Office of Surface Mining, Reclamation and Enforcement, Room 5315, 1100 "L" Street, NW., Washington, D.C. 20240, Telephone: (202) 343–4728

West Virginia Department of Natural Resources, Building 3, Room 630, 1800 Washington Street, East, Charleston, West Virginia 25305, Telephone: (304) 348–9160

FOR FURTHER INFORMATION CONTACT:

Christine M. Struminski, Assistant Regional Director, Division of State and Federal Programs, 603 Morris Street, Charleston, West Virginia 25301, Telephone: (304) 342–8125.

SUPPLEMENTARY INFORMATION:

On April 29, 1981, West Virginia provided a copy of proposed coal refuse disposal regulations to OSM for review (Administrative Record No. WV–400). On June 8, 1981, OSM provided an informal listing of deficiencies found in the proposed regulations (Administrative Record No. WV–401a) and informed the State that the promulgated regulations must be submitted as a formal program amendment which would be subject to public comment.

The regulations were promulgated on October 1, 1981, and submitted as a program amendment on October 29, 1981. On December 21, 1981, notice of opportunity for public hearing on the proposed modifications to the West Virginia program, was published in the **Federal Register** (46 FR 61897). The notice stated that any person interested in making an oral or written presentation at the hearing should contact Ms. Struminski by January 4, 1982, and that if no person contacted Ms. Struminski to express an interest in participating in the hearing by the above date, the hearing would be cancelled.

Because no one expressed an interest in attending the hearing by January 4, 1982, the hearing has been cancelled.

While there is no public hearing, interested persons may still submit written comments on the proposed program elements. Written comments must be received on or before 4:00 p.m., on January 20, 1982, to be considered in the Director's decision on whether the proposed amendments are acceptable.

Dated: January 12, 1982.

J. S. Griles,

Acting Director, Office of Surface Mining.

[FR Doc. 82–1186 Filed 1–14–82; 8:45 am]

BILLING CODE 4310–05–M

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 50

[ORD–FRL–1962–3]

National Ambient Air Quality Measurement Methodology; Proposed Minor Amendments

AGENCY: Environmental Protection Agency (EPA).

ACTION: Notice of proposed rulemaking.

SUMMARY: As described in this notice, the U.S. Environmental Protection Agency (EPA) is proposing to revise Appendixes A, B, and C to 40 CFR Part 50. Appendixes A and B set forth the respective reference methods for measuring sulfur dioxide and total suspended particulates in the atmosphere. Appendix C describes the measurement principle and calibration procurement applicable to reference methods for measuring carbon monoxide in the atmosphere. The revisions are needed to clarify certain provisions of these appendixes, to correct certain identified shortcomings, and to incorporate technical improvements developed subsequent to their 1971 promulgation. Although the proposed text of the revised appendixes is substantially rewritten and reorganized in some cases, the revisions are considered minor from a technical aspect. In particular, technical changes have been specifically limited to those that EPA believes will cause no loss of continuity or comparability between ambient measurements made with the existing methods and those made in accordance with the proposed revised methods.

DATE: Comments should be received no later than February 16, 1982.

ADDRESS: Comments (in duplicate, if possible) should be sent to Public Docket No. A–81–34, U.S. Environmental Protection Agency, Central Docket Section (A–130), West Tower Lobby, Gallery 1, 401 M Street, SW., Washington, D.C. 20460. This docket may be inspected at this address between the hours of 8:00 a.m. and 4:00 p.m. Monday through Friday. A reasonable fee may be charged for copying services.

FOR FURTHER INFORMATION CONTACT:

Larry J. Purdue, Chief, Methods Standardization Branch (CM–77), Quality Assurance Division, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711 (919–541–2665).

SUPPLEMENTARY INFORMATION: When the National Ambient Air Quality Standards (NAAQS) prescribed in 40 CFR Part 50 were promulgated in 1971, an appendix to Part 50 was provided for each standard to describe a reference method for measuring that pollutant. Since then, several of these appendixes (C, D, and F) have undergone major revisions to (1) establish the "measurement principle and calibration procedure" concept for automated reference methods, (2) supersede an existing reference method with a new measurement principle and calibration procedure, or (3) replace a calibration procedure with a substantially better procedure. Also since 1971, it has become apparent that several other appendixes should be changed—but only to a minor extent—to incorporate clarifications and relatively minor technical improvements in the current methodology. Accordingly, such changes are hereby proposed.

Minor revisions are proposed to three appendixes: Appendix A, "Reference Method for the Determination of Sulfur Dioxide in the Atmosphere (Pararosaniline Method)"; Appendix B, "Reference Method for the Determination of Suspended Particulates in the Atmosphere (High-Volume Method)"; and Appendix C, "Measurement Principle and Calibration Procedure for the Continuous Measurement of Carbon Monoxide in the Atmosphere (Non-Dispersive Infrared Spectrometry)". The specific nature, rationale, and technological effect of the revisions for each appendix are subsequently described in detail. In some cases, the text of the appendix is substantially rewritten and somewhat reorganized. However, in all cases, the technical changes are limited to those that will not jeopardize the continuity or comparability of ambient measurements made according to the proposed revised method with those made previously according to the existing method. For Appendix C, the measurement principle and calibration procedure are simply clarified, and no technical changes are proposed.

Comments

The method revisions, particularly those in Appendix B (High-Volume Method) have been extensively reviewed by experts in monitoring methodology, both within and outside of EPA, and their comments have been incorporated into the proposed revisions to the extent possible. During the current comment period, all interested persons are invited to submit written comments on the proposed revisions set forth here.

All such comments received by EPA during the comment period will be available for inspection during normal business hours at the address indicated previously. After consideration of the comments received, the proposed revisions will be modified as appropriate and will become effective upon final promulgation in the **Federal Register.**

Appendix A.—Reference Method For Sulfur Dioxide

Background

On April 30, 1971, the Environmental Protection Agency (EPA) promulgated (36 FR 8187) a Federal Reference Method (FRM) for the determination of sulfur dioxide (SO_2) in the atmosphere. The FRM was presented in Appendix A of 40 CFR Part 50 (National Ambient Air Quality Standards) and prescribed detailed procedures for sample collection and analysis. Measurements of SO_2 with the FRM are based on collection in potassium tetrachloromercurate (TCM) solution and subsequent spectrophotometric analysis after the addition of formaldehyde and pararosaniline. During sample collection, SO_2 is stabilized as the monochlorosulfanatomercurate complex (TCM–SO_2), which resists oxidation by oxygen in the air.

Prior to the promulgation of the FRM in 1971, research (*1*), (*2*), (*3*), (*4*), (*5*), on pararosaniline methods very similar to the FRM indicated that SO_2 samples collected in TCM were subject to a temperature-dependent decay. Most of these earlier studies focused on the stability of collected samples when stored at room temperature (20° to 25° C). Results by the various investigators were generally in good agreement and indicated that TCM–SO_2 samples decay at a rate of about 1 percent per day. One of the investigators (*3*) observed significant SO_2 losses (decay) when samples were stored at 40° C and noted that such temperatures could occur at some sites during strong solar radiation in summer or overheating in winter.

Nevertheless, following promulgation of the FRM in 1971, it became common practice during typical field use to house the sampling equipment in a thermostated sampling unit at 32° C and, after sample collection, to transport the 24-hour samples back to the analytical laboratory in containers that generally had no means of controlling the temperature. Often, collected samples remained in the sampling unit several days before being transported back to the laboratory. Collected samples were sometimes transported to the laboratory by the field operator, but were often sent through the mail. Once received by the analytical laboratory, samples might be stored for several days or even weeks at either room temperature or in a refrigerator. Thus, prior to analysis, samples were exposed to a variety of temperatures for various lengths of time. Temperature exposures were often extreme, especially during the summer months at sites with relatively little protection from the elements (e.g., rooftop locations).

Subsequent to the promulgation of the FRM, additional studies were conducted to determine the effects of temperature and other parameters on the FRM. In a study by the Texas Air Control Board (*6*), the decay rate of TCM–SO_2 samples at temperatures ranging from −15° to 43° C was investigated. The observed decay per day for samples corresponding to the 30-minute ambient SO_2 concentrations of 0.1 and 0.3 ppm was approximately 1 to 1.5 percent at −15° C and 13° C, 2 to 2.5 percent at 30° C, and 14 to 32 percent at 43° C. In another study, the Illinois EPA (*7*) monitored the internal temperature of a typical SO_2 sampler unit under varying ambient air temperature and operating conditions. The internal temperature averaged about 35° C and frequently exceeded 45° C over 10 test days during which the ambient temperature varied from 4.4° to 34.4° C (40° to 94° F). The decay rates of TCM–SO_2 samples corresponding to 24-hour ambient SO_2 concentrations from 33 to 260 $\mu g\ SO_2/m^3$ (0.013 to 0.1 ppm) were examined at temperatures ranging from 10° to 45° C (50° to 112°). Significant losses of SO_2 were observed at temperatures above 27° C (80° F). The observed decay rates were about 10 percent per day at 35° C (95° F) and 40 to 50 percent per day at 45° C (112° F).

EPA Temperature Effect Study

As a result of these reported temperature-related problems with the FRM and other similar reports, EPA undertook an investigation (*8*) to determine the effect of temperature on the stability of collected TCM–SO_2 samples. Simulated field samples representing 24-hour SO_2 concentrations from 35 to 278 $\mu g\ SO_2/m^3$ (0.013 to 0.106 ppm) were exposed to temperatures ranging from 20° C to 50° C. At a given exposure temperature, the rate of decay was found to be independent of SO_2 concentration. The equation best fitting the data was described by an exponential curve of the form:

$$C = C_o e - kt$$

where

C = concentration measured at time = t, $\mu g\ SO_2/mL$

C_o = concentration measured at t = O, $\mu g\ SO_2/mL$

k = rate of decay, day^{-1}

t = time, day

The average decay rates for each exposure temperature are given in Table 1 and indicate about a fivefold increase in decay rate for each 10° C rise in temperature.

TABLE 1.—EFFECT OF TEMPERATURE ON PERCENT DECAY PER DAY

Temperature, °C	Average rate of decay (k)	Percent loss per day
20	0.009 day^{-1}	0.9
30	0.051 day^{-1}	5.0
40	0.287 day^{-1}	25.0
50	1.33 day^{-1}	73.6

Based on the decay data, an equation was derived to relate decay rate to temperature. The equation

$$\ln k = 48.735 - 15661\ (\tfrac{1}{T}),$$

where T = temperature in K° can be used to calculate the rate of decay at any temperature within the range of 20° to 50° C and to estimate the rate of decay outside this range. The calculated rate of decay at 22° C is 1.3 percent per day, which is in good agreement with the value of 1 percent per day reported in the earlier literature.

Using the decay data, Table 2 was constructed showing the percent of SO_2 remaining in the TCM absorbing solution after exposure to various temperatures. The table shows that sample collection at 25° C results in only a 1.1-percent loss in SO_2 during the 24-hour sampling period, but that further exposure of the collected sample for 4 days at this temperature leads to a 10-percent loss in SO_2.

TABLE 2.—EFFECTS OF TIME AND TEMPERATURE ON COLLECTED SO_2-TCM SAMPLES

°C	°F	Percent SO_2 remaining							
		At end of sampling	Day						
			1	2	3	4	5	6	7
5	41	99.9	99.8	99.9	99.8	99.7	99.7	99.6	99.6
10	50	99.9	99.8	99.7	99.6	99.5	99.4	93.5	99.2
15	59	99.8	99.4	99.0	98.6	98.2	97.8	97.4	97.0
20	68	99.6	98.7	97.8	96.9	96.1	95.2	94.3	93.5
25	77	98.9	96.7	94.4	92.2	90.2	88.1	86.1	84.2

TABLE 2.—EFFECTS OF TIME AND TEMPERATURE ON COLLECTED SO_2-TCM SAMPLES—Continued

°C	°F	Percent SO_2 remaining							
		At end of sampling	Day						
			1	2	3	4	5	6	7
30	86	97.4	92.2	87.4	82.8	78.5	74.3	70.4	66.7
35	95	95.1	84.0	74.1	65.5	57.9	51.3	45.2	39.9
40	104	87.6	66.8	50.8	38.7	29.5	22.5	17.2	13.0
45	113	75.3	41.4	22.7	12.5	6.9	3.8	1.9	1.1
50	122	56.3	15.6	4.3	1.2	0.9	0.1	0	0

EPA Bubbler Temperature Study

As a result of the EPA temperature effect study, EPA conducted an investigation (*9*) to (1) characterize the temperatures that TCM absorbing solutions might be exposed to before, during, and after routine ambient air sampling, and (2) evaluate techniques for controlling absorbing solution temperatures during routine use of the method. Two commercially available sampling shelters were utilized in this study. The Indoor Five-Gas Sampler® consists of a thermostated compartment in which the five-gas sampling system is housed and a separate external sampling pump. The All-Weather Five-Gas Sampler® consists of a shelter that houses a temperature-controlled five-gas sampling system in one section and a sampling pump in a separate ventilated section. Both samplers were equipped with a standard heater controlled at 32° C.

Initially, tests were conducted with the two samplers in an environmental chamber capable of maintaining the desired test temperature within ±0.5° C over the range of 0° to 50° C. The tests showed that at chamber temperatures above 20° C, the temperatures inside the samplers were elevated enough to cause significant decay in collected TCM–SO_2 samples.

Additional tests were conducted under actual sampling conditions using the All-Weather sampler equipped with a thermostated heater. Ambient temperature varied from about 5° to 20° C during the tests, while the temperature inside the sampler varied from about 10° to 30° C. The test results were somewhat inconclusive (poor correlation between ambient temperature and sampler temperature) and suggested that the temperature inside the sampler may be affected by other external meteorological conditions such as windspeed and wind direction.

A final test was conducted to determine the temperature of the TCM absorbing solution in the Indoor sampler during sampling under various chamber temperature conditions. The results indicated that decay of SO_2 could be a problem when the sampler is operated with a 32° C controlled heater and when the ambient temperature exceeds 8° C.

The remainder of this study focused on investigating various measures to control the TCM absorbing solution temperature during and after sampling. One of the approaches evaluated was the use of a commercially available, 1.5–ft^3 (42–L) refrigerator to house the SO_2 absorber. Test results showed that the TCM solution temperature could be maintained at 12° ±5° C at ambient temperatures of 25° to 50° C. To prevent solutions from freezing at ambient temperatures below 20° C, a small heater strip was installed in the refrigerator to keep the temperature above 7° C. Condensation in sample inlet lines and in the absorber was a problem when the relative humidity of the sample stream was high. Wrapping the sample inlet line with standard water pipe insulation or a heater tape minimized or eliminated the condensation.

Another approach investigated was the use of a thermoelectrically controlled chamber to house the SO_2 absorber. Three prototype thermoelectric coolers were evaluated and found to be capable of maintaining the TCM solution temperature 12° ±5° C over an ambient temperature range or 0° to 50° C. Two of these devices were designed to be incorporated into the existing Indoor or All-Weather samplers. Minor modifications to each of the samplers were required to provide proper ventilation for the thermoelectric cooler. The third device was a prototype three-gas sampler with cooling capability for one of the three absorbers.

The use of styrofoam containers equipped with a eutectic mixture for cooling was evaluated as a means of shipping collected samples. The temperature of a TCM solution was maintained below 21° C for up to 50 hours at ambient temperatures up to 50° C. In a test in which exposure temperatures were varied from 25° to 40° C for varying periods of time (simulated transit conditions), TCM solution temperature was maintained below 21° C for as long as 62 hours.

Other Reference Method Deficiencies

In addition to the temperature-dependent sample decay problem, several other shortcomings that affect the precision and accuracy of SO_2 measurements obtained with the existing FRM have been identified.

The use of a needle valve/flowmeter combination, currently prescribed as one of two flow control techniques, has been found to be generally unreliable at low flow rates over a 24-hour sampling period. Furthermore, reliance on a single sample flow measurement (at the initiation of sampling) is also recognized as inadequate. Calibration of flow control devices under conditions (temperature and pressure external to the sampling train) different from those encountered during routine sampling can often introduce errors in the air sample volume measurements. The flow measurement procedure as currently prescribed is not sufficiently explicit. No specifications are given regarding the constancy of the flow rate over the sampling period. Quality control measures to ensure acceptable precision and accuracy in the overall measurement process are generally lacking throughout the existing method description.

Although not technically deficient, the currently prescribed dynamic calibration procedure is impractical when applied to the 24-hour procedure. With the current procedure, preparation of calibration standards requires 6 days unless multiple concentrations are generated and sampled simultaneously.

Proposed Revisions

Many of the reference method shortcomings discussed above can be corrected or their effects minimized by:

- The use of adequate temperature control during sample collection, shipment, and storage.
- The use of more reliable flow control and flow measurement techniques during sample collection.
- The incorporation of more explicit specifications, instructions, and quality control measures throughout the method.

In developing the FRM revisions, the comparability between SO_2 measurements obtained with the original and revised versions of the method is an important consideration. Ambient SO_2 measurements obtained with the original FRM under conditions of extreme temperature exposures are highly suspect because of the demonstrated temperature-dependent decay problem. The data base, from promulgation of the FRM in 1971 to early

1976 is, no doubt, biased low. In December 1975 (*10*) the EPA regional offices were apprised of the temperature sensitivity of the SO_2 reference method. At that time, EPA recommended that agencies making SO_2 measurements with the FRM carry out the method in such a manner that TCM absorbing solution temperatures be maintained at 25° C or less during sampling and that the temperature of collected samples be maintained at 20° C or less until analysis. Since that time, most agencies that use the FRM have incorporated temperature control measures in their procedures. Thus, the data base from early 1976 to the present is much less likely to be affected. Furthermore, the comparability between future data and data from 1976 to the present is not expected to change, since the proposed revisions merely formalize the recommendations and guidance given in 1975.

The economic impact on monitoring agencies was also considered and every attempt was made to keep the cost of implementing the proposed changes in the revised method as low as possible. With the exception of one change, the costs should be minimal. Much of the additional equipment required in the revised method is available in most analytical labortories. An exception is the cooler for controlling the absorbing solution temperature during and after sampling (until shipment to the analytical laboratory). Cooler costs range from approximately $150 for a small refrigerator to $600 for a thermoelectric cooler specifically designed for SO_2 sampling.

Section-by-Section Changes

The numbers given below correspond to similarly numbered sections in the revised method.

1.0 *Applicability.*

1.1 Since the principle and applicability of the method are not necessarily related, these descriptive elements have been separated into individual sections. (This, of course, changes the section numbering.) The applicability section is clarified to specifically reference monitoring for compliance with the NAAQS for SO_2. The revised method includes the formal specifications and procedures, with additional quality assurance techniques and other guidance described in other documents, principally 40 CFR Part 58 and the Quality Assurance Handbook.

2.0 *Principle.*

2.1 This section has been expanded and reworded to describe the principle more explicitly and to clarify that the reported measurements are expressed as micrograms per cubic meter of air corrected to EPA reference conditions (25° C, 760 mmHg).

3.0 *Range.*

3.1 This section has been reworded to specify the range limits of the analytical procedure and gives the corresponding ambient SO_2 concentrations when the prescribed short-term and long-term sampling procedures are used.

4.0 *Interferences.*

4.1 This section has been reworded and expanded to include a statement regarding interference by ammonia.

5.0 *Precision and Accuracy.*

5.1 These two descriptive elements have been separated from the discussion of the stability of the TCM–SO_2 complex. This section gives the precision of the analytical procedure.

5.2 This new section has been added to give estimates of the precision (repeatability and reproducibility) of the 24-hour procedure based on collaborative test data. A statement about the accuracy (bias) of the method based on these data has also been added.

6.0 *Stability.*

6.1 The discussion of the stability of the collected TCM–SO_2 complex has been placed in a separate section and the section has been expanded to include a statement regarding retention of the complex during sampling.

7.0 *Apparatus.*

7.1 *Sampling.* This section has been expanded to include descriptions and/or specifications for all equipment and supplies required for both short-term and long-term sampling. Thermoelectric coolers, small modified refrigerators, or other means of controlling temperature are now required to maintain the temperature of the TCM absorbing solution at 15° ± C during sampling.

7.2 *Shipping.* This new section has been added to include a requirement for the use of a shipping container that can maintain the temperature of collected TCM–SO_2 samples at 5° ±5° C during shipment to the analytical laboratory.

7.3 *Analysis.* This section has been expanded to include more explicit requirements for spectrophotometer wavelength calibration and cell matching. A temperature control device is also now required during the color development step of the analytical procedure. The device must be capable of maintaining solution temperatures to ±1° C in the range of 20° to 30° C. A waste receptacle is required for the storage of spent TCM solutions.

8.0 *Reagents.*

8.1 *Sampling.* This section has been expanded to include a procedure for testing the purity of the distilled water used in the preparation of reagents and in the analytical procedure.

8.2 *Analysis.* This section has been expanded to include instructions for the preparation of all reagents used in the calibration and analytical procedures. Procedures for purification and assay of the pararosaniline dye have also been included in the revision.

9.0 *Sampling Procedure.*

9.1 *General Considerations.* The step-by-step procedures for sample collection, analytical calibration, and sample preparation and analysis have been separated into individual sections and have been ordered in the sequence that they would be carried out during routine use of the method. This first section contains general guidance for sampling when the prescribed sampling procedures are not appropriate to meet the special needs of the method user.

9.2 *30-Minute and 1-Hour Sampling.* This section has been expanded to include more explicit instructions for short-term sampling.

9.3 *24-Hour Sampling.* This section has been expanded to include more explicit instructions for long-term sampling. During the sampling period, the absorbing solution temperature must be controlled to 15° ±10° C.

9.4 *Flow Measurement.* This new section has been added to give more explicit instructions for the measurement of sample flow rate. All flow controllers must be calibriated in the sampling train with absorber in solution in place. Sample flow measurements must be obtained both prior to and following the sampling period and the sample must be invalidated if the difference between the intial and final flow rates exceeds 5 percent.

9.5 *Sample Storage and Shipment.* This new section has been added to give instructions regarding sample storage and shipment. After sample collection, a mark is placed on the absorber or shipping bottle indicating the volume of solution remaining. This mark is used later to verify that the solution volume has not changed during shipment of the sample to the analytical laboratory. The sample must be shipped and/or stored at 5° ±5 C unless it is analyzed within 8 hours of the end of the sampling period.

10.0 *Analytical Calibration.*

10.1 *Spectrophotometer Cell Matching.* This new section has been added to incorporate a procedure for spectrophotometer cell matching and determination of corrected absorbance.

10.2 *Static Calibration Procedure (Option 1).* This section has been reworded.

10.3 *Dynamic Calibration Procedures (Option 2).* This section has been expanded to incorporate dynamic calibration procedures applicable to short-term and long-term sampling procedures. The short-term procedure is carried out using sampling conditions identical to those used during field sampling. The long-term procedure is carried out using a high concentration of SO_2 and varying collection times.

11.0 *Sample Preparation and Analysis.*

11.1 *Sample Preparation.* This section has been expanded to include more explicit instructions for sample preparation. Solution temperature and volume are verified and the volume is adjusted as required. The sample must be invalidated if the sample volume differs by more than 10 mL from the original volume.

11.2 *Sample Analysis.* This section has been expanded to include more explicit instructions for sample analysis. The color development step must now be carried out within ±1° C of that temperature used during the analytical calibration. Two control standards must be prepared and analyzed with each batch of field samples.

11.3 *Absorbance Range.* This section has been reworded and expanded to include a recommendation that samples be reanalyzed using a smaller aliquot when dilution ratios greater than 1.1 are required to obtain absorbance readings below 1.0 absorbance units.

11.4 *Reagent Disposal.* This new section has been added and requires that spent reagents containing mercury be stored and disposed of using one of the procedures in Section 13.0.

12.0 *Calculations.*

12.1 *Calibration Slope, Intercept, and Correlation Coefficient.* This new section has been added to give instructions and a data form for calculating the slope, intercept, and correlation coefficient for the analytical calibration curve.

12.2 *Total Sample Volume.* This section has been revised to give an equation for calculating the air sample volume from the initial and final standard flow rates and the sampling time.

12.3 *Sulfur Dioxide Concentration.* This section has been revised to be consistent with the revised calibration procedures and other changes in the method.

12.4 *Control Standards.* This new section has been added to allow for the calculation of the amount of SO_2 in the control standards. The difference between the true and analyzed values of the control standards must not be greater than 5 percent.

12.5 *Conversion of $\mu g/m^3$ to ppm.* This section has not been revised.

13.0 *Disposal of Mercury-Containing Solutions.* This new section describes two procedures for disposing of spent mercury-containing solutions. One procedure is based on the formation of an amalgam with zinc or magnesium and the other uses aluminum foil strips to convert the mercury to its elemental form.

14.0 *References.*

New references have been added as required.

Appendix B.—Reference Method for Total Suspended Particulate Matter

Background

Since its promulgation in 1971 (36 FR 8187), the Federal Reference Method (high-volume method) for total suspended particulates (TSP) has presented a number of significant problems to method users and sampler manufacturers. Specifications and tolerances are insufficiently explicit, the calibration procedure is unclear, pressure and temperature corrections contain errors, and there is little provision for the incorporation of recent technological improvements such as automatic flow control and alternative flow measurement devices. Because of the wide ranges of flow and inlet sizes currently allowed, samplers of similar appearance may have substantially different particle capture air velocities, which could cause differences in the size range of particles collected.

These problems can lead to unnecessary variability in measured TSP values. Many of these shortcomings can be corrected rather readily by more carefully and scientifically selected specifications, which should both reduce the method variability and at the same time allow additional flexibility for sampler manufacturers to develop and incorporate technological improvements.

It is recognized that EPA is currently reviewing the ambient air quality standard for particulate matter. One possible outcome may be the establishment of a standard based on particulates of a defined size range (inhalable particulates). If this course is taken, EPA intends to promulgate a new reference method for measuring inhaled particulates (IP). Nevertheless, promulgation of a revised TSP method is still justified because it will be very useful, during the interim time period prior to the establishment of any new ambient air quality standard, for historical continuity in trend analysis and for the measurement of another criteria pollutant, lead. It is also quite possible that a grandfather clause may be introduced to permit continued use of TSP measurements until such time as new monitoring equipment becomes available and can be purchased and installed by monitoring agencies. Finally, it would be desirous to determine if any quantitative relationship exists between the present TSP method and any new method that may be established for national trend monitoring purposes.

Objectives

The objectives of the revised method are to:

- Clarify specifications and tolerances so that sampler variability is reduced and sampler manufacturers and users will better understand what is acceptable and unacceptable.
- Change, where possible, to functional specifications to allow sampler manufacturers more flexibility to incorporate innovations and improvements.
- Provide more stringent specifications and guidance applicable to the design of new samplers.
- Provide a more explicit calibration procedure and clarify temperature and pressure corrections.

In developing the method revisions, an overriding constraint was to maintain basic comparability between TSP measurements obtained under the original and revised methods. This is extremely important because of the extensive existing data base of TSP measurements and the magnitude of resulting control requirements. Comparable TSP data will likely continue to be important for trend analysis and for ambient lead measurements. Accordingly, all the proposed changes in the revised method are intended to decrease the variability—i.e., reduce the uncertainty in the TSP measurements—without causing any bias or change in the comparability to the previously collected TSP meaurements. In some areas, this severely restricts the extent of any technical changes that can be incorporated.

Another important consideration in revising the method was to minimize the impact on the current TSP monitoring effort. Thus, revised method specifications must attempt to reduce the variability in new samplers without requiring the replacement of large numbers of samplers currently in use. These opposing objectives are particularly challenging because of the lack of clarity in the original specifications and because of the variety of samplers in use, some of which even predate the 1971

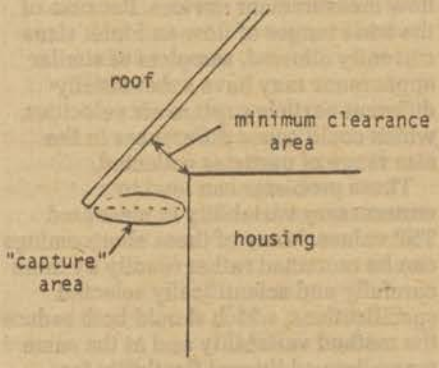
specifications. The revised method strives to accommodate both objectives with a combination of "grandfather" provisions (specific exclusions for existing samplers) and voluntary compliance with suggested "ideal" specifications. Monitoring agencies will be encouraged to check and replace any nonconforming samplers and to phase out older, marginal samplers as funds become available for newer samplers. A new IP standard requiring new monitoring methods would surely reduce the number of TSP samplers required and thereby allow retirement of many old samplers. Finally, many of the proposed changes in the revised method are in the calibration procedures, and such changes can, of course, be readily implemented with little or no economic impact.

In view of the restrictions described above, the changes contained in the revised method are minor to the extent that no changes have been made to the basic principle or comparability of the method, and no substantial impact will result to monitoring agencies. Although the actual language of the method has been changed extensively, the effect of the changes is limited to clarification and control of variability. EPA firmly believes that any apparent differences in ambient measurements that may be observed between the original method and the revised method will be less than the actual *uncertainty* in TSP measurements under the original method. Because of the lack of clarity and wide tolerances of some of the specifications, the maximum variability permitted by the original method is likely to be considerably greater than that observed under typical conditions.

Most Salient Issues

Inlet design. Perhaps the most significant and complex issue is the matter of the sampler air inlet and its relationship to the size of particles collected. Since the original sampler inlet design was only vaguely suggested by a simple line drawing, it was apparently not intended to provide any sort of particle size discrimination. But the inlet geometry does affect the size of particles collected, although the suggested geometric configuration is too simple to provide any sharp cutoff in particle size. A specification is provided for the clearance area between the roof and the main housing at its closest point (580.5 cm^2), but the specified tolerance (±193.5 cm^2) is ±33 percent, which is not very restrictive. Moreover, the specified flow range of 1.13 to 1.70 m^3/min (40 to 60 ft^3/min) allows the inlet velocity to range from 24.3 to 73.2 cm/s—a range of 200 percent. Since the size of collected particles is related to this inlet velocity, such a large velocity range could significantly affect the upper limit of the particle size collected. Changes in the upper particle size limit contribute to overall variability, particularly because of the day-to-day and site-to-site variation in the concentration of large particles (which tend to settle out at low windspeeds).

To complicate matters further, most older samplers (11½" x 14") had clearance area around 450 cm^2, which is near the lower limit of the specified range (387–774 cm^2), and newer (15" x 15") samplers have clearance areas as low as 340 cm^2—apparently below the lower range limit. However, measuring the clearance area at the closest point between the roof and the housing may not be plausible. An alternative concept suggests that whether particles are captured or not captured by the sampler depends on their aerodynamic size and the vertical air velocity in the "capture area" between the lower edge of the roof overhang and the side of the shelter housing.



Once a particle is moving vertically in this capture area, the air velocity increases to a maximum at the minimum clearance area and the particle is collected. Thus, the inlet area used to calculate this "capture air velocity" should be measured in a horizontal plane at the lower edge of the roof overhang, not at the closest point between the roof and the sampler housing.

Of course, this simple concept does not take into account the disturbances caused by windspeed at the air inlet and the fact that the air velocity is not vertical at the inlet. However, in view of the simple design of the sampler inlet, this analysis is not unreasonable. Based on this capture area concept, typical high-volume samplers are estimated to have inlet areas ranging from 700 to 900 cm^2. However, the inlet areas of older samplers may vary widely if the roof is improperly mounted or improperly latched after a filter change, or if the sampler or roof is bent or distorted. Also, on many current samplers, the inlet is not uniform on the four sides of the sampler.

To provide greater sampler-to-sampler uniformity, the particle size sampling characteristics—and hence the capture velocity—should be more closely controlled. The revised method, therefore, uses the above approach and specifies capture velocity rather than inlet area. The inlet area would be required to be sized to provide a capture air velocity of between 20 and 35 cm/s at the recommended operational flow rate. This velocity range is considerably narrower than the currently allowed range. In addition, an "ideal" velocity of 23 ±2 cm's is given as a nominal design specification for new, flow-controlled samplers. This specification is based on the average or typical capture air velocity for most samplers currently in use. Older samplers having capture velocities outside this specified range would have to be modified, either by changing their flow rate or by a physical change to the inlet area, to meet the velocity specifications.

At the specified capture velocity, particles of approximately 60μm and smaller aerodynamic diameter would be collected by the sampler, based on the settling velocity of particles with a density of 1 g/cm^3. This captured size range should be comparable to—but more uniform than—current samplers because the capture velocity would be the same as the current average but would be better controlled. However, this particle size limit is approximate because (1) actual particles are irregularly shaped and vary in density; (2) the simple geometry of the sampler provides a broad rather than a sharp size cutoff characteristic; (3) capture velocity will still vary with changes in temperature, pressure, flow rate, and inlet area; and (4) changes in windspeed significantly alter the sampler characteristics.

A capure velocity of 23 cm/s (0.5 mph) is very low with respect to typical ambient windspeeds. Hence, wind can significantly change the particle size characteristics of the sampler. This

effect has been confirmed by wind tunnel tests. There is very little that can be done to reduce the windspeed effect short of a more sophisticated inlet design. In the interest of data comparability, the revised method specifies no new or improved inlet design. Similarly, there is no attempt to improve the particle size selectivity or the sharpness of the size cutoff.

Another inlet characteristic that causes variability in TSP measurements is the rectangular shape of the sampler and the gabled roof, which obviously lead to directional sensitivity. A circular design with respect to the central vertical axis, with a domed or conical roof and an annular inlet area, would certainly reduce this directional sensitivity. However, such a substantial change in the shape of the sampler could likely lead to apparent, if not actual, loss of comparability with current TSP measurements. Therefore, the rectangular shape and gabled roof are retained in the revised method.

Flow rate measurement device. Because of the large volume of air sampled by the sampler, measurement of the total volume of air sampled is not practical. Hence, the total volume must be computed from the flow rate and the sample time period. The flow rate indicator currently specified is a small, inexpensive rotameter connected to measure a portion of the air flow at the sampler air exhaust. This device has been shown to be subject to a variety of errors, including shifts in calibration due to physical changes that alter the fraction of air sampled, deposition of dirt in the rotameter (primarily carbon from the motor brushes), and flow restrictions in the connecting tubing or rotameter outlet.

The current method does allow other types of flow indicators. Another common type consists of a manometer or aneroid pressure indicator to measure the pressure across an orifice plate mounted in the sampler air exhaust. This device is also inexpensive and eliminates many of the errors associated with the rotameter. It is generally reliable, but requires temperature and pressure corrections and is not without its own particular problems associated with the location of the orifice downstream of the motor where brush carbon, turbulence, and temperature gradients can affect measurement accuracy. In a variation of this flow indicator, the orifice is placed in the clean, less turbulent air stream between the filter and the motor. However, this requires a differential pressure indicator because neither side of the orifice is at atmospheric pressure. Also, the calibration of the device must accommodate the change in pressure drop as the filter loads during sampling.

Electronic mass flowmeters have been applied extensively to TSP samplers in the form of flow controllers, but with a suitable readout, they can certainly serve as flow indicators. Although they do not have the inherent mechanical reliability of a fixed orifice and manometer, these flowmeters appear to have adequate reliability and usually require no manual temperature or pressure corrections. The flow sensor is often mounted in the clean, nonturbulent airstream in the neck of the sampler where it senses a portion of the airflow. During calibration, a clean filter is installed on the sampler to insure that the flow pattern at the sensor is the same as it is during normal sampling.

Most other types of flow measuring devices such as venturis, turbine flowmeters, etc., are impractical because of physical or economic considerations.

The revised method allows any type of flow indicator other than the rotameter but provides a resolution specification (0.02 $stdm^3/min$) applicable to new samplers. Since the orifice/manometer and the electronic mass flowmeters are generally the most commonly used, these two types are addressed in the calibration and operational procedures. The rotameter described in the current method would be specifically disallowed 1 year after the effective date of the revised method.

Flow rate transfer standard. The use of transfer standards other than the conventional orifice/manometer type (such as an electronic mass flowmeter) would be permitted.

Filter conditioning environment. Conditioning filters prior to weighing is quite important, as humidity can substantially affect the filter weight. There is some data to indicate that relative humidities less than about 50 percent RH are suitable and necessary, but there are few or no data on temperature range for the conditioning environment. The upper temperature limit has been reduced slightly from 35° to 30° C, but the conditioning specifications are essentially the same as in the current method.

Calibration relationship. As noted above, the orifice/manometer type of flowmeter is commonly used for both the sampler's internal flow indicator and as a flow rate transfer standard. Therefore, the revised procedure addresses this type of flowmeter in detail, including the necessary temperature and pressure corrections. These corrections can be handled in a number of ways, but the most expedient way, used in the revised procedure, is as follows: During calibration, the I or ΔH values from the manometer (or aneroid instrument) are multiplied by the dimentionless expression, $(P_1/760)\ (298/T_1)$, where P_1 is the barometric pressure and T_1 is the ambient temperature during the calibration. This "corrected" or "normalized" I or ΔH is then used to establish the calibration relationship to the standard flow rate, Q_{std}. During use, the indicated I or ΔH is multiplied by the similar expression $(P_2/760)\ (298/T_2)$, where P_2 and T_2 are the barometric pressure and temperature, respectively, at the time of use. This "corrected" I or ΔH is then used with the calibration relationship to determine Q_{std}. This process allows the calibration relationship to be used at any pressure and temperature.

Although other forms for the "normalization" expression could have been used, the expression form $(P/760)\ (298/T)$ was chosen because (1) it is dimentionless when P is in units of mmHg and T is in kelvins, (2) the expression reduces to 1.00 and can, therefore, be ignored when the pressure and temperature are close to EPA reference conditions of 760 mmHg and 298 K, and (3) at normal conditions the "corrected" I or ΔH is usually very close to the uncorrected I or ΔH which many method users have been using previously. The form of this expression is identical to the expression used in gas volume corrections; however, this similarity is entirely coincidental, as the normalization process is *not* a simple gas volume correction. In fact, to obtain a linear calbration relationship, the expression becomes

$$\sqrt{\Delta H(P/760)(298/T)}.$$

A further provision included in the revised method is a variation off the normalization expression to allow geographic, average barometric pressure and seasonal average temperature to be incorporated into sampler orifice calibrations. These average pressure and temperature values approximate the actual values and permit the sampler flows to be obtained without further pressure or temperature corrections each time the sampler is used. For many sites, these approximations cause relatively small errors and considerably simplify the use of the sampler.

Other variations of the normalization expression are also used for other types of flow indicators. The actual expression to be used is selected from Table 1 of the revised method.

Flow adjustment. Under section 7.2.1 of the revised method, new samplers

would be required to have a means to adjust the sampler flow rate to accommodate changes in line voltage and filter pressure drop. This flow adjustment would likely be effected by an adjustment to the motor voltage. Any such adjustment to the motor changes the motor operating conditions, which may result in a significant change in the temperature of the sampler exhaust air. Such a change in temperature could significantly affect the calibration of an orifice flow indicator located in the sampler exhaust. The magnitude of such an effect varies from sampler to sampler and probably varies with different operating conditions, but it is usually not very large. Also, the extent of any flow adjustment needed to accommodate changes in line voltage and filter pressure drop is likely to be small. Therefore, such flow adjustments are allowed without recalibration, and a warning is provided (in Section 8.8) to alert the operator of the potential problem when making a minor flow adjustment.

Section-by-Section Changes

The numbers given below correspond to similarly numbered sections in the revised method.

1.0 *Applicability*

1.1 Since the principle and applicability of the method are not necessarily related, these descriptive elements have been separated into individual sections. (This, of course, changes the section numbering.) The applicability is clarified to specifically reference monitoring for compliance with the NAAQS for TSP, with the optional possibility of subsequent chemical analysis of the sample. As is the policy for other manual reference methods, the revised method includes the formal specifications and procedures, with quality assurance techniques described in other documents, principally 40 CFR Part 58 and the Quality Assurance Handbook.

2.0 *Principle*

This section has been expanded and reworded to describe the principle more explicitly and to clarify that the reported measurements are expressed as micrograms per cubic meter of air corrected to EPA reference conditions (25° C, 760 mmHg).

3.0 *Range*

3.1 Again, the range, precision, and accuracy are described in separate sections. The approximate upper and lower concentration range limits are stated more explicitly, with explanations of the limiting phenomena. Also included are statements concerning the range of particle sizes collected. Other specifications currently in Section 2.2 (such as weighing resolution) are moved to more appropriate sections.

4.0 *Precision*

4.1 The precision values obtained from collaborative testing are not changed.

5.0 *Accuracy*

5.1 The TSP measurement obtained is essentially defined by the method itself. The "absolute" accuracy is undefined because of the difficulty in defining the "true" particulate concentration. The usefulness of the method does not depend on the absolute accuracy. The ±50 percent error mentioned in the current method is deleted because recent tests and experience indicate that that level of variability is unrealistically pessimistic. Moreover, the new specifications and improved quality assurance procedures should significantly reduce the measurement uncertainty.

6.0 *Inherent Sources of Error*

This new section is added to discuss various recognized sources of error and what, if anything, can be done to minimize these errors. Error sources discussed are (1) air flow variation, (2) air volume measurement, (3) loss of volatiles, (4) artifact (extrinsic) particulates, (5) humidity, (6) filter handling, (7) nonsampled particulates, and (8) timing errors.

The current specification (Section 7.1.2) that the sample is to be collected "from midnight to midnight" has been dropped. The start and stop times of the sampler are of interest here only in determining the elapsed sampling period and have no other bearing on the technical aspects of the method. Omitting this specification is also consistent with § 58.13(b), which covers the operating schedule of manual methods but does not specify starting times for sampling periods.

7.0 *Apparatus*

7.1 *Filter.* Specifications for the collection filter—appearing in various sections of the current method—are brought together in this section, clarified, and augmented with new specifications for maximum pressure drop, pH, integrity, pinholes, tear strength, and brittleness.

7.2 *Sampler.* The sampler specifications are reoriented to reflect functional descriptions allowing more flexibility in sampler design within clearly defined limits. Figure B1 is eliminated, and the specifications are itemized. A new provision applicable only to samplers sold after the effective date of the revision would require samplers to have some sort of flow rate adjustment to accommodate variations in line voltage, filter pressure drop, expected filter loading, or operational preference within the specified flow range. Another new provision requires samplers equipped with flow controllers to have a means to disable the flow controller during calibration of the sampler flow rate indicator.

The flow rate range is reduced slightly (about 12 percent) from 1.13 to 1.70 m^3/min (40 to 60 ft^3/min) to 1.0 to 1.5 m^3/min (35.3 to 53 ft^3/min). This should have no significant effect on the comparability of measurements for two reasons: First, the sampler has been shown to be relatively insensitive to minor changes in flow rate, and second, as discussed under Salient Issues, it is the capture air velocity that affects the particle collection characteristics, not the flow rate alone. A new specification applies to the capture air velocity. Advantages of the slightly reduced flow rate include round-number specifications in metric units (m^3/min), slightly reduced noise and power consumption, greater control range on existing flow-controlled samplers, and extended brush life.

7.3 *Sampler Shelter.* As with the sampler, the shelter specifications are restated as functional specifications. The clearance area specification is replaced with a capture air velocity specification (20 to 35 cm/s) as discussed previously. An "ideal" velocity of 23 ±2 cm/s (1.1 m^3/min flow rate with a capture area of about 800 cm^2) is suggested as a nominal design objective for newly designed samples. Inlet openings of existing samplers that do not allow a capture velocity of 20 to 35 cm/s to be obtained would have to be modified.

7.4 *Flow rate measurement device.* See previous discussion under Most Salient Issues.

7.5 *Thermometer.* May be needed for temperature measurements when using an orifice-type flow indicator.

7.6 *Barometer.* May be needed for pressure measurements when using an orifice-type flow indicator.

7.7 *Timing/control device.* Since most samplers are operated from midnight to midnight, some sort of timer is needed to start and stop the sampler. Emphasis is put on accuracy of the elapsed time rather than the exact start and stop times. An accuracy of time setting specification (± 15 min) is provided for existing sampler timers that have no elasped-time capability. This specification is broad enough to continue to allow mechanical timers, although electronic timers with their much better set-point resolution are recommended. (Note cross reference to section 6.8.)

7.8 *Flow rate transfer standard.* Calibration of the sampler's internal flow rate indicator requires a rather specialized calibration device; therefore, the method includes specifications for such a device. Again, the specifications are functional in nature to allow flexibility in the type of device used rather than to limit it to a specific orifice unit as described in the current method. Consistent with its role and with established policy, the device is referred to as a flow rate transfer standard.

The above notwithstanding, the conventional orifice/manometer type transfer standard is rugged, reliable, inexpensive, and is in wide-spread use. Thus, much of the calibration procedure is addressed to this type of transfer standard. Although the conventional use of various individual resistance plates would still be allowed, a new device having an variable, external resistance adjustment is recommeded. Newer electronic mass flowmeter-type transfer standards are also available. Figure 2 illustrates examples of three commonly used transfer standards.

7.9 *Filter conditioning environment.* See discussion under "Salient Issues."

8.0 *Procedure*

As noted previously, the revised method provides only basic procedural information with the associated quality assurance procedures contained in other sources (references 1 and 2). The procedure section is clarified and restated in a more stepwise format, and the instructions are generalized and oriented toward either a mass flowmeter or an orifice/manometer-type flow rate indicator, since the rotameter is not allowed as a flow indicator. Because orifice/manometer flow indicators generally require temperature and pressure correction, the procedure includes instructions to make these corrections as well as instructions for using geographic barometric pressure and seasonal average temperature to simplify sampler use.

9.0 *Calibration*

9.1 Calibration refers to calibration of the sampler's internal flow rate indicator. The two phases—calibration of the transfer standard and calibration of the flow indicator—are described separately and are illustrated in Figure 2.

9.2 Because the orifice/manometer type of transfer standard is so widely used and because it requires temperature and pressure corrections for accurate, use, the transfer standard calibration procedure applies rather exclusively to that type of transfer standard. Other types of transfer standards—electronic mass flowmeters for example—are allowed, but they would almost certainly require a different calibration procedure. The calibration procedure for any other type of flow transfer standard would have to be approved under 40 CFR Part 58 (Modifications of Methods by Users).

The transfer standard calibration procedure is greatly expanded and more detailed than the current procedure. Temperature and pressure corrections are explicitly specified and a data worksheet (Figure 3) is provided for convenience and accuracy. The entire procedure is designed so that even an inexperienced analyst can obtain a correct result if the steps are followed completely and accurately.

Because of the temperature and pressure sensitivity of orifice-type transfer standards, the calibration relationship (between standard flow and indicated reading) is developed in a way that can be readily applied in the field at any temperature and pressure (see discussion of this procedure under "Salient Issues"). Either a linear or nonlinear graphical method can be used or least-squares regression analysis can be applied to establish the calibration relationship.

9.3 Similarly, the calibration procedure for the sampler is also greatly expanded and detailed, including explicitly specified temperature and pressure corrections and a calibration worksheet (Figure 4). The procedure is designed chiefly for orifice-type flow indicators located downstream of the motor but also covers electronic mass flowmeters; these are the two most common flow indicators. The calibration procedure may have to be modified to accommodate other types of flow indicators.

Again, the calibration procedure is developed so that it can be applied at any temperature or pressure according to the instructions provided. The procedure also allows for incorporation of geographic, average barometric pressure and seasonal average temperature. Either a linear or nonlinear graphical or least-squares regression relationship can be established. For electronic mass flow meters, no temperature or pressure corrections are usually required. The procedure also covers the special case of the pressure recorder which has square-root-function chart paper (e.g., Dixon meter). These various options are accommodated by selection of the appropriate expression from Table 1 to use in the calibration relationship.

For samplers equipped with a flow controller, the controller is disabled during the calibration so that the flow indicator can be calibrated over a range of flow rates rather than just at the controlled flow rate. Normally, the flow rate would be varied by adjusting the flow resistance provided by the transfer standard. However, in the case of an electronic mass flowmeter, the flow could be adjusted equally well by adjusting the voltage or power supplied to the motor.

10.0 *Calculations*

The calculations necessary to determine the ambient TSP concentration are specified in explicit stepwise form and cover the three most common types of flow indicators: electronic mass flowmeter, orifice/manometer flowmeter, and orifice/pressure flow indicator with square-root chart (Dixon). The calculations are facilitated by selection of the proper expression from Table 2 to correspond with the expression from Table 1 selected during calibration. The calculations are greatly simplified when geographic average barometric pressure and seasonal average temperature are incorporated into the calibration. Also provided is an alternate method for determining the average sampler flow rate when using a continuous flow recorder. Finally, a formula is provided for converting the conventional TSP concentration in micrograms per standard cubic meter to the actual concentration in micrograms per actual cubic meter (actual conditions).

Appendix C—Measurement Principle and Calibration Procedure for Carbon Monoxide

Background

Appendix C to Part 50 was amended in 1975 (40 FR 7043) to incorporate the measurement principle and calibration procedure concept for carbon monoxide (CO) reference methods. However, the language of the current measurement principle description and calibration procedure, which were left largely unchanged from the original promulgation in 1971, is in need of additional clarification. The present measurement principle describes a particular photometer design that is not unique to the basic physical principles of the CO measurement intended and thereby leaves doubt as to the qualification of other designs or configurations that also utilize the same basic principle. The present calibration procedure is sketchy and needs supplemental details and specifications to assure adequate calibration of CO reference methods.

To correct these deficiencies, Appendix C has been largely rewritten, but no significant changes are proposed to the basic objectives and intent. The

proposed new language augments the existing measurement principle and calibration procedure with additional technical details and clarification. In addition, the new version is more consistent with the measurement principle and calibration procedure descriptions in other appendixes to Part 50.

Measurement Principle

The revised description of the measurement principle is written in a more generalized, functional form to allows a variety of designs of the photometer. This is important so that the measurement principle description does not inadvertently preclude new, improved designs or new configurations of components that are clearly within the intended scope of the measurement principle. In particular, the new description more clearly allows analyzers using the gas filter correlation technique to qualify as reference methods.

Calibration Procedure

The calibration procedure is augmented with much more technical detail, following a format used for other calibration procedures in other appendixes to Part 50. Two calibration methods are described, one using dilution of a single compressed gas CO standard and the other using multiple compressed gas CO standards. Typical calibration system configurations for each method are shown along with specifications for the major components and for the CO standards. The procedure provides step-by-step instructions for establishing flowing CO atmospheres, calculating diluted CO standard concentrations, adjusting the analyzer's zero and span controls, and preparing calibration curves. Also, section 3.1 allows CO calibration standards to be traceable to either a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to an NBS/EPA-approved gas manufacturer's Certified Reference Material (CRM). This provision is consistent with similar provisions in amendments being proposed to 40 CFR Parts 50 and 58 elsewhere in this **Federal Register.**

Reference (Preamble)

(1) McCaldin, R. O., and E. R. Hendrickson. Use of a Gas Chamber for Testing Air Samplers. J. Amer. Ind. Hyg. Associ., 20:509, 1959.

(2) Perry, W. H., and E. C. Tabor. National Air Sampling Network Measurement of SO_2 and NO_2. Arch. Environ. Health, 4:44 1962.

(3) Lahman, E., The Stability of Absorption Solution for Sulfur Dioxide Determination by the West-Gaeke Method. Staub-Reinhalt. Luft, 29:30, 1969.

(4) Scaringelli, F. P., L. Elfers, D. Norris, and S. Hochheiser. Enhanced Stability of Sulfur Dioxide in Solution. Anal. Chem., 42:1818, 1970.

(5) Shinji, T., E. Kazuhiko, and K. Kazuma. Studies of Analytical Errors in Measurement of Sulfur Dioxide in the Air by the Pararosaniline Method. Jpn. Anal. (Bunseki Kagaku), 20:1097, 1971.

(6) Kasten-Schraufnagel, P., D. L. Ehman, and D. J. Johnson. Telmatic Study Phase II: Effects of Collection and Handling Conditions on the Stability of the Dichlorosulfitomercurate (II) Complex Formed in Sampling for SO_2 by the Modified West-Gaeke Method. Texas Air Control Board, Air Quality Evaluation Division, Austin, Texas, January 15, 1975.

(7) Sweitzer, T. A. The Evaluation of Gas Bubbler Field Performance. Presented at the 32nd Annual Meeting of the East Central Section of the Air Pollution Control Association, Dayton, Ohio, September 17–19, 1975.

(8) Fuerst, R. G., F. P. Scaringelli, and J. H. Margeson. Effect of Temperature of Stability of Sulfur Dioxide Samples Collected by the Federal Reference Method. EPA–600/4–76–024, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, May 1976.

(9) Martin, B. E. Sulfur Dioxide Bubbler Temperature Study. EPA–600/4–77–040, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, August 1977.

(10) Clements, J. B. Memorandum to Directors, Surveillance and Analysis Divisions, Air and Hazardous Materials Divisions, Quality Control Coordinators, EPA Regions I–X; Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, December 29, 1975.

Under Executive Order 12291, EPA must judge whether a regulation is "major" and therefore subject to the requirement of a Regulatory Impact Analysis. This proposed rule is not a major regulation because it principally revises the existing reference methods for SO_2, TSP, and CO to correct identified short-comings and ambiguities. Certain technical improvements have also been incorporated, but all of the proposed changes are designed to improve the quality and comparability of ambient measurements.

This proposed rule was submitted to the Office of Management and Budget for review as required by Executive Order 12291.

Pursuant to the provisions of 5 U.S.C. 605(b), I hereby certify that the attached rule will not, if promulgated, have a significant economic impact on a substantial number of small entities. The proposed rule, if promulgated, would impose no new regulatory requirements; principally, it would correct certain identified shortcomings, clarify ambiguities, and incorporate minor but important technical improvements in the existing reference methods for SO_2, TSP, and CO. The economic impact on monitoring agencies resulting from these method revisions is not considered significant because of the minimal cost of upgrading existing equipment and procedures.

Dated: January 7, 1982.

Anne M. Gorsuch,

Administrator.

PART 50—NATIONAL PRIMARY AND SECONDARY AMBIENT AIR QUALITY STANDARDS

As indicated in the foregoing discussion, it is proposed to amend 40 CFR Part 50 as follows:

1. By revising Appendix A to read as follows:

Appendix A—Reference Method for the Determination of Sulfur Dioxide in the Atmosphere (Pararosaniline Method)

1.1 This method provides a measurement of the concentration of sulfur dioxide (SO_2) in ambient air for determining compliance with the primary and secondary national ambient air quality standards for sulfur oxides (sulfur dioxide) as specified in § 50.4 and § 50.5 of this chapter. The method is applicable to the measurement of ambient SO_2 Concentrations using sampling periods ranging from 30 minutes to 24 hours. Additional quality assurance procedures and guidance are provided in part 58, appendixes A and B, of this chapter and in references (*1*) and (*2*).

2.0 *Principle.* 2.1 A measured volume of air is bubbled through a solution of 0.04 M potassium tetrachloromercurate (TCM). The SO_2 present in the air stream reacts with the TCM solution to form a stable monochlorosulfonatomercurate (*3*) complex. Once formed, this complex resists air oxidation (*4*) (*5*) and is stable in the presence of strong oxidants such as ozone and oxides of nitrogen. During subsequent analysis, the complex is reacted with acid-bleached pararosaniline dye and formaldehyde to form an intensely colored pararosaniline methyl sulfonic acid. (*6*) The optical density of this species is determined spectrophotometrically at 548 nm and is directly related to the amount of SO_2 collected. The total volume of air sampled, corrected to EPA reference conditions (25° C, 760 mm Hg), is determined from the measured flow rate and the sampling time. The concentration of SO_2 in the ambient air is computed and expressed in micrograms per standard cubic meter ($\mu g/std\ m_3$).

3.0 Range. 3.1 The lower limit of detection of SO_2 in 10 mL of TCM is 0.75 μg (based on collaborative test results). (*7*) This represents a concentration of 25 $\mu g\ SO_2/m_3$ (0.01 ppm) in an air sample of 30 standard liters (short-term sampling) and a concentration of 13 $\mu g\ SO_2/m^3$ (0.005 ppm) in an air sample of 288 standard liters (long-term sampling). Concentrations less than 25

$\mu g\ SO_2/m^3$ can be measured by sampling larger volumes of ambient air; however, the collection efficiency falls off rapidly at low concentrations (*8*)(*9*) Beer's law is adhered to up to 34 μg of SO_2 in 25 mL of final solution. This upper limit of the analysis range represents a concentration of 1,130 $\mu g\ SO_2/m^3$ (0.43 ppm) in an air sample of 30 standard liters and a concentration of 590 $\mu g\ SO_2/m^3$ in an air sample of 288 standard liters. Higher concentrations can be measured by collecting a smaller volume of air, by increasing the volume of absorbing solution, or by diluting a suitable portion of the collected sample with solution prior to analysis.

4.0 *Interferences.* 4.1 The effects of the principal potential interferences have been minimized or eliminated in the following manner: nitrogen oxides by the addition of sulfamic acid, (*10*)(*11*) heavy metals by the addition of ethylenediamine tetracetic acid disodium salt (EDTA) and phosphoric acid, (*10*)(*12*) and ozone by time delay. (*10*) Up to 60 μg Fe (III), 22 μg V (V), 10μg Cu (II), 10 μg Mn (II), and 10 μg Cr (III) in 10 mL absorbing reagent can be tolerated in the procedure. (*10*) No significant interference has been encountered with 2.3 $\mu g\ NH^3$. (*13*)

5.0 *Precision and Accuracy.* 5.1 The precision of the analysis is 4.6 percent (at the 95 percent confidence level) based on the analysis of standard sulfite samples.(*10*)

5.2 Collaborative test results (*14*) based on the analysis of synthetic test atmospheres (SO_2 in scrubbed air) using the 24-hour sampling procedure and the sulfite-TCM calibration procedure show that:

- The replication error varies linearly with concentration from $\pm 2.5\ \mu g/m^3$ at concentrations of 100 $\mu g.m^3$ to $\pm 7\ \mu g.m^3$ at concentrations of 400 $\mu g/m^3$.
- The day-to-day variability within an individual laboratory (repeatability) varies linearly with concentration from $\pm 18.1\ \mu g/m^3$ at levels of 100 $\mu g.m^3$ to $\pm 50.9\ \mu g/m^2$ at levels of 400 $\mu g.m^3$.
- The day-to-day variability between two or more laboratories (reproducibility) varies linearly with concentration from $\pm 36.9\ \mu g/m^3$ at levels of 100 $\mu g/m^3$ to $\pm 103.5\ \mu g/m^3$ at levels of 400 $\mu g/m^3$.
- The method has a concentration-dependent bias, which becomes significant at the 95 percent confidence level at the high concentration level. Observed values tend to be lower than the expected SO_2 concentration level.

6.0 *Stability.* 6.1 By sampling in a controlled temperature environment of 15° ±10° C, greater than 98.9 percent of the SO_2—TCM complex is retained at the completion of sampling. (*15*) If kept at 5° C following the completion of sampling, the collected sample has been found to be stable for up to 30 days. (*10*) The presence of EDTA enhances the stability of SO_2 in the TCM solution and the rate of decay is independent of the concentration of SO_2. (*16*)

7.0 *Apparatus.*

7.1 *Sampling.*

7.1.1 *Sample probe:* A sample probe meeting the requirements of section 7 of 40 CFR Part 58, Appendix E (Teflon® or glass with residence time less than 20 sec.) is used to transport ambient air to the sampling train location. The end of the probe should be inverted to preclude the sampling of precipitation, large particles, etc. A suitable probe can be constructed from Teflon® tubing connected to an inverted funnel.

7.1.2 *Absorber—short-term sampling:* An all glass midget impinger having a solution capacity of 30 mL and a stem clearance of 4 ±1 mm from the bottom of the vessel is used for sampling periods of 30 minutes and 1 hour (or any period considerably less than 24 hours). Such an impinger is shown in Figure 1. These impingers are commercially available from distributors such as Ace Glass, Incorporated.

7.1.4 *Absorber—24-hour sampling:* A polypropylene tube 32 mm in diameter and 164 mm long (available from Bel Art Products, Pequammock NJ) is used as the absorber. The cap of the absorber must be a polypropylene cap with two ports (rubber stoppers are unacceptable because the absorbing reagent can react with the stopper to yield erroneously high SO_2 concentrations). A glass impinger stem, 6 mm in diameter and 158 mm long, is inserted into one port of the absorber cap. The tip of the stem is tapered to a small diameter orifice (0.4 ±0.1 mm) such that a No. 79 jeweler's drill bit will pass through the opening but a No. 78 drill bit will not. Clearance from the bottom of the absorber to the tip of the stem must be 6 ±2 mm. Glass stems can be fabricated by any reputable glass blower or can be obtained from a scientific supply firm. Upon receipt, the orifice test should be performed to verify the orifice size. The assembled absorber is shown in Figure 2.

7.1.4 *Moisture trap:* A moisture trap constructed of a glass trap as shown in Figure 1 or a polypropylene tube as shown in Figure 2 is placed between the absorber tube and flow control device to prevent entrained liquid from reaching the flow control device. The tube is packed with silica gel as shown in Figure 2. Glass wool may be substituted for silica gel when collecting short-term samples (1 hour or less) as shown in Figure 1.

7.1.5 *Heat shrinkable tape (24-hour sampling):* A heat skrink seal of appropriate diameter is required for sealing the absorber and cap and the moisture trap and cap to prevent leakage during sampling. A heat gun for shrinking the tape is also required. Figure 2 shows a sampling assembly utilizing the heat shrink seal.

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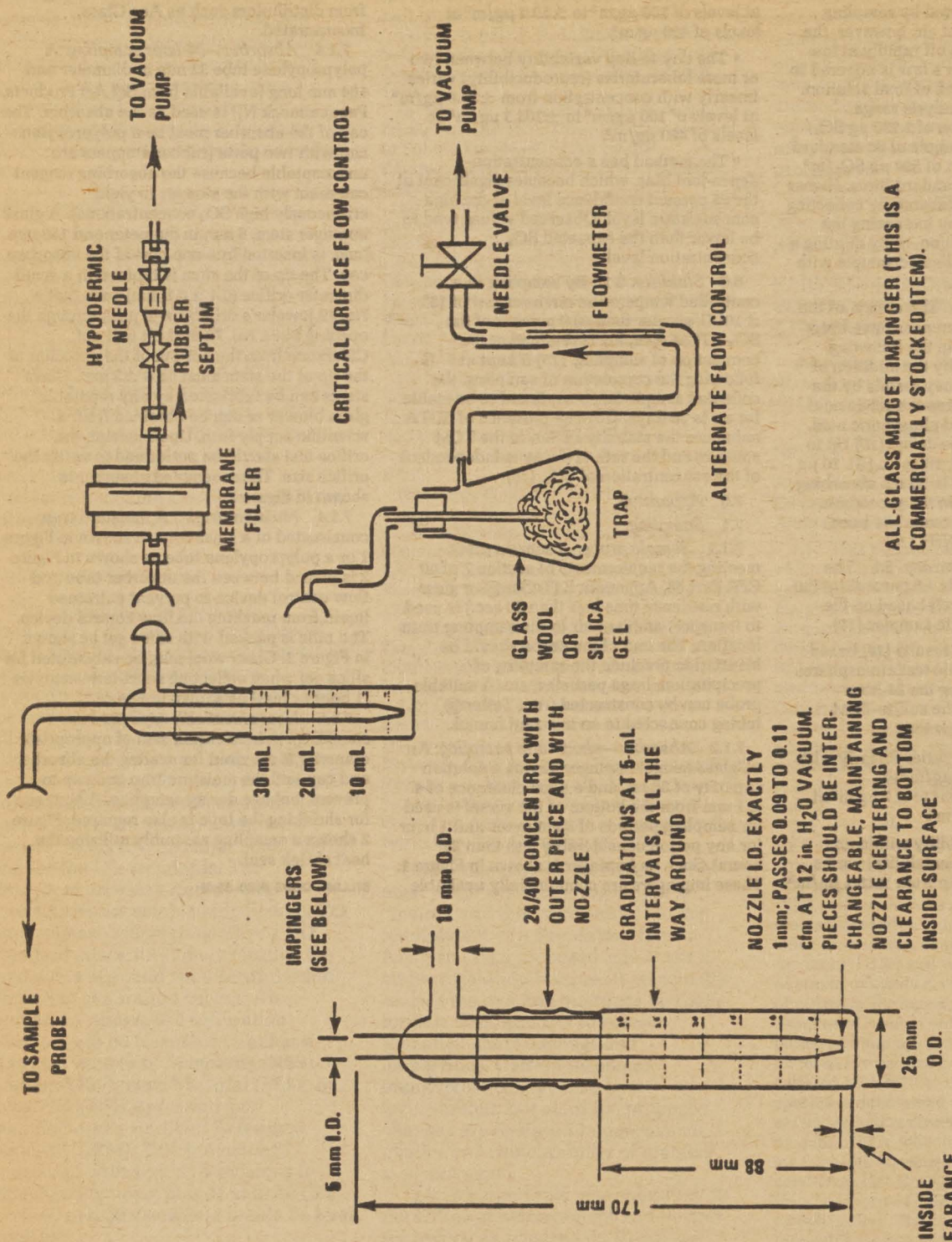

Figure 1. Short-term sampling train.

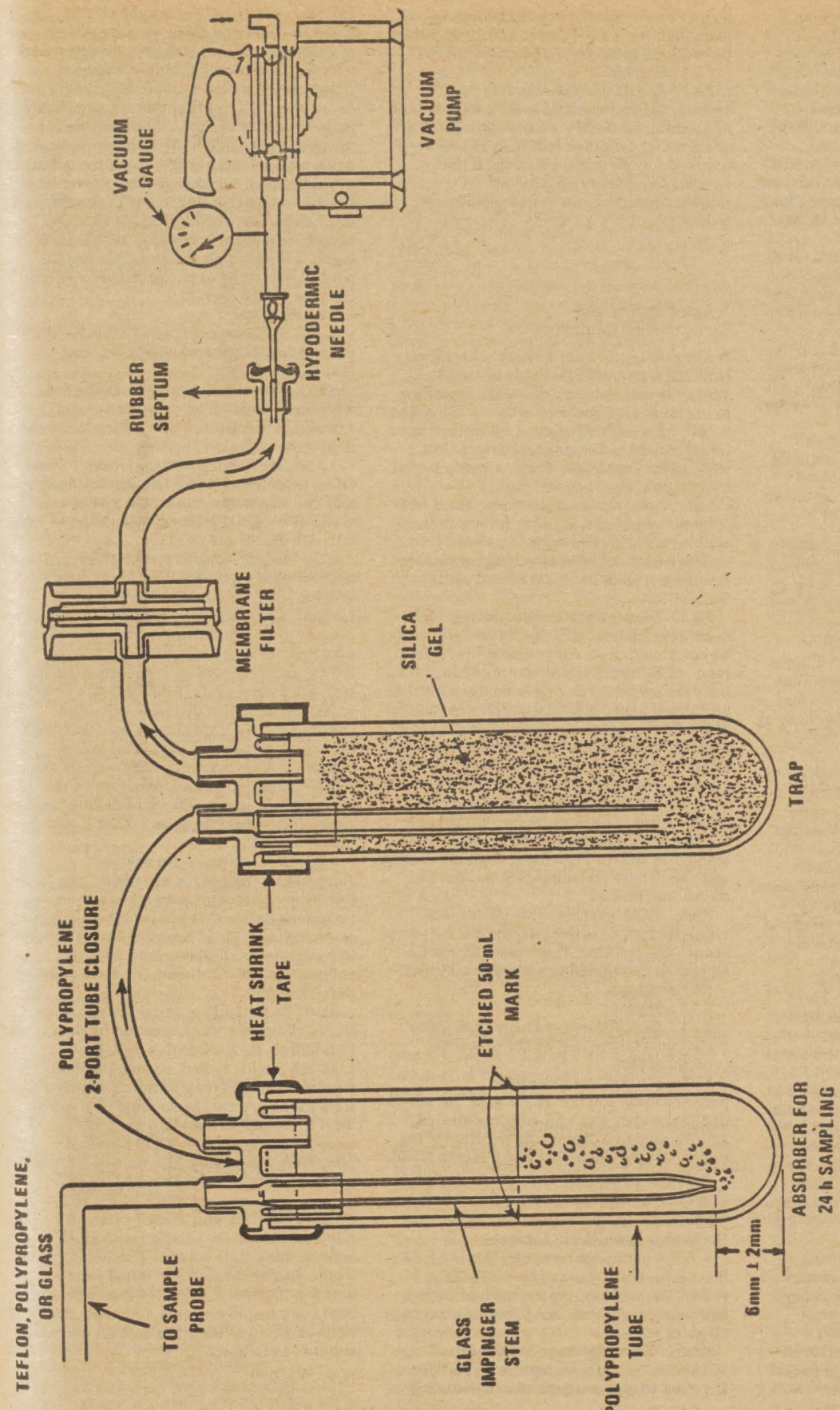

Figure 2. 24-Hour sampling system.

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7.1.6 *Flow control device:* A calibrated rotameter and needle valve combination capable of maintaining and measuring air flow to within ±2 percent is suitable for short-term sampling but may not be used for long-term sampling. Calibrated critical orifices can be used for both long- and short-term sampling. A 22-gauge hypodermic needle 25 mm long may be used as a critical orifice to yield a flow rate of 1 L/min for a 30-minute sampling period. When sampling for 1 hour, a 23-gauge hypodermic needle 16 mm in length will provide a flow rate of approximately 0.5 L/min. Flow control for a 24-hour sample may be provided by a 27-gauge hypodermic needle critical orifice that is 9.5 mm in length. The flow rate should be in the range of 0.18 to 0.22 L/min.

7.1.7 *Membrane particle filter:* A membrane filter of 0.8 to 2 μm porosity is used to protect the flow controller from particles during long-term sampling. This item is optional for short-term sampling.

7.1.8 *Vacuum pump:* A vacuum pump equipped with a vacuum gauge and capable of maintaining at least a 0.7 atm vacuum differential across the flow control device at the specified flow rate is required for sampling.

7.1.9 *Temperature control device:* The temperature of the absorbing solution during sampling must be maintained at 15° ±10° C. As soon as possible following sampling and until analysis, the temperature of the collected sample must be maintained at 5°±5° C. Where an extended period of time may elapse before the collected sample can be moved to the lower storage temperature, a collection temperature near the lower limit of the 15 ±10° C range should be used to minimize losses during this period. Thermoelectric coolers specifically designed for this temperature control are available commercially and normally operate in the range of 5° to 15° C. Small refrigerators can be modified to provide the required temperature control; however, inlet lines must be insulated from the lower temperature to prevent condensation when sampling under humid conditions. A small heating pad may be necessary when sampling at low temperatures (<7° C) to prevent the absorbing solution from freezing. (*17*).

7.1.10 *Sampling train container:* The absorbing solution must be shielded from light during and after sampling. Most commercially available sampler trains are enclosed in a light-proof box.

7.1.11 *Timer:* A timer is recommended to initiate and to stop sampling for the 24-hour period. The timer is not a required piece of equipment; however, without the timer a technician would be required to manually start and stop sampling. An elapsed time meter is also recommended to determine the duration of the sampling period.

7.2 *Shipping.*

7.2.1 *Shipping container:* A shipping container that can maintain a temperature of 5°±5° C is used for transporting the sample from the collection site to the analytical laboratory. Ice coolers or refrigerated shipping containers have been found to be satisfactory. The use of eutectic cold packs instead of ice will give a more stable temperature control. Such equipment is available from Cole-Parmer Company, 7425 North Oak Park Avenue, Chicago, IL 60648.

7.3 *Analysis.*

7.3.1 *Spectrophotometer:* A spectrophotometer suitable for measurement of absorbances at 548 nm with an effective spectral bandwidth of less than 15 nm is required for use during analysis. If the spectrophotometer reads out in transmittance, convert to absorbance as follows:

$$A = \log_{10}(1/T) \quad (1)$$

where

A = absorbance, and
T = transmittance (0<T<1).

A neutral density filter available from the National Bureau of Standards is used to verify the wavelength calibration according to the procedure enclosed with the filter. The wavelength calibration must be verified upon initial receipt of the instrument and after each 160 hours of normal use or every 6 months, whichever occurs first.

7.3.2 *Spectrophotometer cells:* A set of 1-cm path length cells suitable for use in the visible region is used during analysis. If the cells are unmatched, a matching correction factor must be determined according to Section 10.1.

7.3.3 *Temperature control device:* The color development step during analysis must be conducted in an environment that is in the range of 20° to 30° C and controlled to ±1° C. Both calibration and sample analysis must be performed under identical conditions (within 1° C). Adequate temperature control may be obtained by means of constant temperature baths, water baths with manual temperature control, or temperature controlled rooms.

7.3.4 *Glassware:* Class A volumetric glassware of various capacities is required for preparing and standardizing reagents and standards and for dispensing solutions during analysis. These include pipets, volumetric flasks, and burets.

7.3.5 *TCM waste receptacle:* A glass waste receptacle is required for the storage of spent TCM solution. This vessel should be stoppered and stored in a hood at all times.

8.0 *Reagents.*

8.1 *Sampling.*

8.1.1 *Distilled water:* Purity of distilled water must be verified by the following procedure: (*18*)

- Place 0.20 mL of potassium permanganate solution (0.316 g/L), 500 mL of distilled water, and 1 mL of concentrated sulfuric acid in a chemically resistant glass bottle, stopper the bottle, and allow to stand.
- If the permanganate color (pink) does not disappear completely after a period of 1 hour at room temperature, the water is suitable for use.
- If the permanganate color does disappear, the water can be purified by redistilling with one crystal each of barium hydroxide and potassium permanganate in an all glass still.

8.1.2 *Absorbing reagent (0.04 M potassium tetrachloromercurate [TCM]):* Dissolve 10.86 g mercuric chloride, 0.066 g EDTA, and 6.0 g potassium chloride in distilled water and dilute to volume with distilled water in a 1,000-mL volumetric flask. (*Caution:* Mercuric chloride is highly poisonous. If spilled on skin, flush with water immediately.) The pH of this reagent should be between 3.0 and 5.0. (*10*) Check the pH of the absorbing solution by using pH indicating paper or a pH meter. If the pH of the solution is not between 3.0 and 5.0, the solution must be discarded according to one of the disposal techniques described in Section 13.0. The absorbing reagent is normally stable for 6 months. If a precipitate forms, discard the reagent according to one of the procedures described in Section 13.0.

8.2 *Analysis.*

8.2.1 *Sulfamic acid (0.6%):* Dissolve 0.6 g sulfamic acid in 100 mL distilled water. Prepare fresh daily.

8.2.2 *Formaldehyde (0.2%):* Dilute 5 mL formaldehyde solution (36 to 38 percent) to 1,000 mL with distilled water. Prepare fresh daily.

8.2.3 *Stock iodine solution (0.1 N):* Place 12.7 g resublimed iodine in a 250-mL beaker and add 40 g potassium iodide and 25 mL water. Stir until dissolved, then dilute to 1,000 mL with distilled water.

8.2.4 *Iodine solution (0.01 N):* Prepare approximately 0.01 N iodine solution by diluting 50 mL of stock iodine solution (Section 8.2.3) to 500 mL with distilled water.

8.2.5 *Starch indicator solution:* Triturate 0.4 g soluble starch and 0.002 g mercuric iodide (preservative) with enough distilled water to form a paste. Add the paste slowly to 200 mL of boiling distilled water and continue boiling until clear. Cool and transfer the solution to a glass stoppered bottle.

8.2.6 *1 N hydrochloric acid:* Slowly and while stirring, add 86 mL of concentrated hydrochloric acid to 500 mL of distilled water. Allow to cool and dilute to 1,000 mL with distilled water.

8.2.7 *Potassium iodate solution:* Accurately weigh to the nearest 0.1 mg, 1.5 g (record weight) of primary standard grade potassium iodate that has been previously dried at 180° C for at least 3 hours and cooled in a dessicator. Dissolve, then dilute to volume in a 500-mL volumetric flask with distilled water.

8.2.8 *Stock sodium thiosulfate solution (0.1 N):* Prepare a stock solution by dissolving 25 g sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in 1,000 mL freshly boiled, cooled, distilled water and adding 0.1 g sodium carbonate to the solution. Allow the solution to stand at least 1 day before standardizing. To standardize, accurately pipet 50 mL of potassium iodate solution (Section 8.2.7) into a 500-mL iodine flask and add 2.0 g of potassium iodide and 10 mL of 1 N HCl. Stopper the flask and allow to stand for 5 minutes. Titrate the solution with stock sodium thiosulfate solution (Section 8.2.8) to a pale yellow color. Add 5 mL of starch solution (Section 8.2.5) and titrate until the blue color just disappears. Calculate the normality (N_s) of the stock sodium thiosulfate solution as follows:

$$N_s = \frac{W \times 2.80}{M} \quad (2)$$

where
M = volume of thiosulfate required in mL, and
W = weight of potassium iodate in g (recorded weight in Section 8.2.7).

$$2.80 = \frac{10^3 \text{ (conversion of g to mg)} \times 0.1 \text{ (fraction iodate used)}}{35.67 \text{ (equivalent weight of potassium iodate)}}$$

8.2.9 *Working sodium thiosulfate titrant (0.01 N):* Accurately pipet 100 mL of stock sodium thiosulfate solution (Section 8.2.8) into a 1,000-mL volumetric flask and dilute to volume with freshly boiled, cooled, distilled water. Calculate the normality of the working sodium thiosulfate titrant (N_T) as follows:

$$N_T = N_s \times 0.100$$

8.2.10 *Standardized sulfite solution for the preparation of working sulfite-TCM solution:* Dissolve 0.30 g sodium metabisulfite ($Na_2S_2O_5$) or 0.40 g sodium sulfite (Na_2SO_3) in 500 mL of recently boiled, cooled, distilled water. (Sulfite solution is unstable; it is therefore important to use water of the highest purity to minimize this instability.) This solution contains the equivalent of 320 to 400 μg SO_2/mL. The actual concentration of the solution is determined by adding excess iodine and back-titrating with standard sodium thiosulfate solution. To back-titrate, pipet 50 mL of the 0.01 N iodine solution (Section 8.2.4) into each of two 500-mL iodine flasks (A and B). To flask A (blank) add 25 mL distilled water, and to flask B (sample) pipet 25 mL sulfite solution. Stopper the flasks and allow to stand for 5 minutes. Prepare the working sulfite-TCM solution (Section 8.2.11) immediately prior to adding the iodine solution to the flasks. Using a buret containing standardized 0.01 N thiosulfate titrant (Section 8.2.9), titrate the solution in each flask to a pale yellow color. Then add 5 mL starch solution (Section 8.2.5) and continue the titration unitl the blue color just disappears.

8.2.11 *Working sulfite-TCM solution:* Accurately pipet 5 mL of the standard sulfite solution (Section 8.2.10) into a 250-mL volumetric flask and dilute to volume with 0.04 M TCM. Calculate the concentration of sulfur dioxide in the working solution as follows:

$$C_{TCM/SO_2} (\mu g\ SO_2/mL) = \frac{(A-B)(N_T)(32{,}000)}{25} \times 0.02 \quad (4)$$

where
A = volume of thiosulfate titrant required for the blank, mL;
B = volume of thiosulfate titrant required for the sample, mL;
N_T = normality of the thiosulfate titrant, from equation (3); 32,000 = milliequivalent weight of SO_2, μg;
25 = volume of standard sulfite solution, mL; and
0.02 = dilution factor.

This solution is stable for 30 days if kept at 5° C. (*16*) If not kept at 5° C, prepare fresh daily.

8.2.12 *Purified pararosaniline (PRA) stock solution (0.2% nominal):*

8.2.12.1 *Dye specifications—*

• The dye must have a maximum absorbance at a wavelenght of 540 nm when assayed in a buffered solution of 0.1 M sodium acetate-acetic acid;

• The absorbance of the reagent blank, which is temperature sensitive (0.015 absorbance unit/°C), must not exceed 0.170 at 22° C with a 1-cm optical path length when the blank is prepared according to the specified procedure;

• The calibration curve (Section 10.0) must have a slope equal to 0.030 ±0.002 absorbance unit/μg SO_2 with a 1-cm optical path length when the dye is pure and the sulfite solution is properly standardized.

8.2.12.2 *Preparation of stock PRA solution*—A specially purified (99 to 100 percent pure) solution of pararosaniline, which meets the above specifications, is commercially available in the required 0.20 percent concentration (Harleco Co.). Alternatively, the dye may be purified, a stock solution prepared, and then assayed according to the procedure as described below.[10]

8.2.12.3 *Purification procedure for PRA—*

1. Place 100 mL each of 1-butanol and 1 N HCl in a large separatory funnel (250-mL) and allow to equilibrate. Note: Certain batches of 1-butanol contain oxidants that create an SO_2 demand. Before using, check by placing 20 mL of 1-butanol and 5 mL of 20 percent potassium iodide (KI) solution in a 50-mL separatory funnel and shake thoroughly. If a yellow color appears in the alcohol phase, redistill the 1-butanol from silver oxide and collect the middle fraction or purchase a new supply of 1-butanol.

2. Weigh 100 mg of pararosaniline hydrochloride dye (PRA) in a small beaker. Add 50 mL of the equilibrated acid (drain in acid from the bottom of the separatory funnel in 1.) to the beaker and let stand for several minutes. Discard the remaining acid phase in the separatory funnel.

3. To a 125-mL separatory funnel, add 50 mL of the equilibrated 1-butanol (draw the 1-butanol from the top of the separatory funnel in 1.). Transfer the acid solution (from 2.) containing the dye to the funnel and shake carefully to extract. The violet impurity will transfer to the organic phase.

4. Transfer the lower aqueous phase into another separatory funnel, add 20 mL of equilibrated 1-butanol, and extract again.

5. Repeat the extraction procedure with three more 10-mL portions of equilibrated 1-butanol.

6. After the final extraction, filter the acid phase through a cotton plug into a 50-mL volumetric flask and bring to volume with 1 N HCl. This stock reagent will be a yellowish red.

7. To check the purity of the PRA, perform the assay and adjustment of concentration (Section 8.2.12.4) and prepare a reagent blank (Section 11.2); the absorbance of this reagent blank at 540 nm should be less than 0.170 at 22° C. If the absorbance is greater than 0.170 under the conditions, further extractions should be performed.

8.2.12.4 *PRA assay procedure*—The concentration of pararosaniline hydrochloride (PRA) need be assayed only once after purification. It is also recommended that commercial solutions of pararosaniline be assayed when first purchased. The assay procedure is as follows: (*10*)

1. Prepare 1 M acetate-acetic acid buffer stock solution with a pH of 4.79 by dissolving 13.61 g of sodium acetate trihydrate in distilled water in a 100-mL volumetric flask. Add 5.70 mL of glacial acetic acid and dilute to volume with distilled water.

2. Pipet 1 mL of the stock PRA solution obtained from the purification process or from a commercial source into a 100-mL volumetric flask and dilute to volume with distilled water.

3. Transfer a 5-mL aliquot of the diluted PRA solution from 2. into a 50-mL volumetric flask. Add 5 mL of 1 M acetate-acetic acid buffer solution from 1. and dilute the mixture to volume with distilled water. Let the mixture stand for 1 hour.

4. Measure the absorbance of the above solution at 540 nm with a spectrophotometer against a distilled water reference. Compute the percentage of nominal concentration of PRA by

$$\%\ PRA = \frac{A \times K}{W} \quad (5)$$

where
A = measured absorbance of the final mixture (absorbance units);
W = weight in grams of the PRA dye used in the assay to prepare 50 mL of stock solution (for example, 0.100 g of dye was used to prepare 50 mL of solution in the purification procedure; when obtained from commercial sources, use the stated concentration to compute W; for 98% PRA, W = .098 g.); and
K = 21.3 for spectrophotometers having a spectral bandwidth of less than 15 nm and a path length of 1 cm.

8.2.13 *Pararosaniline reagent:* To a 250-mL volumetric flask, add 20 mL of stock PRA solution. Add an additional 0.2 mL of stock solution for each percentage that the stock assays below 100 percent. Then add 25 mL of 3 M phosphoric acid and dilute to volume with distilled water. The reagent is stable for at least 9 months. Store away from heat and light.

9.0 *Sampling Procedure.*

9.1 *General Considerations.* Procedures are described for short-term sampling (30-minute and 1-hour) and for long-term sampling (24-hour). Different combinations of absorbing reagent volume, sampling rate, and sampling time can be selected to meet special needs. For combinations other than those specifically described, the conditions must be adjusted so that linearity is maintained between absorbance and concentration over the dynamic range. Absorbing reagent volumes less than 10 mL are not recommended. The collection efficiency is above 98 percent for the conditions described; however, the efficiency may be substantially lower when sampling concentrations below 25 μg SO_2/m^3. (*8*) (*9*)

9.2 *30-Minute and 1-Hour Sampling.* Place 10 mL of TCM absorbing reagent in a midget impinger and seal the impinger with a thin film of silicon stopcock grease (around the ground glass joint). Insert the sealed impinger into the sampling train as shown in Figure. 1,

making sure that all connections between the various components are leak tight. Greaseless ball joint fittings, heat shrinkable Teflon® tubing, or Teflon® tube fittings may be used to attain leakfree conditions for portions of the sampling train that come into contact with air containing SO_2. Shield the absorbing reagent from direct sunlight by covering the impinger with aluminum foil or by enclosing the sampling train in a light-proof box. Calibrate the critical orifice or flowmeter according to Section 9.4.1. Collect the sample at 1 ±0.10 L/min for 30-minute sampling or 0.500 ±0.05 L/min for 1-hour sampling. Record the exact sampling time in minutes, as the sample volume will later be determined using the sampling flow rate and the sampling time. Record the atmospheric pressure and temperature.

9.3 *24-Hour Sampling.* Place 50 mL of TCM absorbing solution in a large absorber, close the cap, and apply the heat shrink sealant tape as shown in Figure 3. Make a mark on the absorber with a triangular file to indicate the starting volume of absorbing reagent. Insert the sealed absorber into the sampling train as shown in Figure 2. At this time verify that the absorber temperature is controlled to 15° ±10° C. During sampling, the absorber temperature must be controlled to prevent decomposition of the collected complex. From the onset of sampling unitl analysis, the absorbing solution must be protected from direct sunlight. Calibrate the critical orifice according to Section 9.4.1. Collect the sample for 24 hours from midnight to midnight at a flow rate of 0.200 ±0.020 L/min. A start/stop timer is helpful for initiating and stopping sampling and an elapsed time meter will be useful for determining the sampling time.

9.4 *Flow Measurement.*

9.4.1 *Calibration:* Calibrate all flow controllers (i.e., critical orifices) and flow measuring devices against a reliable flow or volume standard such as an NBS traceable bubble flowmeter or calibrated wet test meter. Flow controllers used in the sampling train must meet the flow rate specifications in 9.2 and 9.3. Flow controllers must be calibrated in the sampling train with an absorber solution in place.

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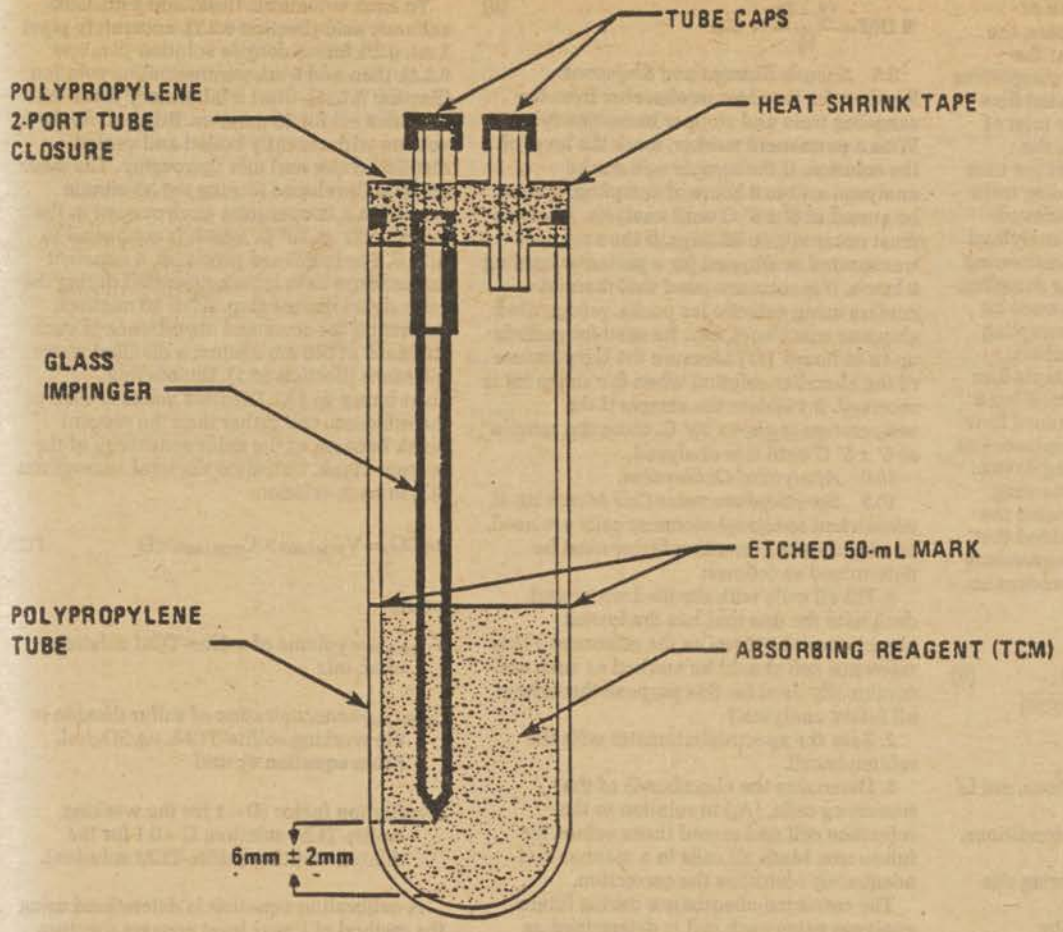

Figure 3. An absorber (24-hour sample) filled and assembled for shipment.

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9.4.2 *Determination of flow rate at sampling site:* For short-term samples, the standard flow rate is determined at the sampling site at the initiation and completion of sample collection with a calibrated flow measuring device connected to the inlet of the absorber. For 24-hour samples, the standard flow rate is determined at the time the absorber is placed in the sampling train and again when the absorber is removed from the train for shipment to the analytical laboratory with a calibrated flow measuring device connected to the inlet of the sampling train. The flow rate determination must be made with all components of the sampling system in operation (e.g., the absorber temperature controller and any sample box heaters must also be operating). Equation 6 may be used to determine the standard flow rate when a calibrated positive displacement meter is used as the flow measuring device. Other types of calibrated flow measuring devices may also be used to determine the flow rate at the sampling site provided that the user applies any appropriate corrections to devices for which output is dependent on temperature or pressure.

$$Q_{std}=Q_{act}\times\frac{P_b-P_{H2O}}{760}\times\frac{298.16}{(T_{meter}+273.16)} \quad (6)$$

where

Q_{std} = flow rate at standard conditions, std L/min (25° C and 760 mm Hg);

Q_{act} = flow rate at monitoring site conditions, L/min;

P_b = barometric pressure at monitoring site conditions, mm Hg;

P_{H2O} = vapor pressure of water at the temperature of the air in the flow or volume standard, mm Hg, (for wet volume standards only, i.e., bubble flowmeter or wet test meter; for dry standards, i.e., dry test meter, $P_{H2O}=0$); and

T_{meter} = temperature of the air in the flow or volume standard, °C (e.g., bubble flowmeter).

If a barometer is not available, the following equation may be used to determine the barometric pressure:

$$P_b=760-[.076(H)] \quad (7)$$

where

H = sampling site elevation above sea level in meters. If the initial flow rate (Q_i) differs from the flow rate of the critical orifice or the flow rate indicated by the flowmeter in the sampling train (Q_c) by more than 5 percent as determined by equation (8), check for leaks and redetermine Q_i.

$$\%\ Diff=\frac{Q_i-Q_c}{Q_c}\times 100 \quad (8)$$

Invalidate the sample if the difference between the initial (Q_i) and final (Q_f) flowrates is more than 5 percent as determined by equation (9)

$$\%\ Diff=\frac{Q_i-Q_f}{Q_f}\times 100 \quad (9)$$

9.5 *Sample Storage and Shipment.* Remove the impinger or absorber from the sampling train and stopper immediately. With a permanent marker, mark the level of the solution. If the sample will not be analyzed within 8 hours of sampling, it must be stored at 5°±5° C until analysis. Analysis must occur within 30 days. If the sample is transported or shipped for a period exceeding 8 hours, it is recommended that thermal coolers using eutectic ice packs, refrigerated shipping containers, etc., be used for periods up to 48 hours. (*17*) Measure the temperature of the absorber solution when the shipment is received. Invalidate the sample if the temperature is above 10° C. Store the sample at 5°±5° C until it is analyzed.

10.0 *Analytical Calibration.*

10.1 *Spectrophotometer Cell Matching.* If unmatched spectrophotometer cells are used, an absorbance correction factor must be determined as follows:

1. Fill all cells with distilled water and designate the one that has the lowest absorbance at 548 nm as the reference. (This reference cell should be marked as such and continually used for this purpose throughout all future analyses.)

2. Zero the spectrophotometer with the reference cell.

3. Determine the absorbance of the remaining cells, (A_c) in relation to the reference cell and record these values for future use. Mark all cells in a manner that adequately identifies the correction.

The corrected absorbance during future analyses using each cell is determined as follows:

$$A=A_{obs}-A_c \quad (10)$$

where

A = corrected absorbance,

A_{obs} = uncorrected absorbance, and

A_c = cell correction.

10.2 *Static Calibration Procedure (Option 1).* Prepare a dilute working sulfite-TCM solution by diluting 10 mL of the working sulfite-TCM solution (Section 8.2.11) to 100 mL with TCM absorbing reagent. Following the table below, accurately pipet the indicated volumes of the sulfite-TCM solutions into a series of 25-mL volumetric flasks. Add TCM absorbing reagent as indicated to bring the volume in each flask to 10 mL.

Sulfite-TCM solution	Volume of sulfite-TCM solution, mL	Volume of TCM, mL	Total μg SO_2 (approximately)[1]
Working	4.0	6.0	28.8
Working	3.0	7.0	21.6
Working	2.0	8.0	14.4
Dilute working	10.0	0.0	7.2
Dilute working	5.0	5.0	3.6
—	0.0	10.0	0.0

[1] Based on working sulfite-TCM solution concentration of 7.2 μg SO_2/mL; the actual total μg SO_2 must be calculated using equation 11 below.

To each volumetric flask, add 1 mL 0.6% sulfamic acid (Section 8.2.1), accurately pipet 2 mL 0.2% formaldehyde solution (Section 8.2.2), then add 5 mL pararosaniline solution (Section 8.2.13). Start a laboratory timer that has been set for 30 minutes. Bring all flasks to volume with recently boiled and cooled distilled water and mix thoroughly. The color must be developed (during the 30-minute period) in a temperature environment in the range of 20° to 30° C, which is controlled to ±1° C. For increased precision, a constant temperature bath is recommended during the color development step. After 30 minutes, determine the corrected absorbance of each standard at 548 nm against a distilled water reference (Section 10.1). Denote this absorbance as (A). Distilled water is used in the reference cell rather than the reagent blank because of the color sensitivity of the reagent blank. Calculate the total micrograms SO_2 in each solution:

$$\mu g\ SO_2=V_{TCM/SO2}\times C_{TCM/SO2}\times D \quad (11)$$

where

$V_{TCM/SO2}$ = volume of sulfite-TCM solution used, mL;

$C_{TCM/SO2}$ = concentration of sulfur dioxide in the working sulfite-TCM, μg SO_2/mL (from equation 4); and

D = dilution factor (D=1 for the working sulfite-TCM solution; D=0.1 for the diluted working sulfite-TCM solution).

A calibration equation is determined using the method of linear least squares (Section 12.1). The total micrograms SO_2 contained in each solution is the x variable, and the corrected absorbance associated with each solution is the y variable. For the calibration to be valid, the slope must be in the range of 0.030 ±0.002 absorbance unit/μg SO_2, the intercept as determined by the least squares method must be equal to or less than 0.170 when the color is developed at 22° C (add 0.015 to this 0.170 specification for each °C above 22° C) and the correlation coefficient must be greater than 0.998. If there criteria are not met, it may be the result of an impure dye and/or an improperly standardized sulfite-TCM solution. A calibration factor (B_s) is determined by calculating the reciprocal of the slope and is subsequently used for calculating the sample concentration (Section 12.3).

10.3. *Dynamic Calibration Procedures (Option 2).* Atmospheres containing accurately known concentrations of sulfur dioxide are prepared using permeation devices. In the systems for generating these atmospheres, the permeation device emits gaseous SO_2 at a known, low, constant rate, provided the termperature of the device is held constant (±0.1° C) and the device has been accurately calibrated at the temperature of use. The SO_2 permeating from the device is carried by a low flow of dry carrier gas to a

mixing chamber where it is diluted with SO_2-free air to the desired concentration and supplied to a vented manifold. A typical system is shown schematically in Figure 4 and this system and other similar systems have been described in detail by O'Keeffe and Ortman; (*19*) Scaringelli, Frey, and Saltzman; (*20*) and Scaringelli, O'Keeffe, Rosenberg, and Bell. (*21*) Permeation devices may be prepared or purchased and in both cases must be traceable either to a National Bureau of Standards (NBS) Standard Reference Material (SRM 1625, SRM 1626, SRM 1627) or to an NBS/EPA-approved commercially available Certified Reference Material (CRM). CRM's are described in Reference 22, and a list of CRM sources is available from the address shown for Reference 22. A recommended protocol for certifying a permeation device to an NBS SRM or CRM is given in Section 2.0.7 of Reference 2. Device permeation rates of 0.2 to 0.4 μg/min, inert gas flows of about 50 mL/min, and dilution air flow rates from 1.1 to 15 L/min conveniently yield standard atmospheres in the range of 25 to 600 μg SO_2/m^3 (0.010–0.230 ppm).

10.3.1 *Calibration option 2A (30-minutes and 1-hour samples):* Generate a series of six standard atmospheres of SO_2 (e.g., 0, 50, 100, 200, 350, 500, 750 μg/m^3) by adjusting the dilution flow rates appropriately. The concentration of SO_2 in each atmosphere is calculated as follows:

$$C_a = \frac{P_r - 10^3}{(Q_d + Q_p)} \quad (12)$$

where

C_a = concentration of SO_2 at standard conditions, μg/m^3;

P_r = permeation rate, μg/min;

Q_d = flow rate of dilution air, stdL/min; and

Q_p = flow rate of carrier gas across permeation device, stdL/min.

Be sure that the total flow rate of the standard exceeds the flow demand of the sample train, with the excess flow vented at atmospheric pressure. Sample each atmosphere using similar apparatus as shown in Figure 1 and under the same conditions as field sampling (i.e., use same absorbing reagent volume and sample same volume of air at an equivalent flow rate). Due to the length of the sampling periods required, this method is not recommended for 24-hour sampling.

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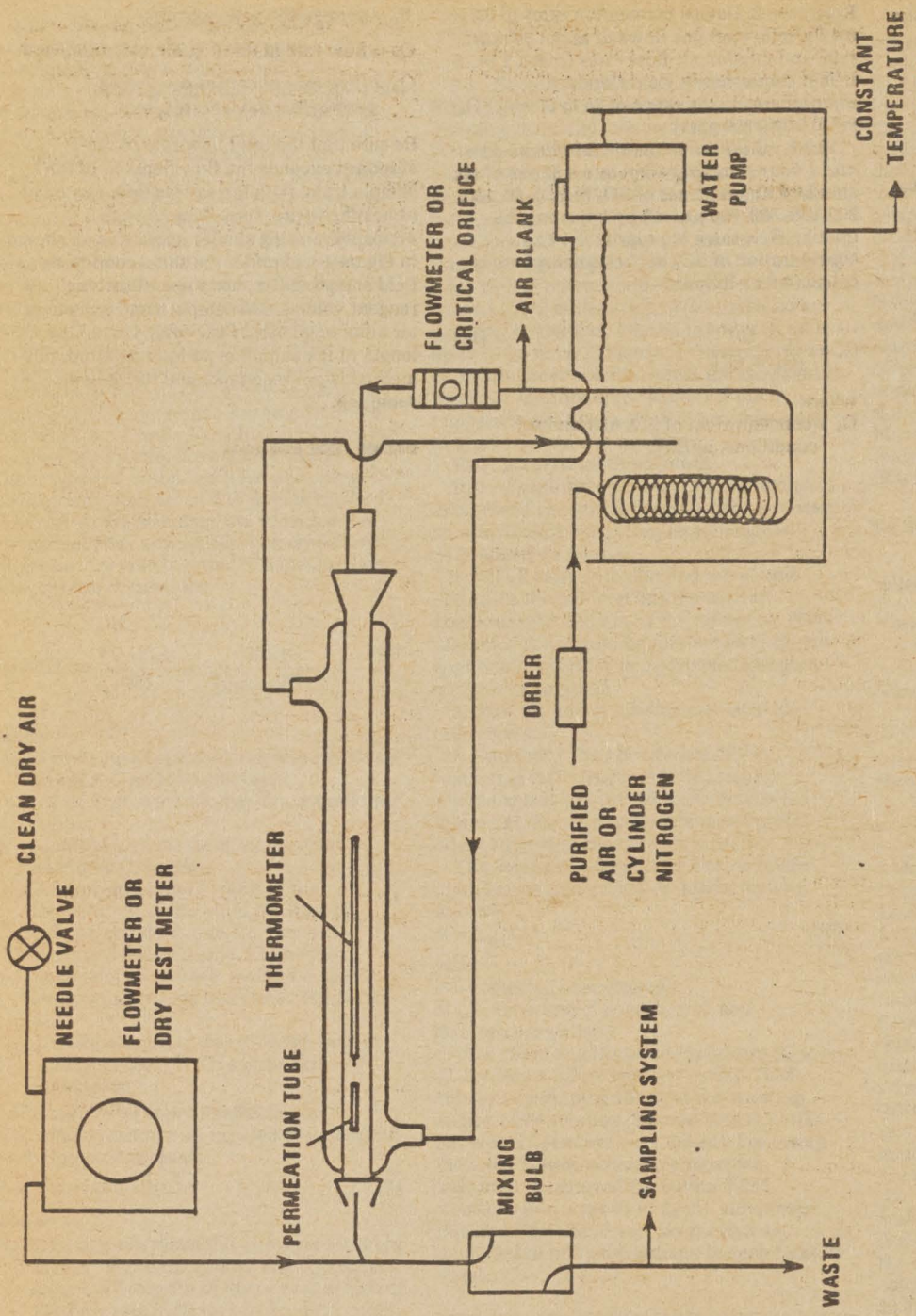

Figure 4. Permeation tube schematic for laboratory use.

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At the completion of sampling, quantitatively transfer the contents of each impinger to a series of 25-mL volumetric flasks (if 10 mL of absorbing solution was used) using small amounts of distilled water for rinse (<5 mL). If >10 mL of absorbing solution was used, bring the absorber solution in each impinger to orignial volume with distilled H_2O and pipet 10-mL portions from each impinger into a series of 25-mL volumetric flasks. If the color development steps are not started within 8 hours of sampling, store the solutions at 5° ±5° C. Calculate the total micrograms SO_2 in each solution as follows:

$$\mu g\ SO_2 = \frac{C_a \times Q_s \times t \times V_a}{V_b} \times 10^{-3} \quad (13)$$

Where:

C_a = concentration of SO_2 in the standard atmosphere, $\mu g/m^3$;

$Q_s = \frac{Q_i + Q_f}{2}$ = sampling flow rate, stdL/min;

t = sampling time, min;

V_a = volume of absorbing solution used for color development (10 mL); and

V_b = volume of absorbing solution used for sampling, mL.

Add the remaining reagents for color development in the same manner as in Section 10.2 for static solution. Calculate a calibration equation and a calibration factor (B_s) according to Section 10.2, adhering to all the specified criteria.

10.3.2 *Calibration option 2B (24-hour samples):* Generate a standard atmosphere containing approximately 1,050 $\mu g\ SO_2/m^3$ and calculate the exact concentration according to equation 12. Set up a series of six absorbers according to Figure 2 and connect to a common manifold for sampling the standard atmosphere. Be sure that the total flow rate of the standard exceeds the flow demand at the sample manifold, with the excess flow vented at atmospheric pressure. The absorbers are then allowed to sample the atmosphere for varying time periods to yield solutions containing 0, 0.2, 0.6, 1.0, 1.4, 1.8, and 2.2 $\mu g\ SO_2$/mL solution. The sampling times required to attain these solution concentrations are calculated as follows:

$$t = \frac{V_b \times C_s}{C_a \times Q_s \times 10^{-3}} \quad (14)$$

Where:

t = sampling time, min;

V_b = volume of absorbing solution used for sampling (50 mL);

C_s = desired concentration of SO_2 in the absorbing solution, $\mu g/mL$;

C_a = concentration of the standard atmosphere calculated according to equation 12, $\mu g/m^3$; and

Q_s = sampling flow rate, stdL/min.

At the completion of sampling, bring the absorber solutions to original volume with distilled water. Pipet 10-mL portion from each absorber into a series of 25-mL volumetric flasks. If the color development steps are not started within 8 hours of sampling, store the solutions at 5°±5° C. Add the remaining reagents for color development in the same manner as in Section 10.2 for static solutions. Calculate the total $\mu g\ SO_2$ in each standard as follows:

$$\mu g\ SO_2 = \frac{t \times C_a \times Q_s \times V_a}{V_b} \times 10^{-3} \quad (15)$$

Where:

V_a = volume of absorbing solution used for color development (10 mL).

All other parameters are defined in equation 14.

Calculate a calibration equation and a calibration factor (B_s) according to Section 10.2 adhering to all the specified critieria.

11.0 *Sample Preparation and Analysis.*

11.1 *Sample Preparation.* Remove the samples from the shipping container. If the shipment period exceeded 8 hours from the completion of sampling, verify that the temperature is below 10° C. Also, compare the solution level to the level marked on the absorber prior to shipping. If either the temperature is above 10° C or there was significant loss (more than 20%) of the sample during shipping, make an appropriate notation in the record and invalidate the sample. Prepare the samples for analysis as follows:

1. For 30-minute or 1-hour samples: Quantitatively transfer the entire 10 mL amount of absorbing solution to a 25-mL volumetric flask and rinse with a small amount (<5 mL) of distilled water.

2. For 24-hour samples: If the volume of the sample is less than the original volume marked on the absorber, adjust the volume back to the original volume with distilled water to compensate for water lost to evaporation during sampling. If the final volume is greater than the original volume, the volume must be measured using a graduated cylinder. To analyze, pipet 10 mL of the solution into a 25-mL volumetric flask.

11.2 *Sample Analysis.* For each set of determinations, prepare a reagent blank by adding 10 mL TCM absorbing solution to a 25-mL volumetric flask, and two control standards containing approximately 5 and 15 $\mu g\ SO_2$, respectively. The control standards are prepared according to Section 10.2 or 10.3. The analysis is carried out as follows:

1. Allow the sample to stand 20 minutes after the completion of sampling to allow any ozone to decompose (if applicable).

2. To each 25-mL volumetric flask containing reagent blank, sample, or control standard, add 1 mL of 0.6% sulfamic acid (Section 8.2.1) and allow to react for 10 min.

3. Accurately pipet 2 mL of 0.2% formaldehyde solution (Section 8.2.2) and then 5 mL of pararosaniline solution (Section 8.2.13) into each flask. Start a laboratory timer set at 30 minutes.

4. Bring each flask to volume with recently boiled and cooled distilled water and mix thoroughly.

5. During the 30 minutes, the solutions must be in a temperature-controlled environment in the range of 20° to 30° C maintained to ±1° C. This temperature must also be within 1° C of that used during calibration.

6. After 30 minutes and before 60 minutes, determine the corrected absorbances of each solution at 548 nm using 1-cm optical path length cells against a distilled water reference (Section 10.1). (*Distilled water is used as a reference instead of the reagent blank because of the color sensitivity of the reagent blank to temperature.*)

7. Do not allow the colored solution to stand in the cells because a film may be deposited. Clean the cells with isopropyl alcohol after use.

8. The reagent blank must be within 0.03 absorbance units of the intercept of the calibration equation determined in Section 10.

11.3 *Absorbance range.* If the absorbance of the sample solution ranges between 1.0 and 2.0, the sample can be diluted 1:1 with a portion of the reagent blank and the absorbance redetermined within 5 minutes. Solutions with higher absorbances can be diluted up to sixfold with the reagent blank in order to obtain scale readings of less than 1.0 absorbance unit. However, it is recommended that a smaller portion (<10 mL) of the original sample be reanalyzed (if possible) if the sample requires a dilution greater than 1:1.

11.4 *Reagent disposal.* All reagents containing mercury compounds must be stored and disposed of using one of the procedures contained in Section 13. Until disposal, the discarded solutions can be stored in closed glass containers and should be left in a fume hood.

12.0 *Calculations.*

12.1 *Calibration Slope, Intercept, and Correlation Coefficient.* The method of least squares is used to calculate a calibration equation in the form of:

$$y = mx + b \quad (16)$$

Where:

y = corrected absorbance,

m = slope, absorbance unit/$\mu g\ SO_2$,

x = micrograms of SO_2,

b = y intercept (absorbance units).

The slope (m), intercept (b), and correlation coefficient (r) are calculated as follows:

$$m = \frac{n\Sigma xy - (\Sigma x)(\Sigma y)}{n\Sigma x^2 - (\Sigma x)^2} \quad (17)$$

$$b = \frac{\Sigma y - m\Sigma x}{n} \quad (18)$$

$$r = \frac{n\Sigma y^2 - (\Sigma y)^2}{m} \quad (19)$$

where n is the number of calibration points.

A data form (Figure 5) is supplied for easily organizing calibration data when the slope, intercept, and correlation coefficient are calculated by hand.

12.2 *Total Sample Volume.* Determine the sampling volume at standard conditions as follows:

$$V_{std} = \frac{Q_i + Q_f}{2} \times t \quad (20)$$

Where:

V_{std} = sampling volume in stdL,
Q_i = standard flow rate determined at the initiation of sampling in stdL/min,
Q_f = standard flow rate determined at the completion of sampling in stdL/min, and
t = total sampling time, min.

12.3 *Sulfur Dioxide Concentration.* Calculate the concentration of each sample as follows:

$$\mu g\ SO_2/m^3 = \frac{(A-A_o)\ (B_x)\ (10^3)}{V_{std}} \times \frac{V_b}{V_a} \qquad (21)$$

Where:
A = corrected absorbance of the sample solution;
A_o = corrected absorbance of the reagent blank;
B_x = calibration factor equal to B_s, B_r, or B_t depending on the calibration procedure used, the reciprocal of the slope of the calibration equation.
V_a = volume of absorber solution analyzed, mL;
V_b = total volume of solution in absorber, mL; and
V_{std} = standard air volume sampled, stdL (from Section 12.2)

DATA FORM FOR HAND CALCULATIONS

Calibration point number	Micrograms SO_2 x	Absorbance units y	x^2	xy	y^2
1					
2					
3					
4					
5					
6					

Σx = ... Σy = ... Σx^2 = ... Σxy = ... Σy^2 = ...
n = (number of pairs of coordinates).

Figure 5. Data form for hand calculations.

12.4 *Control Standards.* Calculate the analyzed micrograms of SO_2 in each control standard as follows:

$$C_q = (A-A_o) \times B_x \qquad (22)$$

Where:
C_q = analyzed μg SO_2 in each control standard,
A = corrected absorbance of the control standard, and
A_o = corrected absorbance of the reagent blank.

The difference between the true and analyzed values of the control standards must not be greater than 1 μg. If the difference is greater than 1 μg, the source of the discrepancy must be identified and corrected and the samples must be reanalyzed.

12.5 *Conversion of $\mu g/m^3$ to ppm (v/v).* If desired, the concentration of sulfur dioxide at reference conditions is converted to ppm SO_2 (v/v) as follows:

$$\text{ppm } SO_2 = \frac{\mu g\ SO_2}{M_3} \times 3.82 \times 10^{-4} \qquad (23)$$

13.0 Disposal of Mercury-Containing Solutions.

13.1 The TCM absorbing solution and any reagents containing mercury compounds must be treated and disposed of by one of the methods discusses below. Both methods remove greater than 99.99 percent of the mercury.

13.2 *Method for Forming an Amalgam.*

(1) For each liter of waste solution, add approximately 10 g of sodium carbonate until neutralization has occurred (NaOH may have to be used).

(2) Following neutralization, add 10 g of granular zinc or magnesium.

(3) Stir the solution in a hood for 24 hours. Caution must be exercised as hydrogen gas is evolved by this treatment process.

(4) After 24 hours, allow the solution to stand without stirring to allow the mercury amalgam (solid black material) to settle to the bottom of the waste receptacle.

(5) Upon settling, decant and discard the supernatant liquid.

(6) Quantitatively transfer the solid material to a container and allow to dry.

(7) The solid material can be sent to a mercury reclaiming plant. It must not be discarded.

13.3 *Method Using Aluminum Strips.*

(1) Place the waste solution in an uncapped vessel in a hood.

(2) Add aluminum foil strips to the solution until the foil is no longer consumed and allow the gas to evolve for 24 hours.

(3) Decant the supernatant liquid and discard.

(4) Transfer the elemental mercury that has settled to the bottom of the vessel to a storage container.

14.0 *References for SO_2 Method.*

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2. By revising Appendix B to read as follows:

Appendix B.—Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High Volume Method)

1.0 *Applicability.* 1.1 This method provides a measurement of the mass concentration of total suspended particulate matter (TSP) in ambient air for determining compliance with the primary and secondary national ambient air quality standards for particulate matter as specified in § 50.6 and § 50.7 of this chapter. The measurement process is nondestructive, and the size of the sample collected is usually adequate for subsequent chemical analysis. Quality assurance procedures and guidance are provided in Part 58, Appendixes A and B, of this chapter and in References (*1*) and (*2*).

2.0 *Principle.* 2.1 An air sampler, properly located at the measurement site, draws a measured quantity of ambient air into a covered housing and through a filter during a 24-hr (nominal) sampling period. The sampler flow rate and the geometry of the shelter favor the collection of particles smaller than approximately 60 μm (aerodynamic diameter). The filters used are specified to have a minimum collection efficiency of 99 percent for 0.3 μm (DOP) particles at face velocities between 150 and 225 cm/sec (see Section 7.1.4).

2.2 The filter is weighed (after moisture equilibration) before and after use to determine the net weight (mass) gain. The total volume of air sampled, corrected to EPA standard conditions (25° C, 760 mm Hg), is determined from the measured flow rate and the sampling time. The concentration of total suspended particulate matter in the ambient air is computed as the mass of collected particles divided by the volume of air sampled, corrected to standard conditions, and is expressed in micrograms per standard cubic meter (μg/std m³). For samples collected at temperatures and pressures significantly different than standard conditions, these corrected concentrations may differ substantially from actual concentrations (in micrograms per actual cubic meter), particularly at high elevations. The actual particulate matter concentration can be calculated from the corrected concentration, using the actual temperature and pressure during the sampling period.

3.0 *Range.* 3.1 The approximate concentration range of the method is 2 to 750 μg/std m³. The upper limit is determined by the point at which the sampler can no longer maintain the specified flow rate due to the increased pressure drop of the loaded filter. This point is affected by particle size distribution, moisture content of the collected particles, and variability from filter to filter, among other things. The lower limit is determined by the sensitivity of the balance (see Section 7.10) and by inherent sources of error (see Section 6). 3.2 At wind speeds between 1.3 and 4.5 m/sec (3 and 10 mph), the high-volume air sampler has been found to collect an aerosol mass equal to the total aerosol mass fraction below about 60 μm³. For the filter specified in Section 7.1 there is effectively no lower limit on the particle size collected.

4.0 *Precision.* 4.1 Based upon collaborative testing, the relative standard deviation (coefficient of variation) for single analyst precision (repeatability) of the method is 3.0 percent. The corresponding value for interlaboratory precision (reproducibility) is 3.7 percent. (*4*)

5.0 *Accuracy.* 5.1 The absolute accuracy of the method is undefined because of the complex nature of atmospheric particulate matter and the difficulty in determining the "true" particulate matter concentration. This method provides a measure of particulate matter concentration suitable for the purpose specified under Section 1.0, Applicability.

6.0 *Inherent Sources of Error.*

6.1 *Airflow variation.* The weight of material collected on the filter represents the (integrated) sum of the product of the instantaneous flow rate times the instantaneous particle concentration. Therefore, dividing this weight by the average flow rate over the sampling period yields the true particulate matter concentration only when the flow rate is constant over the period. The error resulting from a nonconstant flow rate depends on the magnitude of the instantaneous changes in the flow rate and in the particulate matter concentration. Normally, such errors are not large, but they can be greatly reduced by equipping the sampler with an automatic flow controlling mechanism that maintains constant flow during the sampling period. Use of a constant flow controller is recommended.

6.2 *Air volume measurement.* If the flow rate changes substantially or nonuniformly during the sampling period, appreciable errors in the estimated air volume may result by averaging the presampling and postsampling flow rates. Greater air volume measurement accuracy may be achieved by (1) equipping the sampler with a flow controlling mechanism that maintains constant airflow during the sampling period, (2) using a calibrated, continous flow rate recording device to record the actual flow rate during the sampling period and integrating the flow rate over the period, or (3) any other means that will accurately measure the total air volume sampled during the sampling period. Use of a continuous flow recorder is recommended, particularly if the sampler is not equipped with a constant flow controller.

6.3 *Loss of volatiles.* Volatile particles collected on the filter may be lost during subsequent sampling or during shipment and/or storage of the filter prior to the postsampling weighing. (*5*) Although such losses are largely unavoidable, the filter should be reweighed as soon after sampling as practical.

6.4 *Artifact particulate matter.* Artifact particulate matter can be formed on the surface of alkaline glass fiber filters by oxidation of acid gases in the sample air, resulting in a higher than true TSP determination. (*6*) (*7*) This effect usually occurs early in the sample period and is a function of the filter pH and the presence of acid gases. It is generally believed to account for only a small percentage of the filter weight gain, but the effect may become more significant where relatively small particulate weights are collected.

6.5 *Humidity.* Glass fiber filters are comparatively insensitive to changes in relative humidity, but collected particulate matter can be hygroscopic. (*8*) The moisture conditioning procedure minimizes but may not completely eliminate error due to moisture.

6.6 *Filter handling.* Careful handling of the filter between the presampling and postsampling weighings is necessary to avoid errors due to loss of fibers or particles from the filter. A filter paper cartridge or cassette used to protect the filter can minimize handling errors. (See Reference (*2*), Section 2.)

6.7 *Nonsampled particulate matter.* Particulate matter may be deposited on the filter by wind action during periods when the sampler is inoperative. (*9*) It is recommended that errors from this source be minimized by an automatic mechanical device that keeps the filter covered during nonsampling periods, or by timely installation and retrieval of filters to minimize the nonsampling periods prior to the following operation.

6.8 *Timing errors.* Samplers are normally controlled by clock timers set to start and stop the sampler at selected times. Errors in the nominal 1,440-min sampling period may result from a power interruption during the sampling period or from a discrepancy between the start or stop time recorded on the filter information record and the actual start or stop time of the sampler. Such discrepancies may be caused by (1) poor resolution of the timer set-points, (2) timer error due to power interruption, (3) misetting of the timer, or (4) timer malfunction. In general, digital electronic timers have much better set-point resolution than mechanical timers, but require a battery backup system to maintain continuity of operation after a power interruption. A continuous flow recorder or elapsed timer provides an indication of the sampler run-time as well as an indication of any power interruption during the sampling period and is therefore recommended.

7.0 *Apparatus.*

(See References (*1*) and (*2*) for quality assurance information.)

Note.—Samplers purchased prior to the effective date of this amendment are not subject to specifications preceded by (*).

7.1 *Filter.* (Filters supplied by the Environmental Protection Agency can be assumed to meet the following criteria. Additional specifications are required if the sample is to be analyzed chemically.)

7.1.1 *Size:* 20.3±0.2×25.4±0.2 cm (nominal 8 x 10 in).

7.1.2 *Nominal exposed area:* 406.5 cm² (63 in²).

7.1.3 *Material:* Glass fiber or other relatively inert, nonhygroscopic material.[8]

7.1.4 *Collection efficiency:* 99 percent minimum as measured by the DOP test (ASTM–2986) for particles of 0.3 μm diameter at face velocities between 150 and 225 cm/sec.

7.1.5 *Maximum pressure drop:* 43 mm Hg (23 in. water) at a flow rate of 1.5 std m^3/min through nominal exposed area.

7.1.6 *pH:* 6 to 10. (*10*)

7.1.7 *Integrity:* 2.4 mg maximum weight loss. (*10*)

7.1.8 *Pinholes:* None.

7.1.9 *Tear strength:* 500 g minimum for 20 mm wide strip cut from filter in weakest dimension. (See ASTM Test D828–60.)

7.1.10 *Brittleness:* No cracks or material separations after single lengthwise crease.

7.2 *Sampler.* The air sampler shall provide means for drawing the air sample, via reduced atmospheric pressure, through the filter at a uniform face velocity.

7.2.1 The sampler shall have suitable means to:

a. Hold and seal the filter to the sampler housing.

b. Allow the filter to be changed conveniently.

c. Preclude leaks that would cause error in the measurement of the air volume passing through the filter.

d. * Adjust the flow rate to accommodate variations in line voltage and filter pressure drop. This may be accomplished by an automatic flow controller or by a manual flow adjustment device. Any manual adjustment device must be designed with positive detents or other means to avoid unintentional changes in the setting.*

7.2.2 A sampler equipped with a flow regulation mechanism must have the means to temporarily disable the flow controller to allow calibration of the flow indicator over the specified flow range.

7.2.3 *Minimum sample flow rate, heavily loaded filter:* 1.0 std m^3/min.

7.2.4 *Maximum sample flow rate, clean filter:* 1.5 std m^3/min.

7.2.5 *Blower Motor:* The motor must be capable of continuous operation for 24-hr periods.

7.3 *Sampler shelter.* 7.3.1 The sampler shelter shall:

a. Maintain the filter in a horizontal position at least 1 m above the floor or supporting surface so that sample air is drawn downward through the filter.

b. Be rectangular in shape with a gabled roof, similar to the design shown in Figure 1.

c. Cover and protect the filter and sampler from precipitation and other weather.

d. Discharge exhaust air at least 40 cm from the sample air inlet.

e. Be designed to minimize the collection of dust from the supporting surface by incorporating a baffle between the exhaust and the supporting surface.

7.3.2 The sampler cover or roof shall overhang the sampler housing somewhat, and shall be mounted so as to form an air inlet gap between the cover and the sampler housing walls. This sample air inlet should be approximately uniform on all sides of the sampler. The area of the sample air inlet must be sized to provide an effective particle capture air velocity of between 20 and 35 cm/sec at the recommended operational flow rate. The capture velocity is the sample air flow divided by the inlet area measured in a horizontal plane at the lower edge of the cover. Ideally, the inlet area and operational flow rate should be selected to obtain a capture air velocity of 23±2 cm/sec. (A flow rate of 1.1 m^3/min and an inlet area of about 800 cm^2 are recommended.)

7.3.3 Inlet openings of existing samplers that do not permit an inlet velocity within the range of 20 to 35 cm/sec at a flow rate as specified in 7.2.3 and 7.2.4 should be suitably modified to meet these specifications.

7.4 *Flow rate measurement device.* 7.4.1 The sampler shall incorporate a flow rate measurement device capable of indicating the total sampler flow rate. Two common types of flow indicators covered in the calibration procedure are (1) an electronic mass flowmeter and (2) an orifice or orifices located in the sample air stream (downstream of the filter) together with a suitable pressure indicator such as a manometer, or aneroid pressure gauge. A pressure recorder may be used with an orifice to provide a continuous record of the flow. Other types of flow indicators having comparable precision and accuracy are also acceptable.

7.4.2 *The flow rate measurement device must be capable of being calibrated and read in units corresponding to a flow rate which is readable to the nearest 0.02 std m^3/min over the range 0.9 to 1.6 std m^3/min.

Note.—Flow rate devices consisting of a rotameter (e.g., visi-float) connected to measure a portion of the sample flow may be used only until 1 year after the effective date of this amendment.

7.5 *Thermometer,* to indicate approximate air temperature at the flow rate measurement orifice, when temperature corrections are used.

7.5.1 *Range:* −40° to +50°C.

7.5.2 *Resolution:* 2°C.

7.6. *Barometer,* to indicate barometric pressure at the flow rate measurement orifice, when pressure corrections are used.

7.6.1 *Range:* 500 to 800 mm. Hg.

7.6.2 *Resolution:* ±5 mm. Hg.

7.7 *Timing/control device.*

7.7.1 The timing device must be capable of starting and stopping the sampler to obtain an elapsed run-time of 24 hr. ±1 hr. (1,440 ±60 min).

7.7.2 *Accuracy of time setting:* ±15 min., or better. (See Section 6.8.)

7.8. *Flow rate transfer standard,* traceable to a primary standard. (See Section 9.2.)

7.8.1 *Approximate range:* 0.9 to 1.6 std. m^3/min.

7.8.2 *Resolution:* 0.02 std. m^3/min.

7.8.3 *Reproducibility:* ±2 percent (2 times coefficient of variation) over normal ranges of ambient temperature and pressure for the stated flow rate range. (See Reference 2, Section 2.)

7.8.4 The flow rate transfer standard must connect without leaks to the inlet of the sampler and measure the flow rate of the total air sample.

7.8.5 The flow rate transfer standard must include a means to vary the sampler flow rate over the range 0.9 to 1.6 std m^3/min by introducing various levels of flow resistance between the sampler and the transfer standard inlet.

7.8.6 The conventional type of flow transfer standard consists of: an orifice unit with adapter that connects to the inlet of the sampler, a manometer or other device to measure orifice pressure drop, a means to vary the flow through the sampler unit, a thermometer to measure the ambient temperature, and a barometer to measure ambient pressure. Two such devices are shown in Figures 2a and 2b. Figure 2a shows fixed resistance plates, which necessitate disassembly of the unit each time the flow resistance is changed. A preferable design, illustrated in Figure 2b, has a variable flow restriction that can be adjusted externally without disassembly of the unit. Use of a conventional, orifice-type transfer standard is assumed in the calibration procedure (Section 9). However, the use of other types of transfer standards, such as the one shown in Figure 2c, meeting the above specifications may be approved; see the note following Section 9.1.

7.9 *Filter conditioning environment.*

7.9.1 *Controlled temperature:* between 15° and 30° C with less than ±3° C variation.

7.9.2 *Controlled humidity:* less than 50 percent relative humidity, constant within ±5 percent.

7.10 *Analytical balance.*

7.10.1 *Sensitivity:* 0.1 mg.

7.10.2 Weighing chamber designed to accept an unfolded 20.3x25.4 cm (8 x 10 in) filter.

7.11 *Area light source,* similar to x-ray film viewer, to backlight filters for visual inspection.

7.12 *Numbering device,* capable of printing identification numbers on the filters before they are placed in the filter conditioning environment.

8.0 *Procedure.* (See References (*1*) and (*2*) for quality assurance information.)

8.1 Number each filter near its edge with a unique identification number.

8.2 Backlight each filter and inspect for pinholes, particles, and other imperfections; filters with visible imperfections must not be used.

8.3 Equilibrate each filter in the conditioning environment for 24 hr.

8.4 Following equilibration, weigh each filter to the nearest milligram and record this tar weight (W_i) with the filter identification number.

8.5 Do not bend or fold the filter before collection of the sample.

8.6 Open the shelter and install a numbered, preweighed filter in the sampler, following the sampler manufacturer's instructions. During inclement weather, precautions must be taken while changing filters to prevent damage to the clean filter and loss of sample from or damage to the exposed filter. Filter cassettes loaded and unloaded in the laboratory may be used to minimize this problem. (See Section 6.6).

8.7 Close the shelter and run the sampler for at least 5 min to establish run-temperature conditions.

8.8 Record the flow indicator reading and, if needed, the barometric pressure and the ambient temperature (see Note following step

* See note at beginning of Section 7.

8.12). Stop the sampler. Determine the sampler flow rate (see Section 10.1); if it is outside the acceptable range (1.0 to 1.5 std M^3/min), use a different filter, or adjust the sampler flow rate. Warning: Manual flow adjustments may affect the calibration of the orifice-type flow indicators and may necessitate recalibration.

8.9 Record the sample information (filter number, site location or identification number, sample date, and starting time).

8.10 Set the timer to start and stop the sampler at appropriate times.

8.11 As soon as practical following the sampling period, run the sampler for at least 5 min to again establish run-temperature conditions.

8.12 Record the flow indicator reading and, if needed, the barometric pressure and the ambient temperature.

Note.—No onsite pressure or temperature measurements are necessary if the sampler flow indicator does not require pressure or temperature corrections (e.g., a mass flowmeter), or if average barometric pressure and seasonal average temperature for the site are incorporated into the sampler calibration (see step 9.3.9). For individual pressure and temperature corrections, the ambient pressure and temperature can be obtained by onsite measurements or from a nearby weather station. Barometric pressure readings obtained from airports must be station pressure, not corrected to sea level, and may need to be corrected for differences in elevation between the sampler size and the airport. For samplers having flow recorders but not constant flow controllers, the average temperature and pressure at the site *during the sampling period* should be estimated from weather bureau or other available data.

8.13 Stop the sampler and carefully remove the filter, following the sampler manufacturer's instructions. Touch only the outer edges of the filter.

8.14 Fold the filter in half lengthwise so that only surfaces with collected particulates are in contact, and place it in the filter holder (glassine envelope or manila folder).

8.15 Record the ending time or elapsed time on the filter information record, either from the stop set-point time, from an elapsed time indicator, or from a continuous flow record. The sample period must be 1,440 ± 60 min. for a valid sample.

8.16 Record on the filter information record any other factors, such as meteorological conditions, construction activity, fires or dust storms, etc., that might be pertinent to the measurement. If the sample is known to be defective, void it at this time.

8.17 Equilibrate the exposed filter in the conditioning environment for 24 hours.

8.18 Immediately after equilibration, reweigh the filter to the nearest milligram and record the gross weight with the filter identification number.

9.0 *Calibration.* 9.1 Calibration of the high volume sampler's flow indicating device is necessary to establish traceability of the field measurement to a primary standard via the flow rate transfer standard. Figure 3a illustrates the certification of the flow rate transfer standard and Figure 3b illustrates it's use in calibrating the sampler flow indicator. Determination of the corrected flow rate from the sampler flow indicator, illustrated in Figure 3c, is addressed in Section 10.1.

Note.—The following procedure assumes use of a conventional, orifice-type transfer standard. Other types of transfer standards may be used if the manufacturer or user provides an appropriately modified calibration procedure that has been approved by EPA under Section 2.8 of Appendix C to Part 58 of this chapter.

9.2 *Certification of the flow rate transfer standard.* (May be accomplished by either the user or the supplier.)

9.2.1 *Equipment required:* Positive displacement volume standard traceable to the National Bureau of Standards (such as a Roots meter or equivalent), stop-watch, manometer, thermometer, and barometer.

9.2.2 Connect the flow rate transfer standard to the inlet of the volume standard. Connect the manometer to measure the pressure at the inlet of the volume standard. Connect the orifice manometer to the pressure tap on the transfer standard. Connect a high-volume air pump (such as a high volume sampler blower) to the outlet side of the volume standard. See Figure 3a.

9.2.3 Check for leaks by temporarily clamping both manometer lines (to avoid fluid loss) and blocking the orifice with a large-diameter rubber stopper, wide cellophane tape, or other suitable means. Start the high-volume air pump and note any change in the volume standard reading. The reading should remain constant. If the reading changes, locate any leaks by listening for a whistling sound and/or retightening all connections, making sure that all gaskets are properly installed.

9.2.4 After satisfactorily completing the leak check as described above, unclamp both manometer lines and zero both manometers.

9.2.5 Achieve the appropriate flow rate through the system, either by means of the variable flow resistance in the transfer standard or by varying the voltage to the air pump. (Use of resistance plates as shown in Figure 1a is discouraged because the above leak check must be repeated each time a new resistance plate is installed.) A minimum of five different but constant flow rates, evenly distributed with at least three in the specified flow rate interval (1.0 to 1.5 std m^3/min), is required.

9.2.6 Measure and record the certification data on a form similar to the one illustrated in Figure 4 according to the following steps.

9.2.7 Observe the barometric pressure and record as P_1 (item 8 in Figure 4).

9.2.8 Read the ambient temperature in the vicinity of the standard volume meter and record it as T_1 (item 9 in Figure 4).

9.2.9 Start the blower motor, adjust the flow, and allow the system to run for at least 1 min for a constant motor speed to be attained.

9.2.10 Observe the standard volume meter reading and simultaneously start a stopwatch. Record the initial meter reading (V_i) in column 1 of Figure 4.

9.2.11 Maintain this constant flow rate until approximately 5 m^3 have passed through the standard volume meter. Record the standard volume inlet pressure manometer reading as ΔP (column 5 in Figure 4), and the orifice manometer reading as ΔH (column 7 in Figure 4). Be sure to indicate the correct units of measurement.

9.2.12 After at least 5 m^3 of air have passed through the system, observe the standard volume meter reading while simultaneously stopping the stop-watch. Record the final meter reading (V_f) in column 2 and the elapsed time (t) in column 3 of Figure 4.

9.2.13 Calculate the volume measured by the standard volume meter at meter conditions of temperature and pressures as $V_m = V_f - V_i$. Record in column 4 of Figure 4.

9.2.14 Correct this volume to standard volume (std m^3) as follows:

$$V_{std} = V_m \left(\frac{P_1 - \Delta P}{P_{std}}\right)\left(\frac{T_{std}}{T_1}\right) = V_m \left(\frac{P_1 - \Delta P}{760}\right)\left(\frac{298}{T_1}\right)$$

where

V_{std} = standard volume, std m^3;
P_1 = barometric pressure during calibration, mm Hg;
ΔP = differential pressure at inlet to volume meter, mm Hg;
P_{std} = 760 mm Hg;
T_{std} = 298 K;
T_1 = ambient temperature during calibration, K.

Calculate the standard flow rate (std m^3/min) as follows:

$$Q_{std} = \frac{V_{std}}{t}$$

where

Q_{std} = standard volumetric flow rate, std m^3/min.
t = elapsed time, minutes.

Record Q_{std} to the nearest 0.01 std m^3/min in column 6 of Figure 4.

9.2.15 Repeat steps 9.2.9 through 9.2.14 for at least four additional constant flow rates, evenly spaced over the approximate range of 0.9 to 1.6 std m^3/min.

9.2.16 Plot $\Delta H(P_1/760)(298/T_1)$ against Q_{std} or, to obtain a linear curve, plot $\sqrt{\text{Plot } \Delta H(P_1/760)(298/T_1)}$ against Q_{std} as shown in Figure 3a. Draw the orifice transfer standard certification curve or calculate the linear least squares slope and intercept of the certification curve. A certification graph should be readable to 0.02 std m^3.

9.2.17 Recalibrate the transfer standard annually or as required by applicable quality control procedures. (See Reference 2.)

9.3 *Calibration of sampler flow indicator.*

Note.—For samplers equipped with a flow controlling device, the flow controller must be disabled to allow flow changes during calibration of the sampler's flow indicator. For samplers using an orifice-type flow indicator downstream of the motor, do not vary the flow rate by adjusting the voltage or power supplied to the sampler.

9.3.1 A form similar to the one illustrated in Figure 5 should be used to record the calibration data.

9.3.2 Install a clean filter on the sampler and connect the transfer standard to the inlet of the sampler over the filter. Connect the

orifice manometer to the orifice pressure tap, as illustrated in Figure 3b. Make sure there are no leaks between the orifice unit and the sampler.

9.3.3 Operate the sampler for at least 5 minutes to establish thermal equilibrium prior to the calibration.

9.3.4 Measure and record the ambient temperature, T_a, and the barometric pressure, P_a, during calibration.

9.3.5 Adjust the variable resistance or, if applicable, insert the appropriate resistance plate (or no plate) to achieve the desired flow rate.

9.3.6 Let the sampler run for at least 2 min to establish run-temperature conditions. Read and record the pressure drop across the orifice (ΔH) and the sampler flow rate indication (I) in the appropriate columns of Figure 5.

9.3.7 Calculate $\sqrt{\Delta H(P_a/760)(298/T_a)}$ or (whichever form was used in step 9.2.16) and determine the flow rate at standard conditions Q_{std} either graphically from the certification curve or by calculating Q_{std} from the least squares slope and intercept of the transfer standard's certification curve. Record the value of Q_{std}.

9.3.8 Repeat steps 9.3.5, 9.3.6, and 9.3.7 for several additional flow rates distributed over the range of 1.0 to 1.5 std m^3/min.

9.3.9 Determine the calibration curve by plotting values of the appropriate expression involving I against Q_{std} (Table 1). The choice of expression depends on the flow rate measurement device used (see Section 7.4.1) and also on whether the calibration curve incorporates geographic average barometric pressure (P_a) and seasonal average temperature (T_a) for the site to approximate actual pressure and temperature. For many sites, using P_a and T_a is sufficiently accurate and avoids the need for subsequent pressure and temperature calculation when the sampler is used. The geographic average barometric pressure (P_a) may be obtained from an altitude-pressure table or by making an (approximate) elevation correction of −26 mm Hg. for each 305 m (1,000 ft) above sea level (760 mm Hg). The seasonal average temperature (T_a) may be obtained from weather station or other records.

9.3.10 Draw the sampler calibration curve or calculate the linear least squares slope, intercept, and correlation coefficient of the calibration curve. Calibration curves should be readable to 0.02 std m^3/min.

9.3.11 For a sampler equipped with a flow controller, the flow controlling mechanism should be re-enabled and the sample flow rate should be verified at this time with a clean filter installed. Then add two or more filters to the sampler to see if the flow controller maintains a constant flow.

10.0 *Calculation of TSP Concentration.*

10.1 Determine the average sampler flow rate during the sampling period according to either 10.1.1 or 10.1.2 below.

10.1.1 For a sampler without a continuous flow recorder, determine the appropriate expression to be used from Table 2. (The expression will correspond to the one from Table 1 used in step 9.3.9.) Using the appropriate expression, determine Q_{std} for the initial flow rate from the sampler calibration curve, either graphically or from the regression equation. Similarly, determine Q_{std} from the final flow reading, and calculate the average flow Q_{std} as one-half the sum of the initial and final flow rates.

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TABLE 1. EXPRESSIONS FOR PLOTTING SAMPLER CALIBRATION CURVES

Sampler flow rate measuring device	Expression	
	For actual pressure and temperature corrections	For incorporation of geographic average pressure and seasonal average temperature
Mass flowmeter	I	I
Orifice, nonlinear curve	$I\left(\frac{P_2}{760}\right)\left(\frac{298}{T_2}\right)$	$I\left(\frac{P_2}{P_a}\right)\left(\frac{T_a}{T_2}\right)$
Orifice, linear curve	$\sqrt{I\left(\frac{P_2}{760}\right)\left(\frac{298}{T_2}\right)}$	$\sqrt{I\left(\frac{P_2}{P_a}\right)\left(\frac{T_a}{T_2}\right)}$
Pressure recorder having square root scale	$I\sqrt{\left(\frac{P_2}{760}\right)\left(\frac{298}{T_2}\right)}$	$I\sqrt{\left(\frac{P_2}{P_a}\right)\left(\frac{T_a}{T_2}\right)}$

TABLE 2. EXPRESSIONS FOR DETERMINING FLOW RATE DURING SAMPLER OPERATION

Sampler flow rate measuring device	Expression	
	For actual pressure and temperature corrections	For use when geographic average pressure and seasonal average temperature have been incorporated into the sampler calibration
Mass flowmeter	I	I
Orifice	$I\left(\frac{P_3}{760}\right)\left(\frac{298}{T_3}\right)$	I
Orifice, linear curve	$\sqrt{I\left(\frac{P_3}{760}\right)\left(\frac{298}{T_3}\right)}$	$\sqrt{I}$
Pressure recorder having square root scale	$I\sqrt{\left(\frac{P_3}{760}\right)\left(\frac{298}{T_3}\right)}$	I

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10.1.2 For a sampler with a continuous flow recorder, determine the average flow rate device reading I for the period. Determine the appropriate expression from Table 2. (The expression will correspond to the one from Table 1 used in step 9.3.9.) Then using this expression and the average flow rate reading, determine $\overline{Q_{std}}$ from the sampler calibration curve, either graphically or from the regression equation. (If the trace shows substantial flow change during the sampling period, greater accuracy may be achieved by dividing the sampling period into intervals and averaging the individual interval flow rates to find $\overline{Q_{std}}$.

10.2 Calculate the total air volume sampled as:

$$V = \overline{Q_{std}} \times t$$

where

V = total air volume sampled, in standard volume units, std m^3;

$\overline{Q_{std}}$ = average standard flow rate, std m^3/min;

t = sampling time, min.

10.3 Calculate the particulate matter concentration as:

$$TSP = \frac{(W_f - W_i) \times 10^6}{V}$$

where

TSP = mass concentration of total suspended particulate matter, μg/std m^3;

W_i = initial weight of clean filter, g;

W_f = final weight of exposed filter, g;

V = air volume sampled, converted to standard conditions, std m^3;

10^6 = conversion of g to μg.

10.4 If desired, the actual particulate matter concentration (see Section 2.2) can be calculated as follows:

$$(TSP)_a = TSP\ (P_a/760)(298/T_a)$$

where

$(TSP)_a$ = actual concentration at field conditions, $\mu g/m^3$;

TSP = concentration at standard conditions, $\mu g/m^3$;

P_a = average barometric pressure during sampling period, mm Hg;

T_a = average ambient temperature during sampling period, K.

11.0 *References.*

(*1*) Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I, Principles. EPA–600/9–76–005, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, 1976.

(*2*) Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Ambient Air Specific Methods. EPA–600/4–77–027a, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, 1977.

(*3*) Lundgren, D. A., and H. J. Paulus. The Mass Distribution of Large Atmospheric Particles. J. Air Poll. Cont. Assoc., Vol. 25 (1227), 1975.

(*4*) McKee, H. C., et al. Collaborative Testing of Methods to Measure Air Pollutants, I. The High-Volume Method for Suspended Particulate Matter. J. Air Poll. Cont. Assoc., 22 (342), 1972.

(*5*) Clement, R. E., and F. W. Karasek. Sample Composition Changes in Sampling and Analysis of Organic Compounds in Aerosols. The Intern J. Environ. Anal. Chem., 7:109, 1979.

(*6*) Lee, R. E., Jr., and J. Wagman. A Sampling Anomaly in the Determination of Atmospheric Sulfuric Concentration. Am. Ind. Hygiene Assoc. J., 27:266, 1966.

(*7*) Appel, B. R., et al. Interference Effects in Sampling Particulate Nitrate in Ambient Air. Atmospheric Environment, 13:319, 1979.

(*8*) Tierney, G. P., and W. D. Conner. Hygroscopic Effects on Weight Determinations of Particulates Collected on Glass-Fiber Filters. Am. Ind. Hygiene Assoc. J., 28:363, 1967.

(*9*) Chahal, H. S., and D. J. Romano. High-Volume Sampling Effect of Windborne Particulate Matter Deposited During Idle Periods. J. Air Poll. Cont. Assoc., Vol. 26 (885), 1976.

(*10*) EPA Test Procedures for Determining pH and Integrity of High-Volume Air Filters. QAD/M–80.01. Available from the Methods Standardization Branch, Quality Assurance Division, Environmental Monitoring Systems Laboratory (MD–77), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, 1980.

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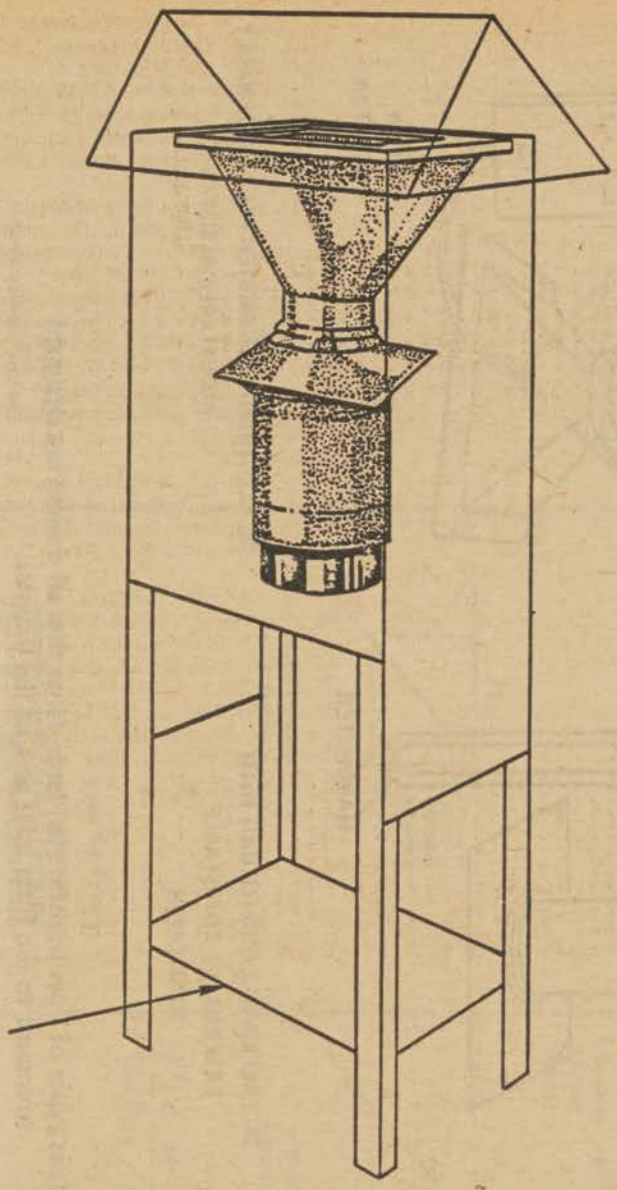
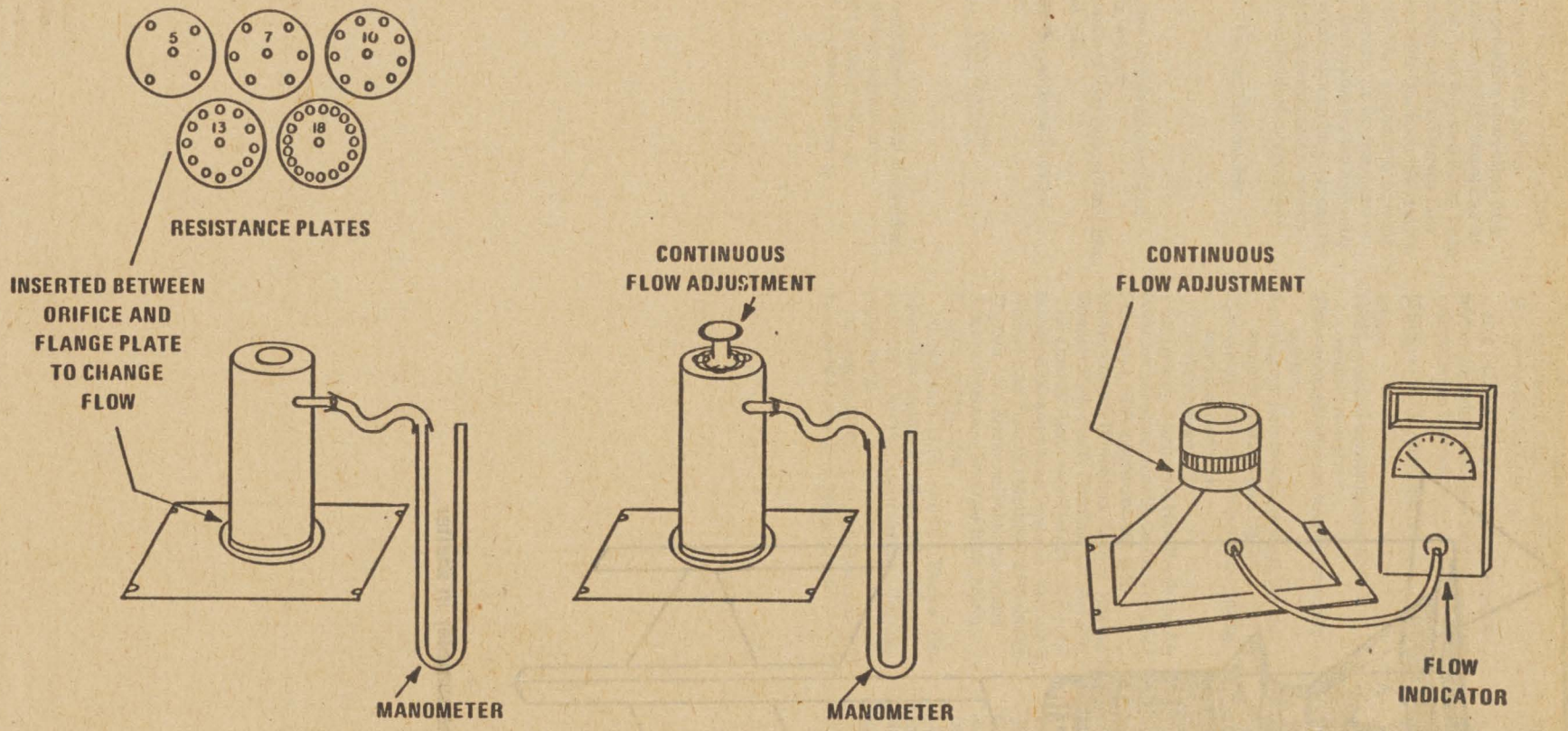


Figure 1. High-volume sampler in shelter.

ORIFICE TYPE FLOW TRANSFER STANDARDS

NONORIFICE TYPE FLOW TRANSFER STANDARD



2a. ORIFICE UNIT USING FIXED RESISTANCE PLATES.

2b. PREFERABLE ORIFICE UNIT WITH EXTERNALLY ADJUSTABLE RESISTANCE.

2c. ELECTRONIC FLOWMETER WITH EXTERNALLY ADJUSTABLE RESISTANCE.

Figure 2. Various types of flow transfer standards. Note that all devices are designed to mount to the filter inlet area of the sampler.

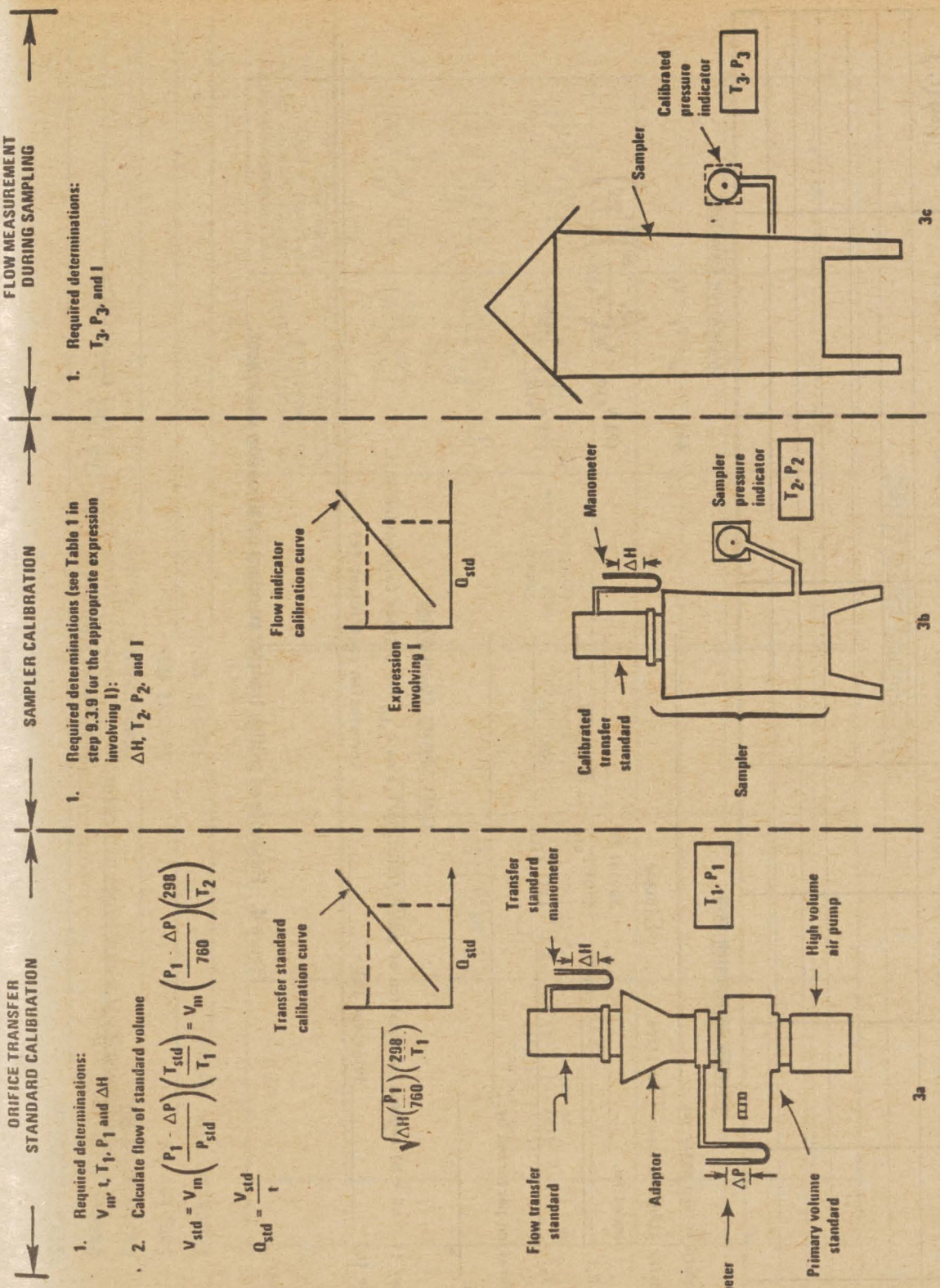


Figure 3. Illustration of the 3 steps in the flow measurement process.

ORIFICE TRANSFER STANDARD CERTIFICATION WORKSHEET

Run No.	(1) Meter reading start V_i (m^3)	(2) Meter reading stop V_f (m^3)	(3) Sampling time t (min)	(4) Volume measured V_m (m^3)	(5) Differential pressure (at inlet to volume meter) ΔP (mm Hg)	(6) Flow rate Q_{std} (std m^3/min)	(7) Pressure drop across orifice ΔH ☐ (in) or ☐ (cm) of water	(7a) ☐ $\Delta H\left(\frac{P_1}{760}\right)\left(\frac{298}{T_1}\right)$ or ☐ $\sqrt{\Delta H\left(\frac{P_1}{760}\right)\left(\frac{298}{T_1}\right)}$
1								
2								
3								
4								
5								
6								

RECORDED CALIBRATION DATA

Standard volume meter no. _____

Transfer standard type: ☐ orifice ☐ other

Model No. _____ Serial No. _____

(8) P_1: _____ mm Hg (10) P_{std}: 760 mm Hg

(9) T_1: _____ K (11) T_{std}: 298 K

Calibration performed by: _____

Date: _____

CALCULATION EQUATIONS

(1) $V_m = V_f - V_i$

(2) $V_{std} = V_m\left(\frac{P_1 - \Delta P}{P_{std}}\right)\left(\frac{T_{std}}{T_1}\right)$

(3) $Q_{std} = \frac{V_{std}}{t}$

LEAST SQUARES CALCULATIONS

Linear ($Y = mX + b$) regression equation of $\sqrt{\Delta H(P_1/760)(298/T_1)}$ on Q_{std} for Orifice Calibration Unit.

Slope (m) = _____ Intercept (b) = _____ Correlation coefficient (r) = _____

Figure 4. Example of orifice transfer standard certification worksheet.

HIGH-VOLUME AIR SAMPLER CALIBRATION WORKSHEET

Site Location: _____

Date: _____ Barometric Pressure, P_2 (mm Hg) _____

Calibrated By: _____ Temperature, T_2 (K) _____

Sampler No. _____ Serial No. _____

Transfer std. type: _____ Serial No. _____

No.	ΔH Pressure drop across orifice ☐(in) or ☐(cm) of water	☐ $\Delta H\left(\frac{P_2}{760}\right)\left(\frac{298}{T_2}\right)$ or ☐ $\sqrt{\Delta H\left(\frac{P_2}{760}\right)\left(\frac{298}{T_2}\right)}$	Q_{std} (from orifice certification) std m^3/min	I Sample flow rate indication (arbitrary)	For specific pressure and temperature corrections: ☐ I; ☐ $I\left(\frac{P_2}{760}\right)\left(\frac{298}{T_2}\right)$; or ☐ $I\sqrt{\left(\frac{P_2}{760}\right)\left(\frac{298}{T_2}\right)}$	For incorporation of average pressure and seasonal average temperature: ☐ I; ☐ $I\left(\frac{P_2}{\bar{P}_a}\right)\left(\frac{T_a}{T_2}\right)$; or ☐ $I\sqrt{\left(\frac{P_2}{\bar{P}_a}\right)\left(\frac{T_a}{T_2}\right)}$
1						
2						
3						
4						
5						
6						

LEAST SQUARES CALCULATIONS

Linear regression of Y on X: $Y = mX + b$

Slope (m) = _____ Intercept (b) = _____ Correlation Coeff. (r) = _____

$X = Q_{std}$, Y = Expression above (see Table 1 in step 9.3.9)

Figure 5. Example of high-volume air sampler calibration worksheet.

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3. By revising Appendix C to read as follows:

Appendix C—Measurement Principle and Calibration Procedure for the Measurement of Carbon Monoxide in the Atmosphere (Non-Dispersive Infrared Photometry)

Measurement Principle

1. Measurements are based on the absorption of infrared radiation by carbon monoxide (CO) in a non-dispersive photometer. Infrared energy from a source is passed through a cell containing the gas sample to be analyzed, and the quantitative absorption of energy by CO in the sample cell is measured by a suitable detector. The photometer is sensitized by CO by employing CO gas in either the detector or in a filter cell in the optical path, thereby limiting the measured absorption to one or more of the characteristic wavelengths at which CO strongly absorbs. Optical filters or other means may also be used to limit sensitivity of the photometer to a narrow band of interest. Various schemes may be used to provide a suitable zero reference for the photometer. The measured absorption is converted to an electrical output signal, which is related to the concentration of CO in the measurement cell.

2. An analyzer based on this principle will be considered a reference method only if it has been designated as a reference method in accordance with Part 53 of this chapter.

3. *Sampling considerations.* The use of a particle filter on the sample inlet line of an NDIR CO analyzer is optional and left to the discretion of the user or the manufacturer. Use of the filter should depend on the analyzer's susceptibility to interference, malfunction, or damage due to particles.

Calibration Procedure

1. *Principle.* Either of two methods may be used for dynamic multipoint calibration of CO analyzers: (1) One method uses a single certified standard cylinder of CO, diluted as necessary with zero air, to obtain the various calibration concentrations needed. (2) The other method uses individual certified standard cylinders of CO for each concentration needed. Additional information on calibration may be found in Section 2.0.9 of Reference (*1*).

2. *Apparatus.* The major components and typical configurations of the calibration systems for the two calibration methods are shown in Figures 1 and 2.

2.1 *Flow controller(s).* Device capable of adjusting and regulating flow rates. Flow rates for the dilution method (Figure 1) must be regulated to ± 1%.

2.2 *Flow meter(s).* Calibrated flow meter capable of measuring and monitoring flow rates. Flow rates for the dilution method (Figure 1) must be measured with an accuracy of ± 2% of the measured value.

2.3 *Pressure regulator(s) for standard CO cylinder(s).* Regulator must have nonreactive diaphragm and internal parts and a suitable delivery pressure.

2.4 *Mixing chamber.* A chamber constructed of glass, Teflon®, or other nonreactive material and designed to provide through mixing of CO and diluent air for the dilution method.

2.5 *Output manifold.* The output manifold should be constructed of glass, Teflon, ® or other nonreactive material and should be of sufficient diameter to insure an insignificant pressuer drop at the analyzer connection. The system must have a vent designed to insure atmospheric pressure at the manifold and to prevent ambient air from entering the manifold.

3. *Reagents.*

3.1 *CO concentration standard(s).* Cylinder(s) of CO in air containing appropriate concentration(s) of CO suitable for the selected operating range of the analyzer under calibration; CO standards for the dilution method may be contained in a nitrogen matrix if the zero air dilution ratio is not less than 100:1. The assay of the cylinder(s) must be traceable either to a National Bureau of Standards (NBS) CO in air Standard Reference Material (SRM) or to an NBS/EPA-approved commercially available Certified Reference Material (CRM). CRM's are described in Reference (*2*), and a list of CRM sources is available from the address shown for Reference (*2*). A recommended protocol for certifying CO gas cylinders against either a CO SRM or a CRM is given in Reference 1. CO gas cylinders should be recertified on a regular basis as determined by the local quality control program.

3.2 *Dilution gas (zero air).* Air, free of contaminants which will cause a detectable response on the CO analyzer. The zero air should contain <0.1 ppm CO. A precedure for generating zero air is given in Reference (*1*).

4. *Procedure Using Dynamic Dilution Method. 4.1* Assemble a dynamic calibration system such as the one shown in Figure 1. All calibration gases including zero air must be introduced into the sample inlet of the analyzer system. For specific operating instructions refer to the manufacturer's manual.

4.2 Insure that all flowmeters are properly calibrated, under the conditions of use, if appropriate, against an authoritative standard such as a soap-bubble meter or wet-test meter. All volumetric flowrates should be corrected to 25° C and 760 mm Hg. A discussion on calibration of flowmeters is given in Reference (*1*).

4.3 Select the operating range of the CO analyzer to be calibrated.

4.4 Connect the signal output of the CO analyzer to the input of the strip chart recorder or other data collection device. All adjustments to the analyzer should be based on the appropriate strip chart or data device readings. References to analyzer responses in the procedure given below refer to recorder or data device responses.

4.5 Adjust the calibration system to deliver zero air to the output manifold. The total air flow must exceed the total demand of the analyzer(s) connected to the output manifold to insure that no ambient air in pulled into the manifold vent. Allow the analyzer to sample zero air until a stable response is obtained. After the response has stabilized, adjust the analyzer zero control. Offsetting the analyzer zero adjustments to +5 percent of scale is recommended to facilitate observing negative zero drift. Record the stable zero air response as Z_{co}.

4.6 Adjust the zero air flow and the CO from the standard CO cylinder to provide a diluted CO concentration of approximately 80 percent of the upper range limit (URL) of the operating range of the analyzer. The total air flow must exceed the total demand of the analyzer(s) connected to the output manifold to insure that no ambient air is pulled into the manifold vent. The exact CO concentration is calculated from:

$$[CO]_{OUT} = \frac{[CO]_{STD} \times F_{CO}}{F_D + F_{CO}} \quad (1)$$

Where:

$[CO]_{OUT}$ = diluted CO concentration at the output manifold, ppm

$[CO]_{STD}$ = concentration of the undiluted CO standard, ppm

F_{CO} = flow rate of the CO standard corrected to 25° C and 760 mm Hg, 1/min

F_D = flow rate of the dilution air corrected to 25° C and 760 mm Hg, 1/min

Sample this CO concentration until a stable response is obtained. Adjust the analyzer span control to obtain a recorder response as indicated below:

$$\text{recorder response (percent scale)} = \frac{[CO]_{OUT}}{URL} \times 100 + X_{CO} \quad (2)$$

Where:

URL = nominal upper range limit of the analyzer's operating range

Z_{CO} = analyzer response to zero air, % scale

If substantial adjustment of the analyzer span control is necessary, it may be necessary to recheck the zero and span adjustments by repeating Steps 4.5 and 4.6. Record the CO concentration and the analyzer's response.

4.7 Generate several additional concentrations (at least three evenly spaced points across the remaining scale are suggested to verify linearity) by decreasing F_{CO} or increasing F_D. Be sure the total flow exceeds the analyzer's total flow demand. For each concentration generated, calculate the exact CO concentration using Equation (1). Record the concentration and the analyzer's response for each concentration. Plot the analyzer responses versus the corresponding CO concentrations and draw or calculate the calibration curve.

5. *Procedure Using Multiple Cylinder Method.* Use the procedure for the dynamic dilution method with the following changes:

5.1 Use a multi-cylinder system such as the typical one shown in Figure 2.

5.2 The flowmeter need not be accurately calibrated provided the flow in the output manifold exceeds the anlayzer's flow demand.

5.3 The various CO calibration concentrations required in Steps 4.6 and 4.7 are obtained without dilution by selecting the appropriate certified standard cylinder.

References

(*1*) Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II—

Ambient Air Specific Methods, EPA–600/4–77–027a, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina 27711, 1977.

(2) A Procedure for Establishing Traceability of Gas Mixtures to Certain National Bureau of Standards Standard Reference Materials. EPA–600/7–81–010, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory (MD–77), Research Triangle Park, North Carolina 27711, January 1981.

BILLING CODE 6560–35–M

[FR Doc. 82–1029 Filed 1–14–82; 8:45 am]

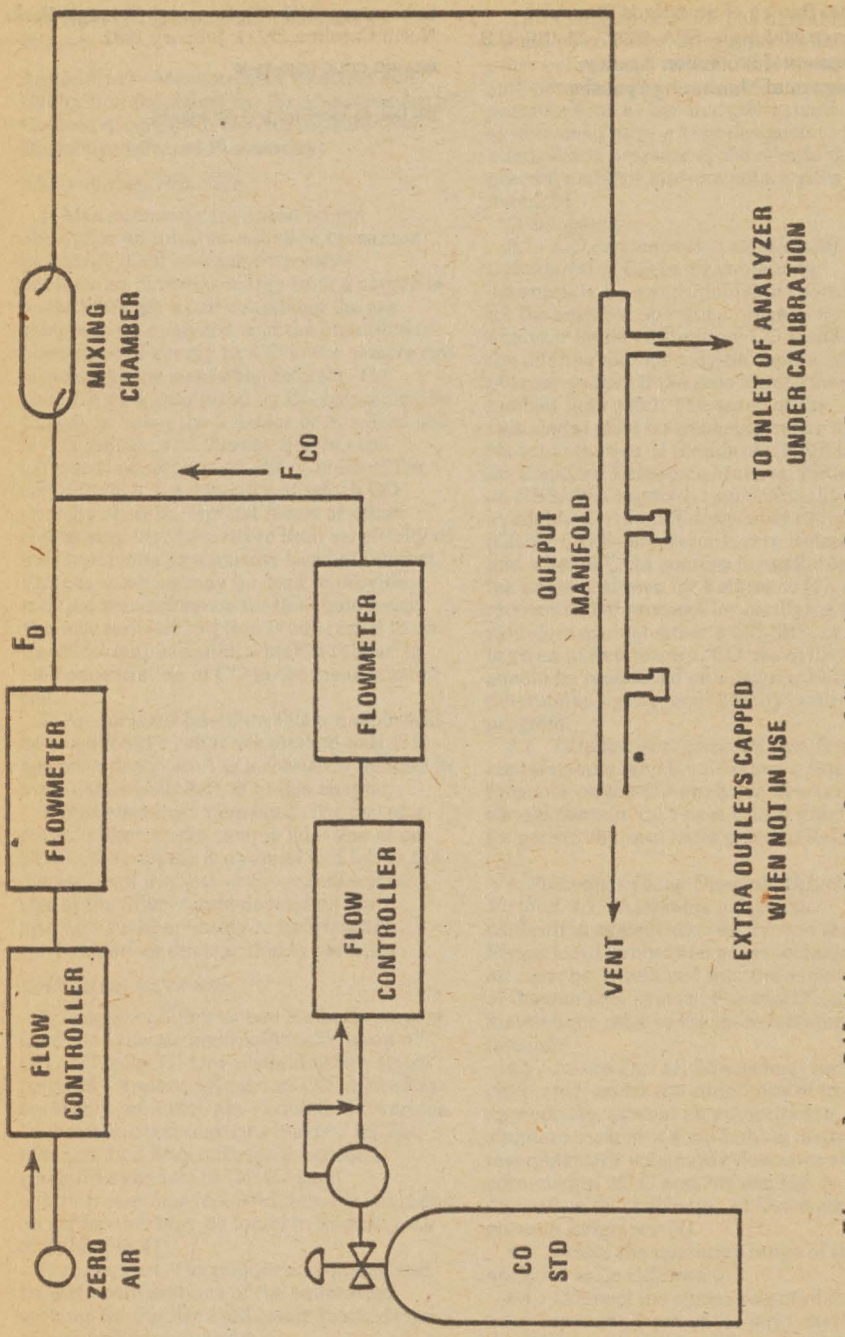

Figure 1. Dilution method for calibration of CO analyzers.

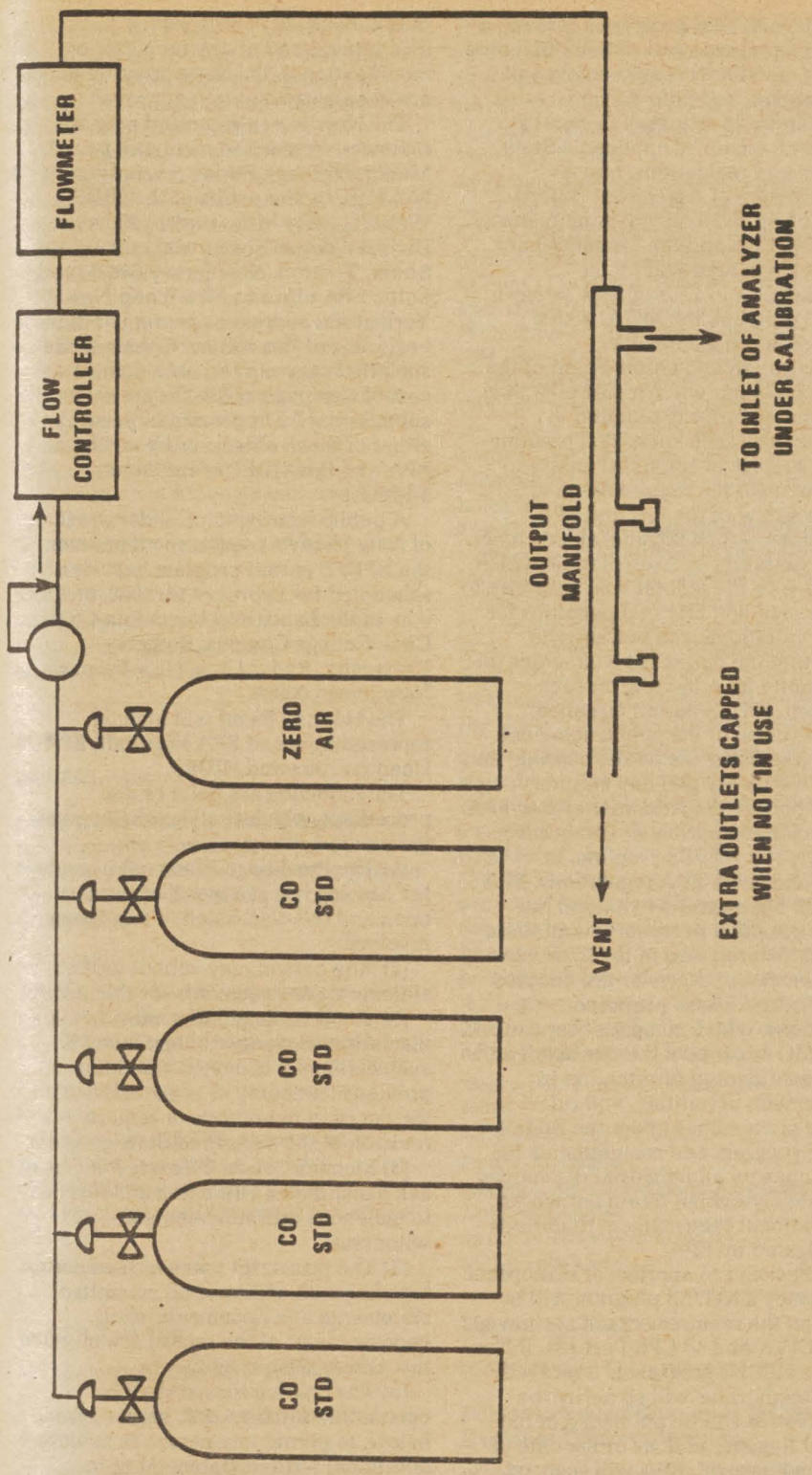

Figure 2. Multiple cylinder method for calibration of CO analyzers.

BILLING CODE 6560-35-C

40 CFR Part 123

[WE-2-FRL 2023-3]

New Jersey's Application to Administer the National Pollutant Discharge Elimination System (NPDES) Program

AGENCY: Environmental Protection Agency.

ACTION: Proposed rule; notice of application.

SUMMARY: The State of New Jersey has submitted a request to the Environmental Protection Agency for approval to administer the National Pollutant Discharge Elimination System (NPDES) program for regulating discharges of pollutants into waters within the State. According to the State's proposal, the NPDES program would be administered by the New Jersey Department of Environmental Protection (NJDEP) under the direction of Jerry Fitzgerald English, Commissioner. This notice provides for a public hearing and a comment period on New Jersey's request. Under EPA regulations, the Administrator shall approve or disapprove a State NPDES program after taking into consideration all comments received.

DATES: Comments must be received on or before March 1, 1982. A public hearing has been scheduled for February 16, 1982, at 10:00 a.m. at the Labor and Education Center, Cook College Campus, Rutgers University, Ryder Lane, New Brunswick, New Jersey 08903.

ADDRESSES: Comments should be addressed to Richard G. Tisch, Chief, Water Enforcement Branch, Enforcement Division, Region II, 26 Federal Plaza, New York, N.Y. 10278.

FOR FURTHER INFORMATION CONTACT: George Pavlou, Water Facilities Branch, New Jersey Management Division, U.S. EPA, 26 Federal Plaza, New York, N.Y. 10278, (212) 264–9878, or Richard Weinstein, Esq., Water Enforcement Branch, Enforcement Division, U.S. EPA, 26 Federal Plaza, New York, N.Y. 10278, (212) 264–4859.

SUPPLEMENTARY INFORMATION: Section 402 of the Federal Clean Water Act created the NPDES under which the Administrator of the United States Environmental Protection Agency (EPA) may issue permits for the discharge of pollutants into waters of the United States under conditions required by that Act.

New Jersey's program submission contains a letter from the Governor requesting NPDES program approval, a complete program description (including funding, personnel requirements and organization, and enforcement procedures), an Attorney General's statement, copies of applicable State statutes and regulations, and a Memorandum of Agreement (MOA) executed by the Regional Administrator, Region II, EPA and the Commissioner, NJDEP. Upon approval by the Administrator of EPA of New Jersey's NPDES program, the MOA, which establishes procedures for administration and enforcement of the State's program, will become effective. The Administrator is required to approve each such submitted program within 90 days of submittal unless it does not meet the requirements of section 402(b) of the Act and EPA regulations, which include, among other things, authority to issue permits which comply with the federal Act, authority to impose civil and criminal penalties for permit violations, and authority to insure that the public is given notice and opportunity for a hearing on each proposed NPDES permit issuance.

At the close of the public comment period (including the public hearing) and within the ninety (90) day statutory review period, the Administrator of EPA will decide to approve or disapprove New Jersey's NPDES program. In accordance with EPA regulations, EPA and DEP have agreed to extend the review period, if necessary, until State regulations proposed in the November 2, 1981 *New Jersey Register* are adopted and effective. These proposed regulations, which comprise Appendix C to the MOA, concern the treatment and use of confidential information in enforcement, permitting, and rule-making proceedings under the State's NPDES program and requirements for compliance by all industrial discharges into publicly-owned treatment works with National Pretreatment Standards promulgated by EPA.

The decision to approve or disapprove New Jersey's NPDES program will be based on the requirements of section 402 of the CWA and 40 CFR Part 123. If New Jersey's NPDES program is approved, the Administrator will so notify the State. Notice will be published in the **Federal Register** and, as of the date of program approval, EPA will suspend issuance of NPDES permits in New Jersey. The State's program will implement federal law and operate in lieu of the EPA administered program. However, EPA will retain the right to object to NPDES permits proposed to be issued by an approved State. If the Administrator disapproves New Jersey NPDES program, the Administrator will notify the State of reasons for disapproval and of any revisions or modification to the State program which are necessary to obtain approval.

The New Jersey submittal may be reviewed from 9:00 a.m. to 4:00 p.m., Monday through Friday, excluding holidays, by the public at the NJDEP, Water Quality Management Element, Division Water Resources, 1474 Prospect Street, Trenton, New Jersey 08625, and at the EPA office in New York, New York at the address appearing at the beginning of this Notice. Copies of the submittal may also be obtained (at a cost of 20¢/page or $70 for the entire submission) by appearing in person at either of those offices, or by writing to EPA and the NJDEP at the same addresses.

A public hearing to consider the State of New Jersey's request to administer the NPDES permit program has been scheduled for February 16, 1982, at 10:00 a.m. at the Labor and Education Center, Cook College Campus, Rutgers University, Ryder Lane, New Brunswick, New Jersey 08903.

The Hearing Panel will include representatives of EPA Region II, EPA Headquarters and NJDEP.

The following are policies and procedures which shall be observed at the public hearing:

(1) The Presiding Officer shall conduct the hearing in a manner that permits open and full discussion of any issues involved;

(2) Any person may submit written statements or documents for the record;

(3) The Presiding Office may, in his discretion, exclude oral testimony if such testimony is overly repetitious of previous testimony or is not relevant to the decision to approve or require revision of the submitted State program;

(4) Members of the Hearing Panel may ask questions of witnesses and respond to questions and statements of witnesses;

(5) The transcript taken at the hearing, together with copies of all submitted statements and documents, shall become a part of the record submitted to the Administrator; and

(6) The hearing record shall be left open until March 1, 1982, as described below, to permit any person to submit additional written statement or to present views or evidence tending to rebut testimony presented at the public hearing; immediately following the public comment period the Regional Administrator shall forward a copy of the complete hearing record to the Adminstrator.

Hearing statements may be oral or written. Written copies of oral

statements are urged for accuracy of the record and for the use of the hearing panel and other interested persons. Statements should summarize any extensive written materials.

All comments or objections received by EPA Region II by March 1, 1982, or presented at the public hearing will be considered by EPA before taking final action on the New Jersey Request for State Program Approval.

Please bring the foregoing to the attention of persons whom you know will be interested in this matter. All written comments and questions on the hearing or the NPDES program should be addressed to Richard G. Tisch, Esq., Chief, Water Enforcement Branch, Enforcement Division, Region II.

The Office of Management and Budget has exempted this rule from the requirements of section 3 of Executive Order 12291.

Richard T. Dewling,

Acting Regional Administrator, Region II.

[FR Doc. 82-288 Filed 1-14-82; 8:45 am]

BILLING CODE 6560-38-M

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40 CFR Part 246

[SW-FRL-1944-1]

Solid Waste Management; Guidelines for Beverage Containers; Resource Recovery Facilities Guidelines; Source Separation for Materials Recovery Guidelines

Correction

In FR Doc. 82-708, appearing at page 1307, in the issue of Tuesday, January 12, 1982, make the following change:

On page 1308, in the middle column, change paragraph "2." to read as follows:

"2. Section 246.100 is amended by removing paragraph (g) and by redesignating paragraph (h) as paragraph (g) and revising it to read as follows:"

BILLING CODE 1505-01-M

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40 CFR Part 799

[OPTS-42002A; TSH-FRL-2030-6]

Fluoroalkenes; Extension of Comment Period

AGENCY: Environmental Protection Agency (EPA).

ACTION: Advance notice of proposed rulemaking; extension of comment period.

SUMMARY: EPA is extending the comment period for the proposed test rule for fluoroalkenes published in the Federal Register of October 30, 1981 (46 FR 53704) to give interested persons additional time to comment on the plan to propose test rules.

DATE: All comments on the proposed rule should be sumbitted on or before February 1, 1982.

ADDRESS: Written comments should bear the document control number OPTS-42002A and should be submitted in triplicate to: Document Control Officer (TS-793), Office of Pesticides and Toxic Substances, Environmental Protection Agency, Rm. E-401, 401 M St. SW., Washington, D.C. 20460.

The administrative record supporting this action is available for public inspection in Rm. E107 at the above address from 8:00 a.m. to 4:00 p.m., Monday through Friday, except legal holidays.

FOR FURTHER INFORMATION CONTACT: Douglas G. Bannerman, Acting Director, Industry Assistance Office (TS-799), Office of Toxic Substances, Environmental Protection Agency, Rm. E-511, 401 M St. SW., Washington, D.C. 20460, Toll Free: (800-424-9065), in Washington, D.C.: (554-1404), outside the USA: (Operator 202-554-1404).

SUPPLEMENTARY INFORMATION: EPA issued an advance notice of proposed rulemaking for fluoroalkenes under section 4 of TSCA in the **Federal Register** of October 30, 1981 (46 FR 53704). The comment period for this proposed test rule will be extended 30 days (from December 29, 1981, to January 29, 1982) to give interested persons additional time to comment on testing requirements for this rule. A notice providing further information will be forthcoming at a later date.

(Sec. 4, Pub. L. 94-469, 90 Stat. 2003 (15 U.S.C. 2601))

Dated: January 5, 1982.

John A. Todhunter,

Assistant Administrator for Pesticides and Toxic Substances.

[FR Doc. 82-1185 Filed 1-14-82; 8:45 am]

BILLING CODE 6560-31-M

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40 CFR Part 761

[OPTS 211004; FRL 1989-8]

Polychlorinated Biphenyls (PCBs); Denial of Citizen's Petition

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed Rule Related Notice.

SUMMARY: This notice announces the Administrator's decision to deny a citizen's petition submitted under section 21 of the Toxic Substances Control Act (TSCA). The petitioner requested that the Agency amend its PCB rule (40 CFR Part 761) to exempt research and development activities from control, integrate the PCB rule with hazardous waste regulations issued under the Resource Conservation and Recovery Act (RCRA) (42 U.S.C. 6901), establish closure and post closure fund requirements, authorize salvage of metals from PCB items, establish performance standards for alternate disposal methods, and give the Administrator approval authority for disposal methods.

ADDRESS: A copy of the petition and all related information is located in: The office of the Document Control Officer (TS-793), Environmental Protection Agency, Rm. E-107, 401 M St., SW., Washington, D.C. 20460.

It is available for viewing and copying from 8 a.m. to 4 p.m., Monday through Friday, except legal holidays.

FOR FURTHER INFORMATION CONTACT: John B. Ritch, Jr., Director, Office of Industry Assistance (TS-799), Office of Toxic Substances, Environmental Protection Agency, Rm. E-511, 401 M St., SW., Washington, D.C. 20460, Toll free: (800-424-9065), In Washington, D.C.: (554-1404), Outside the USA: (Operator 202-554-1404).

SUPPLEMENTARY INFORMATION:

I. Introduction

On August 7, 1981, EPA received a TSCA section 21 petition concerning the PCB rules from EOI, Inc., a PCB waste management company headquartered in Washington, D.C. In general, the petition seeks changes in the PCB rule that would encourage the development of new technology, insure consistency among Regional Office in the implementation of the rule, and integrate the PCB rule with the RCRA hazardous waste regulations. EOI submitted six specific recommendations in its petition to change the PCB regulations.

EPA agrees that EOI has recognized six important areas which require clarification in the PCB regulations. However, EPA feels that the proper clarification can be accomplished without amending the regulation.

II. Petitioner's Arguments and the Agency's Responses

A. EOI has petitioned EPA for the addition to the PCB rule of a general exemption for research and development (R&D) activities involving storage, decontamination, transport, and disposal of PCB materials.

With one very narrow exception,[1] the present PBC rule has no specific

[1] Use of Small Quantities of PCBs for Research and Development is authorized (40 CFR 761.31 (j)).

Continued

provision which allows the use of PCBs in R&D activities. EPA recognizes that R&D for new methods of PCB handling and disposal is most desirable. The PCB rule establishes the Regional Administrators as the approval authority for PCB disposal methods. R&D on disposal methods has usually been regulated using the authority of the Regional Administrators, contained in 40 CFR 761.10(e), to approve alternative disposal methods. Some companies have conducted R&D on PCBs without the EPA Regional Office even being aware of their activities.

If EPA were to amend the PCB rule to deal with R&D activities, the prime objective of the amendment would be to allow legitimate research to proceed with a minimum of red tape while allowing EPA a means of halting research projects that present an unreasonable risk to human health and the environment. It is also desirable that any R&D rules provide a systematic set of procedures so that their application would be as consistent as possible throughout the country. This can be accomplished without rulemaking. EPA Headquarters will issue a guidance memorandum to the Regional Offices in the near future.

B. EOI has petitioned EPA to transfer immediately all regulations for control of PCBs from TSCA to RCRA authority.

The petitioner assumes that it is a simple process to integrate the rules promulgated under TSCA and RCRA. Unfortunately, this is not so. There are many complexities involved in integrating the PCB regulations into the RCRA. The TSCA rules for waste PCBs are in place, and they are well understood by the regulated community. As of this writing, the RCRA rules are the subject of pending litigation. In addition, the RCRA rules are undergoing comprehensive regulatory impact analyses that may result in a number of regulatory amendments. A major effort to integrate the PCB rules now could be confusing to the regulated community and could be inefficient if changes are made in the RCRA rules as a result of the litigation or the regulatory impact analyses. Moreover, effective implementation of the waste PCB regulatory control program could be interrupted.

For these reasons, EPA intends to leave the rules separate at this time, with waste PCBs controlled solely under the TSCA rules. Efforts to integrate the rules will resume after the major RCRA litigation issues are resolved and the RCRA regulatory impact analyses are completed.

C. EOI has petitioned EPA to establish a requirement for closure and post-closure funds for facilities storing, processing, or disposing of PCBs.

Closure and post-closure funds represent money set aside by companies managing wastes to ensure environmentally acceptable closure of the facility. These funds are not required by the PCB rule, but financial assurance for facility closure is required under the RCRA hazardous waste regulations.

The petitioner expresses the concern that some firms are collecting and storing PCBs waiting for an easier and/or cheaper disposal method to be approved. It this does not occur, the petitioner speculates that these firms may not have the resources to dispose properly of the PCBs they have collected. EPA's approach to combat irresponsible activities has been to place the burden on the generator to determine whether a person taking possession of their PCBs is reputable. EPA enunciated this policy in the preamble to the PCB Ban Rule (44 FR 31539, May 31, 1979). In addition, if there are problems with improperly stored PCB waste, EPA has authority under several statutes to take action to clean up or require others to clean up the problem. Although requiring closure and post-closure funds would be an improvement on the present system, the current approach is not unworkable. Therefore, EPA will leave the PCB regulation silent with respect to closure funds, and address this issue when the PCB/RCRA integration takes place.

D. EOI has petitioned EPA to add a provision to the regulation that allows the salvage of copper and steel from PCB Equipment, as defined in 40 CFR 761.2 (w); PCB Transformers, as defined in 40 CFR 761.2 (y); and PCB Containers, as defined in 40 CFR 761.2 (v).

There is some confusion in the regulated community on this subject. In this notice, EPA is seeking to clarify its current policies regarding metal recovery operations. Under the current regulations, there is a way that these metals may be salvaged.

EPA's major concern when developing the PCB Rule was the high human and environmental exposure to PCBs that resulted from the rebuilding and salvage of PCB Transformers. Standard industry practices were very sloppy. Because of their large number and relatively low PCB concentration, PCB-Contaminated Transformers, as defined in 40 CFR 761.2 (z), were permitted to be rebuilt and salvaged. Because of the far smaller number of PCB Transformers and the great uncertainty related to the industry's ability to adequately protect against human and environmental PCB exposure, EPA decided not to include in the PCB Rule any specific provision for the decontamination for salvage of PCB Transformers.

EPA's position is that physical separation of PCBs from the metal portion of the transformers, followed by recycle of the metal by incineration or other destruction of the PCB portion remaining in the transformer, can be approved under the current regulatory structure defined in 40 CFR 761.10(e). However, it must be shown that the total alternate disposal method provides environmental protection equivalent to incineration under Annex I of the PCB rule found in 40 CFR 761.40

The PCB rule prohibits removal of the core from PCB Transformers for servicing and requires that the intact, flushed PCB Transformer, minus the PCB dielectric fluid, be disposed of in an incinerator or EPA-approved chemical waste landfill. The PCB rule provides that waste materials requiring incineration may be disposed of by alternative disposal methods that can be shown to achieve performance equivalent to PCB incinerators. Since metal recovery furnaces operate at very high temperatures for long periods of time, it may be possible for a furnance owner to obtain EPA approval as an alternative disposal method for the PCBs.

If a recycle/incineration system were developed for PCB Transformers that reduced worker and environmental PCB exposures to a level no greater than that which occurs when PCB-Contaminated Transformers are rebuilt, there should be little concern about approving such a system. An additional advantage of such a system, beyond the salvage of valuable metals, would be the reduction in disposal-related transportation costs and in landfill space required for PCB Transformers. The owner of a metals recovery furnace could apply for approval, as an alternative PCB disposal method, under 40 CFR 761.10(e).

Thus, EPA's policy is that metal recyclers can either incinerate metal parts at conditions which destroy PCB molecules, or completely remove PCB molecules from metal parts by use of solvents in the vapor or liquid phase. In addition, the PCB regulation allows transformer owners to lower the contamination classification of their transformers by retrofilling, followed by three months of operation (40 CFR 761.31(a)(5)). During this three-month period, the heat of operation and the

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Small Quantities for Research and Development is defined to include only PCBs originally packaged in one or more hermetically sealed containers of a volume no more than five milliliters (40 CFR 761.2 (ee)).

circulation of dielectric fluid serves to remove PCBs from the transformer core. The disposal of metals from properly reclassified transformers is not controlled under the PCB rule. This process of reclassification might also be accomplished by retrofilling and simulating transformer operation. Any such potential method of metal recovery may be proposed and demonstrated for approval as an alternate method of disposal (40 CFR 761.10 (e)).

There is one other section of the PCB rule which bears an important relationship to the topic of alternate disposal methods. That is 40 CFR 761.30(c)(2), which allows processing and distribution in commerce for purposes of disposal. "Processing for purposes of disposal" has been suggested as a section of the PCB rule which would authorize decontamination of PCB transformers and other equipment for recycle. The section authorizing processing for disposal was intended to facilitate disposal activities. For example, in the case of capacitor disposal, processing for disposal allows grinding of the capacitors prior to incineration. Without this allowance, the regulation would require incineration of whole capacitors, a difficult technical task.

It is theoretically possible to develop a method of physically separating the PCBs from the metals (e.g., solvent extraction). If the method were successful in completely removing all detectable PCBs from the metals, the metal could then be salvaged without subsequent treatment. PCBs removed from the transformer would require incineration. In a case where disposal of the PCB equipment or liquids was regulated, any alternate disposal method requires prior approval under § 761.10(e). This is also the case with PCB-contaminated solvents. These liquids must be incinerated because they are the result of dilution of high concentrations of PCBs.

E. EOI has petitioned EPA to establish separate performance and efficiency standards for alternate disposal techniques.

The advantage of 40 CFR 761.10(e), as it is currently written, is that it can be used to evaluate any new disposal method that may be proposed regardless of the process used. At the time the disposal regulation was issued, EPA recognized that it could not anticipate the technological advances that might be developed toward solving the PCB disposal problem. If EPA set specific parameters, or standards, it would probably be unable to apply them to every possible new disposal technique. In fact, a set of standards would stifle creativity because all new methods would have to be designed to fit the standards.

Section 761.10(e) of the PCB rule states, in part, "Any person who is required to incinerate any PCBs and PCB Items under this subpart and who can demonstrate that an alternative method of distroying PCBs and PCB Items exists and that this alternative method can achieve a level of performance equivalent to Annex I incinerators or high efficiency boilers * * * may submit a written request to the Regional Administrator for an exemption from the incineration requirements." The word "equivalent" is not interpreted to mean "identical", but rather to define a system that provides for the same amount of environmental protection.

One chemical disposal method has been approved under this section and many other varied methods are proposed or under development. Since the present regulation appears to be working, EPA will leave it in place.

F. EOI has petitioned EPA to remove the authority for approval of disposal facilities from the EPA Regional Administration and give it to the Administrator.

The PCB rule gives all the authority for approval of disposal methods to the Regional Administrators. There is a need, however, for uniformity. EPA believes that this consistency can be achieved by improved communication between EPA Headquarters and EPA Regional Offices and among the Regional Offices. Specifically, EPA Headquarters will issue a guidance memorandum to the Regional Offices addressing the need for consistency.

Continuation of decentralized control is desirable because the Regional Offices have traditionally filled this role and are accustomed to it. They have personnel who have learned through experience how best to implement a program for approval of PCB disposal facilities. EPA presently has a contract for technical assistance to the Regional Offices to provide additional review of proposed disposal methods.

EPA therefore intends to leave the approval authority with the Regional Administrators.

Finding

The administrator hereby denies the petition submitted by EOI, Inc., under section 21 of TSCA.

Dated: December 22, 1981.

Anne M. Gorsuch,

Administrator.

[FR Doc. 82-1175 Filed 1-14-82; 8:45 am]

BILLING CODE 6560-31-M

DEPARTMENT OF THE INTERIOR

Office of the Secretary

43 CFR Subtitle A

Flood Insurance for Undeveloped Coastal Barriers; Preliminary Identification

AGENCY: Office of the Secretary, Interior.

ACTION: Notice of Availability of Draft Document.

SUMMARY: This notice is to announce the availability of a draft (Pre-Proposed) document amplifying on the statutory definition and draft maps with supporting information summaries concerning the preliminary identification of undeveloped coastal barriers for initial public review and comments prior to issuance of proposed rule.

DATE: Comments on the draft definitions, draft maps, and draft information summaries should be received no later than March 22, 1982.

ADDRESS: Mr. Ric Davidge, Chairman; Coastal Barriers Task Force; United States Department of the Interior; Washington, D.C. 20240.

FOR FURTHER INFORMATION CONTACT: Mr. Rich Davidge, Chairman; Coastal Barriers Task Force; United States Department of the Interior; Washington, D.C. 20240.

SUPPLEMENTARY INFORMATION: On December 1, 1981, the Secretary of the Interior issued a "Notice of intent to issue a proposed rule" on or about August 13, 1982. As indicated in that Notice, the proposed rule will concern delineation of those areas along the Atlantic Coast and Gulf of Mexico which are determined to be undeveloped coastal barriers, as defined in the Omnibus Budget Reconciliation Act (OBRA) of 1981. 46 Fed. Reg. 58346. Final designation of undeveloped coastal barriers by the Secretary of the Interior will occur thereafter—pursuant to final rulemaking. That action will not occur prior to October 1, 1982. Designation of undevelopd coastal barriers will impact upon the availability of Federal flood insurance after October 1, 1983, pursuant to the National Flood Insurance Act of 1968, as amended by OBRA. Section 1321(a) of that Act provides that "[n]o new flood

insurance coverage shall be provided under this title on or after October 1, 1983, for any new construction or substantial improvements of structures located on undeveloped coastal barriers which shall be designated by the Secretary of the Interior." This final designation will be for Federal flood insurance purposes only.

The December 1, 1981, Notice of Intent outlines the two-fold responsibilities of the Department of the Interior with regard to section 341 of the Omnibus Budget Reconciliation Act, as enacted on August 13, 1981. The Notice was amended slightly on December 8, 1981. 46 FR 69022. As amended, this December Notice served to establish the process the Department of the Interior will follow in order to:

- Conduct a study for the purpose of designating undeveloped coastal barriers and to provide a report to Congress concerning the conclusions of such study and any recommendations regarding the definition of the term "coastal barrier"; and
- Designate undeveloped coastal barriers.

This December Notice also discussed the relationships of the OBRA implementation process to other Federal legal requirements, such as NEPA, and the Department's concern for extensive public review and comment at each step in the preliminary identification and delineation, study, and designation efforts.

The process outlined in the December Notice remains essentially unchanged. The first step was the development of draft definitions and draft maps. This task has now been completed. Consideration was given to comments and suggestions that were received concerning the proper interpretation of OBRA. Initial comments on the draft definitions were then solicited from concerned Members of Congress and Governors of coastal States pursuant to letters from the Secretary of the Interior dated December 9 or 10, 1981. To the degree comments were received prior to January 9, 1982, they have been considered in the preparation of the present draft definitions document and the draft maps. Comments received thereafter will be considered prior to issuance of a proposed rule and submission of the proposed designations of the undeveloped coastal barriers to the Congress.

As indicated in the December Notice, the next step is the public release of these draft definitions, draft maps, and draft summaries of information relevant to designation of undeveloped coastal barriers. This is being done today. The comment period on these draft definitions, draft maps, and draft information summaries will close on March 15, 1982, with any comments received within one week thereafter considered. These comments will then serve as a basis for review and reconsideration of the draft definitions document, draft maps, and draft information summaries and preparation of proposed definitions and proposed maps. Upon completion of that review, proposed designations and supporting material will be made available for additional public review and comment and will be provided to the Congress for their consideration. Consistent with OBRA, this task will be completed prior to August 13, 1982.

As indicated in the December Notice these proposed designations will be based upon the status of the various coastal barriers as of the close of this comment period—March 15, 1982. This date has been chosen to ensure that proposed designations can be provided to the Congress on or before August 13, 1982, as required by OBRA. It is important that public comments on the draft definitions, draft maps, and draft information summaries being released today include a discussion of the factual situation on the coastal barriers as of March 15, 1982. Status of development, nature and extent of infrastructure leading toward development, existence of structures and man's activities on the coastal barriers, and whether an area is otherwise protected as provided by Section 1321(b)(3) of the National Flood Insurance Act of 1968, as amended by OBRA—all of these factors need to be addressed as of the close of the comment period on March 15, 1982. It is for this reason that comments received within one week after March 15, 1982, will still be considered.

It is important to emphasize that this March 15 date is not dictated by OBRA. This legislation did not address the question of what date should be used as a basis for final designations; that is, what date would be used to established the factual situation on each coastal barrier. While it is clear that some date no later than October 1, 1983, must be chosen to ensure that final designations of coastal barriers can be established as of the effective date, it is also obvious that dates other than March 15, 1982, could be utilized. This is important factor still under consideration within the Department of the Interior at this time.

It is also important to emphasize that this discussion does not affect the sale of flood insurance prior to October 1, 1983, for any new construction or substantial improvements. Rather, it concerns the determination of which areas fall within the definitions provided by OBRA and must be designated as undeveloped coastal barriers. Flood insurance in effect prior to October 1, 1983, will remain valid thereafter for those insured structures regardless of designation of a coastal barrier area consistent with the provisions of OBRA. After October 1, 1983, Federal flood insurance will not be available on designated coastal barriers for new construction or substantial improvements.

Another point warrants emphasis. Many other areas not preliminarily identified as undeveloped coastal barriers also contain important wetland and other aquatic habitats. Other Federal laws applicable to these resources (e.g., the Fish and Wildlife Coordination Act or sections 9 and 10 of the Rivers and Harbors Act of 1899) are not affected by this action. The OBRA provision does not affect the applicability of any Federal statutes other than the National Flood Insurance Act of 1968, as amended.

The December Notice also discussed the relationship of the coastal barrier process to other Federal legal requirements. That discussion remains applicable to the present situations. It is contemplated that a Draft Environmental Impact Statement will be available shortly. In this regard, a notice of intent to prepare an environmental impact statement, as published in the **Federal Register** on December 21, 1981, 46 FR 61929, discusses NEPA compliance in greater detail.

The final issue in the December Notice was the Department's concern for extensive public review and comment. As indicated above, this remains an important issue to the Department. It should be emphasized that the draft definitions, draft maps and draft information summaries being released for public review and comment today, are indeed, drafts. A second round of comments will be solicited at the proposed rulemaking stage, on or before August 13, 1982, and proposed definitions and proposed maps will be made available for public review and comment at that time, prior to final designation.

Draft maps, along with the draft definitions document, and draft summaries of information used to tentatively delineate the undeveloped coastal barriers depicted on the maps, are being sent to a number of classes of recipients with special interest in this issue. The Secretary of the Interior has suggested that these recipients seek the widest possible distribution of these draft definitions, draft maps, and draft

information summaries. The classes of recipients of these materials include:

—Senators and Members of Congress from the 16 affected States
—U.S. Fish and Wildlife Service
  —Washington Office
  —Regional Offices
  —Area Offices
  —Ecological Services Field Offices
  —Cooperative Fish and Wildlife Research Units
  —National Coastal Ecosystems Team
—National Park Service
  —Washington Office
  —Regional Offices
  —National Seashores
  —Cooperative Research Units
—Federal Emergency Management Agency
  —Washington Office
  —Regional Offices
—Other Federal Agency Washington Offices
  —Department of Commerce
  —U.S. Army Corps of Engineers
  —Office of Management and Budget
  —Department of Transportation
  —Department of Housing and Urban Development
  —Governors of the 16 affected States
  —A–95 Clearinghouses of the 16 affected States
  —Affected Local Governments
  —Affected Regional governmental entities.

To facilitate public review, the Department has established a system whereby anyone interested may learn where the closest set of maps can be examined. This can be accomplished by calling the U.S. Geological Survey, Eastern National Cartographic Information Center (E–NCIC), at (703) 860–6336 or FTS: 928–6336 between the hours of 8:00 a.m. and 4:00 p.m. EST. Callers must indicate the State and County in which the units of concern are located as well as where they are located. PLEASE NOTE: Maps cannot be ordered by calling this telephone number.

Addresses

Draft undeveloped coastal barrier maps can be purchased from the U.S. Geological Survey at the address indicated below. To cover reproduction and handling costs, a fee of $3.25 will be charged *per map* for each 36 in. x 42 in. paper ozalid copy. Requests for copies must be made using the following ORDER FORM (or a copy thereof) and must be prepaid by check or money order (NO cash or stamps) made payable to: THE UNITED STATES GEOLOGICAL SURVEY. The ORDER FORM and check or money order should be sent to: Eastern National Cartographic Information Center (E–NCIC), U.S. Geological Survey, 536 National Center, Reston, Virginia 22092.

Requests for additional copies of the draft definitions document and draft information summaries must be made in writing and directed to: Ms. Deborah Lanzone, National Park Service—780, Pension Building, Room 201, 440 G Street, NW., Washington, D.C. 20243, (202) 272–3566.

Comments on the draft definitions, draft maps, and draft information summaries should be addressed to: Mr. Ric Davidge, Chairman, Coastal Barriers Task Force, United States Department of the Interior, Washington, D.C. 20240.

Maps may be inspected at and hand-delivered comments may be taken to: Office of the Assistant Secretary for Fish and Wildlife and Parks, Main Interior Building, 18th and C Streets, NW., Room 3148, Washington, D.C. 20240.

For Further Information Contact

Mr. Ric Davidge, Chairman, Coastal Barriers Task Force, U.S. Department of the Interior, Washington, D.C. 20240. (202) 343–5347.

Dated: December 12, 1981.

G. Ray Arnett,

Assistant Secretary for Fish and Wildlife and Parks.

ATTACHMENT A

Order Form

Draft Undeveloped Coastal Barrier Maps

This form will enable you to obtain copies of some or all of the 161 draft Undeveloped Coastal Barrier Maps identified by the U.S. Department of the Interior pursuant to Section 341(d)(1) of the Omnibus Budget Reconciliation Act of 1981 (Pub. L. 97–35). Each paper print which measures 36 inches by 42 inches will cost $3.25.

PLEASE INDICATE THE NUMBER OF MAPS OF EACH UNIT YOU WANT TO ORDER IN THE APPROPRIATE BOX ON THE FOLLOWING LIST OF MAPS. IF YOU MARK THE STATE BOX, THE NUMBER OF SETS OF MAPS INDICATED FOR THE ENTIRE STATE WILL BE MAILED TO YOU.

☐ MAINE (6 maps)
  ☐ A03—Jasper
  ☐ A04—Pond Island
  ☐ A06—Cape Elizabeth
  ☐ A07—Scarborough Beach
  ☐ A08—Crescent Surf
  ☐ A09—Seapoint
☐ MASSACHUSETTS (31 maps)
  ☐ C01—Wingaersheek
  ☐ C02—North Scituate Beach
  ☐ C03—Rivermoor
  ☐ C04—Plymouth Bay
  ☐ C06—Center Hill Complex
  ☐ C08—Scorton
  ☐ C09—Sandy Neck
  ☐ C10—Freemans Pond
  ☐ C11—Namskaket Spits
  ☐ C12—Chatham Roads
  ☐ C13—Lewis Bay
  ☐ C14—Squaw Island
  ☐ C15—Centerville
  ☐ C16—Dead Neck
  ☐ C17—Popponesset Spit
  ☐ C18—Waquoit Bay
  ☐ C19—Black Beach
  ☐ C20—Coatue
  ☐ C21—Sesachacha Pond
  ☐ C22—Cisco Beach
  ☐ C23—Esther Island Complex
  ☐ C24—Tuckernuck Island
  ☐ C25—Muskeget Island
  ☐ C26—Eel Pond Beach
  ☐ C27—Cape Poge
  ☐ C28—South Beach
  ☐ C29—Squibnocket Complex
  ☐ C31—Elizabeth Islands
  ☐ C32—Mishaum Point
  ☐ C33—Little Beach
  ☐ C34—Horseneck Beach
☐ RHODE ISLAND (8 maps)
  ☐ D01—Little Compton Ponds, MA/RI
  ☐ D02—Fogland Marsh
  ☐ D03—Card Ponds
  ☐ D04—Green Hill Beach
  ☐ D05—East Beach
  ☐ D06—Quonochontaug Beach
  ☐ D07—Maschaug Ponds
  ☐ D08—Napatree
☐ CONNECTICUT (7 maps)
  ☐ E01—Wilcox Beach
  ☐ E02—Goshen Cove
  ☐ E03—Jordan Cove
  ☐ E04—Menunketesuck Island
  ☐ E05—Hammonasset
  ☐ E06—Sandy Hook
  ☐ E07—Milford Point
☐ NEW YORK (11 maps)
  ☐ F01—Fishers Island Barriers
  ☐ F02—Eatons Neck
  ☐ F04—Crane Neck
  ☐ F05—Old Field Beach
  ☐ F06—Shelter Island Barriers
  ☐ F07—North Haven
  ☐ F08—Clam Island
  ☐ F9—Gardiners Island Barriers
  ☐ F10—Napeaque
  ☐ F11—Mecox
  ☐ F12—Southhampton Beach
☐ NEW JERSEY (2 maps)
  ☐ G01—Stone Harbor Point
  ☐ G02—Cape May Complex
☐ DELAWARE (1 map)
  ☐ H01—North Bethany Beach
☐ VIRGINIA (5 maps)
  ☐ K01—Assawomen Island
  ☐ K02—Metomkin Island
  ☐ K03—Cedar Island
  ☐ K04—Little Cobb Island
  ☐ K05—Fishermans Island
☐ NORTH CAROLINA (9 maps)
  ☐ L01—Currituck Banks
  ☐ L02—Bodie Island
  ☐ L03—Hatteras Island
  ☐ L04—Bogue Banks
  ☐ L05—Onslow Beach Complex
  ☐ L06—Topsail
  ☐ L07—Lea Island Complex
  ☐ L08—Wrightsville Beach
  ☐ L09—Masonboro Island
☐ SOUTH CAROLINA (13 maps)

- ☐ M01—Waites Island Complex, NC/SC
- ☐ M02—Litchfield Beach
- ☐ M03—Pawleys Inlet
- ☐ M04—Debidue Beach
- ☐ M05—Dewees Island
- ☐ M06—Morris Island Complex
- ☐ M07—Bird Key Complex
- ☐ M08—Captain Sams Inlet
- ☐ M09—Edisto Complex
- ☐ M10—St. Helena Sound Complex
- ☐ M11—Harbor Island
- ☐ M12—St. Phillips Island Complex
- ☐ M13—Daufuskie Island

☐ GEORGIA (4 maps)
- ☐ N01—Little Tybee Island
- ☐ N02—St. Catherines Island
- ☐ N03—Little St. Simons Island
- ☐ N04—Sea Island

☐ FLORIDA (32 maps)
- ☐ P01—Amelia Island
- ☐ P02—Bird/Talbot Islands
- ☐ P04—Guana River
- ☐ P04A—Usinas Beach
- ☐ P05—Conch Island
- ☐ P05A—Matanzas River
- ☐ P07—Ormond-By-The-Sea
- ☐ P09—Ponce Inlet
- ☐ P10—Vero Beach
- ☐ P10A—Blue Hole
- ☐ P11—Hutchinson Island
- ☐ P12—Hobe Sound
- ☐ P13—Jupiter
- ☐ P13A—Lake Worth
- ☐ P14A—North Beach
- ☐ P15—Cape Romano
- ☐ P16—Keewaydin Island
- ☐ P17—Lovers Key Complex
- ☐ P18—Sanibel Island Complex
- ☐ P19—North Captiva Island
- ☐ P20—Cayo Costa
- ☐ P21—Bocilla Island
- ☐ P22—Casey Key
- ☐ P23—Longboat Key
- ☐ P24—The Reefs
- ☐ P25—Atsena Otie Key
- ☐ P26—Pepperfish Keys
- ☐ P28—Dog Island
- ☐ P29—St. George Island
- ☐ P30—Cape San Blas
- ☐ P31—St. Andrew Complex
- ☐ P32—Moreno Point

☐ ALABAMA (2 maps)
- ☐ Q01—Mobile Point
- ☐ Q02—Dauphin Islands

☐ MISSISSIPPI (3 maps)
- ☐ R01—Round Island
- ☐ R02—Deer Island
- ☐ R03—Cat Island

☐ LOUISIANA (14 maps)
- ☐ S01—Bastian Bay Complex
- ☐ S01A—Bay Joe Wise Complex
- ☐ S02—Grande Terre Islands
- ☐ S03—Caminada
- ☐ S04—Bay Champagne
- ☐ S05—Timbalier Island
- ☐ S06—Isles Dernieres (2 maps)
- ☐ S07—Point au Fer
- ☐ S08—Cheniere Au Tigre
- ☐ S09—Rollover
- ☐ S10—Mermentau River Complex
- ☐ S11—Sabine (2 maps)

☐ TEXAS (13 maps)
- ☐ T01—Sea Rim
- ☐ T02—High Island
- ☐ T03—Bolivar Peninsula
- ☐ T04—Follets Island
- ☐ T05—Brazos River Complex
- ☐ T06—Sargent Beach
- ☐ T07—Matagorda Peninsula (2 maps)
- ☐ T08—San Jose Island Complex
- ☐ T09—Mustang Island
- ☐ T10—North Padre Island
- ☐ T11—South Padre Island
- ☐ T12—Boca Chica

☐ COMPLETE SET OF ALL DRAFT UNDEVELOPED COASTAL BARRIER MAPS (161 maps)

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Eastern National Cartographic Information Center (E-NCIC)
U.S. Geological Survey
536 National Center
Reston, Virginia 22092
Telephone: (703) 860-6336 or FTS 928-6336

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STREET ADDRESS _____
CITY STATE ZIP CODE
ORGANIZATION _____

TO BE ABLE TO CONTACT YOU IN THE EVENT THERE ARE QUESTIONS ABOUT YOUR ORDER, PLEASE INCLUDE A TELEPHONE NUMBER WHERE YOU CAN BE REACHED WEEKDAYS BETWEEN 8:00 a.m. AND 4:00 p.m. EST.:

TELEPHONE: AREA CODE ( ) NUMBER

[FR Doc. 82-1197 Filed 1-14-82; 8:45 am]
BILLING CODE 4310-70-M

FEDERAL COMMUNICATIONS COMMISSION

47 CFR Part 73

[BC Docket No. 82-1; FCC No. 82-1]

Subsidiary Communications Authorization (SCA) Operations

AGENCY: Federal Communications Commission.

ACTION: Proposed rule.

SUMMARY: Action taken herein proposes the amendment of §73.593 of the Commission's Rules to permit public broadcasting FM stations to stand on the same footing as commercial FM stations in conducting their Subsidiary Communications Authorization operations.

DATES: Comments must be filed on or before February 11, 1982, and reply comments on or before February 26, 1982.

ADDRESS: Federal Communcations Commission, Washington, D.C. 20554

FOR FURTHER INFORMATION CONTACT: Jonathan David, Broadcast Bureau, (202) 632-7792.

SUPPLEMENTARY INFORMATION:

Adopted: January 5, 1982.

Released: January 11, 1982.

1. The Commission has before it the provisions of § 73.593 of the Commission's Rules which impose restrictions on the use of a Subsidiary Communications Authorization ("SCA") granted to noncommercial educational FM stations, now called public broadcasting stations. Recent amendments to the Communications Act[1] have called upon educational stations to provide more of their own funding. As discussed below, this raises the question whether the current restriction on SCA use should be continued.

2. In addition to the regular broadcast service offered on the main carrier (channel), FM stations have the capacity to program one or more subcarriers[2] on a multiplex basis to provide SCA service.[3] upon grant of the necessary application, both commercial and public broadcast FM stations are permitted to provide SCA service. Unlike the commercial station, the public broadcasting can only transmit programs of a noncommercial nature which are in furtherance of an educational purpose.

3.The placement of restrictions on the use of an SCA by a public broadcasting station reflects the then prevailing view about the nature of these stations as well as the expectation that they would have adequate funding from outside sources. Recently, the situation has changed. Federal funding, once a major source, has been greatly curtailed. Recognizing the consequences of dwindling Federal funding, the Congress acted to let these stations do more to help themselves. In fact, one of the main purposes of the Public Broadcasting

[1] Pub. L. 97-35 (Public Broadcasting Amendment Act of 1981).

[2] One such subcarrier is necessary to carry the second signal if the station operates in stereo.

[3] SCA's can be used for a variety of broadcast-like services. It is frequently used by commercial stations for background music in stores and offices. Public broadcasting stations are not now permitted to use it for such commercial purposes.

Amendments Act of 1981 was to help these broadcasters develop such other funding. To this end, the bill (Pub. L. 97–35) contained (in new Section 399B) provisions allowing public broadcast stations to engage in offering services, facilities or products for remuneration. This provision allows these educational licensees to engage in a variety of remunerative non-broadcast activities.

4. Examination of the new public broadcasting provisions and the Reports and debates which accompany them suggest that it may be inappropriate to continue the restriction on licensees of public radio stations that limits these SCA's to educational purposes and prevents these stations from using their subcarrier SCA capacity for remunerative purposes. In fact, an argument can be made that the current restriction is inconsistent with the new Section 399B. Therefore, we are proposing to consider deletion of the current restriction. With this deletion, commercial and noncommercial educational stations would stand on the same footing in regard to the basis on which they could obtain an SCA and the uses to which it could be put.[4]

5. Regulatory Flexibility Analysis:

I. *Reason for action:* Use of the SCA in the fashion proposed could help educational FM stations be self-supporting and could lead to more efficient use of their subcarrier frequencies, which now sometimes lie fallow.

II. *The objective:* The Commission proposed to allow educational FM stations to employ SCA's for the same purposes now permitted commercial FM stations.

III. *Legal basis:* The action proposed would explore new and improved uses of radio and thus would be in furtherance of Sections 303(g) and 399B of the Communications Act of 1934, as amended.

IV. *Description, potential impact and number of small entities affected:* The proposed removal of the restriction on SCA uses by public broadcasting stations could be expected to enhance the ability of these stations to generate revenues and be more self-supporting. This, in turn, could provide opportunities to enhance competition and increase the availability of SCA services in a community. The rule change, if adopted, would directly affect the almost 1,200 public broadcasting FM stations and indirectly affect the more than 3,500 commercial FM stations which do not now receive competition from public broadcasting station SCA's run on a commercial basis. It is also possible that such a step could have an impact on small governmental or business entities which would gain access to SCA services for the first time. Finally, small entities involved in supplying equipment or services connected with constructing or conducting SCA operations could be affected as such opportunities increased.

V. *Recording, record keeping and other compliance requirements:* None.

VI. *Federal rules which overlap, duplicate or conflict with this rule:* None.

VII. *Any significant alternative minimizing impact on small entities and consistent with stated objective:* The only alternative would be to maintain the status quo and thereby continue to preclude expanded SCA uses by public broadcasting FM stations.

[4]Recently, the Commission adopted a change in permissable use of the commercial station SCA to authorize non-broadcast transmissions for utility load management. Thus, the proposed rule change treating commercial and public broadcasting FM stations on the same footing would permit this use for educational stations as well.

PART 73—RADIO BROADCAST SERVICES

6. Accordingly, it is proposed, that pursuant to the provisions of Sections 4(i), 303 (b), (g) and 399B of the Communications Act of 1934, as amended, § 73.593 of the Commission's Rules be revised to read as follows:

§ 73.593 Subsidiary communications authorizations.

The provisions governing SCA authorizations set forth in § 73.293 are applicable to noncommercial educational FM stations.

7. Authority for the institution of this proceeding is contained in Sections 4(i) and 303 of the Communications Act of 1934, as amended.

8. Pursuant to procedures set forth in § 1.415 of the Commission's Rules, interested persons may file comments on or before February 11, 1982, and reply comments on or before February 26, 1982. The Commission will consider all relevant and timely comments and may also consider other relevant information before it before taking further action in this proceeding.

9. In accordance with the provisions of § 1.419 of the Commission's Rules, an original and five copies of all comments, replies, briefs, and other documents shall be furnished the Commission. Further, members of the general public who wish to participate informally in the proceeding may submit one copy of their comments, specifying the docket number in the heading. All filings in this proceeding will be available for examination by interested persons during regular business hours in the Commission's Public Reference Room at its headquarters, 1919 M Street, N.W., Washington, D.C. 20554.

10. For further information concerning this proceeding, contact Jonathan David, Broadcast Bureau, (202) 632–7792. However, members of the public should note that from the time a Notice of Proposed Rule Making is issued until the matter is no longer subject to Commission consideration or court review, all *ex parte* contacts presented to the Commission in proceedings such as this one will be disclosed in the public docket file.

11. An *ex parte* contact is a message (spoken or written) concerning the merits of a pending rule making other than comments officially filed at the Commission or oral presentations requested by the Commission. If a member of the public does wish to comment on the merits of this proceeding in this manner, he or she should follow the Commission's procedures governing *ex parte* contacts in informal rule making. A summary of these procedures is available from the Commission's Consumer Assistance Office, Federal Communications Commission, Washington, D.C. 20554, (202) 632–7000.

(Secs. 4, 303, 307, 48 Stat., as amended, 1066, 1082, 1083; 47 U.S.C. 154, 303, 307)

Federal Communications Commission.

William J. Tricarico,

Secretary.

[FR Doc. 82–1060 Filed 1–14–82; 8:45 am]

BILLING CODE 6712-01-M

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47 CFR Part 73

[BC Docket No. 81–741]

Transmission of Teletext by TV Stations; Order Extending Time for Filing Comments and Reply Comments; Authorization

AGENCY: Federal Communications Commission.

ACTION: Proposed rule; extension of comment/reply comment period.

SUMMARY: Action taken herein extends the time for filing comments and replies to comments to a Notice of Proposed Rule Making, (46 FR 60851; December 14, 1981) Docket No. 81–741, which proposed amendment of Part 73 of Commission Rules to allow transmission of teletext by TV stations. Several parties filed requests for such an extension.

DATE: Comments are due on or before February 10, 1982 and replies to