METHOD 14XXXSO$_4$

Sulfate (SO_4) by Barium Sulfate Suspension and Photometry

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Disclaimer

This method has been submitted to the U.S. Environmental Protection Agency for use in EPA's water programs but has not been approved for use by EPA. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

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Introduction

This method is a convenient ready to use reagent test kit for Sulfate (SO_4) testing which conforms with "Chemical Analysis of Water and Wastewater", EPA method 375.4. The test kit is suitable for both on-site testing and typical laboratory testing. The test kit consists of pre-measured reagent sets for both sample preparation and analytical determinations. This method's approach, with pre-measured reagents, reduces analytical errors, the amount of hazardous waste, and increases occupational safety.

The method incorporates two ranges (spanning 5 to 1,000 mg/L). This method has increased sensitivity at higher sulfate concentrations, which helps to reduce the amount of sample manipulation, especially in field applications.

Method 14XXXSO_4

Sulfate (SO_4) by Barium Sulfate Suspension and Photometry

1.0 Scope and Application

1.1 This method determines the level of sulfates (SO_4) in waters and wastewater matrices. The sulfate present in the samples is precipitated as barium sulfate in an hydrochloric acid solution with the addition of barium chloride. Barium sulfate is a sparsely soluble compound. The light absorbance of this suspension is measured photometrically.

1.2 This method is based on prior Environmental Protection Agency (EPA) methods for the determination of SO_4 (reference 16.1).

1.3 This method is for use in the United States Environmental Protection Agency's (EPA's) data gathering and monitoring programs under the Clean Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation and Liability Act, and Safe Drinking Water Act.

1.4 This method is intended for the analysis of SO_4 on drinking and surface waters, domestic and industrial wastes.

1.5 This method is capable of measuring SO_4 in the range of 5 to 1,000 mg/L, and may be extended to higher levels by serial dilution.

1.6 The method detection limit (MDL; 40 CFR 136, Appendix B) has been established at 5 mg/L for method 14548 and 100 mg/L for method 14564 (Section 13.2). The Minimum Level (ML) for reporting results is 5.0 mg/L for method 14548 and 100 mg/L for method 14564(Section 13.3).

1.7 Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in Section 9.2.

2.0 Summary of Method

2.1 Depending upon the measurement range (Section 17.0, Table 1), a one or five ml aliquot of un-preserved sample is measured into a Spectroquant® SO_4 cell test and mixed thoroughly. The cell test contains a pre-measured amount of the reagents for barium sulfate precipitation. The contents of the reaction cell are mixed well. To this solution, one dose (70 to 75 mg) of barium chloride reagent (SO_4-1K) is added, and the reagents are dissolved by shaking vigorously.

2.2 After allowing the solution to remain undisturbed for two minutes, the absorbance of the turbidity resulting from the formation of barium sulfate in the solution is measured. The measurements are performed using a photometer at 525-nm for the 5 to 250 mg/L range, and 820-nm for the 100 to 1,000 mg/L range kit.

2.3 The photometric determinations can be conducted on either a filter photometer or other type of photometer.

2.4 Quality is assured through the use of quality control samples (QCS), calibration of the instrumentation by using calibration test solutions (Section 17.0, Table 2), and operation of a formal quality assurance program.

3.0 Definitions

3.1 Definitions for terms used in this method are given in the glossary at the end of the method (Section 18).

4.0 Interferences

4.1 Color and suspended matter, may interfere with the photometric measurement. To counter this potential positive interference, the sample may be filtered using a 0.45 µm membrane filter. A blank of filtered sample (to which no barium chloride has been added) will counter absorbance related to remaining color and turbidity.

4.2 **Method 14548:** EDTA interfers and must be absent. Silver ion interfers if more than 2 mg/L are present, sulfide if more than 10 mg/L, sulfite and dichromate if more than 50 mg/L are present and thiosulfate if more than 25 mg/L are present in the sample.

Method 14564: EDTA interfers and must be absent. Silver ion interfers if more than 10 mg/L are present, sulfide if more than 50 mg/L, sulfite and dichromate if more than 250 mg/L and thiosulfate if more than 100 mg/L are present in the sample.

4.3 In waters containing appreciable amounts of organic material, it may not be possible to precipitate barium sulfate satisfactorily.

5.0 Safety

5.1 This method does not address all safety issues associated with its use. The toxicity or carcinogenicity of reagents used in this method has not been fully established. Each chemical and environmental sample should be regarded as a potential health hazard and exposure should be minimized. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References 16.3 and 16.4.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that the laboratory perform personal hygiene monitoring of each analyst using this method and that the results of this

monitoring shall be made available to the analyst.

5.3 Samples of unknown origin may possess potentially hazardous compounds. Samples should be handled with care (e.g. under a hood), so as to minimize exposure.

5.4 This method employs the use of Spectroquant® SO_4 cell tests. These cells contain pre-measured reagents in small quantities, which limits the handling of hazardous chemicals.

6.0 Equipment and Supplies

NOTE: *Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

6.1 Sample collection bottles-1-L borosilicate amber glass or plastic.

6.2 Analytical balance-Capable of weighing 0.1 mg.

6.3 Volumetric flasks-Various sizes.

6.4 Volumetric pipettes-Various sizes.

6.5 Vacuum source.

6.6 Filtration apparatus.

6.7 Filters-0.45µm.

6.8 Reaction cells-Spectroquant® SO_4 cell test (Section 17.0, Table 1).

6.9 Laboratory timer.

6.10 Rack for cells.

6.11 Dry cloths for cleaning cell tests.

6.12 Photometric device.

6.12.1 Filter photometer equipped with 525-nm and 820-nm wavelength interference filters, with cell compartment for tubes 16 x 100 mm-Spectroquant® NOVA 60, or equivalent.

6.12.2 Spectrophotometer for use at 525-nm and 820-nm wavelength, with cell compartment for tubes 16 x 100 mm.

7.0 Reagents and Standards

7.1 Spectroquant® SO_4 cell test appropriate to the concentration range selected (Section 17.0, Table 1).

7.2 Barium chloride-Spectroquant® SO_4-1K, or equivalent.

7.3 Deionized water.

7.4 Sulfate stock standard, 1,000 mg/L-Spectroquant® item 19813, or equivalent.

8.0 Sample Collection, Preservation, and Storage

8.1 Collect approximately 1 L, or a minimum of 100 ml, of a representative sample in a plastic or glass bottle following conventional sampling techniques (Reference 16.5).

8.2 Refrigerate samples at 0 to 4°C from the time of collection until the time of analysis, 40 CFR 136, Table II.

8.3 Analyze the sample within 7 days of collection.

8.4 The pH of the sample should be between 2.0 and 10.0, adjust if necessary prior to analysis.

8.5 Collect an additional two aliquots of a sample for each batch (of at least 20 samples) for the matrix spike and matrix spike duplicate.

9.0 Quality Control

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the ongoing analysis of laboratory reagent blanks, precision and recovery standards, and matrix-spiked samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data thus generated. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.

9.1.2 Analysis of matrix spike and matrix spike duplicate samples are required to demonstrate method accuracy and precision and to monitor matrix interferences (interferences caused by the sample matrix). The procedure and QC criteria for spiking are described in Section 9.3.

9.1.3 Analyses of laboratory blanks are required to demonstrate freedom from contamination. The procedure and criteria for blank analyses are described in Section 9.4.

9.1.4 The laboratory shall, on an ongoing basis, demonstrate through calibration verification and analysis of the ongoing precision and recovery sample that the analysis system is in control. These procedures are described in Sections 9.5 and 9.6.

9.1.5 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in Sections 9.3.7 and 9.6.3.

9.1.6 Accompanying QC for the determination of SO_4 is required per analytical batch. An analytical batch is a set of samples analyzed, to a maximum of 20 samples. Each analytical batch, of up to 20 samples, must be accompanied by a laboratory blank (Section 9.4), and ongoing precision and recovery sample (OPR, Section 9.6), and a matrix spike and matrix spike duplicate (MS/MSD, Section 9.3).

9.2 Initial demonstration of laboratory capability-The initial demonstration of laboratory capability is used to characterize laboratory performance and method detection limits.

9.2.1 Method detection limit (MDL)-The method detection limit must be established for the analyte, using the QC spiking solution (Section 7.4). To determine MDL values, take seven replicate aliquots of the diluted QC spiking solution and process each aliquot through each step of the analytical method. Perform all calculations and report the concentration values in the appropriate units. MDLs should be determined every year or whenever a modification to the method or analytical system is made that will affect the method detection limit.

9.2.2 Initial Precision and Recovery (IPR) - To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

9.2.2.1 Analyze four samples of the SO_4 standard (Section 7.4) according to the procedure beginning in Section 11.

9.2.2.2 Using the results of the four analyses, compute the average percent recovery (x) and the standard deviation (s, Equation 1) of the percent recovery for SO_4. Use the following equation for calculation of the standard deviation of the percent recovery:

Equation 1

$$s = \sqrt{\frac{\sum x^2 - \frac{(\sum x)^2}{n}}{n-1}}$$

Where:

n = number of samples

x = % recovery in each sample

s = standard deviation

9.2.2.3 Compare s and x with the corresponding limits for initial precision and recovery in Table 3, (which lists EPA's standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB). If s and x meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or x falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem, and repeat the test.

9.3 Matrix Spikes-The laboratory must spike, in duplicate, a minimum of five percent of all samples (one sample in each batch of 20 samples). The two sample aliquots shall be spiked with the SO_4 standard solutions (Sections 7.4).

9.3.1 The concentration of the spike in the sample shall be determined as follows:

9.3.1.1 If, as in compliance monitoring, the concentration of SO_4 in the sample is being checked against a regulatory concentration limit, the spiking level shall be at that limit or at 1 to 5 times higher than the background concentration of the sample (determined in Section 9.3.2), whichever concentration is higher.

9.3.1.2 If the concentration of SO_4 in a sample is not being checked against a limit, the spike shall be at the concentration of the precision and recovery standard (Section 7.4), or at 1 to 5 times higher than the background concentration, whichever concentration is higher.

9.3.2 Analyze one sample aliquot out of each set of 20 samples according to the procedure beginning in Section 11.0 to determine the background concentration (B) of SO_4.

9.3.2.1 If necessary, prepare a standard solution appropriate to produce a level in the sample at the regulatory compliance limit or at 1 to 5 times the background concentration (per Section 9.3.1).

9.3.2.2 Spike two additional sample aliquots with the spiking solution and analyze these aliquots to determine the concentration after spiking (A).

9.3.3 Calculate the percent recovery (P) of in SO_4 each aliquot using the following equation:

Equation 2

$$P = 100 * \frac{(A - B)}{T}$$

where:

P=Percent recovery

A=Measured concentration of SO_4 after spiking

B=Measured concentration of SO_4 before spiking

T=True concentration of the spike

9.3.4 Compare the percent recovery of the SO_4 with the corresponding QC acceptance criteria in Table 3.

9.3.4.1 If the results of the spike fail the acceptance criteria, and the recovery of the QC standard in the ongoing precision and recovery test (Section 9.6) for the analytical batch is within the acceptance criteria in Table 3, (which lists EPA's standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB), an interference is present. In this case, the result may not be reported for regulatory compliance purposes and the analyst must assess the potential cause for the interference. If the interference is attributable to sampling, the site or discharge should be resampled. If the interference is attributable to a method deficiency, the analyst must modify the method repeat the tests required in Section 9.1.2, and repeat the analysis of the sample and the MS/MSD.

9.3.4.2 If the results of both the spike and the ongoing precision and recovery test fail the acceptance criteria, the analytical system is judged to be out of control, and the problem shall be identified and corrected, and the sample re-analyzed.

9.3.5 Compute relative percent difference (RPD) between the two results (not between the two recoveries) using the following equation:

Equation 3

$$RPD = 100 * \frac{|D_1 - D_2|}{(D_1 + D_2)/2}$$

where:

RPD=Relative percent different

D_1=Concentration of SO_4 in the sample

D_2=Concentration of SO_4 in the second (duplicate) sample

9.3.6 The relative percent difference for duplicates shall meet the acceptance criteria in Table 3, (which lists EPA's standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB). If the criteria are not met, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected, and the analytical batch re-analyzed.

9.3.7 As a part of the QC program for the laboratory, method precision and accuracy for samples should be assessed and records should be maintained. After the analysis of five spiked samples, in which the recovery passes the test in Section 9.3.4, compute the average percent recovery (P_a) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from P_a-2s_p to P_a+2s_p. For example, if P_a = 90% and s_p = 10% for five analyses of SO_4 the accuracy interval is expressed as 70-110%. Update the accuracy assessment on a regular basis (e.g., after each five to ten new accuracy measurements).

9.4 Laboratory blanks-Laboratory reagent water blanks are analyzed to demonstrate freedom from contamination.

9.4.1 Prepare and analyze a laboratory blank initially (i.e. with the tests in Section 9.2) and with each analytical batch. The blank must be subjected to the same procedural steps as a sample.

9.4.2 If material is detected in the blank at a concentration greater than the ML (Section 1.6), analysis of samples must be halted until the source of contamination is eliminated and a new blank shows no evidence of contamination. All samples must be associated with an uncontaminated laboratory blank before the results may be reported for regulatory compliance purposes.

9.5 Calibration verification-Verify calibration of the photometric device per Section 10.0 for each analytical batch of up to 20 samples. If calibration curve linearity differs more than 10%, run a new calibration curve.

9.6 Ongoing Precision and Recovery (OPR)-To demonstrate that the analysis system is in control, and acceptable precision and accuracy is being maintained with each analytical batch, the analyst shall perform the following operations:

9.6.1 Analyze a precision and recovery standard (Section 7.4) with each analytical batch according to the procedure beginning in Section 11.0.

9.6.2 Compare the concentration with the limits for ongoing precision and recovery in Table 3, (which lists EPA's standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB). If the concentration is in the range specified, the analysis may proceed. If however, the concentration is not in the specified range, the analytical process is not in control. In this event, correct the problem, re-extract the analytical batch, and repeat the ongoing precision and recovery test.

9.6.3 The laboratory should add results that pass the specification in Section 9.6.2 to IPR and previous OPR data and update QC charts to form a graphic representation of continued laboratory performance. The laboratory should also develop a statement of laboratory data quality for each analyte by calculating the average percent recovery (R) and the standard deviation of the percent recovery (s_r). Express the accuracy as a recovery interval from R - $2s_r$ to R + $2s_r$.

For example, if R = 95%, and s_r = 5%, the accuracy is 85 % to 105 %.

9.7 Quality control sample (QCS)–It is suggested that the laboratory obtain a quality control sample from a source different from the source of the SO_4 used routinely in this method (Sections 7.4).

9.8 The standards used for initial precision and recovery (IPR, Section 9.2.2) matrix spikes (MS/MSD, Section 9.3), and ongoing precision and recovery (OPR, Section 9.6) should be identical, so that the most precise results will be obtained.

10.0 Calibration and Standardization

10.1 The Spectroquant® NOVA 60 is shipped factory calibrated (Reference 16.7), refer to the manufacturer's documents (Reference 16.8). The calibration curve can be verified, and the data from this verification can be stored, modified or re-entered at anytime. However, the factory program setting cannot be changed by the user. When appropriate, the manufacturer supplies a new micro chip (transponder) containing new calibration data.

10.2 For other photometric equipment, other than the Spectroquant® NOVA 60, plot a calibration curve with a minimum of five data points, from standards prepared from a SO_4 solution (Section 7.4), appropriate to the range to be tested.

10.2.1 Prepare a calibration curve using the SO_4 standard solution (Section 7.4), which consists of five data points. Prepare the curves for the two concentration ranges indicated in Table 2. The curves should include the lowest and highest concentrations for the range tested.

10.3 Verify the curve, using a calibration standard (mid-point of the curve), with each analytical batch of samples (Section 9.5).

10.4 Run a new calibration curve with each new lot of reagents, or when calibration curve linearity differs more than 10%, as stated in 40 CFR part 136, Table IB (Section 9.5).

11.0 Procedure

11.1 Choose a Spectroquant® SO_4 cell test concentration range appropriate for the sample matrix to be tested (Section 17.0, Table 1), using prior knowledge of the particular waste stream.

11.2 If suspended solids or turbidity are suspected to cause interferences with the photometric measurements, filter the sample through 0.45 µm filter.

11.3 Carefully pipette the appropriate aliquot (Section 17.0, Table 1) of representative pre-treated sample into a Spectroquant® SO_4 cell test.

11.4 Close tightly with the screw cap and mix vigorously.

11.5 Add one dose (approximately 70 to 75 mg) of barium chloride (Spectroquant® SO_4-1K) to the cell. Close tightly, and shake vigorously until the reagent is completely dissolved.

11.6 Set the time for two minutes and let stand undisturbed.

11.7 After the five minute reaction time, wipe cell with a clean dry cloth, and measure the turbidity. Perform this measurement within five minutes, to avoid settling of barium sulfate fines (swirl gently to re-suspend if five minute time period is exceeded).

11.8 Determination using Spectroquant® NOVA 60.

11.8.1 Switch on the Spectroquant® NOVA 60 filter photometer as per manufacturer's suggestions for operation (Reference 16.8).

11.8.2 Place the Spectroquant® SO_4 cell test into the cell compartment with the vertical line facing you, and push down until the cell clicks into place.

11.8.3 Wait as the Spectroquant® NOVA 60 recognizes the bar code. The Spectroquant® SO_4 cell test product information is displayed, and the instrument is automatically set to the appropriate wavelength and measuring parameters (bar code recognition of item number, test range, cell format, wavelength, and calibration data).

11.8.4 Record the displayed result in mg/L.

11.9 Determination using photometric equipment other than Spectroquant® NOVA 60.

11.9.1 Warm up the instrument as per manufacturer's suggestion for operation.

11.9.2 Set the instrument to a wavelength of 525 nm or 820 nm, depending upon the concentration range tested (Section 17.0, Table 1).

11.9.3 Zero the instrument with a reagent water blank which has been prepared in the same manner as the standards and samples (filtered, pH adjusted, etc.). Do not add barium chloride to the blank.

11.9.4 Place the cell into the cell compartment/cell holder with the vertical line facing you.

11.9.5 Record the absorbance reading on the instrument.

11.9.6 Plot the absorbance reading on the calibration curve, to obtain the concentration SO_4 as mg/L.

12.0 Data Analysis and Calculations

12.1 If no pre-dilution was performed upon the sample, no calculation is necessary.

12.2 If pre-dilution was required, calculate the SO_4 mg/L as follows:

Equation 4

$$SO4 = A * \frac{V_2}{V_1}$$

where:

A = *Measured concentration of analyte from photometric determination (mg/L)*

V_1 = *Volume of sample used for dilution (ml)*

V_2 = *Final total volume of diluted sample (ml)*

12.3 Report results to two significant digits for concentrations found above the ML (Section 1.6) in all samples. Report results below the ML as <5.0 mg/L for SO_4.

13.0 Method Performance

13.1 This method, as equivalent to EPA Method 375.4 (Reference 16.1) should achieve the method performance as cited in Section 8.0 of that method.

13.2 The method detection limit (MDL) study was performed by a single analyst, and was determined as 5 mg/L for method 14548 and 100 mg/L for method 14564.

13.3 The minimum level (ML) is determined as 5.0 mg/L, based on the Spectroquant® SO_4 cell test with the lowest concentration (Section 17.0, Table 2). 5.0 mg/L is the lowest calibration standard used for this range (10.2.1 and 10.2.2).

14.0 Pollution Prevention

14.1 The reagents used in this method pose little threat to the environment, when managed properly.

14.2 Reagents should be ordered consistent with laboratory use, to minimize the amount of expired materials to be disposed.

15.0 Waste Management

15.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restriction. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations.

15.2 For further information on waste management, consult "The Waste Management Manual for Laboratory Personnel" and "Less is Better: Laboratory Chemical Management for Waste Reduction," both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036.

16.0 References

16.1 "Methods for the Chemical Analysis of Water and Wastes," 3rd Edition, Environmental Protection Agency, Environmental Monitoring Systems Laboratory–Cincinnati (EMSL–Ci), Cincinnati, Ohio 45268, EPA–600/4-79-020, Method 375.4.

16.2 "OSHA Safety and Health Standards, General Industry," (29CFR 1910), Occupational Safety and Health Administration, OSHA 2206, revised January 1976.

16.3 "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

16.4 "Standard Practices for Sampling Water," ASTM Annual Book of Standards, Part 31, D3370-76, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103-1187, 1980.

16.5 "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL-Ci, Cincinnati, OH 45268, EPA-600/4-79-019, March 1979.

16.6 "German Standard Methods for the Examination of Water, Wastewater, and Sludge," Deutsches Institut fhr Normung e.V., D-10772, Berlin, DIN Method 38402 Part 51, May 1986.

16.7 Spectroquant® NOVA 60 Manual, Merck KGaA, Frankfurter Strasse 250, Darmstadt 64271, Germany, Release, July 1998.

17.0 Tables and Validation Data

Table 1. Product Range, Number, and Usage Information

Range mg/L	Product Number	Sample Volume (ml)	NOVA 60 Test Code	Wavelength nm
5-250	14548	5	64	525
100-1,000	14564	1	82	820

Table 2. Calibration Standard Preparation

Product # Range (mg/L)	SO_4 stock solution 1000 mg/L	SO_4 working standard Volumes (ml)*	SO_4 Concentration (mg/L)
14548 (5-250)	Cat. No.19813	2.5-7.5-12.5-17.5-22.5	25-75-125-175-225
14564 (100-1,000)	Cat. No.19813	10-30-50-70-100	100-300-500-700-1000

* Dilute all working calibration standards to 100 ml in volumetric flasks.

Table 3. Acceptance Criteria for Performance Tests

Acceptance Criterion	Section	Limit (%)
Initial precision and recovery	9.2.2	
SO_4 Precision (s)	9.2.2.2	15
SO_4 Recovery (X)	9.2.2.2	82 - 112
Matrix spike/matrix spike duplicate	9.3	
SO_4 Recovery	9.3.4	81 - 113
SO_4 RPD	9.3.5	15
Ongoing precision and recovery	9.6	
SO_4 Recovery	9.6	81 - 113

18.0 Definitions

18.1 The definitions and purposes are specific to this method, but have been conformed to common usage as much as possible.

18.1.1 Symbols

°C	degrees Celsius
<	less than
%	percent

18.1.2 Alphabetical Characters

g	gram
L	liter
mg	milligram
mg/L	milligram per liter
ml	milliliter
nm	nanometer
µm	micrometer

18.2 Definitions, acronyms, and abbreviations.

18.2.1 Analyte: SO_4, which is tested for by this method.

18.2.2 Analytical batch: The set of samples analyzed at the same time, to a maximum of 20 samples. Each analytical batch must be accompanied by a laboratory blank (Section 9.4), and ongoing precision and recovery sample (OPR, Section 9.6), a matrix spike and matrix spike duplicate (MS/MSD, Section 9.3).

18.2.3 IPR: See initial precision and recovery.

18.2.4 Initial precision and recovery (IPR): Four aliquots of the diluted SO_4 standard analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed the first time this method is used and any time the method or instrument is modified.

18.2.5 Laboratory blank (method blank): An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with samples. The laboratory blank is used to determine if analyte or interferences are present in the laboratory environment, or the reagents.

18.2.6 Matrix spike (MS) and matrix spike duplicate (MSD): Aliquots of environmental sample to which known quantities of the analyte are added in the laboratory. The MS and MSD are prepared and/or analyzed exactly like a field sample. Their purpose is to quantify any additional bias and imprecision caused by the sample matrix. The background concentration of the analyte in the sample matrix must be determined in a

separate aliquot and the measured values in the MS and MSD corrected for background concentrations.

18.2.7 May: This action, activity, or procedural step is neither required nor prohibited.

18.2.8 May not: This action, activity, or procedural step is prohibited.

18.2.9 Method detection limit (MDL): The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

18.2.10 Memo chip: See transponder.

18.2.11 Minimum level (ML): The lowest level at which the entire analytical system gives a recognizable signal and acceptable calibration point of the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and preparation procedures have been employed.

18.2.12 Must: This action, activity, or procedural step is required.

18.2.13 OPR: See ongoing precision and recovery standard.

18.2.14 Ongoing precision and recovery standard (OPR): A laboratory blank spike with known quantities of analyte. The OPR is treated exactly like a sample. Its purpose is to establish performance of the method by the analyst.

18.2.15 Quality control sample (QCS): A sample containing analyte of interest at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that heave been prepared independently from the normal preparation process.

18.2.16 Reagent water: Water demonstrated to be free from contaminants.

18.2.17 Shall: This action, activity, or procedural step is required.

18.2.18 Should: This action, activity, or procedural step is suggested, but not required.

18.2.19 Spectroquant® NOVA 60: The filter photometer which can be used to perform the photometric measurements of the reacted Spectroquant® SO_4 cell tests.

18.2.20 SO_4: See sulfate.

18.2.21 Spectroquant® SO_4 cell test: The pre-measured reagent test cells for the testing of sulfate.

18.2.22 Sulfate: The analyte which is tested with this method.

18.2.23 Transponder: The memo chip, which contains updated information, which may include new methods and updated calibration information for downloading into the Spectroquant® NOVA 60 filter photometer.