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METHOD 4—DETERMINATION OF MOISTURE CONTENT IN STACK GASES

NOTE: This method does not include all the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 5, and Method 6.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Water vapor (H ₂ O)	7732-18-5	N/A

1.2 Applicability. This method is applicable for the determination of the moisture content of stack gas.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

2.2 The method contains two possible procedures: a reference method and an approximation method.

2.2.1 The reference method is used for accurate determinations of moisture content (such as are needed to calculate emission data). The approximation method, provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content (*e.g.*, drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc.) are also acceptable.

2.2.2 The reference method is often conducted simultaneously with a pollutant emission measurement run. When it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent. These calculations shall not be based upon the results of the approximation method, unless the

approximation method is shown, to the satisfaction of the Administrator, to be capable of yielding results within one percent H₂O of the reference method.

3.0 Definitions [Reserved]

4.0 Interferences

4.1 The moisture content of saturated gas streams or streams that contain water droplets, as measured by the reference method, may be positively biased. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor [capable of measuring to $\pm 1^\circ\text{C}$ (2°F)] to the reference method probe. Measure the stack gas temperature at each traverse point (see section 8.1.1.1) during the reference method traverse, and calculate the average stack gas temperature. Next, determine the moisture percentage, either by: (1) Using a psychrometric chart and making appropriate corrections if the stack pressure is different from that of the chart, or (2) using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternative methods, subject to the approval of the Administrator, shall be used.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Reference Method. A schematic of the sampling train used in this reference method is shown in Figure 4-1.

6.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation, and equipped with a filter, either in-stack (*e.g.*, a plug of glass wool inserted into the end of the probe) or heated out-of-stack (*e.g.*, as described in Method 5), to remove particulate matter. When stack conditions permit, other metals or plastic tubing may be used for the probe, subject to the approval of the Administrator.

6.1.2 Condenser. Same as Method 5, section 6.1.1.8.

6.1.3 Cooling System. An ice bath container, crushed ice, and water (or equivalent), to aid in condensing moisture.

6.1.4 Metering System. Same as in Method 5, section 6.1.1.9, except do not use sampling systems designed for flow rates higher than 0.0283 m³/min (1.0 cfm). Other metering systems, capable of maintaining a constant sampling rate to within 10 percent and determining sample gas volume to within 2 percent, may be used, subject to the approval of the Administrator.

6.1.5 Barometer and Graduated Cylinder and/or Balance. Same as Method 5, sections 6.1.2 and 6.2.5, respectively.

6.2. Approximation Method. A schematic of the sampling train used in this approximation method is shown in Figure 4-2.

6.2.1 Probe. Same as section 6.1.1.

6.2.2 Condenser. Two midget impingers, each with 30-ml capacity, or equivalent.

6.2.3 Cooling System. Ice bath container, crushed ice, and water, to aid in condensing moisture in impingers.

6.2.4 Drying Tube. Tube packed with new or regenerated 6- to 16-mesh indicating-type silica gel (or equivalent desiccant), to dry the sample gas and to protect the meter and pump.

6.2.5 Valve. Needle valve, to regulate the sample gas flow rate.

6.2.6 Pump. Leak-free, diaphragm type, or equivalent, to pull the gas sample through the train.

6.2.7 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume to within 2 percent, and calibrated over the range of flow rates and conditions actually encountered during sampling.

6.2.8 Rate Meter. Rotameter, or equivalent, to measure the flow range from 0 to 3 liters/min (0 to 0.11 cfm).

6.2.9 Graduated Cylinder. 25-ml.

6.2.10 Barometer. Same as Method 5, section 6.1.2.

6.2.11 Vacuum Gauge. At least 760-mm (30-in.) Hg gauge, to be used for the sampling leak check.

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection, Preservation, Transport, and Storage

8.1 Reference Method. The following procedure is intended for a condenser system (such as the impinger system described in section 6.1.1.8 of Method 5) incorporating volumetric analysis to measure the condensed moisture, and silica gel and gravimetric analysis to measure the moisture leaving the condenser.

8.1.1 Preliminary Determinations.

8.1.1.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine points shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a

minimum of twelve traverse points shall be used in all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator. Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack (four total sampling ports) for large stacks, to permit use of shorter probe lengths. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

8.1.1.2 Select a total sampling time such that a minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than 0.021 m³/min (0.75 cfm). When both moisture content and pollutant emission rate are to be determined, the moisture determination shall be simultaneous with, and for the same total length of time as, the pollutant emission rate run, unless otherwise specified in an applicable subpart of the standards.

8.1.2 Preparation of Sampling Train.

8.1.2.1 Place known volumes of water in the first two impingers; alternatively, transfer water into the first two impingers and record the weight of each impinger (plus water) to the nearest 0.5 g. Weigh and record the weight of the silica gel to the nearest 0.5 g, and transfer the silica gel to the fourth impinger; alternatively, the silica gel may first be transferred to the impinger, and the weight of the silica gel plus impinger recorded.

8.1.2.2 Set up the sampling train as shown in Figure 4-1. Turn on the probe heater and (if applicable) the filter heating system to temperatures of approximately 120°C (248°F), to prevent water condensation ahead of the condenser. Allow time for the temperatures to stabilize. Place crushed ice and water in the ice bath container.

8.1.3 Leak Check Procedures. It is recommended, but not required, that the volume metering system and sampling train be leak-checked as follows:

8.1.3.1 Metering System. Same as Method 5, section 8.4.1.

8.1.3.2 Sampling Train. Disconnect the probe from the first impinger or (if applicable) from the filter holder. Plug the inlet to the first impinger (or filter holder), and pull a 380 mm (15 in.) Hg vacuum. A lower vacuum may be used, provided that it is not exceeded during the test. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m³/min (0.020 cfm), whichever is less, is unacceptable. Following the leak check, reconnect the probe to the sampling train.

8.1.4 Sampling Train Operation. During the sampling run, maintain a sampling rate within 10 percent of constant rate, or as specified by the Administrator. For each run, record the data required on a data sheet similar to that shown in Figure 4-3. Be sure to record the dry gas meter reading at the beginning and end of each sampling time increment and whenever sampling is halted. Take other appropriate readings at each sample point at least once during each time increment.

NOTE: When Method 4 is used concurrently with an isokinetic method (*e.g.*, Method 5) the sampling rate should be maintained at isokinetic conditions rather than 10 percent of constant rate.

8.1.4.1 To begin sampling, position the probe tip at the first traverse point. Immediately start the pump, and adjust the flow to the desired rate. Traverse the cross section, sampling at each traverse point for an equal length of time. Add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the silica gel outlet.

8.1.4.2 After collecting the sample, disconnect the probe from the first impinger (or from the filter holder), and conduct a leak check (mandatory) of the sampling train as described in section 8.1.3.2. Record the leak rate. If the leakage rate exceeds the allowable rate, either reject the test results or correct the sample volume as in section 12.3 of Method 5.

8.2 Approximation Method.

NOTE: The approximation method described below is presented only as a suggested method (see section 2.0).

8.2.1 Place exactly 5 ml water in each impinger. Leak check the sampling train as follows: Temporarily insert a vacuum gauge at or near the probe inlet. Then, plug the probe inlet and pull a vacuum of at least 250 mm (10 in.) Hg. Note the time rate of change of the dry gas meter dial; alternatively, a rotameter (0 to 40 ml/min) may be temporarily attached to the dry gas meter outlet to determine the leakage rate. A leak rate not in excess of 2 percent of the average sampling rate is acceptable.

NOTE: Release the probe inlet plug slowly before turning off the pump.

8.2.2 Connect the probe, insert it into the stack, and sample at a constant rate of 2 liters/min (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft³) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as indicated by Figure 4-4.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
Section 8.1.1.4	Leak rate of the sampling system cannot exceed four percent of the average sampling rate or 0.00057 m ³ /min (0.020 cfm)	Ensures the accuracy of the volume of gas sampled. (Reference Method).
Section 8.2.1	Leak rate of the sampling system cannot exceed two percent of the average sampling rate	Ensures the accuracy of the volume of gas sampled. (Approximation Method).

9.2 Volume Metering System Checks. Same as Method 5, section 9.2.

10.0 Calibration and Standardization

NOTE: Maintain a laboratory log of all calibrations.

10.1 Reference Method. Calibrate the metering system, temperature sensors, and barometer according to Method 5, sections 10.3, 10.5, and 10.6, respectively.

10.2 Approximation Method. Calibrate the metering system and the barometer according to Method 6, section 10.1 and Method 5, section 10.6, respectively.

11.0 Analytical Procedure

11.1 Reference Method. Measure the volume of the moisture condensed in each of the impingers to the nearest ml. Alternatively, if the impingers were weighed prior to sampling, weigh the impingers after sampling and record the difference in weight to the nearest 0.5 g. Determine the increase in weight of the silica gel (or silica gel plus impinger) to the nearest 0.5 g. Record this information (see example data sheet, Figure 4-5), and calculate the moisture content, as described in section 12.0.

11.2 Approximation Method. Combine the contents of the two impingers, and measure the volume to the nearest 0.5 ml.

12.0 Data Analysis and Calculations

Carry out the following calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after final calculation.

12.1 Reference Method.

12.1.1 Nomenclature.

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

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 (mm Hg)(m³)/(g-mole)(°K) for metric units and 21.85 (in. Hg)(ft³)/(lb-mole)(°R) for English units.

T_m = Absolute temperature at meter, °K (°R).

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

V_f = Final volume of condenser water, ml.

V_i = Initial volume, if any, of condenser water, ml.

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

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

$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

$V_{wsg(std)}$ = Volume of water vapor collected in silica gel, corrected to standard conditions, scm (scf).

W_f = Final weight of silica gel or silica gel plus impinger, g.

W_i = Initial weight of silica gel or silica gel plus impinger, g.

Y = Dry gas meter calibration factor.

ΔV_m = Incremental dry gas volume measured by dry gas meter at each traverse point, dcm (dcf).

ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

12.1.2 Volume of Water Vapor Condensed.

$$\begin{aligned} V_{wc(std)} &= \frac{(V_f - V_i) \rho_w R T_{std}}{P_{std} M_w} \quad Eq. 4-1 \\ &= K_1 (V_f - V_i) \end{aligned}$$

Where:

$K_1 = 0.001333 \text{ m}^3/\text{ml}$ for metric units,

$= 0.04706 \text{ ft}^3/\text{ml}$ for English units.

12.1.3 Volume of Water Collected in Silica Gel.

$$\begin{aligned} V_{wsg(std)} &= \frac{(W_f - W_i) R T_{std}}{P_{std} M_w K_2} \quad Eq. 4-2 \\ &= K_3 (W_f - W_i) \end{aligned}$$

Where:

$K_2 = 1.0 \text{ g/g}$ for metric units,

$= 453.6 \text{ g/lb}$ for English units.

$K_3 = 0.001335 \text{ m}^3/\text{g}$ for metric units,

$= 0.04715 \text{ ft}^3/\text{g}$ for English units.

12.1.4 Sample Gas Volume.

$$\begin{aligned} V_{m(\text{std})} &= \frac{V_m Y P_m T_{\text{std}}}{P_{\text{std}} T_m} & \text{Eq. 4-3} \\ &= K_4 Y \frac{V_m P_m}{T_m} \end{aligned}$$

Where:

$K_4 = 0.3855$ °K/mm Hg for metric units,

$= 17.64$ °R/in. Hg for English units.

NOTE: If the post-test leak rate (Section 8.1.4.2) exceeds the allowable rate, correct the value of V_m in Equation 4-3, as described in section 12.3 of Method 5.

12.1.5 Moisture Content.

$$B_{ws} = \frac{V_{wc(\text{std})} + V_{wsg(\text{std})}}{V_{wc(\text{std})} + V_{wsg(\text{std})} + V_{m(\text{std})}} \quad \text{Eq. 4-4}$$

12.1.6 Verification of Constant Sampling Rate. For each time increment, determine the ΔV_m . Calculate the average. If the value for any time increment differs from the average by more than 10 percent, reject the results, and repeat the run.

12.1.7 In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see section 4.1), and another based upon the results of the impinger analysis. The lower of these two values of B_{ws} shall be considered correct.

12.2 Approximation Method. The approximation method presented is designed to estimate the moisture in the stack gas; therefore, other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content for the purpose of determining isokinetic sampling rate settings.

12.2.1 Nomenclature.

B_{wm} = Approximate proportion by volume of water vapor in the gas stream leaving the second impinger, 0.025.

B_{ws} = Water vapor in the gas stream, proportion by volume.

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 [(mm Hg)(m³)]/[(g-mole)(K)] for metric units and 21.85 [(in. Hg)(ft³)]/[(lb-mole)(°R)] for English units.

T_m = Absolute temperature at meter, °K (°R).

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

V_f = Final volume of impinger contents, ml.

V_i = Initial volume of impinger contents, ml.

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

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

$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

Y = Dry gas meter calibration factor.

ρ_w = Density of water, 0.09982 g/ml (0.002201 lb/ml).

12.2.2 Volume of Water Vapor Collected.

$$\begin{aligned} V_{wc(std)} &= \frac{(V_f - V_i) \rho_w R T_{std}}{P_{std} M_w} && \text{Eq. 4-5} \\ &= K_5 (V_f - V_i) \end{aligned}$$

Where:

K_5 = 0.001333 m³/ml for metric units,

=0.04706 ft³/ml for English units.

12.2.3 Sample Gas Volume.

$$\begin{aligned} V_{m(std)} &= \frac{V_m Y P_m T_{std}}{P_{std} T_m} && \text{Eq. 4-6} \\ &= K_6 Y \frac{V_m P_m}{T_m} \end{aligned}$$

Where:

K_6 = 0.3855 °K/mm Hg for metric units,

=17.64 °R/in. Hg for English units.

12.2.4 Approximate Moisture Content.

$$B_{ws} = \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + B_{wm} \quad Eq. 4-7$$
$$= \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + (0.025)$$

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

16.1 The procedure described in Method 5 for determining moisture content is an acceptable alternative to Method 4.

16.2 The procedures in Method 6A for determining moisture is an acceptable alternative to Method 4.

16.3 Method 320 is an acceptable alternative to Method 4 for determining moisture.

16.4 Using F-factors to determine moisture is an acceptable alternative to Method 4 for a combustion stack not using a scrubber. If this option is selected, calculate the moisture content as follows:

$$B_{WS} = B_H + B_A + B_F$$

Where:

B_A = Mole Fraction of moisture in the ambient air.

$$B_A = \frac{\%RH}{100 P_{bar}} \left[10^{6.6912 - \left(\frac{3144}{T + 390.86} \right)} \right]$$

B_F = Mole fraction of moisture from free water in the fuel.

$$B_F = \left[\frac{0.0036W^2 + 0.075W}{100} \right] \left[\frac{20.9 - O_2}{20.9} \right]$$

B_H = <Mole fraction of moisture from the hydrogen in the fuel.

$$B_H = \left[1 - \frac{F_d}{F_W} \right] \frac{(20.9 - O_2)}{20.9}$$

B_{ws} = Mole fraction of moisture in the stack gas.

F_d = Volume of dry combustion components per unit of heat content at 0 percent oxygen, dscf/10⁶ Btu (scm/J). See Table 19-2 in Method 19.

F_w = Volume of wet combustion components per unit of heat content at 0 percent oxygen, wet scf/10⁶ Btu (scm/J). See Table 19-2 in Method 19.

%RH = Percent relative humidity (calibrated hydrometer acceptable), percent.

P_{Bar} = Barometric pressure, in. Hg.

T = Ambient temperature, °F.

W = Percent free water by weight, percent.

O₂ = Percent oxygen in stack gas, dry basis, percent.

17.0 References

1. Air Pollution Engineering Manual (Second Edition). Danielson, J.A. (ed.). U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Research Triangle Park, NC. Publication No. AP-40. 1973.
2. Devorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, CA. November 1963.
3. Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases. Western Precipitation Division of Joy Manufacturing Co. Los Angeles, CA. Bulletin WP-50. 1968.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

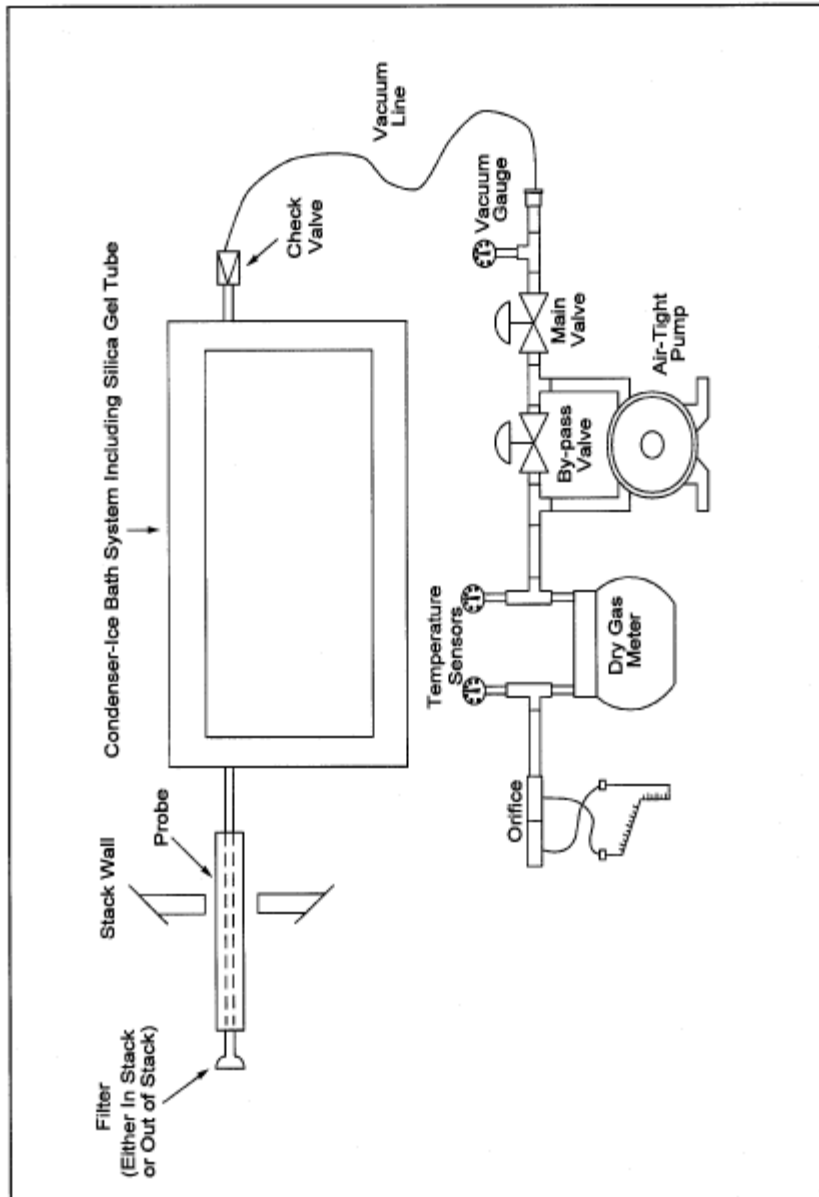


Figure 4-1. Moisture Sampling Train-Reference Method

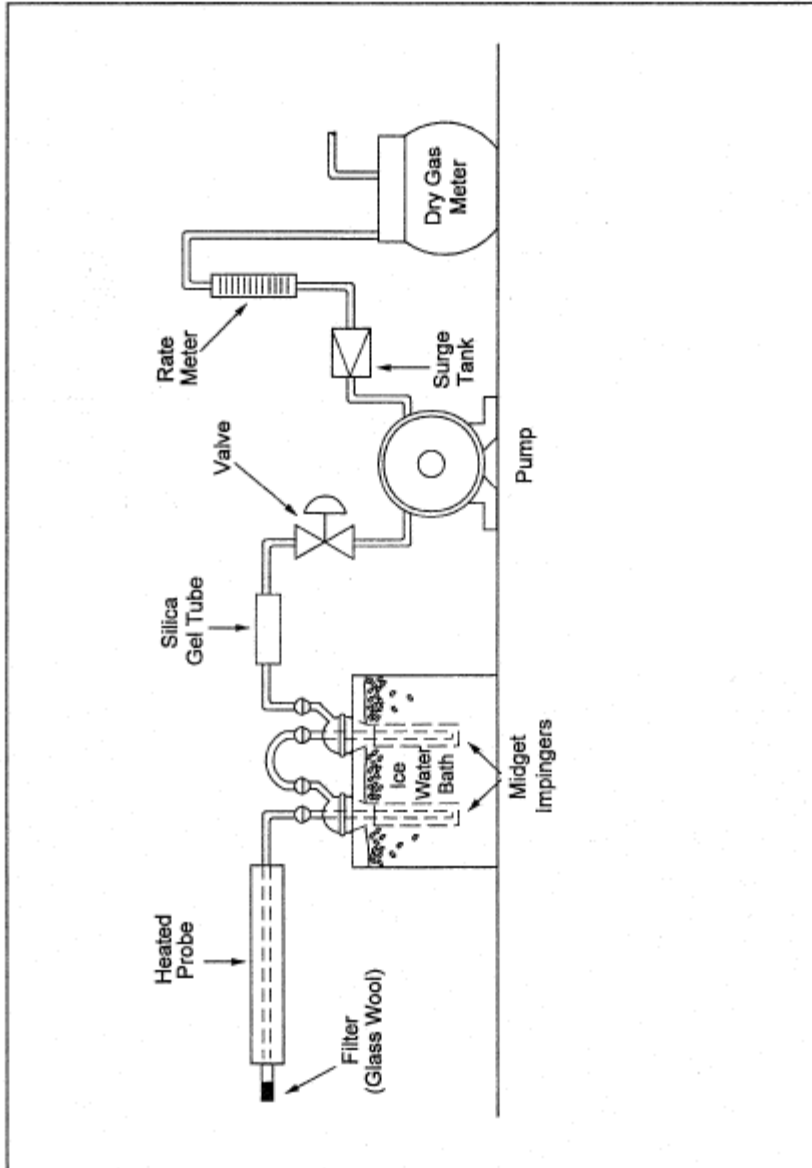


Figure 4-2. Moisture Sampling Train - Approximation Method.

Plant _____
 Location _____
 Operator _____
 Date _____
 Run No. _____
 Ambient temperature _____
 Barometric pressure _____
 Probe Length _____

SCHEMATIC OF STACK CROSS SECTION

Traverse Pt. No.	Sampling time (Δ), min	Stack temperature °C ((°deg;F)	Pressure differential across orifice meter ΔH mm (in.) H ₂ O	Meter reading gas sample volume m ³ (ft ³)	ΔV _m m ³ (ft ³)	Gas sample temperature at dry gas meter		Temperature of gas leaving condenser or last impinger °C ((°deg;F)
						Inlet T _{m_{in}} °C ((°deg;F)	Outlet T _{m_{out}} °C ((°deg;F)	
Average								

Location _____
 Test _____
 Date _____
 Operator _____
 Barometric pressure _____
 Comments: _____

Figure 4-3. Moisture Determination—Reference Method

Clock time	Gas Volume through meter, (V _m), m ³ (ft ³)	Rate meter setting m ³ /min (ft ³ /min)	Meter temperature °C ((°deg;F)

Figure 4-4. Example Moisture Determination Field Data Sheet—Approximation Method

	Impinger volume, ml	Silica gel weight, g
Final		
Initial		
Difference		

Figure 4-5. Analytical Data—Reference Method