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Appendix A to Part 425

Potassium Ferricyanide Titration Method

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Title 40

PART 425 - LEATHER TANNING AND FINISHING POINT SOURCE CATEGORY

Authority: 33 U.S.C. 1311, 1314 (b), (c), (e) and (g), 1316 (b) and (c), 1317 (b) and (c), 1318 and 1361.

Source: 47 FR 52870, Nov. 23, 1982, unless otherwise noted.

GENERAL PROVISIONS

§ 425.01 Applicability.

This part applies to any leather tanning and finishing facility which discharges or may discharge process wastewater pollutants to the waters of the United States, or which introduces or may introduce process wastewater pollutants into a publicly owned treatment works.

§ 425.02 General definitions.

In addition to the definitions set forth in 40 CFR part 401, the following definitions apply to this part:

- (a) "Sulfide" shall mean total sulfide as measured by the potassium ferricyanide titration method described in appendix A or the modified Monier-Williams method described in appendix B.

- (b) "Hide" means any animal pelt or skin as received by a tannery as raw material to be processed.
- (c) "Retan-wet finish" means the final processing steps performed on a tanned hide including, but not limited to, the following wet processes: retan, bleach, color, and fatliquor.
- (d) "Hair pulp" means the removal of hair by chemical dissolution.
- (e) "Hair save" means the physical or mechanical removal of hair which has not been chemically dissolved, and either selling the hair as a by-product or disposing of it as a solid waste.
- (f) "Chrome tan" means the process of converting hide into leather using a form of chromium.
- (g) "Vegetable tan" means the process of converting hides into leather using chemicals either derived from vegetable matter or synthesized to produce effects similar to those chemicals.
- (h) "Raw material" means the hides received by the tannery except for facilities covered by subpart D and subpart I where "raw material" means the hide or split in the condition in which it is first placed into a wet process.
- (i) "Monthly average" means the arithmetic average of eight (8) individual data points from effluent sampling and analysis during any calendar month.
- (j) "Interference" means the discharge of sulfides in quantities which can result in human health hazards and/or risks to human life, and an inhibition or disruption of POTW as defined in 40 CFR 403.3(i).

[47 FR 52870, Nov. 23, 1982, as amended at 53 FR 9181, Mar. 21, 1988]

§ 425.03 Sulfide analytical methods and applicability.

- (a) The potassium ferricyanide titration method described in appendix A to part 425 shall be used whenever practicable for the determination of sulfide in wastewaters discharged by plants operating in all subcategories except the hair save or pulp, non-chrome tan, retan-wet finish subcategory (subpart C, see § 425.30). In all other cases, the modified Monier-Williams method as described in appendix B to part 425 shall be used as an alternative to the potassium ferricyanide titration method for the determination of sulfide in wastewaters discharged by plants operating in all subcategories except subpart C.
- (b) The modified Monier-Williams method as described in appendix B to part 425 shall be used for the determination of sulfide in wastewaters discharged by plants operating in the hair save or pulp, non-chrome tan, retan-wet finish subcategory (subpart C, see § 425.30).

[53 FR 9181, Mar. 21, 1988]

§ 425.04 Applicability of sulfide pretreatment standards.

- (a) A POTW receiving wastewater from a facility subject to this part may require more stringent pretreatment standards for sulfide than those established by this part without EPA approval.
- (b) The pretreatment standards for sulfide established by this part will not apply if the POTW receiving wastewater from a facility subject to this part certifies in writing with explanation of relevant factors considered, in accordance with the provisions of paragraph (c) of this section, that the discharge of sulfide from the facility does not interfere with the operation of the POTW. In making this determination, the POTW shall consider all relevant factors including but not limited to the following:
 - (1) The presence and characteristics, of other industrial wastewaters which can increase or decrease sulfide concentrations, pH, or both.
 - (2) The characteristics of the sewer/interceptor collection system which either minimize or enhance opportunities for release of hydrogen sulfide gas.
 - (3) The characteristics of the receiving POTWs headworks, preliminary and primary treatment systems, and sludge holding and dewatering facilities which either minimize or enhance opportunities for release of hydrogen sulfide gas.
 - (4) The occurrence of any prior sulfide related interference as defined in § 425.02(j).
- (c)

- (1) On October 13, 1983, a POTW which intends to certify that the sulfide pretreatment standard should not apply must publish, in a local newspaper with the largest circulation, a notice that presents the findings supporting this determination consistent with paragraph (a) of this section. Allowance for public hearing of these findings also must be provided. The POTW shall identify all existing facilities to which the sulfide pretreatment standard otherwise established by this part would not apply.
 - (2) On January 11, 1984, a POTW which intends to certify that the sulfide pretreatment standard should not apply must file a written certification with the Regional Water Management Division Director, Environmental Protection Agency, in the appropriate Regional Office. This certification shall include the findings supporting this determination and the results of public comments, and public hearing(s) if held.
 - (3) On February 10, 1984, EPA shall acknowledge to the POTW receipt of any certification submitted under paragraphs (c) (1) and (2) of this section, and shall indicate to the POTW the adequacy of the submission based upon a review of the factors set forth in paragraph (b) of this section.
 - (4) Within 30 days of the date of receipt of adequate submissions under paragraphs (c) (1), (2), and (3) of this section, EPA shall publish a notice in the *FEDERAL REGISTER* identifying those facilities to which the sulfide pretreatment standards of this part shall not apply.
 - (5) A POTW may certify that the sulfide pretreatment standards of this part should not apply to a new source planning to discharge into the POTW. This certification must be submitted prior to the commencement of discharge, and must conform at a minimum with criteria in paragraph (b) of this section and the general procedures and intervals of time contained in paragraphs (c) (1), (2), (3), and (4) of this section.
- (d)
- (1) If, after EPA and the POTW have determined in accordance with this section that the sulfide pretreatment standards of this part are not applicable to specified facilities, a POTW then determines that there have been changed circumstances (including but not limited to changes in the factors specified in paragraph (b) of this section) which justify application of the sulfide pretreatment standards, the POTW shall revoke the certification submitted under paragraph (c) of this section. The POTW and EPA shall then adhere to the general procedures and time intervals contained in paragraph (c) of this section in order to determine whether the sulfide pretreatment standards contained in this part are applicable.
 - (2) If pursuant to paragraph (d)(1) of this section, the sulfide pretreatment standards of this part are applicable to a specified facility, the indirect discharger shall comply with the sulfide pretreatment standards no later than 18 months from the date of publication of the *FEDERAL REGISTER* notice identifying the facility.
- (e) At any time after October 13, 1983, if a POTW determines that there have been changed circumstances (including but not limited to changes in the factors specified in paragraph (b) of this section), it may initiate proceedings contained in paragraph (c) of this section to determine that the sulfide pretreatment standards of this part shall not be applicable. The POTW and EPA shall follow the procedures and time intervals contained in paragraph (c) of this section to make this determination. A final determination that the sulfide pretreatment standards are not applicable must be made prior to the discharge of sulfide not in accordance with the standards set forth in this part.

(The information collection and reporting requirements contained in paragraphs (b) and (c) were approved by the Office of Management and Budget under control number 2040-0032)

[47 FR 52870, Nov. 23, 1982; 48 FR 30117, June 30, 1983, as amended at 48 FR 31404, July 8, 1983; 48 FR 32346, July 15, 1983; 48 FR 35649, Aug. 5, 1983; 53 FR 9181, Mar. 21, 1988]

§ 425.05 Compliance dates.

The compliance date for new source performance standards (NSPS) and pretreatment standards for new sources (PSSES) is the date the new source commences discharge. The compliance date for BPT effluent limitations and guidelines and pretreatment standards for existing sources is no later than March 31, 1989.

[53 FR 9182, Mar. 21, 1988]

§ 425.06 Monitoring requirements.

Compliance with monthly average discharge limitations is required regardless of the number of samples analyzed and averaged.

Subpart A - Hair Pulp, Chrome Tan, Retan-Wet Finish Subcategory

§ 425.10 Applicability; description of the hair pulp, chrome tan, retan-wet finishing subcategory.

The provisions of this subpart are applicable to process wastewater discharges resulting from any tannery which, either exclusively or in addition to other unhairing and tanning operation, processes raw or cured cattle or cattle-like hides into finished leather by chemically dissolving the hide hair, chrome tanning, and retan-wet finishing.

§ 425.11 Effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT):

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 pounds) of raw material	
BOD ₅	9.3	4.2
TSS	13.4	6.1
Oil & Grease	3.9	1.7
Total Chromium	0.24	0.09
pH	(¹)	(¹)

¹ Within the range of 6.0 to 9.0

[53 FR 9182, Mar. 21, 1988]

§ 425.12 Effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT): The effluent limitations are those for BOD₅, TSS, Oil and Grease, and pH contained in § 425.11.

§ 425.13 Effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT): The effluent limitations are those for Total Chromium contained in § 425.11.

§ 425.14 New source performance standards (NSPS).

Any new source subject to this subpart must achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or lb/1,000 lb) of raw material	
BOD ₅	6.0	2.7
TSS	8.7	4.0
Oil and grease	2.5	1.1
Total chromium	0.16	0.06
pH	(¹)	(¹)

¹ Within the range 6.0 to 9.0.

[47 FR 52870, Nov. 23, 1982; 48 FR 30116, June 30, 1983]

§ 425.15 Pretreatment standards for existing sources (PSES).

- (a) Except as provided in § 425.04 and 40 CFR 403.7 and 403.13, any existing source subject to this subpart which introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Maximum for any 1 day	Maximum for monthly average
	Milligrams per liter (mg/l)	
Sulfide	24	
Total chromium	12	8
pH	(¹)	(¹)

¹ Not less than 7.0.

- (b) Any existing source subject to this subpart which processes less than 275 hides/day shall comply with § 425.15(a), except that the total chromium limitations contained in § 425.15(a) do not apply.

[47 FR 52870, Nov. 23, 1982; 48 FR 30116, June 30, 1983, as amended at 53 FR 9182, Mar. 21, 1988; 61 FR 35685, July 8, 1996]

§ 425.16 Pretreatment standards for new sources (PSNS).

Except as provided in 40 CFR 403.7 and 425.04, any new source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the pretreatment standards contained in § 425.15.

Subpart B - Hair Save, Chrome Tan, Retan-Wet Finish Subcategory

§ 425.20 Applicability; description of the hair save, chrome tan, retan-wet finish subcategory.

The provisions of this subpart are applicable to process wastewater discharges resulting from any tannery which processes raw or cured cattle or cattle-like hides into finished leather by hair save unhairing, chrome tanning, and retan-wet finishing.

§ 425.21 Effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT):

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kg (or pound per 1,000 lb) of raw material	
BOD ₅	8.2	3.7
TSS	11.8	5.4
Oil and grease	3.4	1.5
Total chromium	0.21	0.08
pH	(¹)	(¹)

¹ Within the range 6.0 to 9.0.

§ 425.22 Effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT): The effluent limitations are those for BOD₅, TSS, Oil and Grease, and pH contained in § 425.21.

§ 425.23 Effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT): The effluent limitations are those for Total Chromium contained in § 425.21.

§ 425.24 New source performance standards (NSPS).

Any new source subject to this subpart must achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kg (or pound per 1,000 lb) of raw material	
BOD ₅	6.9	3.1
TSS	9.9	4.5
Oil and grease	2.9	1.3
Total chromium	0.18	0.06

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
pH	(¹)	(¹)

¹ Within the range 6.0 to 9.0

§ 425.25 Pretreatment standards for existing sources (PSES).

Except as provided in § 425.04 and 40 CFR 403.7 and 403.13, any existing source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Maximum for any 1 day	Maximum for monthly average
	Milligrams per liter (mg/l)	
Sulfide	24	
Total Chromium	12	8
pH	(¹)	(¹)

¹ Not less than 7.0.

[47 FR 52870, Nov. 23, 1982, as amended at 61 FR 35685, July 8, 1996]

§ 425.26 Pretreatment standards for new sources (PSNS).

Except as provided in 40 CFR 403.7 and 425.04, any new source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the pretreatment standards contained in § 425.25.

Subpart C - Hair Save or Pulp, Non-Chrome Tan, Retan-Wet Finish Subcategory

§ 425.30 Applicability; description of the hair save or pulp, non-chrome tan, retan-wet finish subcategory.

The provisions of this subpart are applicable to process wastewater discharges resulting from any tannery which processes raw or cured cattle or cattle-like hides into finished leather by hair save or pulp unhairing, vegetable tanning or alum, syntans, oils and other agents for tanning, and retan-wet finishing.

§ 425.31 Effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT):

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 pounds) of raw material	
BOD ₅	6.7	3.0
TSS	9.7	4.4
Oil & Grease	2.8	1.3
Total Chromium	0.17	0.06
pH	(¹)	(¹)

¹ Within the range of 6.0 to 9.0

[47 FR 52870, Nov. 23, 1982, as amended at 53 FR 9182, Mar. 21, 1988]

§ 425.32 Effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT): The effluent limitations are those for BOD₅, TSS, Oil and Grease, and pH contained in § 425.31.

§ 425.33 Effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT): The effluent limitations are those for Total Chromium contained in § 425.31.

§ 425.34 New source performance standards (NSPS).

Any new source subject to this subpart must achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pound per 1,000 lb) of raw material	
BOD ₅	5.9	2.7
TSS	8.5	3.9
Oil and grease	2.4	1.1
Total chromium	0.15	0.06
pH	(¹)	(¹)

¹ Within the range 6.0 to 9.0.

§ 425.35 Pretreatment standards for existing sources (PSES).

- (a) Except as provided in § 425.04 and 40 CFR 403.7 and § 403.13, any existing sources subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES limitations	
	Maximum for any 1 day	Maximum for monthly average
	Milligrams per liter (mg/l)	
Sulfide	24	-
Total Chromium	12	8
pH	(¹)	(¹)

¹ Not less than 7.0.

- (b) Any existing source subject to this subpart which processes less than 350 hides/day shall comply with § 425.35(a), except that the Total Chromium limitations contained in § 425.35(a) do not apply.

[47 FR 52870, Nov. 23, 1982; 48 FR 36116, June 30, 1983, as amended at 53 FR 9182, Mar. 21, 1988]

§ 425.36 Pretreatment standards for new sources (PSNS).

Except as provided in 40 CFR 403.7 and § 425.04, any new source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the pretreatment standards contained in § 425.35.

Subpart D - Retan-Wet Finish-Sides Subcategory**§ 425.40 Applicability; description of the retan-wet finish-sides subcategory.**

The provisions of this subpart are applicable to process wastewater discharges resulting from any tannery which processes previously tanned hides and skins (grain side only) into finished leather by retan-wet finishing.

§ 425.41 Effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT):

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kg (or pounds per 1,000 pounds) of raw material	
BOD ₅	8.9	4.0
TSS	12.8	5.8
Oil & Grease	3.7	1.7

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
Total Chromium	0.23	0.08
pH	(¹)	(¹)

¹ Within the range of 6.0 to 9.0.

[47 FR 52870, Nov. 23, 1982, as amended at 53 FR 9182, Mar. 21, 1988]

§ 425.42 Effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT): The effluent limitations are those for BOD₅, TSS, Oil and Grease, and pH contained in § 425.41.

§ 425.43 Effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT): The effluent limitations are those for Total Chromium contained in § 425.41.

§ 425.44 New source performance standards (NSPS).

Any new source subject to this subpart must achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 pounds) of raw material	
BOD ₅	6.5	2.9
TSS	9.3	4.3
Oil & Grease	2.7	1.2
Total Chromium	0.17	0.06
pH	(¹)	(¹)

¹ Within the range of 6.0 to 9.0.

[47 FR 52870, Nov. 23, 1982, as amended at 53 FR 9182, Mar. 21, 1988]

§ 425.45 Pretreatment standards for existing sources (PSES).

Except as provided in 40 CFR 403.7 and 403.13, any existing source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Maximum for any 1 day	Maximum for monthly average
	Milligrams per liter (mg/l)	
Total Chromium	19	12
pH	(¹)	(¹)

¹ Within the range 6.0 to 10.0.

§ 425.46 Pretreatment standards for new sources (PSNS).

Except as provided in 40 CFR 403.7, any new source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the pretreatment standards contained in § 425.45.

Subpart E - No Beamhouse Subcategory

§ 425.50 Applicability; description of the no beamhouse subcategory.

The provisions of this subpart are applicable to process wastewater discharges resulting from any tannery which processes cattle hides, sheepskins, or splits (hair previously removed and pickled) into finished leather by chrome or non-chrome tanning, and retan-wet finishing.

§ 425.51 Effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT):

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 pounds) of raw material	
BOD ₅	8.0	3.6
TSS	11.6	5.3
Oil & Grease	3.4	1.5
Total Chromium	0.21	0.08
pH	(¹)	(¹)

¹ Within the range of 6.0 to 9.0.

[47 FR 52870, Nov. 23, 1982, as amended at 53 FR 9182, Mar. 21, 1988]

§ 425.52 Effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional control technology (BCT): The effluent limitations are those for BOD₅, TSS, Oil and Grease, and pH contained in § 425.51.

§ 425.53 Effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT): The effluent limitations are those for Total Chromium contained in § 425.51

§ 425.54 New source performance standards (NSPS).

Any new source subject to this subpart must achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 lb) of raw material	
BOD ₅	5.3	2.4
TSS	7.7	3.5
Oil & Grease	2.2	1.0
Total Chromium	0.14	0.05
pH	(¹)	(¹)

¹ Within the range 6.0 to 9.0.

§ 425.55 Pretreatment standards for existing sources (PSES).

Except as provided in 40 CFR 403.7 and 403.13, any existing source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Maximum for any 1 day	Maximum for monthly average
	Milligrams per liter (mg/l)	
Total chromium	19	12
pH	(¹)	(¹)

¹ Within the range 6.0 to 10.0.

§ 425.56 Pretreatment standards for new sources (PSNS).

Except as provided in 40 CFR 403.7, any new source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the pretreatment standards contained in § 425.55.

Subpart F - Through-the-Blue Subcategory

§ 425.60 Applicability; description of the through-the-blue subcategory.

The provisions of this subpart are applicable to process wastewater discharges resulting from any tannery which processes raw or cured cattle or cattle-like hides through the blue tanned state by hair pulp unhairing and chrome tanning; no retan-wet finishing is performed.

§ 425.61 Effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT):

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 pounds) of raw material	
BOD ₅	3.2	1.5
TSS	4.7	2.1
Oil & Grease	1.4	0.61
Total Chromium	0.08	0.03
pH	(¹)	(¹)

¹ Within the range of 6.0 to 9.0.

[47 FR 52870, Nov. 23, 1982, as amended at 53 FR 9183, Mar. 21, 1988]

§ 425.62 Effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT): The effluent limitations are those for BOD₅, TSS, Oil and Grease, and pH contained in § 425.61.

§ 425.63 Effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT): The effluent limitations are those for Total Chromium contained in § 425.61.

§ 425.64 New source performance standards (NSPS).

Any new source subject to this subpart must achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 pounds) of raw material	
BOD ₅	3.2	1.5
TSS	4.7	2.1
Oil & Grease	1.4	0.61
Total Chromium	0.08	0.03
pH	(¹)	(¹)

¹ Within the range of 6.0 to 9.0.

[47 FR 52870, Nov. 23, 1982, as amended at 53 FR 9183, Mar. 21, 1988]

§ 425.65 Pretreatment standards for existing sources (PSES).

Except as provided in § 425.04 and 40 CFR 403.7 and 403.13, any existing source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Maximum for any 1 day	Maximum for monthly average
	Milligrams per liter (mg/l)	
Sulfide	24	
Total chromium	12	8
pH	(¹)	(¹)

¹ Not less than 7.0.

[47 FR 52870, Nov. 23, 1982, as amended at 61 FR 35685, July 8, 1996]

§ 425.66 Pretreatment standards for new sources (PSNS).

Except as provided in 40 CFR 403.7 and 425.04, any new source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment must comply with 40 CFR part 403, and must achieve the pretreatment standards contained in § 425.65.

Subpart G - Shearling Subcategory

§ 425.70 Applicability; description of the shearling subcategory.

The provisions of this subpart are applicable to process wastewater discharges resulting from any tannery which processes raw or cured sheep or sheep-like skins with the wool or hair retained into finished leather by chrome tanning, and retan-wet finishing.

§ 425.71 Effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT):

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 pounds) of raw material	
BOD ₅	3.0	1.3
TSS	4.3	1.9
Oil & Grease	1.2	0.55
Total Chromium	0.08	0.03
pH	(¹)	(¹)

¹ Within the range of 6.0 to 9.0.

[47 FR 52870, Nov. 23, 1982, as amended at 53 FR 9183, Mar. 21, 1988]

§ 425.72 Effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT): The effluent limitations are those for BOD₅, TSS, Oil and Grease, and pH contained in § 425.71.

§ 425.73 Effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT): The effluent limitations are those for Total Chromium contained in § 425.71.

§ 425.74 New source performance standards (NSPS).

Any new source subject to this subpart must achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pound per 1,000 lb) of raw material	
BOD ₅	13.2	5.9
TSS	19.1	8.7
Oil and grease	5.6	2.5

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
Total chromium	0.34	0.12
pH	(¹)	(¹)

¹ Within the range 6.0 to 9.0.

§ 425.75 Pretreatment standards for existing sources (PSES).

Except as provided in 40 CFR 403.7 and 403.13, any existing source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Maximum for any 1 day	Maximum for monthly average
	Milligrams per liter (mg/l)	
Total chromium	19	12
pH	(¹)	(¹)

¹ Within the range 6.0 to 10.0.

§ 425.76 Pretreatment standards for new sources (PSNS).

Except as provided in 40 CFR 403.7, any new source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and must achieve the pretreatment standards contained in § 425.75.

Subpart H - Pigskin Subcategory

§ 425.80 Applicability; description of the pigskin subcategory.

The provisions of this subpart are applicable to process wastewater discharges resulting from any tannery which processes raw or cured pigskins into finished leather by chemically dissolving or pulping the hair and tanning with chrome, then retan-wet finishing.

§ 425.81 Effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT):

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kg (or pounds per 1,000 lb) of raw material	
BOD ₅	7.0	3.2

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
TSS	10.1	4.6
Oil and grease	3.0	1.3
Total chromium	0.18	0.07
pH	(¹)	(¹)

¹ Within the range 6.0 to 9.0.

[47 FR 52870, Nov. 23, 1982; 48 FR 30117, June 30, 1983]

§ 425.82 Effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT): The effluent limitations are those for BOD₅, TSS, Oil and Grease and pH contained in § 425.81.

§ 425.83 Effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT): The effluent limitations are those for Total Chromium contained in § 425.81.

§ 425.84 New source performance standards (NSPS).

Any new source subject to this subpart must achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 lb) of raw material	
BOD ₅	5.8	2.6
TSS	8.3	3.8
Oil and grease	2.4	1.1
Total chromium	0.15	0.05
pH	(¹)	(¹)

¹ Within the range 6.0 to 9.0.

§ 425.85 Pretreatment standards for existing sources (PSES).

Except as provided in § 425.04 and 40 CFR 403.7 and § 403.13, any existing source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Maximum for any 1 day	Maximum for monthly average
	Milligrams per liter (mg/l)	
Sulfide	24	
Total chromium	12	8
pH	(¹)	(¹)

¹ Not less than 7.0.

[47 FR 52870, Nov. 23, 1982, as amended at 61 FR 35685, July 8, 1996]

§ 425.86 Pretreatment standards for new sources (PSNS).

Except as provided in 40 CFR 403.7 and 425.04, any new source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the pretreatment standards contained in § 425.85.

Subpart I - Retan-Wet Finish-Splits Subcategory

§ 425.90 Applicability; description of the retan-wet finish-splits subcategory.

The provisions of this subpart are applicable to process wastewater discharges resulting from any tannery which processes previously unhaird and tanned splits into finished leather by retan-wet finishing.

§ 425.91 Effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best practicable control technology currently available (BPT):

Pollutant or pollutant property	BPT limitations	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 pounds) of raw material	
BOD ₅	5.8	2.6
TSS	8.3	3.8
Oil & Grease	2.4	1.1
Total Chromium	0.15	0.05
pH	(¹)	(¹)

¹ Within the range of 6.0 to 9.0.

[47 FR 52870, Nov. 23, 1982, as amended at 53 FR 9183, Mar. 21, 1988]

§ 425.92 Effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best conventional pollutant control technology (BCT): The effluent limitations are those for BOD₅, TSS, Oil and Grease, and pH contained in § 425.91.

§ 425.93 Effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT).

Except as provided in 40 CFR 125.30 through 125.32, any existing point source subject to this subpart must achieve the following effluent limitations representing the degree of effluent reduction attainable by the application of the best available technology economically achievable (BAT): The effluent limitations are those for Total Chromium contained in § 425.91.

§ 425.94 New source performance standards (NSPS).

Any new source subject to this subpart must achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Maximum for any 1 day	Maximum for monthly average
	Kg/kkg (or pounds per 1,000 lb) of raw material	
BOD ₅	3.5	1.6
TSS	5.1	2.3
Oil & Grease	1.5	0.66
Total Chromium	0.09	0.03
pH	(¹)	(¹)

¹ Within the range 6.0 to 9.0.

§ 425.95 Pretreatment standards for existing sources (PSES).

- (a) Except as provided in 40 CFR 403.7 and 403.13, any existing source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and must achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Maximum for any 1 day	Maximum for monthly average
	Milligrams per liter (mg/l)	
Total Chromium	19	12
pH	(¹)	(¹)

¹ Within the range 6.0 to 10.0.

- (b) Any existing source subject to this subpart which processes less than 3,600 splits/day shall comply with § 425.95(a), except that the total chromium limitations contained in § 425.95(a) do not apply.

[47 FR 52870, Nov. 23, 1982; 48 FR 30117, June 30, 1983, as amended at 53 FR 9183, Mar. 21, 1988]

§ 425.96 Pretreatment standards for new sources (PSNS).

Except as provided in 40 CFR 403.7, any new source subject to this subpart that introduces process wastewater pollutants into a publicly owned treatment works must comply with 40 CFR part 403, and achieve the pretreatment standards contained in § 425.95.

Appendix A to Part 425 - Potassium Ferricyanide Titration Method

Source

The potassium ferricyanide titration method is based on method SLM 4/2 described in "Official Method of Analysis," Society of Leather Trades' Chemists, Fourth Revised Edition, Redbourn, Herts., England, 1965.

Outline of Method

The buffered sulfide solution is titrated with standard potassium ferricyanide solution in the presence of a ferrous dimethylglyoxime ammonia complex. The sulfide is oxidized to sulfur. Sulfite interferes and must be precipitated with barium chloride. Thiosulfate is not titrated under the conditions of the determination (Charlot, "Ann. chim. anal.", 1945, 27, 153; Booth; "J. Soc. Leather Trades' Chemists," 1956, 40, 238).

Apparatus

Burette, 10 ml.

Reagents

1. Preparation of 0.02N potassium ferricyanide: Weigh to the nearest tenth of a gram 6.6 g. of analytical reagent grade potassium ferricyanide and dissolve in 1 liter distilled water. Store in an amber bottle in the dark. Prepare fresh each week.
2. Standardization of ferricyanide solution: Transfer 50 ml. of solution to a 250 ml. Erlenmeyer flask. Add several crystals of potassium iodide (about 1 g.), mix gently to dissolve, add 1 ml. of 6N hydrochloric acid, stopper the flask, and swirl gently. Let stand for two minutes, add 10 ml. of a 30 percent zinc sulfate solution, and titrate the mixture containing the gelatinous precipitate with standardized sodium thiosulfate or phenylarsine oxide titrant in the range of 0.025-0.050N. Add 1 ml. of starch indicator solution after the color has faded to a pale yellow, and continue the titration to the disappearance of the blue color. Calculate the normality of the ferricyanide solution using the equation:
3. Preparation of 6M ammonium chloride buffer, pH 9.3: Dissolve 200 g. ammonium chloride in approximately 500 ml. distilled water, add 200 ml. 14M reagent grade ammonium hydroxide and make up to 1 liter with distilled water. The buffer should be prepared in a hood. Store in a tightly stoppered container.
4. Preparation of 0.05M barium chloride solution: Dissolve 12-13 g. barium chloride dihydrate in 1 liter of distilled water.
5. Preparation of ferrous dimethylglyoxime indicator solution: Mix 10 ml. 0.6 percent ferrous sulfate, 50 ml. 1 percent dimethylglyoxime in ethanol, and 0.5 ml. concentrated sulfuric acid.
6. Preparation of stock sulfide standard, 1000 ppm: Dissolve 2.4 g. reagent grade sodium sulfide in 1 liter of distilled water. Store in a tightly stoppered container. Diluted working standards must be prepared fresh daily and their concentrations determined by EPA test procedure 376.1 (see 40 CFR 136.3, Table IB, parameter 66 (49 FR 43234, October 26, 1984, with correction notice at 50 FR 690, January 4, 1985)) immediately prior to use.
7. Preparation of 10N NaOH: Dissolve 400 g. of analytical reagent grade NaOH in 1 liter distilled water.

Sample Preservation and Storage

Samples are to be field filtered (gravity or pressure) with coarse filter paper (Whatman 4 or equivalent) immediately after collection. Filtered samples must be preserved by adjustment to pH>12 with 10N NaOH. Sample containers must be covered tightly and stored at 4 °C until analysis. Samples must be analyzed within 48 hours of collection. If these procedures cannot be achieved, it is the laboratory's responsibility to institute quality control procedures that will provide documentation of sample integrity.

Procedure

1. Transfer 100 ml. of sample to be analyzed, or a suitable portion containing not more than 15 mg. sulfide supplemented to 100 ml. with distilled water, to a 250 ml. Erlenmeyer flask.
2. Adjust the sample to pH 8.5-9.5 with 6N HCl.
3. Add 20 ml. of 6M ammonium chloride buffer (pH 9.3), 1 ml. of ferrous dimethylglyoxime indicator, and 25 ml. of 0.05M barium chloride. Mix gently, stopper, and let stand for 10 minutes.
4. After 10 minutes titrate with standardized potassium ferricyanide to disappearance of pink color. The endpoint is reached when there is no reappearance of the pink color after 30 seconds.

Calculation and Reporting of Results.

where A = volume in ml. of potassium ferricyanide solution used,

and B = normality of potassium ferricyanide solution.

2. Report results to two significant figures.

Quality Control

1. Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of replicate and spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within precision and accuracy limits expected of the method.
2. Before performing any analyses, the analyst must demonstrate the ability to generate acceptable precision and accuracy with this method by performing the following operations.
 - (a) Perform four replicate analyses of a 20 mg./l. sulfide standard prepared in distilled water (see paragraph 6 under "Reagents" above).
 - (b)
 - (1) Calculate clean water precision and accuracy in accordance with standard statistical procedures. Clean water acceptance limits are presented in paragraph 2(b)(2) below. These criteria must be met or exceeded before sample analyses can be initiated. A clean water standard must be analyzed with each sample set and the established criteria met for the analysis to be considered under control.
 - (2) Clean water precision and accuracy acceptance limits: For distilled water samples containing from 5 mg./l. to 50 mg./l. sulfide, the mean concentration from four replicate analyses must be within the range of 50 to 110 percent of the true value.
3. The Method Detection Limits (MDL) should be determined periodically by each participating laboratory in accordance with the procedures specified in "Methods for Chemical Analysis of Municipal and Industrial Wastewater," EPA-660/4-82-057, July 1982, EMSL, Cincinnati, OH 45268. For the convenience of the user, these procedures are contained in appendix C to part 425.

4. A minimum of one spiked and one duplicate sample must be performed for each analytical event, or five percent spikes and five percent duplicates when the number of samples per event exceeds twenty. Spike levels are to be at the MDL (see paragraph 3 above for MDL samples) and at x where x is the concentration found if in excess of the MDL. Spike recovery must be 40 to 120 percent for the analysis of a particular matrix type to be considered valid. If a sample or matrix type provides performance outside these acceptance limits, the analyses must be repeated using the modified Monier-Williams procedures described in appendix B to this part.

5. Report results in mg./liter. When duplicate and spiked samples are analyzed, report all data with the sample results.

[53 FR 9183, Mar. 21, 1988]

Appendix B to Part 425 - Modified Monier-Williams Method

Outline of Method

Hydrogen sulfide is liberated from an acidified sample by distillation and purging with nitrogen gas (N_2). Sulfur dioxide interference is removed by scrubbing the nitrogen gas stream in a pH 7 buffer solution. The sulfide gas is collected by passage through an alkaline hydrogen peroxide scrubbing solution in which it is oxidized to sulfate. Sulfate concentration in the scrubbing solution is determined by either EPA gravimetric test procedure 375.3 or EPA turbidimetric test procedure 375.4 (see 40 CFR 136.3, Table IB, parameter 65 (49 FR 43234, October 26, 1984, and correction notice at 50 FR 690, January 4, 1985)).

Apparatus*

(See Figure 1.) * Catalogue numbers are given only to provide a more complete description of the equipment necessary, and do not constitute a manufacturer or vendor endorsement.

Heating mantel and control (VWR Cat. No. 33752-464)

1000 ml. distilling flask with three 24/40 joints (VWR Cat. No. 29280-215)

Friedricks condenser with two 24/40 joints (VWR Cat. No. 23161-009)

125 ml. separatory funnel with 24/40 joint (VWR Cat. No. 30357-102)

Inlet tube with 24/40 joint (VWR Cat. No. 33057-105)

Adapter joint 24/40 to 19/38 (VWR Cat. No. 62905-26)

Adsorber head (2 required) (Thomas Cat. No. 9849-R29)

Adsorber body (2 required) (Thomas Cat. No. 9849-R32)

Laboratory vacuum pump or water aspirator

Reagents

1. Potassium hydroxide, 6N: Dissolve 340 g. of analytical reagent grade KOH in 1 liter distilled water.

2. Sodium hydroxide, 6N: Dissolve 240 g. of analytical reagent grade NaOH in 1 liter distilled water.

3. Sodium hydroxide, 0.03N: Dilute 5.0 ml. of 6N NaOH to 1 liter with distilled water.

4. Hydrochloric acid, 6N: Dilute 500 ml. of concentrated HCl to 1 liter with distilled water.

5. Potassium phosphate stock buffer, 0.5M: Dissolve 70 g. of monobasic potassium phosphate in approximately 800 ml. distilled water. Adjust pH to 7.0 ± 0.1 with 6N potassium hydroxide and dilute to 1 liter with distilled water. Stock solution is stable for several months at 4 °C.

6. Potassium phosphate buffer, 0.05M: Dilute 1 volume of 0.5M potassium phosphate stock buffer with 9 volumes of distilled water. Solution is stable for one month at 4 °C.
7. Alkaline 3% hydrogen peroxide: Dilute 1 volume of 30 percent hydrogen peroxide with 9 volumes of 0.03N NaOH. Prepare this solution fresh each day of use.
8. Preparation of stock sulfide standard, 1000 ppm.: Dissolve 2.4 g. reagent grade sodium sulfide in 1 liter of distilled water. Store in a tightly stoppered container. Diluted working standards must be prepared fresh daily and their concentrations determined by EPA test procedure 376.1 immediately prior to use (see 40 CFR 136.3, Table IB, parameter 66 (49 FR 43234, October 26, 1984, and correction notice at 50 FR 690, January 4, 1985)).

Sample Preservation and Storage

Preserve unfiltered wastewater samples immediately after collection by adjustment to pH>9 with 6N NaOH and addition of 2 ml. of 2N zinc acetate per liter. This amount of zinc acetate is adequate to preserve 64 mg./l. sulfide under ideal conditions. Sample containers must be covered tightly and stored at 4 °C until analysis. Samples must be analyzed within seven days of collection. If these procedures cannot be achieved, it is the laboratory's responsibility to institute quality control procedures that will provide documentation of sample integrity.

Procedure (See Figure 1 for apparatus layout.)

1. Place 50 ml. of 0.05M pH 7.0 potassium phosphate buffer in Trap No. 1.
2. Place 50 ml. of alkaline 3 percent hydrogen peroxide in Trap No. 2.
3. Sample introduction and N₂ prepurge: Gently mix sample to be analyzed to resuspend settled material, taking care not to aerate the sample. Transfer 400 ml. of sample, or a suitable portion containing not more than 20 mg. sulfide diluted to 400 ml. with distilled water, to the distillation flask. Adjust the N₂ flow so that the impingers are frothing vigorously, but not overflowing. Vacuum may be applied at the outlet of Trap No. 2 to assist in smooth purging. The N₂ inlet tube of the distillation flask must be submerged deeply in the sample to ensure efficient agitation. Purge the sample for 30 minutes without applying heat. Test the apparatus for leaks during the prepurge cycle (Snoop or soap water solution).
4. Volatilization of H₂ S: Interrupt the N₂ flow (and vacuum) and introduce 100 ml. of 6N HCl to the sample using the separatory funnel. Immediately resume the gas flow (and vacuum). Apply maximum heat with the heating mantle until the sample begins to boil, then reduce heat and maintain gentle boiling and N₂ flow for 30 minutes. Terminate the distillation cycle by turning off the heating mantle and maintaining N₂ flow through the system for 5 to 10 minutes. Then turn off the N₂ flow (and release vacuum) and *cautiously* vent the system by placing 50 to 100 ml. of distilled water in the separatory funnel and opening the stopcock carefully. When the bubbling stops and the system is equalized to atmospheric pressure, remove the separatory funnel. Extreme care must be exercised in terminating the distillation cycle to avoid flash-over, draw-back, or violent steam release.
5. Analysis: Analyze the contents of Trap No. 2 for sulfate according to either EPA gravimetric test procedure 375.3 or EPA turbidimetric test procedure 375.4 (see 40 CFR 136.3, Table IB, parameter 65 (49 FR 43234, October 26, 1984, and correction notice at 50 FR 690, January 4, 1985)). Use the result to calculate mg./l. of sulfide in wastewater sample.

Calculations and Reporting of Results

1. Gravimetric procedure:
2. Turbidimetric procedure:

where A = mg./l. of sulfate in Trap No. 2

B = liquid volume in liters in Trap No. 2

and C = volume in ml. of waste sample distilled

3. Report results to two significant figures.

Quality Control

1. Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of replicate and spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within precision and accuracy limits expected of the method.

2. Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision by performing the following operations.

- (a) Perform four replicate analyses of a 20 mg./l. sulfide standard prepared in distilled water (see paragraph 8 under "Reagents" above).
- (b)
 - (1) Calculate clean water precision and accuracy in accordance with standard statistical procedures. Clean water acceptance limits are presented in paragraph 2(b)(2) below. These criteria must be met or exceeded before sample analyses can be initiated. A clean water standard must be analyzed with each sample set and the established criteria met for the analyses to be considered under control.
 - (2) Clean water precision and accuracy acceptance limits: For distilled water samples containing from 5 mg./l. to 50 mg./l. sulfide, the mean concentration from four replicate analyses must be within the range of 72 to 114 percent of the true value.

3. The Method Detection Limit (MDL) should be determined periodically by each participating laboratory in accordance with the procedures specified in "Methods for Chemical Analysis of Municipal and Industrial Wastewater," EPA-600/4-82-057, July 1982, EMSL, Cincinnati, OH 45268. For the convenience of the user, these procedures are contained in appendix C to part 425.

4. A minimum of one spiked and one duplicate sample must be run for each analytical event, or five percent spikes and five percent duplicates when the number of samples per event exceeds twenty. Spike levels are to be at the MDL (see paragraph 3 above for MDL samples) and at x when x is the concentration found if in excess of the MDL. Spike recovery must be 60 to 120 percent for the analysis of a particular matrix type to be considered valid.

5. Report all results in mg./liter. When duplicate and spiked samples are analyzed, report all data with the sample results.

[53 FR 9184, Mar. 21, 1988]

Appendix C to Part 425 - Definition and Procedure for the Determination of the Method Detection Limit¹

The method detection limit (MDL) is defined at the minimum concentration of a substance that can be identified, measured and reported with 99 percent confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

Procedure

1. Make an estimate of the detection limit using one of the following:

- (a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. If the criteria for qualitative identification of the analyte is based upon pattern recognition techniques, the least abundant signal necessary to achieve identification must be considered in making the estimate.
- (b) The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.
- (c) The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations, *i.e.*, a break in the slope of the standard curve.
- (d) The concentration value that corresponds to known instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent water (blank), prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL. (Recommended between 1 and 5 times the estimated MDL.) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL, proceed to Step 4.

If the measured concentration of analyte is less than the estimated MDL, add a known amount of analyte to bring the concentration of analyte to between one and five times the MDL. In the case where an interference is coanalyzed with the analyte:

If the measured level of analyte is greater than five times the estimated MDL, there are two options:

- (1) Obtain another sample of lower level of analyte in same matrix if possible.
- (2) The sample may be used as is for determining the MDL if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If blank measurements are required to calculate the measured level of analyte, obtain separate blank measurements for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less than five times the calculated MDL. To insure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:

- (1) If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.
- (2) If these measurements indicate the sample is not in the correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance (S^2) and standard deviation (S) of the replicate measurements, as follows:

where: the x_i , $i = 1$ to n are the analytical results in the final method reporting units obtained from the n sample aliquots and

refers to the sum of the X values from $i = 1$ to n .

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1, 1-\alpha=.99)} (S)$$

where:

MDL = the method detection

$t_{(n-1, 1-\alpha=.99)}$ = the students' t value appropriate for a 99 percent confidence level and a standard deviation estimate with $n-1$ degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95 percent confidence limits for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (X^2/df) and calculated as follows:

$$MDL_{LCL} = 0.69 MDL$$

$MDL_{UCL} = 1.92 MDL$ where MDL_{LCL} and MDL_{UCL} are the lower and upper 95 percent confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.

- (a) If this is the initial attempt to compute MDL based on the estimated MDL in Step 1, take the MDL as calculated in Step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with Step 4.
- (b) If the current MDL determination is an iteration of the MDL procedure for which the spiking level does not permit qualitative identification, report the MDL as that concentration between the current spike level and the previous spike level which allows qualitative identification.
- (c) If the current MDL determination is an iteration of the MDL procedure and the spiking level allows qualitative identification, use S^2 from the current MDL calculation and S^2 from the previous MDL calculation to compute the F ratio.

then compute the pooled standard deviation by the following equation:

respike at the last calculated MDL and process the samples through the procedure starting with Step 4.

- (d) Use the S_{pooled} as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681 (S_{pooled})$$

where 2.681 is equal to $t_{(12, 1-\alpha=.99)}$

- (e) The 95 percent confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

$$MDL_{LCL} = 0.72 MDL$$

$$MDL_{UCL} = 1.65 MDL$$

where LCL and UCL are the lower and upper 95 percent confidence limits respectively based on 14 aliquots.

Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with the MDL value. Report the mean analyte level with the MDL. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, report the mean recovery, and indicate if the MDL determination was iterated.

If the level of the analyte in the sample matrix exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

Reference

Glaser, J.A., Foerst, D.L., McKee, G.D., Quave, S.A., and Budde, W.L., "Trace Analysis for Wastewaters," Environmental Science and Technology, 15, 1426 (1981).

Table of Students' t Values at the 99 Percent Confidence Level

Number of replicates	Degrees of freedom (n-1)	$t_{(n-1, 1-\alpha = .99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
		2.326

[53 FR 9186, Mar. 21, 1988]

FOOTNOTES - APPENDIX C TO PART 425

[1] Source: "Methods for Chemical Analysis of Municipal and Industrial Wastewater," EPA-600/4-82-057, July 1982, EMSL, Cincinnati, OH 45268
