

**§ 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 BOD<sub>5</sub>, 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 <sub>5</sub> .....	3.5	1.6
TSS .....	5.1	2.3
Oil & 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.

**§ 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 .....	( <sup>1</sup> )	( <sup>1</sup> )

<sup>1</sup> 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:

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

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.

$$1. \text{ mg./l. sulfide} = \frac{A \times B \times 16,000}{\text{vol. in ml. of sample titrated}}$$

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

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\**

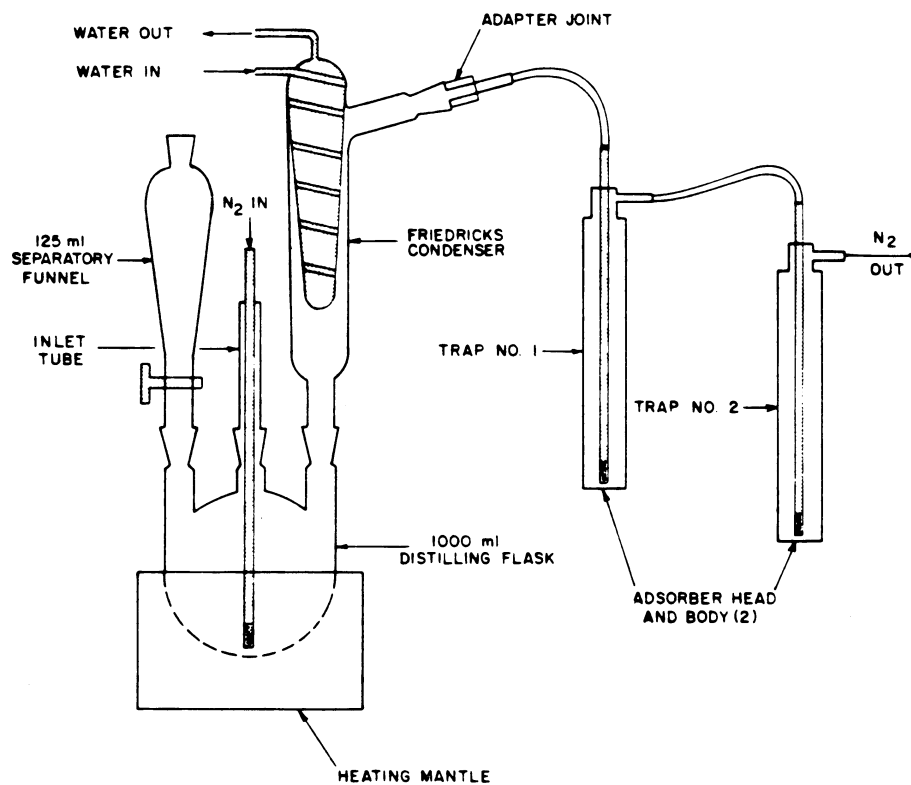
(See Figure 1.) \* Catalogue numbers are given only to provide a more complete description of the equipment necessary, and do not constitute a manufacturer or vendor endorsement.

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)

**FIGURE 1**  
**EQUIPMENT ASSEMBLY**



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  $7.0 \pm 0.1$  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 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 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 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 (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:

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

2. Turbidimetric procedure:

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

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

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 LIMIT<sup>1</sup>

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 ana-

lytical 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

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

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 be-

fore 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<sup>2</sup>) and standard deviation (S) of the replicate measurements, as follows:

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

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

where: the x<sub>i</sub>, 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.

6. (a) Compute the MDL as follows:

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

where:

MDL=the method detection

t<sub>(n-1, 1-a=.99)</sub>=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<sup>2</sup>/df) and calculated as follows:

MDL<sub>LCL</sub>=0.69 MDL  
 MDL<sub>UCL</sub>=1.92 MDL where MDL<sub>LCL</sub> and MDL<sub>UCL</sub> 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<sup>2</sup> from the current MDL calculation and S<sup>2</sup> from the previous MDL calculation to compute the F ratio.

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

then compute the pooled 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 Step 4.

(d) Use the S<sub>pooled</sub> as calculated in 7b to compute the final MDL according to the following equation:

MDL=2.681 (S<sub>pooled</sub>)  
 where 2.681 is equal to t(12, 1-a=.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<sub>LCL</sub>=0.72 MDL  
 MDL<sub>UCL</sub>=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 <sub>(n-1, 1-a=.99)</sub>
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]

**PART 426—GLASS MANUFACTURING POINT SOURCE CATEGORY**

**Subpart A—Insulation Fiberglass Subcategory**

Sec.

426.10 Applicability; description of the insulation fiberglass subcategory.

426.11 Specialized definitions.

426.12 Effluent limitations guidelines representing the degree of effluent reduction attainable by the application of the best