

3.1.3 All sampling train glassware shall be cleaned prior to the test with soap and tap water, water, and rinsed using tap water, water, acetone, and finally, MeCl₂. It is important to completely remove all silicone grease from areas that will be exposed to the MeCl₂ during sample recovery.

3.2 Sample Recovery. Same as in Method 17, Section 2.2, with the following additions:

3.2.1 N₂ Purge Line. Inert tubing and fittings capable of delivering 0 to 28 liters/min of N₂ gas to the impinger train from a standard gas cylinder (see Figure 202-2). Standard 0.95 cm (3/8-inch) plastic tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve may be used.

3.2.2 Rotameter. Capable of measuring gas flow at 20 liters/min.

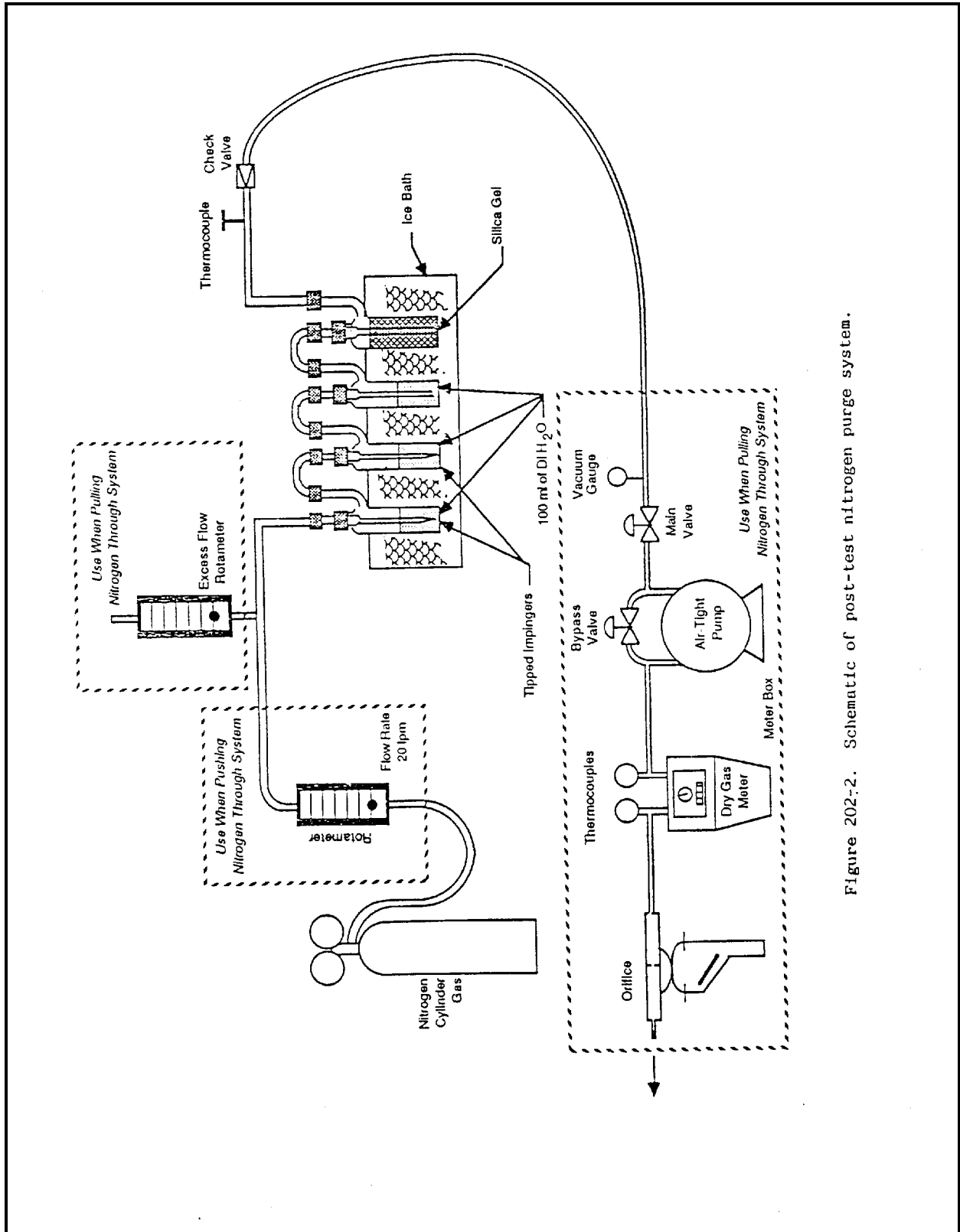


Figure 202-2. Schematic of post-test nitrogen purge system.

3.3 Analysis. The following equipment is necessary in addition to that listed in Method 17, Section 2.3:

- 3.3.1 Separatory Funnel. Glass, 1-liter.
3.3.2 Weighing Tins. 350-ml.
3.3.3 Drying Equipment. Hot plate and oven with temperature control.
3.3.4 Pipets. 5-ml.
3.3.5 Ion Chromatograph. Same as in Method 5F, Section 2.1.6.

4. REAGENTS

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

4.1 Sampling. Same as in Method 17, Section 3.1, with the addition of deionized distilled water to conform to the American Society for Testing and Materials Specification D 1193-74, Type II and the omission of Section 3.1.4.

4.2 Sample Recovery. Same as in Method 17, Section 3.2, with the following additions:

4.2.1 N2 Gas. Zero grade N2 gas at delivery pressures high enough to provide a flow of 20 liters/min for 1 hour through the sampling train.

4.2.2 Methylene Chloride, ACS grade. Blanks shall be run prior to use and only methylene chloride with low blank values (0.001 percent) shall be used.

4.2.3 Water. Same as in Section 4.1.

4.3 Analysis. Same as in Method 17, Section 3.3, with the following additions:

4.3.1 Methylene Chloride. Same as Section 4.2.2.

4.3.2 Ammonium Hydroxide. Concentrated (14.8 M) NH4OH.

4.3.3 Water. Same as in Section 4.1.

transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in Container No. 4 either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Remove a 5-ml aliquot and set aside for later ion chromatographic (IC) analysis of sulfates. (Note: Do not use this aliquot to determine chlorides since the HCl will be evaporated during the first drying step; Section 8.2 details a procedure for this analysis.)

5.3.2.1 Extraction. Separate the organic fraction of the sample by adding the contents of Container No. 5 (MeCl₂) to the contents of Container No. 4 in a 1000-ml separatory funnel. After mixing, allow the aqueous and organic phases to fully separate, and drain off most of the organic/MeCl₂ phase. Then add 75 ml of MeCl₂ to the funnel, mix well, and drain off the lower organic phase. Repeat with another 75 ml of MeCl₂. This extraction should yield about 250 ml of organic extract. Each time, leave a small amount of the organic/MeCl₂ phase in the separatory funnel ensuring that no water is collected in the organic phase. Place the organic extract in a tared 350-ml weighing tin.

5.3.2.2 Organic Fraction Weight Determination (Organic Phase from Container Nos. 4 and 5). Evaporate the organic extract at room temperature and pressure in a laboratory hood. Following evaporation, desiccate the organic fraction for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg.

5.3.2.3 Inorganic Fraction Weight Determination. [Note: If NH₄Cl is to be counted as CPM, the inorganic fraction should be taken to near dryness (less than 1 ml liquid) in the oven and then allow to air dry at ambient temperature. If multiple acid emissions are suspected, the ammonia titration procedure in Section 8.1 may be preferred.] Using a hot plate, or equivalent, evaporate the aqueous phase to approximately 50 ml; then, evaporate to dryness in a 105°C oven. Redissolve the residue in 100 ml of water. Add five drops of phenolphthalein to this solution; then, add concentrated (14.8 M) NH₄OH until the

sample turns pink. Any excess NH4OH will be evaporated during the drying step. Evaporate the sample to dryness in a 105°C oven, desiccate the sample for 24 hours, weigh to a constant weight, and record the results to the nearest 0.1 mg. (Note: The addition of NH4OH is recommended, but is optional when little or no SO2 is present in the gas stream, i.e., when the pH of the impinger solution is greater than 4.5, the addition of NH4OH is not necessary.)

5.3.2.4 Analysis of Sulfate by IC to Determine Ammonium Ion (NH4+) Retained in the Sample. (Note: If NH4OH is not added, omit this step.) Determine the amount of sulfate in the aliquot taken from Container No. 4 earlier as described in Method 5F (Appendix A, 40 CFR Part 60). Based on the IC SO4-2 analysis of the aliquot, calculate the correction factor to subtract the NH4+ retained in the sample and to add the combined water removed by the acid-base reaction (see Section 7.2).

5.3.3 Analysis of Water and MeCl2 Blanks (Container Nos. 6 and 7). Analyze these sample blanks as described above in Sections 5.3.2.3 and 5.3.2.2, respectively.

5.3.4 Analysis of Acetone Blank (Container No. 8). Same as in Method 17, Section 4.3.

6. CALIBRATION

Same as in Method 17, Section 5, except for the following:

6.1 IC Calibration. Same as Method 5F, Section 5.

6.2 Audit Procedure. Concurrently, analyze the audit sample and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for compliance samples and the EPA audit sample. If this condition is met, auditing of subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are

Where:

K = -0.0208, when correcting for NH4+ and H2O.
= 0.354, when only correcting for NH4+.

7.3 Mass of Inorganic CPM.

mi = mr * (V_ic / (V_ic - V_b)) - mc Eq. 202-2

7.4 Concentration of CPM.

C_cpm = (mo + mi - mb) / VM_std Eq. 202-3

8. ALTERNATIVE PROCEDURES

8.1 Determination of NH4+ Retained in Sample by Titration.

8.1.1 An alternative procedure to determine the amount of NH4+ added to the inorganic fraction by titration may be used. After dissolving the inorganic residue in 100 ml of water, titrate the solution with 0.1 N NH4OH to a pH of 7.0, as indicated by a pH meter. The 0.1 N NH4OH is made as follows: Add 7 ml of concentrated (14.8 M) NH4OH to 1 liter of water. Standardize against standardized 0.1 N H2SO4 and calculate the exact normality using a procedure parallel to that described in Section 5.5 of Method 6 (Appendix A, 40 CFR Part 60). Alternatively, purchase 0.1 N NH4OH that has been standardized against a National Institute of Standards and Technology reference material.

8.1.2 Calculate the concentration of SO4-2 in the sample using the following equation.

C_SO4 = (48.03 * Vt * N) / 100 Eq. 202-4

Where:

N = Normality of the NH4OH, mg/ml.
Vt = Volume of NH4OH titrant, ml.

Evaluation of Draft Protocol for Measurement of Condensible Particulate Emissions." Draft Report. November 17, 1989.

3. Texas Air Control Board, Laboratory Division. "Determination of Particulate in Stack Gases Containing Sulfuric Acid and/or Sulfur Dioxide." Laboratory Methods for Determination of Air Pollutants. Modified December 3, 1976.

4. Nothstein, Greg. Masters Thesis. University of Washington. Department of Environmental Health. Seattle, Washington.

5. "Particulate Source Test Procedures Adopted by Puget Sound Air Pollution Control Agency Board of Directors." Puget Sound Air Pollution Control Agency, Engineering Division. Seattle, Washington. August 11, 1983.

6. Commonwealth of Pennsylvania, Department of Environmental Resources. Chapter 139, Sampling and Testing (Title 25, Rules and Regulations, Part I, Department of Environmental Resources, Subpart C, Protection of Natural Resources, Article III, Air Resources). January 8, 1960.

7. Wisconsin Department of Natural Resources. Air Management Operations Handbook, Revision 3. January 11, 1988.

Moisture Determination

Volume or weight of liquid in impingers _____ ml or g
Weight of moisture in silica gel _____ g

Sample Preparation (Container No. 4)

Amount of liquid lost during transport _____ ml
Final volume _____ ml
pH of sample prior to analysis _____
Addition of NH4OH required? _____
Sample extracted 2X with 75 ml MeCl2? _____

For Titration of Sulfate

Normality of NH4OH _____ N
Volume of sample titrated _____ ml
Volume of titrant _____ ml

Sample Analysis

Table with columns: Container number, Weight of Condensable Particulate, mg (Final Weight, Tare Weight, Weight Gain), Total, Less Blank, Weight of Condensable Particulate.

Figure 202-3. Analytical data sheet