Particulate (Including Ammonium Sulfate and Ammonium Sulfite) in Stack Gases Containing Sulfur Oxides and Ammonia

1.0   PRINCIPLE AND APPLICABILITY.

1.1   This method is applicable for the determination of particulate in stack gases containing sulfur oxides and ammonia and is a modification of the U.S. Environmental Protection Agency (EPA) Method 8 (Reference 1) and Method 5 (Reference 2).

1.2   The sampling train consists of a heated, preweighed glass fiber filter immediately in back of the probe followed by three impingers in series, the first containing 80% isopropanol (IPA) in water and the next two containing 6% hydrogen peroxide (H_2O_2) in water (Reference 3).

1.3   The particulate weight determined by this method includes the weight of ammonium sulfate [$(NH_4)_2SO_4$] and ammonium sulfite [$(NH_4)_2SO_3$] created from the sulfuric acid (H_2SO_4), sulfur trioxide (SO_3), sulfur dioxide (SO_2), and ammonia (NH_3) in the stack gas. However, stack $(NH_4)_2SO_4$ and stack $(NH_4)_2SO_3$ are analyzed and reported by this method, so their contributions to the total particulate weight can be determined. Also, this method provides analyses for free (unreacted with NH_3) H_2SO_4 and free SO_2, so weights for both of these entities are reported.

1.3.1   Most of the particulate is collected on the filter; the remainder is collected in the probe wash and in the IPA absorber. This particulate may be water- or acid-soluble and cannot be determined by a filtration technique.

1.3.2   Note: One mole of H_2SO_4, SO_3, or SO_2 reacts with two moles of NH_3.

1.3.3   Sulfuric acid (H_2SO_4) mist may be caught on the heated filter and combined with NH_3 from the stack to form $(NH_4)_2SO_4$. The remaining H_2SO_4 mist and NH_3 are trapped by the IPA absorber, which also collects SO_3 and converts it to H_2SO_4. (Ammonium sulfate may also be found in the probe wash.)

1.3.4   In the presence of sufficient NH_3, SO_2 forms particulate $(NH_4)_2SO_3$, which is trapped in the probe, on the filter, and in the IPA absorber (Reference 4). The remaining (free) SO_2 from the stack passes through the IPA solution to the second and third (H_2O_2) impingers, where it is converted to H_2SO_4.

1.3.5   In the majority of cases, there will be more sulfur oxides in the stack than NH_3. Therefore, NH_3 will be the limiting factor in the reaction with H_2SO_4, SO_3, and SO_2.

1.3.6   If the stack NH_3 is much greater (by at least a factor of 1.2) than twice the total moles of $H_2SO_4 + SO_3 + SO_2$, then this laboratory addresses the problem on an individual basis, taking into account the stack process and the desired results from the analyses.

1.4   Reported data.

1.4.1   Free SO_2. Each H_2O_2 absorber solution is analyzed for sulfate (as SO_2) by ion chromatography (IC) according to the Texas Air Control Board's (TACB's) Laboratory Methods Manual, Method 26a (Reference 5). From these analyses, the total weight of free SO_2 is reported.

1.4.2   Total stack $(NH_4)_2SO_3$, total stack $(NH_4)_2SO_4$, total free H_2SO_4, and total particulate.

1.4.2.1 The filter is extracted with distilled deionized (DDI) water. A portion of the extracting solution is then analyzed for ammonium, sulfite, and sulfate ions by IC according to the TACB's Laboratory Methods Manual, Methods 26b and 26a (Reference 5). From these analyses stack $(NH_4)_2SO_3$ on the filter, stack $(NH_4)_2SO_4$ on the filter, and free H_2SO_4 on the filter are calculated. The remaining portion of the extracting solution (along with the filter and undissolved particulate) is transferred into a clean, tared beaker, is made basic with ammonium hydroxide (NH_4OH) (to fix any free H_2SO_4), and then is evaporated to dryness. Particulate collected on the filter is then calculated from the final weight of the beaker (plus contents), the tared weight of the beaker, the weight of the clean filter, and the weights of stack $(NH_4)_2SO_3$ on the filter, stack $(NH_4)_2SO_4$ on the filter, and free H_2SO_4 on the filter.

1.4.2.2 The probe wash and the IPA absorber are analyzed separately. Each volume is adjusted with DDI water. Then a portion of each resulting solution is analyzed for ammonium, sulfite, and sulfate ions by IC as in Step 1.4.2.1. From these analyses stack $(NH_4)_2SO_3$ in the probe wash and IPA absorber, stack $(NH_4)_2SO_4$ in the probe wash and IPA absorber, and free H_2SO_4 in the probe wash and IPA absorber are calculated. The remaining portion of each solution is transferred into a clean, tared beaker, evaporated down to 100 mL, made basic with NH_4OH (to fix any free H_2SO_4), and then evaporated to dryness. Particulate collected in the probe wash and the IPA absorber is then calculated from the final weight of each beaker (plus contents), the tared weight of each beaker, and the weights of stack $(NH_4)_2SO_3$ in

the probe wash and IPA absorber, stack $(NH_4)_2SO_4$ in the probe wash and IPA absorber, and free H_2SO_4 in the probe wash and IPA absorber.

1.4.2.3 Total stack $(NH_4)_2SO_3$, total stack $(NH_4)_2SO_4$, total free H_2SO_4, and total particulate are calculated from the analyses described in Steps 1.4.2.1 and 1.4.2.2.

2.0   RANGES AND LOWER DETECTABLE LIMITS.

2.1   According to EPA Method 8 (Reference 1), the minimum detectable limits of the method are 0.00005 g SO_3/m^3 and 0.0012 g SO_2/m^3.

2.2   No upper limits have been established.

2.3   The lower detectable limit for the particulate is limited by the weighing process.

3.0   PRECISION.

3.1   The precision of the method, as determined by interlaboratory tests, was found to be 0.00719 g H_2SO_4/m^3 and 0.0223 g SO_2/m^3 (Reference 1).

3.2   The precision of the particulate measurement in stack gas, as a result of a collaborative test program showed a standard deviation percent of mean value of 10.4% (Reference 2).

4.0   ACCURACY.

4.1   The accuracy of the method measured as interlaboratory precision has been shown to be 0.00803 g H_2SO_4/m^3 and 0.0311 g SO_2/m^3 (Reference 1).

4.2   The accuracy of the particulate measurement in stack gas, as a result of an interlaboratory collaborative test program, showed a standard deviation percent of mean value of 12.1% (Reference 2).

5.0   INTERFERENCES.

5.1   Possible interferences with this method include fluorides and water-soluble, non-ammonium salts of H_2SO_4.

5.1.1   Fluorides react with the preweighed glass fiber filter causing it to lose weight as silicon tetrafluoride, thereby interfering with the gravimetric determination of undissolved particulate on the filter.

5.1.2   Water-soluble, non-ammonium salts of H_2SO_4 (such as Na_2SO_4, $MgSO_4$, and amine sulfates) will be reported as free H_2SO_4, causing the reported free H_2SO_4 to be high and the reported particulate to be low.

5.1.3   The greatest source of error occurs during the sampling and collection phase instead of the analysis phase (Reference 2).

6.0   REAGENTS.

All reagents should be ACS reagent-grade or better.

6.1   DDI water.

6.2   Concentrated NH_4OH.

6.3   Ethanol.

6.4   Phenolphthalein indicator. Dissolve 0.05 g phenolphthalein in 50 mL of ethanol, and add 50 mL of DDI water.

7.0   APPARATUS.

7.1   Glassware.

125-mL Erlenmeyer flasks with air condensers.
250-mL graduated cylinders.
250-mL beakers.
50-mL volumetric flasks.
250-mL volumetric flasks.

7.2   Pipettors, 5- and 10-mL.

7.3   Evaporating oven.

Temperature range: 55 to 100° C.
Recirculating airflow.

7.4   Analytical balance.

Sensitivity of 0.0001 g.
Precision of 0.0001 g.

10.0   SAMPLE ANALYSIS.

10.1   Peroxide absorbing solutions.

Analyze the samples in the 50-mL flasks from Step 8.2.2 for sulfate ion (to be reported as free SO_2) by using the IC method.

10.2   Filter extract.

10.2.1 Analyze the samples in the 50-mL flasks from Step 8.3.4 for ammonium, sulfite, and sulfate ions by using the IC methods.

10.2.2 Obtain the final (constant) weight on the beakers from Step 8.3.8 by using the procedures in the TACB's Laboratory Methods Manual, Method 23 (Reference 5).

10.3   Probe wash.

10.3.1 Analyze the probe-wash sample in the 50-mL flask from Step 8.4.4 for ammonium, sulfite, and sulfate ions by using the IC method.

10.3.2 Obtain the final (constant) weight on the probe-wash beaker from Step 8.4.7 by using the procedures used in Step 10.2.2.

10.4   IPA absorber.

10.4.1 Analyze the IPA-absorber sample in the 50-mL flask from Step 8.4.4 for ammonium, sulfite, and sulfate ions by using the IC method.

10.4.2 Obtain the final (constant) weight on the IPA-absorber beaker from Step 8.4.7 by using the procedures used in Step 10.2.2.

11.0   CALCULATIONS.

11.1   Peroxide absorbers.

Calculate and report the weight for total free SO_2 given by:

$$A = (B + C)(10)(100)(0.667)$$

where A is the weight of total free SO_2 in μg,
B and C are the sulfate-ion concentrations in the diluted peroxide solutions from Step 10.1 in $\mu g/mL$,
10 is the dilution factor for each absorber,
100 is the volume for each absorber in mL, and
0.667 is the ratio of the molecular weight of SO_2 to the ionic weight of sulfate ion.

11.2   Filter extract.

11.2.1   Calculate the μmoles of stack ammonium ion in the filter extract given by:

$$D = E(10)(50) / (18)$$

where D is the μmoles of stack ammonium ion in the filter extract,
E is the stack ammonium-ion concentration in the diluted filter extract from Step 10.2.1 in μg/mL,
10 is the dilution factor,
50 is the volume of the extracting solution in mL, and
18 is the ionic weight of ammonium ion.

11.2.2   Calculate the μmoles of sulfite ion in the filter extract given by:

$$F = G(10)(50) / (80)$$

where F is the μmoles of sulfite ion in the filter extract,
G is the sulfite-ion concentration in the diluted filter extract from Step 10.2.1 in μg/mL,
10 is the dilution factor,
50 is the volume of the extracting solution in mL, and
80 is the ionic weight of sulfite ion.

11.2.3   Calculate the μmoles of sulfate ion in the filter extract given by:

$$H = I(10)(50) / (96)$$

where H is the μmoles of sulfate ion in the filter extract,
I is the sulfate-ion concentration in the diluted filter extract from Step 10.2.1 in μg/mL,
10 is the dilution factor,
50 is the volume of the extracting solution in mL, and
96 is the ionic weight of sulfate ion.

11.2.4   Theoretically, sufficient μmoles of stack ammonium ion will exist in the filter extract to fix all of the sulfite ion in the filter extract, so calculate the weight of stack $(NH_4)_2SO_3$ in the filter extract given by:

$$J = F(116)$$

where J is the weight of stack $(NH_4)_2SO_3$ in the filter extract in μg,
F is the μmoles of sulfite ion in the filter extract from Step 11.2.2, and
116 is the molecular weight of $(NH_4)_2SO_3$.

8.0   PROCEDURES.

8.1   Collection of sample.

8.1.1   Before the sampling trip begins, read Steps 8.3 and 8.4 in the TACB's Laboratory Methods Manual, Method 23 (Reference 5) for the preparation of the glass fiber filters and of the beakers used to evaporate the samples.

8.1.2   Prepare the filter and solution blanks to be analyzed by this procedure.

8.1.3   Subject the filter and solution blanks to the same field conditions that the samples experience except for being used in sampling.

8.1.4   Before sampling, perform quality control checks (see Step 12.0) on the IPA batch to be used in the first impinger and on the batch of DDI water or acetone to be used as the probe wash.

8.1.5   After sampling, the person sampling must mark the level of liquid on each sample container in case of loss during shipment. It is assumed that the probe wash will not exceed a volume of 150 mL and that each impinger will contain slightly less than 100 mL of absorber.

8.2   Preparation of the peroxide absorbers.

8.2.1   Using DDI water, adjust the volume of each H_2O_2 absorber to 100 mL.

8.2.2   Using a pipettor, dilute a 5-mL portion of each absorber to 50 mL with DDI water. Each absorber is now ready for analysis.

8.3   Preparation of the filters.

8.3.1   After sampling has been completed, use tweezers to remove the filter from the shipping container. Cut the filter into several pieces before placing it into a 125-mL Erlenmeyer flask equipped with an air condenser. (Extract a clean field filter in a separate flask as a blank.)

8.3.2   Rinse the filter shipping container with two 25-mL portions of DDI water, and pour the rinsings into the Erlenmeyer flask. (The total volume of the filter extract used in the calculations is 50 mL.)

8.3.3   Reflux gently for six to eight hours. Allow the sample to cool. Then verify that the total volume is still 50 mL; adjust with DDI water if necessary.

8.3.4   Allow the undissolved particulate in the extracting solution to settle. Then pipet a 5-mL aliquot of the filter extract into a 50-mL volumetric

flask and dilute to the mark with DDI water. This sample is now ready for analyses.

8.3.5   Place the remaining 45 mL of the filter extract, the filter, and the undissolved particulate from Step 8.3.3 into a clean, tared 250-mL beaker.

8.3.6   Rinse the Erlenmeyer flask with three 10-mL portions of DDI water, and pour the rinsings into the tared beaker.

8.3.7   Add five drops of the phenolphthalein solution and then enough drops of concentrated NH_4OH to turn the sample pink.

8.3.8   Evaporate to dryness in an oven at 95° C. This sample is now ready for analysis.

8.4   Preparation of the probe wash and the IPA absorber (each separately).

8.4.1   Using a clean, dry graduated cylinder, measure the volume of the IPA absorber. If its volume is less than 90 mL, void the sample.

8.4.2   Pour each sample into a 250-mL volumetric flask. (Include a probe wash and an IPA-absorber field blank equal to the volume of the field sample, and put each into a separate volumetric flask.)

8.4.3   Rinse the sample container and the graduated cylinder with DDI water, and pour the rinsings into the volumetric flask. Dilute the flask to the mark with DDI water.

8.4.4   Pipet a 10-mL aliquot of the sample into a 50-mL volumetric flask, and dilute to the mark with DDI water. This sample is now ready for analyses.

8.4.5   Place the remaining 240 mL of sample from Step 8.4.3 into a clean, tared 250-mL beaker.

8.4.6   Add five drops of the phenolphthalein solution and then enough drops of concentrated NH_4OH to turn the sample pink.

8.4.7   Evaporate to dryness in an oven at 95° C (50° C for the probe wash if it contains acetone). This sample is now ready for analysis.

9.0   CALIBRATION.

Follow the procedures given in the TACB's Laboratory Methods Manual, Methods 26b and 26a (Reference 5) for the calibration of the IC instruments.

11.2.5 Calculate the μmoles of stack ammonium ion remaining in the filter extract given by:

$$K = D - 2F$$

where K is the μmoles of stack ammonium ion remaining in the filter extract,
D is the μmoles of stack ammonium ion in the filter extract from Step 11.2.1, and
2F is the μmoles of stack ammonium ion already used to form $(NH_4)_2SO_3$ in the filter extract (F is from Step 11.2.2).

11.2.6 Calculate the weight of stack $(NH_4)_2SO_4$ in the filter extract given by:

$$L = 0.5K(132)$$

where L is the weight of stack $(NH_4)_2SO_4$ in the filter extract in μg,
K is the μmoles of stack ammonium ion remaining in the filter extract (from Step 11.2.5) that can react with 0.5K μmoles of sulfate ion in the filter extract, and
132 is the molecular weight of $(NH_4)_2SO_4$.

11.2.7 Calculate the weight of free H_2SO_4 in the filter extract given by:

$$M = (H - 0.5K)(98)$$

where M is the weight of free H_2SO_4 in the filter extract in μg,
H is the μmoles of sulfate ion in the filter extract from Step 11.2.3,
0.5K is the μmoles of stack $(NH_4)_2SO_4$ from Step 11.2.6, and
98 is the molecular weight of H_2SO_4.

11.2.8 Calculate the weight of particulate (including stack $(NH_4)_2SO_3$ and stack $(NH_4)_2SO_4$) in the filter extract given by:

$$N = P - Q - R + 0.1(J + L) - 0.9M(1.35)$$

where N is the weight of particulate (including stack $(NH_4)_2SO_3$ and stack $(NH_4)_2SO_4$) in the filter extract in μg,
P is the final weight of the filter-extract beaker from Step 10.2.2 in μg,
Q is the tared weight of the filter-extract beaker in μg,
R is the clean weight of the filter in μg,
0.1 is the fraction of the stack $(NH_4)_2SO_3$ and stack $(NH_4)_2SO_4$ removed from the filter extract for analysis,
J is the weight of stack $(NH_4)_2SO_3$ from Step 11.2.4 in μg,
L is the weight of stack $(NH_4)_2SO_4$ from Step 11.2.6 in μg,

0.9 is the fraction of the free H_2SO_4 remaining in the filter extract after removing some of the extract for analyses,
M is the weight of free H_2SO_4 in the filter extract from Step 11.2.7 in μg, and
1.35 is the ratio of the molecular weight of $(NH_4)_2SO_4$ to the molecular weight of H_2SO_4.

11.3   Probe wash.

11.3.1 Calculate the μmoles of stack ammonium ion in the probe wash given by:

$$S = T(5)(250) / (18)$$

where S is the μmoles of stack ammonium ion in the probe wash,
    T is the stack ammonium-ion concentration in the diluted probe wash from Step 10.3.1 in μg/mL,
    5 is the dilution factor,
    250 is the volume of the probe wash in mL, and
    18 is the ionic weight of ammonium ion.

11.3.2 Calculate the μmoles of sulfite ion in the probe wash given by:

$$U = V(5)(250) / (80)$$

where U is the μmoles of sulfite ion in the probe wash,
    V is the sulfite-ion concentration in the diluted probe wash from Step 10.3.1 in μg/mL,
    5 is the dilution factor,
    250 is the volume of the probe wash in mL, and
    80 is the ionic weight of sulfite ion.

11.3.3 Calculate the μmoles of sulfate ion in the probe wash given by:

$$W = X(5)(250) / (96)$$

where W is the μmoles of sulfate ion in the probe wash,
    X is the sulfate-ion concentration in the diluted probe wash from Step 10.3.1 in μg/mL,
    5 is the dilution factor,
    250 is the volume of the probe wash in mL, and
    96 is the ionic weight of sulfate ion.

11.3.4 Theoretically, sufficient μmoles of stack ammonium ion will exist in the probe wash to fix all of the sulfite ion in the probe wash, so calculate the weight of stack $(NH_4)_2SO_3$ in the probe wash given by:

$$Y = Z(116)$$

where Y is the weight of stack $(NH_4)_2SO_3$ in the probe wash in μg,
   Z is the μmoles of sulfite ion in the probe wash from Step 11.3.2, and
   116 is the molecular weight of $(NH_4)_2SO_3$.

11.3.5 Calculate the μmoles of stack ammonium ion remaining in the probe wash given by:

$$a = S - 2U$$

where a is the μmoles of stack ammonium ion remaining in the probe wash,
   S is the μmoles of stack ammonium ion in the probe wash from Step 11.3.1, and
   2U is the μmoles of stack ammonium ion already used to form $(NH_4)_2SO_3$ in the probe wash (U is from Step 11.3.2).

11.3.6 Calculate the weight of stack $(NH_4)_2SO_4$ in the probe wash given by:

$$b = 0.5a(132)$$

where b is the weight of stack $(NH_4)_2SO_4$ in the probe wash in μg,
   a is the μmoles of stack ammonium ion remaining in the probe wash (from Step 11.3.5) that can react with 0.5a μmoles of sulfate ion in the probe wash, and
   132 is the molecular weight of $(NH_4)_2SO_4$.

11.3.7 Calculate the weight of free H_2SO_4 in the probe wash given by:

$$c = (W - 0.5a)(98)$$

where c is the weight of free H_2SO_4 in the probe wash in μg,
   W is the μmoles of sulfate ion in the probe wash from Step 11.3.3,
   0.5a is the μmoles of stack $(NH_4)_2SO_4$ from Step 11.3.6, and
   98 is the molecular weight of H_2SO_4.

11.3.8 Calculate the weight of particulate (including stack $(NH_4)_2SO_3$ and stack $(NH_4)_2SO_4$) in the probe wash given by:

$$d = e - f + 0.04(Y + b) - 0.96c(1.35)$$

where d is the weight of particulate (including stack $(NH_4)_2SO_3$ and stack $(NH_4)_2SO_4$) in the probe wash in μg,
   e is the final weight of the probe-wash beaker from Step 10.3.2 in μg,
   f is the tared weight of the probe-wash beaker in μg,

0.04 is the fraction of the stack $(NH_4)_2SO_3$ and stack
$(NH_4)_2SO_4$ removed from the probe wash for analysis,
Y is the weight of stack $(NH_4)_2SO_3$ from Step 11.3.4 in μg,
b is the weight of stack $(NH_4)_2SO_4$ from Step 11.3.6 in μg,
0.96 is the fraction of the free H_2SO_4 remaining in the probe
wash after removing some of the probe wash for analyses,
c is the weight of free H_2SO_4 in the probe wash from Step
11.3.7 in μg, and
1.35 is the ratio of the molecular weight of $(NH_4)_2SO_4$ to the
molecular weight of H_2SO_4.

11.4   IPA absorber.

11.4.1   Calculate the μmoles of stack ammonium ion in the IPA absorber given
by:

$$h = i(5)(250) / (18)$$

where h is the μmoles of stack ammonium ion in the IPA absorber,
        i is the stack ammonium-ion concentration in the diluted IPA
        absorber from Step 10.4.1 in μg/mL,
        5 is the dilution factor,
        250 is the volume of the IPA absorber in mL, and
        18 is the ionic weight of ammonium ion.

11.4.2   Calculate the μmoles of sulfite ion in the IPA absorber given by:

$$j = k(5)(250) / (80)$$

where j is the μmoles of sulfite ion in the IPA absorber,
        k is the sulfite-ion concentration in the diluted IPA absorber from
        Step 10.4.1 in μg/mL,
        5 is the dilution factor,
        250 is the volume of the IPA absorber in mL, and
        80 is the ionic weight of sulfite ion.

11.4.3   Calculate the μmoles of sulfate ion in the IPA absorber given by:

$$m = n(5)(250) / (96)$$

where m is the μmoles of sulfate ion in the IPA absorber,
        n is the sulfate-ion concentration in the diluted IPA absorber from
        Step 10.4.1 in μg/mL,
        5 is the dilution factor,
        250 is the volume of the IPA absorber in mL, and
        96 is the ionic weight of sulfate ion.

11.4.4   Theoretically, sufficient µmoles of stack ammonium ion will exist in the IPA absorber to fix all of the sulfite ion in the IPA absorber, so calculate the weight of stack $(NH_4)_2SO_3$ in the IPA absorber given by:

$p = q(116)$

where p is the weight of stack $(NH_4)_2SO_3$ in the IPA absorber in µg,
  q is the µmoles of sulfite ion in the IPA absorber from Step 11.4.2, and
  116 is the molecular weight of $(NH_4)_2SO_3$.

11.4.5   Calculate the µmoles of stack ammonium ion remaining in the IPA absorber given by:

$r = h - 2j$

where r is the µmoles of stack ammonium ion remaining in the IPA absorber,
  h is the µmoles of stack ammonium ion in the IPA absorber from Step 11.4.1, and
  2j is the µmoles of stack ammonium ion already used to form $(NH_4)_2SO_3$ in the IPA absorber (j is from Step 11.4.2).

11.4.6   Compare the µmoles of stack ammonium ion remaining in the IPA absorber (r from Step 11.4.5) with the µmoles of stack sulfate ion in the IPA absorber (m from Step 11.4.3). If r is greater than 2m, see Step 1.3.6. Otherwise, continue with Step 11.4.7.

11.4.7   Calculate the weight of stack $(NH_4)_2SO_4$ in the IPA absorber given by:

$s = 0.5r(132)$

where s is the weight of stack $(NH_4)_2SO_4$ in the IPA absorber in µg,
  r is the µmoles of stack ammonium ion remaining in the IPA absorber (from Step 11.4.5) that can react with 0.5r µmoles of sulfate ion in the IPA absorber, and
  132 is the molecular weight of $(NH_4)_2SO_4$.

11.4.8   Calculate the weight of free H_2SO_4 in the IPA absorber given by:

$t = (m - 0.5r)(98)$

where t is the weight of free H_2SO_4 in the IPA absorber in µg,
  m is the µmoles of sulfate ion in the IPA absorber from Step 11.4.3,
  0.5r is the µmoles of stack $(NH_4)_2SO_4$ from Step 11.4.7, and
  98 is the molecular weight of H_2SO_4.

11.4.9 Calculate the weight of particulate (including stack $(NH_4)_2SO_3$ and stack $(NH_4)_2SO_4$) in the IPA absorber given by:

$$u = v - w + 0.04(p + s) - 0.96t(1.35)$$

where u is the weight of particulate (including stack $(NH_4)_2SO_3$ and
stack $(NH_4)_2SO_4$) in the IPA absorber in µg,
v is the final weight of the IPA-absorber beaker from Step 10.4.2
in µg,
w is the tared weight of the IPA-absorber beaker in µg,
0.04 is the fraction of the stack $(NH_4)_2SO_3$ and stack
$(NH_4)_2SO_4$ removed from the IPA absorber for analysis,
p is the weight of stack $(NH_4)_2SO_3$ from Step 11.4.4 in µg,
s is the weight of stack $(NH_4)_2SO_4$ from Step 11.4.7 in µg,
0.96 is the fraction of the free H_2SO_4 remaining in the IPA
absorber after removing some of the IPA absorber for analyses,
t is the weight of free H_2SO_4 in the IPA absorber from Step
11.4.8 in µg, and
1.35 is the ratio of the molecular weight of $(NH_4)_2SO_4$ to the
molecular weight of H_2SO_4.

11.5 Total weights

11.5.1 Calculate and report the weight for total stack $(NH_4)_2SO_3$ given by:

$$x = J + Y + p$$

where x is the weight for total stack $(NH_4)_2SO_3$ in µg,
J is the weight for stack $(NH_4)_2SO_3$ in the filter extract from Step
11.2.4 in µg,
Y is the weight for stack $(NH_4)_2SO_3$ in the probe wash from Step
11.3.4 in µg, and
p is the weight for stack $(NH_4)_2SO_3$ in the IPA absorber from
Step 11.4.4 in µg.

11.5.2 Calculate and report the weight for total stack $(NH_4)_2SO_4$ given by:

$$y = L + b + s$$

where y is the weight for total stack $(NH_4)_2SO_4$ in µg,
L is the weight for stack $(NH_4)_2SO_4$ in the filter extract from Step
11.2.6 in µg,
b is the weight for stack $(NH_4)_2SO_4$ in the probe wash from Step
11.3.6 in µg, and
s is the weight for stack $(NH_4)_2SO_4$ in the IPA absorber from
Step 11.4.7 in µg.

11.5.3   Calculate and report the weight for total free H_2SO_4 in the stack given by:

$z = M + c + t$

where z is the weight for total free H_2SO_4 in the stack in μg,
 M is the weight for free H_2SO_4 in the filter extract from Step 11.2.7 in μg,
 c Is the weight for free H_2SO_4 in the probe wash from Step 11.3.7 in μg, and
 t is the weight for free H_2SO_4 in the IPA absorber from Step 11.4.8 in μg.

11.5.4   Calculate and report the weight for total stack particulate (including $(NH_4)_2SO_3$ and $(NH_4)_2SO_4$) given by:

$A' = N + d + u$

where A' is the weight for total stack particulate in μg,
 N is the weight for stack particulate in the filter extract from Step 11.2.8 in μg,
 d is the weight for stack particulate in the probe wash from Step 11.3.8 in μg, and
 u is the weight for stack particulate in the IPA absorber from Step 11.4.9 in μg.

12.0   QUALITY ASSURANCE.

12.1   Follow the quality assurance procedures for the analyses of ammonium, sulfite, and sulfate ions by IC as outlined in the TACB's Laboratory Methods Manual, Methods 26b and 26a (Reference 5).

12.2   Follow the quality assurance procedures for filters and beakers as outlined in the TACB's Laboratory Methods Manual, Method 23 (Reference 5).

12.3   Prior to sampling, evaporate 200 mL of the DDI water (or acetone) probe wash and 200 mL of the IPA from the batches that will be used for stack sampling.

12.3.1   Evaporate each sample to dryness in clean, tared beakers following the procedures outlined in Step 8.4 in the TACB's Laboratory Methods Manual, Method 23 (Reference 5) for the preparation of clean, tared beakers.

12.3.2 The samples must not be contaminated by more than 0.001% by weight:

Distilled water--not more than 10 mg particulate/liter,
Acetone--not more than 8 mg particulate/liter, and
IPA--not more than 8 mg particulate/liter.

12.3.3 If any sample is contaminated by more than its allowable limit, discard that sample, and test a new batch.

13.0   REFERENCES.

1. EPA Quality Assurance Handbook for Air Pollution Measurement Systems. Volume III - Stationary Source Specific Methods. Method 8. Revision No. 0, May 1, 1979.

2. EPA Quality Assurance Handbook for Air Pollution Measurement Systems. Volume III - Stationary Source Specific Methods. Method 5. Revision No. 0, January 15, 1980.

3. Texas Air Control Board, Sampling Procedures Manual, Chapter 5.

4. EPA Quality Assurance Handbook for Air Pollution Measurement Systems. Volume III - Stationary Source Specific Methods. Method 6. Revision No. 0, May 1, 1979.

5. Texas Air Control Board, Laboratory Methods Manual, Revised 1989. (Method 26b, Analysis for Cations by Ion Chromatography, is under development.)