

RESOLUTION NO. 540

RESOLUTION OF THE BOARD OF DIRECTORS
OF THE PUGET SOUND AIR POLLUTION
CONTROL AGENCY ADOPTING MODIFIED
PARTICULATE SOURCE TEST PROCEDURES

WHEREAS, Regulation I Section 9.09(f) requires procedures for source sampling performed in connection with standards of Regulation I and II for particulate and gases to be done using current Environmental Protection Agency requirements or procedures and definitions adopted by the Board; and

WHEREAS, to conform to current safe and less toxic chemical storage, the particulate measurement procedures currently used by the Agency have been proposed for modification; and

WHEREAS, the Expanded Advisory Council reviewed and approved said source test laboratory procedure modifications; and

WHEREAS, a public hearing was held by the Puget Sound Air Pollution Control Agency Board of Directors on August 11, 1983, to allow public input and critique on the proposal; and

WHEREAS, the Board deems it necessary to adopt said modification to source test procedures; now therefore,

BE IT RESOLVED BY THE BOARD OF PUGET SOUND AIR POLLUTION CONTROL AGENCY:

The Board of Directors does hereby adopt the modifications to the source test procedures, a copy of which is attached hereto and made a part hereof.

PASSED AND APPROVED by the Board of Directors of the Puget Sound Air Pollution Control Agency held this 17th day of August, 1983.

PUGET SOUND AIR POLLUTION CONTROL AGENCY

By [Signature]
Chairman

Attest:

[Signature]
Air Pollution Control Officer

Approved as to form:

[Signature]
Agency Attorney

**Proposed Revised PSAPCA
Particulate Source Test Procedures**

**Engineering Division
Puget Sound Air Pollution Control Agency
200 West Mercer Street, Room 205
P.O. Box 9863
Seattle, Washington 98109**

June 9, 1983

I. Procedures for Particulate Source Sampling

Unless otherwise authorized by the Control Officer, all particulate source sampling performed to demonstrate compliance with the emission standards of Regulation I shall be done using current Environmental Protection Agency Methods 1-5 contained in 40 CFR Part 60, Appendix A, as modified in Section II of this document.

II. Procedure for Determining Particulate Matter in the Impinger Catch (Back Half)

The analysis and calculations for Method 5 shall conform to that described by EPA in the current 40 CFR Part 60, Appendix A, except that the back half catch shall be included as particulate matter. The back half weight is the sum of the impinger catch (organic and inorganic) and the back half acetone rinse weights.

A. Sample Recovery of the Back Half

1. Purging

Whenever SO₂ interference is suspected, purge the impingers immediately after the test run is complete with N₂ or clean air for a minimum of one-half the sample volume.

2. Impinger Liquid

Measure the volume of water collected in all impingers and place the water from the first three impingers in a container. Thoroughly rinse all sample-exposed surfaces between the filter and fourth impinger with water and place in above container.

3. Acetone Rinse

Thoroughly rinse all sample-exposed surfaces between the filter and the fourth impinger with acetone and place the washings in a tared beaker to dry.

B. Analysis of the Back Half

1. Impinger Liquid Extraction

- a. Add 50-100 ml of dichloromethane to the impinger liquid.
- b. Spin for at least ten minutes.

- c. Pour the liquid into a separatory funnel and drain the organic phase into a tared beaker (organic fraction).
- d. Drain the remaining liquid into a beaker and repeat Steps a, b, and c. Perform the extraction several times with fresh dichloromethane until the organic fraction is clear. Keep each organic extraction in a separate beaker.
- e. Following the last extraction, drain the remaining liquid from the separatory funnel into a tared beaker (inorganic fraction).
- f. Allow the organic fraction beakers to dry under a hood at room temperature.
- g. Evaporate the inorganic fraction in such a manner that the beaker contents do not become exposed to temperatures greater than 212°F.
- h. Dry weighed beakers containing a sample of the acetone, dichloromethane and a sample of distilled deionized water to check for blank weight.
- i. Desiccate organic, inorganic and blank beakers for at least 24 hours at room temperature in a desiccator containing silica gel. Weigh to a constant weight and report the results to the nearest 0.1 mg. Constant weight is defined in Section 4.3 of Method 5.

2. Back Half Acetone Rinse

- a. Dry the acetone rinse in a hood at room temperature.
- b. Desiccate and weigh the beaker to constant weight and record.

C. Reagents

1. Water

Use distilled deionized water in the impingers and to rinse all glassware.

2. Acetone

Use reagent grade, \leq 0.001 percent residue in glass bottles.

3. Dichloromethane

Use reagent grade, \leq 0.001 percent residue in glass bottles.

STATE OF WASHINGTON DEPARTMENT OF ECOLOGY
SOURCE TEST METHOD 5

PARTICULATE SAMPLING WITH A HEATED FILTER

1. Principle

Particulate emissions are collected with a heated filter and probe. The method is similar to EPA's Method 5.

2. Description

Stack gas samples are collected by traversing the stack with a heated probe and filter, with adjustments made to the sampling rate to maintain isokinetic conditions. Particulate is collected on the glass fiber filter. The volume sampled is determined by a totalizing dry gas meter. The particulate concentration is then determined by dividing the total particulate collected by the volume sampled.

3. Equipment and ReagentsEquipment:

The design specifications of the particulate sampling train are similar to those used by EPA Method 5 (Figure 1). Commercial models of this train are available.

- a. Nozzle - stainless steel, with sharp, tapered leading edge, and minimum size of 1/4 inch in diameter, if possible.
- b. Probe - Pyrex glass, with a heating system capable of maintaining a minimum gas temperature of 248°F at the exit end during sampling to prevent condensation from occurring. Thermocouple-to-glass junctures should be made near the probe exit to measure gas temperature indirectly. A stainless steel probe may be used when sample gases are low in SO₂ and HCl.
- c. Pitot tube - Type S, or equivalent, with coefficient within ±5% over the working range.
- d. Thermocouple or equivalent to read stack temperature within 1.5% of absolute temperature.
- e. Filter holder - Pyrex glass with heating system capable of maintaining a minimum temperature of 248°F.
- f. Impingers - Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith, or equivalent, design, modified by replacing the tip with a 0.5 inch I.D. glass tube extending one-half inch from the bottom of the flask. The second impinger is of the Greenburg Smith design, or equivalent, with the standard tip.

- g. Metering system - vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5°F , dry gas meter with 2% accuracy, and related equipment or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.
- h. Barometer to measure atmospheric pressure within ± 0.1 in. Hg.
- i. Wash bottle.
- j. Probe brush.
- k. Sample storage containers.
- l. Graduated cylinder - 250 ml.
- m. Analytical balance to weigh filter ± 0.1 mg.
- n. Balance 1000 gm capacity to measure ± 0.1 gm.
- o. Weighing dish.
- p. Oven to maintain temperature of 105°C to $\pm 5^{\circ}\text{C}$.
- q. Adhesive labels or labeling tape.
- r. Stopwatch or timer.
- s. Desiccation chamber with CaSO_4 or equivalent.

Reagents

- a. Distilled water.
- b. Crushed ice.
- c. Acetone.
- d. Filter - glass fiber filter or Pyrex glass without an organic binder, Reeve Angel .934 AH, or equivalent. Preweighed to the nearest 0.1 mg.
- e. Silica gel - indicating type or equivalent.

4. Procedures

Using the WDOE Methods to obtain representative points, measure the stack pressure, temperature, moisture and range of velocity head. On the first run the moisture may be estimated.

Preparation of Sampling Train: Place 100 ml of water in each of the first two impingers. Leave the third impinger empty and place at least 200 g. preweighed to the nearest 0.5 g. of silica gel in the fourth impinger. Set up the train, as in Figure 1. Leak-check the sampling train by plugging the nozzle and pulling 15 inches Hg (or the highest vacuum required during sampling.) A leak rate of less than 0.02 cfm is acceptable. Attach the probe and adjust the heater to provide a gas temperature of 223° to 273° F at the probe outlet. The nozzle, pitot tip, and the thermocouple tip should be at least 3/4 inch apart. Turn on the filter heating system. Adjust filter heating to maintain 248° F. +25° F. Place crushed ice around the impingers. Add more ice during the run to keep the temperatures of the gases, leaving the last impinger as low as possible and preferably at 70° F or less.

Train Operation: For each run, record necessary data; such as location, time, date, operator, nozzle diameter, filter number, initial impinger volume and relevant process information. At each sampling point, and when significant changes in operating conditions necessitate, take readings such as traverse point, sampling time, stack temperature, sample box temperature, and temperature leaving last impinger. Record temperatures within at least 5° F and pressures $\pm 10\%$. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Sample for at least two minutes at each traverse point. Sampling time should be the same for each point, unless some points represent more area than others. In such a case, sample proportionately longer at these points. Adjust the sampling rate to isokinetic as necessary, usually at each traverse point. Turn off the pump at the conclusion of the run and record the final readings. Remove the probe and nozzle from the stack and perform a post-test leak-check, while the sampling train is at the sampling temperature, using the same procedure as during the pre-test leak-check.

Sample Recovery: Exercise care in moving the collection train from the test site to the sample recovery area to minimize the loss of collected samples or the gain of extraneous particulate matter. Place the samples in three containers as follows:

First container: Remove the filter from its holder; place it in this container along with any loose particulate matter brushed from inside the probe and front half of the filter holder, then seal the container.

Second container: Place the water and acetone washings from all sample-exposed surfaces prior to the filter and seal. Use a razor blade, brush, rubber policeman, or similar tool to loosen adhering particles. The acetone wash may be excluded if the particulate is water soluble, such as sodium sulfate.

Third container: If the silica gel was not weighed in the impinger, transfer the silica gel from the fourth impinger to the original container and seal.

Impinger solution: Determine the amount of condensate collected in the impingers either by measuring the volumes of liquid or by weight.

NOTE: Some local air pollution control agencies may require that the material collected in the impingers be analyzed for particulate. For this reason, the agency should be contacted before testing starts to determine how the impinger material is to be treated.

5. Analysis and Calculation

Analysis:

Separately dry the filter catch and the probe wash at 105°C to constant weight, and weigh on an analytical balance to the nearest 0.1 mg.

Calculations:

Add the total of the filter catch and the probe wash to get the particulate weight. The stack flow rate should be calculated from the velocity data. Determine the stack moisture by the moisture collected and by saturation if the gas stream contained entrained water droplets. Use the lower of the two values. Then calculate the particulate emission rate from the flow rate and the concentration. For a sample calculation, see WDOE Source Test Manual, Appendix B.

6. Calibration

The dry gas meters should be calibrated against a wet test meter. If the correction is greater than 2%, adjust the dry gas meter until the correction is less than 2%, or use a correction factor.

Calibrate temperature gauges against a standard thermometer for at least two points; i.e., an ice bath and boiling water. The gauge should read within 1.5% of the absolute temperature.

Barometers should be calibrated against a mercury barometer.

Calibrate pitot tubes against a standard pitot over the normal working range.

Source Test Method 5
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7. References

- a. Martin, Robert, "Construction Detail of Isokinetic Source Sampling Equipment," ERPTD -0581.
- b. Rom, Jerome, "Maintenance, Calibration and Operation of Isokinetic Source Sampling Equipment," EPA, APTD 0576.
- c. Smith, W.S., et al. "A Method of Interpreting Stack Sampling Data," 63rd. Annual Meeting of the APCA, St. Louis, Mo., June 1970.
- d. Smith, W. S., et al. Stack Sampling "Improved Simplified with New Equipment," APCA Paper No. 67-119, 1967.
- e. Federal Register, Vol 36, No. 246, 1971, p. 24888.

December 1, 1976

STATE OF WASHINGTON DEPARTMENT OF ECOLOGY

SOURCE TEST METHOD 9A

VISUAL DETERMINATION OF OPACITY FOR A THREE MINUTE STANDARD

1. Principle

The opacity of emissions from stationary sources is determined visually by a qualified observer.

2. Procedure

The observer must be certified according to the "Criteria for Smoke and Opacity Training School 1970-1971" of the Oregon-Washington Air Quality Committee, except that the average error not exceed 7.5%, all readings are made in percent opacity and no void readings (except in the case of operator error).

The qualified observer shall stand at a distance sufficient to provide a clear view of the emissions with the sun oriented in the 140° sector to his back. Consistent with maintaining the above requirement, the observer shall, as much as possible, make his observations from a position such that his line of vision is approximately perpendicular to the plume direction, and when observing opacity of emissions from rectangular outlets (e.g. roof monitors, open baghouses, noncircular stacks), approximately perpendicular to the longer axis of the outlet. The observer's line of sight should not include more than one plume at a time when multiple stacks are involved, and in any case, the observer should make his observations with his line of sight perpendicular to the longer axis of such a set of multiple stacks (e.g. stub stacks on baghouses).

The observer shall record the name of the plant, emission location type of facility, observer's name and affiliation, and the date on a field data sheet. The time, estimated distance to the emission location, approximate wind direction, estimated wind speed, description of the sky condition (presence and color of clouds), and plume background are recorded on a field data sheet at the time opacity readings are initiated and completed.

The observer should make note of the ambient relative humidity, ambient temperature, the point in the plume that the observations were made, the estimated depth of the plume at the point of observation, and the color and condition of the plume. It is also helpful if pictures of the plume are taken.

Opacity observations shall be made at the point of greatest opacity in that portion of the plume where condensed water vapor is not present. The observer shall not look continuously at the plume, but instead shall observe the plume momentarily at 15-second intervals.

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DOE Source Test Method 9A

Visual Determination of Opacity for a Three Minute Standard

When condensed water vapor is present within the plume as it emerges from the emission outlet, opacity observations shall be made beyond the point in the plume at which condensed water vapor is no longer visible.

When water vapor in the plume condenses and becomes visible at a distinct distance from the emission outlet, the opacity of emissions should be evaluated at the emission outlet prior to the condensation of water vapor and the formation of the steam plume.

Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on an observational record sheet. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period.

3. Analysis

The opacity is determined by the highest 13 observations in any consecutive 60-minute period.

4. References

- (1) Federal Register, Vol. 36, No. 247, Page 24895, Dec. 23, 1971.
- (2) "Criteria for Smoke and Opacity Training School 1970-1971"
Oregon-Washington Air Quality Committee.
- (3) "Guidelines for Evaluation of Visible Emissions." EPA 340/1-75-007.
Environmental Protection Agency, Washington, D.C., April, 1975.

by this Subpart shall constitute a violation of the applicable emission standard of this Subpart.

§ 63.454 Recordkeeping.

(a) The owner or operator shall record and meet the recordkeeping requirements for § 63.10 (a), (b), and (c) for the monitoring parameters specified in § 63.453.

(b) The owner or operator shall record the monitoring parameters specified in § 63.453 and meet the requirements specified in paragraph (a) of this section for any emission point or process wastewater stream that becomes subject to the standards in this Subpart due to an increase in the flow, concentration, or mass parameters equal to or greater than the limits specified in § 63.444(a), § 63.445(a), or § 63.446 (a) or (h).

§ 63.455 Reporting.

(a) Each owner or operator of a source subject to this subpart shall submit the reports listed in paragraphs (a)(1) through (a)(5) of this section.

(1) An Initial Notification described in § 63.9 (a) through (d) and § 63.10(f).

(2) A Notification of Performance Tests specified in § 63.7 and § 63.9(g).

(3) A Notification of Compliance Status specified in § 63.9(h).

(4) Exceedance Reports specified in § 63.10(e)(2) (i) through (v) and (viii).

(i) If actions taken by an owner or operator during a startup, shutdown, or malfunction of an affected source (including actions taken to correct a malfunction) are not completely consistent with the procedures specified in the source's startup, shutdown, and malfunction plan specified in § 63.6(e)(3), the owner or operator shall state such information in the quarterly report. The startup, shutdown, and malfunction report shall consist of a letter, containing the name, title, and signature of the responsible official who is certifying its accuracy, that shall be submitted to the Administrator, and

(ii) If the seals on the secured enclosure openings specified in § 63.453(a) are broken, the duration of the event and an explanation of the reason for breaking the seal shall be included in the exceedance report.

(iii) Separate exceedance reports are not required if the information is included in the quarterly report in paragraph (a)(5) of this section.

(5) A quarterly summary report specified in § 63.10(e)(3). The summary report shall be entitled "Summary Report—Gaseous Excess Emissions and Continuous Monitoring System Performance." The quarterly report must contain any information for the Exceedance Report in paragraph (a)(4) of

this section if an Exceedance Report is required.

(b) The owner or operator shall meet the requirements specified in paragraph (a) of this section for any emission point or process wastewater stream that becomes subject to the standards in this Subpart due to an increase in the flow, concentration, or mass parameters equal to or greater than the limits specified in § 63.444(a), § 63.445(a), and § 63.446 (a) and (h).

§ 63.456 Delegation of authority.

(a) In delegating implementation and enforcement authority to a State under section 112(d) of the Act, the authorities contained in paragraph (b) of this section shall be retained by the Administrator and not transferred to a State.

(b) Authorities which will not be delegated to States: The authority conferred in § 63.6(g) will not be delegated to any State.

§ 63.457 [Reserved]

§ 63.458 [Reserved]

§ 63.459 [Reserved]

3. It is proposed that Appendix A to part 63 be amended by adding Method 308 to read as follows:

Appendix A to Part 63—Test Methods

Method 308—Procedure for Determination of Methanol Emission from Stationary Sources

1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of methanol emissions from specified stationary sources.

1.2 Principle. A gas sample is extracted from the sampling point in the stack. The methanol is collected in deionized distilled water and adsorbed on silica gel. The sample is returned to the laboratory where the methanol in the water fraction is separated from other organic compounds with a gas chromatograph (GC) and is then measured by a flame ionization detector (FID). The fraction adsorbed on silica gel is extracted with an aqueous solution of n-propanol and is then separated and measured by GC/FID.

2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 308-1 and component parts are discussed below.

2.1.1 Probe. Teflon, approximately 6-mm outside diameter.

2.1.2 Impingers. Two 30-mL midjet impingers. The impingers must be connected in series with leak-free glass

connectors. Silicone grease may not be used to lubricate the connectors.

2.1.3 Adsorbent Tube. Glass tubes packed with the required amount of the specified adsorbent.

2.1.4 Valve. Needle valve, to regulate sample gas flow rate.

2.1.5 Pump. Leak-free diaphragm pump, or equivalent, to pull gas through the train. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.1.6 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of about 1000 cc/min.

2.1.7 Volume Meter. Dry gas meter (DGM), sufficiently accurate to measure the sample volume to within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature gauge (dial thermometer, or equivalent) capable of measuring temperature accurately to within 3°C (5.4°F).

2.1.8 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm (0.1 in.) Hg. See the note in Method 5 (40 CFR Part 60, Appendix A), Section 2.1.9.

2.1.9 Vacuum Gauge and Rotameter. At least 760-mm (30-in.) Hg gauge and 0— to 40-cc/min rotameter, to be used for leak-check of the sampling train.

2.2 Sample Recovery.

2.2.1 Wash Bottles. Polyethylene or glass, 500-mL, two.

2.2.2 Sample Vials. Glass 40-mL with Teflon-lined septa, to store impinger samples (one per sample).

2.3 Analysis.

2.3.1 Gas Chromatograph. GC with an FID, programmable temperature control, and heated liquid injection port.

2.3.2 Pump. Capable of pumping 100 mL/min. For flushing sample loop.

2.3.3 Flow Meter. To monitor accurately sample loop flow rate of 100 mL/min.

2.3.4 Regulators. Two-stage regulators used on gas cylinders for GC and for cylinder standards.

2.3.5 Recorder. To record, integrate, and store chromatograms.

2.3.6 Syringes. 1.0- and 10-microliter size, calibrated, for injecting samples.

2.3.7 Tubing Fittings. Stainless steel, to plumb GC and gas cylinders.

2.3.8 Vials. Two 5.0-mL glass vials with screw caps fitted with Teflon-lined septa for each sample. Also one for each standard for adsorbent tube samples.

2.3.9 Vials. Glass 40-mL with Teflon-lined septa, to prepare calibration standards (one per standard) for impinger samples.

3. Reagents

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

3.1 Sampling.

3.1.1 Water. Deionized distilled to conform to ASTM Specification D 1193-77, Type J. At the option of the analyst, the KMnO₄ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.2 Silica Gel. Deactivated chromatographic grade 20/40 mesh silica gel packed in glass adsorbent tubes. The silica gel is packed in two sections. The front section contains 520 mg of silica gel, and the back section contains 260 mg.

3.2 Analysis.

3.2.1 Water. Same as 3.1.1.

3.2.2 *n*-Propanol, 10 Percent. Mix 10 mL of *n*-propanol with 90 mL of water.

3.2.3 Methanol Standards For Impinger samples. Prepare a series of methanol standards by injecting 0, 10, 20, 30, and 40 µg of methanol respectively into five 40-mL glass vials filled with water and capped with Teflon septa.

3.2.4 Methanol Standards for Adsorbent Tube Samples. Prepare a series of methanol standards by injecting 0, 10, 20, 30, and 40 µg of methanol respectively into five 5-mL glass vials capped with Teflon-lined septa and containing 3 mL of a 10% *n*-propanol solution.

3.2.5 GC Column. Capillary column, 30 meters long with an ID of 0.53 mm, coated with DB 624 to a film thickness of 3.0 microns, or an equivalent column.

3.2.6 Helium. Ultra high purity.

3.2.7 Hydrogen. Zero Grade.

3.2.8 Oxygen. Zero grade.

4. Procedure

4.1 Sampling.

4.1.1 Preparation of Collection

Train. Measure 20 mL of water into the midget impinger. The adsorbent tube must contain 520 mg of silica gel in the front section and 260 mg of silica gel in the backup section. Assemble the train as shown in Figure 308-1. Place crushed ice and water around the impinger.

4.1.2 Leak Check. A leak-check prior to the sampling run is optional; however, a leak-check after the sampling run is mandatory. The leak-check procedure is as follows:

Temporarily attach a suitable (e.g., 0- to 40-cc/min) rotameter to the outlet of

the DGM, and place a vacuum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm (10 in.) Hg, and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 percent of the average sampling rate is acceptable.

Note: Carefully release the probe inlet plug before turning off the pump.

4.1.3 Sample Collection. Record the initial DGM reading and barometric pressure. To begin sampling, position the tip of the Teflon tubing at the sampling point, connect the tubing to the impinger, and start the pump. Adjust the sample flow to a constant rate of approximately 200 mL/min as indicated by the rotameter. Maintain this constant rate (±10 percent) during the entire sampling run. Take readings (DGM, temperatures at DGM and at impinger outlet, and rate meter) at least every 5 minutes. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20°C (68°F) or less. At the conclusion of each run, turn off the pump, remove the Teflon tubing from the stack, and record the final readings. Conduct a leak-check as in Section 4.1.2. (This leak-check is mandatory.) If a leak is found, void the test run or use procedures acceptable to the Administrator to adjust the sample volume for the leakage.

4.2 Sample Recovery.

4.2.1 Impingers. Disconnect the impingers. Pour the contents of the midget impingers into a leak-free polyethylene bottle marked for shipment. Rinse the two midget impingers and the connecting tubes with water, and add the washings to the same storage container. Mark the fluid level. Seal and identify the sample container.

4.2.2 Adsorbent Tubes. Seal the silica gel adsorbent tubes and place them in an ice chest for shipment to the laboratory.

4.3 Sample Analysis.

4.3.1 Gas Chromatograph Operating Conditions.

4.3.1.1 Injector. Configured for capillary column, splitless, 200°C.

4.3.1.2 Carrier. Helium at 10 mL/min.

4.3.1.3 Oven. Initially at 45°C for 3 minutes; then raise by 10°C to 70°C; then raise by 70°C/min to 200°C.

4.3.2 Impinger Sample.

4.3.2.1 Note level of liquid in container, and confirm whether any sample was lost during shipment; note this on analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

4.3.2.2 Transfer the contents of the storage container to a 100-mL volumetric flask, and dilute to exactly 100 mL with water.

4.3.2.3 Inject 1 µl of the diluted sample into the gas chromatograph. Repeat the injection until the responses of two successive injections agree within 5%. If the sample response is above that of the highest calibration standard, either dilute the sample until it is in the measurement range of the calibration line or prepare additional calibration standards. If the sample response is below that of the lowest calibration standard, prepare additional calibration standards. If additional calibration standards are prepared, there shall be at least two which bracket the response of the sample. These standards should produce approximately 80% and 120% of the response of the sample.

4.3.3 Silica Gel Adsorbent Sample.

4.3.3.1 Preparation of Samples.

Extract the front and backup sections of the adsorbent tube separately. With a file score the glass adsorbent tube in front of the first section of silica gel. Break the tube open. Remove and discard the glass wool. Transfer the first section of the silica gel to a 5-mL glass vial and stopper the vial. Remove the spacer between the first and second section of the adsorbent tube and discard it. Transfer the second section of silica gel to a separate 5-mL glass vial and stopper the vial.

4.3.3.2 Desorption of Samples. Add 3 mL of the 10% *n*-propanol solution to each of the stoppered vials and shake or vibrate the vials for 30 minutes.

4.3.3.3 Inject 1 µl of the diluted sample into the gas chromatograph. Repeat the injection until the responses of two successive injections agree within 5%. If the sample response is above that of the highest calibration standard, either dilute the sample until it is in the measurement range of the calibration line or prepare additional calibration standards. If the sample response is below that of the lowest calibration standard, prepare additional calibration standards. If additional calibration standards are prepared, there shall be at least two which bracket the response of the sample. These standards should produce approximately 80% and 120% of the response of the sample.

5. Calibration

5.1 Metering System.

5.1.1 Initial Calibration.

5.1.1.1 Before its initial use in the field, first leak-check the metering system (drying tube, needle valve, pump, rotameter, and DGM) as follows: Place a vacuum gauge at the inlet to the drying tube, and pull a vacuum of 250

mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end.

5.1.1.2 Next, remove the drying tube, and calibrate the metering system (at the sampling flow rate specified by the method) as follows: Connect an appropriately sized wet test meter (e.g., 1 liter per revolution) to the inlet of the drying tube. Make three independent calibrations runs, using at least five revolutions of the DGM per run. Calculate the calibration factor, Y (wet test meter calibration volume divided by the DGM volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any Y-value deviates by more than 2 percent from the average, the metering system is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

5.1.2 **Post-Test Calibration Check.** After each field test series, conduct a calibration check as in Section 5.1.1 above, except for the following variations: (a) The leak-check is not to be conducted, (b) three, or more revolutions of the DGM may be used, and (c) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor (determined in Section 5.1.1), then the DGM volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in Section 5.1.1, and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

5.2 **Thermometers.** Calibrate against mercury-in-glass thermometers.

5.3 **Rotameter.** The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

5.4 **Barometer.** Calibrate against a mercury barometer.

5.5 **Gas Chromatograph.**

5.5.1 **Initial Calibration.** Inject 1 μ l of each of the standards prepared in Section 3.3.3 into the GC and record the response. Repeat the injections for each standard until two successive injections agree within 5%. Using the mean response for each calibration standard, prepare a linear least squares equation relating the response to the mass of methanol in the sample. Perform the calibration before analyzing each set of samples.

5.5.2 **Continuing Calibration.** At the beginning of each day, analyze the mid-

level calibration standard as described in Section 5.5.1. The response from the daily analysis must agree with the response from the initial calibration within 10%. If it does not the initial calibration must be repeated.

6. Quality Assurance

6.1 **Applicability.** When the method is used to analyze samples to demonstrate compliance with a source emission regulation, an audit sample must be analyzed, subject to availability.

6.2 **Audit Procedure.** Analyze an audit sample with each set of compliance samples. Concurrently analyze the audit sample and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for the compliance samples and the EPA audit sample.

6.3 **Audit Sample Availability.** Audit samples will be supplied only to enforcement agencies for compliance tests. Audit samples may be obtained by writing: Source Test Audit Coordinator (MD-77B), Quality Assurance Division, Atmospheric Research and Exposure Assessment Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711, or by calling the Source Test Audit Coordinator (STAC) at (919) 641-7834. The audit sample request must be made at least 30 days prior to the scheduled compliance sample analysis.

6.4 **Audit Results.** Calculate the audit sample concentration according to the calculation procedure provided in the audit instructions included with the audit sample. Fill in the audit sample concentration and the analyst's name on the audit response form included with the audit instructions. Send one copy to the EPA Regional Office or the appropriate enforcement agency and a second copy to the STAC. The EPA Regional Office or the appropriate enforcement agency will report the results of the audit to the laboratory being audited. Include this response with the results of the compliance samples in relevant reports to the EPA Regional Office or the appropriate enforcement agency.

7. Calculations

7.1 Nomenclature

E=Mass emission rate of methanol, kg/hr (lb/hr).

M_a =Mass of methanol in the front and back half of the adsorbent tube, μ g.

M_i =Mass of methanol in the impinger portion of the sample train, μ g.

M_{tot} =Total mass of methanol collected in the sample train, μ g.

P_{bar} =Barometric pressure at the exit orifice of the DGM, mm Hg (in. Hg).

P_{std} =Standard absolute pressure, 760 mm Hg (20.92 in. Hg).

Q_{std} =Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/lir).

T_m =Average DGM absolute temperature, K (R).

T_{std} =Standard absolute temperature, 293 K (528 R).

V_a =Volume of sample aliquot titrated, ml.

V_m =Dry gas volume as measured by the DGM, dcm (dcf).

$V_{m(std)}$ =Dry gas volume measured by the DGM, corrected to standard conditions, dscm (dscf).

7.2 **Mass of Methanol.** Calculate the total mass of methanol collected in the sampling train using Equation 308-1.

$$M_{tot} = M_i + M_a \quad \text{Equation 308-1}$$

7.3 **Dry Sample Gas Volume, Corrected to Standard Conditions.** Calculate the volume of gas sampled at standard conditions using Equation 308-2.

$$V_m(\text{std}) = \frac{V_m Y T_{std} P_{bar}}{T_m P_{std}} \quad \text{Equation 308-2}$$

7.4 **Mass Emission Rate of Methanol.** Calculate the mass emission rate of methanol using Equation 308-3.

$$E = \frac{M_{tot} Q_{std}}{V_m(\text{std})} \quad \text{Equation 308-3}$$

8. Bibliography

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3. Westlin, P. R. and R. T. Shigehara. Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards. Source Evaluation Society Newsletter. 3(1):17-30. February 1978.

4. Yu, K. K. Evaluation of Moisture Effect on Dry Gas Meter Calibration. Source Evaluation Society Newsletter. 5(1):24-28. February 1980.

5. NIOSH Manual of Analytical Methods, Volume 2. U. S. Department of Health and Human Services National Institute for Occupational Safety and Health. Center for Disease Control. 4676 Columbia Parkway, Cincinnati, Ohio 45226. May be available from the



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May 4, 2001

Mr. Rick Blackmarr
Pliant Corporation
8039 S. 192nd Street
Kent, Washington 98032

Dear Mr. Blackmarr,

This letter serves as Am Test-Air Quality, LLC's *source test plan* for emissions testing of the 5,000 cfm Dec-E-Tec catalytic oxidizer at Pliant Corporation's facility in Kent, Washington. This thermal oxidizer controls volatile organic compound (VOC) emissions from press #207. The testing will be conducted to determine compliance with Puget Sound Clean Air Agency (PSCAA) requirements for VOC destruction efficiency. For these destruction efficiency tests, Am Test will measure VOC emissions at the oxidizer inlet and outlet. This testing is scheduled for June 20, 2001. (Testing of the #210 press and Smith oxidizer will follow on June 21, 2001).

Testing and analysis procedures to be used for this project are presented in the July 1, 2000 edition of the Environmental Protection Agency (EPA) document Title 40, Code of Federal Regulations, Part 60 (40 CFR 60), Appendix A, Methods 1, 2, 3A, and 4 and in the July 1, 2000 edition of the EPA document Title 40, Code of Federal Regulations, Part 60 (40 CFR 60), Appendix A, and in Part 63 (40 CFR 63), Method 308. Details for the methods to be used are described below:

EPA Methods 1 and 2 - Exhaust Gas Flow Determination

Methods 1 and 2 will be performed to measure the gas velocity and temperature for calculating the volumetric flow rate. Stack gas velocity will be measured using a pitot tube connected to a pressure measurement device (e.g., magnehelic gauge, incline manometer, Shortridge[®] Instruments, Inc. Airdata Multimeter ADM-850, which is a special low flow pressure measurement device, etc.). The pitot tube lines will be leak checked prior to sampling. Stack gas temperature and other necessary temperatures will be measured using K-type thermocouple probes attached to digital thermocouple indicators. Appropriate pressure and temperature measurement device calibration information will be included in the final report. Stack condition measurements will be

made prior to collecting a sample, including measurements of velocity, temperature and a check for cyclonic flow in the duct. Stack gas velocity and temperature measurements will be performed during all test periods.

EPA Method 3A - Molecular Weight Measurements

Method 3A will be performed at the outlet of the thermal oxidizer to measure concentrations of carbon dioxide and oxygen, as needed to calculate the outlet stack gas molecular weight. The inlet gas stream is composed of essentially ambient air, with levels of approximately 0.1% carbon dioxide and 20.9% oxygen. These values will be used to calculate the molecular weight of the inlet gas stream.

EPA Method 4 - Moisture Determination

Method 4 will be performed at the outlet of the thermal oxidizer to measure the moisture content of the stack gas for calculating the volumetric flow rate and molecular weight on a wet basis. The impingers for each Method 4 sample train will be weighed before and after the run. Moisture content of the oxidizer inlet will be determined using wet bulb and dry bulb temperatures and psychrometry.

EPA Method 308 (modified) - Alcohols and Acetates

Alcohol and acetate emissions will be collected using a modified version of Method 308 from 40 CFR 63, Appendix A titled "Procedure for Determination of Methanol Emissions from Stationary Sources." The sample train will contain four (4) midget impingers. The first two (2) impingers will contain 20 milliliters (mL) of Type II deionized (DI) water. The fourth impinger will be empty. The last impinger will contain silica gel. No silica gel adsorbent tube will be included in the sample train. Sampling at the oxidizer inlet will be performed at a sample rate of approximately 200 mL/minute. The oxidizer outlet will be sampled at a rate of approximately 1000 mL/minute (due to the expected lower VOC concentrations at the oxidizer outlet). Three (3) one hour test runs will be performed simultaneously at the thermal oxidizer inlet and outlet. The water from the first three impingers will be recovered into zero headspace sample vials, and analyzed by gas chromatography with a flame ionization detector (GC/FID) for ethyl alcohol, isopropyl alcohol, propyl alcohol, isopropyl acetate, and propyl acetate.

Silica gel adsorbent tubes will not be used in this modified Method 308 sample train. Because each of the target VOCs are polar compounds, these alcohol and acetate



compounds will be collected in the chilled water impingers, meaning the silica gel tubes are not necessary. Silica gel tubes were used in the June 1997 tests at two similar Pliant Corporation sources, and the results were below detection limits on each of the silica gel tubes.

It will be the responsibility of Pliant Corporation personnel to provide Am Test with process information for inclusion in our final report. This process data will include the catalytic oxidizer operating temperature, and the pounds per hour (lb/hr) of solvent use for each individual compound (required during the capture efficiency testing).

Within 45 days after the tests are completed, Am Test-Air Quality, LLC will submit four (4) copies of a final report to Pliant Corporation. The report will be a formal, bound document containing information about the source, dates and times of each test, details of sampling and analysis procedures, quality assurance procedures, and a discussion of how the results were calculated, along with example calculations. Results will be presented in concentration units (e.g., parts per million (ppm)) and emission rate units (e.g. pounds per hour (lb/hr)). Pliant Corporation will be responsible for sending a copy of the report to PSCAAA.

Am Test-Air Quality, LLC looks forward to performing these tests. Please call our office at (425) 222-7746 if we may provide additional information or clarification.

Sincerely,
Am Test-Air Quality, LLC

A handwritten signature in black ink, appearing to read "K. Steven Mackey".

K. Steven Mackey
Sr. Project Engineer

[SC\ksm\c:\mydoc\2001testplans\Pliant2001TP#2]