

## Chapter NR 252

### LEATHER TANNING AND FINISHING

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NR 252.40	Applicability; description of the retan-wet finish-sides subcategory.	NR 252.94	New source performance standards (NSPS).
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NR 252.50	Applicability; description of the no beamhouse subcategory.		

**NR 252.01 Purpose.** The purpose of this chapter is to establish effluent limitations, standards of performance, and pretreatment standards for discharges of process wastes from the leather tanning and finishing category of point sources and its subcategories.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.015 Applicability.** This chapter applies to any leather tanning and finishing facility which discharges or may discharge process wastewater pollutants to the waters of the state, or which introduces or may introduce process wastewater pollutants into a publicly owned treatment works.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.02 General definitions.** In addition to the definitions set forth in 40 CFR Part 401, the following definitions apply to this chapter:

(1) "Chrome tan" means the process of converting hide into leather using a form of chromium.

(2) "Existing source" means any source that is not a new source.

(3) "Hair pulp" means the removal of hair by chemical dissolution.

(4) "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.

(5) "Hide" means any animal pelt or skin as received by a tannery as raw material to be processed.

(6) "Interference" means the discharge of sulfides in quantities which can result in human health hazards and risks to human life, and an inhibition or disruption of a POTW as defined in 40 CFR 403.3 (i).

(7) "Monthly average" means the arithmetic average of 8 individual data points from effluent sampling and analysis during any calendar month.

(8) "New source," as defined for PSES and PSNS, means any building, structure, facility, or installation from which there is or may be a discharge of pollutants, the construction of which commenced after June 2, 1982.

(9) "New source," as defined for BPT, BAT, BCT, and NSPS, means any point source the construction of which commenced after January 6, 1983.

(10) "Raw material" means the hides received by the tannery except for facilities covered by the retan-wet finish-sides and retan-wet finish-splits subcategories where "raw material" means the hide or split in the condition in which it is first placed into a wet process.

(11) "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 fat liquor.

(12) "Sulfide" means total sulfide as measured by the potassium ferricyanide titration method described in s. NR 252.035 or the modified Monier-Williams method described in s. NR 252.036.

(13) "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.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; **am. (12), Register, May, 2001, No. 545, eff 6-1-01.**

$$\text{Normality of Potassium Ferricyanide} = \frac{(\text{ml of thiosulfate added}) \times (\text{normality of thiosulfate})}{\text{K}_3\text{Fe}(\text{CN})_6}$$

(c) 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 one liter with distilled water. The buffer may be prepared in a hood. Store in a tightly stoppered container.

(d) Preparation of 0.05M barium chloride solution: Dissolve 12-13 g. barium chloride dihydrate in one liter of distilled water.

(e) Preparation of ferrous dimethylglyoxime indicator solution: Mix 10 ml. 0.6 percent ferrous sulfate, 50 ml. one percent dimethylglyoxime in ethanol, and 0.5 ml. concentrated sulfuric acid.

(f) Preparation of stock sulfide standard, 1000 ppm: Dissolve 2.4 g. reagent grade sodium sulfide in one 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, param-

**NR 252.03 Sulfide analytical method.** (1) The potassium ferricyanide titration method described in s. NR 252.035 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 section in s. NR 252.30. In all other cases, the modified Monier-Williams method as described in s. NR 252.036 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 sections except s. NR 252.30.

(2) The modified Monier-Williams method as described in s. NR 252.036 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 pursuant to s. NR 252.30.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; **r. and recr. Register, May, 2001, No. 545, eff. 6-1-01.**

**NR 252.035 Potassium ferricyanide titration method.** The following 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 and is to be used for the determination of sulfide in alkaline wastewater.

(1) **OUTLINE OF METHOD.** (a) 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 shall be precipitated with barium chloride. Thiosulfate is not titrated under the conditions of the determination.

(b) Apparatus: burette, 10 ml.

(2) **REAGENT.** (a) 0.1 Preparation of 0.02 N potassium ferricyanide; -32.925 g. per liter - this solution must be kept in the dark. Weigh to the nearest tenth of a gram 6.6 g. of analytical reagent grade potassium ferricyanide and dissolve in one liter distilled water. Store in an amber bottle in the dark. Prepare fresh each week.

(b) Standardization of ferricyanide solution: Transfer 50 ml. of solution to a 250 ml. Erlenmeyer flask. Add several crystals of potassium iodide, about one g., mix gently to dissolve, add one ml. of 6N hydrochloric acid, stopper the flask, and swirl gently. Let stand for 2 minutes, add 10 ml. of a 30% 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 one 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:

eter 66 (49 FR 43234, October 26, 1984, with correction notice at 50 FR 690, January 4, 1985)) immediately prior to use.

(g) Preparation of 10N NaOH: Dissolve 400 g. of analytical reagent grade NaOH in one liter distilled water.

(h) Sample preservation and storage: samples are to be field filtered by either gravity or pressure with coarse filter paper, such as 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 degrees C until analysis. Samples shall 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 shall provide documentation of sample integrity.

(3) **PROCEDURE.** (a) Transfer 100 ml. of sample to be analyzed, or a suitable portion containing not more than 15 mg. sul-

fide supplemented to 100 ml. with distilled water, to a 250 ml. Erlenmeyer flask.

(b) Adjust the sample to pH 8.5–9.5 with 6N HCl.

(c) Add 20 ml. of 6M ammonium chloride buffer (pH 9.3), one ml. of ferrous dimethylglyoxime indicator, and 25 ml. of 0.05M barium chloride. Mix gently, stopper, and let stand for 10 minutes.

(d) 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.

(e) Calculation and reporting of results.

$$\text{mg/l sulfide} = \frac{A \times B \times 16,000}{\text{vol. in ml. of sample titrated}}$$

where

A=volume in ml. of potassium ferricyanide solution used, and  
B= normality of potassium ferricyanide solution.

Report results to 2 significant figures.

**(4) QUALITY CONTROL.** (a) 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 shall be compared with established performance criteria to determine if the results of analyses are within precision and accuracy limits expected of the method.

(b) Before performing any analyses, the analyst shall demonstrate the ability to generate acceptable precision and accuracy with this method by performing the following operations.

1. Perform 4 replicate analyses of a 20 mg/l sulfide standard prepared in distilled water. See sub. (2) (f).

2. a. Calculate clean water precision and accuracy in accordance with standard statistical procedures. Clean water acceptance limits are presented in subd. 2. b. These criteria shall be met or exceeded before sample analyses can be initiated. A clean water standard shall be analyzed with each sample set and the established criteria met for the analysis to be considered under control.

b. 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 4 replicate analyses shall be within the range of 50 to 110% of the true value.

(c) The method detection limits or MDL may 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 s. NR 252.0365.

(d) A minimum of one spiked and one duplicate sample shall be performed for each analytical event, or 5% spikes and 5% duplicates when the number of samples per event exceeds 20. Spike levels are to be at the MDL and at x where x is the concentration found if in excess of the MDL. See par. (c) for MDL samples. Spike recovery shall be 40 to 120% 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 shall be repeated using the modified Monier-Williams procedures described in s. NR 252.036.

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

History: Cr. Register, May, 2001, No. 545, eff. 6-1-01.

#### NR 252.036 Modified Monier-Williams method.

**(1) OUTLINE OF METHOD.** (a) Hydrogen sulfide is liberated from an acidified sample by distillation and purging with nitrogen gas (N<sub>2</sub>). 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)).

(b) (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 mantle 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)

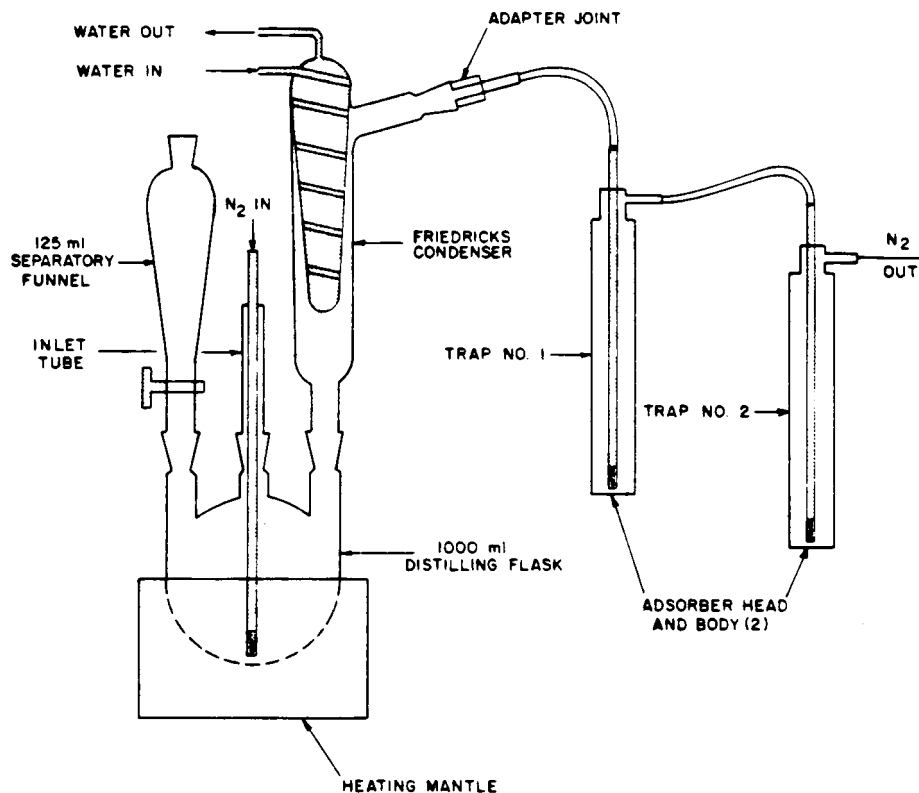
Adapter joint 24/40 to 19/38 (VWR Cat. No. 62905-26)

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

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

Laboratory vacuum pump or water aspirator

**FIGURE 1  
EQUIPMENT ASSEMBLY**



(2) REAGENTS. (a) Potassium hydroxide, 6N: Dissolve 340 g. of analytical reagent grade KOH in one liter distilled water.

(b) Sodium hydroxide, 6N: Dissolve 240 g. of analytical reagent grade NaOH in one liter distilled water.

(c) Sodium hydroxide, 0.03N: Dilute 5.0 ml. of 6N NaOH to one liter with distilled water.

(d) Hydrochloric acid, 6N: Dilute 500 ml. of concentrated HCl to one liter with distilled water.

(e) 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 degrees C.

(f) Potassium phosphate buffer, 0.05M: Dilute one volume of 0.5M potassium phosphate stock buffer with 9 volumes of distilled water. Solution is stable for one month at 4 degrees C.

(g) Alkaline 3% hydrogen peroxide: Dilute one volume of 30% hydrogen peroxide with 9 volumes of 0.03N NaOH. Prepare this solution fresh each day of use.

(h) Preparation of stock sulfide standard, 1000 ppm.: Dissolve 2.4 g. reagent grade sodium sulfide in one liter of distilled water. Store in a tightly stoppered container. Diluted working standards shall 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)).

(i) 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 shall be covered tightly and stored at 4 degrees C until analysis. Samples shall be analyzed within 7 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.

(3) PROCEDURE. (See Figure 1 for apparatus layout).

(a) Place 50 ml. of 0.05M pH 7.0 potassium phosphate buffer in trap no.1.

(b) Place 50 ml. of alkaline 3% hydrogen peroxide in trap no. 2.

(c) Sample introduction and N<sub>2</sub> 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 N<sub>2</sub> 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 N<sub>2</sub> inlet tube of the distillation flask shall 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 using snoop or soap water solution.

(d) Volatilization of H<sub>2</sub>S: Interrupt the N<sub>2</sub> 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 N<sub>2</sub> flow for 30 minutes. Terminate the distillation cycle by turning off the



heating mantle and maintaining N<sub>2</sub> flow through the system for 5 to 10 minutes. Then turn off the N<sub>2</sub> flow, 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 shall be exercised in terminating the distillation cycle to avoid flash-over, draw-back, or violent steam release.

(e) 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.

(f) 1. Calculations and reporting of results

2. Gravimetric procedure:

$$\text{mg sulfide/l} = \frac{(\text{mg. BaSO}_4 \text{ collected in Trap No. 2}) \times (137)}{\text{volume in ml. of waste sample distilled}}$$

3. Turbidimetric procedure:

$$\text{mg sulfide/l} = \frac{A \times B \times 333}{C}$$

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

4. Report results to 2 significant figures.

(4) **QUALITY CONTROL.** (a) Each laboratory that uses this method shall 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 shall maintain performance records to define the quality of data that is generated. Ongoing performance checks shall be compared with established performance criteria to determine if the results of analyses are within precision and accuracy limits expected of the method.

(b) Before performing any analyses, the analyst shall demonstrate the ability to generate acceptable accuracy and precision by performing the following operations.

1. Perform 4 replicate analyses of a 20 mg/l. sulfide standard prepared in distilled water. Refer to sub. (2) (h) under "Reagents."

2. a. Calculate clean water precision and accuracy in accordance with standard statistical procedures. Clean water acceptance limits are presented in subd. 2.b. These criteria shall be met or exceeded before sample analyses can be initiated. A clean water standard shall be analyzed with each sample set and the established criteria met for the analyses to be considered under control.

b. 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 4 replicate analyses shall be within the range of 72 to 114% of the true value.

(c) The method detection limit or MDL may 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 s. NR 252.0365.

(d) A minimum of one spiked and one duplicate sample shall be run for each analytical event, or 5% spikes and 5% duplicates when the number of samples per event exceeds 20. Spike levels are to be at the MDL and at x when x is the concentration found if in excess of the MDL. See par. (c) for MDL samples. Spike recovery shall be 60 to 120% for the analysis of a particular matrix type to be considered valid.

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

**History:** Cr. Register, May, 2001, No. 545, eff. 6-1-01.

## NR 252.0365 Method detection limit method.

(1) **DEFINITION.** "Method detection limit" or "MDL" means the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

(2) **SCOPE AND APPLICATION.** This procedure is designed for applicability to a wide variety of sample types ranging from reagent or blank water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. All sample processing steps of the analytical method shall be included in the determination of the MDL. 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.

(3) **PROCEDURE.** (a) Make an estimate of the detection limit using one of the following:

1. The concentration value that corresponds to an instrument signal or 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 shall be considered in making the estimate.

2. The concentration value that corresponds to 3 times the standard deviation of replicate instrumental measurements for the analyte in reagent water.

3. The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations, such as a break in the slope of the standard curve.

4. The concentration value that corresponds to known instrumental limitations.

(b) Prepare reagent or 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 or interferent. The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

(c) 1. If the MDL is to be determined in reagent or blank water, prepare a laboratory standard such as an analyte in reagent water at a concentration which is at least equal to or in the same concentration range as the estimated MDL. It is recommended to be between one and 5 times the estimated MDL. Proceed to par. (d).

2. 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 5 times the estimated MDL, proceed to par. (d).

3. 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 5 times the MDL. In the case where an interference is coanalyzed with the analyte and the measured level of analyte is greater than 5 times the estimated MDL, there are 2 options:

a. Obtain another sample of lower level of analyte in same matrix if possible.

b. 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.

(d) 1. Take a minimum of 7 aliquots of the sample to be used to calculate the MDL and process each through the entire analyti-

cal 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.

2. It may be economically and technically desirable to evaluate the estimated MDL before proceeding with subd.1. This will prevent repeating this entire procedure when the costs of analyses are high and 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 5 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 2 aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described in subd.1. Evaluate these data:

a. If these measurements indicate the sample is in the desirable range for determining the MDL, take 5 additional aliquots and proceed. Use all 7 measurements to calculate the MDL.

b. If these measurements indicate the sample is not in the correct range, reestimate the MDL, obtain new sample as in par. (c) and repeat either subd.1. or 2.

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

$$S^2 = \frac{1}{n-1} \left[ \sum_{i=1}^n x_i^2 - \frac{(\sum_{i=1}^n x_i)^2}{n} \right]$$

$$s = (S^2)^{0.5}$$

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

$$\sum_{i=1}^n x_i^2$$

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

(f) 1. 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% confidence level and a standard deviation estimate with  $n-1$  degrees of freedom as given in subd. 2.

$S$ =standard deviation of the replicate analyses.

2. The 95% confidence limits for the MDL derived in subd. 1. 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:

$$MDLLCL=0.69 MDL$$

$MDLUCL=1.92 MDL$  where  $MDLLCL$  and  $MDLUCL$  are the lower and upper 95% confidence limits respectively based on 7 aliquots.

(g) 1. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.

2. If this is the initial attempt to compute MDL based on the estimated MDL in par. (a), take the MDL as calculated in par. (f), spike in the matrix at the calculated MDL and proceed through the procedure starting with par. (d)1.

3. 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.

4. 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.

$$\text{if } \frac{S_A^2}{S_B^2} < 3.05$$

then compute the spooled standard deviation by the following equation:

$$s_{\text{pooled}} = \left[ \frac{6S_A^2 + 6S_B^2}{12} \right]^{0.5}$$

$$\text{if } \frac{S_A^2}{S_B^2} > 3.05$$

respike at the last calculated MDL and process the samples through the procedure starting with par. (d).

5. Use the  $S_{\text{pooled}}$  as calculated in subd. 3. to compute the final MDL according to the following equation:

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

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

6. The 95% confidence limits for MDL derived in subd. 4. are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

$$MDLLCL=0.72 MDL$$

$$MDLUCL=1.65 MDL$$

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

(4) REPORTING. The analytical method used shall 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 shall be specified with the MDL value. The sample matrix used to determine the MDL shall 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.

(5) 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).

(6) TABLE OF STUDENTS'  $T$ -VALUES AT THE 99% CONFIDENCE LEVEL.

Number of replicates	Degrees of freedom (n-1)	t (n-1, 1-α=.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

History: Cr. Register, May, 2001, No. 545, eff. 6-1-01.

**NR 252.04 Applicability of sulfide pretreatment standards.** (1) A POTW receiving wastewater from a facility subject to this chapter may require more stringent pretreatment standards for sulfide than those established by this chapter without EPA approval.

(2) The pretreatment standards for sulfide established by this chapter will not apply if the POTW receiving wastewater from a facility subject to this chapter certifies in writing with explanation of relevant factors considered, in accordance with the provisions of sub. (3) 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:

(a) The presence and characteristics of other industrial wastewaters which can increase or decrease sulfide concentrations, pH, or both.

(b) The characteristics of the sewer/interceptor collection system which either minimize or enhance opportunities for release of hydrogen sulfide gas.

(c) The characteristics of the receiving POTW's headworks, preliminary and primary treatment systems, and sludge holding and dewatering facilities which either minimize or enhance opportunities for release of hydrogen sulfide gas.

(d) The occurrence of any prior sulfide related interference as defined in s. NR 252.02 (5).

(3) (a) On October 13, 1983, a POTW which intends to certify that the sulfide pretreatment standard does not apply shall publish, in a local newspaper with the largest circulation, a notice that presents the findings supporting this determination consistent with sub. (1). Allowance for public hearing of these findings shall be provided. The POTW shall identify all existing facilities to which the sulfide pretreatment standard otherwise established by this chapter would not apply.

(b) On January 11, 1984, a POTW which intends to certify that the sulfide pretreatment standard does not apply shall 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 if held.

(c) On February 10, 1984, EPA shall acknowledge to the POTW receipt of any certification submitted under pars. (a) and (b), and shall indicate to the POTW the adequacy of the submission based upon a review of the factors set forth in sub. (2).

(d) Within 30 days of the date of receipt of adequate submissions under pars. (a) to (c), EPA shall publish a notice in the federal register identifying those facilities to which the sulfide pretreatment standards of this part do not apply.

(e) A POTW may certify that the sulfide pretreatment standards of this chapter do not apply to a new source planning to discharge into the POTW. This certification shall be submitted prior to the commencement of discharge, and shall conform to a minimum with criteria in sub. (2) and the general procedures and intervals of time contained in pars. (a) to (d).

**(4) APPLICABILITY OF SULFIDE PRETREATMENT STANDARDS.** (a) If, after EPA and the POTW have determined in accordance with this section that the sulfide pretreatment standards of this chapter are not applicable to specified facilities, a POTW then determines that there have been changed circumstances which justify application of the sulfide pretreatment standards, the POTW shall revoke the certification submitted under sub. (3). Circumstances include, but are not limited to, changes in the factors specified in sub. (2). The POTW and EPA shall then adhere to the general procedures and time intervals contained in sub. (3) in order to determine whether the sulfide pretreatment standards contained in this chapter are applicable.

(b) If pursuant to par. (a) the sulfide pretreatment standards of this chapter 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.

(c) At any time after October 13, 1983, if a POTW determines that there have been changed circumstances which include, but are not limited to, changes in the factors specified in sub. (2), it may initiate proceedings contained in sub. (3) to determine that the sulfide pretreatment standards of this section shall not be applicable. The POTW and EPA shall follow the procedures and time intervals contained in sub. (3) to make this determination. A final determination that the sulfide pretreatment standards are not applicable shall be made prior to the discharge of sulfide not in accordance with the standards set forth in this chapter.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; cr. (4), Register, May, 2001, No. 545, eff. 6-1-01.

**NR 252.05 Compliance date for pretreatment standards for existing sources (PSES).** Existing sources subject to PSES shall comply by November 25, 1985.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.06 Monitoring requirements.** Compliance with monthly average discharge limitations is required regardless of the number of samples analyzed and averaged.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.10 Applicability; description of the hair pulp, chrome tan, retan-wet finishing subcategory.** The provisions of this subcategory 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.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.11 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 125.30-125.32, any existing point source subject to this subcategory shall 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	
	Kg/kg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	9.3	4.2
TSS	13.4	6.1
Oil and grease	3.9	1.7
Total chromium	0.24	0.09
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; am. Table, Register, May, 2001, No. 545, eff. 6-1-01.

**NR 252.12 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 125.30-125.32, any existing point source subject to this subcategory shall 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 BOD<sub>5</sub>, TSS, oil and grease, and pH contained in s. NR 252.11.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.13 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 125.30-125.32, any existing point source subject to this subcategory shall 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 s. NR 252.11.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.14 New source performance standards (NSPS).** Any new source subject to this subcategory shall achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Kg/kgg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	6.0	2.7
TSS	8.7	4.0
Oil and grease	2.5	1.1
Total chromium	0.16	0.06
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.15 Pretreatment standards for existing sources (PSES).** (1) Except as provided in s. NR 252.04 and 40 CFR 403.7 and 403.13, any existing source subject to this subcategory which introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Milligrams per liter (mg/l)	
	Maximum for any 1 day	Maximum for monthly average
Sulfide	24.0	.....
Total chromium	12.0	8.0
pH	(1)	(1)

<sup>1</sup>Not less than 7.0.

(2) Any existing source subject to this subcategory which processes less than 275 hides/day shall comply with sub. (1), except that the total chromium limitations contained in sub. (1) do not apply.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; am. Register, May, 2001, No. 545, eff. 6-1-01.

**NR 252.16 Pretreatment standards for new sources (PSNS).** Except as provided in 40 CFR 403.7 and s. NR 252.04, any new source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the pretreatment standards contained in s. NR 252.15.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.20 Applicability; description of the hair save, chrome tan, retan-wet finish subcategory.** The provisions of this subcategory 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.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.21 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 125.30-125.32, any existing point source subject to this subcategory shall 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	
	Kg/kgg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	8.2	3.7
TSS	11.8	5.4
Oil and grease	3.4	1.5
Total chromium	0.21	0.08
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.22 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 125.30-125.32, any existing point source subject to this subcategory shall 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 BOD<sub>5</sub>, TSS, oil and grease, and pH contained in s. NR 252.21.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.23 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 125.30-125.32, any existing point source subject to this subcategory shall 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 s. NR 252.21.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.24 New source performance standards (NSPS).** Any new source subject to this subcategory shall achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Kg/kgg (or pounds per 1000 lb) or raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	6.9	3.1
TSS	9.9	4.5
Oil and grease	2.9	1.3
Total chromium	0.18	0.06
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.



**NR 252.25 Pretreatment standards for existing sources (PSES).** Except as provided in s. NR 252.04 and 40 CFR 403.7 and 403.13, any existing source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Milligrams per liter (mg/l)	
	Maximum for any 1 day	Maximum for monthly average
Sulfide	24.0	.....
Total chromium	12.0	8.0
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Not less than 7.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; am. Table, Register, May, 2001, No. 545, eff. 6-1-01.

**NR 252.26 Pretreatment standards for new sources (PSNS).** Except as provided in 40 CFR 403.7 and s. NR 252.04, any new source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the pretreatment standards contained in s. NR 252.25.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.30 Applicability; description of the hair save or pulp, nonchrome tan, retan-wet finish subcategory.** The provisions of this subcategory 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.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.31 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 125.30-125.32, any existing point source subject to this subcategory shall 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	
	Kg/kgg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	6.7	3.0
TSS	9.7	4.4
Oil and grease	2.8	1.3
Total chromium	0.17	0.06
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; am. Table, Register, May, 2001, No. 545, eff. 6-1-01.

**NR 252.32 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 125.30-125.32, any existing point source subject to this subcategory shall 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 BOD<sub>5</sub>, TSS, oil and grease, and pH contained in s. NR 252.31.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.33 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 125.30-125.32, any existing point source subject to this subcategory shall 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 s. NR 252.31.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.34 New source performance standards (NSPS).** Any new source subject to this subcategory shall achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Kg/kgg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	5.9	2.7
TSS	8.5	3.9
Oil and grease	2.4	1.1
Total chromium	0.15	0.06
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.35 Pretreatment standards for existing sources (PSES).** (1) Except as provided in s. NR 252.04 and 40 CFR 403.7 and 403.13, any existing source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Milligrams per liter (mg/l)	
	Maximum for any 1 day	Maximum for monthly average
Sulfide	24.0	.....
Total chromium	12.0	8.0
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Not less than 7.0.

(2) Any existing source subject to this subcategory which processes less than 350 hides/day shall comply with s. NR 252.35 (1), except that the total chromium limitations contained in sub. (1) do not apply.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; am. Register, May, 2001, No. 545, eff. 6-1-01.

**NR 252.36 Pretreatment standards for new sources (PSNS).** Except as provided in 40 CFR 403.7 and s. NR 252.04 any new source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the pretreatment standards contained in s. NR 252.35.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.40 Applicability; description of the retan-wet finish-sides subcategory.** The provisions of this subcategory 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.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.41 Effluent limitations representing the degree of effluent reduction attainable by the control technology currently available (BPT).** Except as provided in 40 CFR 125.30–125.32, any existing point source subject to this subcategory shall 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	
	Kg/kg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	8.9	4.0
TSS	12.8	5.8
Oil and grease	3.7	1.7
Total chromium	0.23	0.08
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86; am. Table, Register, May, 2001, No. 545, eff. 6–1–01.

**NR 252.42 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 125.30–125.32, any existing point source subject to this subcategory shall 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 BOD<sub>5</sub>, TSS, oil and grease, and pH contained in s. NR 252.41.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.43 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 125.30–125.32, any existing point source subject to this subcategory shall 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 s. NR 252.41.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.44 New source performance standards (NSPS).** Any new source subject to this subcategory shall achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Kg/kg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	6.5	2.9
TSS	9.3	4.3
Oil and grease	2.7	1.2
Total chromium	0.17	0.06
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86; am. Table, Register, May, 2001, No. 545, eff. 6–1–01.

**NR 252.45 Pretreatment standards for existing sources (PSES).** Except as provided in 40 CFR 403.7 and 403.13, any existing source subject to this subcategory that intro-

duces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Milligrams per liter (mg/l)	
	Maximum for any 1 day	Maximum for monthly average
Total chromium	19.0	12.0
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 10.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.46 Pretreatment standards for new sources (PSNS).** Except as provided in 40 CFR 403.7, any new source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the pretreatment standards contained in s. NR 252.45.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.50 Applicability; description of the no beamhouse subcategory.** The provisions of this subcategory 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 nonchrome tanning, and retan–wet finishing.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.51 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 125.30–125.32, any existing point source subject to this subcategory shall 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	
	Kg/kg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	8.0	3.6
TSS	11.6	5.3
Oil and grease	3.4	1.5
Total chromium	0.21	0.08
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86; am. Table, Register, May, 2001, No. 545, eff. 6–1–01.

**NR 252.52 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 125.30–125.32, any existing point source subject to this subcategory shall 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 BOD<sub>5</sub>, TSS, oil and grease, and pH contained in s. NR 252.51.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.53 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 125.30–125.32, any existing point source subject to this subcategory shall 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 s. NR 252.51.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.54 New source performance standards (NSPS).** Any new source subject to this subcategory shall achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Kg/kg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	5.3	2.4
TSS	7.7	3.5
Oil and grease	2.2	1.0
Total chromium	0.14	0.05
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.55 Pretreatment standards for existing sources (PSES).** Except as provided in 40 CFR 403.7 and 403.13, any existing source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Milligrams per liter (m,g/l)	
	Maximum for any 1 day	Maximum for monthly average
Total chromium	19.0	12.0
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 10.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.56 Pretreatment standards for new sources (PSNS).** Except as provided in 40 CFR 403.7, any new source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the pretreatment standards contained in s. NR 252.55.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.60 Applicability; description of the through-the-blue subcategory.** The provisions of this subcategory 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.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.61 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 125.30-125.32, any existing point source subject to this subcategory shall 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	
	Kg/kg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	3.2	1.5
TSS	4.7	2.1
Oil and grease	1.4	0.61
Total chromium	0.08	0.03
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; **am. Table, Register, May, 2001, No. 545, eff. 6-1-01.**

**NR 252.62 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 125.30-125.32, any existing point source subject to this subcategory shall 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 BOD<sub>5</sub>, TSS, oil and grease, and pH contained in s. NR 252.61.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.63 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 125.30-125.32, any existing point source subject to this subcategory shall 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 s. NR 252.61.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.64 New source performance standards (NSPS).** Any new source subject to this subcategory shall 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 pounds per 1000 lb) of raw material	
BOD <sub>5</sub>	3.0	1.3
TSS	4.3	1.9
Oil and grease	1.2	0.55
Total chromium	0.08	0.03
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; **am. Table, Register, May, 2001, No. 545, eff. 6-1-01.**

**NR 252.65 Pretreatment standards for existing sources (PSES).** Except as provided in s. NR 252.04 and 40 CFR 403.7 and 403.13, any existing source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Milligrams per liter (mg/l)	
	Maximum for any 1 day	Maximum for monthly average
Sulfide	24.0	.....
Total chromium	12.0	8.0
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Not less than 7.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; am. Table, Register, May, 2001, No. 545, eff. 6-1-01.

**NR 252.66 Pretreatment standards for new sources (PSNS).** Except as provided in 40 CFR 403.7 and s. NR 252.04, any new source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the pretreatment standards contained in s. NR 252.65.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.70 Applicability; description of the shearing subcategory.** The provisions of this subcategory 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.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.71 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 125.30-125.32, any existing point source subject to this subcategory shall 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 1000 lb) of raw material	
BOD <sub>5</sub>	15.0	6.8
TSS	21.7	9.9
Oil and grease	6.3	2.8
Total chromium	0.39	0.14
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; am. Table, Register, May, 2001, No. 545, eff. 6-1-01.

**NR 252.72 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 125.30-125.32, any existing point source subject to this subcategory shall 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 BOD<sub>5</sub>, TSS, oil and grease, and pH contained in s. NR 252.71.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.73 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 125.30-125.32, any existing point source subject to this subcategory shall 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 s. NR 252.71.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.74 New source performance standards (NSPS).** Any new source subject to this subcategory shall achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Kg/kg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	13.2	5.9
TSS	19.1	8.7
Oil and grease	5.6	2.5
Total chromium	0.34	0.12
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.75 Pretreatment standards for existing sources (PSES).** Except as provided in 40 CFR 403.7 and 403.13, any existing source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Milligrams per liter (mg/l)	
	Maximum for any 1 day	Maximum for monthly average
Total chromium	19.0	12.0
pH	(1)	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 10.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.76 Pretreatment standards for new sources (PSNS).** Except as provided in 40 CFR 403.7, any new source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the pretreatment standards contained in s. NR 252.75.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.80 Applicability; description of the pigskin subcategory.** The provisions of this subcategory 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.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.



**NR 252.81 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 125.30–125.32, any existing point source subject to this subcategory shall 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 1000 lb) of raw material	
BOD <sub>5</sub>	7.0	3.2
TSS	10.1	4.6
Oil and grease	3.0	1.3
Total chromium	0.18	0.07
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.82 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 125.30–125.32, any existing point source subject to this subcategory shall 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 BOD<sub>5</sub>, TSS, oil and grease, and pH contained in s. NR 252.81.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.83 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 125.30–125.32, any existing point source subject to this subcategory shall 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 s. NR 252.81.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.84 New source performance standards (NSPS).** Any new source subject to this subcategory shall 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 pounds per 1000 lbs) of raw material	
BOD <sub>5</sub>	5.8	2.6
TSS	8.3	3.8
Oil and grease	2.4	1.1
Total chromium	0.15	0.05
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.85 Pretreatment standards for existing sources (PSES).** Except as provided in s. NR 252.04 and 40 CFR 403.7 and 403.13, any existing source subject to this subcategory that introduces process wastewater pollutants into a publicly

owned treatment works shall comply with 40 CFR Part 403, and achieve the following pretreatment standards:

Pollutant or pollutant property	PSES	
	Milligrams per liter (mg/l)	
	Maximum for any 1 day	Maximum for monthly average
Sulfide	24.0	.....
Total chromium	12.0	8.0
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Not less than 7.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86; **am. Table, Register, May, 2001, No. 545, eff. 6–1–01.**

**NR 252.86 Pretreatment standards for new sources (PSNS).** Except as provided in 40 CFR 403.7 and s. NR 252.04, any new source subject to this subcategory that introduces process wastewater pollutants into a publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the pretreatment standards contained in s. NR 252.85.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.90 Applicability; description of the retan-wet finish-splits subcategory.** The provisions of this subcategory are applicable to process wastewater discharges resulting from any tannery which processes previously unhaird and tanned splits into finished leather by retan-wet finishing.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.91 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 125.30–125.32, any existing point source subject to this subcategory shall 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	
	Kg/kg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	5.8	2.6
TSS	8.3	3.8
Oil and grease	2.4	1.1
Total chromium	0.15	0.05
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86; **am. Table, Register, May, 2001, No. 545, eff. 6–1–01.**

**NR 252.92 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 125.30–125.32, any existing point source subject to this subcategory shall 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 BOD<sub>5</sub>, TSS, oil and grease, and pH contained in s. NR 252.91.

**History:** Cr. Register, October, 1986, No. 370, eff. 11–1–86.

**NR 252.93 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 125.30–125.32, any existing point source subject to this subcate-

gory shall 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 s. NR 252.91.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.94 New source performance standards (NSPS).** Any new source subject to this subcategory shall achieve the following new source performance standards (NSPS):

Pollutant or pollutant property	NSPS	
	Kg/kgg (or pounds per 1000 lb) of raw material	
	Maximum for any 1 day	Maximum for monthly average
BOD <sub>5</sub>	3.5	1.6
TSS	5.1	2.3
Oil and grease	1.5	0.66
Total chromium	0.09	0.03
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 9.0.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.95 Pretreatment standards for existing sources (PSES).** (1) Except as provided in 40 CFR 403.7 and 403.13, any existing source subject to this subcategory that introduces process wastewater pollutants into publicly owned treatment works shall 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	19.0	12.0
pH	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup>Within the range 6.0 to 10.0.

(2) Any existing source subject to this subcategory which processes less than 3,600 splits/day shall comply with sub. (1), except that the total chromium limitations contained in sub. (1) do not apply.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86; **am. (2), Register, May, 2001, No. 545, eff. 6-1-01.**

**NR 252.96 Pretreatment standards for new sources (PSNS).** Except as provided in 40 CFR 403.7, any new source subject to this subcategory that introduces process wastewater pollutants into publicly owned treatment works shall comply with 40 CFR Part 403, and achieve the pretreatment standards contained in s. NR 252.95.

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.

**NR 252.99 Cross-references.** The federal citations in this chapter correspond to provisions of the Wisconsin administrative code and Wisconsin statutes. The federal citations can be cross-referenced in the following table:

Code of Federal Regulations	Corresponding state code section
40 CFR Part 425 . . . . .	ch. NR 252
40 CFR 125.30 – 125.32 . . . .	s. NR 211.14, s. 283.13 (3), Stats.
40 CFR Part 401 . . . . .	chs. NR 205, 215, 219
40 CFR 403.3 (i) . . . . .	s. NR 211.03 (5)
40 CFR 403.7 . . . . .	s. NR 211.13
40 CFR 403.13 . . . . .	s. NR 211.14

**History:** Cr. Register, October, 1986, No. 370, eff. 11-1-86.