OTM-59, Method for the correction of residual ammonia in Condensable Particulate Matter Samples Collected according to Method 202

Note: Please submit a copy, either electronic or paper, of any test report from application of this OTM to EPA's Measurement Technology Group.

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Measurement Technology Group Office of Air Quality Planning and Standards U.S. Environmental Protection Agency (Mail Code E143-02) Research Triangle Park, NC 27711

### 1.0 Scope and Applicability

1.1 Scope.

This method was developed to supplement the measurement of condensable particulate matter (CPM) conducted according to Method 202 (40 CFR 63, appendix A), when appropriate under circumstances identified in *the EPA memo titled: "*Condensable PM Adjustment for Ammonium Sulfate Formation in Method 202 Test Results". The procedures in this method regarding sample preparation may be used in addition to requirements in Method 202, when applicable. This method includes requirements for additional analytical measurement of sulfate and cations in the prepared (post-extraction) aqueous fraction (*see section 8.3 of this method*). The method describes the procedures for correcting the measured Method 202 data for any ammonia associated with ammonium sulfate that was collected in the sample.

1.2 Applicability. The procedures included in this method and adjustments to CPM measurements. described in this method are only intended for use in the context of New Source Review (NSR) permitting on a prospective basis, such as 1) to determine applicability or NSR permitting requirements, 2) support a permit application, or 3) derive emissions limitations in a permit. The application of this method is dependent on the relevant stationary source taking the following steps:

(a) demonstrating a measurable gaseous ammonia concentration, according to Method 320 (40 CFR Part 63, appendix A) or an appropriate ammonia monitor, and the presence of ammonia and sulfur dioxide in the stack effluent.

(b) directly measuring ammonia during the time of Method 202 using an approved measurement approach, and;

(c) documenting ammonia being injected by the source for NOx control, including ammonia feed rate, pre-SCR, and post SCR NOx concentrations, when applicable.

1.3 Additional Methods. To obtain reliable results, you should have a thorough knowledge of the following test methods that are found in appendices A-1 through A-3 and A-6 to part 60, and in appendix M to part 51:

### (a) Method 1— Sample and velocity traverses for stationary sources.

- (b) Method 2— Determination of stack gas velocity and volumetric flow rate (Type S pitot tube).
- (c) Method 3— Gas analysis for the determination of dry molecular weight.
- (d) Method 4— Determination of moisture content in stack gases.
- (e) Method 5— Determination of particulate matter emissions from stationary sources.
- (f) Method 17—Determination of particulate matter emissions from stationary sources (in-stack filtration method).
- (g) Method 201A— Determination of PM10 and PM2.5 emissions from stationary sources (Constant sampling rate procedure).
- (h) Method 202 Dry Impinger method for Determining Condensable Particulate Emissions from Stationary Sources
  - (i) Method 320 Measurement of vapor phase organic and inorganic emissions by extractive Fourier transform infrared (FTIR) spectroscopy

1.4 Limitations. The CPM adjustment described in this method is not appropriate for determining compliance with emission limitations that were derived without applying this adjustment methodology and/or unless specifically incorporated into the applicable State Implementation Plan (SIP).

### 2.0 *Summary of Method.*

This method describes the procedures (sampling and analytical) for calculating an adjusted CPM value based on the determination of ammonium and sulfate ions in Method 202 (40 CFR part 51, Appendix M) aqueous fraction of a Method 202 samples via ion chromatography. Depending on the applicability, a source may analyze the aqueous fraction, that includes the filter extraction, prior to neutralization for ammonium and sulfate ions using ion chromatography, using an appropriate column for each ion species. A source could then determine the moles of ammonium sulfate collected in the sample, given the known 2:1 molar ratio of ammonium to sulfate in ammonium sulfate. A source may then correct the CPM measured by Method 202 by subtracting the mass of ammonium in the ammonia sulfate collected.

### 3.0 Definitions

3.1 *Condensable Particulate Matter means* material that is vapor phase at stack conditions, but condenses and/or reacts upon cooling and dilution in the ambient air to form solid or liquid PM immediately after discharge from the stack.

3.2 *Continuing Calibration Verification means* an ongoing verification of an instrument's calibration using standard reference material.

3.3 *Duplicate Analysis means* a separate and distinct measurements of a sample using the same analytical instrument.

3.4 *Independent Calibration Verification* means an independent verification of an instrument's calibration using a different a separate standard reference material than what was used to determine an instruments calibration.

3.5 *Lower limit of quantification or LLOQ* means the lowest concentration of an analyte that can be quantified in a sample with acceptable accuracy and precision and is generally defined as 3 times the MDL value.

3.5 *Matrix Spike* means a separate aliquot of the sample spiked with known concentrations of the analytes of interest. 3.6 *Minimum Detection Limit (MDL)* means the lowest mass a target analyte greater than zero that can be estimated and reported by your candidate analytical technique. The MDL is statistically derived from replicate low level measurements near your analytical instrument's detection level according to the procedures found in Section 15.2 of Method 301 (40 CFR 63, appendix A).

3.7 *NIST* means the National Institute of Standards and Technology, located in Gaithersburg, Maryland. NIST coordinates the Federal Government policy on the conversion to the SI by Federal agencies and on the use of the SI by the United States.

3.8 *New Source Review or NSR* means the Clean Air Act program under sections 110(a)(2)(C), 165, and 173 of the Clean Air Act that require owners or operators of stationary sources of air pollution to obtain permits limiting air emissions before they begin construction that results in increases in emissions.

3.9 *Standard Reference Materials (SRM) are* certified reference materials (CRMs), issued under the NIST trademark that are well-characterized using state-of-the-art measurement methods and/or technologies for the determination of chemical composition and/or physical properties.

3.10 *State Implementation Plan* means a collection of regulations and documents used by a state, territory, or local air district to implement the National Ambient Air Quality Standards, or NAAQS, and to fulfill the requirements of Section 110 and related provisions in Title I of the Clean Air Act.

### 4.0 Interferences.

Possible interferences in this method could be the result of excess anion or cation concentrations which could make it difficult to resolve the target analytes. Interferences can be mitigated by using high quality reagent materials and ensuring proper chromatographic techniques to resolves the target analytes.

# 5.0 Safety.

Disclaimer. Because the performance of this method may require the use of hazardous materials, operations, and equipment, you should develop a health and safety plan to ensure the safety of your employees who are on site conducting the particulate emission test. Your plan should conform with all applicable Occupational Safety and Health Administration, Mine Safety and Health Administration, and Department of Transportation regulatory requirements. Because of the unique situations at some facilities and because some facilities may have more stringent requirements than is required by State or federal laws, you may have to develop procedures to conform to the plant health and safety requirements.

### 6.0 Equipment and Supplies.

The equipment used to collect the samples are described in Section 6.0 of Method 202. In addition, this section includes the equipment and supplied needed to conduct the analysis of anions and cations. This section does not list the common laboratory glassware which could be associated common laboratory practices.

6.1 Ion chromatograph (IC). Capable of delivering 1 to 5 mL of eluent per minute at a pressure of 1000 to 4000 psi (6.5 to 27.5 MPa). The chromatograph must be equipped with an injection valve and set up with the following components identified in this section. You may choose to use dedicated ICs for anion and cation analysis, respectively.

6.1.1 (Optional) Pre-Column(s). A guard column specific for either cation or anion analysis placed before the separator column.

6.1.2 Separator (or analytical) column(s). A column packed with the appropriate exchange resin for the compound of interests. For example, the anion resin column must be suitable for resolving fluoride, bromide, chloride, nitrite, nitrate, phosphate, and sulfate; and the cation resin column must be suitable for resolving ammonium, sodium, and calcium.

6.1.3 (Optional) Conductivity suppressor(s). An ion exchange-based device that can convert or remove the interfering ions from the eluent stream.

6.1.4 Conductivity detector. A low-volume, flow-through, temperature compensated, electrical conductivity cell, equipped with a meter capable of reading from 0 to 1,000 Siemens/cm on a linear scale.

6.1.5 Pump. Capable of delivering a constant flow of approximately 1 to 5 mL/min throughout the test and tolerating a pressure of 1000 to 4000 psi (6.5 to 27.5 MPa).

6.2 Autosampler. System capable of delivering sample to the IC and the associated equipment needed for the delivery, as described by instrument manufacturer's instructions. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory must satisfy itself and document that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. Alternatively, a syringe with a minimum capacity of 1 mL, equipped with a male pressure fitting may be used.

6.3 Data Recorder. Appropriate chromatographic data and control software to acquire data. An integrator or recorder can be used to integrate the area under the chromatographic peaks. If an integrator is used, the maximum area measurement must be within the linear range of the integrator. The recorder should be compatible with the detector output with a full-scale response time of 2 seconds or less.

6.4 Analytical balance. Capable of weighing to the nearest 0.0001 g.

6.5 Pipets, Class A volumetric flasks, beakers. Assorted sizes.

7.0 Reagent and Standards.

7.1 Reagent-grade chemicals. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

7.2 Reagent Water. DI or equivalent water with a conductivity is  $\leq$ 5.0  $\mu$ S/cm.

7.3 0.04 N Sulfuric Acid. To prepare 1 L, slowly add 1 ml of concentrated 17.9 M H2SO4 to about 900 ml of water while stirring and adjust the final volume to 1 L using additional water. Shake well to mix the

solution. Alternative preparation approaches may also be used so long as the desired normality is achieved.

7.3 Eluent. Follow manufacturer guidance for the proper eluent for each specific column and analyte combination. Use laboratory best practices in the preparation and storage of eluent.

7.4 Calibration Standards. All calibrations standards solution must be traceable to an SRM from the National Institute of Standards and Technology .

7.4.1 Primary sulfate standard, 1000 mg/L as SO<sub>4</sub>, other concentrations may be used as appropriate.

7.4.2 Primary ammonium standard 1000 mg/L as  $NH_4$  other concentrations may be used as appropriate.

7.4.3 Independent standard solution. A NIST traceable standard obtained from a source or supplier independent of that for the calibration standards and is used to confirm the integrity of the calibration standards used.

7.5 Preparation of Calibration Standards.

7.5.1 Sulfate Calibration Standards. Prepare a blank and at least four sulfate calibration standards. The sulfate calibrations standards must be prepared using calibrated volumetric equipment, such as Class A volumetric flasks or auto-pipettes using reagent water. The sulfate calibration standards must bracket the expected range of the measurement, with the lowest calibration standard equivalent to the lower limit of quantitation. Sulfate Calibration standards must be prepared monthly.

7.5.2 Ammonium Calibration Standards. Prepare a blank and at least six ammonium calibration standards. The ammonium calibrations standards must be prepared using calibrated volumetric equipment, such as Class A volumetric flasks or auto-pipettes using 0.04N H<sub>2</sub>SO<sub>4</sub>. The calibration standards must bracket the expected range of the measurement, with the lowest calibration standard equivalent to the lower limit of quantitation. Calibration standards must be stored cold (<6° C or per the manufacturer's instructions) and prepared monthly.

8.0 Sample Collection, Preservation, Storage, and Transport.

8.1 You must collect the Method 202 sample(s) as described in Section 8.1 through 8.5.3 of Method 202, however the post-test purge of the entire sampling train for at least two hours instead of the one hour required in Method 202. The purge must be conducted as soon as possible after sampling either at the sampling location or in a clean area on-site of the test. The purge must begin no later than 30 minutes after the conclusion of sampling.

Note: While excess sulfur dioxide is purged from the sampling relatively quickly, it has been shown that it may take up to two hours or more to purge free ammonia from the glassware and aqueous impingers.

8.2 Recover the sampling train in accordance with Section 8.5.4 of Method 202 and transport the sampling train according to Section 5.5.5 of Method 202.

8.3 Conduct the analysis of the sample fractions as described in Method 202, however replace Section 11.2.2.1 of Method 202 with the following:

Determine the inorganic fraction weight. Tare a clean 500-ml or smaller beaker using a balance to the nearest 0.5 g. Transfer the aqueous fraction from the aqueous extraction to the tared beaker and using the balance determine the volume of the prepared aqueous fraction ( $AQ_s$ ) beaker. If necessary, you may use a larger beaker if the aqueous fraction is greater than 500-ml. Collect two aliquots of the sample (AQa) from the beaker using a Class A pipette or calibrated auto-pipette into two separate VOA vials and mark one for sulfate analysis and one for ammonium analysis. The volume removed should not exceed 20% of the total sample volume, to limit higher aliguot factors. Record the volume removed. Add 1:1 concentrated H2SO4 to the ammonium VOA vial to bring solution to 0.04N H2SO4, seal the vial, and shake to mix. Make sure both vials are sealed. Evaporate the aqueous fraction ( $AQ_s$ ) to no less than 10 ml liquid on a hot plate or in the oven at 105°C and allow to dry at room temperature (not to exceed 30°C (85°F)). You must ensure that water and volatile acids have completely evaporated before neutralizing nonvolatile acids in the sample. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight. (See section 3.0 for a definition of constant weight.) Report results to the nearest 0.1 mg on the CPM Worktable (see Figure 6 of section 18) and proceed directly to section 11.2.3. If the residue cannot be weighed to constant weight, re-dissolve the residue in 100 ml of deionized distilled ultra-filtered water that contains 1 ppmw (1 mg/L) residual mass or less and continue to section 11.2.2.2.

8.4 Conduct the analysis to determine the CPM mass as defined in Method 202 and determine the concentration of sulfate and ammonium in the aqueous fraction as detailed in Section 10 and 11 of this method.

8.5 CPM Correction.

8.5.1 Determine the moles of sulfate and ammonium collected in each sample fraction as detailed to Equation 12-1 and 12-2, respectively, of this method.

8.5.2 Determine the maximum number of moles of ammonium sulfate possibly present given the 2:1 molar ratio of ammonium to sulfate in ammonium sulfate.

For example, if there are 2 moles of ammonium and 4 moles of sulfate, then the maximum moles of ammonium sulfate (MMAS) are 1, because it takes 2 moles of ammonium and 1 mole of sulfate to form 1 mole of ammonium sulfate.

Likewise, if there are 4 moles of ammonium and 1 mole of sulfate, then the maximum moles of ammonium sulfate are 1, because, while there are enough moles of ammonium to form 2 moles of ammonium sulfate, there are only enough moles of sulfate to form 1 mole of ammonium sulfate.

8.5.3 Determine the maximum mass adjustment factor (MMAF) for each AQ<sub>a</sub> sample as detailed in Equation 12-3 of this method.

8.5.5 After completing the gravimetric analysis of the relevant 202 fractions, calculated the mass of CPM as detailed in Equation 12-4 of this method, correcting for the volume difference in the mass of the inorganic CPM.

8.5.4 From the results of the associated Method 202 sampling train, corrected the results using the sample specific MMAF as detailed in Equation 12-5 of this method.

## 9.0 *Quality Control.*

QA/QC test or specification	Acceptance Criteria	Frequency	Consequence if not met
Supplementary measurement of	Completed	Each test run	Correction method
Ammonia during the test			cannot be applied
Calibration standards traceable to SRM	NIST traceable	Each test	Repeat calibration
Establish the MDL	Completed	once a year	Correction method
			cannot be applied
Sulfate calibration curve	At least 4 points, bracketing	Each initial	Repeat calibration or
	the expected concentration	calibration	analyze another
			calibration point
Ammonium calibration curve	At least 6 points, bracketing	Each initial	Repeat calibration or
	the expected concentration	calibration	analyze another
			calibration point
Lowest Calibration point	Approximately 3 times the	Each initial	Repeat calibration or
(ammonium and sulfate)	MDL or LLOQ	calibration	analyze another point at
			the LLOQ
Lowest Calibration point	+/- 50% of the true value	Each initial	Repeat lowest calibration
(ammonium and sulfate)		calibration	point
Calibration Correlation	R <sup>2</sup> of 0.995 or higher	Each	Repeat Calibration
		calibration	
Independent Calibration	+/- 10% of the true value	Each initial	Take corrective action,
verification		calibration	repeat ICV
Continuing Calibration Verification	+/- 10% of the true value	prior to and	Take corrective action,
		the end of	repeat CCV, if CCV does
		each batch	not meet criteria, void
		and/or every	any samples results from
		10 samples	the time of the last valid
			CCV
Post-test purge	Within 30 minutes and for	Each test run	Correction method
	2-hours		cannot be applied
Sample Duplicate Analysis	Relative percent difference	Each sample	Repeat the duplicate
	of 10%		injections and use the
			mean response of the
			quadruplicate samples.
Matrix Spike	Percent Recovery of 80 –	Each MS	Flag results
	120 % of the expected		
	mass		
Matrix Duplicate Analysis	Relative percent difference	Each MSD	Flag results
		-	
	of 10%		

Table 9-1 summarizes the QA/QC	performance criteria that are used	to validate the emissions data.
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### 10.0 Calibration and Standardization.

10.1 Establish ion chromatographic operating parameters consistent with the recommendation by the manufacturer and/or internal standard operating procedures develop by the analytical lab for sulfate analysis.

10.2 Prepare the sulfate calibration standards as required in Section 7.5.

10.3 The laboratory must establish the MDL (also known as limit of detection or LOD) for sulfate. The MDL must be established prior to any compliance analysis and must be confirmed at least annually. If an instrument has had any significant maintenance performed, the MDL mut also be reestablished. The MDL must be established according to the procedures in Section 15.2 of Method 301. Flag appropriately any results that lie between the MDL and the LLOQ.

10.4 Inject standards starting with the lowest sulfate concentration standard and increasing in concentration to the highest standard using a fixed injection volume for each calibration standard. Record the peak area responses and retention times for each analyte. The sulfate calibration standards must bracket the expected range of the measurement. The injection volume used for calibrations must match the injection volumes used for standards.

10.5 Establish the sulfate calibration curves by plotting the peak area responses for each standard against the corresponding concentrations. You may use the least squares linear regression to calculate the calibration curve formula, or a weighted least squares regression may also be performed using 1/concentration or 1/(concentration)<sup>2</sup> as the weighting factor. The acceptance criterion for the calibration curve should be a correlation coefficient of 0.995 or higher.

10.6 Verify the reporting limit of the method by evaluating the recovery of the lowest calibration standard. The acceptance criteria for the recovery is 50 - 150% of its true value of the lowest calibration standard. If the criteria is not met, you should re-run the lowest calibration standard.

10.7 Verify the accuracy of the calibration curve by analyzing an independent calibration verification (ICV) standard.

10.7.1 The ICV standard must be prepared from an independent (second source) material at or near the mid-range of the calibration curve.

10.7.2 The acceptance criteria for the ICV standard must be no greater than  $\pm$  10% of its true value. If not, the instrument must be recalibrated before samples are analyzed.

10.8 Repeat the procedures identified in Section 10.1 through 10.7 for ammonium in lieu of sulfate, with the following exception: Ammonium calibration can be quadratic. All other requirements are the same.

10.9 Each day in which the instrument is operating, verify the accuracy of each (i.e., sulfate and ammonium) calibration curve developed in Section 10.5 through the analysis of a continuing calibration verification (CCV) standard. You must also verify the accuracy of each calibration curve for every batch.

10.9.1 The CCV must be made from the same material as the initial calibration standards, at or near mid-range.

10.9.2 The acceptance criteria for the CCV standard must be no greater than  $\pm$  10% of its true value. If the CCV standard result does not meet the acceptance criterion, sample analysis must be discontinued, and the cause determined. Once the cause is determined, the CCV must be reprepared/reinjected and must meet acceptance criteria. If the second analysis does not meet acceptance criteria, the instrument must be recalibrated. All samples analyzed after the last acceptable CCV must be reanalyzed.

### 11.0 Analytical Procedures

#### 11.1 Sample Analysis - Sulfate

11.1.1 Establish ion chromatographic operating parameters for sulfate exactly equivalent to those used for calibration in Section 10. Establish a stable baseline as suggested by the manufacturer and/or internal standard operating procedure develop by the analytical lab for sulfate analysis.

11.1.2 Inject a sample of reagent water and determine if any sulfate ions appear in the chromatogram. If any of these ions are present repeat the load/injection procedure until they are no longer present.

11.1.3 Inject a fraction of the unpreserved AQ<sub>a</sub> sample in duplicate and record the resulting analyte peak sizes in area units as well as the peak retention times. If the peak area responses exceed the highest peak area response from the calibration curve, invalidate the data and dilute the sample in reagent water to bring it within the range of the calibration curve and repeat the sample analysis.

11.1.4 Determine the mean response of the valid duplicate injections to determine the concentration  $(C_s)$  of the AQ<sub>a</sub> sample. The values of the duplicate analysis must be within relative percent difference of 10 percent. If not, repeat the duplicate injections and use the mean response of the quadruplicate samples.

11.1.6 Repeat Section 11.1.3 and 11.1.4 for each AQ<sub>a</sub> sample, including the field train blank.

11.2 Sample Analysis - Ammonium

11.2.1 Establish ion chromatographic operating parameters for ammonium exactly equivalent to those used for calibration in section 10. Establish a stable baseline as suggested by the manufacturer and/or internal standard operating procedure develop by the analytical lab for ammonium analysis.

11.2.2 Inject a sample of reagent 0.04N  $H_2SO_4$  and determine if any ammonium ions appear in the chromatogram. If any of these ions are present, repeat the load/injection procedure until they are no longer present.

11.2.3 Inject a fraction of the H<sub>2</sub>SO<sub>4</sub> preserved AQ<sub>a</sub> sample in duplicate and record the resulting analyte peak sizes in area units as well as the peak retention times. If the peak area responses exceed the highest peak area response from the calibration curve, invalidate the data and dilute the sample in reagent water to bring it within the range of the calibration curve and repeat the sample analysis.

11.2.4 Determine the mean response of the valid duplicate injections to determine the concentration  $(C_a)$  of the AQ<sub>a</sub> sample. The values of the duplicate analysis must be within relative percent difference of 10 percent (see Equation 12-7). If not, repeat the duplicate injections and use the mean response of the quadruplicate samples.

11.2.6 Repeat Section 11.2.3 and 11.2.4 for each AQ<sub>a</sub> sample, including the field blank.

11.3 Quality Control Samples – Matrix Spike (MS) and Matrix Spike Duplicate (MSD).

11.3.1 To determine the presence of any potential bias or matrix effects prepare matrix spikes in duplicate by spiking a known mass (SA) of sulfate to a fraction of the AQ<sub>a</sub> sample. Inject the matrix spike fraction sequentially with the sample fractions in (see Section 11.1.3). Determine the mean of the matrix spike sample results (MSSR) and sample results (SR). Determine the percent recovery of the MS according to Equation 12-6.

11.3.2 The expected spike recoveries are 80-120 percent. If the spike recoveries exceed the limits flag the results.

11.3.3 Determine the relative percent difference of the MSD according to Equation 12-3, if the relative percent difference exceeds 10 percent, flag the results.

11.3.4 Repeat the procedures identified in Section 11.3.1 through 11.3.4 for ammonium using the sample preparation identified in Section 11.2.

12.0 Calculations.

12.1 Nomenclature.

- 18.04 = molar mass of ammonium, g/mol
- 96.06 = molar mass of sulfate, g/mol
- 1000 = conversion of mg to gram
- AQ<sub>s</sub> = Aqueous sample volume, ml.
- $AQ_a$  = Aliquot sample volume, ml.
- *C*<sub>a</sub> = Concentration of ammonium determined from the IC measurement, mg/ml.
- *C*<sub>s</sub> = Concentration of sulfate determined from the IC measurement, mg/ml.
- CM<sub>cpm</sub> = Corrected mass of CPM, mg.
- $M_{CPM}$  = Measured mass of CPM according to Method 202, mg
- D = Duplicate sample result, mg/l
- *FBC*<sub>a</sub> = Concentration of ammonium in field blank, mg/ml.
- *FBC*<sub>s</sub> = Concentration of sulfate in field blank, mg/ml.
- $M_{cpm}$  = Mass of CPM according to Method 202, mg.
- *M<sub>i</sub>* = Mass of inorganic (aqueous) fraction, mg.
- *M*<sub>o</sub> = Mass of organic (aqueous) fraction, mg.
- MMAF = Maximum mass adjustment factor, mg
- MMAS = Maximum molar mass of ammonium sulfate, g/mol
- Mo<sub>a</sub> = moles of ammonium in sample
- Mo<sub>s</sub> = moles of sulfate in sample
- MSSR = Matrix spike sample result, % recovery
- RPD = Relative percent different, &
- S = Sample result, mg/l
- SA = Spike added, mg/l
- SR = Sample Result, mg/l

12.2 Equations. Use the following equations to complete the calculations required in this test method.

12.2.1 Moles of sulfate. Calculate the moles of sulfate in the aqueous fraction according to the following equation:

Equation 12-1 
$$Mo_s = \frac{\left(\frac{C_s \cdot FBC_s}{AQ_s}\right)/1000}{96.06}$$

12.2.2 Moles of ammonium. Calculate the moles of sulfate in the aqueous fraction according to the following equation:

Equation 12-2 
$$Mo_a = \frac{\left(\frac{C_a - FBC_a}{AQ_s}\right)/1000}{18.04}$$

12.2.3 Maximum mass adjustment factor (MMAF). Calculate the MMAF, dependent on the maximum number of moles of ammonium sulfate (MMAS) possibly present in a sample, given the 2:1 molar ratio of ammonium to sulfate in ammonium sulfate:

12.2.4 Calculate the total mass of CPM corrected for volume:

Equation 12-4 
$$Mcpm = M_i(\frac{AQs-AQ_o}{AQ_s}) + M_o$$

12.2.5 Calculate the corrected mass of CPM according to the following equations:

Equation 12-5 
$$CM_{cpm} = M_{cpm} - MMAF$$

12.2.5 Determine the percent recovery for the matrix spike according to the following equation:

Equation 12-6 
$$\% R = \frac{(MSSR - SR)}{SA} \times 100$$

12.2.5 Determine the relative percent difference for each duplicate analysis according to the following equation:

Equation 12-7 
$$RPD = \frac{|S-D|}{(S-D)/2} \times 100$$

13.0 Method Performance [Reserved]

14.0 Waste Management [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures [Reserved]

17.0 References

(1) SW-846 Method 9056A, Determination of Inorganic Anions by Ion Chromatography, February 2007, https://www.epa.gov/sites/default/files/2015-12/documents/9056a.pdf

(2) Condition Test Method 13 (CTM-13), Determination of Sulfuric Acid Vapor Mist and Sulfur Dioxide Emissions from Kraft Recovery Furnaces (NCASI 8A), December 1996, https://www.epa.gov/sites/default/files/2020-08/documents/ctm-013\_0.pdf

(3) Condition Test Method 27 (CTM-27), Procedure for Collection and Analysis of Ammonia in Stationary Sources, August 1997, https://www.epa.gov/sites/default/files/2020-08/documents/ctm-027.pdf

(4) Memorandum, Condensable PM Adjustment for Ammonium Sulfate Formation in Method 202 Test Results, US EPA, 11/21/2024