental Engineering

Gordon Fleisher

Gordon Fleisher:
President

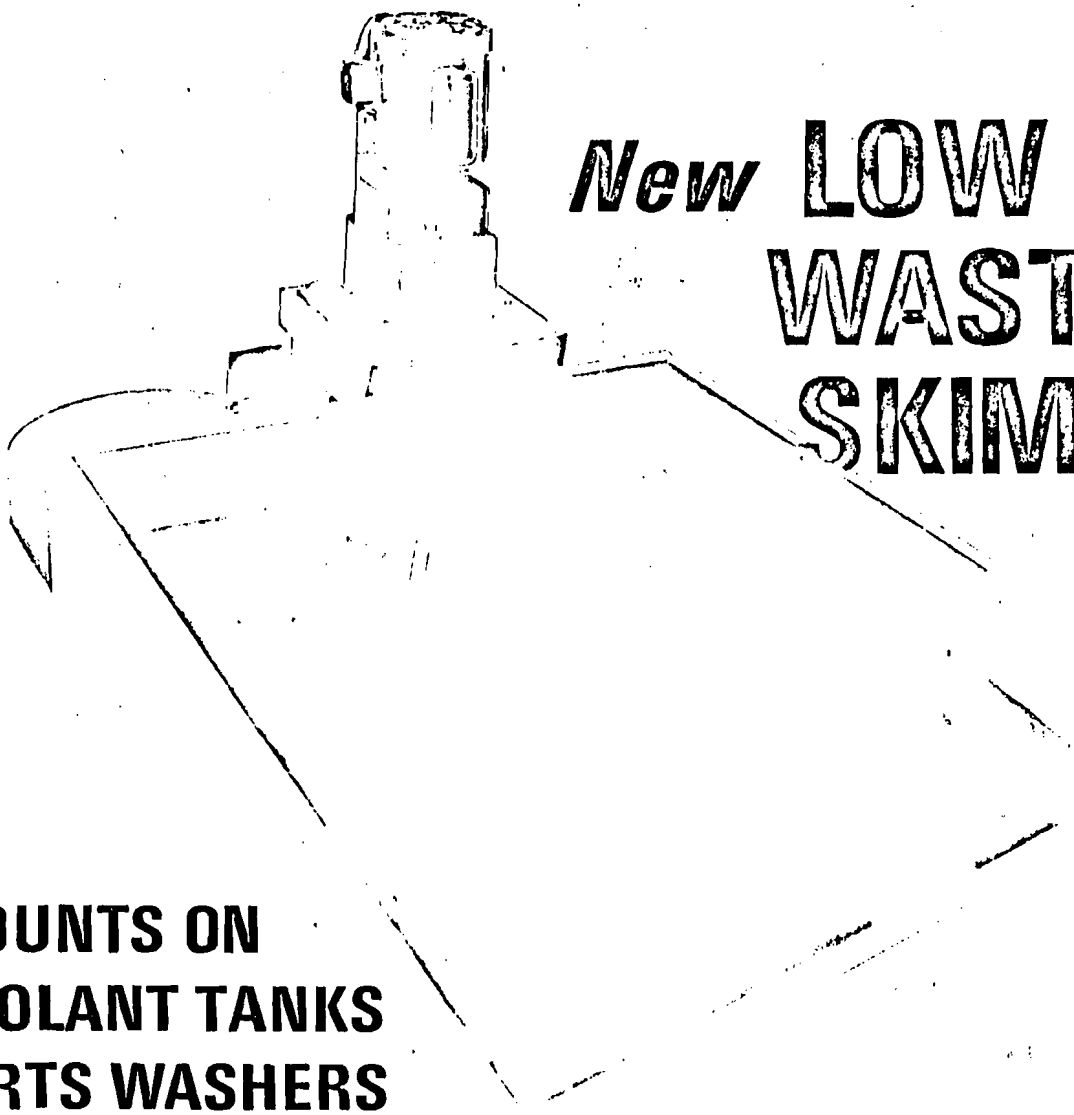
GF:pg
Encl.1

1747 HANCOCK STREET • SAN DIEGO, CALIFORNIA 92101 • TELEPHONE: (714) 296-6524

BRILL SKIMMER MODEL X-4U

WHAT IS YOUR PRESENT SKIMMING PROBLEM?

OIL, GREASE, MINERAL AND VEGETABLE OILS



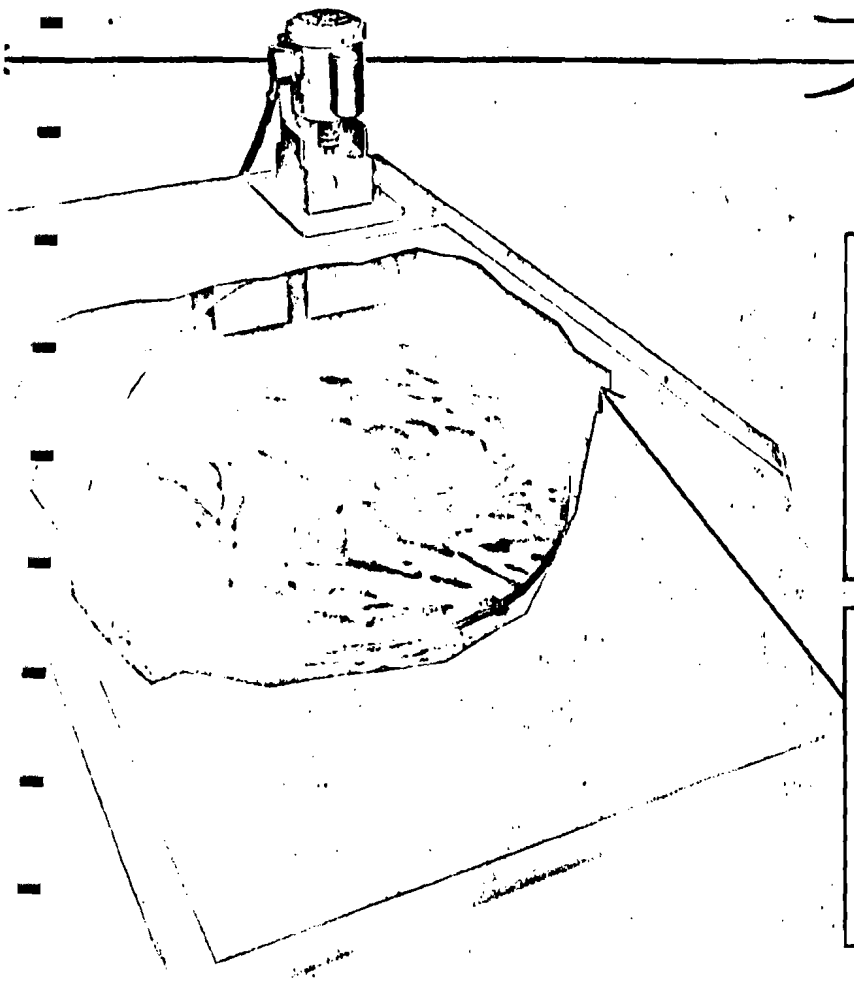
New LOW LIFT WASTE OIL SKIMMER

- MOUNTS ON
- COOLANT TANKS
- PARTS WASHERS
- PROCESS TANKS
- SETTLING TANKS

FOR SURVEY OF SERVICE
YOUR LOCAL SALES ENGINEER REPRESENTATIVE

WESTERN ENVIRONMENTAL ENGINEERING
1747 HANCOCK STREET
SAN DIEGO, CAL. 92101

Manufactured by
OIL SKIMMERS, INC



COVER ILLUSTRATION . . . Model X4U side mounted with an external discharge, commonly used when it is necessary to remove oily materials such as encountered in . . . COOLANT SYSTEMS and PARTS WASHERS

MODEL X4U Top mounted with an internal discharge, commonly used in FUME SCRUBBER applications to maintain a positive or negative atmospheric condition on existing tanks where internal troughs are already in use.

BRILL SKIMMER MODEL X4U

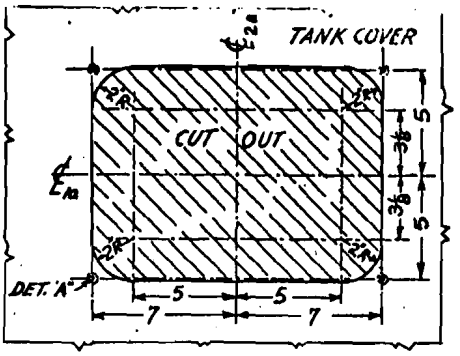
is used in low lift applications (3" to 36") to remove floating oils from water and aqueous solutions in above floor tanks and vats. The oil is picked up by selective adhesion, oils cling to a special formulated plastic tube that is in constant contact with the liquid, this tubing is continually scraped and the oil is stripped from the tube into a trough. The tubing is hollow and floats, adjusting automatically to fluctuating liquid levels.

BRILL SKIMMERS are designed to withstand a wide variety of chemicals over an extreme range of temperatures.

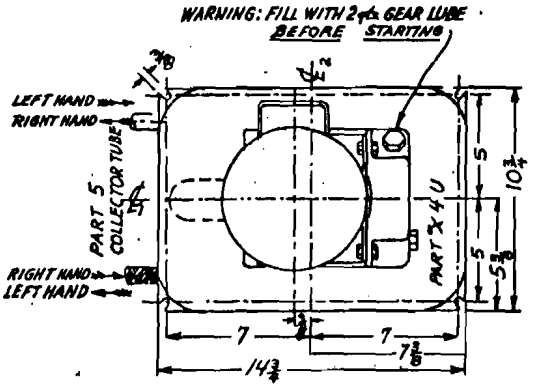
THE MODEL X4U is particularly adapted to COOLANT SYSTEMS, PARTS WASHERS, FUME SCRUBBERS, WET DUST COLLECTORS and general plant service where a compact, reliable and versatile skimmer is required . . . . it is an important factor in rounding out your quality control program and aids in solving air and water pollution problems.

The capacity of the Model X4U is low compared to the Model T6 BRILL SKIMMER, which is used in high lift applications, however its recovery rate is more than adequate compared to other methods, light oils will be skimmed at a rate of 72 GPD, Medium Oils at 240 GPD and Heavy Oils and Greases at 400 GPD.

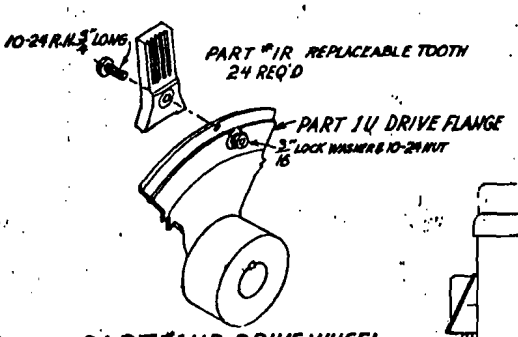
PITS, SUMPS and LAGOON applications can be solved with the use of the Model T6 BRILL SKIMMER.



PLAN VIEW of THE OPENING & BOLT LAYOUT FOR MOUNTING MODEL X4U

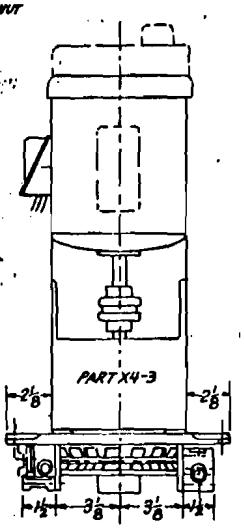


TOP VIEW
STD. RIGHT HAND ARRANGEMENT

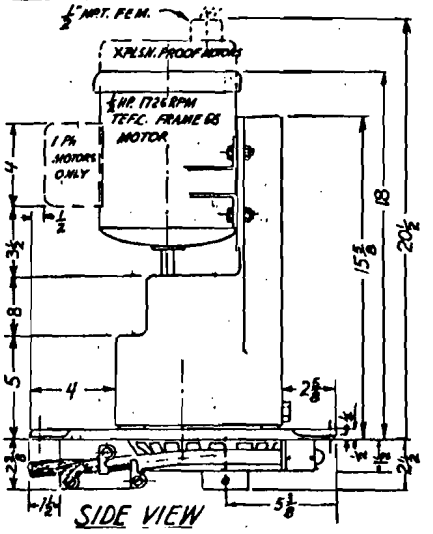


PART #1UR DRIVE WHEEL ASSEMBLY

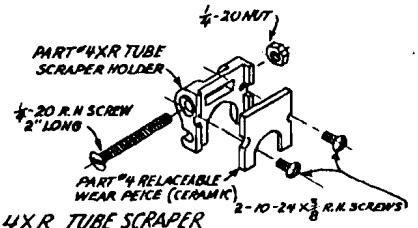
NOTE: USE #1UR WHEEL FOR HIGH ABRASIVE OR HIGH OR LOW PH.



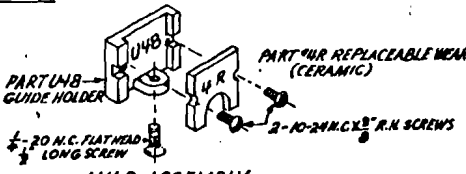
FRONT VIEW



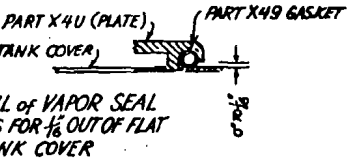
SIDE VIEW



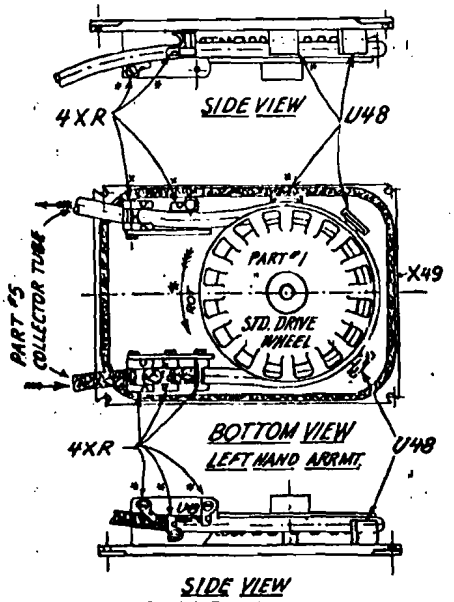
4XR TUBE SCRAPER ASSEMBLY



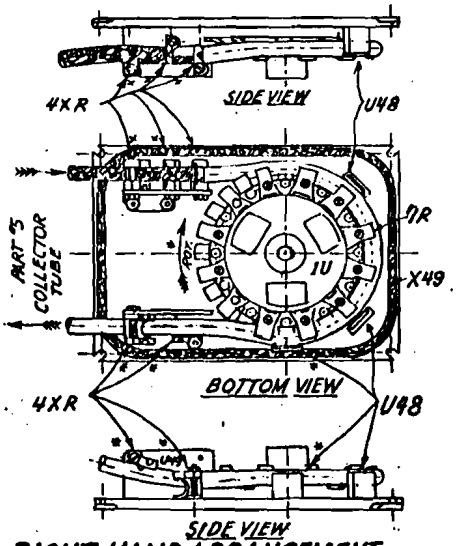
U48 ASSEMBLY



DETAIL of VAPOR SEAL ALLOWS FOR 1/16" OUT OF FLAT ON TANK COVER



LEFT HAND ARRANGEMENT



RIGHT HAND ARRANGEMENT

DIMENSIONS AND PART OF MODEL X4U	
BRILL SKIMMER	DRAWING NUMBER 740819

Why is buying a BRILL SKIMMER the best value?

PERFORMANCE ... it works where others fail. Because of the unique new method of using a FREE FLOATING COLLECTOR TUBE, floating solids and changing liquid levels will not stop the BRILL SKIMMERS from skimming.

CONVENIENT .... Compact model X4U mounts away from congested areas. Only the FREE FLOATING COLLECTOR TUBING floats in the skimming area.

DURABLE ..... New CERAMIC (space age) wear parts will last for years. Heat resistant tubing greatly reduces down time.

EFFICIENT ..... vertical scrapers are used so that waste oils, froth, gunk heavy greases can be DROPPED into collection drum without the use of pumps.
Self adjusting scrapers.

SAVINGS ..... Eliminates costly overflow method of waste oil removal, which removes large amounts of water along with the oil and requires an operator.

Removes oily gum that coats machinery, grinding wheels, and filter media, cuts replacement costs.

Eliminates pipeline build up, plugged sprays, less detergent use.

Eliminates a major culture medium for bacteria in COOLANT SYSTEMS.

Eliminates oily film on washed parts that causes smoke in heat treat furnaces and oily coating on plant ceiling and walls, aids in meeting pollution standards.

AUTOMATIC .... Operates continuously without float switches or timers.

WESTERN ENVIRONMENTAL ENGINEERING
1747 HANCOCK STREET
SAN DIEGO, CAL. 92101
714-296-6524

STOCK ON SHELF
S.O.S. Delivery

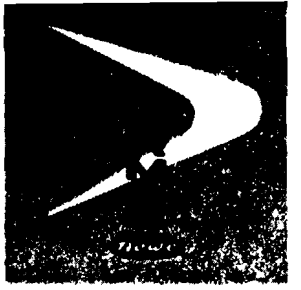
Manufactured by OIL SKIMMERS, INC.
[illegible] CLEVELAND, OHIO [illegible]
PHONE [illegible]

Qty	Description	Serial #	Actual Cost	Date	
line 8020	Permac-Bowe- D/C Plant — Type SE 50ME	180/6507	2250000		
8021	" " " " — Type SE 50ME	181/6507	2250000		
1	Cissell Form Finisher	21672	54820		
1	A.R.U.-46 Concord Automatic Legger	628-Z137-M-31426	149625		
2	42X42 Super Thermatic Tumblers - 6 coil	FT-12507 / FT-12508	253550		
2	D.T.F. Ajax Automatic Pants Toppers	AA-5653044 / AA-5653043	209000		
1	Class 2231 40X36 Cascade Washer Ex 135 lb with Super Jetts Mount	2231-4-2014	984675		
1	Cissell Form Finisher	26747	52820		
1	Model D-4 B-Vac Air Vacuum System	E-092737 motor # 71637B-RB	38000		
1	ALV-46 Concord Automatic Legger	628-Z137-M-31392	149625		
2	C 2800 C-5M-22-80HP Cyclotherm Steam Generators		1298100		
1	Condensate Return System + Water Pump Set	model 154-5	103000		
1	Ingersoll Rand Air Compressor (Code 9678) Type 30 Model 20TA	30T-208904	199500		
2	Over & Under Dyna-Pak Presses - Class 627 - Model 33-9"	627Z 234-M-34801 / 627Z 234-M-34802	307800		
1	Formatic Industrial Work + Dress Shirt - Class 625	6252-M-3123	807025		
2	Formatic Sleeve Finisher - Class 620	6202-74-M-32494 / 6202-74-M-32493	256025		
2	Over + Under Dyna-Pak Presses - Class 627 - Model 33-9"	6272-234M-34994 / 6272-234M-35061	307800		
	Installation of all Machinery		1500000		
1	Formatic Industrial Work Dress Shirt Class 625	6252	807025		
4	Double Sleeve Forms - Class 026		114342		
4					
2	Collar Forms - Code 5464	618-0627	27540		
1	Cissell Puffer Set - 1M	77233	7844		
1	Vibra Steamer - Auto Feed - Model LD / WD Left hand feed 208V 3PH 60 cycle	15371 (Cummings-Laundry)	1512445		
1	Air Pollution Control System - 440/60/3 Phase S/N - VIC Tank A / Tank B	7330652-8 / H-36343688-2072 / H-36-6963-44-1527	1167500		
~~1~~	~~Spencer D/C Machine - Model GT-165TT~~				
1	Spencer D/C Machine - Model GT-165TT	358-P757	3825000		
1	White Machine Company - Conveyor-V-Vega Part 4 200-1	CKT-A45V-1 P4 MAN Model 66 [illegible]	2075000		
1	" " Idler Section	[illegible]	83100		
1	Smokeveyor - Regent Hand		209500		
1	Pantveyor - ~~[illegible]~~		229500		
1	42" Huebsch 6 coil steam Tumbler - Super Model	FTS-25946-TN79582	312886	2/16/78	
1	400 lb. Washer-washer Extractor - New				
1	Water Tower				
1	400 lb Washer - washer Extractor - from Cummings			3/10/78	
1	SI 140 Permac - Recork - from Ray/Verne	Trade in 7500.00 net	2750000 / tax 140000 ? / 2000000 ?	6/20/79	(Ser # 506/2006 - Make

Copy

TELEPHONE: AC 516 MAyfair 3-6655

AMERICAN PERMAC, INC.
EXECUTIVE OFFICES: 1666 MERRICK ROAD, MERRICK, N. Y. 11566



INVOICE

This bill is assigned and payable in New York bankable funds only to our factors, Intercontinental Credit Corporation, Division of Pan American Trade Development Corp., 2 Park Ave., New York, N. Y. 10016

Machine #8013

DATE 8/31/65

TO: NASSAU INDUSTRIAL UNIFORM SERVICES INC.
525 Ray St.
FREEPORT, N.Y.

TERMS: 20,000 S/D
20,000 30 days after del. CSC T/A
8,000 90 days after del. CSC T/A

No. 8013

One drycleaning plant Permac Boewe
Type R/150 M Color: Dk. blue Titan 330lbs.
Serial No. 102/6507
Steamheated $48,000.00

spampinato
marks 8013
from: N.Y. pier

Rel 20,000.00 Sight Draft
9-22-65 22,000.00 check
6000.00 deposit

48000.00

All claims must be made to Intercontinental Credit Corporation, 2 Park Avenue, New York, N. Y. 10016 within 5 days after receipt of goods. If the terms of this bill are not in accordance with those made at the time of sale, please return the bill at once for correction to Intercontinental Credit Corporation, 2 Park Avenue, New York, N. Y. 10016, as we cannot make any change at time of settlement. Interest charges on overdue accounts are incurred at the rate of 6% per annum. Goods delivered to express companies or freight lines are at the risk of purchaser.

Sheet No. _____ Account No. _____

Terms

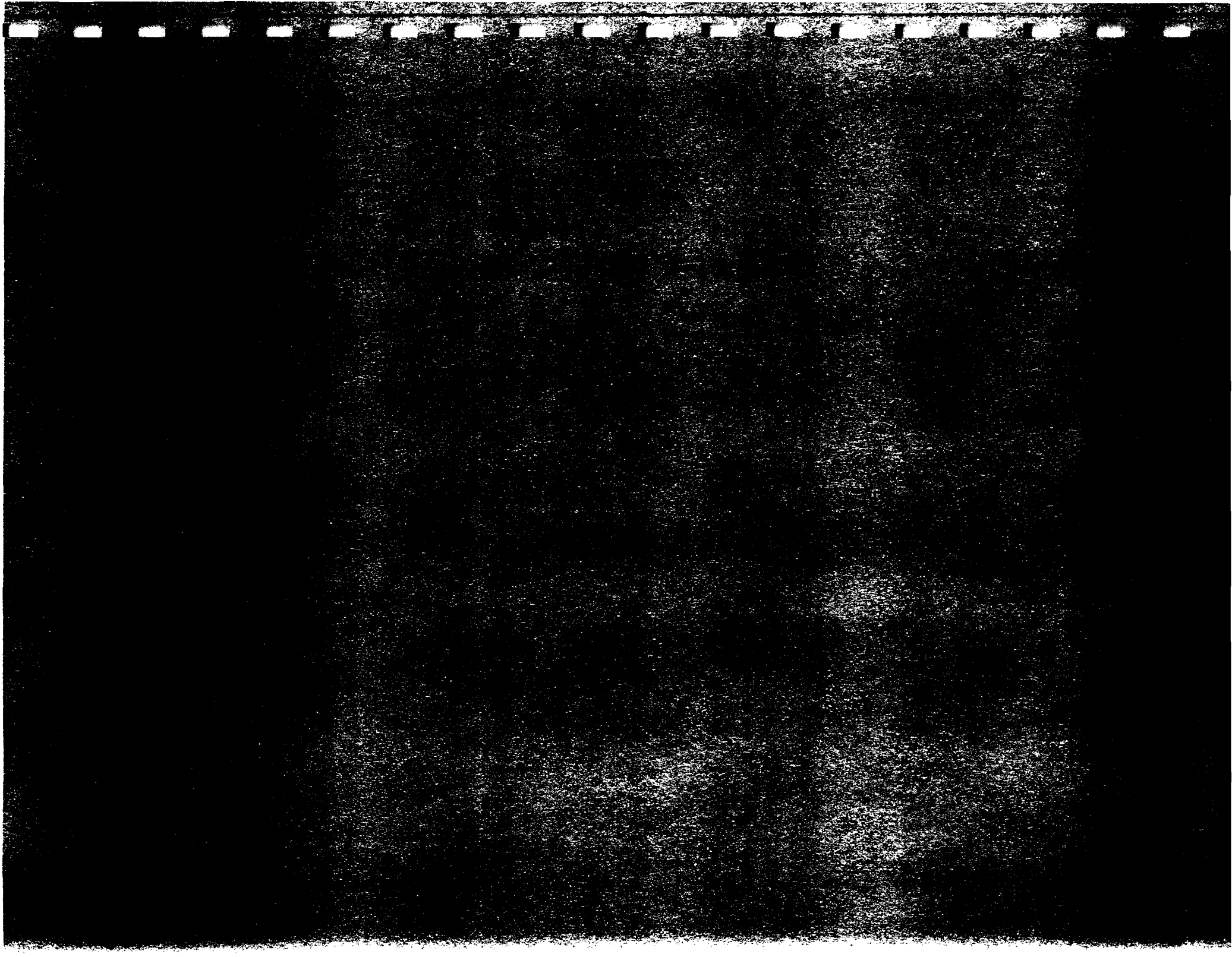
Rating

Credit Limit

NAME Nassau Industrial Dry Cleaning Corp

ADDRESS Machinery Schedule

17-450 HAMMERMILL LEDGER	DATE 19__	ITEMS	FOLIO	✓	DEBITS	DATE 19__	ITEMS	FOLIO	✓	CREDITS
Machine # 8012 1	Permac	Boewe - dry cleaning plant			Type SE 50 ME		Serial # 180/6507			2250 00
8014 1	"	" " " "			SE 50 ME		" " 181/6505			2250 00
returned 8013 1	"	" " " "			R 160 M		" " 102/6507			4800 00
1	Cissell	Form Finisher Serial # 26742								548 00
rec't 2	Colmac	Pants Press Units (4 units)			Serial # S		4145 - L1456, L1457, L1458, L1459			2029 20
1	ALU 46	Concord Automatic Jigger			" "		628 - Z137 - M-31426			1496 62
2	42 x 42	Super Thermatic Tumblers			6 Coil		FT-12507 FT12507 FT12508			2555 52
2	D.T.F.	Ajax Automatic Pants Topper			Serial # S		AA5653014 - AA5653013			2090 00
Rec't 1	Class	2231 40x36 Cascade Washer			Ex L 135 LA		with Super Jtr Mount serial # 2231-mod			9846
1	Cissell	Form Finisher Serial # 26747								548 00
rec't 1	Cissell	Spotting Board Air Vacuum Model A			Serial # 11924					327
1	Model	1-4 B-Ae Air Vacuum Spotter			Serial # E092737 Motor # 71637B-CG					380 00
1	ALU 46	Concord Automatic Jigger			Serial # 628-Z137-M-31392					1496 62
2	C2 800	C-5N-23 80 HP Cyclotherm			Steam generators					12981 00
1	Model	154-5 Condensate Return System			+ water pump set					1030 00
	(Code 9678)	Type # 30 Model 4 30T A			Ingersoll Rand Air Compressor		Serial # 30T 204744			1993 00
2	Class 627	Model 33-9" over & under Dyna-Pak			Presses		234-M-3402, 6272-234-M-34801			3078 00
rec't 1	Class 625	Formatic Industrial Work & Dress			shirt		M3126 6252			807 00
Rec't 2	Class 620	Formatic sleeve finisher			6202 74 M 32492		6202 74-M-32493			2560 00
9	Class 89	Semi Automatic Shirt Folder			8917 M 31211		8917-M-31245 34994			1135 00
2	Class 627	Model 33-9 Over & Under Dyna-Pak			Presses		6272, 234M-35041, 6272 204M			3078 00
		Installation of all machinery								1500 00
1	Class 625	Formatic Industrial Work & Dress shirt					6252 M			807 00
4	Class 626	Double Sleeve Forms								1143 00
2	Amp Boys	Code 5467 626-0616 & Collar Forms					Code 5464 618-0627			275 00
returned 1	Invoice	# 7656 Dated 12/31/65 (Sniffer)					M79/6507 A50 A-680 10V-651V			6066
1	Cissell	1 m Puffer Set			Serial # 77233					18
1	Activated	Carbon Recovery Unit			A50 Serial # 279/6507					2403
1					A50					1981

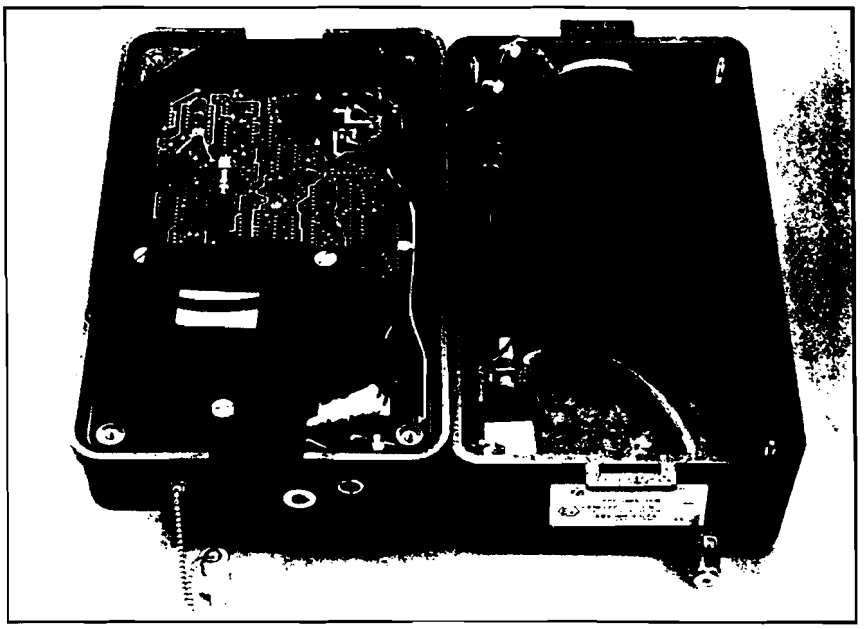


APPENDIX I

FIELD INSTRUMENTS INFORMATION SHEETS

Thermo Environmental Instruments Inc.

MODEL 580S INTRINSICALLY SAFE Organic Vapor Meter Data Logger



CENELEC approved design features an explosion proof detector/lamp housing for operator safety in explosive atmospheres.

The Model 580S Organic Vapor Meter (OVM) combines a highly sensitive photoionization detector, a built-in data logger, and an intrinsically safe design to provide operator safety during field measurement of toxic vapors in explosive environments.

The Institute of National Industry Extractive (INIEX) has tested the Model 580S and has issued CENELEC approval for class EEx d ib IIC T4. The energy limiting protective electrical design is incapable, under normal or abnormal conditions, of causing ignition of hazardous mixtures in the air.

Single-hand operation facilitates isolation of leaking VOCs from petroleum and chemical processing plants.

KEY FEATURES

- Auto Data Logging provides storage of up to 700 data points by date, time, location and alarm status.
- Maximum concentration signal hold.
- Simplified auto-calibration routine.
- Positive displacement pump enables sample collection and remote detection of organic vapors.
- Full 8-hour portable operation from internal lead acid battery.
- RS-232 port allows hard copy generation of collected data.
- Response Factor Settability for direct reading of individual chemical species sample concentrations.
- Detector guard electrode minimizes moisture interference in high humidity environments.
- Interchangeable lamps maximizes number of observable species and compounds.
- Visual display and audible indication of alarm level exceedances.

Specifications

Measurement:	
Technique:	Photoionization Detection of most organic vapors and some inorganic gases are possible – See OSHA Chart.
Ranges:	Digital Readout (LCD) – auto Ranging 0-200 ppm (resolution to 0.1 ppm) 200-2000 ppm (resolution to 1.0 ppm)
Minimum Detectable:	0.1 ppm benzene in air matrix.
Sensitivity:	0.1 ppm benzene on 0-200 ppm scale.
System Time Constant:	2.0 sec. at 300 ml/min. sample flow
Sampling Rate:	Nominal Flow 300 ml/min.
Sample Conditioning:	Changeable ten micron filter on inlet.
Power Requirements:	
Battery:	Internally rechargeable (external charger provided with unit).
Service Life:	8 hours per internal battery charge, operates from charger indefinitely.
Charger Requirements:	115/220 VAC, 60/50 Hz, 4 watts maximum.
Controls, Panel:	
Readout:	Two line alphanumeric display with bargraph.
Keypad:	Seven Touch Pads : PWR, MODE, RESET, LIGHT, +, –, SPKR.
Other Features:	
Audible Alarm:	80 db audible alarm mounted on front panel.
Earphone:	For operation in noisy environment.
Physical	
Case Size:	7.5″ × 5.75″ × 10.0″ (HWD).
Weight:	7.5 lbs.
Communication:	RS-232 port.

Available Options

- Calibration Kit
- Carrying case
- IBM-PC and compatible Communciations Software
- Dilution Probe

For Price and Delivery Information, Contact:

Thermo Environmental Instruments Inc.

8 West Forge Parkway
Franklin, MA 02038

(508) 520-0430
Telex: 200205 THEMO UR

FAX: (508) 520-1460

Solinst *Water Level Meter:* Operating Instructions

Models 101 & 102

Upon receipt of meter the following operational checks should be performed:

Set toggle switch to "on" position, or turn rotary dial fully clockwise.

Circuity check: submerse the electrode (probe) in tap water. This completes the circuit and activates the buzzer.

Test button check: depress button to test the battery and circuitry (excluding the probe).

Routine Care of the Water Level Meter:

The probe and reel assembly is a simple system that will give long and reliable service, if handled with reasonable care.

1. After the depth of water has been recorded the cable should be carefully rewound onto the reel, the probe wiped dry and replaced into the probe holder.
2. The probe, cable and reel can all be cleaned with soap/ detergent and water.
3. Use of a Water Level Meter Carrying Bag adds to the service life of the meter.

NOTES:

Zero measurement point on Model 101 Water Level Meter is at tip of inner electrode, visible near center of probe.

Zero measurement point on Model 102 Water Level Meter is at base of outer body electrode.

The P4 probe has been designed to allow substantial submergence. Use of the P1, P2 or P3 probes to sound the bottom of the well may cause water to enter the probe.

Please call Solinst if any repairs are required:
(416) 873-2255

Water Level Meter Maintenance

SENSITIVITY ADJUSTMENT

- clockwise rotation of rotary dial turns meter on and increases sensitivity.
- always set switch to the highest sensitivity position, then decrease if necessary.

SYMPTOM	CAUSE	REMEDY
No sound when probe immersed in water.	Dead battery.	Replace with 9v Alkaline.
	Water conductivity is very low.	Increase sensitivity switch setting (turn clockwise) or call Solinst for assistance.
	Disconnected wires on circuit board.	Check all connections inside hub of reel for loose/disconnected wires - solder or reconnect.
	Broken wire in tape.	Locate break in tape - splice and seal.
	Disconnected wire inside probe.	Contact Solinst to obtain parts / repair instructions.
Continuous sound after probe is removed from water.	Water conductivity is very high.	Decrease sensitivity switch setting (turn counter-clockwise).
	Damaged components or improper wiring on circuit board.	Contact Solinst to obtain parts / repair instructions.

BATTERY REPLACEMENT

- battery type - alkaline, 9 volt.
- the battery is housed in the reel hub and is replaced by removing the front plate of the reel.
- to remove front plate, unscrew three faceplate screws and carefully lift off to the side to avoid damage to wiring.
- remove battery and put in new one, making sure the polarity is correct.
- replace faceplate of the reel and screws, making sure the wires are fully inside.

Water Level Meter Replacement Parts:

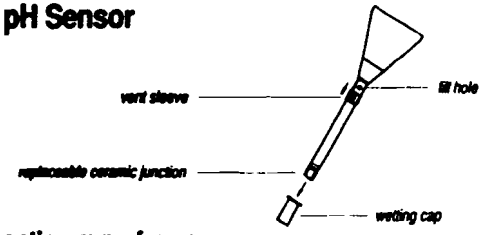
The following parts can be provided should they become lost or damaged.

- probes and probe tips
- tapes and cables
- cable reels
- lights, switches, etc.

Sensor Information

r direct fitting and leaded pH, DO and Conductivity Sensors

pH Sensor



r optimum performance:

Before use remove wetting cap from tip of sensor, and slide the vent sleeve to expose the fill hole.

Make sure that the fill solution is not more than 25 mm (1 inch) below the fill hole. Add KCl solution if necessary.

Gently tap the sensor to remove any air bubbles at the ceramic junction.

Condition the new sensor by soaking in pH 7 buffer for 2 hours. Prolonged soaking is not recommended.

Calibrate and measure samples as described in the M90 instructions.

Allow sufficient time for the sensor to stabilize when measuring samples of different temperatures, or of low ionic strength. Manual endpointing is advised with these samples.

After use, check the level of fill solution, reposition the vent sleeve to cover the fill hole, and replace the wetting cap containing pH 7 buffer (if the sensor will not be used again for more than 2 days, we recommend using saturated KCl in the wetting cap).

cautions and Limitations:

Do not wipe the sensor tip – blot dry with a lint-free tissue.

Do not use KCl saturated with AgCl as this may damage the reference element.

Do not leave the sensor in organic solvents, strong basic solutions, concentrated fluoride solutions, or hydrofluoric acid for extended periods. Measurements made in these solutions should be taken quickly and the sensor rinsed immediately with distilled water. After rinsing, soak in pH 7 buffer for 2 hours.

Do not measure solutions that exceed a temperature range of 0 – 100°C.

intenance and Troubleshooting:

nged use and ageing may reduce performance i.e. slow response, low values, continuous drift or erratic readings. These may be caused by:

n junction – remove air bubbles by gentle tapping.

ss KCl crystals – KCl crystals may build up and settle on the sensor r the KCl may become discolored. Remove the old fill solution and use distilled water to dissolve the crystals. Remove water and refill using KCl solution.

ked junction – KCl crystals can block the junction. To test for this, blot p dry and air dry for one hour. If no KCl crystals appear at the tip of the or the junction is blocked. Remove the ceramic junction using tweezers, nsert new junction (Cat. 477269). Tap gently to remove any air bubbles.

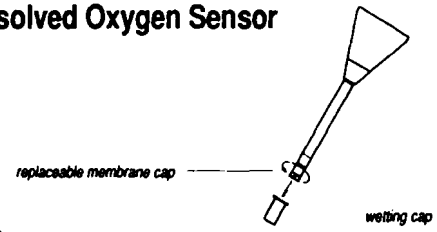
minated pH bulb – i.e. protein/oil contamination.

in – soa... ...nsor i... ...epsin ... adjus... ...H 2 wi...

for 30 minutes. Rinse with distilled water and soak in pH 7 buffer for 2 hours.

Oil – wash sensor tip with 50% water-acetone solution. ***Do not*** soak the sensor in acetone solution as this may cause the seals to deteriorate. Rinse with distilled water and soak in pH 7 buffer for 2 hours.

• Dissolved Oxygen Sensor



Installation:

DO membrane caps are fragile. Handle with care to prevent damage.

The sensor is shipped dry and must be filled before use. Unscrew the membrane cap from the sensor. If the silver/gold tip is tarnished clean carefully using electrode cleaning compound or silver polish, paying particular attention to the gold cathode. Rinse tip with DO electrolyte, and fill membrane cap, avoiding air bubbles. Hold the sensor vertically and gently screw the membrane cap onto the sensor, allowing surplus electrolyte to run out. Fit sensor to the meter and allow 1 hour minimum for polarization. Calibrate as described in M90 instructions.

For optimum performance:

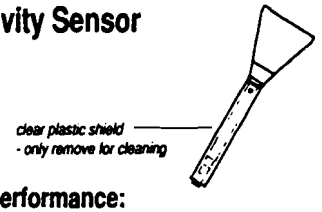
1. Before use remove wetting cap from tip of sensor.
2. For immediate use the sensor should be kept connected to the meter. The sensor may be removed for up to 3 hours as a rechargeable battery in the sensor will maintain polarization. For extended storage remove the membrane cap and rinse with water, and clean the sensor tip. Store dry with the membrane cap loosely fitted. Do not fit wetting cap.
3. When making measurements the sample should be stirred at a constant speed i.e. approximately 20 cm/second (8 inches/second).
4. Allow sufficient time for the sensor to stabilize when measuring samples of different temperatures – in some cases this can be several minutes. Manual endpointing is advised with these samples. Make sure the sensor is immersed to a depth of at least 40 mm (1.5 inches) to cover the temperature sensing element.
5. After use replace wetting cap containing distilled water to prevent electrolyte from drying out.
6. Regular maintenance is important to ensure optimum performance. Replacement of membrane caps depends on usage - we recommend replacement every 2 to 4 weeks.

Maintenance and Troubleshooting:

If the sensor will not calibrate, or becomes sluggish or erratic:

1. The silver/gold sensor tip may become tarnished with time. For optimum performance clean tip and refill cap every 2 weeks as described in Installation.
2. The zero oxygen solution will absorb oxygen if left exposed to air and this will cause inaccurate calibration. Use fresh zero oxygen solution.
3. Make sure there are no air bubbles inside the membrane cap when filling with DO electrolyte. Check by looking up through the membrane from the bottom of the sensor.
4. Check the membrane for damage and replace with new cap (Cat. 473626) as necessary.

• Conductivity Sensor



For optimum performance:

1. Make sure the clear plastic shield is in place when measuring.
2. When measuring make sure the solution is above the cell chamber rings and below the vent hole.
3. To prevent carryover from high to low conductivity solutions rinse with distilled water between measurements.
4. Make sure the cell chamber is bubble free when measuring. To reduce air bubbles, immerse probe in the solution at an angle and then raise to a vertical position.
5. Allow sufficient time for the sensor to stabilize when measuring samples of different temperatures. Manual endpointing is advised with these samples.
6. The sensor is not recommended for low ionic strength solutions (<5 mg/L).
7. Clean the probe and shield with distilled water after use.

General Troubleshooting for all Sensors:

1. To verify meter is working check using the test plug.
2. If the sensor connector becomes damaged or wet the display may read E4 when a sensor is connected.
3. If the temperature sensing element becomes damaged the temperature display may read E1 when a sensor is connected.

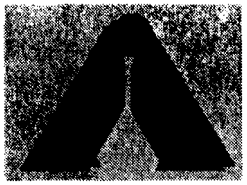
Ordering Information:

Item	Cat.
pH sensor	473619
pH electrode fill solution, 3 x 5 mL	473654
pH 7 buffer sachet (pack of 30)	473650
pH 4 buffer sachet (pack of 30)	473651
pH 10 buffer sachet (pack of 30)	473652
pH multipack, pH 4, 7 and 10 (pack of 30 assorted sachets)	473676
Buffer solution pH 4.00, 2 x 500 mL (red)	478540
Buffer solution pH 7.00, 2 x 500 mL (yellow)	478570
Buffer solution pH 10.01, 2 x 500 mL (blue)	478510
Buffer rainbow pack, pH 4.00, 7.00 and 10.01 (2 x 500 mL of each)	478574
Replaceable ceramic junctions (pH), pack of 3	477269
DO sensor	473620
DO electrolyte, 3 x 5 mL	474594
Zero oxygen solution, 500 mL	473625
DO membrane replacement kit, pack of 2	473626
pO_2 electrode cleaning compound	477656
Conductivity/TDS sensor	473621
1413 µS conductivity standard, 500 mL	473623
12.88 mS conductivity standard, 500 mL	473624
Rinse solution sachet (pack of 30)	473653

Corning Incorporated
Science Products Division
Corning, New York 14831
USA
Tel: 1-607-737-1667
Technical Information Center: 1-(800)-222-7740

APPENDIX J

ACCREDITED LABORATORIES, INC.
STATEMENT
OF
QUALIFICATIONS



ACCREDITED LABORATORIES, INC.

Implementing Tomorrow's Technology, Today™....

Statement of Qualifications

CORPORATE OFFICES: 800-254-LABS 908-541-2025 FAX 908-541-1383
Foot of Pershing Avenue P.O. Box 369 Carteret, New Jersey 07008-0369

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MISSION STATEMENT

The Accredited Laboratories "Strategic Alliance" program is designed to integrate and maximize the resources of the Company and the Client. Our commitment is to offer a variety of comprehensive Analytical, Field Sampling & Testing and Data Management service programs to support current environmental investigations, remedial action programs and regulatory compliance efforts. Our Strategy is to provide cost effective systems which optimize opportunities to meet the competitive challenges of the 90's and the 21st century.

INTRODUCTION

INTRODUCTION

Accredited Laboratories, Inc. (ALI), is a premier environmental laboratory located in Carteret, New Jersey. Established in 1974 as a captive source of analytical services for its former parent company, AMAX, Inc., ALI has become one of the leading full-service environmental laboratories in the Northeast. Its services are as diverse as the environmental requirements that exist today, including air, water, soil, and solid waste analyses.

In 1986, ALI became a wholly-owned subsidiary of AMAX Base Metals Research and Development Group to fill the growing need for analytical services resulting from the proliferation of new and more stringent federal and state regulations. ALI offers full service environmental analyses and field sampling to industrial facilities, municipal and government projects, academic institutions and consulting engineering firms.

On July 8, 1994, ALI was purchased by ARTL Limited Liability, Inc., a corporation founded by Theodore C. Gaydos. Mr. Gaydos served as Accredited's General/Laboratory Manager since 1988, and is an expert in GC/MS and systems automation. In 1993, Mr. Gaydos developed the Syscom*DMP Data Management System, a proprietary Software Program that is second to none. The System is designed to quantify and publish Final Reports of Results, Customized Data Summaries and QA/QC Summary Data Sheets directly from the analytical instrumentation raw quantification files in hardcopy and/or diskette deliverable formats. The program eliminates all requirements for physical manipulation, transcription and reproduction of data packages by clients.

In addition to providing experience, superior client services and sophisticated technology, ALI offers a wide variety of technical disciplines, including:

- Environmental monitoring
- Water, wastewater, soil and sludge analyses
- Analyses of "non-routine" matrices
- Regulatory compliance and permitting
- Environmental sampling services
- Industrial hygiene sampling and analysis

ALI is committed to providing customer service that exceeds the expectations of its clients. Quality service is shaped not only by a combination of sophisticated equipment and experienced personnel, but by the attitude that anything less than the best is completely unacceptable. In today's environmental field, high stakes demand high laboratory standards, consistently reliable data that meet all regulatory standards, quick turnaround, fair prices, and confidentiality.

ALI delivers all of this, along with perhaps the most important service of all - the willingness to do whatever is necessary to assure that each customer's unique requirements are met. This customized approach means that ALI's personnel are always available to establish sampling procedures, discuss test methodologies, interpret results, or handle special requests for customized analytical procedures, reporting or turnaround.

TECHNICAL CAPABILITIES

INSTRUMENTATION

LIST OF MAJOR INSTRUMENTS AND EQUIPMENT

A. Six Gas Chromatograph-Mass Spectrometers equipped with RTE data handling system:

1. Hewlett-Packard A-Series Data Handling System:

   * HP-5890 GC and 5970B MSD (Serial #3033A31866) equipped with Envirochem Thermal Desorption Unit (Model 1260) and Tekmar LSC 2000, ALS 2106 and Sample Heater.

   * HP-5890 GC and 5970B MSD (Serial #2905A11996) equipped with Tekmar LSC 2000, ALS 2016 and Sample Heater.

2. Hewlett-Packard E-Series Data Handling System:

   * HP-5890 GC and 5970B MSD (Serial #2413A00557) equipped with 7673A Auto Sampler.

   * HP 5996A GC/MS (Serial #2413A00557) equipped with Tekmar LSC-2000, ALS-2016 and Sample Heater.

3. Hewlett-Packard A Series (A900) Data Handling System:

   * HP-5890 Series II GC and 5970B MSD (2905A11935) equipped with 7673 Auto Sampler.

   * HP-5890 Series II GC and 5970 MSD (2921A23002) equipped with a 7673 Auto Sampler.

B. Seven Gas Chromatographs:

1. Varian 4600 (Serial #04861674) equipped with Vista 401 data handling system, autosampler, electron capture and flame ionization detector and Hewlett-Packard Headspace sampler Model 19395A.

2. Hewlett Packard A series computer system used on the following GCs:

   * HP-5890 Series II GC (Serial #3033A32207) equipped with PID/ELCD, Tekmar LSC2000, ALS2016 and sample heater.

   * HP-5890 Series II GC (Serial #3033A32222) equipped with 2 ECDs and dual tower 7673 Auto Sampler.

   * Hewlett-Packard 5890A (Serial #2728A12675) equipped with autosampler and two electron capture detectors.

* Hewlett-Packard 5890A (Serial #2413A05663) equipped with Tekmar LSC-2 and ALS as well as PID and Hall detectors.

* Varian 3600 GC (ID#3600-0907) equipped with two ECD detectors, 8100 Auto Sampler and varian star integration package.

3. BaseLine Industries 1030A (Serial #1089) equipped with PID and FID detectors.

4. Sentex portable gas chromatograph equipped with an AID detector and Toshiba personal computer.

C. Three Inductively Coupled Plasmas:

1. Leeman ICP PS-1000 (IC 5006), Axial

2. Leeman ICP 2.5-ps-I (Serial #396), Sequential.

3. Leeman ICP ES-2000 (Serial #1293A), 38 Channel Simultaneous.

D. Two Emission Spectrophotometers:

1. Jarrell Ash 3.5 Ebert (Serial #19261).

2. Baird Direct Reader (Serial #HB-2-262).

E. Eight Atomic Absorption Spectrophotometers:

1. Perkin-Elmer 2380 Atomic Absorption Spectrophotometer (Serial #126854).

2. Perkin-Elmer 3100 Atomic Absorption Spectrophotometer (Serial #LR23329C).

3. Perkin-Elmer 4100 Atomic Absorption Spectrometer (Serial #6058).

4. Perkin-Elmer 5000 Atomic Absorption Spectrophotometer (Serial #117775).

5. Perkin-Elmer Zeeman 5100 Atomic Absorption Spectrophotometer (Serial #133206).

6. Instrument Laboratories 951 Atomic Absorption Spectrophotometer (Serial #2391).

7. Leeman Labs Analyte 5 Simultaneous Graphite Furnace Atomic Absorption Spectrophotometer (Serial #P54002)

8. Leeman Labs AS 200 Automated Mercury Analyzer (Serial #AP-

5009) equipped with PS 200 Mercury Preparation Analyzer (Serial #HG 5009)

F. 1. Buck Scientific Infrared Spectrophotometer.
2. Miran 1A fixed wavelength IR.
3. Horiba OCMA220 fixed wavelength IR.

G. Four HACH DR/3000 Spectrophotometers

H. Ionics Model 115 carbon analyzer equipped with thermolyne type 2100 tube furnace.

I. Lachat Quikchem 8000 F.I.A. Analyzer (Serial #).

J. Two ABC Lahbs Gel Permeation Units.

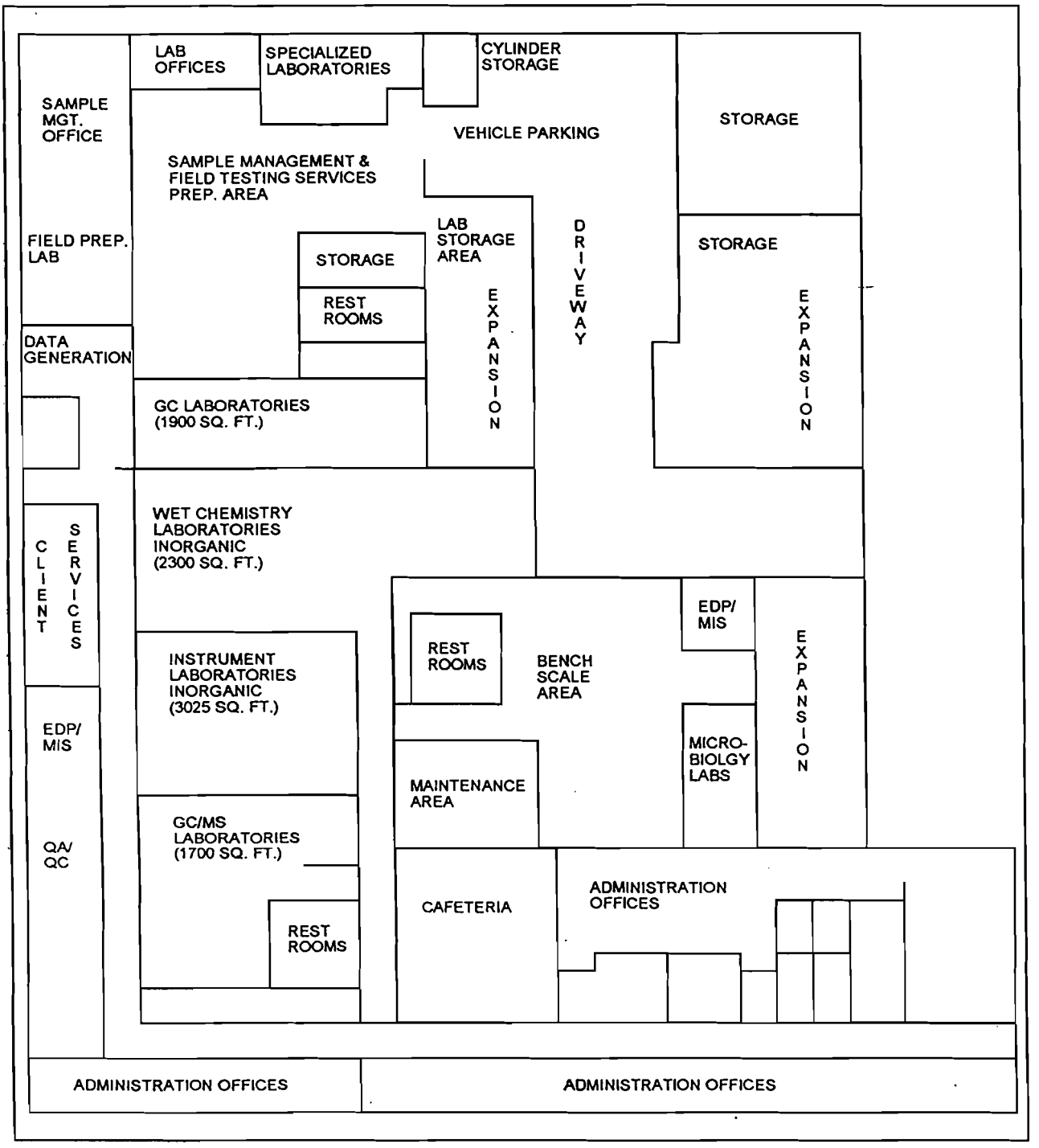
K. Twelve Cyanide Distillation Units.

L. Balances:

1. Ohaus 1500 (Serial #3734)
2. Ohaus 4000-D (Serial #01657)
3. Ohaus E 4000-D (Serial #1731)
4. Ohaus E 4000-D (Serial #1739)
5. Sartorius 1601 (Serial #3404429)
6. Sartorius 1602 (Serial #3405382)
7. Sartorius 1612 (Serial #3403078)
8. Mettler AE240 (Serial #G 17877)
9. Mettler AC100 (Serial #A 33338)
10. Mettler P120 (Serial #232510)
11. Sauter AR1014 (Serial #A 95857)
12. Sauter AR1014 (Serial #B 04445)
13. Cahn 26 (Serial #38747)

ACCREDITED LABORATORIES, INC.

(52,000 SQUARE FEET)



METHODOLOGY

REFERENCE ANALYTICAL METHODS

PHYSICAL PROPERTIES

ANALYSIS	SAMPLE MATRIX	REFERENCE METHOD[1]
Color	Aqueous	EPA 110.2
Conductance	Aqueous	EPA 120.1
Hardness	Aqueous	EPA 130.2
Odor	Aqueous	EPA 140.1
pH	Aqueous	EPA 150.1
Residue		
Filterable	Aqueous	EPA 160.1
Non-filterable	Aqueous	EPA 160.2
Total	Aqueous	EPA 160.3
Volatile	Aqueous	EPA 160.4
Settleable Matter	Aqueous	EPA 160.5
Temperature	Aqueous	EPA 170.1
Turbidity	Aqueous	EPA 180.1

LIMITED CHEMISTRY

ANALYSIS	SAMPLE MATRIX	REFERENCE METHOD[1]
Acidity	Aqueous	EPA 305.1
Alkalinity	Aqueous	EPA 310.1
Bromide	Aqueous	EPA 320.1
Chloride	Aqueous	EPA 325.3
Chlorine	Aqueous	EPA 330.5/.3
Cyanides	Aqueous	EPA 335.2
Reactive Cyanides	Solid	SW846-7.3.3.2

Fluoride	Aqueous	EPA 340.2
Iodine	Aqueous	EPA 345.1
Nitrogen		
Ammonia	Aqueous	EPA 350.3
Kjeldahl, Total	Aqueous	EPA 351.3
Nitrate plus Nitrite	Aqueous	EPA 353.3
Nitrate	Aqueous	EPA 352.1/WeWWG
Nitrite	Aqueous	EPA 354.1
Phosphorus		
Ortho-	Aqueous	EPA 365.3
phosphate	Aqueous	EPA 365.3
Dissolved	Aqueous	EPA 365.3
Hydrolyzable	Aqueous	EPA 365.3
Total	Aqueous	EPA 365.3
Sulfate	Aqueous	EPA 375.3/.4
Sulfide	Aqueous	EPA 376.2/.1
Reactive Sulfide	Solid	SW846-7.3.4.1
Sulfite	Aqueous	EPA 377.1
BOD (5 Day)	Aqueous	EPA 405.1
COD	Aqueous	EPA 410.4
Oil & Grease	Aqueous	EPA 413.2/.1
TPHC	Aqueous Solid	EPA 418.1 EPA 418.1 Modified
Phenolics	Aqueous	EPA 420.1
Organic Carbon	Aqueous	EPA 415.1

HAZARDOUS WASTE CHARACTERISTICS

ANALYSIS	SAMPLE MATRIX	REFERENCE METHOD[1]
Ignitability	Liquid	SW846-1010
Corrosivity (pH)	Liquid Solid	EPA 150.1 SW846-9045
Reactive Cyanide	Liquid Solid	EPA 335.2 SW846-7.3.3.2
Reactive Sulfide	Liquid Solid	EPA 376.2/.1 SW846-7.3.4.1
EP Toxicity Extraction	Solid	SW846-1310
TCLP	Solid	SW846-1311

METALS

ANALYSIS	SAMPLE MATRIX	REFERENCE METHOD[1]
All except Hg, As, Se	Aqueous	EPA 200 series methods for flame, flameless AA and ICAP techniques
All except Hg, As, Se	Solid	SW846-7000 series methods for flame, flameless AA techniques and 6010 method for ICAP technology
As	Aqueous Solid	EPA 206.2 SW846-7060
Se	Aqueous Solid	EPA 270.2 SW846-7740
Hg	Aqueous Solid	EPA 245.1 SW846-7471

ORGANICS - DRINKING WATER ANALYSES

ANALYSIS	SAMPLE MATRIX	REFERENCE METHOD[1]
Chlorinated Herbicides	Potable water	SM509A (GC-ECD)
Trihalomethanes	Potable water	EPA 524.1 (GC/MS) EPA 524.2 (GC/MS)
Purgeable Halocarbons	Potable water	EPA 524.1 (GC/MS) EPA 524.2 (GC/MS)
Purgeable Aromatics	Potable water	EPA 524.1 (GC/MS) EPA 524.2 (GC/MS)
Unregulated VOCs	Potable water	EPA 524.1 (GC/MS) EPA 524.2 (GC/MS)
Chlorinated Pesticides	Potable water	SM 509A (GC-ECD)
Chlorinated Pesticides/PCBs	Potable water	EPA 508 (GC-ECD)

WASTEWATER ANALYSES

ANALYSIS	SAMPLE MATRIX	REFERENCE METHOD[1]
Purgeable Halocarbons	Aqueous	EPA 601 (GC-HECD)
Purgeable Aromatics	Aqueous	EPA 602 (GC-PID)
Chlorinated Pesticides/PCBs	Aqueous	EPA 608 (GC-ECD)
Volatiles	Aqueous	EPA 624 (GC/MS)
Semi-volatile Extractables (BNA)	Aqueous	EPA 625 (GC/MS)

SOLID WASTE ANALYSES

ANALYSES	SAMPLE MATRIX	REFERENCE METHOD[1]
Volatiles (VOA)	Aqueous/Solid	SW846-8240 (GC/MS)
Semi-volatile Extractables (BNA)	Aqueous/Solid	SW846-8270 (GC/MS)

Chlorinated Pesticides/PCBs	Aqueous/Solid	SW846-8080 (GC/ECD)

SW846 protocol is followed for all solid waste analysis.

TARGET COMPOUND LIST

ANALYSES	SAMPLE MATRIX	REFERENCE METHOD[1]
Volatiles	Aqueous/Solid	EPA-CLP-SOW, OLMO 3.0-3.1
Semi-volatile Extractables	Aqueous/Solid	EPA-CLP-SOW, OLMO 3.0-3.1
Metals	Aqueous/Solid	EPA-CLP-SOW, ILMO 3.0-3.1
Cyanide	Aqueous/Solid	EPA-CLP-SOW, ILMO 3.0

[1] Reference methods derived from the following:

a. EPA = US EPA Published Methodologies
b. SW846 = Test Methods for Evaluating Solid Wastes, 3rd ed. 1986
c. EPA-CLP = US EPA Contract Laboratory Program
d. SM = Standard Methods For The Examination of Water and Wastewater

METHODOLOGIES

GC-GC/MS METHODS SUMMARY

DRINKING WATER/SOURCE WATER

TESTING PARAMETER	METHOD
Pesticides, Primary DW	508
Herbicides, 2,4-D Silvex	515
A 280	524.2/508
Trihalomethanes	524.2
Volatile Organics	524.2
Primary DW Parameters (Regulatory)	EPA
Secondary DW Parameters (Non-Regulatory)	EPA

WASTEWATER ANALYSIS

TESTING PARAMETER	METHOD
Halogenated Volatiles	601
Halogenated/Aromatic Volatiles	601/602
Aromatic Volatiles	602
BTEX	602
Acrolein & Acrylonitrile	603
Organochlorine Pest/PCBs	608
Pesticides	608
PCBs	608
BTEX	624
MTBE, DIPE, TBA	624
Volatile Organics	624
Volatiles + 10 (TCL)	624

Volatiles + 15 (PP)	624
Base/Neutrals	625
Base/Neutrals + 10 (TCL)	625
Base/Neutrals + 15 (PP)	625
Acid Extractables	625
Acid Extractables + 10 (TCL)	625
Acid Extractables + 10 (PP)	625
PAHs	625
Base/Neutral, Acid Extract.	625
Base/Neutral, Acid Extract. + 25 (PP)	625
Base/Neutral, Acid Extract. + 20 (TCL)	625
2,3,7,8 TCDD (Dioxin Screen)	625

SOLID WASTE ANALYSES

* Method Utilized

TESTING PARAMETER	METHOD
Halogenated Volatiles	8010
TPHC-GRO	8015M
TPHC-DRO	8015M
Aromatic Volatiles	8020
Acrolein & Acrylonitrile	8030
Phenols	(8040)/8270*
Phthalate Esters	(8060)/8270*
Organochlorine Pest/PCBs	8080
Chlorinated Herbicides	8150
Volatile Organics	8240
Volatile Organics + 15 (PP)	8240
Volatile Organics + 10 (TCL)	8240

Volatile Organics	8260
Acid Extractables	8270
Acid Extractables + 10 (PP)	8270
Acid Extractables + 10 (TCL)	8270
Base/Neutrals	8270
Base/Neutrals + 15 (PP)	8270
Base/Neutrals + 10 (TCL)	8270
Base/Neutrals, Acids	8270
Base/Neutrals, Acids + 25 (PP)	8270
Base/Neutrals, Acids + 20 (TCL)	8270
PAHs	8270
PAHs	8310 (1995)

METALS ANALYSES

Analytical packages:

RCRA HEAVY METALS (8) - TCLP (As, Ba, Cd, Cr, Pb, Hg, Se, Ag)

PRIORITY POLLUTANT METALS (13) - (As, Sb, Be, Cd, Cr, Cu, Ni, Pb, Hg, Se, Ag, Tl, Zn)

TAL METALS (23) - (Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Tl, V, Zn)

TESTING PARAMETER	ACCREDITED STANDARD METHODS WATER/SOLID
Aluminum (Al)	200.7/6010
Antimony (Sb)	200.7/6010
Arsenic (As)	206.2/7060
Barium (Ba)	200.7/6010
Beryllium (Be)	200.7/6010
Boron (B)	200.7/6010

Cadmium (Cd)	200.7/6010
Calcium (Ca)	200.7/6010
Chromium, Total (Cr)	200.7/6010
Chromium, Hexavalent (Cr +6)	218.1/7196
Cobalt (Co)	200.7/6010
Copper (Cu)	200.7/6010
Gold (Au)	200.7/6010
Iron (Fe)	200.7/6010
Lead (Pb)	239.2/7421
Lithium (Li)	SM 3500
Magnesium (Mg)	200.7/6010
Manganese (Mn)	200.7/6010
Mercury (Hg)	245.1/7470
Molybdenum (Mo)	200.7/6010
Nickel (Ni)	200.7/6010
Platinum (Pt)	200.7
Potassium (K)	200.7/6010
Selenium (Se)	270.2/7740
Silicon (Si)	200.7/6010
Silver (Ag)	200.7/6010
Sodium (Na)	200.7/6010
Strontium (Sr)	200.7/6010
Thallium (Tl)	279.2/7841
Tin (Sn)	200.7/6010
Titanium (Ti)	200.7
Vanadium (V)	200.7/6010
Zinc (Zn)	200.7/6010

CHEMICAL AND PHYSICAL PROPERTIES

TESTING PARAMETER	METHODS
Acidity	305.1
Alkalinity	310.1
% Ash	ASTM D482
Bicarbonate	SM 4500
BOD (5 Day)	405.1
Bromide	SM 4500
BTU/lb	ASTM D2382
Carbon	
Total	415.1
Inorganic	415.1
Organic (TOC)	415.1/9060
Carbonate	SM 4500
Carbon dioxide	SM 4500
Cation Exchange Ratio	9080
Chloride	325.3
Chlorine, Total Residual	330.5/.3
Chlorine Demand	SM 2350 B
COD	410.4
Color	110.2
Conductivity, Specific	120.1/9050
Corrosivity (pH)	150.1/9040
Corrosivity (toward steel)	
Cyanide	1110
Reactivity	SW 846
Total	335.2
Amenable	335.1

Fluoride	340.2
Hardness, Total	130.2
Hydrocarbons, Petroleum (TPHC)	418.1
Ignitability, Flash Point	1010
Iodine	SM 4500
Nitrogen	
Ammonia	350.2
Kjeldahl, Total	351.3
Nitrate	SM 4500/352.1
Nitrite	354.1
Nitrate + Nitrite	353.3
Organic, Total	351.3
Odor	140.1
Oil & Grease, Gravimetric	413.1/9070
Oil & Grease, IR	413.2
Oxidation Potential	SM 2580
Oxygen, Dissolved	360.1
Paint Filter Test	9095
Particle Size Analysis (Sieve)	ASTM D422
Particle Size (Hydrometer)	ASTM D422
Phenols, Total	420.1/9066
Phosphorus	
Dissolved	365.3
Ortho	365.3
Total	365.3
pH	150.1
Reactivity, (S/CN)	SW 846
Salinity	SM 2520

Silica, Dissolved	6010
Solids	-
Dissolved (TDS)	160.1
Percent	EPA-CLP
Suspended (TSS)	160.2
Total (TS)	160.3
Total Volatile (TVS)	160.4
Specific Gravity	SM 2710
Sulfate	375.3/.4
Sulfide, Reactive	SW 846
Sulfite	377.1
Sulfur	ASTM D3177
Surfactants (MBAS)	425.1
Thiocyanate	SM 4500
Total Organic Halogens (TOX)	9020
Temperature	170.1
Turbidity	180.1
Viscosity	ASTM
% Water (Karl Fischer)	ASTM E-203

WASTE CLASSIFICATION - ANALYSIS

TCLP Constituents - Full	1311/8240/8270/8080/8150/6010
TCLP (Minus Pest/Herb)	1311/8240/8270/6010
Zero Headspace Extraction	1311
Semi-Volatile/Inorganic Ext.	1311
Volatile Organic Analysis	8240
Base/Neutral Analysis	8270
Acid Extractable Analysis	8270

Metals Analysis	6010
Pesticides/Herbicides Analysis	8080/8150

RCRA CONSTITUENTS - ANALYSIS

ID 27 (All Below)	
Corrosivity	9045
Ignitability	1010
Cyanide Reactivity	SW846 - 7.3.3.2
Sulfide Reactivity	SW846 - 7.3.4.1
PCBs	8080
Petroleum Hydrocarbons (TPHC)	418.1M

MICROBIOLOGY

TESTING PARAMETER	METHOD
Bioassay (Microtox™)*	N/A
Coliform, Fecal (MF/MPN)	SM 9221/2
Coliform, Total (MPN)	SM 9221/2
E. coli	SM 9225
Enterococcus (MPN) (MF)	SM 9230
Giardia	-
Iron Bacteria	SM 9240
Pseudomonas	SM 9213
Streptococci, Fecal	SM 9230
Salmonella (MPN)	SM 9260
Standard Plate Count/Heterotrophic	SM 9215
Water Suitability	SM 9020

FIELD SAMPLING & TESTING SERVICES

SAMPLING CAPABILITY

Wastewater - Stormwater - Monitoring Wells - Hazardous Waste - Industrial Hygiene

NPDES - TSCA - UST - CERCLA - SARA - RCRA - CAAA - ISRA - SDWA
Soil - Sludge - Air - Solids - Aqueous

ANALYTICAL CHEMISTRY FIELD TESTING SERVICES

BTEX	Immunoassay Test Kits
TPHC	Immunoassay Test Kits
PCBs	Immunoassay Test Kits
Chlorine	Meter
Dissolved Oxygen	Meter
Specific Conductivity	Meter
Metals	Portable XRF (1995)
Temperature	
pH	Meter
Volatile Organics	Portable GC

BIOLOGICAL FIELD TESTING SERVICES

Microtox ™ Toxicity Test Systems*(1995)

Wastewater Toxicity	Toxic Waste Minimization
NPDES Toxicity Monitoring	Identify Toxicity Excursions
Biomass Protection Studies	TRE/TIE Evaluations
Storm Water Toxicity Studies	New Product/Raw Material Screen
Waste Treatment Impact Studies	Waste Treatment Technologies Effectiveness

* Microtox ™ is a registered trademark of Microbics Corporation

CERTIFICATIONS AND ACCREDITATIONS

Accredited Laboratories, Inc. is certified to perform environmental analyses in the following states:

STATE	DRINKING WATER	WASTEWATER	SOLID WASTE	CLP
NJ	12007	12007	X	X
PA		X	X	
NY	11109	11109	11109	11109
CT		PH 0110	PH 0110	
CO		X	X	
DE		X	X	
GA		X	X	
KY		X	X	
LA		X	X	
MA		M-NJ 273	X	
MD	223	X	X	
ME	X	X	X	
MS	X	X	X	
NC			X	
SC			X	
VA				
WV			X	
RI			X	
VT	X	X	X	
NH			X	

Lab Accreditation by Private Organizations

AIHA	Accreditation #06966
A2LA	Certificate Number 391.01
X	No Certification Required

State of New Jersey

Christine Todd Whitman
Governor

Department of Environmental Protection
Office of Quality Assurance
CN424
Trenton, NJ 08625-0424
Tel (609) 292-3950
Fax (609) 777-1774

Robert C. Shinn, Jr.
Commissioner

August 1, 1996

12007 ACCREDITED LABORATORIES
FOOT OF PERSHING AVE. BOX 369
CARTERET NJ 07008
ATTN: THEODORE GAYDOS

Dear Laboratory Manager:

On July 1, 1996, the N.J.A.C. 7:18, Regulations Governing the Certification of Laboratories and Environmental Measurements became effective. The mailing of the 1996/1997 renewal applications and fee invoices have been delayed. Therefore, a new annual parameter list was not issued to replace the list which expired June 30, 1996.

Enclosed is a copy of your current laboratory certification status. This document will serve as a temporary annual parameter list. The temporary annual parameter list will be valid until an updated list is issued or June 30, 1997.

The Office of Quality Assurance (OQA) is currently revising the application and printing copies of the new regulations. Once this is complete, OQA will mail the application package to your laboratory. Upon receipt of the application please review it carefully, complete the necessary sections and return it to our office. Based on the information from the application an invoice will be generated and mailed to you. Once our office receives payment from your laboratory an annual certified parameter list will be issued.

Please direct all correspondence and inquiries to me at (609) 633-6752, or by letter to this Office at the letterhead address.

Sincerely,

Michael D. DiBalsi
Supervising Environmental Specialist
Office of Quality Assurance

Enclosure

STATE OF NEW JERSEY

Let's protect our earth

DEPARTMENT OF ENVIRONMENTAL PROTECTION AND ENERGY

Certifies That

Accredited Laboratories, Inc.
Foot of Pershing Avenue
P.O. Box 369
Carteret, New Jersey 07008

having duly met the requirements of the

Regulations Governing Laboratory Certification
And Standards Of Performance N.J.A.C. 7:18 et. seq.

is hereby approved as a

State Certified Environmental Laboratory

To perform the analyses as indicated on the Annual Certified Parameter List
which must accompany this certificate to be valid

12007
PERMANENT CERTIFICATION NUMBER

October 28, 1994
DATE

COMMISSIONER, DEPARTMENT OF
ENVIRONMENTAL PROTECTION AND ENERGY

This certification is subject to unannounced laboratory inspections as specified by N.J.A.C. 7:18-2.11(d) and agreed to by the Laboratory Manager on filing the application

TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY WITH THE ANNUAL CERTIFIED PARAMETER LIST.

STATE OF NEW JERSEY
DEPARTMENT OF ENVIRONMENTAL PROTECTION
OFFICE OF QUALITY ASSURANCE
ANNUAL CERTIFIED PARAMETER LIST FOR 1995-1996

CCREDITED LABPRATORIES, INC. (12007) IS CERTIFIED TO PERFORM THE ANALYSES BELOW UNTIL JUNE 30 1996.

DR NKING WATER LABORATORY CERTIFICATION

MICROBIOLOGY

303	TOT COLI-MEMBRANE FILTER
305	TOT COLI-MPN (10ML/20ML)
307	TOTAL COLIFORM (P-A)
309	TOTAL COLIFORM (ONPG-MUG)
315	FECAL COLIFORM (TC TO EC)
317	E. COLI (EC MEDIUM + MUG)
319	E. COLI (NUT. AGAR + MUG)

LIMITED CHEMISTRY

001	ALKALINITY, TITRIMETRIC
004	CONDUCTIVITY
005	ORTHOPHOSPHATE, COLORIMET
008	TEMPERATURE
019	NITRITE, SPECTROPHOTO
021	NITRITE, CD REDUCTION
034	CYANIDE, SPECTROPHOTO
919	NITRATE,ELECTRODE

DF NKING WATER LABORATORY CERTIFICATION

LIMITED CHEMISTRY

932 NITRATE,CADMIUM REDUCTION

935 FLUORIDE-ELECTRODE

944 TURBIDITY

945 CHLORINE RESIDUAL

947 CHLORIDE, HG OR AG NITRAT

948 COLOR, PLATINUM COBALT

949 ABS/LAS METHYLENE BLUE

950 ODOR, CONSISTENT SERIES

951 PH, GLASS ELECTRODE

952 TOT DISS SOLIDS, TOT RES

953 HARDNESS, EDTA

956 SULFATE, GRAVIM OR TURBID

METALS

010 CALCIUM, ICAP

011 CU, AA/PLATFORM FURNACE

013 PB, AA/PLATFORM FURNACE

017 ALUMINUM, ICAP

025 ANTIMONY, GRAPH FURNACE

028 NICKEL, AA

029 NICKEL, GRAPH FURNACE

030 NICKEL, ICAP

DRINKING WATER LABORATORY CERTIFICATION

METALS

031 THALLIUM, GRAPH FURNACE

036 BERYLLIUM, ICAP

901 BA, ATOMIC ABSORPTION

902 AG, ATOMIC ABSORPTION

903 CU, ATOMIC ABSORPTION

904 FE, ATOMIC ABSORPTION

906 ZN, ATOMIC ABSORPTION

912 HG,MANUAL COLD VAPOR

914 AS, GRAPHITE FURNACE

915 BA, GRAPHITE FURNACE

916 CD, GRAPHITE FURNACE

917 CR, GRAPHITE FURNACE

918 PB, GRAPHITE FURNACE

920 SE, GRAPHITE FURNACE

921 AG, GRAPHITE FURNACE

922 CU, GRAPHITE FURNACE

923 FE, GRAPHITE FURNACE

924 MN, GRAPHITE FURNACE

925 ZN, GRAPHITE FURNACE

954 NA, ATOMIC ABSORPTION

960 ARSENIC, ICAP

961 BARIUM, ICAP

DRINKING WATER LABORATORY CERTIFICATION

METALS

962	CADMIUM, ICAP
963	CHROMIUM, ICAP
965	SILVER, ICAP
966	COPPER, ICAP
967	IRON, ICAP
968	MANGANESE, ICAP
969	ZINC, ICAP
970	SODIUM, ICAP

ORGANICS

508	CHLORINATED PEST/PCBS
515.1	CHLORINATED HERB. (GC)
524.2	VOC (PT/GC-MS)

WATER POLLUTION LABORATORY CERTIFICATION

MICROBIOLOGY

74054	FECAL STREPTOCOCCI
74055	FECAL COLIFORM
74056	TOTAL COLIFORM
74057	ENTEROCOCCI
74058	HETEROTROPHIC PLATE COUNT
74059	PSEUDOMONAS AERUGINOSA

LIMITED CHEMISTRY

00010	TEMPERATURE

LIMITED CHEMISTRY

00076 TURBIDITY

00080 COLOR

00095 SPECIFIC CONDUCTANCE

00299 DISS OXYGEN-ELECTRODE

00300 DISS OXYGEN-WINKLER

00310 BOD(5/20 DAY)

00320 CARBONACEOUS BOD(5/20DAY)

00340 COD

00400 HYDROGEN ION-PH

00410 ALKALINITY

00436 ACIDITY

00500 TOT SOLIDS

00505 TOT VOLATILE SOLIDS

00530 SUSP SOLIDS

00545 SETT SOLIDS-VOLUMETRIC

00546 SETT SOLIDS-GRAVIMETRIC

00556 OIL AND GREASE

00605 ORGANIC NITROGEN

00610 AMMONIA NITROGEN

00615 NITRITE

00625 TOT KJELDAHL NITROGEN

00630 NITRATE

ATER POLLUTION LABORATORY CERTIFICATION

LIMITED CHEMISTRY

00650 PHOSPHORUS, TOT AS PO4

00660 ORTHOPHOSPHATE AS PO4

00665 PHOSPHORUS, TOT AS P

00671 ORTHOPHOSPHATE AS P

00680 ORGANIC CARBON, TOTAL

00681 ORGANIC CARBON, DISSOLVED

00720 CYANIDE, TOTAL

00722 CYANIDE, AMEN TO CHLOR

00740 SULFITE

00745 SULFIDE

00900 HARDNESS

00940 CHLORIDE

00945 SULFATE

00951 FLUORIDE, TOTAL

01032 CR HEX

32730 PHENOLS

38260 SURFACTANTS

50060 CHLORINE RESIDUAL

70300 TOT DISS SOLIDS

METALS

00915 CALCIUM (ICAP)

AT"R POLLUTION LABORATORY CERTIFICATION

METALS

00916	CALCIUM (AA)
00925	MAGNESIUM (ICAP)
00927	MAGNESIUM (AA)
00929	SODIUM (ICAP)
00930	SODIUM (AA)
00935	POTASSIUM (ICAP)
00937	POTASSIUM (AA)
00956	SILICA (ICAP)
01000	ARSENIC (ICAP)
01002	ARSENIC (AA/GF)
01005	BARIUM (ICAP)
01007	BARIUM (AA/GF)
01010	BERYLLIUM (ICAP)
01012	BERYLLIUM (AA/GF)
01020	BORON (ICAP)
01025	CADMIUM (ICAP)
01027	CADMIUM (AA/GF)
01030	CHROMIUM (ICAP)
01032	CHROMIUM VI (AA)
01034	CHROMIUM (AA/GF)
01035	COBALT (ICAP)
01037	COBALT (AA/GF)

ATER POLLUTION LABORATORY CERTIFICATION

METALS

01040	COPPER (ICAP)
01042	COPPER (AA/GF)
01045	IRON (ICAP)
01046	IRON (AA/GF)
01049	LEAD (ICAP)
01051	LEAD (AA/GF)
01055	MANGANESE (ICAP)
01056	MANGANESE (AA/GF)
01057	THALLIUM (ICAP)
01059	THALLIUM (AA/GF)
01060	MOLYBDENUM (ICAP)
01062	MOLYBDENUM (AA/GF)
01065	NICKEL (ICAP)
01067	NICKEL (AA/GF)
01075	SILVER (ICAP)
01077	SILVER (AA/GF)
01085	VANADIUM (ICAP)
01087	VANADIUM (AA/GF)
01090	ZINC (ICAP)
01092	ZINC (AA/GF)
01095	ANTIMONY (ICAP)
01097	ANTIMONY (AA/GF)

ATER POLLUTION LABORATORY CERTIFICATION

METALS

01102	TIN (AA/GF)
01105	ALUMINUM (ICAP)
01106	ALUMINUM (AA/GF)
01145	SELENIUM (ICAP)
01147	SELENIUM (AA/GF)
01152	TITANIUM (AA/GF)
01171	PLATINUM (AA/GF)
01210	PALLADIUM (AA/GF)
27901	RUTHENIUM (AA/GF)
28201	RHODIUM (AA/GF)
71900	MERCURY (COLD VAPOR)
71910	GOLD (AA/GF)

ORGANICS

601	PURGEABLE HALOCARBONS(GC)
602	PURGEABLE AROMATICS (GC)
608	PESTICIDES & PCBS (GC)
624	PURGEABLES (GC/MS)
625	B/N, ACIDS & PEST (GC/MS)

HIS LIST MUST BE CONSPICUOUSLY DISPLAYED WITH THE PERMANENT
EF IFICATE AT THE LABORATORY

State of Connecticut, Department of Public Health and Addiction Services

APPROVED PUBLIC HEALTH LABORATORY

This is to certify that the laboratory described below has been approved by the State Department of Public Health and Addiction Services pursuant to applicable provisions of the Public Health Code and General Statutes of Connecticut for making those examinations, determinations, or tests specified below which have been authorized in writing by that Department.

ACCREDITED LABORATORIES, INC.
Name of Laboratory

Located at Foot of Pershing Avenue *in* Carteret, New Jersey 07008-0369 *and registered in the name of* Theodore Gaydos.

This certificate is issued in the name of Yun-Shen Lee, Ph.D. *who has been designated by the registrant to be in charge of the laboratory work covered by this certificate of approval as follows:*

WASTEWATER AND/OR TRADE WASTE, SEWAGE AND/OR EFFLUENT, SOIL

Examination for:
Inorganic Chemicals
Organic Chemicals

SEE COMPUTER PRINT-OUT FOR SPECIFIC TESTS APPROVED

This certificate expires September 30, 1996 *and is revocable for cause by the State of Connecticut Department of Public Health and Addiction Services.*

Dated at Hartford, Connecticut, this 19th *day of* January, 1994

No. PH-0110

Earl R. Thompson Jr. M.S.
Division Director, Laboratory Standards

Chief, Bureau of Laboratories

OL-181B 2/94

STATE OF NEW YORK
DEPARTMENT OF HEALTH

Wadsworth Center The Governor Nelson A. Rockefeller Empire State Plaza P.O. Box 509 Albany, New York 12201-0509

Barbara A. DeBuono, M.D., M.P.H.
Commissioner

Karen Schimke
Executive Deputy Commissioner

June 26, 1996

Post-it™ brand fax transmittal memo 7671	# of pages ▸ 1
To DR. LEE	From JOYCE JOSLIN
Co. ACCREDITED LABS	Co. NYSDOH - ELAP
Dept.	Phone # 518-485-5570
Fax # 908-541-1383	Fax # 518-485-5568

Dear Laboratory Director:

Please note that your 1995-96 Certificate(s) of Approval, originally extended to June 30, 1996 has/have been further extended to July 31, 1996. This action has been necessitated by the lack of an approved New York State budget.

Enclosed please find your current 1996-97 ELAP fee statement. Upon receipt of at least one quarter of the annual amount due, your 1996-97 Certificate(s) of Approval will be issued.

Verification of your laboratory's approved ELAP status is available by calling the Program Office at (518) 485-5570.

Sincerely,

Joyce Joslin
Administrative Aide
Environmental Laboratory
Approval Program

Enc.

STATE OF NEW YORK
DEPARTMENT OF HEALTH

Wadsworth Center The Governor Nelson A. Rockefeller Empire State Plaza P.O. Box 509 Albany, New York 12201-0509

Barbara A. DeBuono, M.D., M.P.H.
Commissioner

Karen Schimke
Executive Deputy Commissioner

Dear Laboratory Director:

Enclosed are the amended ELAP Certificate(s) of Approval for permit year 1995-96 issued to your environmental laboratory. The Certificate(s) supercede any previously issued and are in effect through March 31, 1996. Please carefully examine the Certificate(s) to insure that the categories, subcategories and analytes for which your laboratory is approved are listed correctly, as well as verifying your laboratory's name, address, director and identification number.

In addition, please destroy your expired 1994-95 ELAP Certificate(s) of Approval.

Please notify this office of any corrections required. We may be reached at (518) 485-5570.

Sincerely,

Linda L. Madlin

Linda L. Madlin
Administrative Assistant
Environmental Laboratory
Approval Program

LLM:saw
Enclosure

STATE OF NEW YORK
DEPARTMENT OF HEALTH

Wadsworth Center The Governor Nelson A. Rockefeller Empire State Plaza P.O. Box 509 Albany, New York 12201-0509

Barbara A. DeBuono, M.D., M.P.H.
Commissioner

Karen Schimke
Executive Deputy Commissioner

MARCH 18, 1996

Dear Laboratory Director:

Please note that although your ELAP Certificate of Approval expires on 12:01 AM April 1, 1996, it is still valid until June 30, 1996 pending receipt of your 1996-97 Certificate(s), as per ELAP Certification Manual, No. 140, Page 7 of 25, dated 4/1/86, Part 55-2.4e NYCRR. "All environmental laboratory approval will, during the pendency of inspections or extension or grace period permitted by this subpart, remain in force beyond the normal expiration dates of certificates unless such approval is specifically revoked or suspended in writing."

Notification regarding the issuance of 1996-97 ELAP Certificate(s) of Approval is pending receipt of all non-governmental laboratories' Total Adjusted Volumes and Approval of the 1996-97 ELAP Budget by the New York State Legislature.

Further verification of your laboratory's approved ELAP status is available by calling the Program Office at (518) 485-5570.

Sincerely,

Linda L. Madlin

Linda L. Madlin
Administrative Assistant
Environmental Laboratory
Approval Program

LLM:saw

BARBARA A. DEBUONO, M.D., M.P.H. Commissioner

Expires 12:01 AM April 1, 1996
ISSUED April 1, 1995
REVISED July 17, 1995

INTERIM CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID No.: 11109

Director: DR. YUN-SHEN LEE
Lab Name: ACCREDITED LABORATORIES INC
Address : FOOT OF PERSHING AVE PO 369
CARTERET NJ 07008-0369

is hereby APPROVED as an Environmental Laboratory for the category

CONTRACT LABORATORY PROTOCOL (CLP)

All approved subcategories and/or analytes are listed below:

Inorganics | CLP PCB/Pesticides | CLP Semi-Volatile Organics | CLP Volatile Organics

Serial No.: 030577

Wadsworth Center

Property of the New York State Department of Health. Valid only at the address shown.
Must be conspicuously posted. Valid certificate has a red serial number.

DOH-3317 (3/95)

BARBARA A. DEBUONO, M.D., M.P.H. Commissioner

Expires 12:01 AM April 1, 1996
ISSUED April 1, 1995
REVISED July 17, 1995

INTERIM CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID No.: 11109

Director: DR. YUN-SHEN LEE
Lab Name: ACCREDITED LABORATORIES INC
Address : FOOT OF PERSHING AVE PO 369
CARTERET NJ 07008-0369

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES NON POTABLE WATER

All approved subcategories and/or analytes are listed below:

Hydrocarbon Pesticides :
4,4'-DDD
4,4'-DDE
4,4 DDT
alp -BHC
Aldrin
beta-BHC
Cap n
Chl dane Total
del -BHC
Dieldrin
Endrin aldehyde
Enc n
Enc ulfan I
Endosulfan II
Endosulfan sulfate
Heptachlor
Hep chlor epoxide
Li ne
Methoxychlor
Toxaphene

Wastewater Miscellaneous :
Boron, Total
Cyanide, Total
Color
Phenols
Oil & Grease Total Recoverable
Hydrogen Ion (pH)
Specific Conductance
Sulfide (as S)
Temperature
Organic Carbon, Total
Residue (ALL)

Chlorophenoxy Acid Pesticides :
2,4-D
2,4,5-TP (Silvex)
Chlorinated Hydrocarbons (ALL)
Haloethers (ALL)
Wastewater Metals I (ALL)
Mineral (ALL)
Nitrosoamines (ALL)
Polynuclear Aromatics (ALL)
Phthalate Esters (ALL)
Purgeable Aromatics (ALL)
TCLP Additional Compounds (ALL)

Acrolein and Acrylonitrile (ALL)
Wastewater Bacteriology (ALL)
Benzidines (ALL)
Demand (ALL)
Wastewater Metals III (ALL)
Wastewater Metals II (ALL)
Nitroaromatics and Isophorone (ALL)
Nutrient (ALL)
Polychlorinated Biphenyls (ALL)
Priority Pollutant Phenols (ALL)
Purgeable Halocarbons (ALL)

Serial No.: 030573

Wadsworth Center

Property of the New York State Department of Health. Valid only at the address shown.
Must be conspicuously posted. Valid certificate has a red serial number.

DOH-3317 (3/95)

NEW YORK STATE DEPARTMENT OF HE

BARBARA A. DEBUONO, M.D., M.P.H. Commissioner

Expires 12:01 AM April 1, 1996
ISSUED April 1, 1995
REVISED July 17, 1995

INTERIM CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID No.: 11109

Director: DR. YUN-SHEN LEE
Lab Name: ACCREDITED LABORATORIES INC
Address : FOOT OF PERSHING AVE PO 369
CARTERET NJ 07008-0369

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES/ POTABLE WATER

All approved subcategories and/or analytes are listed below:

aking Water Non-Metals :
Alkalinity
Calcium Hardness
Chloride
Color
Corrosivity
Fluoride, Total
Nitrate (as N)
Hydrogen Ion (pH)
Solids, Total Dissolved
Sulfate (as SO4)

D.W. Organohalide Pesticides :
Endrin
Lindane
Methoxychlor
Toxaphene

D.W. Chlorinated Acids :
2,4-D
2,4,5-TP (Silvex)
Volatile Aromatics (ALL)

Drinking Water Bacteriology (ALL)
Drinking Water Trihalomethane (ALL)
Drinking Water Metals I (ALL)
Volatile Halocarbons (ALL)

Serial No.: 030574

Wadsworth Center

Property of the New York State Department of Health. Valid only at the address shown.
Must be conspicuously posted. Valid certificate has a red serial number.

DOH-3317 (3/95)

NEW YORK STATE DE

BARBARA A. DEBUONO, M.D., M.P.H. Commissioner

Expires 12:01 AM April 1, 1996
ISSUED April 1, 1995
REVISED July 17, 1995

INTERIM CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID No.: 11109

Director: DR. YUN-SHEN LEE
Lab Name: ACCREDITED LABORATORIES INC
Address : FOOT OF PERSHING AVE PO 369
CARTERET NJ 07008-0369

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES/AIR AND EMISSIONS

All approved subcategories and/or analytes are listed below:

ls (ALL)
ychlorinated Biphenyls (ALL)
Metals I (ALL)
Purgeable Aromatics (ALL)
Metals II (ALL)
Purgeable Halocarbons (ALL)
Polynuclear Aromatics (ALL)

Serial No.: 030575

Wadsworth Center

Property of the New York State Department of Health. Valid only at the address shown.
Must be conspicuously posted. Valid certificate has a red serial number.

DOH-3317 (3/95)

NEW YORK STATE DEPARTMENT OF HEALTH

BARBARA A. DEBUONO, M.D., M.P.H. Commissioner

Expires 12:01 AM April 1, 1996
ISSUED April 1, 1995
REVISED July 17, 1995

INTERIM CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID No.: 11109

Director: DR. YUN-SHEN LEE
Lab Name: ACCREDITED LABORATORIES INC
Address : FOOT OF PERSHING AVE PO 369
CARTERET NJ 07008-0369

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES/SOLID AND HAZARDOUS WASTE

All approved subcategories and/or analytes are listed below:

cteristic Testing :
rrosivity
pait ility
eact ity
CLP
.P. Toxicity

Miscellaneous :
Cyanide, Total
Lead in Paint
Hydrogen Ion (pH)
Sulfide (as S)
Priority Pollutant Phenols (ALL)

Acrolein and Acrylonitrile (ALL)
Chlorinated Hydrocarbons (ALL)
Metals I (ALL)
Nitroaromatics Isophorone (ALL)
Polychlorinated Biphenyls (ALL)
Purgeable Aromatics (ALL)

Chlor. Hydrocarbon Pesticides (ALL)
Haloethers (ALL)
Metals II (ALL)
Polynuclear Arom. Hydrocarbon (ALL)
Phthalate Esters (ALL)
Purgeable Halocarbons (ALL)

Serial No.: 030576

Wadsworth Center

operty of the New York State Department of Health. Valid only at the address shown.
Must be conspicuously posted. Valid certificate has a red serial number.

DH-3317 (3/95)

№ 211

STATE OF MARYLAND

DEPARTMENT OF HEALTH AND MENTAL HYGIENE
LABORATORIES ADMINISTRATION

Certifies That

ACCREDITED LABORATORIES, INC.

FOOT OF PERSHING AVENUE, CARTERET, NEW JERSEY 07008-0369

having duly met the requirements of the

Regulations Governing Laboratory Certification

And Standards Of Performance In Accordance With

The Annotated Code of Maryland,

is hereby approved as a

State Certified Water Quality Laboratory

To perform the analyses indicated on the Annual Certified Parameter List,

which must accompany this certificate.

Certification # 223

Date Issued DECEMBER 16, 1994

Expiration Date DECEMBER 31, 1995
(Not Transferable)

J. Mahsun Joseph
Director, Laboratories Administration

This certification is subject to unannounced laboratory inspections

CONSPICUOUSLY DISPLAY IN THE LABORATORY WITH THE ANNUAL CERTIFIED PARAMETER LIST

CONNECTICUT STATE DEPARTMENT OF HEALTH

LABORATORY DIVISION

THURSDAY NOVEMBER 18, 1993 10:51 AM

REGISTRATION DATE 10/01

PH0110 ACCREDITED LABORATORIES, INC. P.O. BOX 369 CARTERET, N.J. 07008
REGISTRANT THEODORE GAYDOS DIRECTOR YUN-SHEN LEE. PH.D.
CO-DIRECTOR
MEDICARE NUMBER- INTERSTATE NUMBER-

TEST 201	WASTEWATER AND/OR TRADE WASTE
TEST 202	SEWAGE AND/OR EFFLUENT
TEST 203	SOIL
TEST 230	CHEMISTRY
TEST 231	INORGANIC CHEMICALS
TEST 232	PHYSICAL EXAMS
TEST 233	COLOR
TEST 234	ODOR
TEST 235	TURBIDITY
TEST 236	PH
TEST 237	CONDUCTIVITY
TEST 238	TEMPERATURE
TEST 239	MINERALS
TEST 240	ACIDITY
TEST 241	ALKALINITY
TEST 242	HARDNESS
TEST 243	SULFATE
TEST 244	SULFIDE
TEST 245	SULFITE
TEST 247	CHLORIDE
TEST 248	FLUORIDE
TEST 249	CHLORINE
TEST 250	NUTRIENTS
TEST 251	AMMONIA
TEST 252	KJELDAHL NITROGEN
TEST 253	ORGANIC NITROGEN
TEST 254	NITRATE
TEST 255	NITRITE
TEST 256	ORTHO-PHOSPHATE
TEST 257	TOTAL PHOSPHORUS
TEST 258	MISCELLANEOUS
TEST 259	TOTAL SOLIDS
TEST 260	TOTAL DISSOLVED SOLIDS
TEST 261	TOTAL VOLATILE SOLIDS
TEST 262	TOTAL SUSPENDED SOLIDS
TEST 263	CYANIDE
TEST 265	SURFACTANTS
TEST 266	DEMAND
TEST 267	BOD
TEST 268	COD

REGISTRATION DATE 10/01

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TEST 237	CONDUCTIVITY
TEST 238	TEMPERATURE
TEST 239	MINERALS
TEST 240	ACIDITY
TEST 241	ALKALINITY
TEST 242	HARDNESS
TEST 243	SULFATE
TEST 244	SULFIDE
TEST 245	SULFITE
TEST 247	CHLORIDE
TEST 248	FLOURIDE
TEST 249	CHLORINE
TEST 250	NUTRIENTS
TEST 251	AMMONIA
TEST 252	KJELDAHL NITROGEN
TEST 253	ORGANIC NITROGEN
TEST 254	NITRATE
TEST 255	NITRITE
TEST 256	ORTHO-PHOSPHATE
TEST 257	TOTAL PHOSPHORUS
TEST 258	MISCELLANEOUS
TEST 259	TOTAL SOLIDS
TEST 260	TOTAL DISSOLVED SOLIDS
TEST 261	TOTAL VOLATILE SOLIDS
TEST 262	TOTAL SUSPENDED SOLIDS
TEST 263	CYANIDE
TEST 265	SURFACTANTS
TEST 266	DEMAND
TEST 267	BOD
TEST 268	COD

TEST 270	METALS
TEST 271	ALUMINUM
TEST 272	ANTIMONY
TEST 273	ARSENIC
TEST 274	BARIUM
TEST 275	BERYLLIUM
TEST 277	CADMIUM
TEST 278	CHROMIUM TOTAL
TEST 279	CHROMIUM VI
TEST 280	COBALT
TEST 281	COPPER
TEST 282	IRON
TEST 283	LEAD
TEST 285	MAGNESIUM
TEST 286	MANGANESE
TEST 287	MERCURY
TEST 288	MOLYBEDNUM
TEST 289	NICKEL
TEST 290	POTASSIUM
TEST 291	SELENIUM
TEST 292	SILVER
TEST 293	SODIUM
TEST 294	THALLIUM
TEST 295	TIN
TEST 296	TITANIUM
TEST 297	VANADIUM
TEST 298	ZINC
TEST 311	ORGANIC CHEMICALS
TEST 312	PURGEABLE HALOCARBONS
TEST 313	PURGEABLE AROMATICS
TEST 314	PESTICIDES
TEST 315	HERBICIDES
TEST 316	PCB
TEST 320	PHENOLS
TEST 322	PHTHALATE ESTERS
TEST 325	POLYNUCLEAR AROMATIC HYDROCARBONS
TEST 327	CHLORINATED HYDROCARBONS
TEST 329	BASE/NEUTRALS AND ACIDS
TEST 331	OIL AND GREASE
TEST 332	GROSS HYDROCARBONS

DEPARTMENT OF THE ARMY
U.S. ARMY CORPS OF ENGINEERS — MRD
HTRW MANDATORY CENTER OF EXPERTISE
12565 WEST CENTER ROAD
OMAHA, NEBRASKA 68144-3869

REPLY TO
ATTENTION OF

October 18, 1995

Hazardous, Toxic and Radioactive Waste
Center of Expertise

Accredited Laboratories, Inc.
Foot of Pershing Avenue
Carteret, NJ 07008-0369

Gentlemen:

This correspondence addresses the recent evaluation of Accredited Laboratories, Inc. of Carteret, NJ, by the U.S. Army Corps of Engineers (USACE) for hazardous, toxic, and radioactive waste analysis.

Your laboratory has successfully analyzed audit samples as listed below:

METHOD	PARAMETERS	MATRIX
8021A	Volatile Organic Compounds	Water
8020	Aromatic Volatile Organics	Water
SW-846	8 RCRA Metals[1]	Water
SW-846	8 RCRA Metals[1]	Soil
418.1	TRPH	Water
9071/418.1	TRPH	Soil

Remarks: 1) Metals (8 RCRA): Arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver.

Based on the assessment of written qualifications and successful analysis of the performance evaluation samples indicated in the table in paragraph two your laboratory is validated for multimedia sample analysis by the above methods. A full validation of 18 months was approved by the USACE Hazardous, Toxic and Radioactive Waste Center of Expertise (HTRW CX) Laboratory Validation Committee on October 11, 1995. This validation only applies to analysis performed in support of underground storage tank removal projects.

DATA MANAGEMENT

INTRODUCING

Syscom * D M P ™

DATA MANAGEMENT SYSTEMS

"IMPLEMENTING TOMORROW'S TECHNOLOGY, TODAY..."

"In 1993 the Company developed a new, unique and proprietary software program which has considerably increased the efficiencies and sample throughput of the Analytical Laboratories. These new systems now coordinate and manage production schedules, perform continuous and final QA/QC functions and publish Final Reports of Test directly from the system's instrumentation.

We have now upgraded this technology by implementing and incorporating new software programs which produce customized Analytical Data Spreadsheets and QA/QC Summary Formats in Hard Copy or Diskette. These programs are designed to provide supplementary, and final data information packages which require no manipulation, transcription or reproduction.

These Syscom* DMP Data Management Systems are offered in conjunction with our "Strategic Alliance" programs. We would appreciate your inquiry and the opportunity to discuss your analytical and data management requirements. Clients' currently using this system are reporting substantial benefits in reduced operating costs and potential applications to attract or retain clients."

THE STRATEGIC ALLIANCE PROGRAM

The Accredited Laboratories "Strategic Alliance" program is designed to integrate and maximize the resources of the Company and our clients. Our commitment is to offer a variety of comprehensive Analytical and Data Management service programs to support current environmental investigations, remedial action programs and regulatory compliance efforts. Our strategy is to provide "cost effective" systems which optimize opportunities to meet the competitive challenges of the 90's and the 21st Century.

DATA MANAGEMENT SYSTEMS

The Syscom * DMP Data Management System is a new, proprietary, Software Program developed by Accredited Laboratories. The system is designed to quantify and publish Final Reports of Test, Customized Data Summaries and QA/QC Summary Data sheets directly from the analytical instrumentation raw quantification files. The program eliminates all requirements for physical manipulation, transcription and reproduction of data packages by clients who are preparing specialized reporting formats for their customers, regulatory

agencies, or for their own internal information systems documents. Information packages and specialized formats, designed by our clients or regulatory agencies, are offered in Hard Copy or in LOTUS and ASCII diskette deliverables.

PRICE MANAGEMENT PROGRAMS

The Company's Price Management programs are designed to provide competitive analytical fees and offer cost effective, integrated, Data Management Systems which could potentially save clients $10,000 to $75,000 annually. The new Syscom * DMP Software program has considerably reduced the Company's operating costs by increasing efficiencies, sample throughput, and capacity. Client savings are generated by the System's capability to produce supplemental information which substantially reduces operating costs by providing for the re-allocation of engineering resources for managing, and client problem solving rather than data manipulation.

Syscom * *DMP* ™
DISKETTE DELIVERABLE FORMAT

Technical Regulatory Specifications July 93'

Availability: LOTUS Worksheet Format
ASCI II Format

ACCREDITED LABORATORIES, INC.
RESULTS OF VOLATILE ORGANIC ANALYSIS
CASE NUMBER: SAMPLE

SAMPLE NO.:	DAILY BLAN	SAMPLE	SAMPLE	SAMPLE	SAMPLE
CLIENT ID :		SAMPLE	SAMPLE	SAMPLE	SAMPLE
FILE NO. :	SAMPLE	SAMPLE	SAMPLE	SAMPLE	SAMPLE
MATRIX :	Soil	Soil	Soil	Soil	Soil
DIL. FACT.:	1	10	50	1	1
% SOLID :	100	92.3	78.4	96.7	98.9
UNITS :	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG

COMPOUND	CONC.	MDL	CONC.	MDL	CONC.	MDL	CONC.	MDL	CONC.	MDL
Acrolein	U	5	U	54	U	320	U	5	U	5
Acrylonitrile	U	5	U	54	U	320	U	5	U	5
Chloromethane	U	10	U	110	U	640	U	10	U	10
Bromomethane	U	10	U	110	U	640	U	10	U	10
Vinyl Chloride	U	10	U	110	U	640	U	10	U	10
Chloroethane	U	10	U	110	U	640	U	10	U	10
Methylene Chloride	U	5	91	54	U	320	16	5	13	5
Acetone	U	10	210	110	U	640	10(J)	10	11	10
Carbon Disulfide	U	5	U	54	U	320	U	5	U	5
Trichlorofluoromethane	U	10	U	110	U	640	U	10	U	10
1,1-Dichloroethene	U	5	U	54	U	320	U	5	U	5
1,1-Dichloroethane	U	5	U	54	U	320	U	5	U	5
trans-1,2-Dichloroethene	U	5	U	54	U	320	U	5	U	5
Chloroform	U	5	U	54	U	320	U	5	U	5
1,2-Dichloroethane	U	5	U	54	U	320	U	5	U	5
2-Butanone	U	10	120	110	U	640	10(J)	10	7(J)	10
1,1,1-Trichloroethane	U	5	U	54	U	320	U	5	U	5
Carbon Tetrachloride	U	5	U	54	U	320	U	5	U	5
Vinyl Acetate	U	10	U	110	U	640	U	10	U	10
Bromodichloromethane	U	5	U	54	U	320	U	5	U	5
1,2-Dichloropropane	U	5	U	54	U	320	U	5	U	5
cis-1,3-Dichloropropene	U	5	U	54	U	320	U	5	U	5
Trichloroethene	U	5	U	54	U	320	U	5	U	5
Benzene	U	5	210	54	U	320	U	5	U	5
Dibromochloromethane	U	5	U	54	U	320	U	5	U	5
1,1,2-Trichloroethane	U	5	U	54	U	320	U	5	U	5
trans-1,3-Dichloropropene	U	5	U	54	U	320	U	5	U	5
2-Chloroethylvinylether	U	10	U	110	U	640	U	10	U	10
Bromoform	U	5	U	54	U	320	U	5	U	5
2-Hexanone	U	10	U	110	U	640	U	10	U	10
4-Methyl-2-pentanone	U	10	U	110	U	640	U	10	U	10
Tetrachloroethene	U	5	U	54	U	320	U	5	U	5
1,1,2,2-Tetrachloroethane	U	5	U	54	U	320	U	5	U	5
Toluene	U	5	780	54	90(J)	320	1(J)	5	U	5
Chlorobenzene	U	5	U	54	U	320	U	5	U	5

(B) Indicates compound found in associated blank.
(J) Indicates compound concentration found below MDL.
U Indicates compound analyzed for but not detected.

Syscom * *DMP* ™
SYSTEMS CUSTOMIZED SPREADSHEET CAPABILITY

===> Volatiles												
Case Number:												
Client Name:												
Field Number			E-1					E-7				
Sample Number			9407001					9407002				
Matrix			Soil					Soil				
% Moisture			13.7					18.4				
Units	UG/KG	UG/KG	UG/KG					UG/KG				
Dilution Factor			100.0					1.0				
Date Analyzed			05/19/94					05/26/94				
Analyte	RDC	IGW	Result	MDL	Flag	MB	MB MD	Result	MDL	Flag	MB	MB MD
Acrolein	none	none	U	580.0			5.0	U	6.0			5.0
Acrylonitrile	1000	1000	U	580.0			5.0	U	6.0			5.0
Chloromethane	520000	10000	U	1200.0			10.0	U	12.0			10.0
Bromomethane	79000	1000	U	1200.0			10.0	U	12.0			10.0
Vinyl Chloride	2000	10000	U	1200.0			10.0	U	12.0			10.0
Chloroethane	none	none	U	1200.0			10.0	U	12.0			10.0
Methylene Chloride	49000	1000	1700	580.0	(B)	2	5.0	20	6.0	(B)	3	5.0
Acetone	1000000	100000	U	1200.0			10.0	240	12.0	(B)	6	10.0
Carbon Disulfide	none	none	U	580.0			5.0	U	6.0			5.0
Trichlorofluoromethane	none	none	U	1200.0			10.0	U	12.0			10.0
1,1-Dichloroethene	8000	1000	U	580.0			5.0	U	6.0			5.0
1,1-Dichloroethane	57000	10000	U	580.0			5.0	U	6.0			5.0
trans-1,2-Dichloroethene	1000000	50000	U	580.0			5.0	U	6.0			5.0
Chloroform	19000	1000	U	580.0			5.0	U	6.0			5.0
1,2-Dichloroethane	6000	1000	U	580.0			5.0	U	6.0			5.0
2-Butanone	1000000	50000	U	1200.0			10.0	68	12.0			10.0
1,1,1-Trichloroethane	210000	50000	U	580.0			5.0	U	6.0			5.0
Carbon Tetrachloride	20000	1000	U	580.0			5.0	U	6.0			5.0
Vinyl Acetate	none	none	U	1200.0			10.0	U	12.0			10.0
Bromodichloromethane	11000	1000	U	580.0			5.0	U	6.0			5.0

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QUALITY ASSURANCE MANAGEMENT
QA/QC ORGANICS SUMMARY*

(Internal Standards & Surrogates)

* Available Hard Copy only

BFB File >C6524 Injected at 08:57 on the date of 08/03/94

Reviewed By:_____ Date:_____.

Checked By:_____ Date:_____.

			Internal Standards			Surrogates				Chrom.	Premlim. Data
	ALI Sample #	Data File	1 area	2 area	3 area	DIC %	TOL %	BRO %	Flags	Acceptable Y or N	Acceptable Y or ReRun
AL CHECK		>C6525	73495	256981	194378	NA	NA	NA	*O	_____	_____.
_____	DAILY BLAN	>C6527	81247	278335	203244	99	97	98		_____	_____.
_____	SAMPLE	>C6528	74331	248518	181034	99	98	113		_____	_____.
_____	SAMPLE	>C6529	48087	160448	118477	102	98	104		_____	_____.
_____	SAMPLE	>C6530	52889	177494	129171	102	100	110		_____	_____.
_____	SAMPLE	>C6533	51738	171265	118724	105	103	113		_____	_____.
_____	SAMPLE	>C6534	52885	180630	134353	106	96	94		_____	_____.
_____	SAMPLE	>C6535	56768	195804	144297	107	96	100		_____	_____.
_____	SAMPLE	>C6536	69444	229220	165969	104	100	106		_____	_____.
_____	SAMPLE	>C6537	59439	196743	141768	103	98	100		_____	_____.
_____	SAMPLE	>C6538	68244	229697	168386	103	97	115		_____	_____.
_____	SAMPLE	>C6539	66376	221862	170329	102	94	98		_____	_____.
_____	SAMPLE	>C6540	63782	212850	156510	102	96	113		_____	_____.
_____	SAMPLE	>C6541	63348	211670	159241	101	95	114		_____	_____.

BFB Inj. Time is 08:57 Daily calibration inj. Time :06/31/94 09:22

Last Inj. Time is 20:28 Blank injection Time :06/31/94 11:02

SURROGATE LIMITS	WATER	SOIL
S1 (DIC) = 1,2-Dichloroethane-d4	(76-114)	(70-121)
S2 (TOL) = Toluene-d8	(88-110)	(81-117)
S3 (BRO) = Bromofluorobenzene	(86-115)	(74-121)

*A = Area is > 200% or < 50% of Cal Check

*R = RT is Plus or Minus .5 min. of Cal Check

*O = There is a compound in the sample over 200

* Values outside of contract required QC Limits

Syscom * DMP ™
VOLATILE ORGANICS REPORT
ISRA* FOOTNOTES

* Groundwater Quality Criteria

ACCREDITED LABORATORIES, INC.
VOLATILE ORGANIC ANALYSIS DATA

CASE NUMBER	EXAMPLE	MATRIX	Aqueous
SAMPLE NUMBER	EXAMPLE	DILUTION FACTOR	EXAMPLE
DATA FILE	EXAMPLE	DATE EXTRACTED	EXAMPLE
CLIENT NAME	EXAMPLE	DATE ANALYZED	EXAMPLE
FIELD ID	EXAMPLE	ANALYZED BY	EXAMPLE

CAS #	COMPOUND	UG/L		MDL	CAS #	COMPOUND	UG/L		MDL
107028	Acrolein	U		25000	78875	1,2-Dichloropropane	U		25000
107131	Acrylonitrile	U		25000	10061015	cis-1,3-Dichloropropene	U		25000
74873	Chloromethane	U		50000	79016	Trichloroethene	U		25000
74839	Bromomethane	U		50000	71432	Benzene	46000	W	25000
75014	Vinyl Chloride	U		50000	124481	Dibromochloromethane	U		25000
75003	Chloroethane	U		50000	79005	1,1,2-Trichloroethane	U		25000
75092	Methylene Chloride	U		25000	10061026	trans-1,3-Dichloropropene	U		25000
67641	Acetone	U		50000	110758	2-Chloroethylvinylether	U		50000
75150	Carbon Disulfide	U		25000	75252	Bromoform	U		25000
75694	Trichlorofluoromethane	U		50000	591786	2-Hexanone	U		50000
75354	1,1-Dichloroethene	U		25000	108101	4-Methyl-2-pentanone	U		50000
75343	1,1-Dichloroethane	U		25000	127184	Tetrachloroethene	U		25000
156605	trans-1,2-Dichloroethene	U		25000	79345	1,1,2,2-Tetrachloroethane	U		25000
67663	Chloroform	U		25000	108883	Toluene	640000	W	25000
107062	1,2-Dichloroethane	U		25000	108907	Chlorobenzene	U		25000
78933	2-Butanone	U		50000	100414	Ethylbenzene	200000	W	25000
71556	1,1,1-Trichloroethane	U		25000	100425	Styrene	U		25000
56235	Carbon Tetrachloride	U		25000	1330207	m,p-Xylene	840000	W	25000
108054	Vinyl Acetate	U		50000	95476	o-Xylene	320000	W	25000
75274	Bromodichloromethane	U		25000	156592	cis-1,2-Dichloroethene	U		25000

SURROGATE COMPOUNDS	RECOVERY	LIMITS	STATUS
1,2-Dichloroethane-d4	101 %	76-114	OK
Toluene-d8	91 %	88-110	OK
Bromofluorobenzene	115 %	86-115	OK

J - Indicates compound concentration found below MDL.
U - Indicates compound analyzed for but not detected.
B - Indicates compound found in associated blank.
W - Result exceeds specific ground water quality criteria.*

* Flags are based on Specific Ground Water Quality Criteria from New Jersey Register dated February 1, 1993.

Syscom * DMP ™*
VOLATILE ORGANICS REPORT
ISRA* FOOTNOTES

* Industrial Surface Water Standards
* Residential Surface Soil Standards

ACCREDITED LABORATORIES, INC.
VOLATILE ORGANIC ANALYSIS DATA

CASE NUMBER	EXAMPLE	MATRIX	Soil
SAMPLE NUMBER	EXAMPLE	DILUTION FACTOR	EXAMPLE
DATA FILE	EXAMPLE	DATE EXTRACTED	EXAMPLE
CLIENT NAME	EXAMPLE	DATE ANALYZED	EXAMPLE
FIELD ID	EXAMPLE	ANALYZED BY	EXAMPLE

CAS #	COMPOUND	UG/KG		MDL	CAS #	COMPOUND	UG/KG		MDL
107028	Acrolein	U		26000	78875	1,2-Dichloropropane	U		26000
107131	Acrylonitrile	U		26000	10061015	cis-1,3-Dichloropropene	U		26000
74873	Chloromethane	U		52000	79016	Trichloroethene	U		26000
74839	Bromomethane	U		52000	71432	Benzene	48000	I	26000
75014	Vinyl Chloride	U		52000	124481	Dibromochloromethane	U		26000
75003	Chloroethane	U		52000	79005	1,1,2-Trichloroethane	U		26000
75092	Methylene Chloride	U		26000	10061026	trans-1,3-Dichloropropene	U		26000
67641	Acetone	U		52000	110758	2-Chloroethylvinylether	U		52000
75150	Carbon Disulfide	U		26000	75252	Bromoform	U		26000
75694	Trichlorofluoromethane	U		52000	591786	2-Hexanone	U		52000
75354	1,1-Dichloroethene	U		26000	108101	4-Methyl-2-pentanone	U		52000
75343	1,1-Dichloroethane	U		26000	127184	Tetrachloroethene	U		26000
156605	trans-1,2-Dichloroethene	U		26000	79345	1,1,2,2-Tetrachloroethane	U		26000
67663	Chloroform	U		26000	108883	Toluene	670000		26000
107062	1,2-Dichloroethane	U		26000	108907	Chlorobenzene	U		26000
78933	2-Butanone	U		52000	100414	Ethylbenzene	210000		26000
71556	1,1,1-Trichloroethane	U		26000	100425	Styrene	U		26000
56235	Carbon Tetrachloride	U		26000	1330207	m,p-Xylene	880000	I	26000
108054	Vinyl Acetate	U		52000	95476	o-Xylene	330000	I	26000
75274	Bromodichloromethane	U		26000	156592	cis-1,2-Dichloroethene	U		26000

SURROGATE COMPOUNDS	RECOVERY	LIMITS	STATUS
1,2-Dichloroethane-d4	101 %	70-121	OK
Toluene-d8	91 %	81-117	OK
Bromofluorobenzene	115 %	74-121	OK

Percent solid of 95.5 is used for all target compounds.

R - Result exceeds residential surface soil standards.*
J - Indicates compound concentration found below MDL.
U - Indicates compound analyzed for but not detected,
I - Result exceeds industrial surface soil standards.*
B - Indicates compound found in associated blank.

* Flags are based on New Jersey Soil Cleanup Criteria from Site Remediation News Volume 06 Number 1.

Syscom * DMP ™

DISCHARGE MONITORING REPORT

INDUSTRIAL DISCHARGER NAME: _____ REPORT PERIOD: 02/01/94 THROUGH: 02/28/94

	SAMPLE DATE: SAMPLE TYPE: SAMPLE I.D.#:	02/24/94 G 9402268	02/24/94 C 9402269	1/31/94 C 9401084	1/31/94 G 9401085	REPORT MONTHY AVERAGE	REPORT # OF DAILY EXCURSIONS
PARAMETER	DISCHARGE LIMITS						
FLOW-MGD	0.035 (1)						
pH (SU)	6.0 - 9.0					5.77	1
MG/L BOD: LBS:	2,000 (2) 584 (2)		4,390.0			1,977.00	2
MG/L COD: LBS:	4,000 (2) 1,170 (2)		6,830.0			3,569.40	2 0
MG/L TSS: LBS:	600 175		1,263.0			793.50	1
SS (ML/L)	10.0					< 1.0	0
OIL & GREASE	50.0						
PHENOLS	10.0					32.90	1
SULFIDES	0.2					< 0.20	0
AMMONIA-NITROGEN	22.0					< 0.10	0
CADMIUM (TOTAL)	4.0					< 0.01	0
CHROMIUM (TOTAL)	———					< 0.03	0
COPPER (TOTAL)	3.0					2.060	0
IRON (TOTAL)	15.0					2.330	0
NICKEL (TOTAL)	5.0					0.0877	0
ZINC (TOTAL)	5.0					2.870	0
ARSENIC (TOTAL)	1.0					0.0898	0
CHROMIUM (HEX.)	3.0					< 0.10	0
LEAD (TOTAL)	———					0.140	0
SILVER (TOTAL)	———					< 0.03	0
CYANIDE (TOTAL)	2.0					< 0.01	0

NOTE: ALL LIMITS EXPRESSED IN MG/L UNLESS OTHERWISE SPECIFIED.

MGD = MILLION GALLONS PER DAY
SU = STANDARD UNITS
ML/L = MILLILITERS PER LITER
(1) = THE FLOW LIMIT IS A MONTHLY AVERAGE LIMIT
(2) = THE BOD AND COD LIMITS ARE APPROVED MODIFICATIONS TO THE LRSA LOCAL ORDINANCE. BOTH CONCENTRATION AND MASS LIMITS WILL APPLY FOR COMPLIANCE DETERMINATION.

QUALITY ASSURANCE/QUALITY CONTROL OVERVIEW

LABORATORY QUALITY ASSURANCE PROGRAM

OBJECTIVES AND POLICIES

Accredited Laboratories, Inc. utilizes only USEPA-NIOSH approved methods, such as EPA 500 series methods for potable water analysis, EPA 600 series methods for wastewater analysis, EPA SW846 methods for solid waste analysis, EPA TO series methods for ambient air analysis, NIOSH-OSHA methods for industrial hygiene analysis, the most current EPA-CLP statement of work for the remediation projects required to adopt this analytical protocol. The quality assurance procedures recommended in USEPA analytical methods are adopted in ALI's Laboratory Standards Operation Procedure (SOP). The general QA/QC practices described in the "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", "Test Methods for Evaluating Solid Waste Physical/Chemical Methods", "Manual of Analytical Quality Control for Pesticides and Related Compounds" and "EPA Contract Laboratory Program" are the basis for ALI's quality assurance program. The objectives of this laboratory's quality assurance program are to provide valid, documented and court defensible results. It is realized that the improvement in laboratory quality is a continuing and progressive process. However, the whole quality system of the laboratory must be reviewed and revised by management at least once per year.

ALI's quality assurance program requires specific laboratory operating procedures which establish, maintain and document the desired, valid, acceptable analytical results specified in the program objectives. Key components of this quality assurance program include strict adherence to sampling and analytical method criteria, sampling and analytical chain-of-custody procedures and documentation, continual instrument performance specifications, mandatory instrument standardization schedules verified with systematic calibration checks, and routine surveillance and documentation of acceptable analytical accuracy and precision through systematic inclusion of quality control samples into all laboratory analyses.

Quality control is an integral part of the analytical process at ALI. Whenever it is feasible, duplicate spike and/or surrogate preparation and analysis is included in all of the SOPs. At least one in every twenty samples is prepared and analyzed as a matrix spike and matrix spike duplicate. Surrogate recovery is routinely performed on all samples analyzed by GC/MS. Standard series preparation and analysis is also included in all SOPs.

Standard method blanks, duplicate samples, spike samples and surrogates are analyzed along with the samples. Samples are not analyzed until criteria are met. The acceptable QA/QC criteria specified in the EPA-NIOSH approved methods are strictly enforced at ALI.

The procedures involved in sample log-in, chain-of-custody, analysis request, sample tracking and handling and data reporting, etc. are all described in detail in ALI's Standard Operation Procedures Manual. In general, every step involving data generation at ALI is

checked by a second person prior to the release of the data in order to minimize any possibility of human error. The sample tracking and checking flow chart is contained herein.

It is ALI's policy that the unused portion of samples will be archived and/or disposed of after "a proper period of time" (either specified in the project or determined by the laboratory) upon completion of sample analysis. Any samples exhibiting hazardous waste characteristics or known to be hazardous according to 40CFR Part 261 will be returned to the client or charged for ultimate disposal.

ALI generates six types of data reporting packages. They are:

Standard Package

- Cover Page
- Chain-of-custody form
- Methodology
- Results with MDLs

NJDEPE - Reduced Deliverables (ISRA, NJ Reduced Data Package)

- Results with MDLs and other applicable QC results (e.g. method, trip and field blanks, spike/spike duplicate, and/or surrogate recovery)
- Non-Conformance Summary
- Laboratory Deliverable Check List
- Laboratory Certificate
- GC/MS Conformance/Non-conformance Summary Format
- GC/MS Result Summary
- GC/MS Surrogate Summary
- Methodology Summary
- GC/MS Tuning, Calibration, TICs and Quant Report
- GC Chromatograms
- Chain-of-Custody Form
- Laboratory Chronicles

NJDEPE - Full Format (Non-CLP Format, Tier I Data Package)

- Results with MDLs and other applicable QC results (e.g. method, trip and field blanks, spike/spike duplicate, and/or surrogate recovery)
- Non-Conformance Summary
- Methodology Summary
- GC/MS Tuning, Calibration, TICs and Quant Report
- GC/MS Internal Standard Area Check Report
- Mass Spectra for target and/or tentatively identified compounds

- External standard calibration associated with GC analysis
- GC Chromatograms of sample analysis
- Instrument Performance Report associated with ICAP
- Standard Addition Report associated with AA-Furnace
- Chain-of-Custody Form
- Laboratory Chronicles

NJDEPE - CLP II Format

- Title Page
- Table of Contents
- Sample Analysis Request Form and Chain-of-Custody Documentation
- Methodology Review
- Non-conformance Summary
- Organic Data Deliverables:
  - Sample and Method Blank Analysis Data Sheet (Form I)
  - Surrogate Recovery Summary (Form II)
  - Matrix Spike/Matrix Spike Duplicate Recovery (Form III)
  - Method Blank Summary (Form IV)
  - Instrument Performance Check (Form V)
  - Organics Initial Calibration Data (Form VI)
  - Continuing Calibration Check (Form VII)
  - Internal Standard Area Summary (Form VIII)
  - Pesticide Analytical Sequence (Form VIIID)
  - Pesticide Identification Summary (Form X)
  - Mass Spectra, Quant Report and Chromatograms for Sample and Method Blank Analysis
- Inorganic Data Deliverables:
  - Inorganic Analysis Data Sheet (Form I)
  - Initial and Continuing Calibration Verification (Form II, Part 1)
  - CRDL Standard for AA and ICP (Form II, Part 2)
  - Blanks (Form III)
  - ICP Interference Check Sample (Form IV)
  - Spike Sample Recovery (Form V, Part 1)
  - Post Digest Spike Sample Recovery (Form V, Part 2)
  - Duplicates (Form VI)
  - Laboratory Control Sample (Form VII)
  - Standard Addition Results (Form VIII)
  - ICP Serial Dilution (Form IX)
  - Instrument Detection Limits - Quarterly (Form X)
  - ICP Interelement Correction Factors - Annually (Form XI)
  - ICP Linear Ranges - Quarterly (Form XII)
  - Preparation Log (Form XIII)
  - Analysis Run Log (Form XIV)

NJDEPE - CLP I Format

- Title Page
- Table of Contents
- Sample Analysis Request Form and Chain-of-Custody Documentation
- Methodology Review
- Non-conformance Summary
- Organic Data Deliverables:
  - Sample and Method Blank Analysis Data Sheet (Form I)
  - Surrogate Recovery Summary (Form II)
  - Matrix Spike/Matrix Spike Duplicate Recovery (Form III)
  - Method Blank Summary (Form IV)
  - Instrument Performance Check (Form V)
  - Organics Initial Calibration Data (Form VI)
  - Continuing Calibration Check (Form VII)
  - Internal Standard Area Summary (Form VIII)
  - Pesticide Analytical Sequence (Form VIIID)
  - Pesticide Identification Summary (Form X)
  - Raw Sample Data as per USEPA-CLP (3/90) deliverable requirements
  - Raw Standard Data as per USEPA-CLP (3/90) deliverable requirements
  - Raw Quality Control Data as per USEPA-CLP (3/90) deliverable requirements
- Inorganic Data Deliverables:
  - Inorganics Analysis Data Sheet (Form I)
  - Initial and Continuing Calibration Verification (Form II, Part 1)
  - CRDL Standard for AA and ICP (Form II, Part 2)
  - Blanks (Form III)
  - ICP Interference Check Sample (Form IV)
  - Spike Sample Recovery (Form V, Part 1)
  - Post Digest Spike Sample Recovery (Form V, Part 2)
  - Duplicates (Form VI)
  - Laboratory Control Sample (Form VII)
  - Standard Addition Results (Form VIII)
  - ICP Serial Dilution (Form IX)
  - Instrument Detection Limits - Quarterly (Form X)
  - ICP Interelement Correction Factors - Annually (Form XI)
  - ICP Linear Ranges - Quarterly (Form XII)
  - Preparation Log (Form XIII)
  - Analysis Run Log (Form XIV)
  - Raw Sample Data as per USEPA-CLP deliverable requirements
  - Raw Standard Data as per USEPA-CLP deliverable requirements
  - Raw Quality Control Data as per USEPA-CLP deliverable requirements

USEPA - CLP FORMAT

- Complete Sample Delivery Group File (CSF)
- SDG Narrative
- Chain-of-Custody Forms
- Traffic Reports
- Volatile Data Deliverables:
  - QC Summary
    - System Monitoring Compound Summary (Form II)
    - Matrix Spike/Matrix Spike Duplicate Summary (Form III)
    - Method Summary (Form IV)
    - GC/MS Instrument Performance Check (Form V)
    - Internal Standard Area and RT Summary (Form VIII)
  - Sample Data
    - Target Compound Results (Form I)
    - Tentatively Identified Compounds (Form I-TIC)
    - Reconstructed Total Ion Chromatograms (RIC) and Quant Reports
    - Mass Spectra of all target compounds detected
    - Mass Spectra of all TICs detected
  - Standards Data
    - Initial Calibration Data (Form IV) including RICs and Quant Reports
    - Continuing Calibration Data (Form VII) including RICs and Quant Reports
  - Raw QC Data
    - BFB including RICs, Bar Graph Spectrum and Mass Listing
    - Blank Data including Tabulated Results (Form I), TICs (Form-TIC), RICs, Quant Reports, Mass Spectra for both Target and non-target Compounds detected
    - Matrix Spike Data including Tabulated Results (Form I), RICs and Quant Reports
    - Matrix Spike Duplicate Data including Tabulated Results (Form I), RICs and Quant Reports

- Semi-volatile Data Deliverables:
  - QC Summary
    - Surrogate Percent Recovery Summary (Form II)
    - Matrix Spike/Matrix Spike Duplicate Summary (Form III)
    - Method Blank Summary (Form IV)
    - GC/MS Instrument Performance Check (Form V)
    - Internal Standard Area and RT Summary (Form VIII)
  - Sample Data
    - Target Compound Results (Form I)
    - Tentatively Identified Compounds (Form I-TIC)
    - Reconstructed Total Ion Chromatograms (RIC) and Quant Reports

- Mass Spectra of all Target Compounds detected
- Mass Spectra of all TICs detected

Standards Data
- Initial Calibration Data (Form VI) including RICs and Quant Reports
- Continuing Calibration Data (Form VII) including RICs and Quant Reports

Raw QC Data
- DFTPPP including RICs, Bar Graph Spectrum and Mass Listing
- Blank Data including Tabulated Results (Form I), TICs (Form-TIC), RICs, Quant Reports, Mass Spectra for both Target and non-target Compounds detected
- Matrix Spike Data including Tabulated Results (Form I), RICs and Quant Reports
- Matrix Spike Duplicate Data including Tabulated Results (Form I), RICs and Quant Reports
- GPC Data including UV Tracing Chromatograms, RICs and Quant Reports for Standards and GPC Blank Analyses

- Pesticides/Aroclor Data Deliverables:

QC Summary
- Surrogate Percent Recovery Summary (Form II)
- Matrix Spike/Matrix Spike Duplicate Summary (Form III)
- Method Blank Summary (Form IV)

Sample Data
- Target Compound Results (Form I)
- GC Chromatograms and Quant Reports for both Primary and Secondary Columns

Standards Data
- Initial Calibration of Single Component Analytes (Form VI)
- Initial Calibration of Multicomponent Analytes (Form VI)
- Analyte Resolution Summary (Form VI)
- Calibration Verification Summary (Form VII) for all Performance Evaluation Mixtures and Instrument Blanks
- Calibration Verification Summary (Form VII) for all Midpoint Concentration of Individual Standard Mixtures A and B and Instrument Blanks
- Analytical Sequence (Form VIII)
- Florisil Cartridge Check (Form IX)
- Pesticide GPC Calibration (Form IX)
- Pesticide Identification Summary for Single Component Analytes (Form X)
- Pesticide Identification Summary for Multicomponent Analytes (Form X)
- GC Chromatogram and Quant Reports for all Standards Raw QC

The expiration date of validation is April 11, 1997. The HTRW CX may schedule and conduct an on-site audit at any time during the 18 month validation period to evaluate lab performance if deemed necessary. USACE reserves the right to conduct laboratory audits or to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of any analytical samples from a USACE Contracting Office Representative.

If you have any questions or comments, please contact Ms. Elena Webster at (402) 697-2574.

Sincerely,

Marcia C. Davies

Marcia C. Davies, Ph.D.
Director, USACE Hazardous,
Toxic and Radioactive Waste
Center of Expertise

The American Industrial Hygiene Association

is proud to acknowledge that

Accredited Laboratories, Inc.

Carteret, NJ

Laboratory ID # 6966

has fulfilled the requirements for Industrial Hygiene Laboratory Accreditation and has earned distinguished recognition as an

AIHA IH Accredited Laboratory

Originally Accredited February 1, 1975, current certificate effective February 1, 1996 until February 1, 1999, subject to continued compliance with AIHA accreditation criteria.

President
American Industrial Hygiene Association

Chair
Analytical Accreditation Board

Chairman
IH Laboratory Accreditation Committee

January 1, 1996
Date Prepared

028
Certificate Number

State of Maryland

LABORATORIES ADMINISTRATION
DEPARTMENT OF HEALTH AND MENTAL HYGIENE
201 WEST PRESTON STREET • P.O. BOX 2355 • BALTIMORE,MARYLAND 21203-2355 • (410) 225-6150

Nelson J. Sabatini
Secretary

J. Mehsen Joseph, Ph.D.
Director

November 15, 1993

Accredited Laboratories, Inc.
Attn.: Mr. Yun-shen Lee
Foot of Pershing Avenue
P. O. Box 369
Carteret, New Jersey 07008-0369

Dear Mr. Lee:

Enclosed please find your certificate of reciprocity for drinking water laboratory certification in the State of Maryland. The reciprocity is good for a period of three (3) years. The certificate and fee are renewable annually. After the 3-year period, a new application must be made. If you have any changes in methods, supervisory personnel or major equipment pertinent to drinking water analysis during the year, you are required to advise this office within 30 days.

On the certificate is a statement that the subject laboratory is approved "To perform the analyses indicated on the Annual Certified Parameter List, which must accompany this certificate". Please note that this statement applies only to laboratories located in Maryland, not those certified by reciprocity. Where the laboratory is located outside this state, the approved list provided by their home state serves as the list of certified tests.

If you have any questions, please do not hesitate to call.

Sincerely,

Deanna Murphy Baxam
Water Quality Laboratory
Certification Officer

DMB:ncw

Enclosure (certificate)

TDD for The Deaf:
Baltimore Area 383-7555
D.C. Metro Area 565-0451

THE AMERICAN
ASSOCIATION
FOR LABORATORY
ACCREDITATION

ACCREDITED LABORATORY

A2LA has accredited

ACCREDITED LABORATORIES, INC.
Carteret, NJ

for technical competence in the field of

Environmental Testing

The accreditation covers the specific tests and types of tests listed on the agreed scope of accreditation. This laboratory meets the requirements of ISO/IEC guide 25-1990 "General Requirements for the Competence of Calibration and Testing Laboratories" (equivalent to relevant requirements of the ISO 9000 series of standards) and any additional program requirements in the identified field of testing.

Presented this 12th day of April, 1995.

President
For the Accreditation Council

Certificate Number 391.01

Valid to January 31, 1997

Commonwealth of Massachusetts
Executive Office of Environmental Affairs
Department of Environmental Protection
Senator William X. Wall Experiment Station

William F. Weld
Governor
Trudy S. Coxe
Secretary, EOEA
Thomas B. Powers
Acting Commissioner

April 29, 1994 Certified Mail # P 273 849 965

M-NJ273
Dr. Yun-Shen Lee
Accredited Laboratories, Inc.
Foot of Pershing Avenue
P.O. Box 369
Carteret, New Jersey 07008-0369

Dear Dr. Lee:

Enclosed is your Massachusetts Certificate for Environmental Analysis. This certificate is in effect through December 31, 1994 and is subject to revision throughout the year to reflect your laboratory's performance on proficiency tests and the status of your certification in your resident state. Please examine the certificate carefully to ensure that the categories and analytes for which your laboratory is approved appear correct. In addition, please verify the accuracy of your laboratory's name, address, telephone number and director.

Renewal of this certificate is contingent upon timely receipt of the results of your participation in the EPA Water Supply and Water Pollution Performance Evaluation Studies. Massachusetts requires full participation (i.e. all analytes in all the certified categories) twice yearly in the WS and WP studies. Satisfactory results must be achieved in the analyte categories for which certification is requested. In addition, you are required to submit to this office current copies of your resident state certificate and resident state on-site audit report as renewals occur. Any change in the status of your resident state certification must be reported immediately to this office.

Your laboratory's quality assurance plan has been reviewed according to 310 CMR 42.08(5)(a). The following is noted:

1. The use of quality control charts by the laboratory needs to be addressed.

2. Laboratory safety plans must be included.

If you have any questions, please do not hesitate to contact me.

Sincerely,

Ann Marie Allen

Ann Marie Allen
Director, Laboratory Certification Office

The Commonwealth of Massachusetts

Department of Environmental Protection
Division of Environmental Analysis

Certifies

Laboratory ID #: M-NJ273

Accredited Laboratories, Inc.
Foot of Pershing Ave.
P.O. Box 369
Carteret, NJ 07008-0369

for the ***Chemical Analysis of Non-Potable Water***

pursuant to 310 CMR 42.00

Laboratory Director: Dr. Yun-Shen Lee

Expiration Date: 06/30/97

This certificate supersedes all previous Massachusetts certificates issued to this laboratory. The laboratory is regulated by and shall be responsible for being in compliance with Massachusetts regulations at 310 CMR 42.00.

This certificate is valid only when accompanied by the latest dated Certified Parameter List as issued by the Massachusetts D.E.P.

Certification is no guarantee of the validity of the data. This certification is subject to unannounced laboratory inspections.

Director, Division of Environmental Analysis

07/01/96

Issued

Commonwealth of Virginia
Department of General Services
Division of Consolidated Laboratory Services

Certifies That

ACCREDITED LABORATORIES, INC.

Having Duly Met the Requirements of the
Regulations for the Certification of Laboratories Analyzing Drinking Water
Is hereby Approved as a

Certified Drinking Water Laboratory

To Perform the Analyses as indicated on the Annual Certified Parameter List
Which must accompany this to be valid.

Laboratory ID Number 00043 Effective 7/1/96 Through 6/30/97

Virginia Laboratory Officer
Safe Drinking Water Program

This certification is subject to unannounced laboratory inspections.
Conspicuously display in the laboratory with the annual certified parameter list.

This laboratory has met the minimum requirements for certification to analyze drinking water.
THIS CERTIFICATION DOES NOT GUARANTEE ACCURATE RESULTS.

Certificate Not Transferable Surrender upon Revocation

Data

- Blank Data including tabulated Results (Form I), GC Chromatograms and Quant Reports
- Matrix Spike Data including Tabulated Results (Form I) GC Chromatograms and Quant Reports
- Matrix Spike Duplicate Data including Tabulated Results (Form I), GC Chromatograms and Quant Reports
- GPC Data including UV Tracing Chromatograms, GC Chromatograms and Quant Reports for Standards and GPC Blank Analyses
- Florisil Data including GC Chromatograms and Quant Reports for Florisil Cartridge Performance Check Analyses

- Inorganic Data Deliverables:

  Results - Inorganic Analysis Data Sheet (Form I)

  Quality Control Data
  - Initial and Continuing Calibration Verification (Form II)
  - CRDL Standard for AA and Linear Range Analysis for ICP (Form II)
  - Blanks (Form III)
  - ICP Interference Check Sample (Form IV)
  - Spike Sample Recovery (Form V)
  - Post Digest Spike Sample Recovery (Form V)
  - Duplicates (Form VI)
  - Laboratory Control Sample (Form VII)
  - Standard Addition Results (Form VIII)
  - ICP Serial Dilutions (Form IX)
  - Holding Times (Form X)

  Verification of Instrument Parameters
  - Instrument Detection Limits (Quarterly) (Form XI)
  - ICP Instrument Correction Factors (Annually) (Form XII)
  - ICP Interelement Correction Factors (Annually) (Form XII)
  - ICP Linear Ranges (Quarterly) (Form XIII)

  Raw Data

  The order of raw data in the data deliverables shall be the following: ICP, Flame AA, Furnace AA, Mercury and Cyanide. All Flame and Furnace AA data shall be grouped by elements. Raw data must be labeled and identified with the following information: Calibration standards including sources and prep. date, initial and continuing calibration blanks and preparation blanks, initial and continuing calibration verification standards, interference check samples, ICP serial dilution samples, CRDL standard for ICP and AA, Laboratory Control Sample and Post Digestion Spike, diluted and undiluted samples and all weights, dilutions and volumes used to obtain the reported values, duplicate, spikes including the indication of standard solution used and final spike concentrations, volumes involved, instrument used, any

instrument adjustments, data corrections or other apparent anomalies on the measurement record, information for furnace analysis as analytical spike data, percent recovery, coefficient of variation, full MSA data, MSA correlation coefficient, slope and intercepts of linear fit, final sample concentration and type of background correction used, instrument run logs; integration times for AA analyses.

The Standard Operation Procedure regarding the Data Package Assembly and the deliverable contents of each type of data package is described and specified in "Data Package Assembly".

The laboratory quality control measurements are intended to ensure the generation of the analytical data of high quality and court-defensibility. However, the continuing generation of reliable data cannot be guaranteed without routine, consistent monitoring and checking the system in place. Such monitoring systems can be broadly divided into two main categories, both of which are discussed below. The interlaboratory quality control program which is participated through the laboratory certification/accreditation programs is to assess the laboratory performance on a national or interlaboratory basis. The intralaboratory quality control program which is monitored through the internal audit procedure is to assess the single laboratory performance meeting the established quality control criteria and to set up the warning system to prevent the generation of out of control data. With both programs in active status, the known quality of analytical data generated from this laboratory can be predicted and maintained.

INTERLABORATORY PERFORMANCE EVALUATION PROGRAM

Accredited Laboratories, Inc. (ALI) participates in the National Performance Evaluation (PE) Program through the laboratory certification/accreditation program, by receiving and analyzing a defined set of PE samples. The analytical results are evaluated on a national basis to indicate the quality of data (accuracy). A list of various Performance Evaluation (PE) samples derived from the laboratory certification/accreditation programs is tabulated as follows:

GOVERNING AGENT	PE SET	ESTIMATED ARRIVING (MONTH)	CATEGORY [1]
NYDOH [2]	1	January	Nonpotable/Solid Waste/Air Emission
	2	April	Potable
	3	July	Nonpotable
	4	October	Potable

USEPA	WS	April	Water Supply
(NJDEPE/PADER)	WP	September	Water Pollution
USEPA	DMR	February	NPDES
NIOSH AIHA[3]	PAT[4]-1 PAT[4]-2 PAT[4]-3 PAT[4]-4	January April July October	Metals/Solvents for industrial hygiene testing

[1] Both Organic and Inorganic parameters are involved
[2] New York State Department of Health
[3] American Industrial Hygiene Association
[4] Proficiency Analytical Testing

A total of 11 sets of PE samples are received and analyzed at Accredited Laboratories annually. The analysis evaluated in these participated interlaboratory QC programs consists of all organic, inorganic and wet chemistry parameters analyzed routinely from the laboratory. The performance evaluation results derived from these participating PE samples would be a best indicator regarding the laboratory performance at a large-pool scale. The out-of-control parameter requires a systematic evaluation of the laboratory procedure in order to determine whether the out-of-control is due to "random" error or a "systematic" error. In general, the evaluation procedure is described as follows when the out-of-control data is anticipated from the interlaboratory QC programs:

1. A level II corrective action is initiated by the QA/QC supervisor when any "Not Acceptable" PE results are received.
2. The assigned investigator will inspect all raw data files associated with the generation of PE results to determine the cause(s) and to recommend the solution(s) in order to prevent the same errors from happening again.
3. If the error is determined to be systematic, all sample analyses associated with the affected parameters must be stopped immediately. The cause(s) must be corrected prior to the sample analysis. A level III corrective action will then be automatically initiated.
4. It is the responsibility of the QA/QC supervisor to ensure the proper correction being carried out in the laboratory practice for any error(s) defined in the corrective action. The QA/QC supervisor will also be responsible to file and execute the checking procedure for all corrective actions documented. The final results of the above-mentioned processes will be reported to management.
5. It is important as a part of the QC function to monitor the laboratory performance by continuously checking the laboratory process for any defined errors being corrected through the internal audit program described below:

As a part of the requirements under laboratory certification/accreditation protocol, the

laboratory will be audited/inspected by the governing authority periodically. The audit report will serve as guidance to improve the quality of the laboratory performance. Any deficiencies must be evaluated and corrected immediately and the corrective actions will be documented for future checking and reported to management. Any recommendations derived from the audit report will be seriously considered to be adopted. Below is the list of governing authorities performing the formal lab audits periodically at ALI:

- USEPA Region II
- NJDEPE
- NYDOH
- PADER
- AIHA

In addition to the above-mentioned authorities, several private firms have also performed audits on ALI. The results of the private audit reports were treated the same as the official audits. It is the policy of ALI to welcome any interested party to audit/inspect ALI to continuously improve laboratory performance.

INTRALABORATORY INTERNAL AUDIT PROGRAM

The intralaboratory control program is defined as a continuing, systematic, in-house quality control monitoring program being carried out by the QA/QC department to ensure the highest possible quality of analytical data generated through the exercises of all pertinent quality control (QC) measurements associated with each analytical protocol. In general, there are three levels of internal audit processes involved in ALI's internal audit program as follows:

1. Laboratory Self-Monitoring Procedure

Each analyst who is responsible to generate the analytical results based on a defined protocol must generate all pertinent QC data associated with the sample results. The acceptable criteria of each monitored QC measurement must then be plotted and documented. Such QC measurements are included, but not limited to, blank contamination, sample duplicate, matrix spike/matrix spike duplicate, surrogate recovery and laboratory control sample results, etc. in either a tabulated form or on a control chart. When enough data points have been accumulated, e.g. 30 points, the standard deviation (SD) of all results can then be calculated. The control limits are usually set at +/- 3 S.D. level with a warning level at +/- 2 S.D. If any QC measurements fall outside the control limits, the situation must be corrected prior to the sample analysis. With seven consecutive points at the warning level on the same trend, it warrants the needs of investigation. It should be emphasized that the acceptable criteria for the QC measurements under the defined protocols are

usually pre-determined either by project specification or by protocol specification. It is the responsibility of the analyst to monitor continuously the performance of the QC measurements.

2. *Data Validation Procedure*

It is required to check and/or correct any error or deficiency discovered during the data validation process. It is the responsibility of the QA/QC administrator to monitor and document any errors or deficiencies discovered from the data validation process. If the situation warrants, e.g. repeated same errors, an investigation will be initiated to determine the extent of the problems. The results of the investigation must be documented and filed properly.

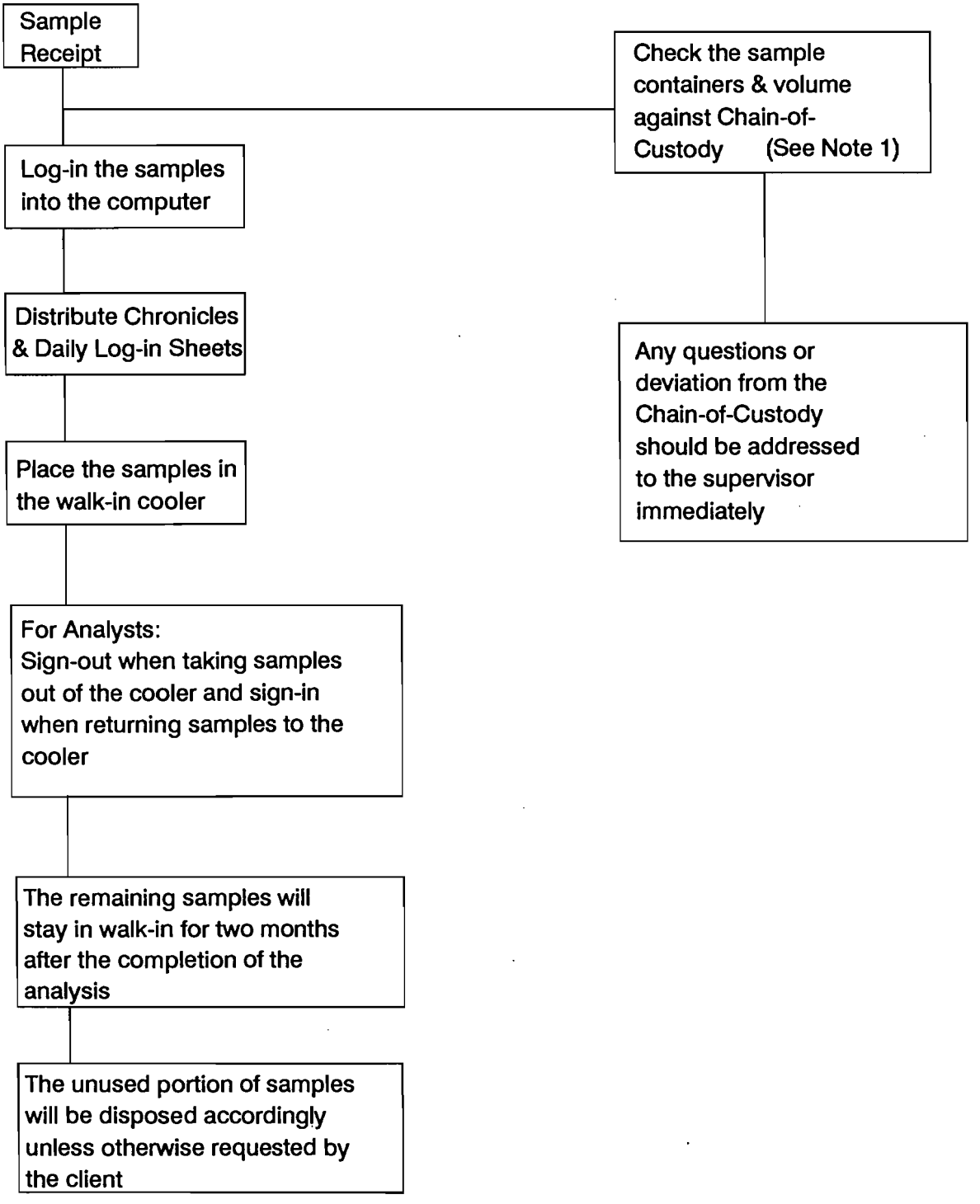
3. *Internal Audit Procedure*

Periodically it is required to conduct the lab internal audit. This is a systematic evaluation of all QC protocols carried out by the whole laboratory. The QA/QC supervisor or his/her designator is responsible to perform such a task. The corrected deficiencies derived from the interlaboratory audit process, self-monitoring procedure and data validation process are also required to be covered in the internal audit evaluation procedures. The ultimate objectives of the internal audit program are to provide the information listed as follows for the continuing improvement of laboratory performance.

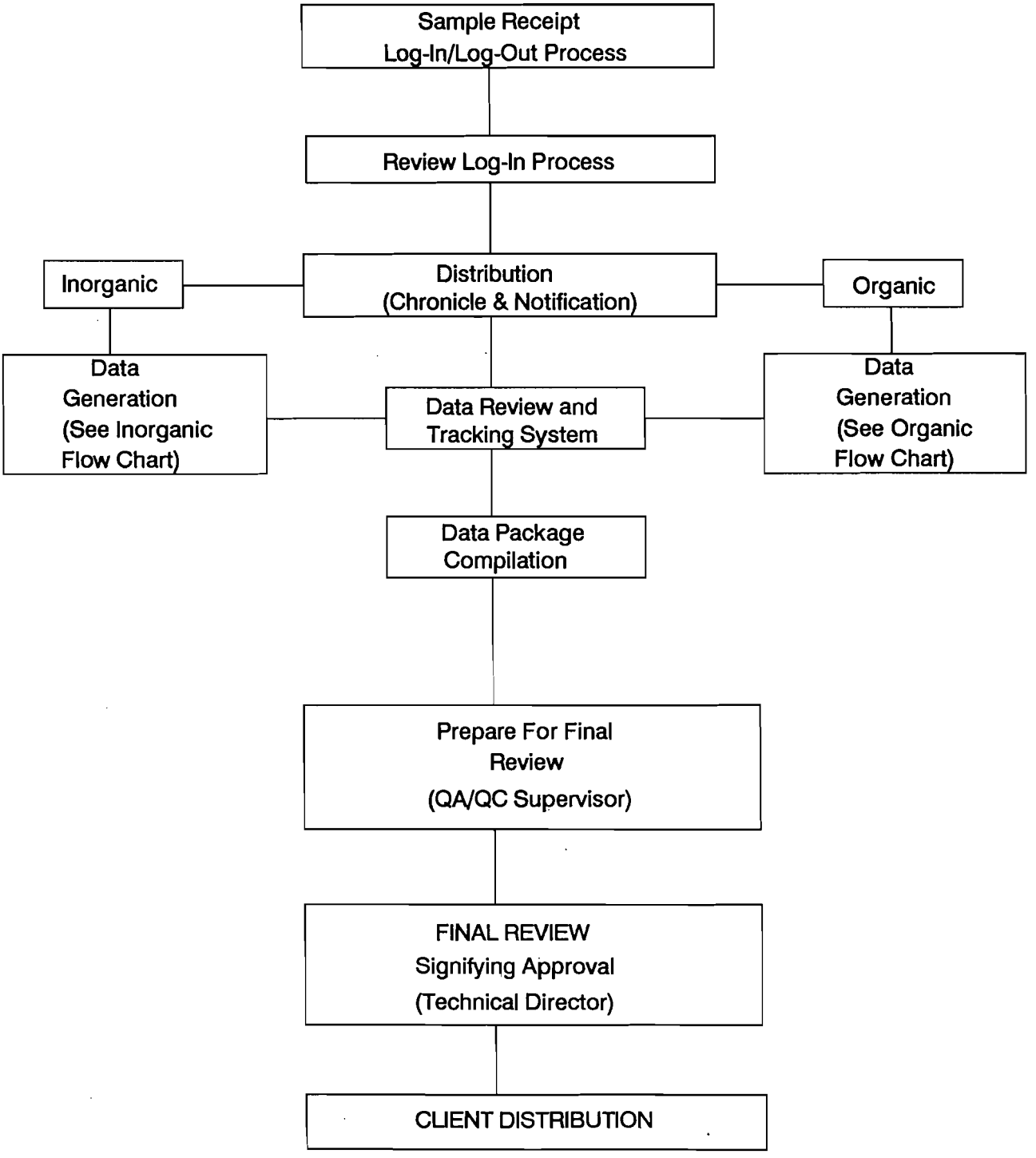
- To establish the predicted accuracy and precision of all analytical methods.
- To maintain and ensure the data quality at a defined standard.
- To identify any weak links e.g. personnel, equipment or methodology associated with the QC problems and to suggest the solution(s) of such problems.
- To recommend the procedures for the future data quality improvement and to reestablish the data quality standard for the future adoption.

Environmental Sample Flow Chart Within ALI

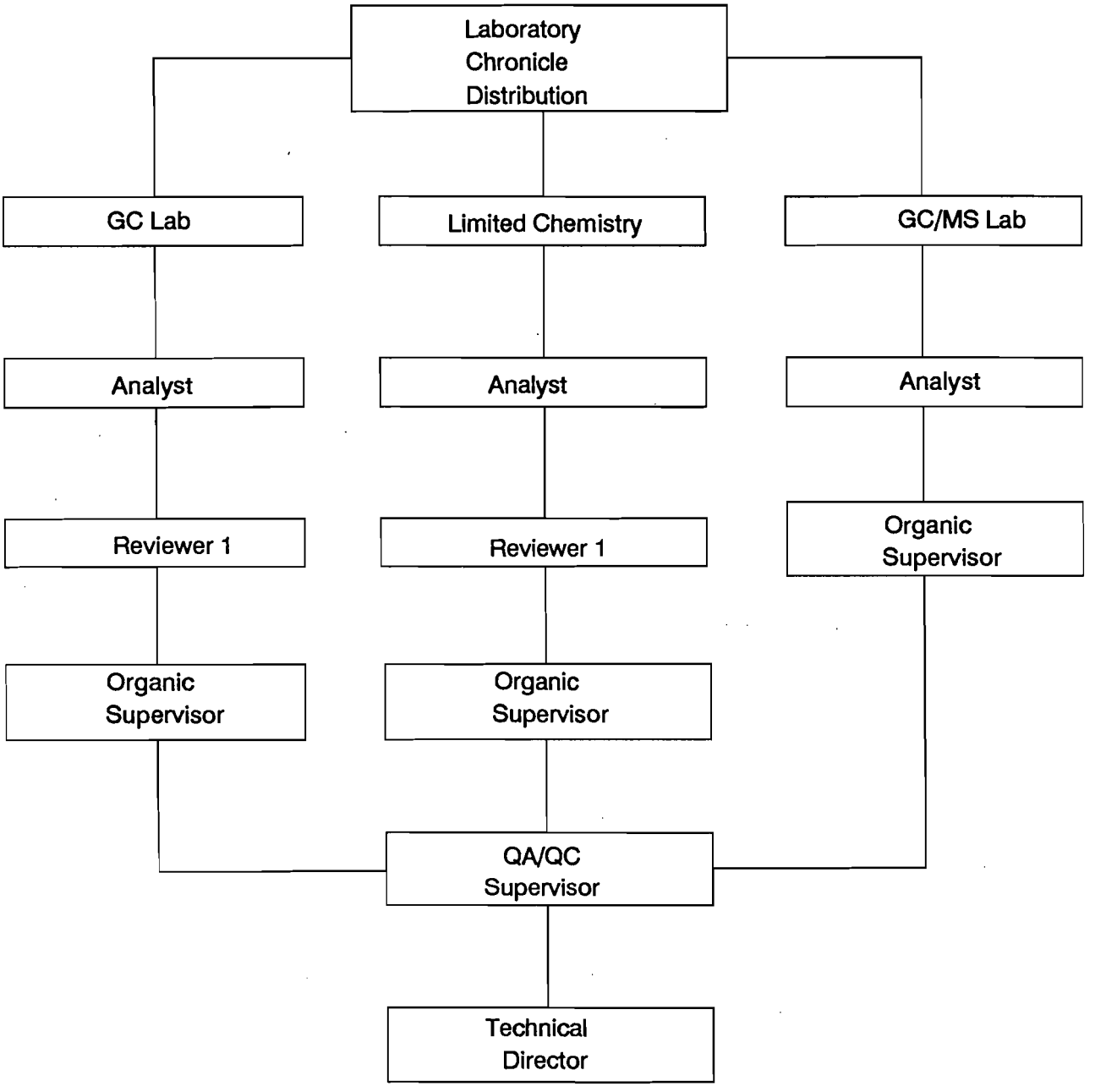
(Maintaining Sample Integrity)



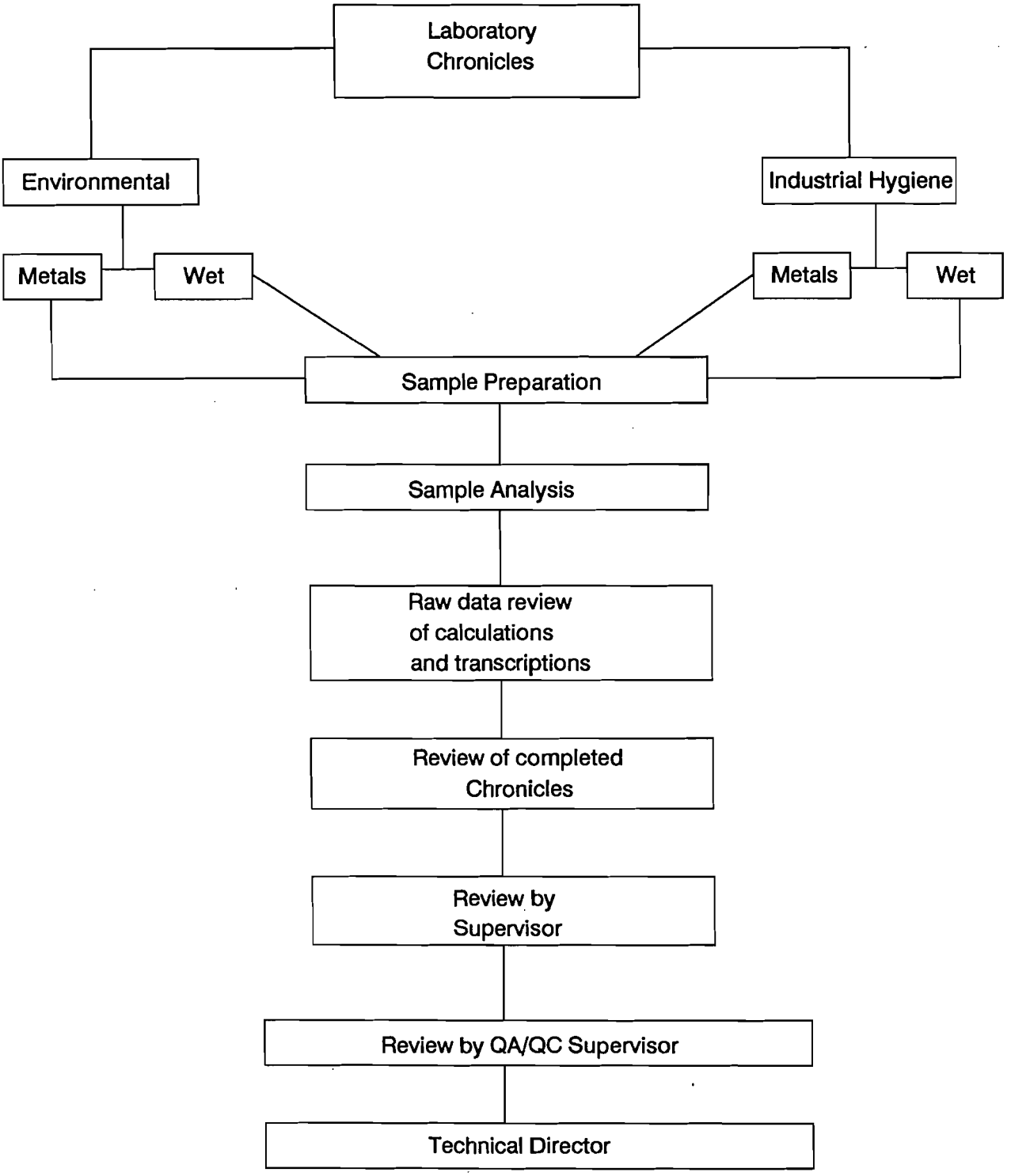
DATA INTEGRITY: QA/QC DEPARTMENT



DATA REVIEW
ORGANIC LABORATORY



DATA REVIEW
INORGANIC LABORATORY



SAMPLE INTEGRITY (Note 1)

In order to maintain sample integrity, the following precautions need to be exercised during the sample log-in procedure.

1. One chain-of-custody form must accompany each sampling episode with all the necessary information regarding the sampling such as, sampling date and time, sample volume and container, analysis requested, preservatives, turnaround time, type of data package, client name, project name, contact person and phone number, purchase order number, special comments and other pertinent information regarding the project.

2. Contact the supervisor immediately if the information or the chain-of-custody form associated with the samples is improper or incomplete.

3. The supervisor must resolve the problem and/or discrepancy associated with the chain-of-custody with the client immediately before the case can be logged in the computer.

4. Check to see if any requested analysis exceeds its legal holding time, particularly those very short holding time parameters, such as Cr+6, fecal coliform, etc.

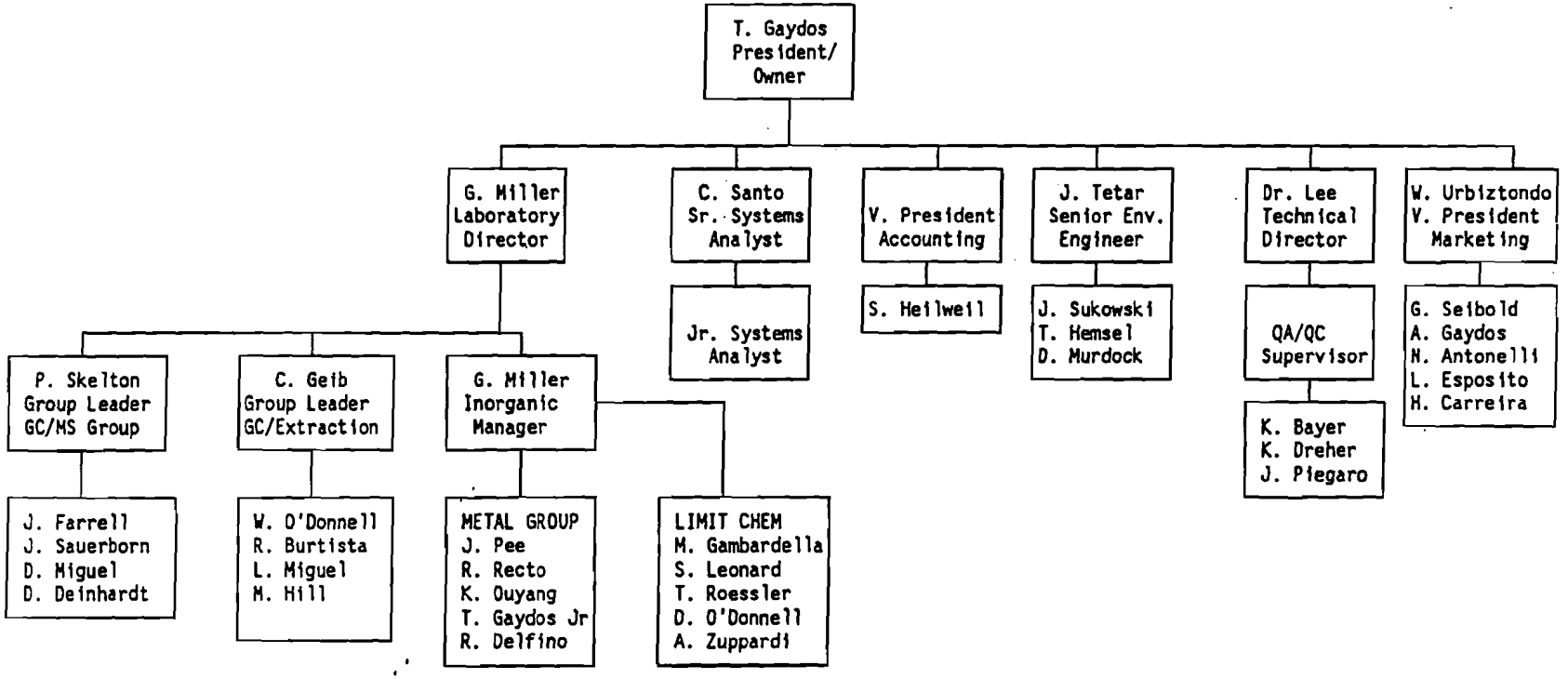
5. Report to supervisor immediately if the time period between sampling date and receiving date.

6. Start logging in the samples only when all the information on the chain-of-custody form has been checked properly and all containers of samples are in proper condition.

PERSONNEL

ACCREDITED LABORATORIES, INC.

Organization Chart



10/96

January 1971 - October 1986:

Specialist, Exxon Research and Engineering, New Jersey

Worked for Exxon Research and Engineering in a variety of capacities outlined below:

1986: Engineered the building of a reactor to study the aromatization of hydrocarbon feed material via catalytic activity. The system consisted of an HP 5995C GC/MS analyzer interfaced to an HP 1000 mini computer.

1985: Engineered the installation of an Omnisorp 360; instrumentation designed to study the microporosity of compounds along with BET surface area and total pore volume. The 360 was interfaced with an HP Series 200 computer.

1984: Designed an automated system for a collection of units termed Naphtha Aromatization Units, which were interfaced to an HP 85B computer.

1975-82: Large scale pilot projects in the plant and laboratory environment.

1971-75: Organic synthesis and analytical responsibilities utilizing HP 5840 GC analysis, NMR, Scanning IR and HPLC.

FORMAL TRAINING:

Hewlett Packard 5 Day Seminar, "GC/MS Operation and Maintenance", El Segundo, CA, 1985

Intro to Mass Spec Interpretation

THEODORE GAYDOS
Accredited Laboratories, Inc.
President - CEO

EDUCATION:

A.A.S. Chemistry, Middlesex County Community College, May 1969. 40 credits in Chemistry (transcript)

EXPERIENCE:

July 1994 to Present:

President/CEO - Accredited Laboratories, Inc., Carteret, New Jersey

Responsible for all aspects of daily business and operations

February 1992 - July 1994:

General Manager - Accredited Laboratories, Inc., Carteret, New Jersey

Responsible for the future development of the laboratory along with direction of the overall operations. Oversaw Quality Control and Assurance programs to insure the integrity of data was met, based on EPA protocols. Guaranteed client confidentiality and service, exceeding the standards demanded within the environmental business.

October 1988 - February 1992:

Analytical Laboratory Manager, Accredited Laboratories, Inc., Carteret, New Jersey

Primarily responsible for supervision of entire analytical laboratory; responsible for data generated to QA/QC. Responsible for running of GC/MS instrumentation (volatiles and semivolatiles).

October 1986 - October 1988:

Technical Specialist, Engelhard Research Laboratory, New Jersey

Worked for Engelhard Research as a specialist to design automated units used to study the combustion effects on automotive catalysts utilizing mass spectrometry (VG instrumentation).

January 1971 - October 1986:

Specialist, Exxon Research and Engineering, New Jersey

Worked for Exxon Research and Engineering in a variety of capacities outlined below:

1986: Engineered the building of a reactor to study the aromatization of hydrocarbon feed material via catalytic activity. The system consisted of an HP 5995C GC/MS analyzer interfaced to an HP 1000 mini computer.

1985: Engineered the installation of an Omnisorp 360; instrumentation designed to study the microporosity of compounds along with BET surface area and total pore volume. The 360 was interfaced with an HP Series 200 computer.

1984: Designed an automated system for a collection of units termed Naphtha Aromatization Units, which were interfaced to an HP 85B computer.

1975-82: Large scale pilot projects in the plant and laboratory environment.

1971-75: Organic synthesis and analytical responsibilities utilizing HP 5840 GC analysis, NMR, Scanning IR and HPLC.

FORMAL TRAINING:

Hewlett Packard 5 Day Seminar, "GC/MS Operation and Maintenance", El Segundo, CA, 1985

Intro to Mass Spec Interpretation

YUN-SHEN LEE
Accredited Laboratories, Inc.
Technical Director

EDUCATION:

Ph.D. Environmental Sciences, Rutgers, The State University of New Jersey - July 1981

M.S. Environmental Health Sciences, School of Public Health, University of California, Berkeley, California - December 1977

B.S. Plant Pathology, Chung-Hsing University, Taiwan - June 1970

EXPERIENCE:

August 1988 - Present

Technical Director - Accredited Laboratories, Inc., Carteret, New Jersey

Oversees all aspects of technical functions within the laboratory operation, including quality assurance/quality control functions, as well as establishing and implementing the laboratory's internal auditing system to ensure that all analytical data meets USEPA and State agency requirements to be court defensible without sacrificing efficient laboratory operation. In addition, monitors trends in environmental law and EPA policy for incorporation into the laboratory's functions, implementing and advising about newly developed techniques which can be incorporated into the laboratory's practices. Finally, makes recommendations on major equipment purchases, new employee hiring and long-term business approaches.

June 1982 - July 1988

Laboratory Supervisor, NJDEPE, Bureau of Environmental Laboratories, West Trenton, New Jersey

Directed the operation of New Jersey DEPE's laboratory, including the Pesticide Enforcement, Organic, Inorganic and GC/MS sections. Working with the QA supervisor, coordinated all four sections to maximize efficiency while ensuring accuracy and precision of the data generated. In addition, reviewed and approved all data, and interacted with clients to develop project plans and interpret analytical results. Administrative duties also included the recommendation of laboratory equipment purchases, establishment of training plans for laboratory personnel, operational safety plans and personnel performance evaluations.

May 1981 - May 1982

Analytical Chemist, SCA Chemical Service, Inc., Newark, New Jersey

Performed analyses to evaluate the characteristics of various hazardous wastes to determine the treatment processes for these wastes. Also established the analytical procedures to measure the components in the wastes qualitatively and quantitatively via both wet chemistry and instrumental analytical techniques.

June 1978 - June 1981 (Part Time)

Research Assistant, Environmental Sciences Department, Rutgers University, New Brunswick, New Jersey

Performed research utilizing the computerized GC/MS to analyze the EPA priority pollutants in ground and surface waters.

October 1975 - December 1977 (Part Time)

Research Assistant, Environmental Health Science Department, University of California at Berkeley, Berkeley, California

Research centered on developing the "environmental weathering control chamber", studying the relationship between the weathering factors such as ozone/dust and the disappearance rates of various organophosphorus insecticides and the production rates of their oxidative derivatives on citrus foliage.

January 1972 - January 1975

Research Assistant, Pesticide Residue Division, Taiwan Plant Protection Center, Taiwan

Analyzed and generated results for pesticide residue in environmental samples.

FORMAL TRAINING:

Interpretation of Spectra, Rutgers, The State University of New Jersey, 1979.

GAEL MILLER
Accredited Laboratories, Inc.
Laboratory Director

Education:

B.A. Chemistry, Rutgers, The State University of New Jersey, Cook College. June, 1988.

Experience:

January 1996:
Laboratory Director, Accredited Laboratories, Inc., Carteret, New Jersey

Primary responsibility is to develop and manage the entire ALI laboratory operation. Reports directly to the President.

July, 1984 to August, 1988:

Chemist, Lever Research Laboratories, Edgewater, New Jersey

Perform R & D analysis on various commercial product lines using ICP.

July, 1984 to August, 1988;

Laboratory Technician, Lawler, Matusky & Skelly Engineers, Pearl River, New York

Perform limited chemistries and AA Spectroscopy for environmental samples.

Formal Training:

Inductively Coupled Argon Plasma Spectroscopy, March, 1986.
Ward Scientific Programming
Supervision Seminar
Orion Selective Electro Seminar

CLIFFORD GEIB
Accredited Laboratories, Inc.
Group Leader
GC Laboratory
Extraction Laboratory

EDUCATION:

2 years towards AA	Middlesex County Community College, 1989-90
	Kean College 1986-87
	NJIT 1973-75

EXPERIENCE:

June 1993 - Present

Group Leader, Accredited Laboratories, Inc., Carteret, New Jersey

Coordinates and reviews Organic Department workload including Extractions, GC operation and personnel.

March 1988 - May 1992

Senior Laboratory Technician, Accredited laboratories, Inc., Carteret, New Jersey

Primary responsibility included GC operation, emission spectroscopy, X-ray fluorescence and ICP equipment. Also performed extraction of organic environmental samples.

July 1976 - February 1988

Laboratory Technician IV, U.S. Metals Refining Company, Carteret, New Jersey

Operated emission spectroscopy, x-ray fluorescence and wet chemistries.

FORMAL TRAINING:

Wastewater Operations I & II, Middlesex County Community College
Industrial Spectroscopy, Arizona State College
HP 5890 Trouble Shooting and Maintenance - H.P.
Introduction to GCD Technology

PAUL ROBERT SKELTON
Accredited Laboratories, Inc.
Group Leader
GC/MS Laboratory

EDUCATION:

B.S. Biology, Utica College of Syracuse University, Utica, New York - May 1990

Concentration in Ecological Studies

EXPERIENCE:

June 1993 - Present

Group Leader - GC/MS Department, Accredited Laboratories, Inc., Carteret, New Jersey

Responsible for the scheduling and organization of the GC/MS Department. Also responsible for the analysis of organic samples for semi-volatile compounds utilizing Methods 625 and 8270.

December 1990 - May 1993

GC/MS Analyst, Accredited Laboratories, Inc., Carteret, New Jersey

Operated Hewlett Packard 5890 Gas Chromatograph with 5970 Mass Selective Detector. Used Tekmar ALC 2016 and LCS 2000 Purge and Trap. Utilized Aquarius based operating software. Expertise in EPA Methods 624, 8240, 524.2, TCLP and CLP SOW 90, covering all Organic Volatiles.

FORMAL TRAINING:

Introduction to Mass Spec Interpretation - H.P.
Capillary GC Columns & Detectors - H.P.
Capillary GC Sample Introduction Techniques - H.P.
GC/MS and other Hypenated Techniques - H.P.

CARLOS SANTO
Accredited Laboratories, Inc.
Senior Systems Analyst

EDUCATION:

B.S. Electrical Engineering, New Jersey Institute of Technology - June 1990

EXPERIENCE:

October 1990 - Present

Senior Systems Analyst, Accredited Laboratories, Inc., Carteret, New Jersey

Experience in all aspects of managing and ensuring quality control of all computer systems, as well as generating and updating all in-house automated deliverables. Also installs and operates software.

August 1989 - September 1990

Independent Consultant

Worked in all aspects of programming, installation and operation of laboratory software.

FORMAL TRAINING:

Computer Courses - "C" Programming, Fortran Programming, Pascal Programming, Assembly Programming System 1 and System 2

KATHLEEN BAYER
Accredited Laboratories, Inc.
QA/QC Administrator

EDUCATION:

A.S. Secretarial Science, Middlesex County College, New Jersey - June 1979

EXPERIENCE:

August 1990 - Present

QA/QC Administrator, Accredited Laboratories, Inc., Carteret, New Jersey

Responsible for sample and data tracking system; reviewing and assembling data packages; data package filing and retrieving; all QA/QC documentation associated with data generation including lab notebook control, QC data reviewing and control, and the initiation and control on QC corrective actions.

August 1979 - July 1990

Planner/Scheduler, Dranetz Technologies, Inc., Edison, New Jersey

Responsible for all phases of documentation systems.

FORMAL TRAINING:

Word Perfect Course, 1990
How To Supervise People, October 1991

JOHN TETAR
Accredited Laboratories, Inc.
Senior Environmental Engineer

EDUCATION:

B.S. Chemical Engineering, New Jersey Institute of Technology - 1979

EXPERIENCE:

October 1989 - Present

Senior Environmental Engineer, Accredited Laboratories, Inc., Carteret, New Jersey

Manages the Environmental Service Development and Field Service personnel. Additional responsibilities include:

Development and implementation of environmental studies and audits
Permit preparation and attainment
Environmental control research and recommendations
Industrial hygiene audits
Interaction with regulatory agencies
Technical Advisor for on-site wastewater treatment plant
Environmental reporting and recordkeeping
Waste material management
Emergency Response Planning

January 1986 - October 1989

Environmental Engineer, Accredited Laboratories, Inc., Carteret, New Jersey

Assisted in converting a research and development laboratory into a full service commercial environmental laboratory. Responsibilities included development and implementation of sampling plans, project cost estimations, report writing, and the procurement of required instrumentation.

December 1983 - January 1986

Environmental Engineer/Industrial Hygienist, United States Metals Refining Company (AMAX), Carteret, New Jersey

Assured through monitoring and training that the company was in compliance with all applicable Federal and State environmental and safety regulations.

July 1982 - December 1983

Safety Engineer, United States Metals Refining Company, Carteret, New Jersey

Included those Industrial Hygiene duties above with additional responsibilities in the area of safety and fire protection.

February 1980 - July 1982

Environmental Engineer, United States Metals Refining Company, Carteret, New Jersey

Entry level position. Responsible for the water monitoring program. Conducted research work in the area of heavy metal removal from process waste streams.

CERTIFICATIONS:

NJDEP N-2 Industrial Wastewater Treatment Operator License
Certified Hazardous Materials Manager (CHMM) Master's Level

PROFESSIONAL SOCIETIES:

American Institute of Chemical Engineers
Air and Waste Management Association
American Industrial Hygiene Association
Institute of Hazardous Materials Management

PERSONNEL TRAINING

Accredited Laboratories, Inc. recognizes the importance and value of the continuing professional development through personnel training for its staff. The training program is carried out through both internal and external training seminars.

Periodically, the QA/QC department is responsible for conducting in-house training seminars. The purpose of this program is to inform the operation staff of the current changes and/or adoptions of new governing regulations imposed by either federal or state agents, as well as the analytical protocols associated with those new regulations. This is considered to be part of the internal communication network ensuring the staff is informed on all current requirements. For each individual, the job-related training program is carried out by the department and is the responsibility of his/her immediate supervisor. All internal training activities must be recorded by the sponsor's department, then forwarded to their personnel files.

Each year the department supervisor/manager is required to submit an annual external training activity schedule for his/her staff. Each program will be scheduled and approved by the General Manager or his/her designee. The objective of the external training is to improve the techniques of the staff, by experts within the field. Such techniques, i.e. QA/QC practices, instrument trouble-shooting, better efficiency in laboratory operation and management, newly developed sample preparation systems, improving laboratory automation systems both in sample receipt and sample process procedures are covered.

Implementing both internal and external training programs at Accredited Laboratories, Inc. ensures that the professional quality of the staff is maintained at the highest levels. In effect, the quality of analytical data is maintained at the highest level possible.

APPENDIX

SCHEDULE OF YOUR CURRENT INSURANCE

INSURED: Accredited Laboratories, Inc.

DATE: 7-12-96

COVERAGE	AMOUNT/LIMITS	EXP. DATE	COMPANY	POLICY NUMBER	PREMIUM	DED. AMOUN
*GENERAL LIABILITY	1,000,000 Occ. 2,000,000 Agg.	7-8-97	MONTICELLO	0005-95-01748	10,000	2,500
*EXCESS LIABILITY	1,000,000	7-8-97			3,780.10	
*PROFESSIONAL LIABILITY	1,000,000	8-1-97	GULF Ins.	G6110580	8,500	15,000
*BOILER & MACHINERY	5,000,000	7-8-97	CHUBB	7835-21-93	1,637	1,000
WORKERS COMPENSATION		7-13-97	ITT HARTFORD	123	9,711	
BODILY INJURY BY ACCIDENT	1,000,000					
BODILY INJURY BY DISEASE-POLICY LIMIT	1,000,000					
BODILY INJURY BY DISEASE-EACH EMPLOYEE	1,000,000					
PROPERTY		8-1-97	CHUBB	3534-72-91		
BUILDING	3,120,000					
BUSINESS PERSONAL PROPERTY	1,000,000					
BUSINESS INCOME	1,200,000					
AUTOMOBILE INSURANCE		8-1-97	CHUBB	BAP9773229787		
LIABILITY	1,000,000					
MEDICAL PAYMENTS	10,000					
UNINSURED MOTORIST	1,000,000					
NON-OWNED AUTO/HIRED AUTO	1,000,000					
INLAND MARINE		8-1-97	CHUBB	3534-72-91		
EDP	1,550,000					
PROPERTY IN TRNSIT	155,000					

date: 1/23/95
written by: L PETRACCARO
reviewed by: P GLEASON

METHOD 624

SCOPE AND APPLICATION

This method covers the determination of a number of purgeable organics as seen in Table 1. This is a purge and trap gas chromatographic/mass spectrometer method applicable to the determination of these compounds in municipal and industrial discharges. The method detection limit for each parameter is also listed in Table 1. This method is restricted to use by or under the supervision of analysts experienced in the use of purge and trap gas chromatograph/mass spectrometer systems and skilled in the interpretation of mass spectra. The MDL is calulated using the accepted EPA determined method.

METHOD SUMMARY

This is a purge and trap gas chromatograph/mass spectrometer method for the determination of volatile organics in aqueous samples. The analytical system consists of: a Tekmar LSC 2000 purge and trap concentrator (or a Tekmar 3000) with a Tekmar ALS 2016 autosampler, a Hewlett Packard 5890 series 2 gas chromatograph with a Hewlett Packard 5970 MSD mass spectrometer. Data is analyzed using Aquarius data package revision D on a Hewlett Packard 1000 computer system. Data is archived using a Hewlett Packard 7970E tape drive.

Ultra high purity helium is bubbled through a 5mL aliquot of sample held in appropriate glassware on the purge and trap autosampler. The purgeables in the sample are effectively transferred by the inert helium purge gas from the aqueous to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed the sorbent trap is heated and backflushed with ultra high purity helium to desorb the purgeables from the trap onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeable organics which are then detected by the mass spectrometer.

INTERFERENCES

The analytical system is proven to be free from contamination on a daily basis. A reagent blank is analyzed every day and must show that interferences from the analytical system are under control before sample analysis is to proceed.

SAFETY AND HAZARDS

Most of the compounds analyzed are known carcinogens. All standards and samples should be handled with extreme care. Proper precautions must be taken, including wearing protective gloves and eyewear, wearing a labcoat, and operating under a lab hood.

EQUIPMENT-ANALYTICAL SYSTEM

1. Tekmar ALS 2016 autosampler fitted with clean glassware.
   - A. Glassware
     - a. 5mL fritted
     - b. 5mL frittless

2. Tekmar LSC 2000 (or 3000) purge and trap concentrator
   - A. Traps
     - a. Tekmar purge trap F (SP-2100/TENAX/Silica gel)
3. Hewlett Packard 5890 series 2 gas chromatograph
   - A. Columns
     - a. Restek 105m,0.53mm ID,3.0um RTx- 502.2
4. Hewlett Packard 5970 MSD mass selective detector
5. Hewlett Packard 1000 computer system
   - A. Aquarius data package revision D
6. Hewlett Packard 7970E magnetic tape drive

REAGENTS AND STANDARDS

1. Methanol - Baxter (Burdick and Jackson) high purity solvent
2. Supelco Purgeable A w/DCB cat# 7969
3. Supleco Purgeable B w/DCB cat# 7970
4. Supelco Purgeable C cat# 8853
5. Absolute Internal Standards Mix cat# 20009
6. Absolute System Monitoring Compounds Mix cat# 20010
7. Supelco 4-Bromofluorobenzene cat# 8800
8. Nanopure water proven to be free of volatile organics
9. EM Science Omnisolv water

See Table 2 for contents and concentrations of standards

SAMPLE PREPARATION

Samples are collected without headspace in 40mL vials with teflon septa seal screw caps. Nonpreserved aqueous samples must be analyzed within seven days of collection. Preserved aqueous samples must be analyzed within fourteen days of collections.

STANDARD PREPERATION

NOTE: ALWAYS USE CLEAN APPROPRIATE SYRINGES WHEN TRANSFERING STANDARDS TO WORKING STOCK SOLUTIONS. WASH SYRINGES BETWEEN USES WITH METHANOL.

1. 624 calibration working stock standard solution
   A. Prepare for all target analytes (except gases) at 25 ppm and for system monitoring compounds at 15 ppm.
   B. Store in labeled 2 mL teflon screw cap vials.

2. Purgeable C working stock standard solution
   A. Prepare for Purgeable C mix compounds at 25 ppm.
   B. Store in labeled 2 mL teflon screw cap vials.

3. 624 internal standards working stock standard solution
   A. Prepare for Purgeable Internal Standards mix compounds at 15 ppm.
   B. Store in labeled 2 mL teflon screw cap vials.

4. 624 internal standard/surrogate working stock solution
   A. Prepare for Purgeable Internal Standards mix compounds and system monitoring compounds at 15 ppm.
   B. Store in labeled 2 mL teflon screw cap vials.

1. Tekmar LSC 2000 (or 3000) purge and trap concentrator
   A. Purge gas - Ultra high purity helium
   B. Purge time/temp - 11 min/32 degrees C
   C. Desorb time/temp - 1.5 min/180 degrees C
   D. Bake time/temp - 10 min/200 degrees C
   E. Equilibrium temp - 30 degrees C
   F. 2016 Transfer line temp - 125 degrees C

2. Hewlett Packard 5890 series 2 gas chromatograph
   A. Carrier gas - Ultra high purity helium
   B. Initial time/temp - 10 min/30 degrees C
   C. Ramping rate - 6 degrees C/min
   D. Final temp - 200 degrees C
   E. Transfer line temp - 120 degrees C

3. Flow rates
   A. Purge gas - 40mL/min
   B. Carrier gas - 30mL/min

SAMPLE ANALYSIS

1. Mass spectrometer tuning
   A. Using a 50ppm working stock standard solution of 4-Bromofluorobenzene, inject 1uL onto the gas chromatographic column during a single sample run of the GC/MS.
   B. The mass spectra of the injected BFB must pass the EPA conditions seen in Table 3.
   C. The time of injection for an acceptable BFB tune begins the 24 hour time clock for sample analysis.

- TABLE 3 -

GC/MS PERFORMANCE STANDARD
Bromofluorobenzene (BFB)

m/z	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5-9% of mass 174
176	95-101% of mass 174
177	5-9% of mass 176

2. Initial calibration
   A. A three point calibration curve is necessary for EPA method 624. Analyze three points at 2ppb, 20ppb, and 200ppb.
   B. Using the working stock standards solutions for the 624 Calibration mix and the Purgeable C mix, inject the proper amounts into a 5mL syringe filled with contamination free water. Be sure to use a clean 10uL syringe and wash with methanol between uses. Then add 10uL of 624 Internal Standards mix into the same syringe.
   C. Follow above procedure for each of the three points.
   D. If the three point calibration is acceptably linear (all parameters show <35 % rsd) then analysis may proceed.
   E. If necessary a calibration curve using the first degree equation of the line can be used as per EPA test method 624 for purgeables.

3. Daily calibration
   A. A daily calibration may be run against an existing three point to prove the systems linearity for the day.
   B. A single point 20ppb standard is set up the same way as in the initial calibration.
   C. If the single point passes against the existing three point (all parameters show <35 % diff) then analysis may proceed. If not then a new calibration curve must be generated.

4. Daily blank
   A. A blank is run after an acceptable calibration to prove the system is free from contamination.
   B. Three blanks may be run to prove system clean.
   C. Once system is proven clean sample analysis may begin.

5. QC sample spikes
   A. Every 20 samples or 30 days, whichever is more frequent a 20ppb spiked sample is run for quality control purposes.
   B. Every 20 samples or 30 days, whichever is more frequent a 20ppb blank spike is run for quality control purposes.
   c. Every 20 samples or 30 days, whichever is more frequent a sample duplicate is run for quality control purposes.

ANALYSIS OF VOLATILE ORGANICS GC/MS
(Method 8240)

Written By: Ted Gaydos
Reviewed By: Yun-shen Lee Date: 7/13/89

METHOD SUMMARY

This is a Purge and Trap - Gas Chromatograph/Mass Spectrometer Method for the determination of volatile organics in aqueous samples. This procedure is conducted in accordance with EPA Method 8240, SW-846. The analytical system consists of: a Tekmar LSC purge and trap with a Tekmar ALS automated sampler, a Hewlett Packard 5996 gas chromatograph/mass spectrometer, a Hewlett Packard 1000 computer utilizing the aquarius data package revision D. A Hewlett Packard 7070E magnetic tape drive is used for archiving data.

SCOPE OF THE METHOD

The following compounds can be analyzed by this procedure.

Acrolein	Acrylonitrile
Acetone	Carbon Disulfide
Chloromethane	trans 1,3-Dichloropropene
Bromomethane	Trichloroethylene
Vinyl Chloride	Benzene
Chloroethane	cis 1,3-Dichloropropene
Methylene Chloride	1,1,2-Trichloroethane
Trichlorofluoromethane	Chlorodibromomethane
1,1-Dichloroethene	Bromoform
1,1-Dichloroethane	Tetrachloroethylene
trans 1,2-Dichloroethene	1,1,2,2-Tetrachloroethane
Chloroform	Toluene
1,2-Dichloroethane	Chlorobenzene
1,1,1-Trichloroethane	Ethyl Benzene
Carbon Tetrachloride	1,2-Dichlorobenzene
Bromodichloromethane	1,3-Dichlorobenzene
1,2-Dichloropropane	1,4-Dichlorobenzene
2-chloroethyl vinyl ether	2-Butanone
2-Hexanone	4-Methyl-2-Pentanone
Vinyl Acetate	Styrene
Xylenes	cis 1,2-Dichloroethene

The detection limit of these compounds in aqueous samples is 5 ug/l.

SAMPLE PREPARATION

Samples are collected without head space in 40 ml vials with teflon lines screw caps. Aqueous samples must be analyzed within fourteen days of collection.

INTERFERENCES

A reagent blank is analyzed daily to check for impurities and laboratory contamination. The system interferences must be minimal and under control before sample analysis begins.

PURPOSE

To analyze aqueous/soil samples for volatile organics by Purge and Trap GC/MS.

EQUIPMENT

1. Tekmar ALS automated sampler.
2. Tekmar LSC purge and trap.
3. Hewlett Packard 5996 gas chromatograph/mass spectrometer.
4. Hewlett Packard 1000 computer.
5. Aquarious Data package Revision D.
6. Hewlett Packard 7970E magnetic tape drive.
7. Column: Supelco 1% SP1000 on 60/80 carbopack B 2mm ID x6'.
8. Purge Tubes: Tekmar 5ml.

REAGENTS AND STANDARDS

1. Methanol: Burdick and Jackson high purity solvent.

2. Standard solution.

   a. Supelco purgeable A kit, quantitative standard #4-8851 containing the following in methanol;

Carbontetrachloride	200ug/ml
Chlorobenzene	200ug/ml
2-Chloroethylvinyl ether	200ug/ml
Chloroform	200ug/ml
Dibromochloromethane	200ug/ml
1,1-Dichloroethane	200ug/ml
1,1-Dichlorethylene	200ug/ml
1,2-Dichloropropane	200ug/ml
Methylene Chloride	200ug/ml
Tetrachlorethylene	200ug/ml
1,1,2-Trichloroethane	200ug/ml
Trichloroethylene	200ug/ml
Trichlorfluoromethane	200ug/ml

   b. Supelco purgeable B kit, quantitative standard #4-8852 containing the following in methanol:

Benzene	200ug/ml
Bromodichloromethane	200ug/ml
Bromoform	200ug/ml
1,2-Dichloroethane	200ug/ml
trans-1,2-Dichloroethylene	200ug/ml
1,3-Dichloropropene	400ug/ml
Ethyl benzene	200ug/ml

1,1,2,2-Tetrachloroethane	200ug/ml
Toluene	200ug/ml
1,1,1-Trichloroethane	200ug/ml

c. Supelco purgeable C kit, quantitative standard #48853 containing the following in methanol:

Bromomethane	200ug/ml
Chloromethane	200ug/ml
Chloromethane	200ug/ml
Vinyl chloride	200ug/ml

d. 2-Chloroethyl vinyl ether — Supelco P/N 4-8516

e. 1,2-Dichlorobenzene — Supelco P/N 4-8522

f. 1,3-Dichlorobenzene — Supelco P/N 4-8523

g. 1,4-Dichlorobenzene — Supelco P/N 4-8524

3. Surrogate. Consisting of:
   a. Supelco reference standard #4-8800.
   4-Bromofluorobenzene, Toluene-d8 and 1,2-Dichloroethane-d4 in methanol.

   b. Supelco reference standard #4-8876.
   Bromochloromethane, 1-Chloro-2-Bromopropane and 1,4-Dichlorobutane in methanol.

4. Type II laboratory water proven to be free of volatile organics.

HAZARDS

Most of the compounds analyzed are known carcinogens. Standard should be handled with extreme care. See appropriate MSDS sheets for more information.

PROCEDURE

PREPARATION OF STANDARDS

1. Using a 250 microliter syringe, deliver exactly 125 ul each of purgeable A, B and C (Supelco Catalog numbers 8851, 8852 and 8853, respectively). Then add 12.5 ul of hazardous substance liquid volatiles using a 25 microliter syringe (Supelco Catalog #8920) and dilute to a total volume of 1000 microliters. This is your working stock standard solution.

2. Accurately weigh to 4 significant figures 0.100g each of 2-Chloroethyl vinyl ether, 1,2-Dichlorobenzene, 1,3-Dichlorobenzene and 1,4-Dichlorobenzene, into a 100 ml volumetric flask. Dilute to volume with methanol and mix thoroughly. this is a 1000 ppm "other purgeables" stock solution.

3. Using a 100 ul syringe deliver 50 ul of the solution prepared in Step #3 into

1ml of methanol and mix thoroughly. This is a 50 ul/ml "other purgeables" solution.

PREPARATION OF INTERNAL STANDARD/SURROGATE SOLUTION

4. Using a 25 microliter (ul) syringe, deliver exactly 25 ul of internal standard mix #4-8876 (Supelco) and dilute to a volume of 1000 ul with methanol. this is a 50 ug/ml stock internal standard solution.

5. Using a 100 microliter syringe deliver exactly 100 ul of surrogate standard (Supelco Catalog #4-8835) and dilute to a volume of 100 ul with methanol. This is equivalent to a 50 ug/ml stock surrogate standard solution.

6. The stock solutions in Step 1 and 2 may be combined into one working stock solution.

SAMPLE ANALYSIS

7. At the beginning of each day, check the chromatographic conditions to ensure they have not been changed. The chromatographic conditions for volatile organic analysis should be:

Trap:	Tekmar 12" x 1/8" 3 ring trap containing Tenex/Silica Gel/Charcoal
Gas:	He @40ml/min
Purge:	11 min @ 30 degrees C
Desorb:	4 min @ 180 degrees C
Bake:	12 min @ 220 degrees C
Equilibrium Temp:	30 degrees C

Gas Chromatographic Conditions:

Column:	Supelco 1% SP1000 60/80 Carbopack B 2mm ID x 6'
Initial:	3 min @ 45 degrees C
Ramp:	8 degrees C/min
Final:	15 min @ 220 degrees C
Carrier Gas:	He @ 30 ml/min
Transfer Line Temp:	220 degrees C

Mass Spectrophotometer Conditions:

Analyzer Temp:	220 degrees C
Electronic Voltages:	70 electron volts
Scan Delay:	1 min
Scan:	35-260 amu

8. Check the instrument tuning by analyzing p-Bromofluorobenzene. Using Supelco Standard #4-8800 (25mg/ml), prepare a 25mg working standard of p-BFB. Using a 10 microliter syringe, accurately deliver 10ul of p-BFB standard (25mg/ml) and then dilute to a volume of 10ml using high purity methanol and mix thoroughly. This is equal to a 25mg/ul working stock. Inject 2ul of this solution directly onto to column.

9. Bromofluorobenzene must meet the EPA tune conditions listed below:
BFB ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	<1% of base peak
174	>50% of mass 95
175	5-9% of mass 174
176	>95% but <101% of mass 174
177	5-9% of mass 176

The voltages applied to the entrance lens, ion focus, x-ray repeller and source electron multiplier may be adjusted to meet BFB requirements.

10. After autotuning using PFTBA; inject 2 microliters of the p-BFB working stock and tune to meet the above criteria. Once the tune criteria has been successfully meet, go on to Step 8.
11. Initially calibrate the GC/MS with 5 point standards. The %RSD for each individual CCC should be less than 30%. The minimum RF for each individual SPCC is 0.300 (0.25 for Bromoform). Run a 50 ug/ml standard every 12 hours. Accurately measure 5ml of laboratory water into a 5ml syringe. Using a 10ul syringe, inject exactly 10 ul of working stock standard solution, Step #1, and 5 ul of "other purgeables" solution, Step #2, into the water. Using a 10 ul syringe, inject exactly 10 ul of the internal standard/surrogate solution, Steps #4, 5 and 6 into the water.

12. Inject the water containing standards and surrogates into the appropriate purge tube and initiate analysis.

13. If the continuing calibration standard is within 25% of it's original calibration range then proceed with sample analysis, Step #16. If not, rerun the standard as per Steps #11 and 12.

14. If the continuing calibration standard does not pass a second time run three calibration standard as per Step #11 using 1, 5 and 20 ul aliquots of the standard preps Steps #1. The relative standard deviation (RSD) of the response factors for the three standards should be less than 30%.

15. If the RSD of the response factors is greater than 30%, prepare a new standards and analyze as per Step #11.

16. Analysis of quality control blank. Using a 5ml syringe, accurately measure 5mls of nano-pure grade laboratory water. Using a 10ul syringe, inject exactly 10ul of the 50ug/ml internal standard/surrogate solution through the 5ml syringe needle into the QC blank sample.

17. Deliver this sample into the appropriate purge tube and initiate the analysis.

18. Once the analyst has successfully shown that the laboratory quality control blank is free of interferences or contaminants, analysis of samples may begin.

19. Analysis of samples. Using a 5 ml syringe, accurately measure 5 mls of sample. Using a 10 ul syringe, inject exactly 10 ul of internal standard/surrogate solution through the 5 ml syringe needle into the sample.

20. Deliver this sample into the appropriate purge tube and initiate analysis.

21. All sample analysis are to be performed within 12 hours of the p-BFB injection that has met all ion abundance criteria.

22. Analyze one matrix spike sample for every twenty samples exactly as the sample was analyzed, Steps #19 and #20, except deliver 10ul of 50ug/ml stock standard matrix spike solution into the 5ml of sample. Trip blank or field blanks are never allowed for use in spiked sample analysis.

23. Analyze one matrix spike duplicate sample exactly as in Steps #19 and #20.

24. Soil samples are analyzed by weighing 1-5 gr. of soil into the sparging vessels. Next, follow Steps #19 and 20. After this addition, heat sample to 60°C while purging. All other procedures are as in an aqueous sample

25. The results are calculated, report printed and data stored on magnetic tape by Aquaraus data package in the Hewlett-Packard 1000 computer. Concentration is determined using the following equation:

$$\text{Concentration of Analyte} = \frac{(As)\ (Cis)}{(Ais)\ (RF)\ (V)}$$

where: As is the area of the characteristic m/z for the compound or surrogate to be measured.
Ais is the area of the characteristic m/z for the internal standard.
Cis is the concentration of the internal standard.
RF is the response factor: RF =(Ast x Cis)/(Ais x Cst)

where: Cst is the concentration of the standard compound to be measured.
Ast is the area of the characteristic m/z for the standard compound analyzed.

STANDARD OPERATION PROCEDURE

ANALYTICAL METHODS

for

VOLATILES

EPA-CLP-SOW

Document #OLMO3.0-3.1

TABLE OF CONTENTS

(Volatiles)

ANALYTICAL METHODS FOR VOLATILES

Scope and Application

This method covers the determination of the target volatile (purgeable) organics as listed in Table 8. The actual analysis is based on a purge and trap gas chromatographic/mass spectrometer (GC/MS) method. For soil/sediment samples, the purge device is heated.

Sample Storage and Holding Times

Analysis of water and soil/sediment samples must be completed within 10 days of validated time of sample receipt (VTSR).

GC/MS ANALYTICAL PROTOCOL OF VOLATILES

Summary of Methods

1. Water Samples

   An inert gas is bubbled through a 5 ml sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

   An aliquot of the sample is diluted with reagent water when dilution is necessary. A 5 ml aliquot of the dilution is taken for purging.

2. Soil/Sediment Samples

   a. Low level - an inert gas is bubbled through a mixture of reagent water and 5 g of sample contained in a specifically designed purging chamber that is held at an elevated temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

   b. Medium level - a measured amount of soil is extracted with methanol. A portion of the methanol extract is diluted to 5 ml with reagent water. An inert gas is bubbled through this solution in a specifically designed purging chamber at ambient temperature. The purgeables are effectively transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

Interferences

1. Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in QA/QC requirements. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

2. Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling. A holding blank prepared from reagent water and carried through the holding period and the analysis protocol serves as a check on such contamination. **One holding blank per case must be analyzed. Data must be retained by the laboratory and be made available for inspection during on-site laboratory evaluations.**

3. Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it must be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution between analyses, rinse it with distilled water, and then dry it in a 105°C oven. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. The laboratory where volatile analysis is performed should be completely free of solvents.

Apparatus and Materials

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance meeting the requirements of this SOW is the responsibility of the laboratory.

1. Micro syringes - 25 ul and larger, 0.006 inch ID needle.

2. Syringe valve - two-way, with Luer ends (three each), if applicable to the purging device.

3. Syringe - 5ml, gas-tight with shut-off valve.

4. Balance - analytical, capable of accurately weighing ± 0.0001 g, and a top-loading balance capable of weighing ± 0.1 g.

5. Glassware

   a. Bottle - 15 ml, screw cap, with Teflon cap liner.

   b. Volumetric flasks - class A with ground-glass stoppers.

   c. Vials - 2 ml for GC autosampler.

6. Purge and trap device - consists of three separate pieces of equipment: the sample purger, trap and desorber. Several complete devices are now commercially available.

   a. The sample purger must be designed to accept 5 ml samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 ml. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.

   b. The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following minimum lengths of adsorbents: 15 cm of 2,6-diphenylene oxide polymer (Tenax-GC, 60/80 mesh) and 8 cm of silica gel (Davison Chemical, 35/60 mesh, grade 15, or equivalent). The description of this trap is presented in SDG narrative.

   c. The desorber should be capable of rapidly heating the trap to 180°C and the remaining sections should not exceed 220°C during bakeout mode.

   d. The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph.

   e. A heater or heated bath capable of maintaining the purge device at 40°C ± 1°C is to be used for low level soil analysis, but <u>not</u> for waters or medium level soil analyses.

7. GC/MS System

   a. Gas Chromatograph - the gas chromatograph (GC) system must be capable of temperature programming and have a flow controller that maintains a constant column flow rate throughout desorption and temperature program operations. The system must include or be interfaced to a purge and trap system as specified in paragraph 6d and have all required accessories including syringes, analytical columns, and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used.

   b. Gas Chromatography Columns

      - 105 m long x 0.53 mm ID VOCOL (Supelco, Inc., or equivalent) fused silica wide-bore capillary column with 3 um film thickness. The decription of the column is presented in the SDG narrative.

   c. Mass Spectrometer - must be capable of scanning from 35 to 300 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the instrument performance acceptance criteria when 50 ng of p-bromofluorobenzene (BFB) is injected through the gas chromatograph inlet. The instrument conditions required for the acquisition of the BFB mass spectrum are given in Instrument Operating Conditions 4c.

   d. Data system - a computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion

compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent lease of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

e. Magnetic tape storage device - must be capable of recording data and must be suitable for long-term, off-line storage.

f. pH paper - wide range.

Reagents

1. Reagent water - defined as water in which an interferant is not observed at or above the CRQL of the parameters of interest.

   a. Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

   b. A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

   c. Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a narrow-mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

2. Sodium thiosulfate - (ASC) granular.

3. Methanol - pesticide quality or equivalent.

Standards

1. The laboratory must provide all standards to be used with this contract. These standards may be used only after they have been certified according to the procedure in QA/QC requirements. The laboratory must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the laboratory and present upon request. All stock standard solutions are purchased.

2. Stock Standard Solutions

Stock standard solutions may be purchased or may be prepared in methanol from pure standard materials.

a. Prepare stock standard solutions by placing about 9.8 ml of methanol into a 10.0 ml ground-glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes, or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

b. Add the assayed reference material as described below.

(1) If the compound is a liquid, using a 100 ul syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

(2) If the compound is a gas at room temperature, fill a 5 ml valved gas-tight syringe with the reference standard to the 5.0 ml mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The gas will rapidly dissolve in the methanol. This may also be accomplished by using a lecture bottle equipped with a Hamilton Lecture Bottle Septum (#86600). Attach Teflon tubing to the side-arm relief valve and direct a gentle stream of the reference standard into a methanol meniscus.

c. Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. For non-gaseous and compounds, calculate the concentration in micrograms per microliter from the net gain in weight. When compound purity is assayed to be 97 percent or greater, the weight may be used, without correction, to calculate the concentration of the stock standard. If the compound purity is assayed to be less than 97 percent, the weight must be corrected when calculating the concentration of the stock solution. For gaseous compounds, calculate the concentration in micrograms per microliter, using the Ideal Gas Law, taking into account the temperature and pressure conditions within the laboratory.

d. Prepare fresh stock standards every two months for gases or for reactive compounds such as styrene. All other stock standards for non-gases/non-reactive purgeable compounds must be replaced after six months, or sooner if standard has degraded or evaporated.

3. Secondary Dilution Standards

a. Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. Secondary dilution standard solutions should be prepared at concentrations that can be easily diluted to prepare working standard solutions.

b. Prepare fresh secondary dilution standards for gases and for reactive compounds such as styrene every month, or sooner, if standard has degraded or evaporated. Secondary dilution standards for the other purgeable compounds must be replace after six months, or sooner if standard has degraded or evaporated.

4. Working Standards

a. Instrument Performance Check Solution - p-Bromofluorobenzene (BFB)

Prepare a 25 ng/ul solution of BFB in methanol. Prepare fresh BFB solution every six months, or sooner, if the solution has degraded or evaporated.

b. Calibration Standard Solution

Prepare the working calibration standard solution containing all of the purgeable target compounds in methanol. The recommended concentration of the target compounds is 100 ug/ml. Prepare fresh working calibration standard solutions weekly, or sooner, if solutions have degraded or evaporated.

c. Internal Standard Spiking Solution

Prepare an internal standard spiking solution containing Bromochloromethane, Chlorobenzene-d_5, and 1,4-Difluorobenzene in methanol at the concentration of 25.0 ug/ml for each internal standard. Add 10 ul of this spiking solution into 5.0 ml of sample or calibration standard for a concentration of 50 ug/l. Prepare fresh spiking solution weekly, or sooner if the solution has degraded or evaporated.

d. System Monitoring Compound (SMC) Spiking Solution

Prepare a system monitoring compound spiking solution containing Toluene-d_8, p-Bromofluorobenzene, and 1-2-Dichloroethane-d_4 in methanol at a concentration of 25.0 ug/ml. Add 10.0 ul of this spiking solution into 5.0 ml of sample, for a concentration of 50 ug/l. Prepare fresh spiking solution weekly, or sooner if the solution has degraded or evaporated.

e. Volatile Matrix Standard Spiking Solution

(1) Prepare a spiking solution in methanol that contains the following compounds at a concentration of 25.0 ug/ml: 1,1-Dichloroethane, Trichloroethene, Chlorobenzene, Toluene, and Benzene. Prepare fresh spiking solution weekly, or sooner if the solution has degraded or evaporated.

(2) Matrix spikes are analyzed in duplicate; therefore, add an aliquot of this solution to each of two portions from one sample chosen for spiking.

5. Aqueous Calibration Standard Solutions

a. Prepare five aqueous initial calibration standard solutions containing all of the purgeable target compounds and system monitoring compounds at the 10, 20, 50, 100, and 200 ug/l levels. Note: These are not the same levels as have been used in previous Statements of Work. It is required that all three Xylene isomers (o-, p-, and m-Xylene) be present in the calibration standards at concentrations of each isomer equal to that of the other target compounds (i.e., 10, 20, 50, 100, and 200 ug/l). Similarly, the cis and trans isomers of 1,2-dichloroethene must be present in the standards at concentrations of each isomer equal to that of the other target compounds.

b. Aqueous calibration standards may be prepared in a volumetric flask or in the syringe used to inject the standard into the purging device.

(1) Volumetric flask - add an appropriate volume of working calibration standard solution to an aliquot of reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcohol standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Bring to volume. Mix by inverting the flask three times only. Discard the contents contained in the head of the flask.

(2) Syringe - remove the plunger from a 5 ml "Luerlock" syringe. Pour reagent water into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the water. Invert the syringe, open the syringe valve and vent any residual air. Adjust the water volume to 5.0 ml minus the amount of calibration standard to be added. Withdraw the plunger slightly and add an appropriate volume of working calibration standard through the valve bore of the syringe. Close the valve and invert three times.

c. The 50 ug/l aqueous calibration standard solution is the continuing calibration standard.

d. The methanol purged in each of the aqueous calibration standards must not exceed 1% by volume.

6. Storage of standards

a. Stock the stock standards in Teflon-sealed screw-cap bottles with zero headspace at -10°C to -20°C. Protect the standards from light. Once one of the bottles containing the stock standard solution has been opened, it may be used for no longer than one week.

b. Store secondary dilution standards in Teflon-sealed screw-cap bottles with minimal headspace at -10°C to 20°C. Protect the standards from light. The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to preparing the working calibration standards from them.

c. Aqueous standards may be stored for up to 24 hours if held in Teflon-sealed screw cap vials with zero headspace at 4°C. Protect the standards from light. If not so stored, they must be discarded after one hour unless they are set up to be purged by an autosampler. When using an autosampler, the standards may be kept for up to 12 hours in purge tubes connected via the autosampler to the purge and trap device.

d. Purgeable standards must be stored separately from other standards.

Instrument Operating Conditions

1. Purge and Trap Device

The following are the recommended purge and trap analytical condition except as stated below:

Purge Conditions:

Purge Gas:	Helium or Nitrogen
Purge Time:	11.0 ± 0.1 min
Purge Flow Rate:	25-40 ml/min
Purge Temperature:	Ambient (water or medium level soil), required 40°C (low level soil), required.

Desorb Conditions:

Desorb Temperature:	180°C
Desorb Flow Rate:	15 ml/min
Desorb Time:	4.0 ± 0.1 min

Trap Reconditioning Conditions:

Reconditioning Temperature:	180°C
Reconditioning Time:	7.0 min ± 0.1 min

Before initial use, condition the trap overnight at 180°C by backflushing with at least 20 ml/min flow of inert gas. Vent the trap effluent to the room and not to the analytical column. Prior to daily use, condition the trap by heating at 180°C for 10 minutes while backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

2. Gas Chromatograph

The following are the recommended GC analytical conditions:

a. Packed Columns

Carrier Gas:	Helium
Flow Rate:	30 ml/min
Initial Temperature:	45°C
Initial Hold Time:	3 min
Ramp Rate:	8°C/min
Final Temperature:	220°C
Final Hold Time:	15 min
Transfer Line Temperature:	250-300C

b. Capillary Columns

Carrier Gas:	Helium
Flow Rate:	15 ml/min
Initial Temperature:	10°C
Initial Hold Time:	1.0 - 5.0 min (±0.1 min)
Ramp Rate:	6°C/min
Final Temperature:	160°C
Final Hold Time:	Until all target compounds elute

c. Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, matrix spikes, and matrix spike duplicates.

3. Mass Spectrometer

The following are the required mass spectrometer conditions:

Electron Energy:	70 Volts (nominal)
Mass Range:	35-300 amu
Scan Time:	To give at least 5 scans per peak, not to exceed 2 seconds per scan for capillary column.
	To give at least five (5) scans per peak, not to exceed 3 seconds per scan for packed column.

4. The GC/MS system must be tuned to meet the manufacturer's specifications, using a suitable calibrant such as FC-43 or perfluorokerosene (PFK). The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution (Working Standards 4a).

a. Prior to the analyses of any samples, blanks, or calibration standards, the laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing p-bromofluorobenzene (BFB).

b. The analysis of the instrument performance check solution may be performed as follows:

- As an injection of up to 50 ng of BFB into the GC/MS.
- By adding 50 ng of BFB to 5.0 ml of reagent water and analyzing the resulting solution as if it were an environmental sample (see Sample Analysis).

BFB may not be analyzed simultaneously with a calibration standard.

c. The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background substraction is required, and must be accomplished using a single scan prior to the elution of BFB.

d. The analysis of the instrument performance check solution must meet the ion abundance criteria given below.

TABLE 1
BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	8.0 - 40.0 percent of mass 95
75	30.0 - 66.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent relative abundance
173	less than 2.0 percent of mass 174
174	50.0 - 120.0 percent of mass 95
175	4.0 - 9.0 percent of mass 174
176	93.0 - 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

e. The criteria listed above are based on adherence to the acquisition specifications identified in paragraph 4c and were developed for the specific target compound list associated with this Statement of Work. The criteria are based on performance characteristics of instruments currently utilized in routine support of Program activities. These specifications, in conjunction with relative response factor criteria for 23 target compounds (see Table 2), are designed to control and monitor instrument performance associated with the requirements of this Statement of Work.

f. The instrument performance check solution must be injected once at the beginning of each 12-hour period, during which samples or standards are to be analyzed. The twelve (12) hour time period for GC/MS Instrument Performance Check (BFB), standards calibration (initial or continuing calibration criteria) and method blank analysis begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after twelve (12) hours has elapsed according to the system clock.

Calibration

1. Prior to the analysis of samples and required blanks, and after the instrument performance check solution criteria have been met, each GC/MS system must be calibrated at a minimum of five concentrations to determine instrument sensitivity and the linearity of GS/MS response for the purgeable target compounds.

2. Assemble a purge and trap device that meets the specification in Item 6 of Apparatus and Materials. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 ml/min. Daily, prior to use, condition the traps for 10 minutes while backflushing at 180°C with the column at 220°C.

3. Connect the purge and trap device to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in Item 1 of Instrument Operating Conditions. Calibrate the purge and trap-GC/MS system using the internal standard technique.

4. Internal standard calibration procedure. The three internal standards are Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d_5, at 50 ug/l at time of purge. Separate initial and continuing calibrations must be performed for water samples, and low level soil samples (unheated purge vs. heated purge). Extracts of medium level soil samples may be analyzed using the calibrations for water samples.

a. Prepare calibration standards at a minimum of five concentration levels for each target compound and system monitoring compound, as specified in Item 5 of Standards. Standards may be stored up to 24 hours, following the procedures in Item 6 of Standards.

b. Prepare a spiking solution containing each of the internal standards using the procedures described in paragraph 4c of Standards.

c. Verify that the GC/MS system meets the instrument performance criteria in paragraph 4d of Instrument Operating Conditions by injecting BFB. Analyze each calibration standard, according to Item 1 of Calibration, adding 10 ul of internal standard spiking solution directly to the syringe.

Tabulate the area response of the characteristic ions in the extracted ion current profile (EICP) against concentration for each compound and internal standard and calculate relative response factors (RRF) for each compound as follows:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (See Table 4)

A_{is} = Area of the characteristic ion (CICP) for the specific internal standard (See Table 3)

C_{is} = Concentration of the internal standard

C_x = Concentration of the compound to be measured

d. The average relative response factor (RRF) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of RRF values over the working range of the curve.

$$\%RSD = \frac{\text{Standard deviation}}{\text{mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1} \right|^{1/2}$$

Where,

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

e. The response factors of the compounds listed below (Table 2) must meet the minimum RRF criteria at each concentration level and maximum %RSD criteria for the initial calibration, with allowance made for up to two volatile compounds. However, the RRFs for those two compounds must be greater than or equal to 0.010, and the %RSD of those two compounds must be less than or equal to 40.0% for the initial calibration to be acceptable.

TABLE 2
RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF VOLATILE ORGANIC COMPOUNDS

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Bromomethane	0.100	20.5	25.0
Vinyl chloride	0.100	20.5	25.0
1,1-Dichloroethene	0.100	20.5	25.0
1,1-Dichloroethane	0.200	20.5	25.0
Chloroform	0.200	20.5	25.0
1,2-Dichlorethane	0.100	20.5	25.0
1,1,1-Trichloroethane	0.100	20.5	25.0
Carbon tetrachloride	0.100	20.5	25.0
Bromodichloromethane	0.200	20.5	25.0
cis-1,3-Dichloropropene	0.200	20.5	25.0
Trichloroethene	0.300	20.5	25.0
Dibromochloromethane	0.100	20.5	25.0
1,1,2-Trichloroethane	0.100	20.5	25.0
Benzene	0.500	20.5	25.0
trans-1,3-Dichloropropene	0.100	20.5	25.0
Bromoform	0.100	20.5	25.0
Tetrachloroethene	0.200	20.5	25.0
1,1,2,2-Tetrachloroethane	0.500	20.5	25.0
Toluene	0.400	20.5	25.0
Chlorobenzene	0.500	20.5	25.0
Ethylbenzene	0.100	20.5	25.0
Styrene	0.300	20.5	25.0
Xylenes (total)	0.300	20.5	25.0
Bromofluorobenzene	0.200	20.5	25.0

f. The following compounds have no Maximum %RSD, or Maximum %Difference criteria; however, these compounds must meet a minimum RRF criterion of 0.010:

Acetone	1,2-Dichloropropane
2-Butanone	2-Hexanone
Carbon disulfide	Methylene chloride
Chloroethane	4-Methyl-2-pentanone
Chloromethane	Toluene-d_8
1,2-Dichloroenthene (total)	1,2-Dichloroethane d_4

g. A check of the calibration curve must be performed once every 12 hours (see Item 4f of Instrument Operating Conditions) for the definition of the twelve-hour time period). Check the relative response factors of those compounds for which RRF values have been established. If these criteria are met, the relative response factors for all compounds are calculated and reported. A percent difference of the daily relative response factor (12-hour) compared to the average relative response factor from the initial curve is calculated. Calculate the percent difference criteria listed above. For negative percent difference values, the value must be greater than or equal to -25.0%, but less than 0%. As with the initial calibration, up to two volatile compounds in Table 2 may fail to meet the minimum RRF or maximum %D criteria, but the RRFs of those two compounds must be greater than or equal to 0.010, and the percent differences must be less than or equal to 40.0% for the continuing calibration to be acceptable.

h. Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standards changes by more than 0.50 minutes (30 seconds) from the latest daily (12-hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each standard. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction, and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

5. Each GC/MS system must be calibrated upon award of the contract, whenever the laboratory takes corrective action which may change or affect the initial calibration criteria (i.e., ion source cleaning or repair, column removal or replacement, etc.), or if the continuing calibration acceptance criteria have not been met.

6. If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. It is not necessary to analyze a continuing calibration standard, if the initial calibration meets the calibration acceptance criteria above. A method blank is necessary. Quantify all sample results against the initial calibration standard that is the same concentration as the continuing calibration standard (50 ug/l).

7. If time does not remain in the 12-hour period beginning with the injection of the instrument performance check solution, a new injection of the instrument performance check solution must be made. If the new injection meets the ion abundance criteria for BFB, then a continuing calibration standard may be injected.

8. The concentrations of volatile target compounds in the continuing calibration standard are given in paragraph 5c of Standards.

9. The response factors for the continuing calibration standard must meet the criteria given in paragraph 4e of Calibration prior to the analysis of any blanks or samples.

Sample Analysis

1. Water Samples

   a. All water samples must be allowed to warm to ambient temperature before analysis.

   b. Prior to the analysis of samples, establish the appropriate GC/MS operating conditions, as outlined in paragraphs of Instrument Operation Conditions, analyze the instrument performance check solution (Item 4 of Instrument Operating Conditions), and calibrate the GC/MS system according to paragraphs of Calibration.

   c. If time remains in the 12-hour period (as described in paragraph 6 of Calibration), samples may be analyzed without analysis of a continuing calibration standard.

   d. If time does not remain in the 12-hour period since the injection of the instrument performance check solution, both the instrument performance check solution and the continuing calibration standard must be analyzed before sample analysis may begin (see paragraphs 7-9 of Calibration).

   e. Adjust the purge gas (helium) flow rate to 25-40 ml/min. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly Chloromethane and Bromoform.

   f. Remove the plunger from a 5 ml syringe and attach a closed syringe valve. Open the sample or standard bottle which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 ml. This process of taking an aliquot destroys the validity of the sample for future analysis so, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time as the analyst has determined that the first sample has been analyzed properly. Filling one 5 ml syringe would allow the use of only one syringe. If an analysis is needed from the second 5 ml syringe, it must be performed within 24 hours. Care must also be taken to prevent air from leaking into syringe.

   g. Once the sample aliquots have been taken from the VOA vial, the pH of the water sample must be determined. The purpose of the pH determination is to ensure that all VOA samples were acidified in the field. Test the pH by placing one or two drops of sample on the pH paper (do not add pH paper to the vial). Record the pH of each sample, and report these data in the laboratory chronicle. No pH adjustment is to be performed by the laboratory.

   h. Add 10.0 ul of the system monitoring compound spiking solution (paragraph 4d of Standards) and 10.0 ul of the internal standard spiking solution (paragraph 4c of Standards) through the valve bore of the syringe, then close the valve. The system monitoring compounds and internal standards may be mixed and added as a single spiking solution. The addition of 10 ul of the system monitoring compound spiking solution to 5 ml of sample is equivalent to a concentration of 50 ug/l of each system monitoring compound.

   i. Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

j. Close both valves and purge the sample for 11.0 ± 0.1 minutes at ambient temperature.

k. At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 180°C while backflushing the trap with an inert gas between 20 and 60 ml/min for four minutes. If this rapid heating requirement cannot be met, the gas chromatographic column must be used as a secondary trap by cooling it to 30°C (or subambient, if problems persist) instead of the recommended initial temperature of 45°C.

l. While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5 ml flushes of reagent water to avoid carryover of target compounds.

m. After desorbing the sample for four minutes, recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180°C. Trap temperatures up to 220°C may be employed, however the higher temperature will shorten the useful life of the trap. After approximately seven minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

n. Each analytical run must be checked also for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound. The initial calibration requires that the system should not be saturated for high response compounds at 200 ug/l for VOA target compounds. In addition, the system must not be saturated by the two Xylene isomers that co-elute on the GC column used for analysis when the co-eluting peak represents 400 ug/l, or for the two 1,2-Dichloroethene isomers that may co-elute when the co-eluting peak represents 400 ug/l. Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the non-conformance. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by the analysis of a reagent water blank. If the blank is not free of interferences, the system must be contaminated. Sample analysis may not resume until a blank has been analyzed that demonstrates that the system is free of interferences. Once the system is free of interferences, the sample that saturated the detection must be diluted and reanalyzed.

o. To prepare a matrix spike and matrix spike duplicate for water samples, add 10 ul of the matrix spike solution (paragraph 4e of Standards) to each of the 5 ml aliquots of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 ug/l of each matrix spike compound. The frequency of MS/MSD analysis is given in paragraph 7 of Quantitative Analysis.

p. A volatile method blank must be analyzed at least once during every twelve-hour period, on each GC/MS system used for volatile analysis (see paragraph 4f of Instrument Operating Conditions for the definition of the twelve-hour time period).

(1) For water samples, a volatile method blank consists of a 5 ml volume of reagent water (paragraph 1 of Reagents) spiked with the system monitoring compounds and internal standards, and carried through the analytical procedure.

(2) An acceptable volatile method blank for water samples must contain less than or equal to five times (5x) the Contract Required Quantitation Limit (CRQL, see Table 8) of Methylene chloride, Acetone, and 2-Butanone, and less than or equal to the CRQL of any other volatile target compound.

(3) All volatile analyses associated with a blank that does not meet the requirements above, (i.e., a contaminated blank) must be repurged, reanalyzed, and reported at no additional cost to the Agency.

(4) The volatile method blank must be analyzed after the calibration standards, to ensure that there is not carryover of material from the standards into samples.

q. The laboratory must demonstrate that there is no carryover from a contaminated sample before data from subsequent analyses may be submitted. After a sample that contains a target compound at a level exceeding the initial calibration range, the laboratory must either:

(1) Analyze a method blank immediately after the contaminated sample. If an autosampler is used, a method blank must also be analyzed using the same purge inlet that was used for the contaminated sample. The method blanks must meet the technical acceptance criteria for blank analysis (see Item P above), or

(2) Monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample and that exceeded the limits above. The maximum contamination criteria are as follows: the sample must not contain a concentration above the CRQL for the target compounds that exceeded the limits in the contaminated sample. If an autosampler is used, the next sample analyzed using the same purge inlet that was used for the contaminated sample also must meet the maximum contamination criteria.

2. Soil/Sediment Samples

Two approaches may be taken to determine whether the low-level or medium-level method must be followed.

If peaks are saturated from the analysis of a 5 g sample, a smaller sample size must be analyzed to prevent saturation. However, the smallest sample size permitted is 1 g. If smaller than 1 g sample size is needed to prevent saturation, the medium level method must be used.

a. Low-Level Soil Method

The low level soil method is based on a heated purge of a soil/sediment sample mixed with reagent water containing the system monitoring compounds and the internal standards. Analyze all method blanks and standards under the same conditions as the samples.

(1) The GC/MS system should be set up as in paragraphs under Calibration. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and sample. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions (under Calibration), but increase the purge temperature to 40°C.

(2) To prepare the reagent water containing the system monitoring compounds and the internal standards, remove the plunger from a 5 ml "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 ml. Add 10 ul of the system monitoring compound spiking solution and 10 ul of the internal standard solution to the syringe through the valve.

(3) The sample (for volatile organics) consists of the <u>entire</u> contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in paragraph 2a of Sample Analysis into a tared purge device. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g.

(4) Add the spiked reagent water to the purge device and connect the device to the purge and trap system.
Note: Prior to the attachment of the purge device, the steps in paragraphs (2) and (4) above must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

(5) Heat the sample to 40°C ± 1°C and purge the sample for 11.0 ± 0.1 minutes.

(6) Proceed with the analysis as outlined in paragraphs 1j - 1m of Water Sample Analysis. Requirements for dilution of samples are given in paragraphs 2 of Soil/Sediment Samples.

(7) To prepare a matrix spike and matrix spike duplicate for low-level soils/sediment, add 10 ul of the matrix spike solution (4e of Standards) to the 5 ml of water added to each of the two aliquots of the soil from the sample chosen for spiking (paragraph 2a(2) of Soil/Sediment Samples). The concentration for a 5 g sample would be equivalent to 50 ug/kg of each matrix spike compound. The frequency of MS/MSD analysis is given in paragraph 7 of Quantitative Analysis.

(8) A volatile method blank must be analyzed at least once during every twelve-hour time period, on each GC/MS system used for volatile analysis (see paragraph 4f of Instrument Operating Conditions for the definition of the twelve-hour time period.

(8a) For low level soil/sediment samples, a volatile method blank consists of a 5 ml of reagent water, spiked with the system monitoring compounds and internal standards, and carried through the analytical procedure.

(8b) An acceptable volatile method blank for low-level soil samples must contain less than or equal to five times (5x) the Contract Required Quantitation Limit (CRQL, see Table 8) of Methylene chloride, Acetone, and 2-Butanone, and less than or equal to the CRQL of any other volatile target compound.

(8c) <u>All</u> volatile analyses associated with a blank that does <u>not</u> meet the requirements above, (i.e., a contaminated blank) <u>must</u> be repurged, reanalyzed, and reported.

(8d) The volatile method blank <u>must</u> be analyzed <u>after</u> the calibration standards, to ensure that there is not carryover of material from the standards into samples.

2. Medium-Level Soil Method

The medium level soil method is based on extracting the soil/sediment sample with methanol. An aliquot of the methanol extract is added to reagent water containing the system monitoring compounds and the internal standards. The reagent water containing the methanol extract is purged at ambient temperature. If saturated peaks occurred, or would occur, when a 1 g sample was analyzed, the medium level method must be used.

a. The GC/MS system should be set up as in paragraphs under Instrument Operation Conditions. This should be done prior to the addition of the methanol extract to reagent water. Because the methanol extract and reagent water mixture is purged at ambient temperature, the instrument performance check, initial calibration, and continuing calibration for water samples may be used for analyses of medium soil sample extracts.

b. The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 4 g (wet weight) into a tared 15 ml vial. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g. Determine the percent moisture.

c. Quickly add 9.0 ml of methanol to the vial. Then add 1.0 ml of the system monitoring compound spiking solution to the vial. Cap and shake for 2 minutes.
Note: The steps in paragraphs b and c of this section must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

d. Using a disposable pipette, transfer approximately 1 ml of extract into a GC vial for storage. The remainder may be discarded. Transfer approximately 1 ml of the reagent methanol to a GC vial for use as the method blank for each Case or day on which medium soil sample extractions are performed, whichever is most frequent. These extracts may be stored in the dark at 4°C (±2°C) prior to analysis.

e. The following table can be used to determine the volume of methanol extract to add to the 5 ml of reagent water for analysis. If the sample was submitted as a medium level sample, start with 100 ul.

All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of linear range of the curve.

X Factor	Estimated Concentration Range	Take this Volume of Methanol Extract
	ug/kg	ul
0.25 - 5.0	500 - 10,000	100
0.5 - 10.0	1,000 - 20,000	50
2.5 - 50.0	5,000 - 100,000	10
12.5 - 250	25,000 - 500,000	100 of 1/50 dilution

f. Remove the plunger from a 5 ml "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 ml. Pull the plunger back to 5 ml to allow volume for the addition of sample and standards. Add 10 ul of the internal standard solution. Also add the volume of methanol extract determined in paragraph e. above and a volume of clean methanol to total 100 ul (excluding methanol in standards).

g. Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

h. Proceed with the analysis as outlined in paragraphs of Sample Analysis. Analyze all method blanks on the same instrument as the samples. Requirements for dilution of samples are given in paragraph 2 of Soil/Sediment Samples.

i. To prepare a matrix spike and matrix spike duplicate for the medium level soil/sediment samples, add 8.0 ml of methanol, 1.0 ml of matrix spike solution (paragraph 4e of Standards) as in paragraph 2c of Medium Level Soil Method, to each of the two aliquots of the soil sample chosen for spiking. This results in a 6,200 ug/kg concentration of each matrix spike compound when added to a 4 g sample. Add a 100 ul aliquot of this extract to 5 ml of water for purging (as per paragraph f above). The frequency of MS/MSD analysis is given in paragraph 7 of Quantitative Analysis.

j. A volatile method blank must be analyzed at least once during every twelve-hour time period, on each GC/MS system used for volatile analysis (see paragraph 4f of Instrument Operation Conditions for the definition of the twelve-hour time period).

(1) For medium level-soil/sediment samples, a volatile method blank consists of a 4 g of a purified solid matrix spiked with the system monitoring compounds, extracted with methanol, and carried through the analytical procedure. If a purified solid matrix is not available, the reagent water will be used as method blank matrix.

(2) An acceptable volatile method blank for medium-level soil/sediment samples must contain less than or equal five times (5x) the Contract Required Quantitation Limit (CRQL, See Table 8) of Methylene chloride, Acetone, and 2-Butanone, and less than or equal to the CRQL of any other volatile target compound.

(3) <u>All</u> volatile analyses associated with a blank that does <u>not</u> meet the requirements above, (i.e. a contaminated blank) <u>must</u> be repurged, reanalyzed, and reported at not additional cost to the Agency.

(4) The volatile method blank <u>must</u> be analyzed <u>after</u> the calibration standards, to ensure that there is no carryover of material from the standards into samples.

k. The laboratory must demonstrate that there is no carryover from a contaminated sample before data from subsequent analyses may be submitted.

Qualitative Analysis

1. The compounds listed in the Target Compound List (TCL), Table 8, shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample mass spectrum to a standard of the suspected compound. Two criteria must be satisfied to verify the identification: (1) elution of the sample component at the same GC relative retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

   a. For establishing correspondence of the GC relative retention time (RRT), the sample component RRT of the standard component. For reference, the standard must be run in the same 12-hour time period as the sample. If samples are analyzed during the same 12-hour time period as the initial calibration standards, use the RRT values from the 50 ug/l standard. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

   b. For comparison of standard and sample component mass spectra, mass spectra obtained on the laboratory's GC/MS are required. Once obtained, these standard spectra may be used for identification purposes, only if the laboratory's GC/MS meets the daily instrument performance requirements for BFB. These standard spectra may be obtained from the run used to obtain reference RRTs.

   c. The requirements for qualitative verification by comparison of mass spectra are as follows:

      (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

      (2) The relative intensities of ions specified in paragraph (1) above must agree within ± 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent).

      (3) Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. In Task III, the verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the CRQL report the actual value followed by a "J", e.g., "3J."

   d. If a compound cannot be verified by all of the criteria in paragraph 1c above, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that identification and proceed with quantification in paragraphs of Quantitative Analysis.

2. A library search shall be executed for non-target sample components for the purpose of tentative identification. For this purpose, the most recent release of the NIST/EPA/MSDC mass spectral library, shall be used. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

   a. Up to 10 organic compounds of greatest apparent concentration not listed in Exhibit C for the purgeable organic fraction, excluding the system monitoring compounds, shall be tentatively identified via a forward search of the NIST/EPA/MSDC Library (substances with responses less than 10% of the internal standard are not required to be search in this fashion). Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

   b. Guidelines for making tentative identification:

      (1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

      (2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50 percent of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent).

      (3) Molecular ions present in reference spectrum should be present in sample spectrum.

      (4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

      (5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds. Data system library reduction programs can sometimes create these discrepancies.

   c. If, in the technical judgement of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

Quantitative Analysis

1. Target components identified shall be quantified by the internal standard method. The internal standard used shall be that which is assigned in Table 5 of this Section. The EICP area of the characteristic ions of analytes listed in Tables 3 and 4 in this Section are used.

   In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator must identify such edit or manual procedures by initializing and dating the changes made to the report.

2. Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12-hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. For samples analyzed during the same 12-hour time period as the initial calibration standards, compare the internal standard responses and retention times against the 50 ug/l calibration standard. The extracted iron current profile (EICP) of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is necessary.

   a. If after re-analysis, the EICP areas for all internal standards are inside the contract limits (-50% to +100%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, submit only data from the analysis with EICPs within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

   b. If after re-analysis of the sample does not solve the problem, i.e., the EICP areas are outside the contract limits for both analyses, then submit the EICP data and sample data from both analyses. Distinguish between the initial analysis and the re-analysis on all data deliverables.

3. The relative response factor (RRF) from the continuing calibration standard is used to calculate the concentration in the sample. Use the relative response factor as determined in paragraph 4c of Calibration and the equations below. When target compounds are below contract required quantitation limits (CRQL), but the spectra meet the identification criteria, report the concentration with a "J." For example, if CRQL is 10 ug/l and concentration of 3 ug/l is calculated, report as "3J."

   Water

   $$\text{Concentration ug/l} = \frac{(A_x)\,(I_s)\,(Df)}{(A_{is})\,(RRF)\,V_o}$$

   Where,

   A_x = Area of the characteristic ion (EICP) for the compound to be measured (see Table 4)

   A_{is} = Area of the characteristic ion (EICP) for the specific internal standard (see Table 3)

   I_s = Amount of internal standard added in nanograms (ng)

   RRF = Relative response factor from the ambient temperature purge of the calibration standard.

   V_o = Volume of water purged in milliliters (ml)

   Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (ml) of water purged (i.e., V_o above) to the number of ml of the original water sample used for purging. For example, if 2.5 ml of sample is diluted to 5.0 ml with reagent water and purged, Df = 5.0 ml/2.5 ml = 2.0. If no dilution is performed, Df = 1.0.

Low Soil

Concentration (Dry weight basis) ug/Kg = $\frac{(A_x)(I_s)}{(A_{is})(RRF)(W_s)(D)}$

Where,

A_x, I_s, A_{is} are as given for water.

RRF = Relative response factor from the heated purge of the calibration standard.

D = $\frac{100 - \%\ moisture}{100}$

W_s = Weight of sample added to the purge tube, in grams (g).

Medium Soil

Concentration (Dry weight basis) ug/Kg = $\frac{(A_x)(I_s)(V_t)(1000)(Df)}{(A_{is})(RRF)\ (V_a)(W_s)(D)}$

Where,

A_x, A_{is}, I_s are as given for water above.

RRF = Relative response factor from the ambient temperature purge of the calibration standard.

V_t = Total volume of the methanol extract in milliliters (ml). **Note:** This volume is typically 10.0 ml, even though only 1.0 ml is transferred to the vial in paragraph d of Medium Level Soil Method.

V_a = Volume of the aliquot of the methanol extract in microliters (ul) added to reagent water from purging.

W_s = Weight of soil extracted, in grams (g).

D = $\frac{100 - \%\ moisture}{100}$

Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for volatiles by the medium-level method is defined as the ratio of the number of microliters (ul) of methanol added to the reagent water for purging i.e., V_a above, to the number of microliters of the methanol extract of the sample contained in that volume V_a. The dilution factor is equal to 1.0 in all cases other than those requiring dilution of the methanol extract. Dilution of the extract is required when the "X" factor (paragraph e of Medium Level Soil Method) is $\geq$ 12.5.

The factor of 1,000 in the numerator converts the value of V_t from ml to ul.

4. An estimated concentration for non-target components tentatively identified shall be determined by the internal standard method. For quantification, the nearest internal standard free of interferences shall be used.

   The formula for calculating concentrations is the same as in paragraph 3 above. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A relative response factor (RRF) of one (1) is to be assumed. The resulting concentration shall be qualified as "J" (estimated, due to lack of a compound-specific response factor), and "N" (presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target component. An estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

5. Do not submit data for more than two analyses.

6. Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis.

7. Calculate the recovery of each system monitoring compound in all samples, blank, matrix spikes, and matrix spike duplicates. Determine if the recovery is within limits (see Table 6), and report on appropriate form.

   a. Calculate the concentration of the system monitoring compounds using the same equations as used for target compounds.
   Calculate the recovery of each system monitoring compound as follows:

   $$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

   b. If the recovery of any one system monitoring compound is not within limits, the following are required:

      - Check to be sure that there are no errors in calculations, formulation of the system monitoring compound spiking solutions, and internal standards. Also check instrument performance.

      - Re-analyze the sample if none of the above steps reveal a problem.

      - If an undiluted analysis with acceptable monitoring compound recoveries is being submitted, do not re-analyze diluted samples if the system monitoring compound recoveries are outside the limits.

      - Never re-analyze the matrix spike or matrix spike duplicate (MS/MSD), even if the system monitoring compound recoveries are outside the limits.

      - If the sample associated with the matrix spike and matrix spike duplicate does not meet specification, it should be re-analyzed only if the MS/MSD system monitoring compound recoveries are within the limits. If the sample and associated MS/MSD show the same pattern (i.e., outside the limits), then the sample does not require re-analysis and a re-analysis must not be submitted. Document in the non-conformance the similarity in recoveries of the system monitoring compounds in the sample and associated MS/MSD.

b. If the re-analysis of the sample solves the problem, then the problem was within the laboratory's control. Therefore, submit only data from the analysis with system monitoring compound recoveries within the limits. This shall be considered the initial analysis and shall be reported as such on all data deliverables.

c. If the re-analysis of the sample does not solve the problem (i.e., the system monitoring compound recoveries are outside the limits for both analyses), then submit the data from both analyses. Distinguish between the initial analysis and the re-analysis on all data deliverables.

d. For medium-level soil analyses, involving methanol extraction, the treatment of system monitoring compound recoveries is similar to that for semi-volatile surrogate recoveries. If any system monitoring compound recovery is outside the limits, re-analyze the methanol extract first, to determine if the problem was with the analysis. If re-analysis of the extract does not solve the problem, then re-extract the medium soil sample and analyze the second extract. Follow paragraphs b and c above when determining which analyses to submit.

e. If the recovery of any one system monitoring compound in a method blank is outside the limits, then the method and all associated samples must be re-analyzed at no additional cost to the Agency.

8. A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix, for the following, whichever is most frequent:

- Each Case of field samples received, OR
- Each 20 field samples in a Case, OR
- Each group of field samples of a similar concentration level (soils only), OR
- Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which field samples in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group).

a. Calculate the concentrations of the matrix spike compounds using the same equations as used for target compounds.
Calculate the recovery of each matrix spike compound as follows:

$$\text{Matrix Spike Recover} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spiked sample result

SR = Sample result

SA = Spike added

b. Calculate the relative percent difference (RPD) of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

$$RPD = \frac{|MSR - MSDR|}{(1/2)(MSR+MSDR)} \times 100$$

Where,

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

c. The limits for matrix spike compound recovery and RPD are given in Table 7. As these are only advisory, no further action by the laboratory is required, however, frequent failures to meet the limits for recovery or RPF warrant investigation by the laboratory, and may result in questions from the Agency.

9. Determine the concentrations of any target compounds detected in the volatile method blank, using the equations in paragraph 3 of Quantitative Analysis. The method blank must contain less than or equal to the Contract Required Quantitation Limit (CRQL) of the volatile target compounds in Table 8, except Methylene chloride, Acetone, and 2-Butanone, which must be less than or equal to five times (5x) the CRQL. For soil/sediment method blanks, CRQL value must be adjusted for percent moisture.

If a laboratory method blank exceeds these criteria, the laboratory must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measure MUST be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) MUST be re-extracted/repurged and re-analyzed at no additional cost to the Agency. The Laboratory Manager, or his designee, must address problems and solutions in the non-conformance.

TABLE 3
CHARACTERISTIC IONS FOR
SYSTEM MONITORING COMPOUNDS AND
INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
SYSTEM MONITORING COMPOUNDS		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane-d_4	65	102
Toluene-d_8	98	70, 100
INTERNAL STANDARDS		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d_5	117	82, 119

TABLE 4
CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS

Analyte	Primary Ion*	Secondary Ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49, 51, 86
Acetone	43	58
Carbon disulfide	76	78
1,1-Dichloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	43**	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon tetrachloride	117	119, 121
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropane	63	65, 114
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	-
cis,-1,3-Dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100
Tetrachloroethene	164	129, 131, 166
Toluene	91	92
Chlorobenzene	112	114
Ethyl benzene	106	91
Styrene	104	78, 103
Total Xylenes	106	91

* The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.
**-m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

TABLE 5
VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SYSTEM MONITORING COMPOUNDS ASSIGNED FOR QUANTITATION

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d_5
Chlormethane	1,1,1-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	1,2-Dichloropropane	1,1,2,2-Tetrachloroethane
Methylene Chloride	trans-1,3-Dichloropropene	Toluene
Acetone	Trichloroethene	Chlorobenzene
Carbon Disulfide	Dibromochloromethane	Ethylbenzene
1,1-Dichloroethene	1,1,2-Trichloroethane	Styrene
1,1-Dichloroethane	Benzene	Xylene (total)
1,2-Dichloroethene (total)	cis-1,3-Dichloropropene	Bromofluorobenzene (smc)
Chloroform	Bromoform	Toleune d_8 (smc)
1,2-Dichloroethane		
2-Butanone		
1,2-Dichloroethane-d_4 (smc)		

(smc) = system monitoring compound

TABLE 6
SYSTEM MONITORING COMPOUND RECOVERY LIMITS

Compound	%Recovery Water	%Recovery Soil
Toluene-d_8	88-110	84-138
Bromofluorobenzene	86-115	59-113
1,2-Dichloroethane-d_4	76-114	70-121

TABLE 7
MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	%Recovery Water	RPD Water	%Recovery Soil	RPD Soil
1,1-Dichloroethene	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

VOLATILE QA/QC REQUIREMENTS

Introduction

This section is a guide to the specific QC operations that must be considered for volatile analyses. At a minimum, the laboratory is expected to address these operations in preparing the quality assurance plan and QA/QC Standard Operating Procedures discussed in here.

These QC requirements include the following:

- GC/MS Mass Calibration and Ion Abundance Patterns
- GC/MS Initial and Continuing Calibration
- Stability of Internal Standard Responses and Retention Times
- Blank Analysis
- System Monitoring Compound Recoveries
- Matrix Spike and Matrix Spike Duplicate Analyses

GC/MS Mass Calibration and Ion Abundance Patterns

Prior to initiating any data collection activities involving samples, blanks, or standards, it is necessary to establish that a given GC/MS system meets the instrument performance criteria specified in paragraphs of Instrument Operating Conditions. The purpose of this instrument performance check is to assure correct mass calibration, mass resolution, and mass transmission. This is accomplished through the analysis of Bromofluorobenzene (BFB).

1. The required frequency of BFB analysis (once every 12 hours on each GC/MS system) is described in SOP.

2. The key ions produced during the analysis of BFB and their respective ion abundance criteria are given in Table 1.

3. The documentation includes Form V VOA, and a mass listing and bar graph spectrum of each BFB analysis.

GC/MS Initial Calibration for Target Compounds and System Monitoring Compounds

Prior to the analysis of samples and required blanks and after instrument performance criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing target compound and system monitoring compound standards.

1. The concentrations of the initial calibration standards for volatile target compounds and system monitoring compounds are 10, 20, 50, 100, and 200 ug/l, as described in paragraph 5a of Standards.

2. The standards are to be analyzed according to the procedures given in paragraphs of Calibration, and at the frequency given in that paragraph.

3. The relative response factors (RRFs) are determined according to the procedures in paragraph 4 of Calibration, using the assignment of internal standard to target compounds and system monitoring compounds give in paragraph 4 of Calibration and Table 5.

4. The calibration of the GC/MS is evaluated on the basis of the magnitude and stability of the relative response factors of each target compound and system monitoring compound. The minimum RRF of each compound at each concentration level in the initial calibration and the percent relative standard deviation (%RSD) across all five points must meet the criteria given in paragraphs 4e and 4f of Calibration and Table 2. Allowance is made for any two volatile compounds that fail to meet these criteria. The minimum RRFs of those two compounds must be greater than or equal to 0.010, and the %RSD must be less than or equal to 40.0% for the initial calibration to be acceptable.

5. The documentation includes Form VI VOA, a GC/MS data system printout for the analysis of each volatile calibration standard.

GC/MS Continuing Calibration for Target Compounds and System Monitoring Compounds

Once the GC/MS system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

1. The concentration of the continuing calibration standard for volatile target compounds and system monitoring compounds is 50 ug/l, as described in paragraph 5c of Standards.

2. The standard is to be analyzed according to the procedures given in paragraphs of Standards, and at the frequency given in these paragraphs.

3. The continuing calibration of the GC/MS system is evaluated on the basis of the magnitude of the relative response factors and the percent difference between the average RRF of each compound from the initial calibration standard. The minimum RRF of each compound in the continuing calibration and the percent difference must meet the criteria given in paragraphs 4e to 4g of Calibration and Table 2. Allowance is made for any two volatile compounds that fail to meet these criteria. The minimum RRFs of those two compounds must be greater than or equal to 0.010, and the percent difference must be less than or equal to 40.0% for the continuing calibration to be acceptable.

4. The documentation includes Form VII VOA, a GC/MS data system printout for the analysis of the volatile calibration standard.

Internal Standard Responses and Retention Times

The response of each of the internal standards in all calibration standards, samples, and blanks is crucial to the provision of reliable analytical results, because the quantitative determination of volatile compounds by these procedures is based on the use of internal standards added immediately prior to analysis.

1. The specific compounds used as internal standards are given in paragraph 4c of Standards. The concentration of each internal standard in the aliquot of the sample analyzed by GC/MS must be 50 ug/l at the time of purging.

2. The retention time and the extracted ion current profile (EICP) of each internal standard must be

monitored for all analyses.

3. The area response of each internal standard from the EICP and the retention time of the internal standard are evaluated for stability, according to the procedures in paragraph 2 of Quantitative Analysis. The area of the internal standard in a sample must not vary by more than a factor of 2 (i.e. -50% to +100%) from the area of the same internal standard in the associated continuing calibration standard. Likewise, the retention time of an internal standard must be within ± 0.50 minutes (30 seconds) of its retention time in the continuing calibration standard (see paragraph 2 of Quantitative Analysis).

4. Requirements for re-analysis of samples when internal standards do not meet specifications are given in paragraph 2 of Quantitative Analysis.

5. The documentation includes Form VIII VOA, and the GC/MS data system printout for the analysis of each sample, blank, matrix spike, matrix spike duplicate, and standard.

Blank Analysis

There are three different types of blanks required by this method.

Method Blank:
A method blank is a volume of a clean reference matrix (deionized distilled water for water samples, or a purified solid matrix for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume of weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

1. For volatile analysis, a method blank must be analyzed once every 12 hours on each GC/MS system, as described in detail in paragraphs 1p, 2a (8) of Sample Analysis.

2. For the purposes of this protocol, an acceptable method blank must meet the criteria in paragraphs a and b below.

   a. A method blank for volatile analysis must contain less than or equal to 2.5 times the Contract Required Quantitation Limit (CRQL, see Table 8) of Methylene Chloride and contain less than or equal to five times of CRQL for Acetone and 2-Butanone.

   b For all other target compounds, the method blank must contain less than or equal to the Contract Required Quantitation Limit (CRQL, see Table 8) of any single target compound.

3. If a method blank exceeds the limits for contamination above, the laboratory must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for re-analysis of associated samples are given in paragraph 9 of Quantitative Analysis.

4. The documentation includes Form I VOA for the blank analysis, Form IV VOA, associating the samples and the blank, and a GC/MS data system printout for the analysis of the method blank.

Storage Blank:
Upon receipt of the first samples in an SDG, two 40 ml screw-cap volatile vials with a PTFE-faced silicone septum are filled with reagent water. The vials are stored with the samples in the SDG under the same conditions. After all samples in the SDG have been analyzed, the storage blank is analyzed. The storage blank

indicates whether contamination may be occurred during storage of samples. A minimum of one storage blank must be analyzed per SDG.

Instrument Blank:
A 5 ml aliquot of reagent water that is carried through the entire analytical procedure is considered as an instrument blank. It is analyzed after a sample/dilution which contains a target compound exceeding the initial calibration range. The results from the instrument blank analysis indicate whether there is contamination from a previous sample. Instrument blanks analyzed during the instrument decontamination process which exceed the requirements do not need to be reported.

System Monitoring Compound Recoveries

The recoveries of the three system monitoring compounds are calculated from the analysis of each sample, blank, matrix spike and matrix spike duplicate. The purpose of the system monitoring compounds is to evaluate the performance of the entire purge and trap-gas chromatograph-mass spectrometer system. Poor purging efficiency, leaks, and cold spots in transfer lines are only a few of the potential causes of poor recovery of these compounds.

1. The system monitoring compounds are added to each sample, blank, matrix spike, and matrix spike duplicate prior to purging or extraction (medium soils only), at the concentration described in paragraph 4d of Standards.

2. The recoveries of the system monitoring compounds are calculated according to the procedures in paragraph 7a of Quantitative Analysis.

3. The recoveries must be within the quality control limits given in Table 6. If the recovery of any one system monitoring compound is outside these limits, the laboratory must follow the steps outlined in paragraphs 7b to 7e of Quantitative Analysis.

4. The documentation includes Form II VOA, and a GC/MS data system printout for the analysis of each sample, blank, matrix spike, and matrix spike duplicate.

Matrix Spike and Matrix Spike Duplicate Analysis

In order to evaluate the effects of the sample matrix on the methods used for volatile analyses, the Agency has prescribed a mixture of volatile target compounds to be spiked into two aliquots of a sample and analyzed in accordance with the appropriate method.

1. The frequency of matrix spike and matrix spike duplicate (MS/MSD) analysis is described in paragraph 8 of Quantitative Analysis.

2. The recoveries of the matrix spike compounds are calculated according to the procedures in paragraph 8a of Quantitative Analysis. The relative percent difference between the results for each spiked analyte of the matrix spike and the matrix spike duplicate are calculated according to the procedures in paragraph 8b of Quantitative Analysis.

3. The quality control limits for recovery and relative percent difference are given in Table 7. These limits are only advisory at this time, and no further action is required when the limits are exceeded.

4. The documentation includes Form I VOA for both the MS and MSD analyses, Form III VOA, and a GC/MS printout for each analysis.

TABLE 8

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

	Volatiles	CAS Number	Quantitation Limits Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg
1.	Chloromethane	74-87-3	10	10	1200
2.	Bromomothane	74-83-9	10	10	1200
3.	Vinyl Chloride	75-01-4	10	10	1200
4.	Chloroethane	75-00-3	10	10	1200
5.	Methylene Chloride	75-09-2	10	10	1200
6.	Acetone	67-64-1	10	10	1200
7.	Carbon Disulfide	75-15-0	10	10	1200
8.	1,1-Dichloroethene	75-35-4	10	10	1200
9.	1,1-Dichloroethane	75-34-3	10	10	1200
10.	1,2-Dichloroethene (total)	540-59-0	10	10	1200
11.	Chloroform	67-66-3	10	10	1200
12.	1,2-Dichloroethane	107-06-2	10	10	1200
13.	2-Butanone	78-93-3	10	10	1200
14.	1,1,1-Trichloroethane	71-55-6	10	10	1200
15.	Carbon Tetrachloride	56-23-5	10	10	1200
16.	Bromodichloromethane	75-27-4	10	10	1200
17.	1,2-Dichloropropane	78-87-5	10	10	1200
18.	cis-1,3-Dichloropropene	10061-01-5	10	10	1200
19.	Trichloroethene	79-01-6	10	10	1200
20.	Dibromochloromethane	124-48-1	10	10	1200
21.	1,1,2-Trichloroethane	79-00-5	10	10	1200
22.	Benzene	71-43-2	10	10	1200
23.	trans-1,3-Dichloropropene	10061-02-6	10	10	1200
24.	Bromoform	75-25-2	10	10	1200
25.	4-Methyl-2-pentanone	108-10-1	10	10	1200
26.	2-Hexanone	591-78-6	10	10	1200
27.	Tetrachloroethene	127-18-4	10	10	1200
28.	Toluene	108-88-3	10	10	1200
29.	1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200
30.	Chlorobenzene	108-90-7	10	10	1200
31.	Ethyl Benzene	100-41-4	10	10	1200
32.	Styrene	100-42-5	10	10	1200
33.	Xylenes (Total)	1330-20-7	10	10	1200

STANDARD OPERATING PROCEDURE

ANALYTICAL METHODS

for

SEMI-VOLATILES

EPA-CLP-SOW

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Semi-Volatiles - (BNA)

ANALYTICAL METHODS FOR SEMI-VOLATILES

SCOPE AND APPLICATION

1. This method covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. These target compounds and the contract required quantitation limits are listed in Table 9.

2. The method involves solvent extraction of the matrix sample, characterization to determine the appropriate analytical protocol to be used, and GC/MS analysis to determine semi-volatile organic compounds present in a sample.

SAMPLE STORAGE AND HOLDING TIMES

Procedures for Sample Storage

1. The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

2. Samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.

Contract Required Holding Time

1. Extraction of water samples by continuous liquid-liquid procedures shall be started within 5 days of VTSR (Validated Time of Sample Receipt): Extraction of soil/sediment samples by sonication procedures shell be completed within 10 days of VTSR. NOTE: Separatory funnel extraction procedures are not permitted.

2. Extracts of either water or soil/sediment samples must be analyzed within 40 days following extractions.

SAMPLE PREPARATION FOR WATER

Summary of Sample Preparation Method

A one liter aliquot of sample is acidified to pH 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. Separatory funnel extraction is NOT permitted. The methylene chloride extract is dried and concentrated to a volume of 1.0 ml.

Interferences

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source-to-source.

Apparatus and Materials

1. Glassware

   a. Continuous liquid-liquid extractors - equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication.

   b. Concentrator tube - Kuderna-Danish, 10 ml, graduated. Calibration must be checked at the volumes employed in the test. Ground-glass stoppers are used to prevent evaporation of extracts.

   c. Evaporative flask - Kuderna-Danish, 500 ml. Attached to concentrator tube with springs.

   d. Snyder column - Kuderna-Danish, three-ball macro.

   e. Snyder column - Kuderna-Danish, two-ball micro.

   f. Vials - amber glass, 2 ml capacity with Teflon-lined screw cap.

   g. Syringes - 0.2 ml, 0.5 ml, and 5 ml volumes.

2. Silicon carbide boiling chips - approximately 10/40 mesh.

3. Water bath - heated, with concentric ring cover, capable of temperature control ($\pm$ 2°C). The bath should be used in a hood.

4. Balance - analytical, capable of accurately weighing $\pm$ 0.0001 g.

5. Nitrogen evaporation device equipped with a bath that can be maintained at 35-40°C.

Reagents

1. Reagent water - defined as water in which an interferent is not observed at or above the CRQL of each parameter of interest.

2. Sodium thiosulfate - (ACS) granular.

3. Sulfuric acid solution (1+1) - slowly add 50 ml of H_2SO_4 (sp gra 1.84) to 50 ml of reagent water.

4. Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent.

5. Sodium sulfate - (ACS) powdered, anhydrous. Purify by heating at 400°C for four hours in a shallow tray, cool in a desiccator and store in a glass bottle.

6. Surrogate standard spiking solution.

   a. Surrogate standards are added to all samples and calibration solutions; the compounds specified for this purpose are Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, 2-Chlorophenol-d_4, and 1,2-Dichlorobenzene-d_4. Additional surrogates may be added at the laboratory's discretion.

b. Prepare a surrogate standard spiking solution that contains Nitrobenzene-d_5, Terpheyl-d_{14}, 2-Fluorobiphenyl, and 1,2-Dichlorobenzene-d_4 at a concentration of 100 ug/ml; Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, and 2-Chlorophenol-d_4 at a concentration of 150 ug/ml. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

7. BNA Matrix standard spiking solution - the matrix spike solution consists of the following:

Bases/Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

Prepare a spiking solution that contains each of the base/neutral compounds above at 100 ug/1.0 ml in methanol and the acid compounds at 150 ug/1.0 ml in methanol. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replace after twelve months, or sooner if comparison with quality control check samples indicates a problem.

Water Sample Extraction

1. Continuous liquid-liquid extraction is used to extract the samples.

a. Add methylene chloride to the bottom of the extractor and fill it to a depth of at least 1 inch above the bottom side arm.

b. Using a 1 liter graduated cylinder, measure out a 1.0 liter sample aliquot. Transfer the 1 liter sample aliquot to the continuous extractor. Pipet 0.5 ml of surrogate standard spiking solution into the sample and mix well. Check the pH of the sample with wide-range pH paper and adjust the pH to 2.0 with 1:1 H_2SO_4.

c. Following the procedures in 1a and 1b above, prepare two additional 1.0 liter aliquots of the sample chosen for spiking. Add 0.5 ml of the BNA Matrix Spiking Solution to each of the additional aliquots. The frequency of MS/MSD analysis is given in paragraph 6 of Quantitation.

d. Add 500 ml of methylene chloride to the distilling flask. Add sufficient reagent water to ensure proper operation. Extract for 18 hours. Allow to cool, then detach the distilling flask, and label the flask.

e. Prepare a method blank with each group of water samples extracted. For semi-volatile analyses, a method blank for water samples consists of a 1 L volume of reagent water (see paragraph 1 of Reagents), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in paragraph 7 of Quantitation.

2. Concentrating the Extracts

a. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 ml concentrator tube to a 500 ml evaporative flask. Other concentration devices or techniques may be used in place of the K-D, if equivalency is demonstrated for all the semi-volatile target compounds listed in Table 9.

b. Transfer the extract by pouring the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate, and collect the extract in a K-D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 ml of methylene chloride to complete the quantitative transfer.

c. Add one or two clean boiling chips and attach a three-ball Snyder column to the evaporative flask. Pre-wet the Snyder column by adding about 1 ml methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60°C to 80°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation, the balls of the column will chatter actively, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 ml, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 ml of methylene chloride. a 5 ml syringe is recommended for this operation.

5. Nitrogen Evaporation Technique

a. Place the concentrator tube with an open micro Snyder attached in a warm water bath (30°C to 35°C) and evaporate the solvent volume to just below 1 ml by blowing a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon) above the extract. **CAUTION: Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or Teflon tubing. The internal wall of the concentrator tube must be rinsed down several times with methylene chloride during the operation and the final volume brought to 1.0 ml with methylene chloride. During the evaporation, the tube solvent level must be kept below the water level of the bath. The extract must never be allowed to become dry. Transfer the extract to a Teflon-sealed screw-cap bottle, label the bottle and store at 4°C (±2°C).**

6. The sample extracts are ready for GC/MS analysis.

SAMPLE PREPARATION FOR SOIL/SEDIMENT

It is mandatory that all soil/sediment samples be characterized as to concentration level so that the appropriate analytical protocol is chosen to ensure proper quantitation limits for the sample. Note that the terms "low-level" and "medium-level" are not used here as a judgement of degree of contamination but rather as a description of the concentration ranges that are encompassed by the "low" and "medium" level procedures.

The laboratory is at liberty to determine the method characterization. The following two screening methods may be used for soil/sediment sample characterization:

- Screen an aliquot from "low-level" 30 g extract or an aliquot from the "medium level" 1 g extract.
- Screen using either GC/FID or GC/MS as the screening instrument.

The concentration ranges covered by these two procedures may be considered to be approximately 330 ug/kg - 10,000 ug/kg for the low-level analysis and >10,000 ug/kg for medium-level analysis for semi-volatile extractables.

Screen from the Medium-Level Method

Take 5.0 ml from the 10.0 ml total extract and concentrate to 1.0 ml and screen. If the sample concentration is >10,000 ug/kg, proceed with GC/MS analysis of the organics. If the sample concentration is <10,000 ug/kg, proceed with concentration and the remainder of the low-level method.

Screen from the Low-Level Method

Take 5.0 ml from the 300 ml (approximate) total extract from the 30 g sample and concentrate to 1.0 ml and screen. If the original sample concentration is >10,000 ug/kg, discard the 30 g extract and follow the medium level methods for organics, using medium level surrogates. If the sample concentration is <10,000 ug/kg, proceed with concentration and the remainder of the low-level method.

Mandatory GPC Cleanup

All soil/sediment sample extracts must be subjected to cleanup by Gel Permeation Chromatography (GPC).

Medium Level Preparation for Screen and Analysis of Semi-volatiles

Scope and Application

This procedure is designed for the preparation of soil/sediment samples which may contain organic chemicals at a level greater than 10,000 ug/kg.

1. The extracts and sample aliquots prepared using this method are screened by GC/MS or FID, using capillary columns for semi-volatile priority pollutants and related organic chemicals. The results of these screens will determine whether sufficient quantities of pollutants are present to warrant analysis by the medium protocol.

2. If the screenings indicate no detectable pollutants at the lower limits of quantitation, the sample should be prepared by the low-level protocol described below.

Summary of Method

1. Approximately 1 g portions of soil/sediment are transferred to vials and extracted with methylene chloride. The methylene chloride extract is screened for extractable organics by GC/FID or GC/MS.

2. If organic compounds are detected by the screen, the methylene chloride extract is subjected to GPC clean up and analyzed by GC/MS for extractable organics.

3. If no organic compounds are detected by the medium level screen, then a low-level sample preparation is required.

Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample-processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source-to-source.

Limitations

1. The procedure is designed to allow quantitation limits for screening purposes as low as 10,000 ug/kg for extractable organics. For analysis purposes, the quantitation limits are 10,000 ug/kg for extractable organics. If peaks are present based on the GC/FID screen, the sample is determined to require a medium-level analysis by GC/MS. Some samples may contain high concentrations of chemicals that interfere with the analysis of other components at lower levels; the quantitation limits in those cases may be significantly higher.

2. These extraction and preparation procedures were developed for rapid and safe handling of high concentration hazardous waste samples. The design of the methods thus does not stress efficient recoveries of low limits of quantitation of all components. Rather the procedures were designed to screen, at moderate recovery and sufficient sensitivity, a broad spectrum of organic chemicals. The results of the analyses thus may reflect only a minimum of the amount actually present in some samples.

Reagents

1. Sodium Sulfate - anhydrous powdered reagent grade, heated at 400°C for four hours, cooled in a desiccator and stored in a glass bottle (Baker anhydrous powder, catalog #73898 or equivalent).

2. Acetone, Methanol, Methylene chloride - pesticide residue analysis grade or equivalent.

3. Base/neutral and Acid Surrogate Spiking Solution

   Surrogate standards are added to all samples and calibration solutions. The compounds specified are Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, 2-Chlorophenol-d_4, and 1,2-Dichlorobenzene-d_4. Prepare a surrogate standard spiking solution that contains Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, and 1,2-Dichlorobenzene-d_4 at a concentration of 100 ug/ml; Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol and 2-Chlorophenol-d_4 at concentration of 150 ug/ml. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

4. Base/Neutral and Acid Matrix Spiking Solution

Prepare a spiking solution in methanol that contains the following compounds at a concentration of 100 ug/ml for base/neutrals and 150 ug/ml for acids. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

Bases/Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

Equipment

1. Glass scintillation vials - at least 20 ml, with screw cap and Teflon or aluminum foil liner.
2. Spatula - stainless steel or Teflon.
3. Balance - capable of weight 100 g to ± 0.01 g.
4. Vials and caps - 2 ml for GC auto sampler.
5. Disposable pipets - Pasteur; glass wool rinsed with methylene chloride.
6. Concentrator tubes - 15 ml.
7. Ultrasonic cell disrupter - Heat Systems, Ultrasonics, Inc., Model W-385 SONICATOR (475 watt with pulsing capability, No. 200, 1/2 inch tapped disrupter horn and No. 419, 1/8 inch standard tapered MICROTIP probe), or equivalent device with a minimum of 375 Watt output capability. **NOTE:** In order to ensure that sufficient energy is transferred to the sample during extraction, the MICROTIP probe must be replaced if the tip begins to erode. Erosion of the tip is evidenced by a rough surface.
8. Sonabox acoustic enclosure - recommended with above disrupters for decreasing cavitation sound.
9. Test tube rack.
10. Oven - drying.
11. Desiccator.
12. Crucibles - porcelain.
13. Syringes - 0.5 ml volume.

Medium Level Sample Preparation

1. Transfer the sample container into a fume hood. Decant and discard any water layer on a sediment sample. Mix samples thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves and rocks. Transfer approximately 1 g (record weight to the nearest 0.1 g) of sample to remove any sample material. Record the exact weight of sample taken. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

   a. Transfer 50 g of soil/sediment to a 100 ml beaker. Add 50 ml of water and stir for 1 hour. Determine pH of sample with glass electrode and pH meter while stirring. Report pH value on appropriate data sheets. If the pH of the soil is greater than 11 or less than 5, contact the Technical Project Officer cited in the contract for instructions on how to handle the sample. Document the instructions in the SDG Narrative. Discard this portion of sample. **NOTE:** If limited sample volume is received, use 5 g of soil and 5 ml of water for pH determination. Note this is a SDG Narrative.

2. Immediately after weighing the sample for extraction, weigh 5-10 g of the sediment into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\frac{\text{g of sample - g of dry sample}}{\text{g of sample}} \times 100 = \%\ \text{moisture}$$

3. Add 2.0 g of anhydrous powdered sodium sulfate to the sample in the 20 ml vial from paragraph 1 above and mix well.

4. Surrogates are added to all samples, spikes and blanks. Add 0.5 ml of surrogate spiking solution to sample mixture.

5. Add 0.5 ml of matrix standard spiking solution to each of two 1 g portions from the sample chosen for spiking. The frequency of MS/MSD analysis is given in paragraph 6 of Quantitation.

6. Immediately add 9.5 ml of methylene chloride to the sample and disrupt the sample with the 1/8 inch tapered MICROTIP ultrasonic probe for 2 minutes at output control setting 5, in continuous mode (if using a sonicator other than Models W-375 or W-385, contact the Project Officer for appropriate output settings). Before extraction, make certain that the sodium sulfate is free flowing and not a consolidated mass. As required, breakup large lumps with a clean spatula or, very carefully, with the tip of the unenergized probe.

   Add only 9.0 ml of methylene chloride to the matrix spike samples to achieve a final volume of 10 ml.

7. Prepare a method blank with each group of medium soil/sediment samples extracted. For semi-volatile analyses, a method blank for medium soil/sediment samples consists of 1 g sodium sulfate (see paragraph 1 of Reagents), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in paragraph 7 of Quantitation.

8. Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extract through the glass wool and collect at least 8.0 ml in a concentrator tube.

9. If the extract is to be screen prior to GPC, concentrate 5.0 ml of the extract collected in paragraph 7 above to 1.0 ml using the nitrogen evaporation technique described in paragraph of Final Extract Concentration. Transfer the concentrate to an autosampler vial for GC/FID or GC/MS for screening. The quantitation limits for the screening procedures in Screening of Semi-Volatile Organics are approximately 10,000 ug/kg.

10. If the extract is to be cleaned up using GPC without screening, take at least 8.0 ml of the extract in paragraph 7 above and proceed to paragraph Extract Cleanup by GPC of this section. Following GPC, the 5.0 ml of extract collected must be concentrated to 0.5 ml by the nitrogen evaporation technique described in paragraph of Final Extract Concentration, and screened according to the procedures in Screening of Semi-Volatile Organics. In this case, the quantitation limits for the screening procedures in this section are approximately 20,000 ug/Kg.

Low Level Preparation for Screening and Analysis of Semi-Volatiles

Summary of Method

A 30-gram portion of sediment is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone using an ultrasonic probe. If the optional low level screen is used, a portion of this dilute extract is concentrated fivefold and is screened by GC/FID or GC/MS. If peaks are present at greater than 10,000 ug/kg, discard the extract and prepare the sample by the medium level method. If no peaks are present at greater than 10,000 ug/kg, the entire extract is concentrated, subjected to GPC cleanup, and analyzed by GC/MS for extractable organics.

Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample-processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source-to-source.

Apparatus and Materials

1. Brand names, suppliers, and part numbers ar for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance meeting the requirements of this SOW is the responsibility of the laboratory.

    a. Apparatus for determining percent moisture.

        (1) Oven - drying.

        (2) Desiccator.

        (3) Crucibles - porcelain.

    b. Disposable Pasteur glass pipets - 1 ml.

c. Ultrasonic cell disrupter, Heat Systems, Ultrasonics, Inc., Model W-385 SONICATOR.

Sonabox acoustic enclosure - recommended with above disruptors for decreasing cavitation sound.

d. Beakers - 400 ml

e. Vacuum filtration apparatus.

(1) Buchner funnel.

(2) Filter paper - Whatman No. 41 or equivalent.

f. Kuderna-Danish (K-D) apparatus.

(1) Concentrator tube - 10 ml, graduated.

(2) Evaporative flask - 500 ml.

(3) Snyder column - three-ball macro.

g. Silicon carbide boiling chips - approximately 10/40 mesh.

h. Water bath - heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

i. Balance - capable of accurately weighing ± 0.01 g.

j. Vials and caps - 2 ml for GC auto sampler.

k. Balance - analytical, capable of accurately weighing ± 0.0001 g.

l. Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C.

m. Pyrex glass wool.

n. Pasteur pipets - disposable.

o. Syringes - 0.5 ml volume.

Reagents

1. Sodium Sulfate - anhydrous powdered reagent grade, heated at 400°C for four hours, cooled in a desiccator, and stored in a glass bottle.

2. Methylene chloride, methanol, acetone, isoctane, 2-propanol, and benzene - pesticide residue analysis grade or equivalent.

3. Reagent water - defined as water in which an interferent is not observed at or above the CRQL of each parameter of interest.

4. Sodium Sulfite - reagent grade.

5. Base/Neutral and Acid Surrogate Spiking Solution

Surrogate standards are added to all samples and calibration solutions. The compounds specified are Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, 2-Chlorophenol-d_4, and 1,2-Dichlorobenzene-d_4. Prepare a surrogate standard spiking solution that contains Nitrobenzene-d_5, Terphenyl-d_{14}, 2-Fluorobiphenyl, and 1,2-Dichlorobenzene-d_4 at a concentration of 100 ug/ml; Phenol-d_5, 2,4,6-Tribromophenol, 2-Fluorophenol, and 2-Chlorophenol-d_4 at a concentration of 150 ug/ml. Store the spiking solutions at 4°C (±2°C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

6. Base/Neutral and Acid Matrix Spiking Solution

Prepare a spiking solution in methanol that contains the following compounds at a concentration of 100 ug/ml for base/neutrals and 150 ug/ml for acids. Store the spiking solutions at 4°C (±2C) in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after twelve months, or sooner if comparison with quality control check samples indicates a problem.

Bases/Neutral	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenapthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

Low Level Sample Preparation

1. Decant and discard any water layer on a sediment sample. Mix samples thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks.

2. The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30 g of sample to the nearest 0.1 g into a 400-ml beaker and add 60 g of anhydrous powdered sodium sulfate. Mix well. The sample should have a sandy texture at this point. Immediately, add 100 ml of 1:1 methylene chloride-acetone to the sample, then add the surrogates according to paragraph c below.

   a. Immediately after weighing the sample for extraction, weigh 5-10 g of the sediment into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\frac{\text{g of sample - g of dry sample}}{\text{g of sample}} \times 100 = \% \text{ moisture}$$

   b. Weigh of two 30 g (record weight to nearest 0.1 g) portions for use as matrix spike duplicates

according to paragraph 2 above. Add 0.5 ml of the BNA matrix spike solution to each of two portions. The frequency of MS/MSD analysis is given in paragraph 6 of Quantitation.

c. Add 0.5 ml of base/neutral and acid surrogate standard to sample and each of the aliquots in paragraph b above.

d. Prepare a method blank with each group of low soil/sediment samples extracted. For semi-volatile analyses, a method blank for low-soil/sediment samples consists of 30 g of sodium sulfate (see paragraph 1 of Reagents), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in paragraph 7 of Quantitation.

3. Place the bottom surface of the tip of the 3/4 inch disrupter horn about 1/2 inch below the surface of the solvent but above the sediment layer.

4. Sonicate for 1-1/2 minutes with the W-385 (or 3 minutes with W-375), using No. 208, 3/4 inch standard disrupter horn with output control knob set at 10 (or No. 305, 3/4 inch tapped high gain "Q" disrupter horn at 5) and mode switch on "1 sec. pulse" and % duty cycle knob set at 50% (if using a sonicator other than Models W-375 or W-385, contact the Project Officer for appropriate output settings).

5. Decant and filter extracts through Whatman #41 filter paper using vacuum filtration or centrifuge and decant extraction solvent.

6. Repeat the extraction two more times with 2 additional 100 ml portions of 1:1 methylene chloride-acetone. Before each extraction, make certain that the sodium sulfate is free flowing and not a consolidated mass. As required, break up large lumps with a clean spatula or, very carefully, with the tip of the unenergized probe. Decant the extraction solvent after each sonication. On the final sonication, pour the entire sample into the Buchner funnel and rinse with 1:1 methylene chloride-acetone.

a. If the sample is to be screened from the low-level method prior to GPC, take 5.0 ml and concentrate to 1.0 ml following paragraph of Final Concentration of Extract, but not that the final volume for screening is 1.0 ml, not 0.5 ml.

b. After GC/FID or GC/MS screening, transfer the remainder of the 1 ml back to the total extract from paragraph 6 above. **CAUTION:** To minimize sample loss, autosamplers which pre-flush through the syringe should not be used.

Concentration and Solvent Exchange

1. Low-level soil/sediment samples prepared by the procedures in paragraph of Low Level Sample Preparation will result in extracts containing a mixture of acetone and methylene chloride. Because all soil/sediment sample extracts must be subjected to GPC clean up prior to analysis, the majority of the acetone must be removed from the extract, otherwise it will have adverse effects on the GPC column. To remove the acetone from the sample extract, follow the steps below.

2. Transfer the extract to a Kuderna-Danish (K-D) concentrator consisting of a 10 ml concentrator tube and a 500 ml evaporative flask. Other concentration devices or techniques may be used if equivalency is demonstrated for all extractable compounds listed in Table 9.

3. Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 ml methylene chloride to the top. Place the K-D apparatus on a hot water bath (60 to 80°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation in the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reached 1 ml, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. Do not allow the evaporator to go dry.

4. Dilute the extract to 10.0 ml with methylene chloride, and proceed with GPC clean up (see paragraph below.

Extract Cleanup by Gel Permeation Chromatography

Gel permeation chromatography (GPC) is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of synthetic macromolecules. The packing gel is porus and is characterized by the range or uniformity (exclusion range) of the pore size. In the choice of gels, the exclusion range must be larger than the molecular size of the molecules to be separated. A cross-linked divinyl benzenestyrene copolymer (SX-3 Bio Beads or equivalent) is specified for this method.

GPC is required for all soil/sediment samples, regardless of concentration level, for the elimination of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds from the sample extract. GPC is appropriate for both polar and non-polar analytes, therefore, it can be used effectively to cleanup extracts containing a broad range of analytes.

Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a gas chromatograph (GC) or in the front of the GC column. This residue ultimately will reduce the chromatographic separation efficiency or column capacity because of adsorption of the target analytes on the active sites. Pentachlorophenol especially is susceptible to this problem.

Apparatus and Materials

1. Gel permeation chromatography (GPC) cleanup device.

   Gel permeation chromatography system - GPC Autoprep Model 1002 A or B (Analytical Biochemical Laboratories, Inc., or equivalent) Systems that perform very satisfactorily also have been assembled from the following components - an HPLC pump, an auto sampler or a valving system with sample loops, and a fraction collector. All systems, whether automated or manual must meet the calibration requirements in paragraph of Calibration of the GPC Column described below.

   a. Chromatographic column - 700 mm x 25 mm i.d. glass column. Flow is upward. To simplify switching from the UV detector during calibration to the GPC collection device during extract cleanup, an optional double 3-way valve may be attached so that the column exit flow can be shunted either to the UV flow-through cell or to the GPC collection device.

   b. Guard column - (Optional) 5 cm, with appropriate fittings to connect to the inlet side of the analytical column.

c. Bio beads (S-X3) - 200-400 mesh, 70 gm. An additional 5g of Bio Beads is required if the optional guard column is employed. The quality of Bio Beads may vary from lot-to-lot because of excessive fines in some lots. In addition to fines having a detrimental effect on chromatography, they also can pass through the column screens and damage the valve.

d. Ultraviolet detector - fixed wavelength (254 nm) with a semi-prep flow-through cell.

e. Strip chart recorder, recording integrator or laboratory data system.

f. Syringe = 10-ml with Luerlok fitting.

g. Syringe filter assembly, disposable - Bio-Rad "Prep Disc" sample filter assembly #343-0005, 25 mm, and 5 micron filter discs or equivalent. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

Reagents

1. GPC Calibration Solution - prepare a calibration solution in methylene chloride containing the following analytes (in elution order):

Compound	mg/ml
corn oil	25.0
bis (2-ethylhexyl) phthalate	1.0
methoxychlor	0.2
perylene	0.02
sulfur (optional)	0.08

Column Preparation

1. Weigh out 70 gm of Bio Beads (SX-3). Transfer them to a quart bottle with a Teflon-lined cap or a 500 ml separatory funnel with a large bore stopcock, and add approximately 300 ml of methylene chloride. Swirl the container to ensure the wetting of all beads. Allow the beads to swell for a minimum of 2 hours. Maintain enough solvent to cover the beads sufficiently at all times. If a guard column is to be used, repeat the above with 5 gm of Bio Beads in a 125 ml bottle or a beaker, using 25 ml of methylene chloride.

2. Turn the column upside down from its normal position, and remove the inlet bed support plunger (the inlet plunger is longer than the outlet plunger). Position and tighten the outlet bed support plunger as near the end as possible, but no closer than 5 cm (measured from the gel packing to the collar).

3. Raise the end of the outlet tube to keep the solvent in the GPC column, or close the column outlet stopcock. Place a small amount of solvent in the column to minimize the formation of air bubbles at the base of poured column packing.

4. Swirl the bead/solvent slurry to get a homogeneous mixture and, if the wetting was done in a quart bottle, quickly transfer it to a 500 ml separatory funnel with a large bore stopcock. Drain the excess methylene chloride directly into the waste beaker, and then start draining the slurry into the column by placing the separatory funnel tip against the column wall. This will help to minimize bubble formation.

Swirl occasionally to keep the slurry homogeneous. Drain enough to fill the column. Place the tubing from the column outlet into a waste beaker below the column, open the stopcock and allow the excess solvent to drain. Raise the tube to stop the flow and close the stopcock when the top of the gel begins to look dry. Add additional methylene chloride to just re-wet the gel.

5. Wipe any remaining beads and solvent from the inner walls to the top of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

6. Compress the column as much as possible without applying excessive force. Loosen the seal and gradually pull out the plunger. Rinse and wipe off the plunger. Slurry any remaining beads and transfer them into the column. Repeat the step in paragraph 5 above and reinsert the plunger. If the plunger cannot be inserted and pushed in without allowing beads to escape around the seal, continue compression of the beads without tightening the seal, and loosen and remove the plunger as described. Repeat this procedure until the plunger is inserted successfully.

7. Push the plunger until it meets the gel, then compress the column bed about four centimeters.

8. Pack the optional 5 cm column with approximately 5 gm of pre-swelled beads. Connect the guard column to the inlet of the analytical column.

9. Connect the column inlet to the solvent reservoir (reservoir should be placed higher than the top of the column) and place the column outlet tube in a waste container. Placing a restrictor in the outlet tube will force air out of the column more quickly. A restrictor can be made from a piece of capillary stainless steel tubing of 1/16" OD x 10/1000" ID x 2". Pump methylene chloride through the column at a rate of 5 ml/min for one hour.

10. After washing the column for at least one hour, connect the column outlet tube, without the restrictor, to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. A restrictor in the outlet tube from the UV detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not effect flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi backpressure is achieved. Push the plunger in to increase pressure to slowly pull outward to reduce pressure.

11. When the GPC column is not to be used for several days, connect the column outlet line to the column inlet to prevent column drying and/or channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, methylene chloride should be pumped through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify retention volumes have not changed.

<u>Calibration of the GPC Column</u>

1. Using a 10 ml syringe, load sample loop #1 with calibration solution (paragraph of Reagents above). With the ABC automated system, the 5 ml sample loop requires a minimum of 8 ml of the calibration solution. Use a firm, continuous pressure to push the sample onto the loop. Switch the valve so that GPC flow is through the UV flow-through cell.

2. Inject the calibration solution and obtain the UV trace showing a discrete peak for each component. Adjust the detector and/or recorder sensitivity to produce a UV trace that meets the following

requirements. Differences between manufacturer's cell volumes and detector sensitivities may require a dilution of the calibration solution to achieve similar results. An analytical flow-through detector cell will require a much less concentrated solution than the semi-prep cell and, therefore, the analytical cell is not acceptable for use.

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit >85% resolution.
- Phthalate and methoxychlor peaks must exhibit >85% resolution.
- Methoxychlor and perylene peaks must exhibit >85% resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

3. Using the information from the UV trace, establish appropriate collect and dump time periods to ensure collection of all target analytes. Initiate column eluate collection just before elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Collection should be stopped before sulfur elutes. Use a "wash" time of 10 minutes after the elution of sulfur. Each laboratory is required to establish its specific time sequences.

4. Verify the flow rate by collecting column eluate for 10 minutes in a graduated cylinder and measure the volume, which should be 45-55 ml (4.5-5.5 ml/min). If the flow rate is outside of this range, corrective action must be taken, as described above. Once the flow rate is within the range of 4.5-5.5 ml/min, record the column pressure (should be 6-10 psi) and room temperature. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared. A UV trace that does not meet the criteria in paragraph 2 above would also indicate that a new column should be prepared. It may be necessary to obtain a new lot of Bio Beads if the column fails all the criteria.

5. Re-inject the calibration solution after appropriate collect and dump cycles have been set, and the solvent flow and column pressure have been established.

   a. Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed may be used to indicate problems with the system during sample processing.

   b. The retention times of bis(2-ethylhexyl)phthalate and perylene must not vary more than ±5% between calibrations. If the retention time shift is >5%, take corrective action. Excessive retention time shifts are caused by the following:

      - Poor laboratory temperature control or system leaks.
      - An unstabilized column that requires pumping methylene chloride through it for several more hours or overnight.
      - Excessive laboratory temperatures causing outgassing of the methylene chloride.

6. Analyze a GPC blank by loading 5 ml of methylene chloride into the GPC. Concentrate the methylene chloride that passes through the system during the collect cycle using a Kuderna-Danish (KD) evaporator. Analyze the concentrate by GC/MS. If the blank exceeds one-half the CRQL of any analyte, pump additional methylene chloride through the system for 1-2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

Sample Extract Cleanup

It is very important to have consistent laboratory temperatures during an entire GPC run, which could be 24 hours or more. If temperatures are not consistent, retention times will shift, and the dump and collect times determined by the calibration standard no longer will be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C.

1. In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with viscosity greater than that of a 1:1 glycerol:water solution must be diluted and loaded into several loops. Similarly, extracts containing more than 500 mg of nonvolatile residue per 5 ml of extract must be diluted and loaded into several loops. The nonvolatile residue may be determined by evaporating a 100 ul aliquot of the extract to dryness in a tared aluminum weighing pan, or other suitable container.
2. Particles greater than 5 microns may scratch the valve, which may result in a system leak and cross contamination of sample extracts in the sample loops. To avoid such problems, filter the extract through a 5 micron filter disc by attaching a syringe filter assembly containing the filter disc by attaching a syringe filter assembly containing the filter disc to a 10 ml syringe. Draw the sample extract through the filter assembly before transferring the sample extract into a small glass container, e.g., a 15 ml culture tube with a Teflon lined screw cap. Alternatively, draw the extract into a syringe without the filter assembly. Attach the filter assembly and force the extract through the filter and into the glass container. Draw a minimum of 8 ml of extract into a 10 ml syringe.

3. Attach the syringe to the turn lock on the injection port. Use firm, continuous pressure to push the sample onto the 5-ml sample loop. If the sample is difficult to load, some part of the system may be blocked. Take appropriate corrective action. If the back pressure is normal (6-10 psi) the blockage is probably in the valve. Blockage may be flushed out of the valve by reversing the inlet and outlet tubes and pumping solvent through the tubes (this should be done before sample loading).

   NOTE: Approximately 2 ml of the extract remains in the lines between the injection port and the sample loop; excess sample also passes through the sample loop to waste.

4. After loading a loop, and before removing the syringe from the injection port, index the GPC to the next loop. This will prevent loss of sample caused by unequal pressure in the loops.

5. After loading each sample loop, wash the loading port with methylene chloride in a PTFE wash bottle to minimize cross contamination. Inject approximately 10 ml of methylene chloride to rinse the common tubes.

6. After loading all the sample loops, index the GPC to the 00 position, switch to the "RUN" mode and start the automated sequence. Process each sample using the collect and dump cycle times established in Calibration of GPC Column.

7. Collect each sample in a 250-ml Erlenmeyer flask, covered with aluminum foil to reduce solvent evaporation, or directly into a Kuderna-Danish evaporator. Monitor sample volumes collected. Changes in sample volumes collected may indicate one or more of the following problems:

   - Change in solvent flow rate, caused by channeling in the column or changes in column pressure.
   - Increase in column operating pressure due to the absorption of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.
   - Leaks in the system or significant variances in room temperature.

8. Concentrate the extract as per paragraph of Final Concentration of Extract below.

9. Calibrate the GPC at least once per week, following the procedure outlined in Calibration of GPC Column. The UV trace must meet the requirements. In addition, the retention times of the calibration compounds must be within ±5% of their retention times in the previous calibration. A copy of the UV trace of the calibration solution must be submitted with the data for the associated samples.

10. If the requirements in paragraphs of Calibration of GPC Column cannot be met, the column may be cleaned by processing several 5 ml volumes of butyl chloride through the system. Butyl chloride removes the discoloration and particles that may have precipitated out of the methylene chloride extracts. If a guard column is being used replace it with a new one. This may correct the problem. If column maintenance does not restore the performance of the column, the column must be replaced with new packing re-calibrated.

Final Concentration of Extract

1. Nitrogen evaporation technique

   Place the concentrator tube in a warm bath (35°C) and evaporate the solvent volume to below 0.5 ml using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

   The internal wall of the tube must be rinsed down several times with methylene chloride during the operation. During evaporation, the tube solvent level must be kept below the level of the bath. The extract must never be allowed to become dry. Concentrating the extract to 0.5 ml will result in no loss of sensitivity despite the volume of extract (5 ml) not recovered after GPC.

2. Store all extracts at 4°c (±2° C) in the dark in Teflon-sealed containers. The extracts are ready for GC/MS analysis.

Screening of Semi-Volatile Organic Extracts

Summary of Method

The solvent extracts of water and soil/sediment are screened on a gas chromatograph/flame ionization detector (GC/FID) using a fused silica capillary column (FSCC). For water samples, the results of the screen may be used to determine an appropriate dilution factor for the GC/MS analysis of the sample extract. For soil/sediment samples, the results of the screen are used to determine which of the two sample preparation procedures (low or medium is required, and to determine an appropriate dilution factor for GC/MS analysis. The results of the screen may be used also to assist the analyst in performing Gel Permeation Chromatography (GPC) clean up procedures on extracts of either water or soil/sediment samples.

Apparatus and Materials

1. Gas chromatograph - an analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns and gases. The injection port must be designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

   a. Above GC equipped with flame ionization detector.

   b. GC column - 30 m x 0.32 mm, 1 micron film thickness, silicone coated, fused silica capillary column (J & W Scientific DB-5 or equivalent).

Reagents

1. Methylene chloride - pesticide residue analysis grade or equivalent.

2. GC calibration standard - prepare a standard solution containing phenol, phenanthrene and di-n-octylphthalate.

   a. Stock standard solutions (1.00 ug/ul) - Stock standard solutions can be prepared from pure standard materials or purchased solutions.

      (1) Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality methylene chloride and dilute to volume in a 10 ml volumetric flask. Larger volumes may be used at the convenience of the analyst. If compound purity is assayed at 97% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

      (2) Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at -10°C to -20°C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Stock standard solutions must be replaced after six months, or sooner, if comparison with quality control check samples indicates a problem. Standards prepared from gases or reactive compounds such as styrene must be replace after two months, or sooner, if comparison with quality control check samples indicates a problem.

   b. Prepare a working standard mixture of the three compounds in methylene chloride. The concentration must be such that the volume injected equals 50 ng of each compound. The storage and stability requirements are the same as specified in paragraph a(2) above.

GC Calibration

At the beginning of each 12-hour shift, inject the GC calibration standard. The following criteria must be met:

1. The GC must be standardized for half scale response from 50 ng of phenanthrene.

2. The GC must adequately separate phenol from the solvent front.

3. A minimum of quarter scale response for 50 ng of di-n-octylphthalate must be exhibited.

GC/FID Screening

1. Suggested GC operating conditions are as follows:

   - Initial Column Temperature Hold - 50°C for 4 minutes.
   - Column Temperature Program - 50-280°C at 8 degrees/min.
   - Final Column Temperature Hold - 280°C for 8 minutes.
   - Injector - Grob-type, splitless.
   - Sample Volume - 1-2 ul.
   - Carrier Gas - Helium at 30 ml/sec.

2. Inject the GC calibration standard and ensure the criteria specified in GC Calibration are met before injecting samples. Estimate the response for 10 ng of phenanthrene.

3. Inject the appropriate extracts, including blanks.

Interpretation of Chromatogram

1. Water

   a. If no sample peaks are detected, or all are less than full scale deflection, the undiluted extract is analyzed on GC/MS.

   b. If any sample peaks are greater than full scale deflection, calculate the dilution necessary to reduce the major peaks to between half and full scale deflection. Use this dilution factor to dilute the extract for GC/MS analysis.

2. Soil/Sediment

   a. If no sample peaks from the extract (from low or medium level preparation) are detected, or all are less than 10% full scale deflection, the sample must be prepared by the low level protocol specified above.

   b. Peaks are detected at greater than 10% full scale deflection and less than or equal to full scale deflection.

      (1) If the screen is from the medium level extract, proceed with GC/MS analysis of this extract with appropriate dilution if necessary.

      (2) If screen is from the low level extract, discard extract and prepare sample by medium level method for GC/MS analysis.

c. Peaks are detected at greater than full scale deflection.

(1) If the screen is from the medium level preparation, calculate the dilution necessary to reduce the major peaks to between half and full scale deflection. Use this dilution factor to dilute the extract. This dilution is analyzed by GC/MS for extractable organics.

(2) If the screen is from the low level preparation, discard the extract and prepare a sample by the medium level method for GC/MS analysis.

GC/MS Analysis

1. Use the information from paragraph of Interpretation of Chromatograms to perform the GC/MS analysis, beginning GC/MS Analysis of Semi-volatiles.

2. The information from screening may be useful also in processing sample extracts through GPC cleanup.

NOTE: The choice of screening sample extracts before or after GPC cleanup is left to the laboratory.

GC/MS ANALYSIS OF SEMI-VOLATILES

This method is to be used for the GC/MS analysis of semi-volatiles and for confirmation of pesticides/Aroclors identified by GC/EC, if concentrations permit.

Apparatus and Materials

Gas Chromatograph/Mass Spectrometer System.

1. Gas chromatograph - an analytical system complete with a temperature programmable gas chromatograph suitable for splitless injection and all required accessories including syringes, analytical columns and gases.

2. Column - 30 m x 0.25 mm ID (or 0.32 mm) bonded-phase silicone coated fused silica capillary column (J&W Scientific DB-5 or equivalent). A film thickness of 1.0 micron is recommended because of its larger capacity. A film thickness of 0.25 micron may be used. The description of GC column is presented in SDG narrative.

3. Mass Spectrometer - capable of scanning from 35 to 500 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the instrument performance criteria in Table 1 when 50 ng of decafluorotriphenylphosphine (DFTFF) is injected through the GC inlet. The instrument conditions required for the acquisition of the DFTPP mass spectrum are given in paragraph of Instrument Operating Conditions below.

4. GC/MS interface - any gas chromatograph to mass spectrometer interface that gives acceptable calibration points, at 50 ng or less per injection, for each of the parameters of interest, and achieves all acceptable performance criteria may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be de-activated by silanizing with dichlorodimethylsilane.

5. Data system - computer system must be interfaced to the mass spectrometer that allow the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. This type of plot is defines as an Extracted Ion Current Profile (EICP). Software must be available that allow integrating the abundance in any EICP between specified time or scan number limits. Also for the non-target compounds software must be available that allow comparing sample spectra against reference spectra. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

6. Syringes - 2 ul and 10 ul volumes.

Reagents

1. Internal standards - 1,4 Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, Perylene-d_{12}.

   An internal standard solution can be prepared by dissolving 100 mg of each compound in 50 ml of methylene chloride. It may be necessary to use 5 to 10 percent benzene or toluene in this solution and a few minutes of ultrasonic mixing in order to dissolve all the constituents. The resulting solution will contain each standard at a concentration of 2000 ng/ul. A 10 ul portion of this solution should be added to each 1 ml of sample extract. This will result in 40 ng of each internal standard in the 2 ul volume of extract injected into the GC/MS.

2. Prepare calibration standards at a minimum of five concentration levels (20, 50, 80, 120, and 160 total ng per 2 ul). Each calibration standard should contain each compound for interest and each surrogate. Eight compounds 2,4-Dinitrophenol, 2,4,5-Trichlorophenol, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, 4-Nitrophenol, 4,6-Dinitro-2-methylphenol and Pentachlorophenol will require only a four-point initial calibration at 50, 80, 120 and 160 total ng, since detection at less than 50 ng per injection is difficult. Great care must be taken to maintain the integrity of all standard solutions. Store all standard solutions at -10°C to -20°C in screw-cap amber bottles with Teflon liners. Fresh standards should be prepared every twelve months at minimum. The continuing calibration standard (50 ng) should be prepared weekly and stored at 4°C (±2°C).

3. Instrument performance check solution - prepare a solution of decafluorotripehnylphosphine (DFTPP), such that a 2-ul injection will contain 50 ng of DFTPP. The DFTPP also must be included in the calibration standards at this level.

Instrument Operating Conditions

1. Gas Chromatograph

   The following are the recommended GC analytical conditions:

Initial column Temperature Hold	-	40°C for 4 minutes.
Column Temperature Program	-	40-270°C at 10 degrees/min.
Final Column Temperature Hold	-	270°C for 10 minutes

Injector Temperature	-	250-300°C
Transfer Line Temperature	-	250-300°C
Source Temperature	-	according to manufacturer's specifications
Injector	-	Grob-type, splitless
Sample Volume	-	2 ul
Carrier Gas	-	Helium at 30 ml/sec.

Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks, matrix spikes, and matrix spike duplicates.

2. Mass Spectrometer

The following are the required mass spectrometer analytical conditions:

Electron Energy	- 70 volts (nominal)
Mass Range	- 35 to 500 amu
Scan Time	- not to exceed 1 second per scan

3. The GC/MS system must be tuned to meet the manufacturer's specifications, using a suitable calibrant such as FC-43 or perfluorokerosene (PFK). The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution (paragraph 3 of Reagents).

   a. Prior to the analysis of any samples, blanks or calibration standards, the laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing decafluorotriphenylphosphine (DFTPP).

   b. The analysis of the instrument performance check solution may be performed as follows:

      - As an injection of up to 50 ng of DFTPP into the GC/MS.
      - By adding 50 ng of DFTPP to the calibration standards (paragraph 2 of Reagents) and analyzing the calibration standard.

   c. The analysis of the instrument performance check solution must meet the ion abundance criteria given below.

TABLE 1
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA FOR QUADRAPOLE MASS SPECTROMETERS

Mass	Ion Abundance Criteria
51	30.0 - 80.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	Present
70	Less than 2.0 percent of mass 69
127	25.0 - 75.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	Greater than 0.75 percent of mass 198
441	Present but less than mass 443
442	40.0 - 110.0 percent of mass 198
443	15.0 - 24.0 percent of mass 442

e. The abundance criteria listed above must be met for a 50 ng injection of DFTPP. The mass spectrum of DFTPP must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required and must be accomplished using a single scan prior to the elution of DFTPP. Note: All subsequent standards, samples, MS/MSD and blanks associated with a DFTPP analysis must use identical mass spectrometer instrument conditions.

f. The criteria above are based on adherence to the acquisition specifications identified in paragraph e above. The criteria are based on performance characteristics of instruments currently utilized in routine support of Program activities. These specifications, in conjunction with relative response factor criteria for 54 target compounds (see Table 2), are designed to control and monitor instrument performance associated with the requirements of this Statement of Work.

g. The instrument performance check solution must be analyzed once at the beginning of each 12-hour period during which samples or standards ar analyzed.

The twelve (12) hour time period for a GC/MS system instrument performance check and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after twelve (12) hours has elapsed according to the system clock.

Calibration

1. Prior to the analysis of samples and required blanks, and after the instrument performance check solution criteria have been met, each GC/MS system must be calibrated at a minimum of five concentrations to determine instrument sensitivity and linearity of GC/MS response for the semi-volatile target compounds.

2. The internal standards are added to all calibration standards and all sample extracts (including blanks, matrix spikes and matrix spike duplicates) just prior to analysis by GC/MS. A 10 ul aliquot of the internal standard solution should be added to a 1 ml aliquot of calibration standards. The internal standards specified in paragraph 1 of Reagents should permit most of the semi-volatile target compounds to have relative retention times of 0.80 to 1.20, using the assignments of internal standards to target compounds given in Table 2.

3. The quantitation ions for each internal standard are given in Table 3. Use the primary ion listed in Table 3 for quantitation, unless interferences are present. If interferences prevent the use of the primary ion for a given internal standard, use the secondary ion(s) listed in Table 3.

4. Prepare calibration standards at a minimum of five concentration levels for each target compound and surrogate, as specified in paragraph 2 of Reagents. Analyze 2 ul of each calibration standard and tabulate the area of the primary characteristic ion against concentration for each compound including the surrogate compounds. A 2 ul injection is required. Calculate relative response factors (RRF) for each compound using Equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured (See Table 4)

A_{is} = Area of the characteristic ion (CICP) for the specific internal standard (See Table 3)

C_{is} = Concentration of the internal standard

C_x = Concentration of the compound to be measured

5. The average relative response factor (RRF) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of RRF values over the working range of the curve.

$$\%RSD = \frac{\text{Standard deviation}}{\text{mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1} \right|^{1/2}$$

Where,

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

TABLE 2

SEMI-VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}	Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
Phenol	Nitrobenzene	Hexachlorocyclo-pentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl-phthalate
bis (2-Chloroethyl) ether	Isophorone	2,4,6-Trichloro-phenol	N-nitrosodi-phenylamine	Butylbenzyl phthalate	Benzo(b)fluor-anthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichloro-phenol	4-Bromophenyl phenyl ether	3,3'-Dichloro-benzidine	Benzo(k)fluor-anthane
1,3-Dichlorobenzene	2,4-Dimethyl-phenol	2-Chloronaphthalene	Hexachloro-benzene	Benzo(a)-anthracene	Benzo(a)pyrene
1,4-Dichlorobenzene	bis(2--Chloro-ethoxy)methane	2-Nitroaniline	Pentachloro-phenol	bis(2-Ethyl-hexyl)phthalate	Indeno(1,2,3-cd) pyrene
1,2-Dichlorobenzene	2,4-Dichloro-phenol	Dimethyl Phthalate	Phenanthrene	Chrysene	Dibenz(a,h)-anthracene
2-Methylphenol	1,2,4-Trichloro-benzene	Acenaphthylene	Carbazole	Terphenyl-d_{14} (surr)	Benzo(g,h,i)-perylene
2,2'-oxybis (1-Chloropropane)	Naphthalene	3-Nitroaniline	Anthracene		
4-Methylphenol	4-Chloroaniline	Acenaphthene	Di-n-butyl-phthalate		
N-Nitroso-Di-n propylamine	Hexachloro-butadiene	2,4-Dinitrophenol			
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol			
2-Fluorophenol (surr)	2-Methylnaphth-alene	Dibenzofuran			
Phenol-d_5 (surr)	Nitrobenzene-d_5 (surr)	2,4-Dinitrotoluene			
2-Chlorobenzene-d_4 (surr)		2,6-Dinitrotoluene			
1,2-Dichlorobenzene-d_4 (surr)		Diethyl phthalate			
		4-Chlorophenyl phenyl ether			
		Fluorene			
		4-Nitroaniline			
		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromo-phenol (surr)			

surr = surrogate compound

TABLE 3

CHARACTERISTIC IONS FOR INTERNAL STANDARDS FOR SEMI-VOLATILE COMPOUNDS

INTERNAL STANDARDS	Primary Ion	Secondary Ions
1,4-Dichlorobenzene-d_4	152	115
Naphthanlene-d_8	136	68
Acenapthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

TABLE 4
CHARACTERISTIC IONS FOR SEMI-VOLATILE TARGET COMPOUNDS AND SURROGATES

Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
2,2'-oxybis(1-Chloropropane)	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(2-Chloroethyoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	194, 164
Acenaphthylene	152	151 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141

TABLE 4 (continued)
CHARACTERISTIC IONS FOR SEMI-VOLATILE TARGET COMPOUNDS AND SURROGATES

Parameter	Primary Ion	Secondary Ion(s)
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Carbazole	167	166, 139
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-Octyl phthalate	149	---
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,H)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
SURROGATES		
Phenol-d_5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
Nitrobenzene-d_5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl-d_{14}	244	122, 212
2-Chlorophenol-d_4	132	68, 134
1,2-Dichlorobenzene-d_4	152	115, 150

6. Response factor criteria have been established for the calibration of the semi-volatile target compounds and semi-volatile surrogate compounds.

   a. The response factors of the compounds listed in Table 5 must meet the minimum RRF criteria at each concentration level and maximum %RSD criteria for the initial calibration, with allowance made for up to four semi-volatile target and surrogate compounds. However, the RRFs for those four compounds must be less than or equal to 40.0% for the initial calibration to be acceptable.

TABLE 5
RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF SEMI-VOLATILE TARGET COMPOUNDS

Semi-volatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Phenol	0.800	20.5	25.0
bis(-2-Chloroethyl)ether	0.700	20.5	25.0
2-Chlorophenol	0.800	20.5	25.0
1,3-Dichlorobenzene	0.600	20.5	25.0
1,4-Dichlorobenzene	0.500	20.5	25.0
1,2-Dichlorobenzene	0.400	20.5	25.0
2-Methylphenol	0.700	20.5	25.0
4-Methylphenol	0.600	20.5	25.0
N-Nitroso-Di-propylamine	0.500	20.5	25.0
Hexachloroethane	0.300	20.5	25.0
Nitrobenzene	0.200	20.5	25.0
Isophorone	0.400	20.5	25.0
2-Nitrophenol	0.100	20.5	25.0
2,4-Dimethylphenol	0.200	20.5	25.0
bis(-2-Chloroethoxy)methane	0.300	20.5	25.0
2,4-Dichlorophenol	0.200	20.5	25.0
1,2,4-Trichlorobenzene	0.200	20.5	25.0
Naphthalene	0.700	20.5	25.0
4-Chloro-3-methylphenol	0.200	20.5	25.0
2-Methylnaphthalene	0.400	20.5	25.0
2,4,6-Trichlorophenol	0.200	20.5	25.0
2,4,5-Trichlorophenol	0.200	20.5	25.0
2-Chloronaphthalene	0.800	20.5	25.0
Acenaphthylene	1.300	20.5	25.0
2,6-Dinitrotoluene	0.200	20.5	25.0
Acenaphthene	0.800	20.5	25.0
Dibenzofuran	0.800	20.5	25.0
2,4-Dinitrotoluene	0.200	20.5	25.0
4-Chlorophenyl-phenylether	0.400	20.5	25.0
Fluorene	0.900	20.5	25.0
4-Bromophenyl-phenylether	0.100	20.5	25.0
Hexachlorobenzene	0.100	20.5	25.0
Pentachlorophenol	0.050	20.5	25.0
Phenanthrene	0.700	20.5	25.0
Anthracene	0.700	20.5	25.0
Fluoranthene	0.600	20.5	25.0
Pyrene	0.600	20.5	25.0
Benzo(a)anthracene	0.800	20.5	25.0
Chrysene	0.700	20.5	25.0
Benzo(b)fluoranthene	0.700	20.5	25.0
Benzo(k)fluoranthene	0.700	20.5	25.0
Benzo(a)pyrene	0.700	20.5	25.0
Indeno(1,2,3-cd)pyrene	0.500	20.5	25.0

TABLE 5 (Continued)
RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF SEMI-VOLATILE TARGET COMPOUNDS

Semi-volatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Dibenzo(a,h)anthrance	0.400	20.5	25.0
Benzo(g,h,i)perylene	0.500	20.5	25.0
Nitrobenzene-d_5	0.200	20.5	25.0
2-Fluorobiphenyl	0.700	20.5	25.0
Terphenyl-d_{14}	0.500	20.5	25.0
Phenol-d_5	0.800	20.5	25.0
2-Fluorophenol	0.600	20.5	25.0
2-Chlorophenol-d_4	0.800	20.5	25.0
1,2-Dichlorobenzene-d_4	0.400	20.5	25.0

b. The following compounds have no Maximum %RSD, or Maximum %Difference criteria; however, these compounds must meet a minimum RRF criterion of 0.010:

2,2'-oxybis(1-Chloropropane)	4-Nitroaniline
4-Chloroaniline	4,6-Dinitro-2-methylphenol
Hexachlorobutadiene	N-Nitrosodiphenylamine
Hexachlorocyclopentadiene	Di-n-butylphthalate
2-Nitroaniline	Butylbenzylphthalate
Dimethylphthalate	3,3'-Dichlorobenzidine
3-Nitroaniline	bis(2-Ethylhexyl)phthalate
2,4-Dinitrophenol	Di-n-octylphthalate
4-Nitrophenol	2,4,6-Tribromophenol
Diethylphthalate	Carbazole

7. A check of the calibration curve must be performed once every 12 hours. Check the relative response factors of those compounds for which RRF values have been established. If these criteria are met, the relative response factors for all compounds are calculated and reported. A percent difference of the daily relative response factor (12-hour) compared to the average relative response factor from the initial curve is calculated. Calculate the percent difference for each compound and compare with the maximum percent difference criteria listed above. For negative percent difference values, the value must be greater than or equal to -25.0%, but less than 0%.

As with the initial calibration up to four semi-volatile target compounds in Table 5 may fail to meet the minimum RRF or maximum %D criteria, but the RRFs of those four compounds must be greater than or equal to 0.010, and the percent differences must be less than or equal to 40.0% for the continuing calibration to be acceptable.

8. Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 0.50 minutes (30 seconds) from the latest daily (12-hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. The extracted iron current profile (EICP) of the internal standards must be monitored and evaluated for each standard. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction, and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is necessary.

9. Each GC/MS system must be calibrated upon award of the contract, whenever the laboratory takes corrective action which may change or affect the initial calibration criteria (i.e., ion source cleaning or repair, column removal or replacement, etc.), or if the continuing calibration acceptance criteria have not been met.

10. If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. It is not necessary to analyze a continuing calibration standard, if the initial calibration standard that is the same concentration as the continuing calibration standard meets the continuing calibration acceptance criteria. Quantify all sample results against the initial calibration standard that is the same concentration as the continuing calibration stand (50ng/2ul).

11. If time does NOT remain in the 12-hour period beginning with the injection of the instrument performance check solution, a new injection of the instrument performance check solution must be made. The DFTPP may be included in the continuing calibration standard.

12. If the injection of the instrument performance check solution meets the criteria in Table 1, calculate the response factors for the continuing calibration standard and the percent difference of the response factors from the mean response factors in the initial calibration.

13. The response factors from the continuing calibration standard must meet the criteria in Table 5 prior to the analysis of any blanks or samples.

<u>Sample Analysis</u>

1. Sample extracts may be analyzed only after the GC/MS system has met the instrument performance check, initial calibration, and continuing calibration requirements above. The same instrument conditions must be employed for the analysis of samples as were used for calibration.

2. Internal standard solution is added to each sample extract. Add 10 ul of internal standard solution to each accurately measured 1.0 ml of water sample extract. For soil samples and water samples subjected to GPC, add 5 ul of internal standard solution to each accurately measured 0.5 ml of sample extract. This will result in a concentration of 20 ng/ul of each internal standard.

3. Make any extract dilution indicated by characterization prior to the addition of internal standards. If any further dilutions of water or soil/sediment extracts are made, additional internal standards must be added to maintain the required 40 ng (20 ng/ul) of each internal standard in the extract volume.

4. Inject 2 ul of the sample extract into the GC/MS. This 2 ul volume must contain 40 ng of each internal standard.

Qualitative Analysis

1. The compounds listed in the Target Compound List (TCL), Table 9, shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications:

   - Elution of the sample component at the GC relative retention time as the standard component.
   - Correspondence of the sample component and standard component mass spectra.

   a. For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.06 RRT units of the RRT of the standard component. For samples analyzed during the same 12-hour time period as the initial calibration standards, compare the sample retention times to those from the 50 ng calibration standard. For reference, the standard must be run on the same shift as the sample. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

   b. For comparison of standard and sample component mass spectra, mass spectra obtained on the contractor's GC/MS are required. Once obtained, these standard spectra may be used for identification purposes, only if the contractor's GC/MS meets the DFTPP daily instrument performance requirements. These standard spectra may be obtained from the run used to obtain reference RRTs.

   c. The requirements for qualitative verification by comparison of mass spectra are as follows:

      (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

      (2) The relative intensities of ions specified in paragraph (1) above must agree within ±20% between the standard and sample spectra.

      (3) Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. In Task III, the verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra.

   d. If a compound cannot be verified by all of the criteria in paragraph c above, but in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that identification and proceed with quantification in paragraph of Quantitation below.

2. A library search shall be executed for non-target sample components for the purpose of tentative identification. For this purpose the most recent release of the NIST/EPA/MSDC mass spectral library shall be used.

a. Up to 20 non-surrogate organic compounds of greatest apparent concentration not listed in Table 9 for the semi-volatile fraction shall be identified tentatively via a forward search of the NIST/EPA/MSDC mass spectral library. Substances with responses less than 10% of the nearest internal standard are not required to be searched in this fashion. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

Peaks that are suspected as aldol-condensation reaction products (i.e., 4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) shall be searched and reported but not counted as part of the 20 most intense non-target semi-volatile compounds.

b. Guidelines for making tentative identification.

(1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ±20%.

(3) Molecular ions present in reference spectrum should be present in sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds.

c. If the technical judgement of the mass interpretation spectral specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound).

Quantitation

1. Target components identified shall be quantified by the internal standard method. The internal standard used shall be the one nearest the retention time to that of a given analyte (see Table 2). The EICP area of characteristic ions of analytes listed in Table 4 are used for quantitation. In all instances where the data system report has been edited, or where manual integration of quantitation has been performed, the GC/MS operator must identify such edits or manual procedures by initializing and dating the changes made to the report.

Internal standard responses and retention times in all samples must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 0.50 minutes (30 seconds) from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. For samples analyzed during the same 12-hour time period as the initial calibration standards, compare the internal standard responses and retention times to those of the 50 ng calibration standard. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate. The criteria are described in detail in the instructions of Form VIII, Internal Standard Area Summary. If the EICP are for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate.

If the analysis of a subsequent sample or standard indicates that the system is functioning properly, then corrections may not be required. The samples or standard with EICP areas outside the limits must be re-analyzed, and treated according to paragraphs a and b below. If corrections ar made, then the laboratory must demonstrate that the mass spectrometric system is functioning properly. This must be accomplished by the analysis of a standard or sample that does meet the EICP criteria. After corrections are made, the re-analysis of samples analyzed while the system was malfunctioning is required.

a. If after re-analysis, the EICP areas for all internal standards are inside the contract limits (-50% to +100%), then the problem with the first analysis is considered to have been within control of the laboratory. Therefore, submit only data from the analysis with EICPs within the contract limits. This is considered the initial analysis and must be reported as such on all data deliverables.

b. If the re-analysis of the sample does not solve the problem, i.e., the EICP areas are outside the contract limits for both analyses, then submit the EICP data and sample data from both analyses. Distinguish between the initial analysis and the re-analysis on all data deliverables.

c. Do not re-analyze MS/MSD samples that do not meet the EICP area limits.

2. The relative response factor (RRF) from the daily standard analysis is used to calculate the concentration in the sample. For samples analyzed during the same 12-hour time period as the initial calibration standards, use the RRF values from the 40 ng calibration standard. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reasons. The area of a secondary ion cannot be substituted for the area of a primary ion unless a relative response factor is calculated using the secondary ion.

When target compounds are below contract required quantitation limits (CRQL) but the spectrum meets the identification criteria, report the concentration with a "J".

Calculate the concentration in the sample using the relative response factor (RRF) as determined in paragraph 4 of Calibration and the following equation:

Water

$$\text{Concentration ug/l} = \frac{(A_x)(I_s)(V_t)Df)}{(A_{is})(RRF)(V_o)(V_i)}$$

Where,

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

I_s = Amount of internal standard injected in nanograms (ng).

V_o = Volume of water extracted in milliliters (ml)

V_i = Volume of Extract injected in microliters (ul).

V_t = Volume of the concentrated extract in microliters (ul)

Df = Dilution Factor. The dilution factor for analysis of water samples for semi-volatiles by this method is defined as follows:

$$\frac{\text{ul most conc. extract used to make dilution + ul clean solvent}}{\text{ul most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0

Soil/Sediment

$$\text{Concentration (Dry weight basis)} \quad \text{ug/kg} = \frac{(A_x)(I_s)(V_t)(Df)(2.0)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

Where,

A_x,I_s,A_{is} are given for water, above.

V_t = Volume of the concentrated extract in microliters (ul).

V_i = Volume of extract injected in microliters (ul)

$D = \frac{100 - \%\ \text{moisture}}{100}$

W_s = Weight of sample extracted in grams (g).

Df = Dilution Factor. The dilution factor for analysis of soil sampled for semi-volatiles by this method is defined as follows:

$$\frac{\text{ul most conc. extract used to make dilution + ml clean solvent}}{\text{ul most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

The factor of 2.0 in the numerator is used to account for the amount of extract that is not recovered from the mandatory use of GPC cleanup. Concentrating the extract collected after GPC to 0.5 ml, rather than 1.0 ml for water samples not subjected to GPC, maintains the sensitivity of the soil method comparable to that of the water method, but correction of the numerical result is still required.

3. An estimated concentration for non-target components tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard free of interferences shall be used. The formula for calculating concentrations is the same as in paragraph 2 above. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A relative response factor (RRF) of one (1) is to be assumed. The resulting concentration shall be qualified as "J" (estimated due to lack of a compound-specific response factor), and "N" (presumptive evidence presence), indicating the quantitative and qualitative uncertainties associated with this non-target component. An estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

4. If the on-column concentration of any compound in any sample exceeds the initial calibration range, that sample extract must be diluted, the internal standard concentration re-adjusted and the sample extract re-analyzed. Guidance in performing dilutions and exceptions to this requirement are given below.

   a. Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

   b. The dilution factor chosen should keep the response of the largest peak for a target compound in the upper half of the initial calibration range of the instrument.

   c. Do not submit data for more than two analyses, i.e., the original sample extract and one dilution, or if the semi-volatile screening procedure was employed, from the most concentrated dilution analyzed and one further dilution.

   d. Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within the calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis, and note the problem in the non-conformance.

5. Calculate surrogate standard recovery on all samples, blanks and spikes. Determine if recovery is within limits (see Table 6) and report on appropriate form.

   a. Calculate the concentrations of the surrogate compounds using the same equations as used for the target compounds. Calculate the recovery of each surrogate as follows:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

   b. Determine if the sample surrogate recovery meets specifications as follows:

      - The eight semi-volatile surrogates can be divided into three groups: base/neutral compounds (Nitrobenzene-d_5, 2-Fluorobiphenyl and Terpheyl-d_{14}); acid compounds (Phenol-d_5, 2-Fluorophenol and 2,4,6-Tribromophenol); and compounds with advisory QC limits (2-Chlorophenol-d_4 and 1-2-Dichlorobenzene-d_4).

      - If a single surrogate recovery from any group is not within the contract windows, the sample does not require re-analysis or re-extraction.

      - If a single surrogate recovery from the base/neutral group and a single surrogate recovery from the acid group are not within the contract windows the sample does not require re-analysis or re-extraction.

      - Do not re-analyze or re-extract if only surrogates with advisory QC limits are not within the contract windows.

   c. If the sample surrogate recovery does not meet specifications (i.e., if two base/neutral or two acid surrogates are out of limits or if recovery of any one base/neutral or acid surrogate is below 10%), the following are required:

      - Check to be sure that there are no errors in calculations, surrogate solutions and internal standards. Also check instrument performance.

- Re-analyze the sample if none of the above reveal a problem.
- If surrogate recoveries in a blank do not meet specifications the blank may be analyzed alone.
- Do not re-analyze dilutions if surrogate recoveries are outside the limits.
- Never re-analyze the matrix spike or matrix spike duplicate (MS/MSD), even if surrogate recoveries are outside the limits.
- If the sample associated with the matrix spike and matrix spike duplicate does not meet specifications, it should be re-analyzed only if the MS/MSD surrogate recoveries are within the limits. If the sample and associated MS/MSD show the same pattern (i.e., outside the limits), then the sample does not require re-analysis and a re-analysis must not be submitted.

  Document in the non-conformance the similarity in surrogate recoveries.

d. If the re-analysis of the sample solves the problem, then the problem was within the laboratory's control. Therefore, submit only data from the analysis with surrogate spike recoveries within the contract windows. This shall be considered the initial analysis and shall be reported as such on all data deliverables.

e. If none of the steps in paragraph c or d above solves the problem, then, except as noted below, re-extract and re-analyze the sample. If the re-extraction and re-analysis of the sample solves the problem then the problem was within the laboratory's control. Therefore, submit only data from the analysis with surrogate recoveries within the contract windows. This shall be considered the initial analysis and shall be reported as such on all data deliverables.

- If surrogate recoveries in a blank do not meet specifications even after re-analysis, all of the samples associated with that blank must be re-extracted along with the blank. The blank is intended to detect contamination in samples processed at the same time.
- Do not re-extract diluted samples if surrogate recoveries are outside the limits.
- Never re-extract the matrix spike or matrix spike duplicate (MS/MSD), even if surrogate recoveries are outside the limits.
- If the sample associated with the matrix spike and matrix spike duplicate does not meet specifications after re-analysis, it should be re-extracted only if the re-analysis surrogate recoveries are not within the limits and MS/MSD surrogate recoveries are within the limits. If the sample and associated MS/MSD show the same pattern (i.e., outside the limits), then the samples does not require re-analysis and a re-analysis must not be submitted.

  Document in the non-conformance the similarity in surrogate recoveries.

f. If the re-extraction and re-analysis of the sample does not solve the problem then submit the surrogate recovery data and sample analysis data from the initial analysis of both sample extracts. Distinguish between the initial analysis and the analysis of the re-extracted sample on all data deliverables.

TABLE 6
SURROGATE RECOVERY LIMITS

Compound	%Recovery Water	%Recovery Soil
Nitrobenzene-d_5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
Terphenyl-d_{14}	33-141	18-137
Phenol-d_5	10-110	24-113
2-Fluorophenol	21-110	25-121
2,4,6-Tribromophenol	10-123	19-122
2-Chlorophenol-d_4	33-110	20-130 (advisory)
1,2-Dichlorobenzene-d_4	16-110	20-130 (advisory)

6. A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix, for the following, whichever is most frequent.

   - Each Case of field samples received, OR
   - Each 20 field samples in a Case, OR
   - Each group of field samples of a similar concentration level (soils only), OR
   - Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which field samples in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group).

   Calculate the recovery of each matrix spike compound in the matrix spike and matrix spike duplicate report on appropriate form.

   a. Calculate the concentrations of the matrix spike compounds using the same equations as used for target compounds. Calculate the recovery of each matrix spike compound as follows:

   $$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

   Where,

   SSR = Spike sample result
   SR = Sample result
   SA = Spike added

   b. Calculate the relative percent difference of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

   $$RPD = \frac{|MSR - MSDR|}{(1/2)(MSR + MSDR)} \times 100$$

Where,

RPD = Relative Percent Difference

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

c. The limits for matrix spike compound recovery and RPD are given in Table 7. As these limits are only advisory, no further action by the laboratory is required, however, frequent failures to meet the limits for recovery or RPD warrant investigation by the laboratory and may result in questions from the Agency.

TABLE 7
MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	%Recovery Water	RPD Water	%Recovery Soil	RPD Soil
Phenol	12-110	42	26- 90	35
2-Chlorophenol	27-123	40	25-102	50
1,4-Dichlorobenzene	36- 97	28	28-104	27
N-Nitroso-di-n-propylamine	41-116	38	41-126	38
1,2,4-Trichlorobenzene	39- 98	28	38-107	23
4-Chloro-3-methylphenol	23- 97	42	26-103	33
Acenaphthene	46-118	31	31-137	19
4-Nitrophenol	10- 80	50	11-114	50
2,4-Dinitrotoluene	24- 96	38	28- 89	47
Pentachlorophenol	9-103	50	17-109	47
Pyrene	26-127	31	35-142	36

7. Method blank analysis must be performed once for the following, on each GC/MS system used to analyze samples, whichever is most frequent:

- Each Case, OR
- Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which samples in a Case are received (said period beginning with the receipt of the first sample in that Sample Delivery Group), OR
- Each 20 samples in a Case, including matrix spikes and re-analyses, that are of similar matrix (water or soil) or similar concentration (soil only), OR
- Whenever samples are extracted by the same procedure (continuous liquid-liquid extraction or sonication).

Determine the concentrations of any target compounds detected in the semi-volatile method blank, using the equations in the semi-volatile method blank, using the equations in paragraph 2 of Quantitation. The method blank must contain less than or equal to the Contract Required Quantitation Limit (CRQL) of the semi-volatile target compounds in Table 9, Except the phthalate esters, which must be less than or equal to five times (5x) the CRQL. For soil/sediment method blanks, CRQL value must be adjusted for percent moisture.

If a laboratory method blank exceeds these criteria, the laboratory must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) MUST be re-extracted and re-analyzed at no additional cost to the Agency. The Laboratory Manager, or his designee, must address problems and solutions in the non-conformance.

TABLE 8

CHARACTERISTIC IONS FOR PESTICIDES/AROCLORS

Parameter	Primary Ion	Secondary Ion(s)
alpha-BHC	183	181, 109
beta-BHC	181	183, 109
delta-BHC	183	181, 109
gamma-BHC (Lindane)	183	181, 109
Heptachlor	100	272, 274
Aldrin	66	263, 220
Heptachlor epoxide	353	355, 351
Endosulfan I	195	339, 341
Dieldrin	79	263, 279
4,4'-DDE	246	248, 176
Endrin	263	82, 81
Endrin ketone	317	67, 319
Endrin aldehyde	67	250, 345
Endosulfan II	337	339, 341
4,4'-DDD	235	237, 165
Endosulfan sulfate	272	387, 422
4,4'-DDT	235	237, 165
Methoxychlor	227	228
Chlordane (alpha and/or gamma)	373	375, 377
Toxaphene	159	231, 233
Aroclor-1016	222	260, 292
Aroclor-1221	190	222, 260
Aroclor-1232	190	222, 260
Aroclor-1242	222	256, 292
Aroclor-1248	292	362, 326
Aroclor-1254	292	362, 326
Aroclor-1260	360	362, 394

SEMI-VOLATILE QA/QC REQUIREMENTS

The purpose of this section is to outline the minimum quality control (QC) operations necessary to satisfy the analytical requirements associated with the determination of the semi-volatile organic target compound listed in Table 9 using the procedures described above for water and soil/sediment samples.

These requirements include the following:

- GC/MS Mass Calibration and Ion Abundance Patterns
- GC/MS Initial and Continuing Calibration
- Stability of Internal Standard Responses and Retention Times
- Method Blank Analysis
- Surrogate Recoveries
- Matrix Spike and Matrix Spike Duplicate Analyses

GC/MS Mass Calibration and Ion Abundance Patterns

Prior to initiating any data collection activities involving samples, blanks, or standards, it is necessary to establish that a given GC/MS system meets the instrument performance criteria specified in paragraph of Instrument Operating Conditions. The purpose of this instrument performance check is to assure correct mass calibration, mass resolution, and mass transmission. This is accomplished through the analysis of Decafluorotriphenyl phosphine (DFTPP).

1. The required frequency of DFTPP analysis (once every 12 hours on each GC/MS system) is described in detail in paragraph 3g of Instrument Operating Conditions.

2. The key ions produced during the analysis of DFTPP and their respective ion abundance criteria are given in Table 1, paragraph 3c of Instrument Operating Conditions.

3. The documentation includes Form V SV, and a mass listing and bar graph spectrum of each DFTPP analysis.

GC/MS Initial Calibration for Target Compounds and Surrogates

Prior to the analysis of samples and required blanks, and after instrument performance criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing target compound and surrogate standards.

1. The levels of the initial calibration standards for semi-volatile target compounds and surrogates are 20, 50, 80, 120 and 160 ng, in a 2 ul injection volume, as described in paragraph of Reagents.

2. The standards are to be analyzed according to the procedures given in paragraph of Calibration, and at the frequency given in that paragraph.

3. The relative response factors (RRFs) are determined according to the procedures in paragraph of Calibration, using the assignment of internal standard to target compounds and surrogates given in paragraph of Calibration and Tables 3 and 4.

4. The calibration of the GC/MS is evaluated on the basis of the magnitude and stability of the relative response factors of each target compound and surrogate. The minimum RRF of each compound at each concentration level in the initial calibration and the percent relative standard deviation (%RSD) across all five points must meet the criteria given in paragraph of Calibration and Table 5. Allowance is made for any four semi-volatile compounds that fail to meet these criteria. The minimum RRFs of those four compounds must be greater than or equal to 40.0% for the initial calibration to be acceptable.

5. The documentation includes Form VI SV, a GC/MS data system printout for the analysis of each semi-volatile calibration standard.

GC/MS Continuing Calibration for Target Compounds and Surrogates

Once the GC/MS system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC/MS system.

1. The level of the continuing calibration standard for semi-volatile target compounds and surrogates is 50 ng, in a 2 ul injection volume, as described in paragraph of Reagents.

2. The standard is to be analyzed according to the procedures given in paragraph of Calibration, and at the frequency given in that paragraph.

3. The continuing calibration of the GC/MS system is evaluated on the basis of the magnitude of the relative response factors and the percent difference between the average RRF of each compound from the initial calibration and the RRF of that compound in the continuing calibration standard. The minimum RRF of each compound in the continuing calibration and the percent difference must meet the criteria given in paragraphs of Calibration and Table 5. Allowance is made for any four semi-volatile compounds that fail to meet these criteria. The minimum RRFs of those four compounds must be greater than equal to 0.010, and the %D must be less than or equal to 40.0% for the continuing calibration to be acceptable.

4. The documentation includes From VII SV, a GC/MS data system printout for the analysis of the semi-volatile calibration standard.

Internal Standard Responses and Retention Times

The response of each of the internal standards in all calibration standards, samples, and blanks is crucial to the provision of reliable analytical results because the quantitative determination of semi-volatile compounds by these procedures is based on the use of internal standards added immediately prior to analysis.

1. The specific compounds used as internal standards are given in paragraph of Reagents. The amount of each internal standard in the injection volume (2 ul) of the sample extract analyzed by GC/MS must be 40 ng (20 ug/ul).

2. The retention time and the extracted ion current profile (EICP) of each internal standard must be monitored for all analyses.

3. The area response of each internal standard from the EICP and the retention time of the internal standard are evaluated for stability, according to the procedures in paragraph 1 of Quantitation. The area of the internal standard in a sample must not vary by more than a factor of 2 (i.e., -50% to +100%) from the area of the same internal standard in the associated continuing calibration standard.

Likewise, the retention time of an internal standard must be within ± 0.50 minutes (30 seconds) of its retention time in the continuing calibration standard(see paragraph 1 of Quantitation).

4. Requirements for re-analysis of samples when internal standards do not meet specifications are given in paragraph 1 of Quantitation.

5. The documentation includes Form VIII SV, and the GC/MS data system printout for the analysis of each sample, blank, matrix spike, matrix spike duplicate and standard.

Method Blank Analysis

A method blank is a volume of a clean reference matrix (deionized distilled water for water samples, or a purified sodium sulfate for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

1. For semi-volatile analysis, one method blank must be extracted with each group of samples of a similar matrix and concentration level (soils only), as described in paragraph 7 of Quantitation.

2. For the purposes of this protocol, an acceptable method blank must meet the criteria in paragraphs a and b below.

   a. A method blank for semi-volatile analysis must contain less than or equal to five times (5x) the Contract Required Quantitation Limit (CRQL, see Table 9) of the phthalate esters listed in Table 9.

   b. For all other target compounds, the method blank must contain less than or equal to the Contract Required Quantitation Limit (CRQL, see Table 9) of any single target compound.

3. If a method blank exceeds the limits for contamination above, the laboratory must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for re-extraction and re-analysis of associated samples are given in paragraph 7 of Quantitation.

4. The documentation includes Form I SV for the blank analysis, Form IV SV, associating the samples and the blank, and a GC/MS data system printout for the analysis of the method blank.

Surrogate Recoveries

The recoveries of the eight surrogates are calculated from the analysis of each sample, blank, matrix spike and matrix spike duplicate. The purpose of the surrogates is to evaluate the preparation and analysis of samples.

1. The surrogates are added to each sample, blank, matrix spike and matrix spike duplicate prior to extration, at the concentrations described in paragraph 5 of Reagents.

2. The recoveries of the surrogates are calculated according to the procedures in paragraph 5a of Quantitation.

3. The recoveries must be within the quality control limits given in Table 6. If the recovery of any surrogate is outside these limits, the laboratory must follow the steps outlined in paragraphs 5b to 5f of Quantitation to determine whether or not re-extraction and/or re-analysis is required.

4. The documentation includes Form II SV, and a GC/MS data system printout for the analysis of each sample, blank, matrix spike, and a matrix spike duplicate.

Matrix Spike and Matrix Spike Duplicate Analysis

In order to evaluate the effects of the sample matrix on the methods used for semi-volatile analyses, the Agency has prescribed a mixture of semi-volatile target compounds to be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method.

1. The frequency of matrix spike and matrix spike duplicate (MS/MSD) analysis is described in paragraph 6 of Quantitation.

2. The recoveries of the matrix spike compounds are calculated according to the procedures in paragraph 6a of Quantitation. The relative percent difference between the results for each spiked analyte of the matrix spike and the matrix spike duplicate is calculated according to the procedures in paragraph 6b of Quantitation.

3. The quality control limits for recovery and relative percent difference are given in Table 7. These limits are only advisory at this time, and no further action is required when the limits are exceeded.

4. The documentation includes Form I SV for both the MS and MSD analyses, Form III SV and a GC/MS printout for each analysis.

TABLE 9

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

	Semi-volatiles	CAS Number	Quantitations Limits Water ug/l	Low Soil ug/Kg	Med. Soil ug/Kg
34.	Phenol	108-95-2	10	330	10000
35.	bis(2-Chloroethyl) ether	111-44-4	10	330	10000
36.	2-Chlorophenol	95-57-8	10	330	10000
37.	1,3-Dichlorobenzene	541-73-1	10	330	10000
38.	1,4-Dichlorobenzene	106-46-7	10	330	10000
39.	1,2-Dichlorobenzene	95-50-1	10	330	10000
40.	2-Methylphenol	95-48-7	10	330	10000
41.	2,2'-oxybis (1-Chloropropane)#	108-60-1	10	330	10000
42.	4-Methylphenol	106-44-5	10	330	10000
43.	N-Nitroso-di-n-propylamine	621-64-7	10	330	10000
44.	Hexachloroethane	67-72-1	10	330	10000
45.	Nitrobenzene	98-95-3	10	330	10000
46.	Isophorone	78-59-1	10	330	10000
47.	2-Nitrophenol	88-75-5	10	330	10000
48.	2,4-Dimethylphenol	105-67-9	10	330	10000
49.	bis(2-Chloroethoxy) methane	111-91-1	10	330	10000
50.	2,4-Dichlorophenol	120-83-2	10	330	10000
51.	1,2,4-Trichlorobenzene	120-82-1	10	330	10000
52.	Naphthalene	91-20-3	10	330	10000
53.	4-Chloroaniline	106-47-8	10	330	10000
54.	Hexachlorobutadiene	87-68-3	10	330	10000
55.	4-Chloro-3-methylphenol	59-50-7	10	330	10000
56.	2-Methylnaphthalene	91-57-6	10	330	10000
57.	Hexachlorocyclopentadiene	77-47-4	10	330	10000
58.	2,4,6-Trichlorophenol	88-06-2	10	330	10000
59.	2,4,5-Trichlorophenol	95-95-4	25	800	25000
60.	2-Chloronaphthalene	91-58-7	10	330	10000
61.	2-Nitroaniline	88-74-4	25	800	25000
62.	Dimethylphthalate	131-11-3	10	330	10000
63.	Acenaphthylene	208-96-8	10	330	10000
64.	2,6-Dinitrotoluene	606-20-2	10	330	10000
65.	3-Nitroaniline	99-09-2	25	800	25000
66.	Acenaphthene	83-32-9	10	330	10000
67.	2,4-Dinitrophenol	51-28-5	25	800	25000
68.	4-Nitrophenol	100-02-7	25	800	25000

#Previously known by the name bis(2-Chloroisopropyl)ether

TABLE 9 (Continued)

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

	Semi-volatiles	CAS Number	Quantitations Limits Water ug/l	Low Soil ug/Kg	Med. Soil ug/Kg
69.	Dibenzofuran	132-64-9	10	330	10000
70.	2,4-Dinitrotoluene	121-14-2	10	330	10000
71.	Diethylphthalate	84-66-2	10	330	10000
72.	4-Chlorophenyl-phenyl ether	7005-72-3	10	330	10000
73.	Fluorene	86-73-7	10	330	10000
74.	4-Nitroaniline	100-01-6	25	800	25000
75.	4,6-Dinitro-2-methylphenol	534-52-1	25	800	25000
76.	N-nitrosodiphenylamine	86-30-6	10	330	10000
77.	4-Bromophenyl-phenylether	101-55-3	10	330	10000
78.	Hexachlorobenzene	118-74-1	10	330	10000
79.	Pentachlorophenol	87-86-5	25	800	25000
80.	Phenanthrene	85-01-8	10	330	10000
81.	Anthracene	120-12-7	10	330	10000
82.	Carbazole	86-74-8	10	330	10000
83.	Di-n-butylphthalate	84-74-2	10	330	10000
84.	Fluoranthene	206-44-0	10	330	10000
85.	Pyrene	129-00-0	10	330	10000
86.	Butylbenzylphthalate	85-68-7	10	330	10000
87.	3,3'-Dichlorobenzidine	91-94-1	10	330	10000
88.	Benzo(a)anthracene	56-55-3	10	330	10000
89.	Chrysene	218-01-9	10	330	10000
90.	bis-(2-Ethylhexyl)phthalate	117-81-7	10	330	10000
91.	Di-n-octylphthalate	117-84-0	10	330	10000
92.	Benzo(b)fluoranthene	205-99-2	10	330	10000
93.	Benzo(k)fluoranthene	207-08-9	10	330	10000
94.	Benzo(a)pyrene	50-32-8	10	330	10000
95.	Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10000
96.	Dibenz(a,h)anthracene	53-70-3	10	330	10000
97.	Benzo(g,h,i)perylene	191-24-2	10	330	10000

STANDARD OPERATION PROCEDURE

ANALYTICAL METHODS

for

PESTICIDES/AROCLORS

EPA-CLP-SOW

Document #OLMO3.0-3.1

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(Pesticides/Aroclors)

SCOPE OF APPLICATION

The analytical method that follows is designed to analyze water, sediment and soil from hazardous waste sites to determine the presence and concentration of the chlorinated pesticides and Aroclors found in the Target Compound List (Table 1). The method can be used for determining analyte concentrations in the range from the contract required quantitation limits (CRQL) to one million times the CRQL in these matrices. The method is based on EPA Method 608.

SAMPLE STORAGE AND HOLDING TIMES

The samples must be protected from light and refrigerated at 4°C (±2°C) from the time of receipt until 60 days after delivery of a complete reconciled sample data package. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

Samples, sample extracts and standards must be stored separately.

Contract Required Holding Times

The extraction of water samples by separatory funnel procedures must be completed within five days of the Validated Time of Sample receipt (VTSR). Extraction of water samples by continuous liquid-liquid extraction procedures must be started within five days of VTSR. Extraction of soil/sediment samples by sonication must be completed within 10 days of VTSR.

Analysis of sample extracts must be completed within 40 days following the start of extraction.

SAMPLE PREPARATION FOR EXTRACTABLE PESTICIDES AND AROCLORS

Summary of Sample Preparation Methods

1. Water Samples

   A 1-l volume of sample is spiked with the surrogate solution and is extracted with methylene chloride by using a separatory funnel or a continuous extractor. The methylene chloride extract is dried and concentrated (see Extraction of Water Samples). The extract is then cleaned up by GPC (GPC is required when higher molecular weight compounds are present that interfere with the analyses of target compounds; GPC is optional for all other circumstances), exchanged to hexane, cleaned up by Florisil cartridge, and adjusted to a final volume of 1.0 ml or 2.0 ml as described beginning at paragraph of Solvent Exchange into Hexane.

2. Soil/sediment Samples

   A 30 g aliquot of sample is spiked with the surrogate solution and then mixed with sodium sulfate and extracted with a 1:1 acetone/methylene chloride solvent mixture by sonication. The extract is then filtered, dried, concentrated by K-D, and the solvent exchanged into methylene chloride (see Soil Extract Concentration). The extract is then cleaned up by GPC (mandatory), exchanged to hexane, cleaned up by Florisil cartridge, and adjusted to a final volume of 1.0 or 2.0 ml (see Solvent Exchange into Hexane).

Interferences

1. Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware. These contaminants lead to discrete artifacts or to elevated baselines in gas chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

2. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source-to-source, depending upon the nature of the site being sampled. The cleanup procedures must be used to remove such interferences in order to achieve the Contract Required Quantitation Limits.

Apparatus and Materials

1. Continuous liquid-liquid extractors - with Teflon or glass connecting lines for use with methylene chloride.

2. Separatory funnel - 2000 ml with Teflon stopcock.

3. Apparatus for determining percent moisture

   a. Oven - drying

   b. Desiccator

   c. Crucibles - porcelain (optional)

   d. Aluminum weighing pans (optional)

4. Sonic cell disrupter - Heat Systems, Ultrasonics, Inc., Model W-385 (475 watt with pulsing capability, No. 207 3/4-inch tapped disrupter horn) or equivalent device with a minimum 375 Watt output capability.

5. Sonabox (or equivalent) for use with disrupter to decrease noise level.

6. Beakers - 400-ml.

7. Kuderna-Danish (K-D) apparatus.

   a. Concentrator tube - 10 ml, graduated (Kontes K-570040-1029, or equivalent).

   b. Evaporative flask - 500 ml (Kontes K-470001-0500, or equivalent).

   c. Snyder column - three-ball macro (Kontes K-5030000-0121, or equivalent).

8. Funnels and Filter Paper

   a. Powder funnels - 10 cm diameter (optional), for filtration/drying.

   b. Buchner funnels - 9 cm diameter, for filtration (optional).

   c. Filter paper - No. 41 Whatman (or equivalent), 9 cm circles (optional).

9. Boiling chips - Silicon carbide boiling chips - approximately 10 to 40 mesh.

10. Water bath - heated, with concentric ring cover, capable of temperature control. NOTE: The water bath should be used in a hood.

11. Top loading balance - capable of weighing accurately to ± 0.01 g.

12. Balance - analytical, capable of weighing accurately to ± 0.0001 g.

13. Nitrogen evaporation device equipped with a heated bath that can be maintained at 35 to 40°C.

14. Vials and caps - 2 ml for GC auto sampler.

15. Gel permeation chromatography (GPC) cleanup device. NOTE: GPC cleanup is required for all extracts for all soils and for water extracts containing higher molecular weight contaminants that interfere with the analyses of the target compounds (see paragraph of Extract Cleanup by GPC).

    Gel permeation chromatography system - GPC Autoprep Model 1002 A or B, Analytical Biochemical Laboratories, Inc, or equivalent. Systems that perform very satisfactorily also have been assembled from the following components - an HPLC pump, an autosampler or a valving system with sample loops, and a fraction collector. All systems, whether automated or manual, must meet the calibration requirements of Calibration of the GPC Column.

    a. Chromatographic column - 700 mm x 25 mm i.d. glass column. Flow is upward. To simplify switching from the UV detector during calibration to the GPC collection device during extract cleanup, an optional double 3-way valve (Rheodyne Type 50 Teflon Rotary Valve #10-262 or equivalent) may be attached so that the column exit flow can be shunted either to the UV flow-through cell or to the GPC collection device.

    b. Guard column (Optional) 5 cm, with appropriate fittings to connect to the inlet side of the analytical column (Supelco 5-8319 or equivalent).

    c. Bio Beads (S-X3) - 200-400 mesh, 70 gm. An additional 5 gm of Bio Beads is required if the optional guard column is employed. The quality of Bio Beads may vary from lot-to-lot because of excessive fines in some lots. In addition to fines having a detrimental effect on chromatography, they also can pass through the column screens and damage the valve.

    d. Ultraviolet detector - fixed wavelength (254 nm) with a semi-pre flow-through cell.

    e. Strip chart recorder, recording integrator or laboratory data system.

    f. Syringe - 10 ml with Luerlok fitting.

g. Syringe filter assembly, disposable - Bio-Rad "Prep Disc" sample filter assembly #343-0005, 25 mm, and 5 micron filter discs or equivalent. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

h. Glass bottle - 1 liter volume, for use in preparation of Bio beads for packing into column.

16. Florisil - 500 mg or 1 g cartridges.

17. Vacuum system for eluting multiple cleanup cartridges.

a. Vac Elute Manifold - The manifold design must ensure that there is no contact between plastics containing phthalates and sample extracts.

b. Vacuum trap made from a 500 ml sidearm flask fitted with a one-hole stopper and glass tubing.

c. Vacuum pressure gauge.

d. Rack for holding 10 ml volumetric flasks in the manifold.

18. Pyrex glass wool - rinsed with methylene chloride and dried before use.

19. Bottle or test tube - 20 ml with Teflon-lined screw cap for sulfur removal.

20. Glass vials - minimum of 20 ml, with screw cap and Teflon or aluminum foil liner.

21. Spatula - stainless steel or Teflon.

22. pH Paper - wide range.

23. Pipet - Volumetric 1.00 ml or 2.00ml (optional).

24. Syringe - 1.00 ml or 2.00 ml (optional).

25. Flask - Volumetric 10.00 ml.

26. Flask - Volumetric 1.00 ml or 2.00 ml (optional).

27. Vials- 10 ml, with screw cap and Teflon liner (optional).

28. Tube - centrifuge, 12 to 15 ml with 19 mm ground glass joint (optional).

29. Snyder Column - micro two or three-ball with a 19 mm ground glass joint.

30. Centrifuge - table top (optional).

31. Vortex mixer - Genie, Model 550-6, Scientific Industrial, Inc., Bohemia, NY or equivalent.

32. pH Meter with a combination glass electrode.

33. Magnetic stirrer motor - Model PC353, Corning Co., Corning NY or equivalent.

34. Magnetic stir bar - Teflon coated, at least 4 cm long.

35. Graduated cylinder - one (1) L capacity.

Reagents

1. Sodium sulfate - granular-anhydrous reagent grade, heated at 400°C for 4 hours, or at 120°C for 16 hours, cooled in a desiccator, and stored in a glass bottle.

2. Methylene chloride, hexane, acetone, toluene, iso-octane, and methanol (optional) - pesticide quality or equivalent.

3. Copper powder (optional) - fine, granular. Copper may be used for sulfur cleanup. Remove oxides by treating with dilute nitric acid, rinse with distilled water to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen.

4. Sodium hydroxide solution (10N) - Carefully dissolve 40 g of NaOH in reagent water and dilute the solution to 100 ml.

5. Concentrated sulfuric acid - 18 N.

6. Reagent water - defined as a water in which no interferant is observed at one-half the CRQL of any pesticide/Aroclor when one liter of the reagent water is extracted and prepared by using the same workup procedure as for a water sample.

7. Ten percent acetone in hexane (v/v) - prepare by adding 10.0 ml of acetone to 90.0 ml of hexane. NOTE: Prepare this mixture accurately or the results from the Florisil cartridge cleanup will be adversely affected. Water in the acetone also will adversely affect Florisil performance.

8. Standards

   a. The laboratory must provide all standards to be used with this protocol. These standards may be used only after they have been certified according to the procedure in QA/QC Requirements. The laboratory must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the laboratory and presented upon request.

   b. Stock standard solutions (1.00 ug/ul) - can be prepared from pure standard materials or purchased as certified solutions.

      (1) Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in toluene, dilute to volume in a 10-ml volumetric flask with toluene or acetone.

      (2) Transfer the stock standard solutions into a bottle/vial with Teflon-lined cap or septa. Store at 4°C(±2°C) and protect from light. Stock standard solutions must be replaced after six months or sooner, if comparison with check standards indicates a problem.

c. GPC calibration solution - prepare a solution in methylene chloride that contains the following analytes in the concentrations listed below:

Analyte	mg/ml
corn oil	25
bis-2-ethylhexyl phthalate	1.0
methoxychlor	0.2
perylene	0.02
sulfur	0.08

NOTE: Sulfur is not very soluble in methylene chloride, however, it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds.

Store the calibration solution in an amber glass bottle with a Teflon lined screw-cap at 4°C, and protect from light. (Refrigeration may cause the corn oil to precipitate. Before use, allow the calibration solution to stand at room temperature until the corn oil dissolves.) Replace the calibration standard solution every six months or more frequently if necessary.

d. Surrogate solution - the surrogates, Tetrochloro-m-xylene and Decachlorobiphenyl, are added to all standards, samples, matrix spikes and blanks. Prepare a surrogate spiking solution of 0.2 ug/ml of each of the two compounds in acetone. The solution should be check frequently for stability. The solution must be replace after six months, or sooner, if comparison with quality control check samples indicates a problem. **CAUTION:** Analysts must allow all spiking solutions to equilibrate to room temperature before use.

e. Pesticide matrix spiking solution - prepare a spiking solution in acetone or methanol that contains the following pesticides in the concentrations specified:

Pesticide	ug/ml
gamma-BHC (Lindane)	0.5
4,4'-DDT	1.0
Endrin	1.0
Heptachlor	0.5
Aldrin	0.5
Dieldrin	1.0

The solution must be prepared every six months, or sooner if the solution has degraded or concentrated.

f. Florisil cartridge check solution.

Prepare a solution of 2,4,5-trichlorophenol in acetone, at a concentration of 0.1 ug/ml.

g. Store all standard solutions in amber glass bottles or vials with a teflon-lined screw cap at 4°C(±2°C) and protect from light.

EXTRACTION OF WATER SAMPLES

Water samples may be extracted by either a separatory funnel procedure or a continuous liquid-liquid extraction procedure. If an emulsion prevents acceptable solvent recovery with the separatory funnel procedure, continuous liquid-liquid extraction must be employed.

1. Separatory Funnel Extraction

   a. Measure out each 1.0 L sample aliquot in a separate graduated cylinder. Measure and record the pH of the sample with wide-range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or conc. sulfuric acid, if required. Samples requiring pH adjustment must be noted. Place the sample into a 2-L separatory funnel.

   b. For each sample selected for matrix spike and matrix spike duplicate analyses, measure out two additional 1-L portions and transfer those portions into separate funnels. Adjust the pH of each, if required, and fortify each with 1.0 ml of matrix spike solution before continuing the extraction. The frequency of MS/MSD analysis is given in paragraph of Matrix Spike/Matrix Spike Duplicate below.

   c. Using a syringe or a volumetric pipet, add 1.0 ml of the surrogate solution to all water samples, matrix spikes and blanks.

   d. Add 60 ml methylene chloride to the separatory funnel and extract the sample by spiking the funnel for two minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation or other physical means. Drain the methylene chloride into a 250 ml Erlenmeyer flask.

   e. Add a second 60 ml volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

   f. Prepare a method blank with each group of water samples extracted. For pesticide/Aroclor analyses, a method blank for water samples consists of a 1 L volume of reagent water (see paragraph 6 of Reagents), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in paragraph of Blanks below.

2. Continuous Liquid-Liquid Extraction

   a. Add methylene chloride (100 to 250 ml) to the bottom of the extractor and fill it to a depth of at least one-inch above the bottom sidearm.

   b. Measure out each 1.0 L sample aliquot in a separate graduated cylinder. Measure and record the pH of the sample with wide-range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Samples requiring pH adjustment must be noted. Place the sample into the continuous extractor.

c. With some samples it may be necessary to place a layer of glass wool between the methylene chloride and the water layers in the extractor to prevent precipitation of suspended solids into the methylene chloride during extraction.

d. For each sample selected for matrix spike and matrix spike duplicate analyses, measure out two additional l-L portions and transfer those portions into separate funnels. Adjust the pH of each, if required, and fortify each with 1.0 ml of matrix spike solution before continuing the extraction. The frequency of MS/MSD analysis is given in paragraph of Matrix Spike/Matrix Spike Duplicate below.

e. Using a syringe or a volumetric pipet, add 1.0 ml of the surrogate solution to all water samples, matrix spikes, and blanks.

f. Adjust the level of methylene chloride in the extractor so that the bottom sidearm is half filled with solvent.

g. Add sufficient methylene chloride to the distilling flask to ensure proper solvent cycling during operation and extract the solution for 18 hours. Allow to cool, then detach the distillation flask and label.

h. Prepare a method blank with each group of water samples extracted. For pesticide/Aroclor analyses, a method blank for water samples consists of a 1 L volume of reagent water (see paragraph 6 of Reagents), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in paragraph of Blanks below.

3. Extract Drying and Concentration

a. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 ml concentrator tube to a 500 ml evaporative flask. Other concentration devices or techniques may be used in place of the K-D if equivalency is demonstrated for all the pesticide/Aroclor target compounds listed in Table 1.

b. Pour the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and the column with at least two additional 20 to 30 ml portions of methylene chloride to complete the quantitative transfer.

c. Add one of two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 ml of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (60° - 80°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface to the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 30 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 3 to 5 ml, remove the K-D apparatus. Allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATED TO GO DRY.

d. If no GPC cleanup is required, proceed with the hexane exchange described in paragraph of Solvent Exchange into Hexane. If GPC cleanup is to be used, remove the Snyder column, rinse the flask and its lower joint and collect the rinsate in the concentrator tube, adjust the volume to 10.0 ml with methylene chloride. Proceed to paragraph of Extract Cleanup by GPC.

EXTRACTION OF SOIL/SEDIMENT SAMPLES

1. Sample Preparation

   a. Mix samples thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Also, decant and discard any standing aqueous phase.

   b. pH Determination - transfer 50 g of soil/sediment to a 100-ml beaker. Add 50 ml of water and stir the solution with a magnetic stirrer for one (1) hour. Determine the pH of the sample by using a glass electrode and the pH meter while the sample is stirred. Report pH value on the appropriate data sheet. If the pH of the soil is > 9 or <5, document any subsequent problems in the analysis related to pH but do not attempt to adjust the pH of the sample. Discard the portion of the sample used for pH determination.

   c. Percent Moisture - Weigh 5 to 10 g of the sediment to the nearest 0.01 g into a tared crucible or aluminum weighing pan. Determine the weight percent volatilized by drying overnight at 105°C (hereafter referred to as percent moisture). After the sample is dry, remove the sample and pan and allow them to cool in a desiccator before weighing. Calculate the percent moisture according to equation below. Concentrations of individual analytes will be reported relative to the dry weight of sediment. **CAUTION:** Gases volatilized from some soil/sediment samples may require that this drying procedure be carried out in a hood.

$$\text{Percent Moisture} = \frac{\text{Wt of Sample - Wt of Dry Sample}}{\text{Wt of Sample}} \times 100$$

2. Extraction with Sonication

   a. Tune the sonicator according to the manufacturer's directions prior to extracting samples by this procedure.

   b. Weight approximately 30 g of sample (to the nearest 0.1 g) into a 250 or 400-ml beaker and add 60 g of anhydrous sodium sulfate (granular).

   c. For a sample to be used for matrix spike and matrix spike duplicate analysis, weigh out two additional 30 g (record weight to nearest 0.1 g) portions of sample and add 1.0 ml of the pesticide matrix spike solution to each soil aliquot. The frequency of MS/MSD analysis is given in paragraph of Matrix Spike/Matrix Spike Duplicate below.

   d. Add 2.0 ml of surrogate solution to all soil samples, matrix spikes, and blanks by using a volumetric pipet or a syringe. Mix the solution well. The sample and the added sodium sulfate should be a homogeneous, granular mixture at this point. (Twice as much of the surrogate solution is added to soil samples than to water samples because of the increased likelihood that the soil extracts will require dilution).

   e. Immediately add 80 to 100 ml of 1:1 methylene chloride/acetone to the sample.

   f. Place the bottom surface of the sonicator probe about 1/2 inch below the surface of the solvent but above the sediment layer.

   g. Sonicate for 3 minutes using a 3/4-inch horn at full power (output control knob at 10) with pulse on and percent duty cycle know set at 50 percent.

h. The extracted sample can be filtered by using gravity filtration.

Prepare a filtration/drying bed by placing a plug of glass wool in the neck of a 10-cm powder funnel and filling the funnel to approximately half its dept (4 or 5 cm) with anhydrous sodium sulfate (80-100 g). Decant the extract through the packed funnel and collect it in a 500-ml evaporation (K-D) flask.

i. Repeat the extraction two more times with additional 80 to 100 ml portions of the 1:1 methylene chloride/acetone. Before each extraction, thoroughly mix the solid residue, and make certain that the sodium sulfate is free flowing and not a consolidated mass. As required, break up large lumps with a clean spatula. Decant and filter the extraction solvent after each sonication by using the same funnel described in paragraph h above. After the final sonication, pour the entire sample into the funnel and rinse the beaker and funnel with 60 ml of 1:1 methylene chloride/acetone.

j. Prepare a method blank with each group of soil/sediment samples extracted. For pesticide/Aroclor analyses, a method blank for soil/sediment samples consists of 30 g of sodium sulfate (see paragraph 1 of Reagents), spiked with the surrogates and carried through the entire analytical procedure. The frequency of method blank analysis is given in paragraph of Blanks below.

3. Soil Extract Concentration

a. Add one or two clean boiling chips to the evaporative flask and attach a three-ball macro Snyder column. Pre-wet the Snyder column by adding about 1 ml of methylene chloride to the top. Place the K-D apparatus on a hot water bath (60 to 80°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 30 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. Reduce the volume of liquid to less than 10 ml. Remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

b. In order to remove most of the acetone, it is absolutely necessary to further reduce the volume of all soil/sediment extracts to 1.0 ml. This is the best accomplished using the nitrogen evaporation technique (see paragraph of Nitrogen Evaporation Techniques below). The presence of acetone will cause a dead volume to develop in the GPC column and thus will cause loss of surrogates and analytes during GPC cleanups.

c. Adjust the extract volume to 10.0 ml with methylene chloride. Proceed to Extract Cleanup, below, for mandatory GPC and Florisil cartridge cleanup of soil extracts.

Extract Cleanup

There are three cleanup procedures specified in this method: GPC, Florisil cartridge, and sulfur cleanup. GPC must be performed for all soil extracts. GPC must be performed for water extracts that contain higher molecular weight contaminants that interfere with the analysis of the target analytes. Florisil cartridge cleanup is mandatory for all extracts. Sulfur cleanup must be performed for all sample extracts contaminated with sulfur. Blanks and matrix spike and matrix spike duplicate samples must be subjected to the same cleanup as the unspiked samples.

1. Extract Cleanup by Gel Permeation Chromatography (GPC)

a. GPC is mandatory for all soil/sediment extracts. GPC must be performed for water extracts that contain higher molecular weight contaminants that interfere with the analysis of the target analytes. Gel permeation chromatography (GPC) is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of synthetic macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the molecular size of the molecules to be separated. A cross-linked divinyl benzenestyrene copolymer (SX-3 Bio Beads or equivalent) is specified for this method.

GPC is recommended for the elimination from the sample of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds. GPC is appropriate for both polar and non-polar analytes,therefore, it can be used effectively to clean up extracts containing a broad range of analytes.

Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a gas chromatograph (GC) or in the front of the GC column. This residue ultimately will reduce the chromatographic separation efficiency or column capacity because of absorption of the target analytes on the active sites. Pentachlorophenol especially is susceptible to this problem. GPC system performance must be validated at least once every seven calendar days by demonstrating 80-110 percent recovery of the pesticide matrix spike mixture and examining the pattern of peaks from an Aroclor 1016/1260 mixture.

b. GPC Column Preparation

(1) Weigh out 70 gm of Bio Beads (SX-3). Transfer them to a one (1) liter bottle with a Teflon-lined cap or a 500 ml separatory funnel with a large bore stopcock, and add approximately 300 ml of methylene chloride. Swirl the container to ensure the wetting of all beads. Allow the beads to swell for a minimum of 2 hours. Maintain enough solvent to cover the beads sufficiently at all times. If a guard column is to be used, repeat the above with 5 gm of Bio Beads in a 125 ml bottle or a beaker, using 25 ml of methylene chloride.

(2) Turn the column upside down from its normal position, and remove the inlet bed support plunger (the inlet plunger is longer than the outlet plunger). Position and tighten the outlet bed support plunger as near the end as possible, but no closer than 5 cm (measured from the gel packing to the collar).

(3) Raise the end of the outlet tube to keep the solvent in the GPC column, or close the column outlet stopcock. Place a small amount of solvent in the column to minimize the formation of air bubbles at the base of poured column packing.

(4) Swirl the bead/solvent slurry to get a homogeneous mixture and, if the wetting was done in a quart bottle, quickly transfer it to a 500 ml separatory funnel with a large bore stopcock. Drain the excess methylene chloride directly into the waste beaker, and then start draining the slurry into the column by placing the separatory funnel tip against the column wall. This will help to minimize bubble formation. Swirl occasionally to keep the slurry homogeneous. Drain enough to fill the column. Place the tubing from the column outlet into a waste beaker below the column outlet into a waste below the column, open the stopcock (if attached) and allow the excess solvent

to drain. Raise the tube to stop the flow, and close the stopcock when the top of the gel begins to look dry. Add additional methylene chloride to just re-wet the gel.

(5) Wipe any remaining beads and solvent from the inner walls of the top of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

(6) Compress the column as much as possible without applying excessive force. Loosen the seal and gradually pull out the plunger. Rinse and wipe off the plunger. Slurry any remaining beads and transfer them into the column. Repeat the step in paragraph (5) above and reinsert the plunger. If the plunger cannot be inserted and pushed in without allowing beads to escape around the seal, continue compression of the beads without tightening the seal, and loosen and remove the plunger as described. Repeat this procedure until the plunger is inserted successfully.

(7) Push the plunger until it meets the gel, then compress the column bed about four centimeters.

(8) Pack the optional 5 cm column with approximately 5 gm of pre-swelled beads (different guard columns may require different amounts). Connect the guard column to the inlet of the analytical column.

(9) Connect the column inlet to the solvent reservoir (reservoir should be place higher than the top of the column) and place the column outlet tube in a waste container. Placing a restrictor in the outlet tube will force air out of the column more quickly. A restrictor can be made from a piece of capillary stainless steel tubing of 1/16" OD x 10/1000" ID x 2". Pump methylene chloride through the column at a rate of 5 ml/min for one hour.

(10) After washing the column for at least one hour, connect the column outlet tube, without the restrictor, to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. A restrictor (same size as the one in paragraph (9) above) in the outlet tube from the UV detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not effect flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi backpressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

(11) When the GPC column is not to be used for several days, connect the column outlet line to the column inlet to prevent column drying and/or channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, methylene chloride should be pumped through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify retention volumes have not changed.

(12) The GPC calibration procedure is based on monitoring the elution of standards with a UV detector connected to the GPC column. Care must be taken to account for any difference in volume (elution time) between the GC column and detector and between the GPC column and the collection vial.

c. Calibration of the GPC Column

(1) Using a 10 ml syringe, load sample loop #1 with calibration solution (paragraph 8c of Reagents). With the ABC automated system, the 5 ml sample loop requires a minimum of 8 ml of the calibration solution. Use a firm, continuous pressure to push the sample onto the loop. Switch the valve so that GPC flow is through the UV flow-through cell.

(2) Inject the calibration solution and obtain a UV trace showing a discrete peak for each component. Adjust the detector and/or recorder sensitivity to produce a UV trace that meets the following requirements. Differences between manufacturer's cell volumes and detector sensitivities may require a dilution of the calibration solution to achieve similar results. An analytical flow-through detector cell will require a much less concentrated solution than the semi-prep cell and, therefore, the analytical cell is not acceptable for use.

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit >85% resolution.
- Phthalate and methoxychlor peaks must exhibit >85% resolution.
- Methoxychlor and perylene peaks must exhibit >85% resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

(3) Determine the elution times for the phthalate, methoxychlor and perylene. Phthalate will elute first, perylene last.

(4) Choose a "DUMP" time which removes >85 percent of the phthalate. Choose a "COLLECT" time so the >95 percent of the methoxychlor is collected, and continue to collect until just prior to the elution time of sulfur. Us a "WASH" time of 10 minutes.

(5) NOTE: The DUMP and COLLECT times must be adjusted to compensate for the difference in volume of the lines between the detector and the collection flask.

(6) Verify the flow rate by collecting column eluate for 10 minutes in a graduated cylinder and measure the volume, which should be 45-55 ml (4.5-5.5 ml/min). If the flow rate is outside of this range, corrective action must be taken to achieve this flow rate. Once the flow rate is within the range of 4.5-5.5 ml/min, record the column pressure (should be 6-10 psi) and room temperature. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared. A UV trace that does not meet the criteria in paragraph (2) above would also indicate that a new column should be prepared. It may be necessary to obtain a new lot of Bio Beads if the column fails all the criteria.

(7) Re-inject the calibration solution after appropriate collect and dump cycles have been set, and the solvent flow and column pressure have been established.

(7a) Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed may be used to indicate problems with the system during sample processing.

(7b) The retention times for bis(2-ethylhexyl)phthalate and perylene must not vary more than ±5% between calibrations. If the retention time shift is >5%, take corrective action. Excessive retention time shifts are caused by the following:

- Poor laboratory temperature control or system leaks.
- An unstabilized column that requires pumping methylene chloride through it for several more hours or overnight.
- Excessive laboratory temperatures causing outgassing of the methylene chloride.

(8) Analyze a GPC blank by loading 5 ml of methylene chloride into the GPC. Concentrate the methylene chloride that passes through the system during the collect cycle using a Kuderna-Danish (KD) evaporator. Exchange the solvent to hexane and analyze the concentrate by GC/EC. If the blank exceeds one-half the CRQL of any analyte, assuming that the blank represents the extract from a one liter water sample, pump additional methylene chloride through the system for 1-2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

c. GPC Calibration Check

No Florisil cleanup is used in the GPC calibration check.

(1) At least once every 7 day, the calibration of the GPC must be verified with two check mixtures. The first mixture is prepared by concentrating 2.0 ml of the matrix spiking solution (paragraph 8e of Reagents) to less than 1 ml under a stream of nitrogen (see paragraph of Nitrogen Evaporation Technique below), and adjusting the final volume to 10.0 ml with methylene chloride. The second mixture is prepared with 2 ug of Aroclor 1016 and 2 ug of Aroclor 1260 in a final volume of 10.0 ml methylene chloride.

(2) Load the first 5.0 ml sample loop by using a 10 ml syringe containing 8 ml of the dilutes pesticide matrix spike solution (paragraph (1) above). The Aroclor mixture is loaded into Loop 2 in the same manner. Fractions are collected in an auto sequence by using the GPC program established by the UV detector calibration procedure (paragraph of Calibration of GPC Column).

(3) The collected GPC calibration fraction is transferred to a K-D apparatus and the collection vessel is rinsed with two additional 10-ml portions of methylene chloride to complete the transfer. The volume of methylene chloride is reduced (described in paragraph of Nitrogen Evaporation Technique below). After cooling, the solvent is exchanged to hexane according to the instruction in paragraph of Solvent Exchange to Hexane below. The final volume is adjusted to 10.0 ml, and the sample is analyzed by

GC according to the procedures in GC/EC Analysis of Pesticides and Aroclors below. The analysis must be performed on at least one of the GC columns used for samples analysis.

(4) The pattern of the Aroclor quantitation peaks and the recovery of each single component analyte must be determined for evaluation and reporting purposes. If the recovery of each of the single component analytes is 80 to 110 percent and if the Aroclor pattern is the same as with previously run standards, the analyst may continue to use the column. If recoveries are out of the acceptance window or if changes in the relative peak heights of the patterns of the aroclor are observed, the column must be replace and recalibrated according to the instructions in paragraph of Calibration of GPC Column.

(5) Some samples may contaminate the SX-3 Bio Beads and change the retention volume of the GPC column. Therefore system calibration and analyte recovery must be checked whenever a sample causes significant discoloration of the GPC column. Even if no darkening is visible, GPC calibration must be checked not less than once every seven days. In many cases, the SX-3 Bio Beads may be used for several months as long as the column calibration and flow rate remain constant.

d. Daily UV calibration check (optional)

The calibration of the GPC may be monitored daily by use of the UV-GPC calibration solution (paragraph 8c of Reagents) and the UV Detector Calibration Procedure (see paragraph of Calibration of GPC Column). The UV detector should be used to monitor the elution times for the phthalate, methoxychlor, and perylene, in that order. The precalibrated GPC program should "DUMP" > 85 percent of the phthalate and should "COLLECT" > 95 percent of the methoxychlor and perylene. Significant changes in elution times of the analytes (e.g., > 0.5 minutes) indicate that the column is out of calibration and must be recalibrated or replaced.

e. Sample Extract Cleanup

It is very important to have consistent laboratory temperatures during an entire GPC run, which could be 24 hours or more. If temperatures are not consistent, retention times will shift, and the dump and collect times determined by the calibration standard no longer will be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C.

(1) In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 glycerol:water solution must be diluted and loaded into several loops. Similarly, extracts containing more than 500 mg of non-volatile residue per 5 ml of extract must be diluted and loaded into several loops. The non-volatile residue may be determined by evaporating a 100 ul aliquot of the extract to dryness in a tared aluminum weighing pan, or other suitable container.

(2) Particles greater than 5 micron may scratch the valve, which may result in a system leak and cross contamination of sample extracts in the sample loops. To avoid such problems, filter the extract through a 5 micron filter disc by attaching a syringe filter assembly and into the 10 ml syringe. Disconnect the filter assembly before transferring the sample extract into a small glass container, e.g., a 15 ml culture tube with a Teflon lined screw cap. Alternatively, draw the extract into the syringe without the filter

assembly. Attach the filter assembly and force the extract through the filter and into the glass container. Draw a minimum of 8 ml of extract into a 10 ml syringe.

(3) Prior to loading samples, put the GPC into the "LOAD" mode, set the instrument terminal for the number of loops to be loaded, and set the "DUMP", "COLLECT", and "WASH" times for the values determined by the calibration procedure described in paragraph of Calibration of GPC Column.

(4) Attach the syringe to the turn lock on the injection port. Use firm, continuous pressure to push the sample onto the 5-ml sample loop. If the sample is difficult to load, some part of the system may be blocked. Take appropriate corrective action. If the back pressure is normal (6-10 psi) the blockage is probably in the valve. Blockage may be flushed out of the valve by reversing the inlet and outlet tubes an pumping solvent through the tubes (this should be done before sample loading).

(5) After loading a loop, and before removing the syringe from the injection port, index the GPC to the next loop. This will prevent loss of sample caused by unequal pressure in the loops.

(6) After loading each sample loop, wash the loading port with methylene chloride in PTFE wash bottle to minimize cross contamination. Inject approximately 10 ml of methylene chloride to rinse the common tubes.

(7) After loading all the sample loops, index the GPC to the 00 position, switch to the "RUN" mode and start the automated sequence. Process each sample using the collect and dump cycle times established in paragraph of Calibration of GPC Column.

(8) Collect each sample in a 250-ml Erlenmeyer flask, covered with aluminum foil to reduce solvent evaporation, or directly into a Kuderna-Danish evaporator. Monitor sample volumes collected. Changes in sample volumes collected may indicate one or more of the following problems:

- Change in solvent flow rate, caused by channeling in the column or changes in column pressure.
- Increase in column operating pressure due to the absorption of articles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.
- Leaks in the system or significant variances in room temperature.

(9) After the appropriate GPC fraction has been collected for each sample, the solvent must be exchanged to hexane as described in paragraph of Solvent Exchange into Hexane below.

(10) Any samples that were loaded into two or more loops must be recombined before proceeding to solvent exchange.

2. Solvent Exchange Into Hexane

This procedure applies to both extracts of water samples and extracts of soil samples.

a. With the extract in a K-D apparatus, remove the Snyder column, add 50 ml of hexane and a new boiling chip, and reattach the Snyder column. Pre-wet the column by adding about 1 ml of hexane to the top. Concentrate the solvent extract as described previously. When the apparent volume of liquid reaches 3 to 5 ml, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

b. Remove the Snyder column; using 1 to 2 ml of hexane, rinse the flask and its lower joint into the concentrator tube. Complete quantitative transfer of the extract to a 10 ml vial by using hexane.

c. For samples which have <u>not</u> been subjected to GPC cleanup, adjust the volume of the hexane extract to 10.0 ml. For samples which <u>have</u> been subjected to GPC cleanup, concentrate the hexane extract to 5.0 ml using nitrogen evaporation, as described in paragraph of Nitrogen Evaporation Technique below. Proceed to paragraph of Florisil Cartridge Cleanup below for Florisil cartridge cleanup.

3. Florisil Cartridge Procedure

Florisil cartridge cleanup is required for all extracts. Cleanup significantly reduces matrix interferences caused by polar compounds.

a. Cartridge Performance Check - every lot number of Florisil cartridges must be tested by the following procedure before they are used for sample cleanup. Add 0.5 ml of 2,4,5-trichlorophenol solution (0.1 ug/ml in acetone) and 0.5 ml of Standard Mixture A, mid-point concentration (paragraph 3 of Calibration Standards below) to 4 ml of hexane. Reduce the final volume to 0.5 ml using nitrogen (paragraph of Nitrogen Evaporation Technique below). Place the mixture onto the top of a washed Florisil cartridge (paragraph (4) of Florisil Cartridge Cleanup below), and elute it with 9 ml of hexane/acetone [(90:10)(V/V)]. Use two additional 1-ml hexane rinses to ensure quantitative transfer of standard from the cartridge. Reduce the final volume to 1.0 ml using nitrogen (see paragraph of Nitrogen Evaporation Technique below) and analyze the solution by GC/EC using at least one of the GC columns specified for sample analyses. The recovery of each analyte must be determined for evaluation and reporting purposes. Calculate the percent recovery using the equation below. The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 120 percent, if the recovery of trichlorophenol is less than 5%, and if not peaks interfering with the target analytes are detected.

EQ. 1

$$\text{Percent Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where,

Q_d = Quantity determined by analysis.

Q_a = Quantity added to sample/blank

b. Nitrogen Evaporation Technique

(1) Place the concentrator tube with an open mini-Snyder column attached in a heating bath (30 to 35°C) and evaporate the solvent to the final volume by blowing a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon) onto the solvent. DO NOT ALLOW THE EXTRACT TO GO TO DRYNESS.

(2) New plastic tubing must not be used between the carbon trap and the sample as it may introduce interferences. The internal wall of new tubing must be rinsed several times with hexane and then dried prior to use.

c. Florisil Cartridge Cleanup

(1) Attach the vacuum manifold to a water aspirator or to a vacuum pump with a trap installed between the manifold and the vacuum source. Adjust the vacuum pressure in the manifold to between 5 and 10 pounds of vacuum.

(2) Place on Florisil cartridge into the vacuum manifold for each sample extract.

(3) The required Florisil cartridge size and the final volume of the extract after Florisil cleanup are a function of the GC autosampler that a laboratory uses. If the autosampler operates reliably with 1.0 ml of sample extract, then a 500-mg cartridge is used and the required final volume is 1.0 ml. If the autosampler requires more sample, prepare 2.0 ml of sample extract using a 1-g cartridge. Manual injection requires only a 1.0 ml final extract volume and a 500-mg cartridge.

(4) Prior to cleanup of samples, the cartridges must be washed with hexane/acetone (90:10). This is accomplished by placing the cartridge in the vacuum manifold, by pulling a vacuum, and by passing at least 5 ml of the hexane/acetone solution through the cartridge. While the cartridges are being washed, adjust the vacuum applied to each cartridge so that the flow rate through each cartridge is approximately equal. DO NO ALLOW THE CARTRIDGES TO GO DRY AFTER THEY HAVE BEEN WASHED.

(5) After the cartridges in the manifold are washed, the vacuum is released, and a rack containing labeled 10-ml volumetric flasks is placed inside the manifold. Care must be taken to ensure that the solvent line from each cartridge is placed inside the appropriate volumetric flask as the manifold top is replaced.

(6) After the volumetric flasks are in place, vacuum to the manifold is restored, and a volume of extract equal to the required final volume (1.0 or 2.0 ml) from each sample, blank or matrix spike extract is transferred to the top frit of the appropriate Florisil cartridge.

(7) Because the volumes marked on concentrator tubes are not necessarily accurate at the 1-ml level, the use of a syringe or a volumetric pipet is required to transfer the extract to the cleanup cartridge.

(8) The pesticides/Aroclors in the extract concentrates are then eluted through the column with 8 ml of hexane/acetone (90:10) and are collected into the 10-ml volumetric flasks held in the rack into the vacuum manifold.

Appendix K

(9) Transfer the eluate in each volumetric flask to a clean centrifuge tube or 10-ml vial. Use two additional 1-ml hexane rinses to ensure quantitative transfer of the cartridge eluate.

(10) Concentrate the extract to 1.0 or 2.0 ml as required in paragraph of Nitrogen Evaporation Technique above using nitrogen blowdown. Measure the final volume with a syringe or by transferring the extract to a volumetric flask.

(11) Sulfur contamination will cause a rise in the baseline of the chromatogram that may interfere with the analyses of the later eluting pesticides. If crystals of sulfur are evident or if the presence of sulfur is suspected, proceed to paragraph of Sulfur Removal below. Sample analyses showing the presence of sulfur are not acceptable and must be cleaned up and re-analyzed.

(12) If sulfur is not present, transfer the sample to a GC vial and label the vial. The extract is ready for GC/EC analysis. Proceed to GC/EC Analysis of Pesticides/Aroclors below. Store the extracts at 4°C (±2°C) in the dark.

4. Sulfur Removal

Sulfur can be removed by the copper technique described below. Interference which is due to sulfur is not acceptable. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean centrifuge tube or clean concentrator tube before proceeding with further sulfur cleanup.

a. If only part of a set of samples requires sulfur cleanup, then two method blanks are required for that set: one that is shaken with copper, and one that is not.

Sulfur cleanup blank - add 1.0 ml of surrogate to 10 ml of hexane in a clean centrifuge tube or 10-ml vial. Concentrate the solution to 2.0 ml by using nitrogen evaporation. The concentrated volume of the blank must be the same as the final volume of the samples associated with the blank. Measure the volume with a syringe or by transferring the solution to a volumetric flask. Proceed with the sulfur removal using the same technique as the samples associated with the blank.

b. Copper Technique

Add approximately 2 g of cleaned copper powder to the extract in the centrifuge or concentrator tube. (2 g will fill the tube to about the 0.5 ml mark). Mix the copper and extract for at least one minute on a mechanical shaker. Separate the extract from the copper powder by drawing off the extract with a disposable pipet, and transfer the extract to a clean vial. The extract transferred to the vial still represents the 2.0 ml final volume. The separation of the extract from the copper powder is necessary to prevent degradation of the pesticides. If the copper appears bright, proceed to GC/EC Analysis protocol below and analyze the extract. If the copper changes color, repeat the sulfur removal procedure as necessary.

GC/EC ANALYSIS OF PESTICIDES AND AROCLORS

Summary of GC/EC Analysis

1. The analysis of samples is accomplished by using a wide-bore (0.53 mm ID) fused silica capillary column.

2. Sample extracts, standards, and blanks must be analyzed within an analytical sequence as defined in paragraph of Analysis Sequence for Standards and Samples below. GC/EC analysis begins with an initial demonstration of instrument performance and the calibration of all pesticides and Aroclors. Acceptable initial calibration is defined in paragraph of Initial Calibration below. Initial calibration must be repeated whenever the calibration verification stipulated in paragraph of Calibration Verification below, or when major instrument maintenance or modification is performed.

3. An instrument blank, a Performance Evaluation Mixture, and a second instrument blank and the mid-point concentration of Individual Standard Mixtures A and B are analyzed no less than once in every 12-hour analytical sequence in order to monitor retention times, calibration factors, and column performance. Data can be collected only as long as the results for these standards and instrument blanks fall within the limits defined in paragraph of Calibration Verification below. If two consecutive unacceptable standards are run, all extracts run since the previous acceptable standard must be re-analyzed. Additional standards and blanks are recommended when highly contaminated samples are suspected.

4. Calibration and run sequence specifications of the GC/EC method apply independently to both GC columns.

5. Matrix spike and a matrix spike duplicate analyses must be prepared and analyzed at least once for each matrix type or once per Sample Delivery Group (SDG), whichever is most frequent.

6. Analysis of a sample on both GC columns is required for all samples, blanks, matrix spikes, and matrix spike duplicates.

7. A single component pesticide is identified if a peak is detected within its appropriate retention time window on each of two columns. Toxaphene and Aroclors are identified primarily by pattern recognition, but RTs of three to five major peaks must also be taken into consideration. Guidance on quantitation of Aroclors is given beginning at paragraph 9 of Quantitation of Analyte.

8. Standards for all tentatively identified Aroclors must be run within 72 hours of the sample analysis in which they were observed. These standard are used to verify identification only; quantitation is based on the standard analyzed during initial calibration.

9. Quantitative analysis of pesticides/Aroclors must be accomplished by the external standard method. Three-point calibration curves for single component analytes and the surrogates must be generated during the initial calibration. A linear response range must be demonstrated from the CRQL to high point at least 16 times greater than the CRQL. Single-point calibrations for multi-component analytes are sufficient for quantitation by this method.

10. The ECD response for single component analytes must be within the three-point calibration range in order for quantitative measurements to be made. The ECD response for the Aroclors/toxaphene must not be larger than the response for the high point calibration analysis of the single component analytes. The extracts must be diluted if the ECD response exceeds the calibration range. Quantitation must be performed and reported for both GC columns.

11. Absolute retention times (RTs) are used for the identification of pesticides/Arclors. The absolute retention time window is calculated during initial calibration from the RT of the standard, using the retention time window specifications in paragraph 4 of Determination of Absolute Retention Times below.

Gas Chromatograph/Election Capture Detector (GC/EC)

1. Gas Chromatograph

   a. The gas chromatograph (GC) system must adequately regulate temperature in order to give a reproducible temperature program and have a flow controller that maintains a constant column flow rate throughout temperature program operations. The system must be suitable for splitless injection and have all required accessories including syringes, analytical columns, and gases.

   b. Gas chromatographs that are available from some manufacturers may have difficulty in meeting certain method QC requirements because of Endrin and DDT breakdown in the injector. This problem can be minimized by operating the injector at 200-205°C, using a Pyrex (not quartz) methyl silicone deactivated injector liner, and deactivating any metal parts in the injector with dichlorodimethyl silane. In some cases, using a 0.25 inch packed column injector converted for use with 0.53 mm capillary columns works better than a Grob-type injector. If a Grob-type injector is used, a 4 mm liner may be required to meet breakdown criteria.

2. Gas Chromatograph Columns

   a. Two wide-bore (0.53 mm ID) fused silica GC columna are required. A separate detector is required for each column. The specified analytical columns are a DB-1701, 30 m x 0.53 mm ID, 1.0 um film thickness, (J&W Scientific, Folsom, CA or equivalent) and a DB-608, 30 m x 0.53 mm ID, 0.5 to 1.0 um film thickness. Equivalent columns may be employed if they meet the requirements for resolution, initial calibration, and calibration verification listed in this sections.

   b. Columns are mounted in 0.25-inch injector ports by using glass adapters available from a variety of commercial sources. The two columns may be mounted into a single injection port with a tee adapter. Use of this adapter allows simultaneous injection onto both columns. The laboratory should follow manufacturer's recommendations for mounting 0.53 mm capillary columns in injector ports.

3. The carrier gas for routine application is helium. Laboratories may choose to use hydrogen as a carrier gas, but they must clearly identify its use in the non-conformance and on all divider pages preceding raw chromatographic data in submissions to the Agency. Laboratories that choose to use hydrogen are advised to exercise caution in its use. Use of a hydrogen leak detector is highly recommended when hydrogen is used as the carrier gas. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluorethylene (PTFF) thread sealants or flow controllers with rubber components are not to be used.

4. Electron Capture Detector - the make-up gas must be P-5, P-10 (argon/methane) or nitrogen according to the instrument specification. The linearity of the response of the ECD may be greatly dependent ont he flow rate of the make-up gas. Care must be taken to maintain stable and appropriate flow of make-up gas to the detector. The GC/EC system must be in a room in which the atmosphere has been demonstrated to be free of all contaminants which may interfere with the analysis. The instrument must be vented to outside the facility or to a trapping system which prevents the release of contaminants into the instrument room.

5. Data System - a data system must be interfaced to the GC/EC. The data system must allow the continuous acquisition of data throughout the duration of the chromatographic program and must permit, at the minimum, the output of time vs. intensity (peak height or peak area) data. Also, the data system must be able to rescale chromatographic data in order to report chromatograms meeting the requirements listed within this method.

Calibration Standards

1. Resolution Check Mixture - prepare the mixture of pesticides in hexane or iso-octane at the concentrations listed below. The mixture must be prepared every six months, or sooner if the solution has degraded or concentrated.

gamma-Chlordane	10.0 ng/ml	Endrin ketone	20.0 ng/ml
Endosulfan I	10.0 ng/ml	Methoxychlor	100.0 ng/ml
p,p'-DDE	20.0 ng/ml	Tetrachloro-m-xylene	20.0 ng/ml
Dieldrin	20.0 ng/ml	Decachlorobiphenyl	20.0 ng/ml
Endosulfan sulfate	20.0 ng/ml		

2. Performance Evaluation Mixture (PEM) - prepare the PEM in hexane or iso-octane at the concentration levels listed below. The PEM must be prepared weekly, or more often if the solution has degraded or concentrated.

gamma-BHC	10.0 ng/ml	Endrin	50.0 ng/ml
alpha-BHC	10.0 ng/ml	Methoxychlor	250.0 ng/ml
4,4'-DDT	100.0 ng/ml	Tetrachloro-m-xylene	20.0 ng/ml
beta-BHC	10.0 ng/ml	Decachlorobiphenyl	20.0 ng/ml

3. Individual Standard Mixtures A and B - the single components pesticide standards must be prepared in hexane or iso-octane at three concentrations for each analyte, including the surrogates. Two separate calibration mixtures, A and B, (listed below) are used to ensure that each peak is adequately resolved. The low point concentration corresponds to the CRQL for each analyte. The mid-point concentration must be 4 times the low point concentration. The high point concentration must be at least 16 times that of the low point, but a higher concentration may be chosen by the laboratory. The high point concentration defines the upper end of the concentration range for which the concentration is valid. The solution must be prepared every six months, or sooner if the solution has degraded or concentrated.

Individual Standard Mixture A - Low Point Concentration

alpha-BHC	5.0 ng/ml
Heptachlor	5.0 ng/ml
gamma-BHC	5.0 ng/ml
Endosulfan I	5.0 ng/ml
Dieldrin	10.0 ng/ml
Endrin	10.0 ng/ml

p,p'-DDD	10.0 ng/ml
p,p'-DDT	10.0 ng/ml
Methoxychlor	50.0 ng/ml
Tetrachloro-m-xylene	5.0 ng/ml
Decachlorobiphenyl	10.0 ng/ml

Individual Standard Mixture B- Low Point Concentration

beta-BHC	5.0 ng/ml
delta-BHC	5.0 ng/ml
Aldrin	5.0 ng/ml
Heptachlor expoxide	5.0 ng/ml
alpha-Chlordane	5.0 ng/ml
gamma-Chlordane	5.0 ng/ml
p,p'-DDE	10.0 ng/ml
Endosulfan sulfate	10.0 ng/ml
Endrin aldehyde	10.0 ng/ml
Endrin ketone	10.0 ng/ml
Endosulfan II	10.0 ng/ml
Terachloro-m-xylene	5.0 ng/ml
Decachlorobiphenyl	10.0 ng/ml

4. Multicomponent Standards - Toxaphene and Aroclor standards must be prepared individually except for Aroclor 1260 and Aroclor 1016 which may be combined in one standard mixture. The calibration standards for the Aroclors must be prepared at concentrations of 100 ng/ml, except for Aroclor 1221 which must be prepared at 200 ng/ml. Toxaphene must be prepared at 500 ng/ml. All multicomponent standards must contain the surrogates at 20.0 ng/ml. The Aroclor and Toxaphene solutions must be prepared in hexane or iso-octane. Each solution must be prepared every six months, or sooner if the solution has degraded or evaporated.

Gas Chromatograph Operating Conditions

The following are the gas chromatographic analytical conditions. The conditions are recommended unless otherwise noted.

Carrier Gas:	Helium (Hydrogen may be used, see paragraph 3 of GC/EC above)
Column Flow:	5 ml/min
Make-up Gas	P-5/P-10 or N_2 (required)
Injector Temperature	≥ 200°C
Injection:	On-column
Injection Volume:	1 or 2 ul (see paragraph 1 below)
Injector:	Grob-type, splitless
Initial Temperature:	150°C
Initial Hold Time:	1/2 min.
Temperature Ramp:	5°C to 6°C/min
Final Temperature:	275°C
Final Hold Time:	Until after Decachlorobiphenyl has eluted (approximately 10 minutes)

Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks and MS/MSDs.

The linearity of the ECD may be greatly dependent on the flow rate of the make-up gas. Care must be taken to maintain stable and appropriate flow of make-up gas to the detector.

1. Manual injections must be 2.0 ul. Auto injectors may use 1.0 ul volumes. The same injection volume must be used for all standards, blanks and samples.

Analysis Sequence for Standards and Samples

1. All acceptable samples must be analyzed within a valid analysis sequence as given below.

Time	Injection	Material Injected
	1 - 15	First 15 steps of the Initial Calibration
0 hr.	16	Instrument Blank at end of Initial Calibration
	17	PEM at end of Initial Calibration
	18	First Sample
	o	
	o	Subsequent Samples
	o	
12 hr.	o	Last Sample
	1st injection past 12:00 hr	Instrument Blank
	2nd and 3rd injections past 12:00 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	o	
	o	
Another 12 hr.	o	Last Sample
	1st injection past 12:00 hr.	Instrument Blank
	2nd injection	Performance Evaluation Mixture
	o	Sample
	o	
	o	Subsequent Samples
	o	
	o	
Another 12 hr.	o	Last Sample
	lst injection past 12:00 hr.	Instruction Blank
	2nd and 3rd injections past 12 hr.	Individual Standard Mixtures A and B
	o	Sample
	o	
	o	Subsequent Samples
	o	
	o	
	etc.	

NOTE: The first 12 hours are counted from the injection #16 (the Instrument Blank at the end of the initial calibration sequence), not from injection #1. Samples may be injected until 12:00 hours have elapsed. All subsequent 12-hour periods are timed from the injection of the instrument blank that

brackets the front end of the samples. Because the 12-hour time period is timed from injection of the instrument blank until the injection of the last sample, each 12-hour period may be separated by the length of one chromatographic run, that of the analysis of the last sample. While the 12-hour period may not be exceeded, the laboratory may run instrument blanks and standards more frequently, for instance to accommodate staff working on 8-hour shifts.

2. Before any samples are analyzed, it is necessary for the laboratory to complete an acceptable initial calibration sequence (see paragraph of Initial Calibration below).

3. After the initial calibration, the analysis sequence may continue as long as acceptable instrument blanks, Performance Evaluation Mixtures, and Individual Standard Mixtures A and B are analyzed at the required frequency (see paragraph of Calibration Verification). This analysis sequence shows only the minimum required blanks and standards. More blanks and standards may be run at the discretion of the laboratory; these must also satisfy the criteria presented in paragraph of Calibration Verification in order to continue the run sequence.

4. An analysis sequence must also include all required matrix spike/matrix spike duplicate analyses and method blanks, but the laboratory may decide at what point in the sequence they are to be analyzed.

5. A standard of any identified Aroclor must be run within 72 hours of its detection in a sample chromatogram.

Initial Calibration

1. Initial Calibration Sequence

   a. Before any samples are analyzed, it is necessary for the laboratory to complete the initial calibration sequence given below:

      NOTE: Steps 16 and 17 are used as part of the calibration verification as well (see paragraph of Calibration Verification below).

      Initial Calibration Sequence.

      1. Resolution Check
      2. Performance Evaluation Mixture
      3. Aroclor 1016/1260
      4. Aroclor 1221
      5. Aroclor 1232
      6. Aroclor 1242
      7. Aroclor 1248
      8. Aroclor 1254
      9. Toxaphene
      10. Low Point Standard A
      11. Low Point Standard B
      12. Midpoint Standard A
      13. Midpoint Standard B
      14. High Point Standard A
      15. High Point Standard B
      16. Instrument Blank
      17. Performance Evaluation Mixture

b. Samples may be analyzed only after the initial calibration acceptance criteria (see paragraph 2 below) are met. Otherwise, the analytical system is not functioning adequately for use with this protocol.

c. The initial calibration may continue to be used as long as the analytical system remains under control. The proof that the analytical system is under control is provided by the analyses of the Performance Evaluation Mixtures. If those analyses do not meet the criteria described in paragraph of Calibration Verification, appropriate corrective action must be taken, and the initial calibration sequence must be repeated. The calibration sequence must also be repeated if any major change in instrument hardware or instrument parameters is made (e.g., if a new column is installed or if the detector temperature is changed).

2. Initial Calibration Acceptance Criteria (apply to each GC column independently).

a. The initial calibration sequence must be analyzed in the order listed in paragraph 1 above using the optimized GC/EC operating conditions described in paragraph of Gas Chromatograph Operating Conditions above. The standards must be prepared according to paragraph of Calibration Standards. Calculate the calibration factors and retention times according to paragraphs of Determination of Absolute Retention Times and of Calibration Factors below.

b. The resolution criterion is that the depth of the valley between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60.0% of the height of the shorter peak. The poorest resolution on the DB-608 column probably will be between DDE and Dieldrin, between Methoxychlor and Endrin ketone and between Endosulfan I and gamma-chlordane. On the DB-1701 column, resolution difficulties most frequently occur between Endosulfan I and gamma-Chlordane, and between Methoxychlor and Endosulfan sulfate.

c. The breakdown of DDT and Endrin in both of the Performance Evaluation Mixtures must be less than 20.0 percent, and the combined breakdown of DDT and Endrin must be less than 30.0 percent where,

EQ.2

$$\% \text{ Breakdown DDT} = \frac{\text{Amount found in ng (DDD + DDE) * 100}}{\text{Amount in ng of DDT injected}}$$

EQ.3

$$\% \text{ Breakdown Endrin} = \frac{\text{Amount found in ng (Endrin aldehyde + Endrin Ketone) * 100}}{\text{Amount of Endrin injected in ng}}$$

EQ.4

$$\text{Combined \% Breakdown} = \%\text{Breakdown DDT} + \%\text{Breakdown Endrin}$$

d. All peaks in both of the Performance Evaluation Mixtures must be 100 percent resolved on both columns.

e. The absolute retention times of each of the single component pesticides and surrogates in both of the PEMs must be within the retention time windows determined from the three-point initial calibration, in paragraph 4 of Determination of Absolute Retention Times.

f. The relative percent difference of the calculated amount of the true amount for each of the single component pesticides and surrogates in both of the PEMs must be less than or equivalent to 25.0 percent, using equation 5.

g. At least one chromatogram from each of the two Individual Standard Mixtures A and B, run during the initial calibration, must yield peaks that give recorder deflections of 50 to 100 percent of full scale.

h. The resolution between any two adjacent peaks in the midpoint concentration of Individual Standard Mixtures A and B in the initial calibration must be greater than or equal to 90.0 percent.

i. The % RSD of the calibration factor for each single component target compound must be less than or equal to 20.0 percent, except as noted below. The % RSD of the calibration factor for the two surrogates must be less than or equal to 30.0 percent. Up to two single component target compounds (but not surrogates) per column may exceed the 20.0 percent limit for % RSD, but those compounds must have a % RSD of less than or equal to 30.0 percent.

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1} \right|^{1/2}$$

Where,

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

3. Corrective Action

a. If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

b. Contamination should be suspected as a cause if the detector cannot achieve acceptable linearity using this method. In the case of light contamination, baking out the detector at an elevated temperature (350°C) should be sufficient to achieve acceptable performance. In the case of heavy contamination passing hydrogen through the detector 1-2 hours at an elevated temperature may correct the problem.

c. If a laboratory cleans out a detector using an elevated temperature, the ECD electronics must be turned off during the bake out procedure.

d. After bake out or hydrogen reduction, the detector must be recalibrated using the initial calibration sequence.

e. Initial calibration technical acceptance criteria MUST be met before any samples or required blanks are analyzed. Any samples or required blanks analyzed after the initial calibration criteria have not been met will require reanalysis at no additional cost to the Agency.

Calibration Verification

1. Three types of analyses are used to verify the calibration and evaluate instrument performance. The analyses of instrument blanks, Performance Evaluation Mixtures (PEM), and the mid-point concentration of Individual Standard Mixtures A and B constitute the continuing calibration. Sample data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEM, and both Individual Standard Mixtures A and B.

2. An instrument blank and the Performance Evaluation Mixture must bracket one end of a 12-hour period during which sample data are collected, and a second instrument blank and the mid-point concentration of Individual Standard Mixtures A and B must bracket the other end of the 12-hour period.

3. For the 12-hour period immediately following the initial calibration sequence, the instrument blank and the PEM that are the last two steps in the initial calibration sequence bracket the front end of that 12-hour period. The injection of the instrument blank starts the beginning of that 12-hour period (see paragraph of Analysis Sequence for Standards and Samples). Samples may be injected for 12 hours from the injection of the instrument blank. The three injections immediately after that 12-hour period must be an instrument blank, Individual Standard Mixture A, and Individual Standard Mixture B. The instrument blank must be analyzed first, before either standard. The Individual Standard Mixtures may be analyzed in either order (A,B or B,A).

4. The analyses of the instrument blank and Individual Standard Mixtures A and B immediately following one 12-hour period may be used to begin the subsequent 12-hour period, provided that they meet the acceptance criteria in paragraphs 8-14 of this section. In that instance, the subsequent 12-hour period must be bracketed by the acceptable analyses of an instrument blank and PEM, in that order. Those two analyses may in turn be used to bracket the front end of yet another 12-hour period. This progression may continue every 12 hours until such time as any of the instrument blanks, PEMs or Individual Standard Mixtures fails to meet the acceptance criteria in paragraphs 8-14 of this section. The 12-hour time period begins with the injection of the instrument blank. Standards (PEM or Individual Standard Mixtures), samples and required blanks may be injected for 12:00 hours from the time of injection of the instrument blank.

5. If more than 12 hours have elapsed since the injection of the instrument blank that bracketed a previous 12-hour period, an acceptable instrument blank and PEM must be analyzed in order to start a new sequence. This requirement applies even if no analyses were performed since that standard(s) was injected.

6. After a break in sample analyses, the laboratory may only resume the analysis of samples using the current initial calibration for quantitation by analyzing an acceptable instrument blank and a PEM.

7. If the entire 12-hour period is not required for the analyses of all samples to be reported and all data collection is to be stopped, the incomplete sequence must be ended with either the instrument blank/PEM combination or the instrument blank/Individual Standard Mixtures A and B combination, whichever was due to be performed at the end of 12-hour period.

8. Analysts are cautioned that running an instrument blank and a performance evaluation mixture once every 12 hours is the minimum contract requirement. Late eluting peaks may carry over from one injection to the next if highly complex samples are analyzed or if the GC conditions are unstable. Such carryover is unacceptable. Therefore, it may be necessary to run instrument blanks and performance evaluation mixtures more often to avoid discarding data.

9. The requirements for running the instrument blanks, Performance Evaluation Mixture, and Individual Standard Mixtures A and B are waived when no samples, method blanks, or matrix spikes are run during that 12-hour period. After a break in sample analysis, a laboratory may resume the analysis of samples, method blanks, and matrix spikes and may use the current initial calibration for quantitation only after an acceptable PEM is run (paragraphs 2-6 of this section). If a successful PEM cannot be run before an interruption, an acceptable initial calibration must be run before sample data may be collected. All acceptable sample analyses must be bracketed by acceptable performance evaluation mixtures and instrument blanks.

10. Technical Acceptance Criteria (apply to each GC column independently)

    a. All single component pesticides and surrogates in the Performance Evaluation Mixtures used to demonstrate continuing calibration must be 100 percent resolved. The resolution between any two adjacent peaks in the mid-point concentrations of Individual Standard Mixtures A and B in the initial calibration must be greater than or equal to 90.0 percent.

    b. The absolute retention time for each of the single component pesticides and surrogates in the PEMs and mid-point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be within the retention time window determined from the three-point initial calibration in paragraph 4 of Determination of Absolute Retention Times.

    c. The relative percent difference of the calculated amount and the true amount for each of the single component pesticides and surrogates in the PEM and mid-point concentration of the Individual Standard Mixtures used to demonstrate continuing calibration must be less than or equal to 25.0 percent, using Equation 5.

EQ.5

$$RPD = \frac{|C_{nom} - C_{calc}|}{C_{nom}}$$

C_{nom} = true concentration of each analyte

C_{calc} = calculated concentration of each analyte from the analyses of the standard.

Note: The vertical bars in the equation indicate the absolute value, hence RPD is always a positive number.

    d. The percent breakdown of DDT and Endrin in the PEM must be less than or equal to 20.0 percent on both columns. The combined breakdown of DDT and Endrin must be less than or equal to 30.0 percent on both columns.

    e. All instrument blanks must meet the acceptance criteria in paragraph of Instrument Blank below.

11. Corrective Action

a. If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective action to achieve the acceptance criteria.

b. Major corrective actions such as replacing the GC column or baking out the detector will require that a new initial calibration be performed and meets the technical acceptance criteria in paragraph 2 of Initial Calibration.

c. Minor corrective actions may not require performing a new initial calibration, provided that a new analysis of the standard (PEM or Individual Mixture) that originally failed the criteria and an associated instrument blank immediately after the corrective action do meet all the acceptance criteria.

d. If the analysis of the standard and instrument blank in paragraph c above fail any of the technical acceptance criteria, a new initial calibration must be performed.

Determination of Absolute Retention Times

1. During the initial calibration sequence, absolute retention times (RT) are determined for all single response pesticides, the surrogates, and at least three major peaks of each multi-component analyte.

2. For single component pesticides, an RT is measured in each of three calibration standards and the mean RT is calculated as the average of the three values. An RT is measured for the surrogates in each of the three analyses of Individual Mixture A during the initial calibration and the mean RT is calculated as the average of the three values.

3. A retention time window is calculated for each single component analyte and surrogate by using the list in paragraph 4 of this section. Windows are centered around the mean absolute retention time for the analyte established during the initial calibrations.

4. Retention time windows for single and multi-component analytes and surrogates.

Compound	Retention Time Window in Minutes
alpha-BHC	± 0.05
beta-BHC	± 0.05
gamma-BHC	± 0.05
delta-BHC	± 0.05
Heptachlor	± 0.05
Aldrin	± 0.05
alpha-chlorodane	± 0.07
gamma-Chlorodane	± 0.07
Heptachlor epoxide	± 0.07
Dieldrin	± 0.07
Endrin	± 0.07
Endrin aldehyde	± 0.07
Endrin ketone	± 0.07
DDD	± 0.07
DDE	± 0.07
DDT	± 0.07
Endosulfan I	± 0.07
Endosulfan II	± 0.07
Endosulfan sulfate	± 0.07

Methoxychlor	± 0.07
Aroclors	± 0.07
Toxaphene	± 0.07
Tetrachloro-m-xylene	± 0.05
Decachlorobiphenyl	± 0.10

5. For each multi-component analyte, the RTs for three to five peaks are calculated from the initial calibration standard analysis. An RT window of ±0.07 minutes is used for all multi-component analyte peaks.

6. Analytes are identified when peaks are observed in the RT window for the compound on both GC columns.

Calibration Factors for Single Component Pesticides

1. During the initial calibration sequence, the laboratory must establish the magnitude of the linear ECD response range for each single component pesticide and surrogate on each column and for each GC system. This is accomplished by analyzing the Individual Standard Mixtures A and B at three concentrations during the initial calibration sequence in paragraph of Initial Calibration.

2. The linearity of the instrument is determined by calculating a percent relative standard deviation (%RSD) of the calibration factors from a three-point calibration curve for each single component pesticide and surrogate. Either peak area or peak height may be used to calculate calibration factors used in the %RSD equation. For example, it is permitted to calculate linearity for Endrin based on peak area and to calculate linearity for Aldrin based on peak height. It is not permitted within a %RSD calculation for an analyte to use calibration factors calculated from both peak area and peak height. For example, it is not permitted to calculate the calibration factor for the low point standard for Endrin using peak height and calculate the mid-point and high point stand calibration factors for Endrin using peak area.

   a. Calculate the calibration factor for each single component pesticide and surrogate over the initial calibration range using Equation 6. The calibration factors for the surrogates are calculated from the three analyses of Individual Standard Mixture A only.

   b. Calculate the mean and the %RSD of the calibration factors for each single component pesticide and surrogate over the initial calibration range using Equations 7 and 8.

EQ.6

$$CF = \frac{\text{Peak Area (or Height) of the Standard}}{\text{Mass Injected (ng)}}$$

EQ.7

$$\overline{CF} = \sum_{i=1}^{n} \frac{CF_i}{n}$$

EQ.8

$$\% \text{ RSD} = \frac{\text{Standard Deviation}}{\overline{CF}} \times 100$$

CF = Calibration factor

Where,

$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1} \right|^{1/2}$$

Where,

x_i = each individual value used to calculate the mean

$\bar{x}$ = the mean of n values

n = the total number of values

c. The linearity of the calibration is considered acceptable when the % RSD of the three-point calibration is less than 20.0 percent except as noted in the following.

The % RSD of the two surrogates must be less than or equal to 30.0 percent. Up to two single component target compounds (but not surrogates) per column may exceed the 20.0 percent limit for % RSD., but those compounds must have a % RSD of less than or equal to 30.0 percent.

d. If the linearity requirements listed above are met, the calibration factor from the mid-point concentration standard is used for quantitation of each single component pesticide.

3. Sample analysis may not proceed until a satisfactory calibration has been demonstrated.

Calibration Factors for Toxaphene and Aroclors

1. Toxaphene and Aroclors require only a single-point calibration and they present special analytical difficulties. Because of the alternation of these materials in the environment, it is probable that samples which contain multi-component analytes will give patterns similar to, but not identical with, those of the standards.

2. A set of three to five major peaks is selected for each multi-component analyte. Retention time (see paragraph 4 of Determination of Absolute Retention Times above) and calibration factors are determined from the initial calibration analysis for each peak. Guidance for the choice of which peaks to use is given in paragraph 9 of Quantitation Analysis below.

Acceptance Criteria for Chromatograms of Calibration Standards

The identification of single component pesticides by gas chromatographic methods is based primarily on retention time data. The retention time of the apex of a peak can be verified only from an on-scale chromatogram. The identification of multi-component analytes is based primarily on recognition of patterns of retention times displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single component and multi-component analytes.

1. The chromatograms that result from the analyses of the Resolution Check Mixture, the Performance Evaluation Mixture, and Individual Standard Mixtures A and B during the initial calibration sequence must display the single compound analytes present in each standard at greater than 10 percent of full scale but less than 100 percent of full scale.

2. The chromatograms, for at least on of the three analyses each of Individual Standard Mixtures A and B from the initial calibration sequence, must display the single component analytes at greater than 50 percent and less than 100 percent of full scale.

3. The chromatograms of the standards for the multi-component analytes analyzed during the initial calibration sequence must display the peaks chosen for identification of each analyte at greater than 20 percent and less than 100 percent of full scale.

4. For any standard containing alpha-BHC, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

5. If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

6. If the chromatogram of any standard needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

Sample Analysis

1. Unless ambient temperature on-column injection is used, the injector must be heated to at least 200°C. The optimized gas chromatographic conditions from paragraph of Gas Chromatograph Operating Conditions above must be used.

2. The injection must be made on-column by using either automatic or manual injection. If auto injectors are used, 1.0 ul injection volumes may be used. Manual injection shall use at least 2.0 ul injection volumes. The same injection volume must be used for all standards, samples, and blanks, associated with the same initial calibration. If a single injection is used for two GC columns attached to a single injection port, it may be necessary to use an injection volume greater than 2 ul. However, the same injection volume must be used for all analyses.

3. Analysis of a sample on both GC columns is required for all samples, blanks, matrix spikes, and matrix spike duplicates.

4. The requirements for the analysis sequence apply to both GC columns and for all instruments for these analyses.

5. The laboratory will identify and quantitate analyte peaks based on RT and calibration factor established during the initial calibration sequence, as long as an acceptable calibration verification (see paragraph of Calibration Verification above) is performed every 12 hours.

6. The protocol is intended to achieve the quantitation limits shown in Table 1 whenever possible. If sample chromatograms have interfering peaks, a high baseline, or off-scale peaks, then those samples must be re-analyzed following dilution, further cleanup, or re-extraction. Samples which cannot be made to meet the given specifications after one re-extraction and three-step cleanup (GPC, Florisil, and sulfur

removal) are reported in the non-conformance and do not require further analysis. No limit is placed on the number of re-extractions of samples that may be required because of contaminated method blanks.

7. The sample must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography (defined below). If dilution is employed solely to bring a peak within the calibration range or to get a multi-component pattern on scale, the results for both the more and the less concentrated extract must be reported. The resulting changes in quantitation limits and surrogate recovery must be reported also for the diluted samples.

8. If the laboratory has reason to believe that diluting the final extracts will be necessary, an undiluted run may not be required. If an acceptable chromatogram (as defined below) is achieved with the diluted extract, an additional extract 10 times the concentration of the dilute sample must be injected and reported with the sample data.

9. No target analyte concentrations may exceed the upper limit of the initial calibration.

10. A standard for any identified multi-component analyte must be analyzed during a valid analytical sequence on the same instrument, within 72 hours of its detection in a sample.

11. The identification of single component by gas chromatographic methods is based primarily on retention time data. The retention time of the apex of a peak can be verified only from an on-scale chromatogram. The identification of multi-component analyte is based primarily on recognition of patterns of retention times displayed on a chromatogram. Therefore, the following requirements apply to all data presented for single components and multi-component analytes.

    a. When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low point standard of the initial calibration associated with those analyses.

    b. Chromatograms must display single component pesticides detected in the sample at less than full scale.

    c. Chromatograms must display the largest peak of any multi-component analyte detected in the sample of less than full scale.

    d. If an extract must be diluted, chromatograms must display single component pesticides between 10 and 100 percent of full scale.

    e. If an extract must be diluted, chromatograms must display the peaks for quantitation of multi-component analytes between 25 and 100 percent of full scale.

    f. For any sample, the baseline of the chromatogram must return to below 50 percent of full scale before the elution time of alpha-BHC, and return to below 25 percent of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl.

    g. If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

    h. If the chromatogram of any sample needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

Quantitation of Analytes

1. Quantitation of target analytes and surrogates must be performed and reported on both columns.

2. Analytes must be quantitated with an electronic integrator or with a laboratory data system. The analyst can use either peak height or peak area as the basis for quantitation. The use of an electronic integrator or a laboratory data system is required.

3. The chromatograms of all samples must be reviewed by a qualified pesticide analyst before they are reported.

4. In order to be quantitated, the detector response (peak area or peak height) of all of the single component analytes must lie between the response of the low and high concentrations in the initial calibration. If the analytes are detected below the CRQL, they are reported as present below the CRQL, and flagged accordingly. If they are detected at a level greater than the high calibration point, the sample must be diluted either to a maximum of 1:100,000 or until the response is within the linear range established during calibration. Guidance in performing dilutions and exceptions to this requirement are given below.

   a. If the response is still above the high calibration point after the dilution of 1:100,000, the laboratory shall contact the SMO immediately.

   b. Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

   c. The dilution factor chosen should keep the response of the largest peak for a target compound in the upper half of the initial calibration range of the instrument.

   d. Do not submit data for more than two analyses, i.e., the original sample extract and one dilution, or, if a screening procedure was employed, from the most concentrated dilution analyzed and one further dilution.

   e. Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within the calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis and note the problem in the non-conformance.

5. The concentration of the single component pesticides are calculated separately for both GC columns by using the following equations:

   a. Water EQ. 9

   $$\text{Concentration ug/l} = \frac{(A_x)(V_t)(D_f)}{(CF)(V_o)(V_i)}$$

   Where

   A_x = Area of the peak for the compound to be measured.

   CF = Calibration factor for the mid-point concentration external standard (area per ng)

V_o = Volume of water extracted in milliliters (ml)

V_i = Volume of extract injected in microliters (ul) (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto to each column.)

V_t = Volume of the concentrated extract in microliters (ul) (this volume must be 10000 ul, see paragraph c of Solvent Exchange into Hexane).

Df = Dilution Factor. The dilution factor for analysis of water samples by this method is defined as follows:

$$\frac{\text{ul most conc. extract used to make dilution + ul clean solvent}}{\text{ul most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

If GPC is performed on a water sample extract, V_t becomes 5000 ul, and a factor of 2 must be added to the numerator, as described below for soil/sediment samples.

b. Soil/Sediment

EQ. 10

$$\text{Concentration ug/Kg (Dry weight basis)} = \frac{(A_x)(V_t)(D_f)(2.0)}{(CF)(V_i)(W_s)(D)}$$

Where

A_x and CF are as given for water, above.

V_t = Volume of the concentrated extract in microliters (ul) (this volume must be 5000 ul, see paragraph c of Solvent Exchange into Hexane.

V_i = Volume of extract injected in microliters (ul) (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto to each column.)

$D = \frac{100 - \%\ \text{moisture}}{100}$

W_s = Weight of sample extracted in grams (g)

Df = Dilution Factor. The dilution factor for analysis of soil samples by this method is defined as follows:

$$\frac{\text{ul most conc. extract used to make dilution + ul clean solvent}}{\text{ul most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0

The factor of 2.0 in the numerator is used to account for the amount of extract that is not recovered from the mandatory use of GPC cleanup. Concentrating the extract collect after GPC to 5.0 ml rather than 10.0 ml for water samples not subjected to GPC (see paragraph c of Solvent Exchange into Hexane), maintains the sensitivity of the soil method comparable to that of the water method, but correction of the numerical result is still required.

c. Note that the calibration factors used for the quantitation of the single component pesticides are the calibration factors from the mid-point concentration standard for each analyte.

d. Because of the likelihood that compounds co-eluting with the target compounds will cause positive interferences and increase the concentration determined by the method, the lower of the two concentrations calculated for each single component pesticide is reported on From I. In addition, the concentrations calculated for both the GC columns are reported on Form X, along with a percent difference comparing the two concentrations. The percent difference is calculated according to Equation 11.

EQ. 11

$$\%D = \frac{Conc_H - Conc_L \times 100}{Conc_L}$$

Where,

$Conc_H$ = The higher of the two concentrations for the target compound in question.

$Conc_L$ = The question of the two concentrations for the target compound in question.

Note that using this equation will result in percent difference vales that are always positive. The value will also be greater than a value calculated using the higher concentration in the denominator, however, given the likelihood of a positive interference raising the concentration determined on one GC column, this is a conservative approach to comparing the two concentrations.

6. The concentrations of the surrogates are calculated separately for both GC columns in a similar manner as the other analytes, using Equations 9 and 10. Use the calibration factors from the mid-point concentration of Individual Standard Mixture A. The recoveries of the surrogates are calculated for both GC columns according to Equation 12.

EQ. 12

$$\text{Surrogate Percent Recovery} = \frac{Q_d \times 100}{Q_a}$$

Where,

Q_d = Quality determined by analysis

Q_a = Quantity added to sample/blank

The limits for the recovery of the surrogates are 60-150 percent for both surrogate compounds. As these limits are only advisory, no further action is required by the laboratory is required, however, frequent failures to meet the limits for surrogate recovery warrant investigation by the laboratory, and may result in questions from the Agency. Surrogate recovery data from both GC columns are reported.

7. The quantitative determination of Toxaphene or Aroclors is somewhat different from that of single component pesticides. Quantitation of peaks within the detector linear range CRQL to > 16 times CRQL is based on a single calibration point assuming linear detector response. Alternatively, a linear calibration range may be established during a run sequence by a three point calibration curve for any multi-component analyte. If the concentration is calculated to be 10^6 times the CRQL, the laboratory shall contact the SMO immediately.

8. The quantitation of Toxaphene or Aroclors must be accomplished by comparing the heights or the areas of each of the three to five major peaks of the multi-component analyte in the sample with the calibration factor for the same peaks established during the initial calibration sequence. The concentration of multi-component analytes is calculated by using Equations 9 and 10, where A_x is the area for each of the major peaks of the multi-component analyte. The concentration of each peak is determined and then a mean concentration for three to five major peaks is determined on both columns. The following table lists the number of potential quantitation peaks for each Aroclor and Toxaphene.

Analyte	No. of Potential Quantitation Peaks
Aroclor 1016/1260	5/5
Aroclor 1221	3
Aroclor 1232	4
Aroclor 1242	5
Aroclor 1248	5
Aroclor 1254	5
Toxaphene	4

9. The reporting requirements for Toxaphene and the Aroclors are similar to those for the single component analytes, except that the lower mean concentration (from three to five peaks) is reported on Form X, and the two mean concentrations are compared using Equation 11.

10. The choice of the peaks used for multi-component quantitation and the recognition of those peaks may be complicated by the environmental alteration of the Toxaphene or Aroclors, and by the presence of co-eluting analytes or matrix interferences, or both.

11. If more than one multi-component analyte is observed in a sample, the laboratory must choose separate peaks to quantitate the different multi-component analytes. A peak common to both analytes present in the sample must not be used to quantitate either compound.

Sample Data Acceptance Criteria

1. The requirements below apply to both columns, and quantitation must be performed on both GC columns and reported.

2. All samples must be analyzed as part of a valid analysis sequence (paragraph of Analysis Sequence for Standards and Samples above). They must be bracketed by acceptable instrument blanks (paragraph of Instrument Blanks below), acceptable Performance Evaluation Mixture, and acceptable Individual Standard Mixture A and B (paragraph of Calibration Verification above) that were analyzed at the required frequency.

3. The retention times for both of the surrogates must be within the retention time windows as calculated in paragraph of Determination of Absolute Retention Times above for both GC columns.

4. Reportable data for a sample must include a chromatogram in which a baseline returns to below 50 percent of full scale before the elution time of alpha-BHC, and to below 25 percent of full scale after alpha-BHC and before decachlorobiphenyl.

5. If dilution has been applied and if no peaks are detected above 25 percent of full scale, analysis of a more concentrated sample is required.

6. Reportable sample data must include chromatogram(s) which meet the criteria in paragraph 11 of Sample Analysis above.

Blanks

There are two types of blanks always required by this method: the method blank and the instrument blank. A separate sulfur cleanup blank may be required if all samples associated with a given method blank are not subjected to sulfur cleanup. Samples that are associated with a sulfur cleanup blank are also associated with the method blank with which they were extracted. Both the method and sulfur cleanup blanks must meet the respective acceptance criteria for the sample analysis acceptance criteria to be met.

1. Method blank

   a. Method blanks are spiked with the surrogate solution, extracted, cleaned up, and analyzed by following the same procedure that is used with the samples. A water method blank is one liter of reagent water treated as the water sample aliquot. A soil method blank is 30 g of sodium sulfate treated as the soil sample aliquot.

      Method blank analysis must be performed once for the following, whichever is most frequent, and analyzed on each GC/EC system used to analyze samples:

      - Each Case, OR
      - Each 14 calendar day period (7 calendar day period for 14-day data turnaround contracts) during which samples in a Case are received (said period beginning with the receipt of the first sample in that Sample Delivery Group), OR
      - Each 20 samples in a Case, including matrix spikes and re-analyses, that are of similar matrix (water or soil), OR
      - Whenever samples are extracted by the same procedure (separatory funnel, continuous liquid-liquid extraction, or sonication).

b. In order to be acceptable, a method blank analysis cannot contain any of the analytes listed in Table 1 at greater than the CRQL. The surrogate retention times must be within the retention time windows calculated from the initial calibration sequence mean retention time for both tetrachloro-m-xylene and decachlorobiphenyl.

c. All samples associated with an unacceptable method blank (see Form IV) must be re-extracted and re-analyzed at no additional cost to the Agency.

2. Sulfur Cleanup Blank

a. The sulfur cleanup blank is a modified form of the method blank. The sulfur cleanup blank is hexane spiked with the surrogates and passed through the sulfur cleanup procedure (see paragraph of Sulfur Removal above).

b. The sulfur cleanup blank is prepared when only part of a set of samples extracted together requires sulfur removal. A method blank is associated with the entire set of samples. The sulfur cleanup blank is associated with the part of the set which required sulfur cleanup. If all the samples associated with a given method blank are subjected to sulfur cleanup,then the method blank must be subjected to sulfur cleanup, and no separate sulfur cleanup blank is required.

c. In order to be acceptable, a sulfur blank analysis cannot contain any of the analytes listed in Table 1 at greater than the CRQL, assuming that the material in the sulfur blank resulted from the extraction of a 1 (one) L water sample. Calculate the concentration of each analyte using the Equation 9. Compare the results to the CRQL values for water samples in Table 1. The surrogate retention times must be within the retention time windows calculated from the initial calibration sequence mean retention time for both tetrachloro-m-xylene and decachlorobiphenyl.

d. All samples associated with an unacceptable sulfur bank (see Form IV) must be re-extracted and re-analyzed at no additional cost to the Agency.

3. Instrument Blank

a. An instrument blank is a hexane or iso-octane solution containing 20.0 ng/ml of tetrachloro-m-xylene and decachlorobiphenyl.

b. The first analysis in a 12-hour analysis sequence must be an instrument blank. All acceptable samples analyses are to be bracketed by acceptable instrument blanks, as described in paragraph 1 of Analysis Sequence for Standards and Samples.

c. An acceptable instrument blank must be analyzed within a 12-hour analysis sequence and must demonstrate that no analyte in Table 1 is detected at greater than 0.5 times the CRQL and that the surrogate retention times are within the retention time windows. For comparing the results of the instrument blank analysis to the CRQLs, assume that the material in the instrument resulted from the extraction of a one (1) L water sample and calculate the concentration of each analyte using Equation 9. Compare the results to one-half the CRQL values for water samples in Table 1.

d. If analytes are detected at greater than half the CRQL, or the surrogate RTs are outside the RT windows, all data collection must be stopped, and corrective action must be taken. Data for samples which were run between the last acceptable instrument blank and the unacceptable blank are considered suspect. An acceptable instrument blank must be run before additional

data are collected. After an acceptable instrument blank is run, all samples which were run after the last acceptable instrument blank must be re-injected during a valid run sequence at no additional cost to the Agency and must be reported.

e. Analysts are cautioned that running an instrument blank once every 12 hours is the minimum contract requirement. Late eluting peaks may carry over from one injection to the next if highly complex samples are analyzed or if the GC conditions are unstable. Such carryover is unacceptable. Therefore, it may be necessary to run instrument blanks more often to avoid discarding data.

Matrix Spike/Matrix Spike Duplicate

1. A matrix spike and matrix spike duplicate must be extracted and analyzed at least once with every 20 samples of each matrix. NOTE: There is not differentiation between "low" and "medium" soil samples in this method. Therefore only one soil MS/MSD is to be submitted per SDG.

2. The surrogate retention times must be within the retention time windows specified.

3. The percent recoveries and the relative percent difference between the recoveries of each of the six (6) compounds in the matrix spike samples will be calculated and reported by using the following equations:

EQ.12

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike sample result
SR = Sample result
SA = Spike added

EQ.13

$$RPD = \frac{|MSR - MSDR|}{1/2(MSR + MSDR)} \times 100$$

Where,

RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

4. The laboratory shall report matrix spike and matrix spike duplicate recoveries and percent difference values with the analytical results. The limits for matrix spike compound recovery and RPD are given below. As these limits are only advisory, no further action by the laboratory is required, however, frequent failures to meet the limits for recovery or RPD warrant investigation by the laboratory, and may result in questions from the Agency.

MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	% Recovery Water	RPD Water	%Recovery Soil	RPD Soil
gamma-BHC (Lindane)	56-123	15	46-127	50
Heptachlor	40-131	20	35-130	31
Aldrin	40-120	22	34-132	43
Dieldrin	52-126	18	31-134	38
Endrin	56-121	21	42-139	45
4,4'-DDT	38-127	27	23-134	50

PESTICIDE/AROCLOR QA/QC REQUIREMENTS

The purpose of this Section is to outline the minimum quality control(QC) operations necessary to satisfy the analytical requirements associated with the determination of the pesticide/Aroclor target compounds listed in Table 1.

These requirements include the following:

- GC Column Resolution
- GC/EC Initial and Continuing Calibration
- Determination of Retention Times and Retention Time Windows
- Analytical Sequence
- Blank Analyses
- Surrogate Recoveries
- Matrix Spike and Matrix Spike Duplicate Analyses

GC Column Resolution

Prior to initiating any data collection activities involving samples, blanks, or standards, it is necessary to establish that a given GC column meets the analyte resolution criteria specified in paragraph 2b of Initial Calibration above. The purpose of this resolution check is to demonstrate that at the time of the initial calibration, the GC column is capable of chromatographically resolving the target compounds. This is accomplished through the analysis of the Resolution Check Mixture which contains the nine target compounds that are most difficult to resolve.

1. The Resolution Check Mixture must be analyzed at the beginning of every initial calibration sequence, on each GC column and instrument used for analysis.

2. Additional resolution criteria apply to the target compounds in the standards used for initial calibration and calibration verification, as described in paragraphs of Initial Calibration and Calibration Verification above.

3. The documentation includes Form VI PEST-4, chromatograms and data system printouts for the analysis of the Resolution Check Mixture on each GC column and instrument used for analysis.

GC/EC Initial Calibration for Target Compounds and Surrogates

Prior to the analysis of samples and required blanks, the GC/EC system must be initially calibrated at a minimum of three concentrations to determine the linearity of response utilizing single component target compound and surrogate standards. Multi-component target compounds are calibrated at a single point.

1. The concentrations of the low point initial calibration standards for single component pesticide target compounds and surrogates are described in paragraph 3 of Calibration Standards above. The concentration of the mid-point initial calibration standards is specified in the same paragraph as 4 times the low point concentration. The concentration of the high point initial calibration standard must be at least 16 times the low point concentration, and may be higher as described in the same paragraph.

2. The standards are to be analyzed according to the procedures given in paragraph of Analysis Sequence for Standards and Samples above, using the GC operating conditions in paragraph GC Operating Conditions above, and at the frequency given in paragraph 1 of Initial Calibration above.

3. The calibration factors are determined according to the procedures in paragraphs of Calibration Factors above.

4. The initial calibration of the GC/EC is evaluated on the basis of the stability of the calibrations factors and retention times of each target compound and surrogate, described in paragraphs 2e to 2i of Initial Calibration.

5. The calibration is also evaluated on the basis of the extent of breakdown of two target compounds, Endrin and 4,4'-DDT, as described in paragraph 2c of Initial Calibration.

6. The documentation includes Form VI PEST, chromatograms and data system printouts of all standards for the pesticide/Aroclor calibration standards.

<u>GC/EC Continuing Calibration for Target Compounds and Surrogates</u>

Once the GC/EC system has been calibrated, the calibration must be verified each twelve (12) hour time period for each GC column and instrument used for analysis. The calibration is verified through the analysis of instrument blanks, Performance Evaluation Mixtures (PEM), and the mid-point concentrations of Individual Standard Mixtures A and B.

1. The concentrations of the PEM and Individual Standard Mixtures used for continuing calibration are given in paragraph 2 and 3 of Calibration Standards above.

2. The instrument blank is described in paragraph 3 of Blanks above.

3. The instrument blank and the standards must be analyzed once every twelve hours according to the procedures in paragraph of Analysis Sequence for Standards and Samples, bracketing the sample analyses, as described in paragraph of Calibration Verification.

4. The continuing calibration is evaluated on the basis of the stability of the retention times of the target compounds in the standards.

5. The continuing calibration is evaluated on the basis of the stability of the instrument response to the target compounds in the PEM, as judged by the reproducibility of the determinations of the concentrations of these compounds in the standard, as described in paragraph 10 of Calibration Verification.

6. The continuing calibration is evaluated on the basis of the extent of breakdown of two target compounds in the PEM, Endrin and 4,4'-DDT, as described in paragraph 10 of Calibration Verification.

7. The continuing calibration is evaluated on the basis of the levels of contamination that are found in the instrument blank, as described in paragraph 3 of Blanks.

8. The documentation includes From VII PEST, Form VIII PEST, chromatograms and data system printouts for all standards and instrument blanks analyzed.

Determination of Retention Times and Retention Time Windows

The identification of single component pesticides by gas chromatographic methods is based primarily on retention time data. The identification of multi-component analytes is based primarily on recognition of patterns of retention times displayed on a chromatogram. Therefore, the determination of retention times and retention time windows is crucial to the provision of valid data for these target compounds.

1. The identification of all target compounds analyzed by the procedures described in GC/EC Analysis of Pesticides and Aroclors is based on the use of absolute retention time. The mean retention time of each target compound, or each peak in a multi-component target compound, is determined from the initial calibration standards, according to the procedures outlined in paragraph of Determination of Absolute Retention Times.

2. The retention time window of each target compound peak is determined as described in Ex. D paragraph 4 of Determination of Absolute Retention Time.

3. The retention time shifts of the surrogates are used to evaluate the stability of the gas chromatographic system during analysis of samples and standards. The retention time of the surrogates must be within the retention time windows determined during the initial calibration.

4. The documentation includes Form VI PEST, Form VII PEST, Form VIII PEST, chromatograms and data system printouts for all standards for the Pesticide/Aroclor initial and continuing calibrations, on each instrument and GC column used for analysis.

Analytical Sequence

The standards and samples analyzed according to the procedures in GC/EC Analysis of Pesticides and Aroclors must be analyzed in a sequence described in paragraphs of Analysis Sequence for Standards and Samples. This sequence includes requirements that apply to the initial and continuing calibrations, as well as to the analysis of samples. The documentation includes Form VIII PEST.

Blank Analysis

Two types of blanks are required for analyses using the procedures in GC/EC Analysis of Pesticides and Aroclors. They are method blanks and instrument blanks. A third type of blank, a sulfur clean up blank, may be required.

1. A method blank is a volume of a clean reference matrix (deionized distilled water for water samples, or purified sodium sulfate for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

   a. The frequency of method blank extraction is described in paragraph 1a of Blanks.

   b. The method blank must be analyzed on each GC column and instrument used for the analysis of associated samples.

   c. For the purpose of this protocol, an acceptable method blank must meet the criteria in paragraph 1b of Blanks.

2. The instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

   a. The frequency of instrument blank analysis is part of the initial and continuing calibration requirements described in paragraphs of Analysis Sequence, Initial Calibration and Calibration Verification.

   b. For the purposes of this protocol, an acceptable instrument blank must meet the criteria in paragraph 1b of Blanks.

3. The sulfur cleanup blank is a volume of clean solvent spiked with the surrogates and carried through the sulfur clean up and analysis steps. The purpose of the sulfur clean up blank is to determine the levels of contamination associated with the separate sulfur clean up steps.

   a. The sulfur clean up blank is only required when all the samples associated with a particular method blank are <u>not</u> subjected to sulfur clean up, as described in paragraph 2b of Blanks.

   b. The sulfur clean up blank must be analyzed on all GC column and instruments used for analysis of samples that received sulfur clean up.

   c. For the purposes of this protocol, an acceptable sulfur clean up blank must meet the criteria in paragraph 2c of Blanks.

4. If a method blank exceeds the limits for contamination above, the laboratory must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for re-extraction and re-analysis of associated samples are given in paragraph 1c of Blanks.

5. If an instrument blank exceeds the limits for contamination above, the laboratory must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for re-analysis of associated samples are given in paragraph 3d of Blanks.

6. If a sulfur clean up blank exceeds the limits for contamination above, the laboratory must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. The requirements for re-extraction and re-analysis of associated samples are given in paragraph 2d of Blanks.

7. The documentation includes Form I PEST for the analysis of each type of blank; Form IV PEST, associating the samples and the method and sulfur clean up blank; Form VIII PEST, associating the samples and the instrument blanks; and chromatograms and GC/EC data system printouts for the analysis of each blank.

Surrogate Recoveries

The recoveries of the two surrogates are calculated from the analysis on each GC column of each sample, blank, matrix, spike and matrix spike duplicate. The purpose of the surrogates is to evaluate the preparation and analysis of samples.

1. The surrogates are added to each sample, blank, matrix spike, and matrix spike duplicate prior to extraction, at the concentrations described in paragraph 8d of Reagents.

2. The recoveries of the surrogates are calculated according to the procedures in paragraph 6 of Quantitation of Analytes.

3. The quality control limits for surrogate recovery, given in paragraph 6 of Quantitation of Analytes are 60-150 percent. These limits are only advisory, and no further action by the laboratory is required if the limits are exceeded, however, frequent failures to meet the limits for surrogate recovery warrant investigation by the laboratory, and may result in questions from the Agency.

4. The documentation includes Form II PEST, a chromatogram and a GC/EC data system printout for the analysis of each sample, blank, matrix spike, and matrix spike duplicate.

Matrix Spike and Matrix Spike Duplicate Analysis

In order to evaluate the effects of the sample matrix on the methods used for pesticide/Aroclor analyses, the Agency has described a mixture of pesticide/Aroclor target compounds to be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method.

1. The frequency of matrix spike and matrix spike duplicate (MS/MSD) analysis is described in paragraph 1 of Matrix Spike/Matrix Spike Duplicate.

2. The recoveries of the matrix spike compounds are calculated according to the procedures in paragraph 3 of Matrix Spike/Matrix Spike Duplicate. The relative percent difference for each spiked analyte between the results of the matrix spike and the matrix spike duplicate are calculated according to the procedures in paragraph 3 of Matrix Spike/Matrix Spike Duplicate.

3. The quality control limits for recovery and relative percent difference are give in paragraph 4 of Matrix Spike/Matrix Spike Duplicate. These limits are only advisory at this time, and no further action is required when the limits are exceeded.

4. The documentation includes Form I PEST for both the MS and MSD analyses, Form III PEST, and chromatograms and a GC/EC data system printout for each analysis.

TABLE 1

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Pesticides/Aroclors	CAS Number	Quantitation Limits* Water ug/l	Soil ug/Kg
98. alpha-BHC	319-84-6	0.05	1.7
99. beta-BHC	319-85-7	0.05	1.7
100. delta-BHC	319-86-8	0.05	1.7
101. gamma-BHC (Lindane)	58-89-9	0.05	1.7
102. Heptachlor	76-44-8	0.05	1.7
103. Aldrin	309-00-2	0.05	1.7
104. Heptachlor epoxide	1024-57-3	0.05	1.7
105. Endosulfan I	959-98-8	0.05	1.7
106. Dieldrin	60-57-1	0.10	3.3
107. 4,4'-DDE	72-55-9	0.10	3.3
108. Endrin	72-20-8	0.10	3.3
109. Endosulfan II	33213-65-9	0.10	3.3
110. 4,4'-DDD	72--54-8	0.10	3.3
111. Endosulfan sulfate	1031-07-8	0.10	3.3
112. 4,4'-DDT	50-29-3	0.10	3.3
113. Methoxychlor	72-43-5	0.50	17.0
114. Endrin ketone	53494-70-5	0.10	3.3
115. Endrin aldehyde	7421-36-3	0.10	3.3
116. alpha-Chlordane	5103-71-9	0.05	1.7
117. gamma-Chlordane	5103-74-2	0.05	1.7
118. Toxaphene	8001-35-2	5.0	170.0
119. Aroclor-1016	12674-11-2	1.0	33.0
120. Aroclor-1221	11104-28-2	2.0	67.0
121. Arcolor-1232	11141-16-5	1.0	33.0
122. Arcolor-1242	53469-21-9	1.0	33.0
123. Arcolor-1248	12672-29-6	1.0	33.0
124. Arcolor-1254	11097-69-1	1.0	33.0
125. Arcolor-1260	11096-82-5	1.0	33.0

*Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of Pesticides/Aroclors.

APPENDIX K

FREEDOM OF INFORMATION SEARCH VILLAGE OF FREEPORT

APPLICATION FOR ACCESS TO RECORDS

54/315/98

DATE: 12/5/96

TO: RECORD ACCESS OFFICER, VILLAGE OF FREEPORT

I hereby apply to inspect the following record(s) for 525 Ray St. - Nassau Uniforms (Address)

- [X] Certificate(s) of Occupancy
- [X] Survey(s)
- [X] Violations
- [X] Plans ☐ Original ☐ Other: (Specify plans desired)
- [X] Permits ☐ Original Dwelling ☐ Additional work done
- ☐ Other CO being amended.

[X] I wish to obtain copies of said records and understand that the cost to reproduce said records will be 25¢ per photocopy up to 9"x14', or the actual cost of reproduction for other records.

Jeff Bohlen (Print Applicant's name)

Jeff Bohlen (Applicant's signature)

33 Gerard St (Street Address)

VC Jason Environmental Ltd (Representing)

Huntington N.Y. 11743 (City, State, Zip)

351-3555 (Phone Number)

RECEIVED DEC 11 2 04 PM '96 CLERK'S OFFICE VILLAGE OF FREEPORT, N.Y.

(Do not write below this line)

___ APPROVED - records will be made available on or about_____

___ DENIED - for the reason(s) checked below:

___ Exempted from disclosure by state or federal statute

___ Disclosure would result in an unwarranted invasion of personal privacy

___ Disclosure would impair contract awards or collective bargaining agreements

___ Records are compiled for law enforcement purposes and, if disclosed, would

___ interfere with law enforcement and/or judicial proceedings

___ deprive a person of a right to fair trial

___ identify a confidential source or disclose confidential info re: investigation

___ reveal non-routine criminal investigative techniques or procedures

___ Disclosure would endanger the life of safety of any person

___ Records are inter/intra-agency communications that are not 1)statistical or factual tabulations or data, 2) instructions to staff that affect the public, 3) final agency policy or determinations, or 4) external audits

___ OTHER - called 1-15-97 will be here tomorrow

Anna Knoller (Authorized signature)

VC (Title)

12-11-96 (Date)

NOTICE: You have a right to appeal a denial of the application. You must appeal, in writing, to the Board of Trustees of the Village of Freeport within thirty (30) days of the date of the denial. If the Board further denies access the reasons will be given to you in writing within ten (10) business days of your denial of appeal.

VILLAGE OF FREEPORT
MUNICIPAL BUILDING
46 NORTH OCEAN AVENUE
FREEPORT, NEW YORK 11520
(516) 377-2241
(516) 377-2242
FAX (516) 377-2493

RICHARD R. WISSLER
MAYOR

JOSEPH MADIGAN
ACTING SUPERINTENDENT
BUILDING DEPARTMENT

RE: 525 RAY STREET, FREEPORT, NY

Listed below is the information requested:

C.O. #1846 attached

No violations

PERMIT NUMBER	DATE ISSUED	DATE COMPLETED	DESCRIPTION
218	9/22/48	C.O.	Dry Cleaning
356	8/7/59	Cancelled	One-story addition
811	9/26/62	2/26/63	2-story masonry addition
1698	1/11/65	1/18/65	Stockade fence
1899	6/4/65	7/15/66	2nd story addition
2202	3/16/66	7/25/66	Replace & repair fire damage
4395	12/12/73	1/7/74	Install water tower on roof

Incorporated Village of Freeport
DEPARTMENT OF BUILDINGS

CERTIFICATE OF OCCUPANCY NO. 1846 *Dated* 12/19/52

THIS Certifies that THE BUILDING LOCATED ON Sec. No. 54 *Block No.* 315 *Lot No.* 98-107

Nassau County Tax Map Address S/W C/O Ray Street & Westend Avenue

Location 525 Ray Street

issued according to the requirements of Building Permit No. 218 *Date* 9/22/48 *and in compliance with the Building Code of The Village of Freeport, Nassau County, NY. at the time of issuance.*

Zone Industrial and Residence A *Occupancy* C3.2 Dry Cleaning. **********

****

DUPLICATE _____ *AMENDED* X

Copy Prepared 1/14/97 *Reason* For correct address. **********

****

VARIANCE granted by the Zoning Board of Appeals _____

Superintendent of Buildings
Joseph Madigan

* This is not valid without the Village Seal.