STATE OF WASHINGTON DEPARTMENT OF ECOLOGY December 1, 1976
SOURCE TEST METHOD 14

CARBONYL DETERMINATION BISULFITE ABSORPTION

1. Principle

Carbonyls are collected in a sodium bisulfite solution and lab-analyzed using an iodine titration.

2. Description

Stack gases are sampled through a non-reactive probe and bubbled into a solution of sodium bisulfite forming an aldehyde bisulfite complex. Excess bisulfite is precipitated out with an iodine solution. By adjusting the pH of the solution, the complex is decomposed, freeing the bisulfite ion equivalent to the aldehydes in the sample. The liberated bisulfite ion is then titrated with the standard iodine solution.

3. Equipment and Reagents

Equipment:

a. Non-reactive probe, such as stainless steel.

b. Connecting tubing such as Teflon.

c. Collection system comprised of three Greenburg-Smith impingers and drying tube. The first and third impingers can be modified by replacing the tip with a half-inch O.D. glass tube, extending to a half-inch of the bottom of the flask. The drying tube contains approximately 200 grams of silica gel.

d. Leakless valve to regulate flow.

e. Leakless pump.

f. Dry test gas meter with a thermometer to indicate temperature.

g. Barometer to measure atmospheric pressure within 0.1 in. Hg.

Reagents:

a. 1.0% $NaHSO_3$.

b. 1.0% starch solution.

c. 0.05 N sodium thiosulfate.

d. 0.1 N (approx.) iodine.

by adding 0.05 N sodium thiosulfite drop-wise. Add 0.005 N iodine to a faint blue end point. Cool thoroughly in an ice bath and add 15 ml of chilled buffer solution. Allow to stand in the ice bath for 10 to 15 minutes after buffer solution is added, then titrate the liberated bisulfate with 0.005 N iodine to the same faint blue end point. Make a blank using the same volume of $NaHSO_3$ as used in the aliquot.

Calculations:

The concentration of carbonyls is determined by multiplying the number of moles CH_2O collected by the molar volume and divided by the sample volume corrected to standard conditons and expressed in ppm.

6. Calibration

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

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

Barometers should be calibrated against a mercury barometer.

7. References

a. Goldman, F.A. and Yagoda, H., Ind. Eng. Chem., Anal. Ed. Vol. 15:377 (1943.

b. Levaggi, D.A. and Feldstein M., Journal of Air Pollution Control Association Vol. 19, No. 1, 43.

c. Jacobs, M.B., "The Chemical Analysis of Air Pollutants, 1960."

d. Regulation 3, Bay Area Air Pollution Control District, San Francisco, Calif., 1969.

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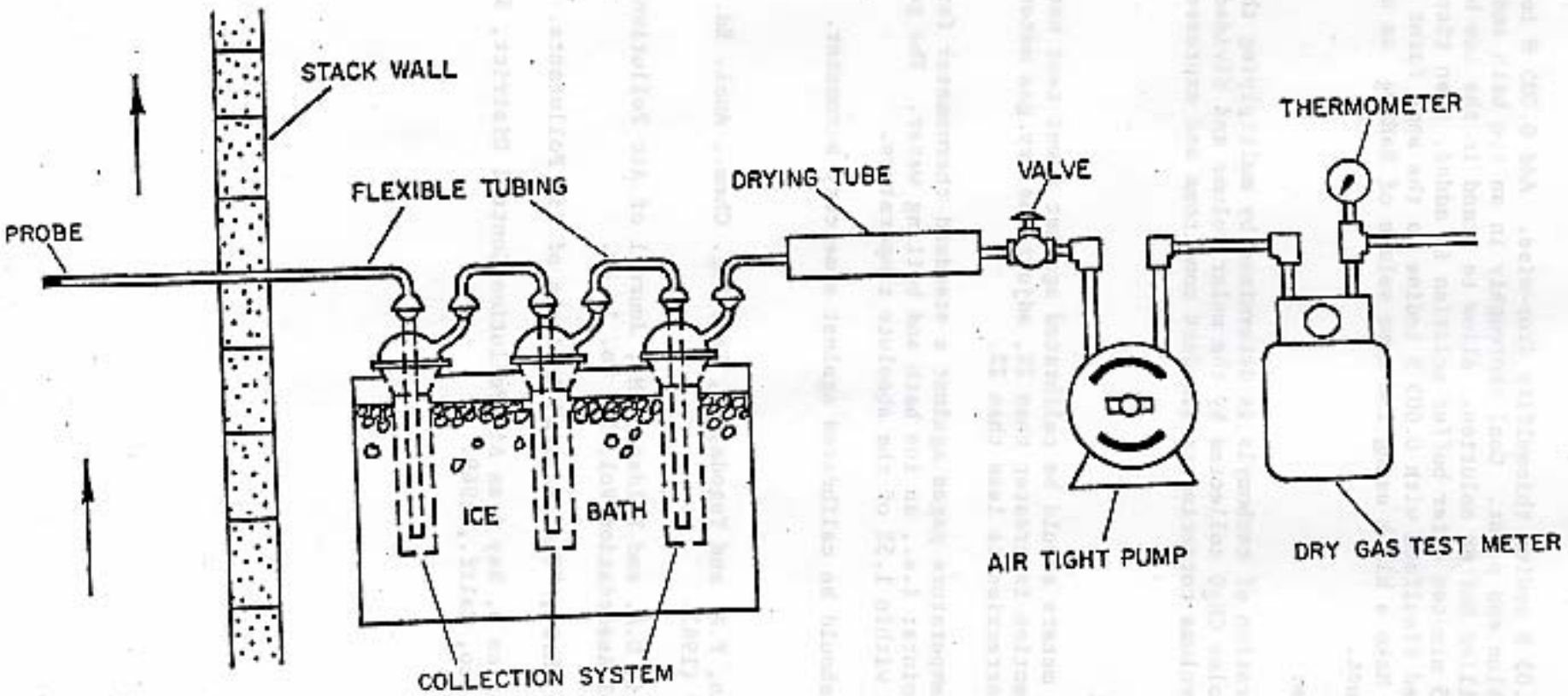

Figure 1: DOE METHOD 14 - CARBONYLS SAMPLING TRAIN.

Test No. _____

Date _____

STATE OF WASHINGTON DEPARTMENT OF ECOLOGY

TOTAL CARBONYLS

DATA AND CALCULATIONS

SAMPLING STATION _____ SAMPLE NUMBER _____

Sampling Time	Gas Meter			Operating Conditions
	Reading	Pressure in H_g	Temperature	
	G Ft.^{3}	H "Hg	°F	

Analysis Method _____

A. Absorbant Volume; ml _____

B. Normality of Standard Reagent _____

C. Dilution Factor: $\frac{A}{\text{ml Aliquot}}$ _____

D. Reagent Titration Volume for Blank; ml _____

E. Reagent Titration Volume for Sample; ml _____

Lab Analyst _____ Date in _____ Date out _____

F. Mass Collected: (E-D) x B x C x 30.0/2, mg _____

J. Sampled Gas Standard Volume: $\frac{520}{(460 + 1)} \times \frac{H \times G}{29.9}$ SDCF _____

K. Average Sampling Rate: $\frac{J}{\text{Time}}$, SDCFM _____

L. Stack Gas Flow Rate, SDCFM _____

M. Volume Concentration: $\frac{F \times 0.8363 \text{ CF/gmol} \times 10^6}{J \times 30.0 \times 10^3 \text{ mg/gmol}}$, PPM _____

N. Mass Concentration @ 60°F, 35.3 x F/J, mg/M^3 _____

P. Emission Rate: $M \times 10^{-6} \frac{(30.0)}{(28.9)} (0.07636 \frac{\text{lbs}}{\text{CF}}) \times L \times 60$, lbs/hr _____

ECY 020-75