METHOD 0020

SOURCE ASSESSMENT SAMPLING SYSTEM (SASS)

1.0 PRINCIPLE AND APPLICATION

    1.1 Principle

        1.1.1   Particulate and semivolatile organic materials are withdrawn from a source at a constant rate near isokinetic conditions and are collected in a multicomponent sampling train.

        1.1.2   Three heated cyclones and a heated high-efficiency fiber filter remove and collect the particulate material from the sample and a packed bed of porous polymeric resin adsorbs the condensible organic vapors.

        1.1.3   Chemical analyses of the sample are conducted to determine the concentration and identity of the semivolatile organic species and gravimetric determinations are performed to approximate particulate emissions.

    1.2   Application: This method is applicable to the preparation of semiquantitative estimates (within a factor of three) of the amounts and types of semivolatile organic and particulate materials that are discharged from incineration systems.

2.0 APPARATUS

    2.1   Sampling Train: A schematic of the sampling train used in this method is given in Figure 1. This sampling train configuration is that of the Source Assessment Sampling System (SASS), as supplied by the manufacturer. Basic operating and maintenance procedures are described in the "Operating and Service Manual: Source Assessment Sampling System" supplied on purchase of the sampling system (Blake, 1977).   Users should refer to this document and adopt, but not limit themselves to, its operating and maintenance procedures. The SASS train components and specifications are detailed below.

        2.1.1   **Probe nozzles:** The probe nozzles are constructed of Type 316 seamless stainless steel tubing and have sharp leading edges. The nozzles are a hybrid elbow/buttonhook design, obtainable in diameters ranging from 0.31 to 1.91 cm (1/8 to 3/4 in.), and are interchangeable. Each nozzle should be calibrated according to the procedure outlined in Paragraph 7.2 of this method.
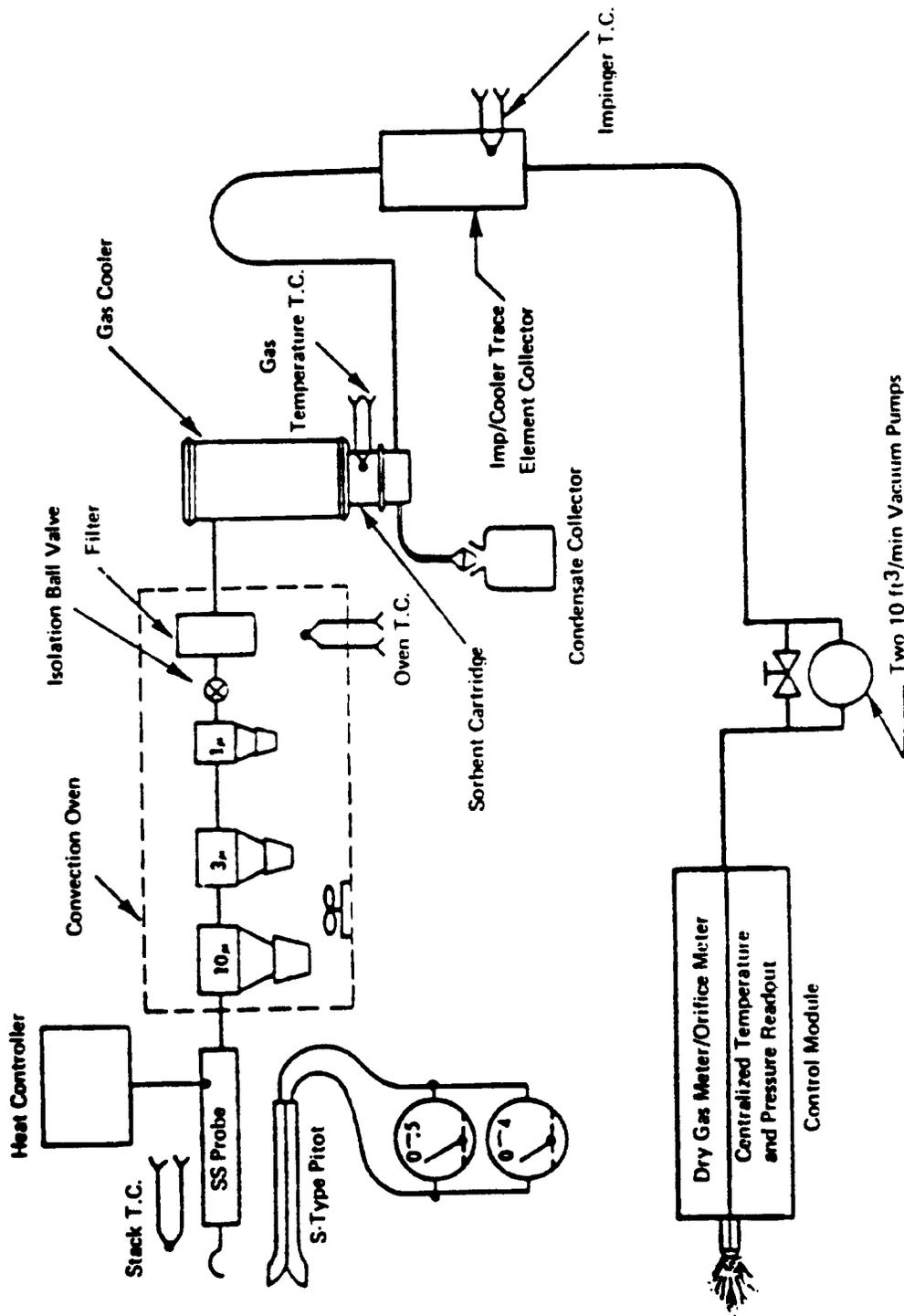
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Figure 1. SASS Schematic Diagram.

2.1.2   **Probe Liner:**

2.1.2.1 The probe liner is also constructed of Type 316 seamless stainless steel tubing; attached to the liner is a proportional temperature controller capable of maintaining the liner surface temperature at 204 $\pm$ 20°C (400 $\pm$ 36°F) during sampling. The use of the proportional controller to control the liner surface temperature at the control module is preferred because the oven often cannot be reached for adjustment during sampling.

2.1.2.2 It should be noted that the measurement of the probe liner surface temperature is not an accurate measurement of the internal gas stream temperature, which is the temperature of interest. This source of error is caused by the temperature gradient that exists between the inner and outer walls of the liner. Monitoring of the actual gas stream temperature is impractical with the SASS trains as presently constructed. It is suggested that a one-time calibration be conducted in which the internal gas stream temperature is compared to the liner surface at various temperatures and at the standard SASS flow rate of 4.0 scfm.

2.1.2.3 The probe and probe liner can withstand points up to 370°C (700°F), at which temperature they will soften. However, stack temperatures greater than 288°C (550°F) may result in gas temperatures at the 10-um cyclone inlet greater than the recommended 204°C (400°F) and hence require the use of a special water- or forced-air-cooled probe.

2.1.3   **Pitot tubes:** The pitot tubes are Type S, designed to meet the specifications of EPA Method 2 (see Reference below); these are attached to the probe sheath to allow constant monitoring of the stack gas velocity. The point of attachment to the sheath is such that the impact (high pressure) opening plane of the pitots is level with or above the sampling nozzle entry plane, as required by Method 2, to eliminate nozzle interference in velocity measurements. If calibration is not required, the pitot tubes are assigned a nominal coefficient of 0.84, as described in the calibration section of this method.

2.1.4   **Differential pressure gauges:** Three Magnehelic-type gauges are used. One gauge (0 to 0.5 in. H_2O) monitors the pressure drop across the orifice meter (ΔH_i); the other two gauges (0 to 0.5 and 0 to 4.0 in. H_2O) are connected in parallel and indicate the pressure differential across the pitot tubes used for measuring stack gas velocity.

2.1.5   **Filter holder and filter support:** The filter holder and filter support screen are constructed of Type 316 stainless steel with a

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Teflon gasket providing an airtight seal around the circumference. The holder is attached immediately to the outlet of the 1-um cyclone or the cyclone bypass.

2.1.6 **Cyclone/Filter heating system:** The cyclone/filter heating system is an insulated double-walled oven, capable of maintaining the temperature in the area of the cyclones and filter holder around the recommended 204°C (400°F). A chromel-alumel thermocouple for temperature sensing allows feedback control of the temperature to within approximately 10%.

2.1.7 **Cyclone train:** The cyclone train consists of three cyclone separators in series, having nominal particle-size cutoff diameters of 10, 3, and 1 um respectively. The material of construction is Type 316 stainless steel with Teflon gaskets sealing the hoods and collector cups. The compact design of the 10-um cyclone is achieved by incorporating flow-interrupting vanes in the collection cup.

2.1.8 **Organic module:** The organic module consists of a thin-film heat exchanger/gas cooler, a sorbent cartridge, and a condensate collection trap. The temperature of the heat exchanger fluid is regulated by activating an immersion heater or routing the coolant through another heat exchanger in the impinger ice water bath. Water from the impinger bath is continually circulated through the inner reservoir of the gas cooler for additional cooling capacity. The sorbent cartridge encloses the polymeric adsorbent bed in a cylinder covered on both ends by 80-mesh, Type 316 stainless steel wire cloth. The cartridge holds approximately 150 grams of XAD-2 adsorbent resin. Condensed moisture from the gas stream is collected in a reservoir located directly beneath the packed sorbent bed. The drain valve of the reservoir should be coupled with a Teflon line to an appropriately sized (1- to 5-liter) glass storage container, as the capacity of the reservoir will typically be exceeded during a run.

2.1.9 **Impinger train:** The four impingers have a capacity of approximately 3 liters each and are constructed of pyrex glass. The caps are Teflon with stainless steel fittings and the impingers are interconnected by flexible Teflon or stainless steel tubing. The first two impingers are equipped with splash guards to minimize fluid carryover and the last impinger with a thermocouple mounted in the cap for monitoring the impinger train exit gas temperature.

2.1.10 **Pump/Metering system:** Two leak-free vane-type vacuum pumps connected in parallel are used to maintain the 4.0-scfm flow in the sampling system. Vacuum and differential pressure gauges, thermocouples capable of measuring temperature to within 3°C (5.4°F) and a dry gas meter capable of measuring volume to within 2% are supplied as the other necessary components for maintaining isokinetic sampling rates.

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2.1.11 **Barometer:** An aneroid barometer, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.5 in. Hg), is required, unless the barometric reading is obtained from a nearby National Weather Station; the station value (i.e., the absolute barometric pressure) must be corrected for elevation differences between the weather station and the sampling point. The corrected value should reflect a decrease of 2.5 mm Hg (0.1 in. Hg) per 30-m (100-ft) elevation increase, and vice versa for elevation decrease. (See Paragraph 7.7).

2.1.12 **Gas density determination apparatus:** The length of a SASS run is typically sufficient to determine the average gas stream density during the run. EPA Method 3 should be consulted for detailed specifications for an integrated fixed gas sampling system. Analysis of the collected samples should be performed with an ORSAT analyzer or a GC/TCD system outfitted specifically for this purpose.

2.1.13 **Calibration/Field preparation log:** For documentation of calibration and preparation procedures, a permanently bound laboratory notebook is recommended, in which carbon copies are made of the data as they are being recorded. The carbon copies should be detachable and used only for separate storage in the test program archives.

2.2 <u>Sample recovery</u>:

2.2.1 **Probe liner brush:** The brush must have nylon bristles, a stainless steel wire handle, and extensions of stainless steel, Teflon, or other inert material. The combined extensions must be equal to or greater than the length of the probe.

2.2.2 **Probe nozzle brush:** The brush must have nylon bristles and a stainless steel wire handle, and be properly sized and shaped for cleaning the inner surfaces of the nozzle.

2.2.3 **Cyclone and filter holder brushes:** The brushes must have nylon bristles and a stainless steel wire handle, and be properly sized for cleaning the inner walls of these components. It is strongly recommended that a separate brush be used for sample recovery from each of these components to avoid cross contamination of one particle size fraction by another.

2.2.4 **Wash bottles:** Three are needed. Teflon or glass is required to avoid contamination of organic solvents; Teflon is preferred because it is unbreakable.

2.2.5 **Glass sample storage containers:** The containers must be chemically resistant, borosilicate glass bottles, 500-mL or 1,000-mL. Screw-cap liners should be Teflon or constructed so as to be leak-free and resistant to chemical attack by organic recovery solvents (narrow-mouth glass bottles have been found to exhibit less tendency toward leakage).

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2.2.6 **Petri dishes:** These must be glass and sealed around the circumference with Teflon tape for storage and transport of filter samples.

2.2.7 **Graduated cylinder and triple-beam balance:** to measure condensed water to the nearest 1 mL or 0.5 g. Graduated cylinders must have subdivisions no greater than 2 mL. Equipment made of glass must be used for measuring the volume of any solution that will be subject to organic analysis. Laboratory triple-beam balances must be capable of weighing to $\pm$0.5 g or better.

2.2.8 **High-density linear polyethylene (HDLP) storage containers:** These are used for storage of the impingers.

2.2.9 **Plastic storage containers:** Airtight containers are necessary for storage of silica gel.

2.2.10 **Funnels:** Glass funnels must be used in recovering samples for organic analysis. Glass or plastic funnels may be used in other processes but care must be taken to segregate the two types.

3.0 REAGENTS AND MATERIALS

3.1 <u>Filters</u>: Glass fiber filters, 15.24 cm (6.0 in.) in diameter without organic binder, exhibiting 99.95% efficiency (<0.05% penetration) on 0.3-micron dioctyl phthalate smoke particles, conforming to the specifications outlined in ASTM Standard Method D2986-71. Test data from the supplier's quality control program are sufficient for this purpose. The filter material must also be unreactive to SO_2 and SO_3.

3.2 <u>Adsorbent resin</u>: Porous polymeric resin, XAD-2, is used. The resin must be cleaned prior to use. The resin must not exhibit a blank higher than 4 mg/kg of total chromatographable organics (TCO) prior to use. Once cleaned, the resin should be stored in a wide-mouth amber glass container and the headspace purged with nitrogen to limit exposure to ambient air. Resin should be used within 2 wk of preparation.

3.3 <u>Silica gel</u>: Indicating type, 6 to 16 mesh. If previously used, dry at 175°C (350°F) for 2 hr. New silica gel may be used as received.

3.4 <u>Impinger solutions</u>: Since the impinger solutions are typically used for the determination of gas-stream water-vapor content, Type II water should be used. If specific inorganic species are to be determined (e.g., hydrochloric acid when burning chlorinated organic material), then other appropriate collecting solutions (in the above example, dilute base) must be used.

3.5 <u>Crushed ice</u>: Commercially available. Quantities ranging from 50 to 100 lb may be necessary during a run, depending upon ambient air temperatures.

3.6   Methanol/Methylene chloride: Distilled-in-glass or pesticide-grade methanol and methylene chloride are required.

4.0 SAMPLING PROCEDURE

    4.1   Sampling equipment calibration:
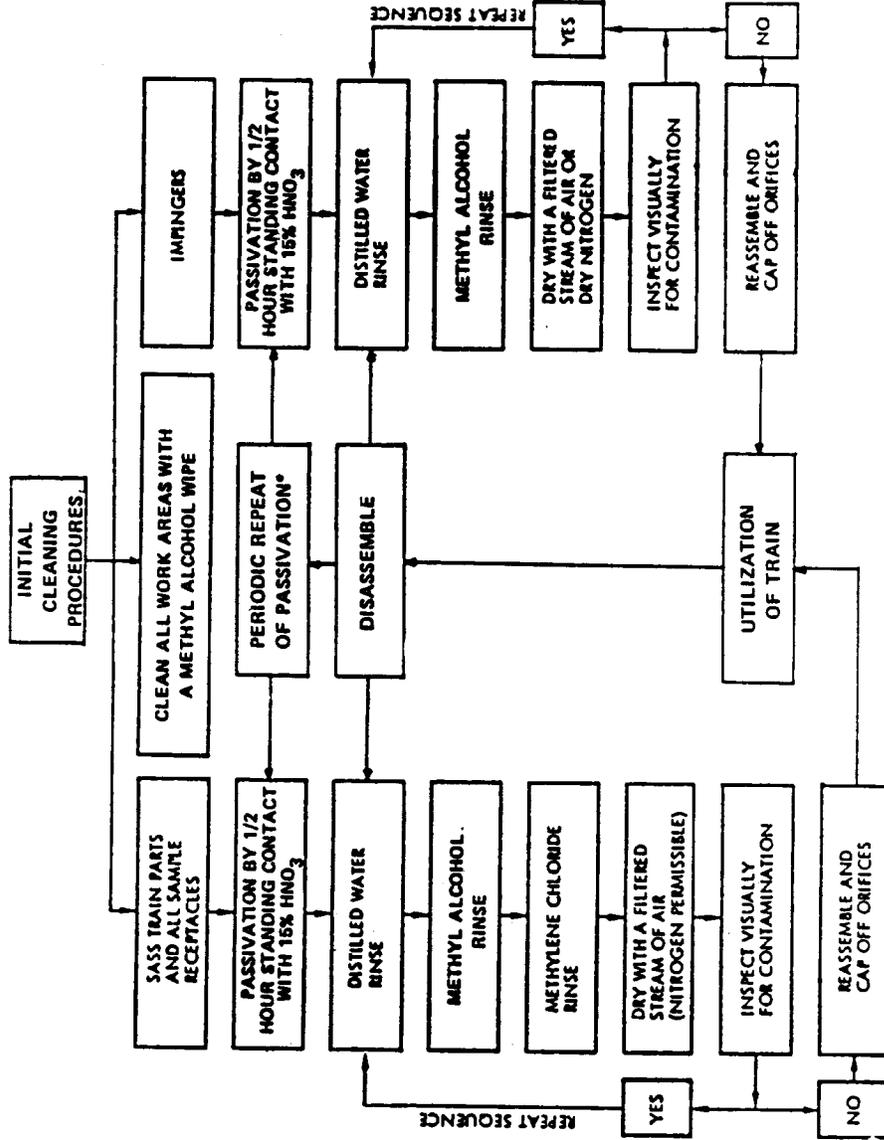
        4.1.1   The probe tips, pitot tubes, dry gas meter, thermocouples, and any thermometers must be calibrated before and after each field sampling trip according to the procedures outlined in APTD-0576 (Rom, 1972) and below in Section 7.0. During extended sampling trips where the train will routinely be used more than 10 times, it is strongly recommended that a calibrated orifice, a set of micrometers (Vernier calipers), and a standard mercury-in-glass thermometer accompany the train to verify that the calibrations of the dry gas meter, probe nozzles, and thermocouples, respectively, have not changed significantly (more than $\pm$2%). The aneroid barometer should be calibrated on a daily basis against a mercury barometer when in the laboratory and periodically in the field by consulting the local weather station and correcting for elevation (see Paragraph 7.7).

    4.2   Laboratory preparation:

        4.2.1   Weigh several 700- to 800-g portions of silica gel in airtight containers to $\pm$0.1 g. Record the weight of the silica gel plus the container on the container and in a field sampling preparation notebook.

        4.2.2   Holding with blunt-tipped tweezers, check filters visually against light for irregularities, flaws, or pinhole leaks. Label the shipping containers (glass Petri dishes) and keep the filters in these containers at all times except during sampling and weighing. The filters themselves need not be labeled if strict compliance with the above instruction is ensured. Desiccate the filters in a desiccator over Drierite or silica gel with the Petri dishes open at 20 $\pm$ 5.6°C (68 + 10°F) and ambient pressure for at least 24 hr and weigh. Thereafter weigh at 6-hr (minimum) intervals to a constant weight, i.e., previous weight $\pm$0.5 mg; record the weight to the nearest 0.1 mg, along with the date and time, in the field sampling preparation notebook. Alternatively, the filters may be oven-dried at 105°C (220°F) for 2 to 3 hr, desiccated for 2 hr, weighed, and weighed thereafter at 6-hr intervals to a constant weight. During each weighing, the filter must not be exposed to the laboratory atmosphere for longer than 2 min with a relative humidity greater than 50%.

        4.2.3   Passivate all SASS train parts and sample storage containers, referring to the procedure that appears in Figure 2, adapted from Level I requirements. Passivation is required of all new train components and sample storage containers before their initial use in the field. Thereafter, passivation should be conducted every 6 months when the frequency of tests is once per month or less, and every 3 months when the

Figure 2. SASS Cleaning Procedure

Source: "IERL-RTP Procedures Manual: Level 1 Environmental Assessment (Second Ed.) EPA-600/7-78-201

SASS Cleaning Procedures

*Refer to text for passivation time schedule.

frequency is between once per week and once per month. If testing is more frequent, passivation should be conducted proportionately more often. Whenever corrosion has occurred, the corrosion must be removed and the passivation repeated. The passivation and rinse solutions should be replaced every fourth use, or discarded weekly.

4.2.4 Prepare recycled sample containers by detergent washing (using a stiff nylon brush where necessary), followed by rinsing with Type II water, methanol and methylene chloride. As each part is treated with the final solvent, dry with filtered air or dry nitrogen and inspect for any contaminating residue. Discard any container exhibiting visual contamination. Cover all open surfaces with aluminum foil or Teflon film, using elastic bands to secure.

4.2.5 Assembly and leak-checking of the entire train in the laboratory is highly recommended to reveal the need for replacement of gaskets or defective components. The leak-check procedure is described in Paragraph 4.4.3.11. Substitution of Viton-A gaskets for Teflon may facilitate meeting the allowable leak rate. A length of Teflon tape stretched around the circumference of each flanged connection underneath the ring clamp also greatly reduces inward air leakage.

4.3 Preliminary field determinations:

4.3.1 Select the sampling site and remove any accumulated scale and corrosion from the sampling portholes. Determine the stack static pressure, temperature, and velocity profile using EPA Method 2 (see References); a leak-check of the pitot lines prior to conducting these measurements is highly recommended. Approximate the moisture content using EPA Method 4 (Approximation Method) or alternate means such as drying tubes, wet bulb/dry bulb or condensation techniques, stoichiometric calculations, or previous experience. Determine the dry molecular weight of the stack gas by performing an ORSAT or GC/TCD analysis for CO, CO_2, O_2 and N_2 on an average of three grab samples taken from either the center of the duct or a point no closer to the stack walls than **1.0 m (3.3 ft).**

4.3.2 Select a nozzle size based upon the calculations below, ensuring that it will not be necessary to change the nozzle during the sampling run to maintain near-isokinetic sampling rates.

4.3.2.1 To calculate the required nozzle diameter, first calculate the Average Stack Gas Velocity:

$$(V_s)_{avg} = K_p C_p (\sqrt{(\Delta P)})_{avg} \left[\sqrt{\frac{(T_s)_{avg}}{P_s M_s}} \right]$$

where:

$(V_s)_{avg}$ = Average stack gas velocity, ft/sec;

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$$K_p = 85.48 \left[\frac{ft}{sec} \left[\frac{(lb/lb\text{-}mole)(in.\ Hg)}{°R(in.\ H_2O)} \right]^{1/2} \right];$$

C_p = Pitot tube coefficient, dimensionless;

ΔP = Velocity head of stack gas, in. H_2O;

$(T_S)_{avg}$ = Average stack gas temperature, °R;

P_S = Absolute stack gas pressure, in. Hg; and

M_S = Molecular weight of stack gas (wet basis), lb/lb-mole.

4.3.2.2 Then calculate the required <u>Nozzle Diameter</u> (D_n):

$$D_n = 0.831 \sqrt{\frac{(T_S)_{avg}}{(V_S)_{avg}\ P_S}}$$

4.3.3 Select a suitable probe length such that one or more points of average velocity can be sampled. Determine the total length of sampling time by comparing the anticipated average sampling rate to the volume requirement of 30 dscm (approximately 1,060 dscf).

4.3.3.1 The anticipated <u>Average Sampling Rate</u> is calculated as follows:

$$Q_{mo} = (1-B_{ws})Q_{st} \frac{P_{st}\ (T_m)_{avg}}{P_m\ (T_{st})}$$

where:

Q_{mo} =   Flow rate through orifice at meter conditions, ft^3/min (dry);

B_{ws} =   Volume fraction of water vapor in the gas stream, dimensionless;

Q_{st} =   Design sampling flowrate for SASS train, 4.0 scfm;

P_{st} =   Standard presssure, 29.92 in. Hg;

$(T_m)_{avg}$ =   Average gas temperature (estimated) at the dry gas meter, °R;

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P_m = Absolute meter pressure, in. Hg, calculated by

$$P_m = P_b + \frac{(\Delta H)_{est.avg.}}{13.6}$$

where:

$(\Delta H)_{est.avg.}$ = Estimated average ΔH across orifice, 3-4 in. H_2O, and

P_b = Barometric pressure (corrected), in. Hg; and

T_{st} = Standard temperature, 528°R.

None of these definitions has an English/metric equivalent.

4.3.3.2 Using this result, obtain the approximate sampling time by dividing the required sample volume by the estimated sampling flowrate.

4.3.4 Finally, calculate the <u>Orifice Pressure Drop</u> needed to maintain near-isokinetic sampling conditions from the equation:

$$\Delta H_i = \frac{P_m}{(T_m)_{avg}} \left[\frac{0.1924\ Q_{mo}}{J_i D_{oi}^2}\right]^2$$

where:

ΔH_i = Required ΔH across the orifice, in. H_2O;

P_m = Absolute meter pressure, in. Hg (calculated the same way as for Average Sampling Rate above);

$(T_m)_{avg}$ = Estimated average gas temperature at the dry gas meter, °R;

J_i = Orifice coefficient for orifice "i" (see Blake, 1977, and Section 7.0 of this method for determining orifice coefficients); and

D_{oi} = Orifice diameter, in. (information supplied upon purchase of the SASS train; the largest diameter orifice is typically best suited for the SASS sampling rate of 4.0 scfm).

4.3.5 It is desirable, but not required, to sample more than one point of average velocity during a SASS run. Allocate equal intervals of the total sampling time estimated above to each sampling point chosen if more than one point will be sampled.

4.4 Preparation of collection train:

 4.4.1   An integral part of preparing the collection train is
securing sufficient electrical power to operate for an extended period of
time without interruption. Three separate circuits -- two 30-amp and one
20-amp -- are required. It is highly recommended that one sampling pump and
one control box power cord (probe heater) be placed on one of the 30-amp
circuits, and the other sampling pump and control box power cord (oven
heater and temperature readout) be placed on the other 30-amp circuit. The
organic module coolant pump and temperature controller should be placed on
the smaller 20-amp circuit.

 4.4.2   During assembly of the train, keep the inner surfaces of each
component covered until it is integrated into the system and sampling is
about to begin. Fill the sorbent trap section of the organic module with
approximately 150 g of clean adsorbent XAD-2 resin. To avoid contamination,
the trap should be placed upon a clean surface (i.e., aluminum foil rinsed
with methylene chloride and air-dried) while filling; gloves should be worn.
Pack the trap uniformly to eliminate potential channeling. Place 500 mL of
Type II water or other appropriate solution into the first and second
impingers, leave the third impinger empty, and place a preweighed portion
of silica gel into the fourth.
NOTE: The choice of impinger solutions depends upon whether these will be
      used to collect selected inorganic species or simply to condense
      water vapor from the gas stream to measure percent moisture. For
      example, in an incinerator combusting chlorinated organic material,
      a solution of dilute base would typically be used to collect
      hydrochloride acid emissions.

 Using blunt-tipped tweezers, place a tared filter into the filter
holder. Ensure that the filter is centered and the gasket properly placed
to prevent the gas stream from circumventing the filter. On the probe, mark
the locations of the chosen sampling points with heat-resistant tape or
paint.

 4.4.3   The stepwise procedure for assembly of the train follows:

 4.4.3.1 Place the oven on a table or rollers that will be used as
a support throughout the run.

 4.4.3.2 Assemble the three cyclones, using the vortex breaker
supplied with the cyclone in the 10-um cyclone only. (To minimize
leaks throughout the system, a strip of Teflon tape should be
stretched around the circumference of each flanged seal and the ring
clamp placed over and secured.)   Do not use the vortex breakers
supplied with the 3- and 1-um cyclones in the 3- and 1-um cyclones.
Actual calibration data has shown that the use of the vortex breakers
in the two smaller cyclones may result in unreproducible particle-
size cutoff diameters (the particle size at which 50% collection
efficiency is exhibited).

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4.4.3.3 Attach the filter holder to the outlet of the 1-um cyclone and place the cyclones and filter holder together in the oven. Preheat a spare filter holder containing a tared filter on the oven floor; cover the holder openings with aluminum foil.

4.4.3.4 Attach the probe to the oven and to the 10-um cyclone.

4.4.3.5 Place the impingers in the tray in the impinger case and make the apropriate interconnections.

4.4.3.6 Connect the organic module inlet to the filter housing outlet and the organic module outlet to the first impinger inlet.

4.4.3.7 Connect the vacuum pumps in parallel to the fourth impinger outlet.

4.4.3.8 Connect all temperature sensors and power lines to the control unit. Check temperature indicators and controllers at ambient temperature.

4.4.3.9 Activate gas cooling system. Begin monitoring the XAD-2 temperature. Always check coolant level before supplying power. Ensure proper gas cooling system temperature before proceeding.
NOTE: IT IS EXTREMELY IMPORTANT THAT THE XAD-2 RESIN TEMPERATURE NEVER EXCEED 50°C, AS DECOMPOSITION WILL OCCUR. DURING TESTING, THE XAD-2 TEMPERATURE MUST NOT EXCEED 20°C FOR EFFICIENT CAPTURE OF THE SEMIVOLATILE ORGANIC SPECIES OF INTEREST.

4.4.3.10   Heat oven and probe to 204°C (400°F).

4.4.3.11   Run gas flow leak-check. The following instructions will facilitate the leak-checking procedure:

a.   Open the isolation ball valve and plug the inlet to the probe with a rubber stopper or appropriate airtight cap.

b.   Start the pumping system with the bypass valves fully open and the coarse valves completely closed. Partially open the coarse valves and slowly close the bypass valves until a vacuum of 127 mm Hg (5 in. Hg) is reached. <u>Do not reverse the direction of the bypass valves as backflushing of the impinger solutions into the organic module will result.</u> If the desired vacuum is exceeded, either leak-check at the higher vacuum or terminate the leak-check and begin again. Allow the system to equilibrate and measure the leakage rate. The allowable leak rate for the SASS train is 0.0014 m^3/min (0.05 ft^3/min) at this vacuum. Close the isolation ball valve and evacuate the train to 281 mm Hg (15 in. Hg). The leak rate through the back half of the train should be less than 0.0014 m^3/min (0.05 ft^3/min) at this vacuum.

c.   When the leak-check is complete, slowly remove the plug from the probe tip and then slowly open the isolation ball valve.

d.   When the vacuum drops to 127 mm Hg (5 in. Hg) or less, immediately close both coarse control valves together. Switch off the pumping system and reopen the bypass valves. The bypass valves should not be opened until the coarse valves have been closed.

4.4.4   Only post-test leak-checks are mandatory; however, experience has shown that pre-test leak-checks and leak-checks following component changes are necessary to ensure that invaluable sampling time is not lost as a result of an oversight or defective component.

4.5   <u>Sample Collection</u>: Constant monitoring of train operations before, during, and after the particulate run is essential in maintaining sample integrity. Listed below are sample collection guidelines:

4.5.1   With the coarse valves closed and bypass valves open, turn on the vacuum pumps and allow them to warm up. As the probe and oven are heating, prepare a SASS run data sheet as shown in 40 CFR Appendix A (see References below). Barometric pressure data should be recorded at least at the beginning and end of the run; once per hour is preferred.

4.5.2   When operating temperatures have been reached, place the probe in the stack at the first designated sampling point, turn on the vacuum pumps, adjust the sampling flowrate to achieve the calculated ΔH_i, and start the elapsed timer. If, however, the gas stream is under medium or high negative pressure, it becomes extremely important to start the vacuum pumps just before placing the probe in the gas stream, and to continue to operate the pumps until just after the probe has been removed from the gas stream. This will eliminate the possibility of lifting of the filter or backflushing of the filter and cyclone particulate catches at any time.

4.5.3   Seal the sampling port around the probe to prevent introduction of dilution air at this point. Record the clock time of the start of the test.

4.5.4   Using the criteria outlined above under Paragraph 4.3, Preliminary Field Determination, place the integrated fixed gas bag or bulb sampling probe into the gas stream and begin sampling. Collect three samples during the SASS run; record the initial and final clock times of each integrated fixed gas sample.

4.5.5   Monitor and maintain all temperatures and the calculated ΔH and record the data at equal intervals of 10-15 min.

4.5.6   Add crushed ice to the impinger section and drain excess water as necessary.

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4.5.7   Without interrupting sampling, drain the condensate initially every 30-45 min, and afterward as necessary. Ensure that the vessel into which the reservoir is drained forms an airtight system with the reservoir using a connecting Teflon line, and is placed well below the level of the reservoir itself. To drain the reservoir, close the solation ball valve and open the drain valve. Allow the system to evacuate for 10-20 sec. Carefully open the isolation valve. The condensate should siphon from the reservoir into the storage vessel. Close the drain valve when the siphoning action of the condensate ceases.

4.5.8   Replace the filter when it becomes impossible to maintain near-isokinetic sampling rates but not more frequently than every 20 to 30 min. Always terminate and initiate sampling by adjustment of the coarse pump valves and then the bypass valves. A spare filter holder and filter, if available, should be preheating in the oven at all times. Conduct leak-checks before and after changing the filter. Recall previous instructions concerning removal and reintroduction of the probe into the duct.

4.5.9   At the same time, check the 1-um cyclone reservoir for remaining capacity, taking care not to contaminate the contents during this inspection.

4.5.10   When replacing a filter, start and stop the fixed gas sampling concurrently with the SASS sampling; record the clock time and dry-gas-meter reading whenever sampling is interrupted.

4.5.11   Upon collection of the required 30 dscm (1,060 dscf), remove the probe from the gas stream and shut down the pumps as previously instructed. Record the final dry-gas-meter reading and clock time; turn off all heaters. Conduct the post-test leak-check when the probe tip can be safely handled. Do not cap the probe while initially cooling, because this will create a vacuum inside that will cause disruption of the cyclone and filter particulate catches when it is released. Instead, use aluminum foil to cover probe openings. Before the probe is transported, secure the aluminum foil covers with elastic bands. Leak-check the pitot lines per EPA Method 2 to validate velocity heat data.

5.0 SAMPLE RECOVERY

The sample handling and transfer procedures outlined in this section have been adopted from the Level 1 procedures. The flow diagrammatic representation of the sample recovery procedures shown in Figures 3, 4, and 5 can be found in the Level 1 Sampling and Analysis Procedures Manual.

5.1 Disassembly of the SASS Train: At the conclusion of the sampling run, the train is disassembled and transported to the prepared work area as follows:
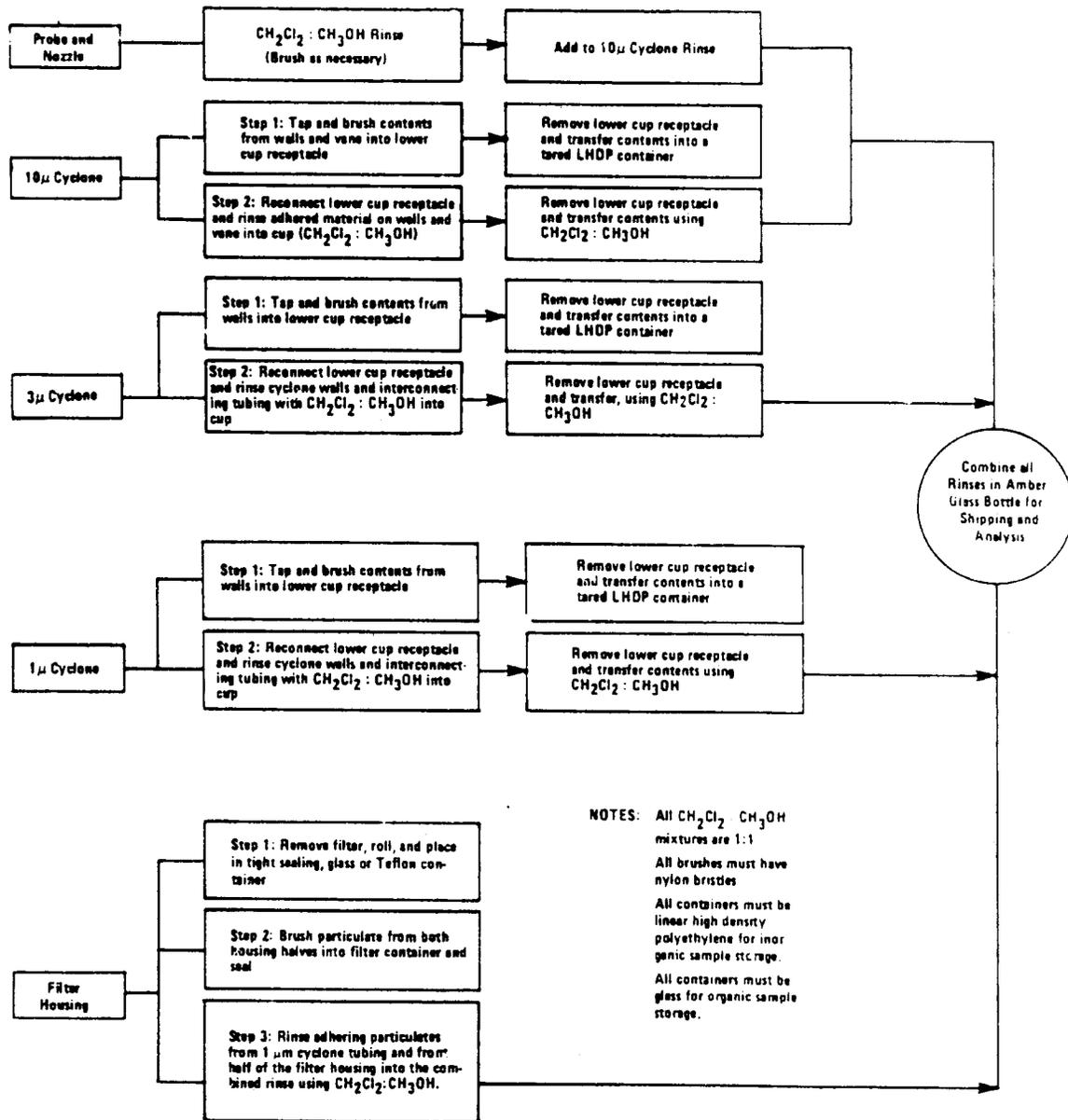
Probe and Nozzle → CH$_2$Cl$_2$: CH$_3$OH Rinse (Brush as necessary) → Add to 10μ Cyclone Rinse

10μ Cyclone
- Step 1: Tap and brush contents from walls and vane into lower cup receptacle → Remove lower cup receptacle and transfer contents into a tared LHDP container
- Step 2: Reconnect lower cup receptacle and rinse adhered material on walls and vane into cup (CH$_2$Cl$_2$: CH$_3$OH) → Remove lower cup receptacle and transfer contents using CH$_2$Cl$_2$: CH$_3$OH

3μ Cyclone
- Step 1: Tap and brush contents from walls into lower cup receptacle → Remove lower cup receptacle and transfer contents into a tared LHDP container
- Step 2: Reconnect lower cup receptacle and rinse cyclone walls and interconnecting tubing with CH$_2$Cl$_2$: CH$_3$OH into cup → Remove lower cup receptacle and transfer, using CH$_2$Cl$_2$: CH$_3$OH

1μ Cyclone
- Step 1: Tap and brush contents from walls into lower cup receptacle → Remove lower cup receptacle and transfer contents into a tared LHDP container
- Step 2: Reconnect lower cup receptacle and rinse cyclone walls and interconnecting tubing with CH$_2$Cl$_2$: CH$_3$OH into cup → Remove lower cup receptacle and transfer contents using CH$_2$Cl$_2$: CH$_3$OH

Filter Housing
- Step 1: Remove filter, roll, and place in tight sealing, glass or Teflon container
- Step 2: Brush particulate from both housing halves into filter container and seal
- Step 3: Rinse adhering particulates from 1 μm cyclone tubing and from half of the filter housing into the combined rinse using CH$_2$Cl$_2$:CH$_3$OH.

Combine all Rinses in Amber Glass Bottle for Shipping and Analysis

NOTES: All CH$_2$Cl$_2$: CH$_3$OH mixtures are 1:1

All brushes must have nylon bristles

All containers must be linear high density polyethylene for inorganic sample storage.

All containers must be glass for organic sample storage.

Figure 3. Sample Handling and Transfer Nozzle, Probe, Cyclones and Filter.

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INTACT SORBENT MODULE
AFTER SAMPLING RUN

RELEASE CLAMP JOINING
SORBENT CARTRIDGE SEC·
TION TO THE UPPER GAS
CONDITIONING SECTION

REMOVE SORBENT CAR·
TRIDGE FROM HOLDER.
REMOVE SCREEN FROM
TOP OF CARTRIDGE.
EMPTY RESIN INTO A
WIDE-MOUTH AMBER JAR
USING CLEAN FUNNEL

REMOVE CONDENSATE
RESERVOIR AND DRAIN
CONDENSATE THROUGH
VALVE INTO THE
CONDENSATE DRAIN
CONTAINER USED TO
COLLECT CONDENSATE
DURING THE SASS RUN

CLOSE CONDENSATE VALVE
AND REASSEMBLE TO MOD·
ULE TO COLLECT WASHINGS

RINSE SCREEN AND CAR·
TRIDGE INTO RESIN
CONTAINER WITH CH_2Cl_2

MEASURE VOLUME AND
pH

RELEASE UPPER CLAMP
AND LIFT OUT INNER WELL

REASSEMBLE MODULE
TO COLLECT WASH·
INGS

RINSE WITH
CH_2Cl_2

EXTRACT AT pH 2

COMBINE CH_2Cl_2
EXTRACTS AND
SHIP TO LAB IN
AMBER GLASS
CONTAINER

RINSE WITH TEFLON WASH
BOTTLE (CH_2Cl_2) ALONG
INNER WELL SURFACE
AND CONDENSER WALL

EXTRACT AT pH 11

PLACE INNER WELL
ASIDE IN CLEAN AREA

LABEL RESIN
CONTAINER
AND RINSINGS

MEASURE AQUEOUS
VOLUME REMAINING

MEASURE VOLUME
OF FIRST IMPINGER

RINSE ENTRANCE TUBE AND
BACK HALF OF FILTER HOUS·
ING INTO MODULE. RINSE
DOWN CONDENSER WALL

COMBINE

RELEASE CENTRAL CLAMP
TO SEPARATE CLEAN CON·
DENSER SECTION FROM
LOWER SECTION. RINSE LOW·
ER SECTION INTO CONDEN·
SATE CUP. RELEASE THE
BOTTOM CLAMP AND RINSE
INTO CONDENSATE CUP.
DRAIN INTO AMBER BOTTLE
VIA DRAIN VALVE

CLEAN ALL MODULE METAL
PARTS BY CLEANING PROCE·
DURE

Figure 4. Sample Handling and Transfer XAD-2 Module.

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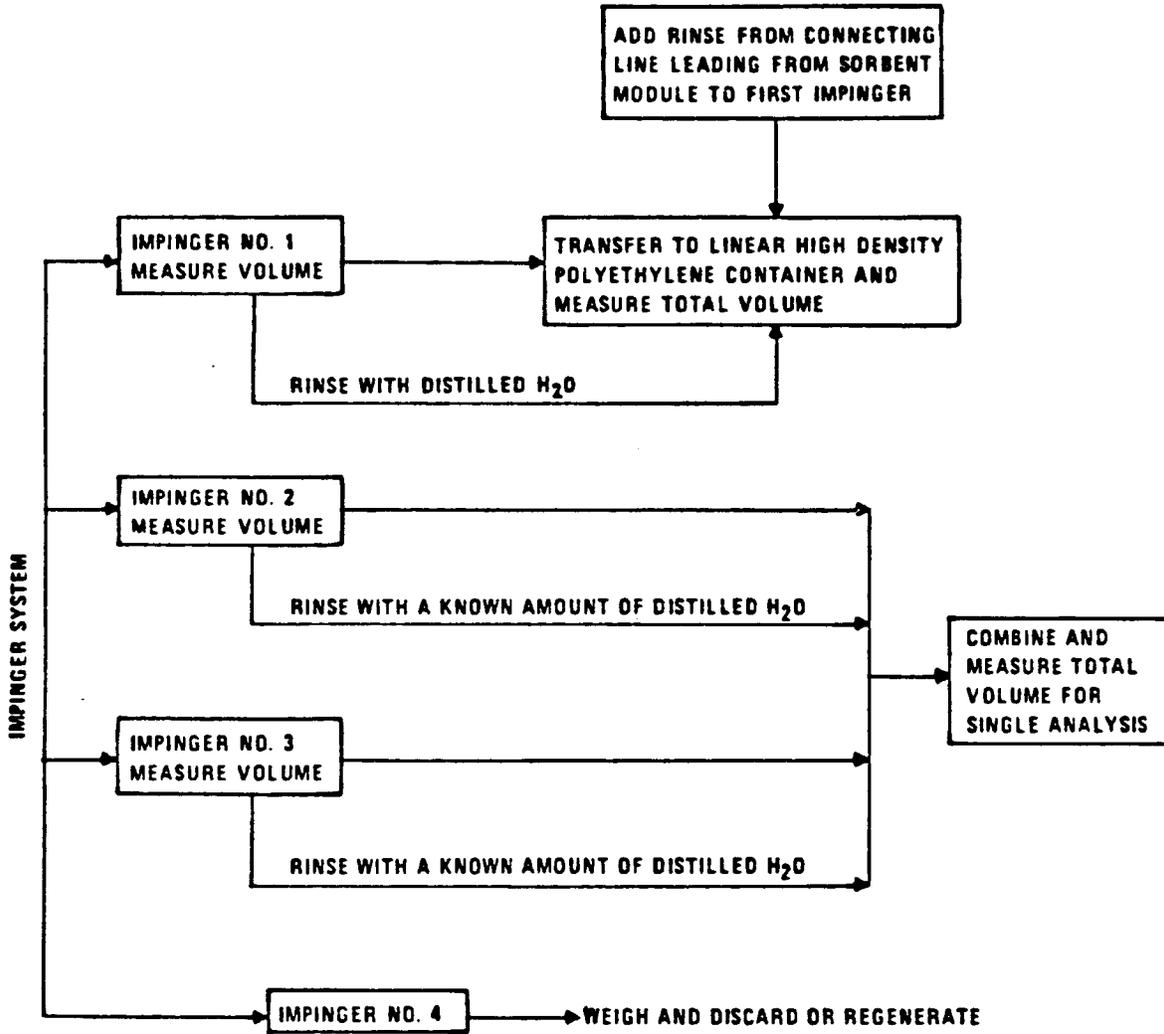
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Figure 5. Sample Handling and Transfer Impingers.

5.1.1 Leaving the fan operating, open the cyclone oven door to expedite cooling. When the probe can be safety handled, disconnect from the 10-um cyclone inlet. Wipe off external particulate matter near the probe tip and place a cap over each end. The probe must remain level throughout this procedure.

NOTE: CARE MUST BE TAKEN TO AVOID TIGHTLY CAPPING TRAIN COMPONENTS AS THEY ARE COOLING FROM STACK OR OVEN TEMPERATURES. THIS WILL ELIMINATE THE POSSIBILITY OF CREATING A VACUUM INSIDE WHICH, WHEN RELEASED, MAY DISRUPT AND BACKFLUSH THE CYCLONE AND FILTER PARTICULATE CATCHES INTO ONE ANOTHER.

5.1.2 Disconnect the line joining the filter outlet to the XAD-2 module and cap off:

a.   The 10-um cyclone inlet;

b.   The filter holder outlet; and

c.   The inlet of the line just disconnected from the filter holder outlet.

5.1.3 Disconnect the filter holder and cap the inlet. Set aside with the inlet facing upward. Cap the outlet of the 1-um cyclone. The cyclones must remain upright throughout this procedure. The cyclones can now be disconnected from one another or moved to the recovery area as a single unit.

5.1.4 Disconnect the line joining the XAD-2 module to the impinger system at the organic module outlet. Cap the organic module outlet.

5.1.5 Disconnect the silica gel impinger outlet from the vacuum line to the pumps; cap off the first impinger inlet and the fourth impinger outlet.

5.2 Nozzle, Probe, Cyclones, and Filter: The step-by-step procedures for the recovery of particulate material collected in the nozzle, probe, and cyclones, and on the filter are detailed below:

5.2.1 Carefully transfer the filter from the filter housing to its original glass Petri dish; a pair of clean blunt-tipped tweezers and a flat spatula should be used for handling the filter. Using a clean nylon-bristled brush, add any particulate material from the front half of the filter housing to the Petri dish; seal the Petri dish around the circumference with 1-in.-wide Teflon tape; store with the collected particulate material facing upward.

5.2.2 Tap and brush any particulate material adhering to the walls of the upper chamber of the 1-um cyclone into the lower cup; remove the cup and quantitatively transfer the bulk contents to a wide-mouth amber glass jar. Rinse the brush with methanol/methylene chloride (1:1 v/v) into the probe rinse container.

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5.2.3 Recover the contents of the 3-um cyclone in the same manner, using a separate wide-mouth amber glass jar.

5.2.4 Recover the contents of the 10-um cyclone in the same manner, using a separate wide-mouth amber glass jar.

5.2.5 Reconnect the lower cups of each cyclone and rinse any particulate material adhering to the walls down into the cups with the methanol/methylene chloride mixture until the walls appear clean. Remove the lower cups and transfer the contents to the probe rinse container. Rinse the interconnecting tubing among the cyclones into the probe rinse in the same manner.

5.2.6 Carefully remove the probe nozzle and clean the inside surface by rinsing with the methanol/methylene chloride (1:1 v/v) from a wash bottle and brushing with a nylon-bristle brush. Brush until the rinse shows no visible particles; make a final rinse of the inside surface.

5.2.7 Rinse the probe liner (preferably with two people so as to minimize the possibility of accidental sample loss) with methanol/ methylene chloride (1:1 v/v) by tilting and rotating while spraying solvent into the upper end and allowing the lower end to drain into the sample container. Follow rinsing with brushing and rinsing from the upper to the lower end. Push the brush through the liner with a twisting action; ensure that the sample container is placed under the lower end. Brush until the rinse appears clean; perform a final rinse. Inspect the inner surface of the liner for cleanliness. Rinse any particulate material remaining on the brush into the sample container.

5.2.8 Clearly label all containers according to the coding scheme given in Table 1; cover each label completely with transparent tape; mark liquid levels and store all liquid samples on ice.

5.3 XAD-2, condensate, and organic module: Sample recovery of the entire organic module may be conducted independently from the previous steps. The step-by-step procedure for recovery of this stage is given below:

5.3.1 Rinse a 1-ft x 1-ft square of aluminum foil (dull side) with methylene chloride and allow to air dry.

5.3.2 Release the clamp joining the XAD-2 cartridge section to the upper gas conditioning system (second clamp); remove the XAD-2 cartridge from the holder and place upon the clean aluminum foil. GENTLY pry loose or unscrew (depending upon the design) the ring securing the fine mesh screen on the top of the cartridge. Remove the screen and quantitatively transfer the XAD-2 to a clean glass amber jar. A large, clean glass funnel should be used for the transfer. Rinse the inner surfaces of the cartridge and the funnel with methylene chloride as necessary to remove adhering XAD-2. Any XAD-2 that escapes onto the aluminum foil should be retrieved and added to the sample.

TABLE 1. SUGGESTED FORMAT FOR SAMPLE CODING AND IDENTIFICATION

Sample Code	Container	Size	Sample description
1C	Amber glass	100 mL, wide-mouth	1-m cyclone catch
3C	Amber glass	100 mL, wide-mouth	3-m cyclone catch
10C	Amber glass	100 mL, wide-mouth	10-m cyclone catch
PF-a,b,c,...	Glass Petri dish	>6-in. diam.	Particulate filter(s)
PR	Amber glass	1 liter	Methylene chloride/ methanol front-half rinse
PRB	Amber glass	500 mL	Methylene chloride/ methanol blank
MRX	Amber glass	1 liter	Methylene chloride back half rinse
MRXB	Amber glass	500 mL	XAD-2 resin blank
CD-LE	Amber glass	1 liter	Methylene chloride condensate extract
CD-LEB	Amber glass	500 mL	Methylene chloride blank
AR-I1	HDLP[a]	1 liter	Aqueous residue of condensate combined with first impinger catch
I1B	HDLP	500 mL	First impinger blank (distilled H_2O or other appropriate solution)
I23	HDLP	1 liter	Second and third impinger catches
I23B	HDLP	500 mL	Second and third impinger blank (distilled H_2O or other appropriate solution)

[a]HDLP = High Density Linear Polyethylene.

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5.3.3   Replace the screen on the cartridge, reinsert the cartridge into the module, and reassemble the module. One person can accomplish this task by butting the lower section in its proper sealing position up against the upper section while securing the ring clamp. One or more wooden spacers approximately 1/2 in. thick are suggested for this purpose.

5.3.4   Open the condensate reservoir valve and drain the remaining condensate into the condensate storage container. Measure and record the volume and pH (using narrow-range pH paper) of the entire condensate.

5.3.5   Transfer the entire condensate to an appropriately-sized separatory funnel. Adjust the pH of the condensate (as indicated by the narrow-range pH paper) to 1-2 using ultrapure or reagent grade nitric acid. Extract the condensate three times with methylene chloride, each time with fresh portions measuring 8-10% of the total condensate volume. If the volume of the condensate is extremely large (>1800 mL), the condensate may be extracted in portions, but fresh volumes of methylene chloride must be used for each and every extraction. After each addition of methylene chloride to the separatory funnel, the funnel must be shaken with periodic venting through the stopcock to relieve any vapor pressure. For safety, the tip of the separatory funnel should <u>always</u> be directed away from the face and eyes while venting. When no further vapor pressure can be vented after shaking, the funnel should be mounted upright on a ring stand, the cap removed, the layers allowed to separate, and the methylene chloride (bottom) layer removed. If an emulsion forms equal to more than one-third the size of the solvent layer, reagent-grade sodium chloride should be added until the emulsion is broken or reduced to meet the above criterion. The emulsion interface should <u>not</u> be included as part of the methylene chloride extract.

5.3.6   Following the third extraction of the acidified condensate, adjust the pH of the aqueous residue to 11-12 with a 50% w/w solution of sodium hydroxide (as indicated by narrow-range pH paper), extract with methylene chloride in the same manner, and combine the methylene chloride extracts of the condensate at the high and low pH readings.

5.3.7   Transfer the aqueous residue from this extraction to a clean Nalgene container; retain for later addition of the first impinger solution.

5.3.8   Ensure that the condensate reservoir valve is closed, release the upper clamp, and lift the inner well halfway out of the module. Rinse the inner well into the XAD-2 module using a Teflon wash bottle containing methylene chloride, so that the rinse travels down the module and into the condensate collector. Then remove the well entirely and place to one side on a clean surface (aluminum foil prerinsed with methylene chloride). Rinse the entrance tube into the module interior; rinse the condenser wall allowing solvent to flow down through the system and collect in the condensate cup.

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5.3.9   Release the central clamp again and separate the lower section (XAD-2 cartridge holder and condensate cup) from the upper.

5.3.10 Lift the empty XAD-2 cartridge halfway out of the mid-section and rinse the outer surface down into the condensate cup. Remove the cartridge completely to a clean surface (aluminum foil rinsed with methylene chloride).

5.3.11 Rinse the empty XAD-2 section into the condensate cup. Open the condensate reservoir valve and drain into the XAD-2 sample storage container (wide-mouth amber glass jar).

5.3.12 Rinse the condensate reservoir and combine the rinse with the XAD-2 resin as above.

5.3.13 Clearly label all containers according to the coding scheme presented in Table 1; cover each label completely with transparent tape; mark liquid levels and store all liquid samples on ice.

5.4   Impingers: Sample recovery from the impingers may also be accomplished independently of the other two sections of the SASS train.   The procedures are described below.

5.4.1   **First impinger:**

5.4.1.1 Measure the volume of liquid in the impinger with a graduated cylinder; combine with the aqueous residue from the condensate.

5.4.1.2 Rinse the line connecting the XAD-2 module to the first impinger with Type II water; transfer the rinse to the same graduated cylinder. Rinse the impinger twice more with Type II water, combining all rinses in the graduated cylinder. Measure the total rinse volume and add to the sample. Rinse the graduated cylinder with a known amount of Type II water and add to the sample. Record all volumes on the sample recovery sheet.

5.4.2   **Second and third impingers:**

5.4.2.1 Measure and record the combined volume of liquid in the impingers in a large (1,000-mL) graduated cylinder; transfer to a clean sample storage container.

5.4.2.2 Rinse the line connecting the first and second impinger into the second impinger and the line connecting the second and third impinger into the third impinger. Transfer the rinses to the same graduated cylinder. Rinse each impinger twice again with Type II water, combining all rinses in the graduated cylinder. Measure and record the combined rinse volume and add to the sample. Rinse the graduated cylinder with a known amount of Type II water and add to the sample. Record this additional rinse volume and add to the impinger rinse volume above.

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5.4.2.3 Clearly label all sample containers according to the coding scheme presented in Table 1; cover each label completely with transparent tape; mark fluid levels and store all liquid samples on ice.

5.4.3 **Fourth impinger:**

5.4.3.1 Transfer the silica gel to its original container. Weigh to the nearest 0.1 g on a triple-beam balance, and record the weight.

5.4.3.2 Discard or regenerate.

6.0 SAMPLE PREPARATION FOR SHIPMENT

6.1 Prior to shipment, recheck all sample containers to ensure that the caps are securely tightened. Seal the lids of all Nalgene containers around the circumference with vinyl tape and those of glass containers with Teflon tape. Ship all liquid samples on ice and all particulate filters with the particulate catch facing upward. Ship peroxide solutions (impinged) in a separate container.

7.0 CALIBRATION

7.1 All calibration results should be recorded on appropriate data sheets and fastened securely into a separate section in the field sampling notebook. Samples of blank data appear in 40 CFR 60 (1979), Appendix A.

7.2 <u>Probe nozzles</u>:

7.2.1 Probe nozzles must be calibrated before each initial use in the field. Using Vernier calipers or micrometers, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Perform ten separate measurements using different diameters; obtain the average of the ten measurements. The difference between the highest and lowest measurement results must not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they must be reshaped, sharpened, and recalibrated before reuse. Recalibration of the nozzle before each run in gas streams that are highly corrosive is strongly recommended, as the nozzle diameter may be changing slightly from one run to the next. Each nozzle must be permanently and uniquely engraved.

7.3 <u>Pitot tube</u>

7.3.1 If the Type-S pitot tube conforms to the construction specifications (the face openings are not visibly nicked, dented, or corroded) and the pitot tube/probe assembly meets the intercomponent spacings outlined in EPA Method 2 (see References), the pitot tube need not be calibrated to meet federal and many state testing requirements; a correction coefficient may be assigned in these cases. Some states, however, require that, once used, pitot tubes must be calibrated in a

wind tunnel. Specific state requirements such as this must be unequivocally stated prior to testing. In either case, pitot tube face openings should be inspected before each run to ensure that there has been no change in appearance since their construction or most recent calibration.

7.4 Metering system:

7.4.1 Before each initial use in the field, the metering system shall be calibrated using a standard bell prover of the proper size. (A standard bell prover is recommended for this procedure because the displacement volume of commercially available wet test meters is typically insufficient.) A meter stick should be used to indicate the distance travelled by the inner tank during the measurement. Figure 6 illustrates a suitable arrangement for the calibration. It is highly recommended that the dry gas meter be adjusted until the ratio of the dry gas meter volume to the standard bell prover volume equals 1.00 ± 0.01, to ensure that the calculated ΔH_i will result in near-isokinetic sampling rates. The calibration procedure follows:

7.4.1.1 Perform both a positive (pressure) and a negative (vacuum) leak-check of the metering system. For the negative leak-check, include only the orifice Magnehelics (reg. trademark), dry gas meter, and two vacuum pumps by removing the vacuum line connecting the fourth impinger to the vacuum pumps at the common side of the pump inlet tee, and replacing the line with a plain-end male quick connect. Tightly cap this end and leak-check in the manner outlined above under 4.4.3.11. For the positive leak-check, connect a short length of rubber tubing to the "gas exhaust" port on the SASS control module. Disconnect and vent the low side of the orifice magnehelic; close off the low-side orifice tap. Pressurize the system to 13-18 cm H_2O (5-7 in. H_2O) by blowing into the rubber tubing; pinch off the tubing and observe the magnehelic for one minute. The magnehelic reading must remain unchanged during that time period. Any loss of magnehelic pressure indicates a leak that must be corrected.

7.4.1.2 Upon obtaining satisfactory leak-checks, connect the metering system to the standard bell prover.

7.4.1.3 Using the control box Magnehelic (reg. trademark) indicator, set the pumping rate corresponding to a ΔH of 1 in. Hg. Turn the pumps off using the switches.

7.4.1.4 Record the initial temperature and pressure of the bell prover and the initial temperature and reading of the dry gas meter. Record the barometric pressure every hour.
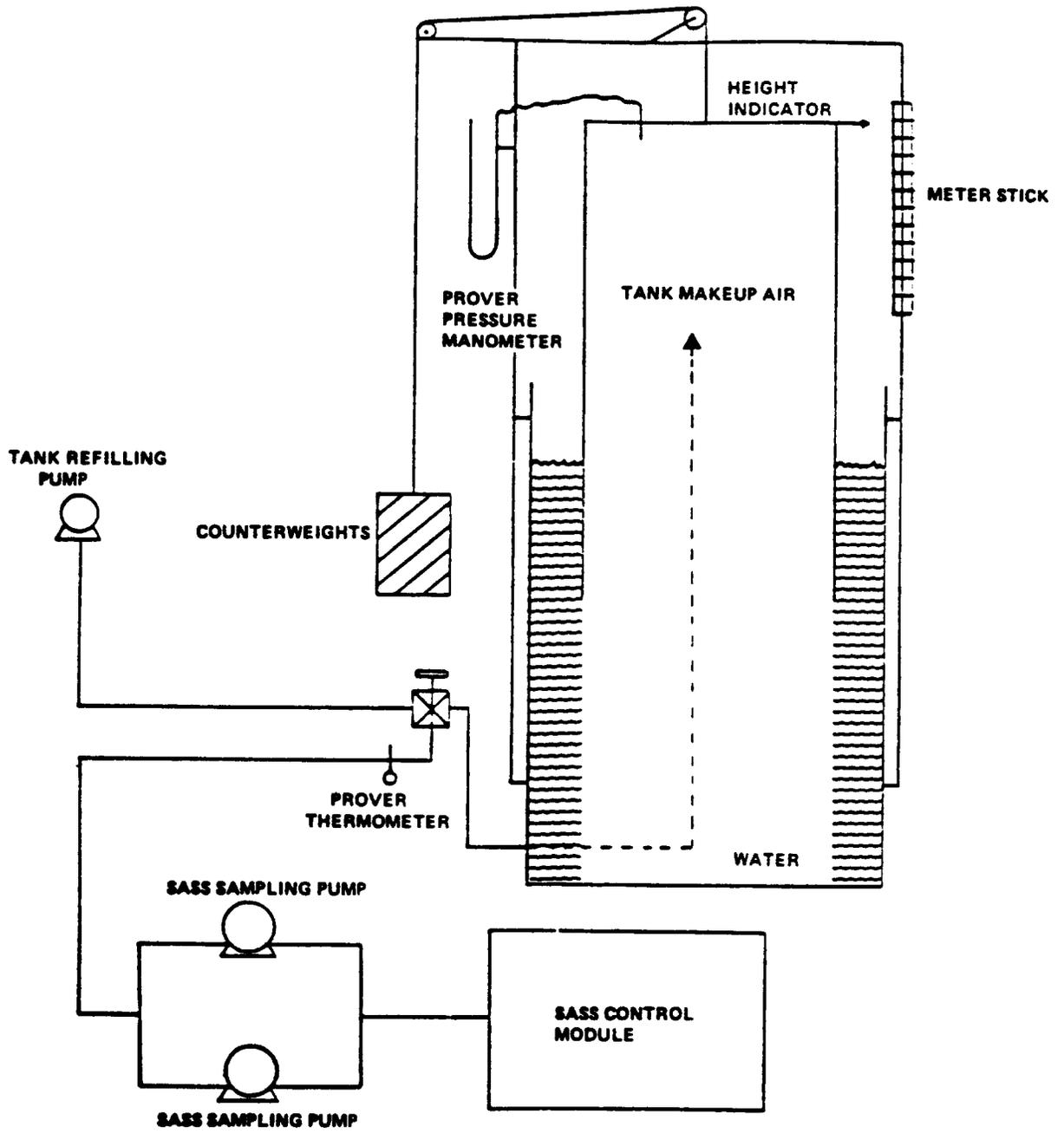
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Figure 6. Schematic Diagram of Standard Bell Prover Arrangement for SASS Dry-Gas-Meter Calibration.

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7.4.1.5 Disconnect the metering system and pump the inner tank of the bell prover to a convenient height. Reconnect the metering system and record the height.

7.4.1.6 Start the pumps and a stopwatch simultaneously; evacuate the tank for 3 min.

7.4.1.7 After 3 min, turn off the pumps using the switches. Record the final inner tank height, the final dry gas meter reading and temperature, and the bell prover final temperature and pressure.

7.4.1.8 Repeat steps 3-7 using ΔH settings of 2, 4, and 6 in. H_2O.

7.4.1.9 Duplicate the entire procedure as a check; repeat the entire procedure after each adjustment of the dry gas meter.

7.4.1.10 Calculate the <u>Dry-Gas-Meter Correction Coefficient</u>, the ratio of the volume of gas measured by the dry gas meter to the standard bell prover, both corrected to standard conditions and on a dry basis. The ratio reduces to:

$$\gamma = \frac{V_{pvr(std)}}{V_{dgm(std)}} = \frac{V_{pvr} \, P_{pvr} \, T_{dgm}}{V_{dgm} \, P_{dgm} \, T_{pvr}}$$

where:

γ = Dry gas meter correction coefficient, dimensionless;

$V_{dgm(std)}$ = Volume of gas measured by the dry gas meter on a dry basis, corrected to standard conditions, dscm (dscf);

$V_{pvr(std)}$ = Volume of gas measured by the standard bell prover on a dry basis, corrected to standard conditions, dscm (dscf);

V_{dgm} = Volume of gas measured at dry-gas-meter conditions, $m^3(ft^3)$;

= Final volume reading - initial volume reading;

V_{prv} = Volume of gas measured at standard bell prover conditions, $m^3(ft^3)$

= K_{pvr} x (difference in meter stick height readings),

where:

K_{pvr} = number of ft^3 of air displaced represented by each cm of movement along the meter stick, $m^3/cm(ft^3/cm)$;

P_{dgm} =   Absolute meter pressure, mm Hg (in. Hg)

=   Barometric pressure + $\Delta H/13.6$;

P_{pvr} =   Absolute prover pressure, mm Hg (in. Hg)

=   Barometric pressure - [(ΔP) prover manometer]/13.6;

T_{pvr} =   Absolute bell prover temperature, °K (°R); and

T_{dgm} =   Absolute dry-gas-meter temperature, °K (°R).

   7.4.1.11   Calculate the <u>Orifice Constants</u> using the following equations:

a. $Q_{mo} = (1-B_{ws})\ Q_{std}\ \dfrac{P_{std}T_{m(avg)}}{P_m T_{std}}$

   where:

   Q_{mo} =   Sampling flowrate at orifice, ft^3/min (dry);

   B_{ws} =   Proportion by volume of water in ambient air, dimensionless;

   Q_{std} =   Standard sampling flowrate for SASS, 4.0 scfm (wet);

   P_{std} =   Standard absolute pressure, 29.92 in. Hg;

$T_{m(avg)}$ =   Average meter temperature, °R;

   P_m =   Absolute meter pressure (barometric pressure + $\Delta H/13.6$), in. Hg; and

   T_{std} =   Standard temperature, 528°R.

b. $J_i = \dfrac{Q_{mo}}{A_{oi}}\left[\dfrac{T_{m(avg)}}{P_m M_m}R\Delta H_i 2g_c\right]^{-1/2}$

   where:

   J_i =   Orifice coefficient for orifice "i";

   Q_{mo} =   Sampling flowrate at orifice, ft^3/min (dry);

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$$A_{oi} = \text{Orifice area } [\pi(\text{diameter})^2]/4, \text{ in.}^2;$$

$$T_{m(avg)} = \text{Average meter temperature, } °R;$$

$$P_m = \text{Absolute meter pressure (see equation above), in. Hg;}$$

$$M_m = \text{Molecular weight of air, 29.0 } lb_m/lb\text{-mole;}$$

$$R = \text{Gas law constant, 1545 ft-lb}_f/°R \text{ lb-mole;}$$

$$\Delta H_i = \text{Orifice "i" pressure drop, in. } H_2O; \text{ and}$$

$$g_c = \text{Gravitational constant, 32.17 } lb_m\text{-ft/lb}_f \text{ sec}^2.$$

7.4.1.12 The orifice constants may be determined without the bell prover by noting the dry-gas-meter volumes obtained by pumping at 1, 2, 3, and 6 in. H_2O for 3-min periods. The obtaining of consistent values when checking orifice constants in the field may be used as a rough indication of a valid calibration during extended field use.

7.4.2 After each series of field tests, the calibration of the metering system must be checked by performing three calibration measurements at a single intermediate orifice setting at or near the average used during the field testing. If the calibration has changed by more than 5%, recalibrate the meter over the full range of orifice settings. Calculations for the test series should then be performed using whichever calibration results in the lower value for total sample volume.

7.5 Probe heater: The probe heating system shall be calibrated before each initial use in the field and checked after each series of field tests according to the procedure outlined in APTD-0576.

7.6 Thermocouples: Each thermocouple must be permanently and uniquely marked on the casing; all mercury-in-glass reference thermometers must conform to ASTM-E-1 #63C or 63F specifications. Thermocouples should be calibrated in the laboratory without the use of extension leads. If extension leads are used in the field, the thermocouple reading at ambient air temperatures, with and without the extension lead, must be noted and recorded. Correction is necessary if the use of an extension lead produces a change greater than 1.5%. Calibration for the various kinds of thermocouples proceeds as follows:

7.6.1 **Impinger and organic module thermocouples:** For the thermocouples used to measure the temperature of the gas leaving the impinger train and the XAD-2 resin bed, a three-point calibration at ice water, room-air, and boiling-water temperatures is necessary. Accept the thermocouples only if the readings at all three temperatures agree within 2°C (3.6°F) of the absolute value of the reference thermometer.

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7.6.2 **Dry-gas-meter thermocouples:** For the thermocouples used to indicate the dry-gas-meter inlet and outlet temperatures, a three-point calibration at ice-water, room-air, and boiling-water temperatures must be performed. The values must be within 2°C (3.6°F) of the absolute reference thermometer value at all three calibration points.

7.6.3 **Probe and stack thermocouple:** For the thermocouples used to indicate the probe and stack temperatures, a three-point calibration at ice-water, boiling-water, and boiling cooking-oil temperatures must be performed; it is highly recommended that room-air temperature be added as a fourth calibration point. If the absolute values of the reference thermometer and the thermocouple agree within 1.5% at each of the calibration points, a calibration curve (equation) may be constructed (calculated), and the data extrapolated to cover the entire temperature range suggested by the manufacturer.

7.7 Barometer: Adjust the field barometer initially and before each test series to agree within 2.5 mm Hg (0.1 in. Hg) of the mercury barometer, or within the station barometric pressure value reported by a nearby National Weather Service station and corrected for elevation.

7.8 Triple-Beam Balance: Calibrate the triple beam balance before each test series using Class-S standard weights; the weights must be within 0.5 g of the standards, or the balance adjusted to meet these limits.

7.9 Analytical Balance: Calibrate the analytical balance with Class-S weights before initially tare-weighing each set of filters. The balance must agree or be adjusted to within 2 mg of the standards. Run at least one standard each time one or more of the filters is reweighed.

8.0 CALCULATIONS

8.1 Dry gas volume:

8.1.1 From the SASS run sheet, average the dry-gas-meter temperatures and orifice pressure drops readings throughout the run. Calculate the Volume of Dry Gas Sampled at standard conditions (20°C, 760 mm Hg [528°R, 29.92 in. Hg]) using the equation:

$$V_{m(std)} = V_m Y \frac{(T_{std})}{T_m} \frac{(P_{bar} + (\Delta H/13.6))}{P_{std}}$$

$$= K_1 V_m Y \frac{(P_{bar} + (\Delta H/13.6))}{T_m}$$

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where:

$V_{m(std)}$ = Volume of dry gas sampled at standard conditions, dscm (dscf);

V_m = Volume of dry gas sampled at dry-gas-meter conditions, dcm (dcf);

γ = Dry-gas-meter calibration factor, dimensionless;

T_m = Average dry-gas-meter temperature, °K (°R);

T_{std} = Standard absolute temperature, °K (°R);

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg);

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

ΔH = Average orifice pressure drop during the sampling run, mm H_2O (in. H_2O); and

K_1 = 0.358°K/mm for metric units
= 17.64°R/in. Hg for English units.

8.1.2 The above equation must be modified whenever the leakage rate observed during any of the mandatory leak-checks (i.e., the post-test leak-checks or leak-checks made prior to component changes) exceeds the maximum allowed. The modification follows:

8.1.2.1 <u>Case I</u> (No component changes have been made during the sampling run, and the allowable leakage rate has been exceeded during the post-test leak-check): Replace V_m with the expression:

$$V_m - [(L_p - L_a)\theta]$$

where:

L_p = Leakage rate observed during post-test leak-check, m^3/min (cfm);

L_a = Maximum allowed leakage rate, 0.0014 m^3/min (0.05 ft^3/min); and

θ = Total sampling time, min.

8.1.2.2 <u>Case II</u> (One or more component changes made during the sampling run, and the allowable leakage rate has been exceeded

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during one or more of the leak-checks prior to component changes or during the post-test leak-check): Replace V_m with the expression:

$$V_m - \sum_{i=1}^{n} \left[\theta_{i-1}(L_i - L_a) - \theta_p(L_p - L_a) \right]$$

where:

L_i = Leakage rate observed prior to "i^{th}" component change if the allowable leakage rate has been exceeded while sampling with the "i^{th}" component, m^3/min (cfm);

L_a = Maximum allowed leakage rate, 0.0014 m^3/min (0.05 ft^3/min);

θ_{i-1} = Sampling time interval between the successive component changes in which the allowable leakage rate has been exceeded, min;

L_p = Leakage rate observed during post-test leak-check, if the allowable leakage rate has been exceeded, m^3/min (cfm); and

θ_p = Sampling time interval, from the final (n^{th}) component change until the end of the sampling run, if the allowable leakage rate has been exceeded during the post-test leak-check, min.

8.2 Moisture content:

8.2.1 Calculate the Volume of Water Vapor at standard conditions:

$$V_{w(std)} = \left[\frac{\rho_w}{M_w} \right] \left[\frac{RT_{std}}{P_{std}} \right] = K_2 V_{1c}$$

where:

$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, dscm (dscf);

V_{lc} = Volume of liquid collected in the condensate reservoir added to the net increase in impinger solution volumes and silica gel weight gain during the run, mL;

ρ_w = Density of water, 0.9982 g/mL (0.002201 lb/mL);

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M_W = Molecular weight of water, 18.0 g/g-mole (lb/lb-mole);

R = Ideal gas constant, 0.06236 mm Hg-m^3/°K-g-mole (21.85 in. Hg-ft^3/°R-lb-mole);

T_{std} = Standard absolute temperature, °K (°R);

P_{std} = Standard absolute pressure, mm Hg (in. Hg); and

K_2 = 0.001333 m^3/mL for metric units
= 0.04707 ft^3/mL for English units.

8.2.2 Calculate the <u>Stack Gas Moisture Content</u> (equal to B_{ws} x 100 for conversion to percent):

$$\%M = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} = B_{ws} \times 100$$

where:

B_{ws} = Proportion of water vapor in the gas stream by volume, dimensionless;

$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, dscm (dscf); and

$V_{m(std)}$ = Volume of gas measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

8.2.3 In saturated or water-droplet-laden gas streams, make two calculations of the moisture content, one from the total volume of liquid collected in the train and one from the assumption of saturated gas-stream conditions. Use whichever method results in the lower value. To determine the moisture content based upon saturated conditions, use the average stack gas temperature in conjunction with: (1) a psychrometric chart, correcting for difference between the chart and the absolute stack pressure; or (2) saturation vapor pressure tables.

8.3 <u>Particulate concentration</u>:

8.3.1 Calculate the <u>Unit Methanol/Methylene Chloride Blank Correction</u> for all front-half samples:

$$C_{mm} = \frac{M_{mm}}{V_{mm}\rho_{mm}}$$

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where:

C_{mm} = Methanol/methylene chloride blank correction, mg/g;

M_{mm} = Mass of methanol/methylene chloride after evaporation, mg;

V_{mm} = Volume of methanol/methylene chloride used in wash, mL; and

p_{mm} = Density of 50:50 mix of methanol/methylene chloride, mg/mL (see labels on bottles).

8.3.2 Calculate the <u>Total Methanol/Methylene Chloride Blank Weight Correction</u> for each individual front-half sample:

$$W_{mm} = C_{mm} V_{mm} \rho_{mm}$$

where:

W_{mm} = Weight of residue in methanol/methylene chloride front-half wash, mg;

C_{mm} = Methanol/methylene chloride unit blank correction, mg/g;

V_{mm} = Volume of methanol/methylene chloride used for front-half wash, mL; and

ρ_{mm} = Density of 50:50 mixture of methanol and methylene chloride, mg/mL.

8.3.3 Calculate <u>Total Particulate Weight</u>:

$$W_p = (W_{pf\text{-}a} + W_{pf\text{-}b} + \ldots\ldots) + (W_{10c} + W_{3c} + W_{1c}) + (W_{pr} - W_{mm})$$

where:

W_p = Total particulate weight, mg;

$W_{pf\text{-}a+\ldots}$ = Particulate weight from filter Pf-a + Pf-b + ... ;

W_{10c}, W_{3c}, W_{1c} = Particulate weight catch from the 10-, 3-, and 1-um cyclones, respectively, mg;

W_{pr} = Weight of front-half rinse residue before blank correction, mg; and

W_{mm} = Methanol/methylene chloride blank weight correction, mg.

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8.3.4 Calculate the <u>Total Particulate Concentration</u>:

$$C_p = (0.001 \text{ g/mg}) \, (W_p/V_{m(std)})$$

where:

C_p = Concentration of particulate material in the stack gas, g/dscm (gr/dscf);

W_p = Weight of particulate material collected during run, mg; and

$V_{m(std)}$ = Volume of gas sampled, dscm (dscf).

8.3.5 To convert the above concentration to units of gr/ft^3 or lb/ft^3 for comparison with established or projected values, the following conversion factors are useful:

<u>From</u>:	<u>To</u>:	<u>Multiply By</u>:
scf	m^3	0.02832
g/ft^3	gr/ft^3	15.43
g/ft^3	lb/ft	2.205×10^{-3}
g/ft^3	g/m^3	35.315

8.4 <u>Concentration of organic material</u>:

8.4.1 Calculate the <u>Volumetric Flow Rate</u> (Q_{sd}) during the run. Determine the average stack gas velocity and volumetric flow rate from actual run data in the same manner that these were calculated during preliminary determinations (see Paragraph 4.3).

8.4.2 Calculate the <u>POHC Concentration</u>:

$$C_{pohc} = \frac{M_{pohc}}{Q_{sd}} = \frac{M_{cd-le} + M_{mrx}}{Q_{sd}}$$

where:

C_{pohc} = Concentration of POHCs in stack gas, ug/dscm;

M_{pohc} = Total mass of POHCs collected in XAD-2 and organic module rinse, and in the condensate extract, ug;

M_{cd-le} = Mass of POHCs extracted from the condensate (corrected for methylene chloride blank extraction residue), ug;

M_{mrx} = Mass of POHCs extracted from the XAD-2 sorbent and organic module rinse (corrected for methylene chloride blank extraction residue), ug; and

Q_{sd} = Volumetric flow rate during the run, dscm.

8.5 Isokinetic variation:

   8.5.1 Having calculated T_s, $V_{m(std)}$, V_s, A_n, P_s, and B_{ws}, determine the Isokinetic Variation using the equation:

$$I = K_4 \frac{T_s V_{m(std)}}{P_s V_s A_n \theta (1-B_{ws})}$$

where:

   I = Isokinetic variation, %;

   T_s = Absolute average stack gas temperature, °K (°R);

   $V_{m(std)}$ = Volume of gas sampled, dscm (dscf);

   P_s = Absolute stack gas pressure, mm Hg (in. Hg);

   V_s = Stack gas velocity, m/sec (ft/sec);

   A_n = Cross-sectional area of nozzle, m^3 (ft^3);

   θ = Net sampling time, min;

   B_{ws} = Proportion of water vapor in gas stream by volume, dimensionless; and

   K_4 = 4.320 for metric units
   = 0.09450 for English units.

   8.5.2 For the accuracy of Level 1 requirements (factor of 3) for measured particulate emissions, the isokinetic variation must be within 70-150%.

8.6 Cyclone particle-size cutoff diameter:

   8.6.1 The particle-size cutoff diameter represents that particle diameter (assuming spherical particles of unit density) at which the cyclone exhibits 50% collection efficiency; it is expressed as the "d_{50}." The range of particle size collected in each cyclone and on the filter is dependent upon the operating temperature and flow rate through each of

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these components. The particle-size cutoff diameters of 10, 3, and 1 um in the cyclones are the result of calibration of these at 400°F and 4.0 scfm (6.5 acfm). When the determined isokinetic sampling rate is not 4.0 scfm, or when it is necessary to maintain a constant subisokinetic sampling rate (still within the limits of Level 1 accuracy) during the SASS run, the particle-size cutoff diameters for the cyclones must be extrapolated.

8.6.2 Existing calibration data is insufficient to determine exac^{-4} t mathematical relationships for variations of particle-size cutoff diameter with temperature and with volumetric flow rate. The best estimates (McCain, 1983) suggest that a square, and an inverse square root dependence, respectively, exist; the extrapolation equation is presented below.

8.6.2.1 Calculate the <u>Gas Viscosity</u> from the equation:

$$\mu = (1.68 \times 10^{-4}) + (2.292 \times 10^{-7})(T)$$

where:

μ = Gas viscosity, poise; and

T = Gas temperature, °F.

8.6.2.2 Extrapolate the <u>Particle Size Cutoff Diameter</u> from:

$$D_{T_a,F_a} = D_{400,4.0} \frac{\left[\dfrac{\mu_{400}}{\mu_{T_a}}\right]^2}{\left[\dfrac{V_{4.0}}{V_{F_a}}\right]^{1/2}} = D_{400,4.0} \frac{3.37\sqrt{V_{F_a}}}{\mu^2 T_a}$$

where:

D_{T_a,F_a} =   Particle size cutoff diameter at cyclone operating a temperature and flow rate, um (note that the volumetric flow rate must be corrected to standard conditions);

$D_{400,4.0}$ =   Particle size cutoff diameter at an operating temperature of 400°F and flow rate of 4.0 scfm, um;

μ_{400} = Gas viscosity at 400°F, poise;

μ_{T_a} = Gas viscosity at operating conditions, poise;

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$$V_{4.0} = \text{Cyclone volumetric flow rate of 4.0 scfm; and}$$

$$V_{F_a} = \text{Cyclone volumetric flow rate at operating conditions, scfm.}$$

This equation reduces to:

1.
$$\frac{33.7\sqrt{V_{F_a}}}{\mu_{T_a}^2} \quad \text{for the 10-um cyclone,}$$

2.
$$\frac{10.1\sqrt{V_{F_a}}}{\mu_{T_a}^2} \quad \text{for the 3-um cyclone,}$$

3.
$$\frac{3.37\sqrt{V_{V_a}}}{\mu_{T_a}^2} \quad \text{for the 1-um cyclone,}$$

8.7 <u>Cumulative particulate weight percent less than calculated size</u>:

8.7.1 Divide the weight collected in the individual cyclones and on the filter by the total weight of particulate collected; express these as a percentage, using the following equations:

$$\% \ W_{pf} = \frac{W_{pf-a} + W_{pf-b} + \ldots}{W_p} \times 100$$

$$\% \ W_{10c} = \frac{W_{10c}}{W_p} \times 100$$

$$\% \ W_{3c} = \frac{W_{3c}}{W_p} \times 100$$

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$$\% \ W_{1c} = \frac{W_{1c}}{W_p} \times 100$$

where:

W_p = Total particulate weight collected, mg;

$W_{pf} = W_{pf-a} + W_{pf-b} + \ldots$

= Particulate weight collected on filters PF-a + PF-b, etc., mg;

W_{10c} = Particulate weight collected in 10-um cyclone, mg;

W_{3c} = Particulate weight collected in 3-um cyclone, mg;

W_{1c} = Particulate weight collected in 1-um cyclone, mg; and

100 = Conversion to percent.

8.7.2 Calculate the <u>Cumulative Weight Percent Less than the Calculated Particle Size Cutoff Diameter</u> by adding, to each weight percent, the weight percent of all fractions having a smaller particle- size cutoff diameter. Tabulate the data, using the form below as an example:

PRESENTATION OF SASS PARTICLE SIZING DATA

Stage	Weight % Collected in Stage	Cumulative Weight % Less than Calculated Particle Size Cutoff Diameter	Calculated Particle Size Cutoff Diameter
10-um cyclone			
3-um cyclone			
1-um cyclone			
Glass fiber filter			

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