

VII. REFERENCES

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VIII. APPENDIX

SAMPLING AND ANALYTICAL METHOD FOR FURFURYL ALCOHOL IN AIR

This method is NIOSH Method No. S-365 [50].

Principle of the Method

A known volume of air is drawn through a glass tube containing Porapak Q to adsorb furfuryl alcohol vapor. Furfuryl alcohol is desorbed from the Porapak Q with acetone, and the sample is analyzed by gas chromatography.

Range and Sensitivity

This method was validated over the range of 117-469 mg/cu m at an atmospheric temperature of 22 C (71.6 F) and atmospheric pressure of 765 mmHg, using a 6-liter sample. This sample size is based on the capacity of the Porapak Q to collect vapors of furfuryl alcohol in air at high relative humidity. The method may be capable of measuring smaller amounts if the desorption efficiency is adequate. Desorption efficiency must be determined over the range used.

The upper limit of the range of the method depends on the adsorptive capacity of the Porapak Q. This capacity may vary with the concentrations of furfuryl alcohol and other substances in the air. Breakthrough is defined as the time that the effluent concentration from the collection tube (containing 150 mg of Porapak Q) reaches 5% of the concentration in the test gas mixture. Breakthrough did not occur after sampling for 7 hours at an average sampling rate of 0.044 liter/minute and relative humidity of greater than 80% and temperature of 19 C (66.2 F). The breakthrough test was conducted at 418 mg/cu m.

Interferences

When other compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

Any compound that has the same retention time as furfuryl alcohol at the operating conditions described in this method will interfere. Retention-time data on a single column should not be considered proof of chemical identity.

Precision and Accuracy

The coefficient of variation for the total analytical and sampling method in the range of 117-469 mg/cu m was 0.072. This value corresponds to a 34 mg/cu m standard deviation at the limit of 200 mg/cu m.

On the average, the concentrations obtained in the laboratory validation study at 100, 200, and 400 mg/cu m were 1.6% lower than the "true" concentrations for 18 samples. Any difference between the "found" and "true" concentrations may not represent a bias in the sampling and analytical method, but rather a random variation from the experimentally determined "true" concentration. Therefore, the method has no evident bias. The coefficient of variation is a good measure of the accuracy of the method, since the recoveries and storage stability were good. Storage stability studies on samples collected from a test atmosphere at 224.2 mg/cu m indicate that collected samples are stable for at least 7 days.

Advantages and Disadvantages

The sampling device is small and portable and involves no liquids. Interferences are minimal, and most of those that occur can be eliminated by altering chromatographic conditions. The tubes are analyzed by means of a quick instrumental method.

One disadvantage of the method is that the amount of sample that can be taken is limited by the mass of furfuryl alcohol that the tube will hold before overloading. When the amount of furfuryl alcohol found on the backup section of the Porapak Q tube exceeds 25% of that found on the front section, the probability of sample loss exists.

The precision of the method is limited by the reproducibility of the pressure drop across the tubes. This drop will affect the flowrate and cause the measured volume to be imprecise because the pump is usually calibrated for one tube only.

Apparatus

(a) Personal sampling pump: A calibrated personal sampling pump, the flowrate of which can be determined within 5% at the recommended flowrate.

(b) Porapak Q tubes: Glass tube with both ends unsealed, 8.5-cm long with a 6-mm OD and a 4-mm ID, containing two sections of 50/80 mesh Porapak Q separated by a 2-mm portion of urethane foam. The adsorbing section of the tube contains 150 mg of Porapak Q, and the backup section contains 75 mg. A plug of silylated glass wool is placed at each end of the tube. The pressure drop across the tube must be less than 10 mmHg at a flowrate of

0.05 liter/minute. Immediately prior to packing, the tubes should be acetone rinsed and dried to eliminate the problem of Porapak Q adhering to the walls of the glass tubes. The Porapak Q tubes are capped with plastic caps at each end.

Sorbent washing procedure: Prior to usage, Porapak Q is washed with acetone and dried to reduce or eliminate the effects of unreacted monomers, solvents, and manufacturer's batch-to-batch differences in production. A quantity of Porapak Q is placed in a sintered glass filter fitted to a large vacuum flask. Reagent grade acetone, of a volume equal to twice that of Porapak Q, is added to the sorbent and mixed, and the pressure is reduced. Repeat the operation of wash-mix-vacuum six times. The sorbent is then transferred to an evaporating dish and dried in a vacuum oven at 120 C (248 F) under slight vacuum (635 mmHg) for 4 hours.

- (c) Gas chromatograph equipped with a flame-ionization detector.
- (d) Column (.94 m long x 3-mm OD stainless steel) packed with 50/80 mesh Porapak Q.
- (e) An electronic integrator or some other suitable method of determining peak areas.
- (f) Sample containers: 2-ml glass sample containers with glass stoppers or Polyseal caps (or equivalent).
- (g) Microliter syringes: 10- μ l and other convenient sizes for preparing standards.
- (h) Pipets: Delivery type, 1.0-ml and other convenient sizes.
- (i) Volumetric flasks: 10-ml and other convenient sizes for preparing standard solutions.
- (j) Stopwatch.

Reagents

- (a) Acetone, reagent grade.
- (b) Furfuryl alcohol, Baker grade (or equivalent). Furfuryl alcohol is a clear viscous liquid. If furfuryl alcohol is received from the manufacturer as a dark-colored liquid, it should be distilled before use. The boiling point of furfuryl alcohol is 170 C (338 F).
- (c) Benzene, chromatographic quality.
- (d) Nitrogen, purified.

- (e) Hydrogen, prepurified.
- (f) Filtered compressed air.

Procedure

(a) Cleaning of Equipment

All glassware used for the laboratory analysis should be detergent washed, thoroughly rinsed with tapwater and distilled water, and dried.

(b) Calibration of Sampling Pumps

Each personal sampling pump must be calibrated with a representative Porapak Q tube in the line to minimize errors associated with uncertainties in the volume sampled. Figure IX-2 shows a typical setup for calibrating sampling pumps with a soapbubble meter.

(c) Collection and Shipment of Samples

(1) Immediately before sampling, the caps from the ends of the Porapak Q tube should be removed. All tubes must be packed with Porapak Q from the same manufacturer's lot.

(2) The smaller section of Porapak Q is used as a backup and should be positioned nearer the sampling pump.

(3) The tube should be placed in a vertical direction during sampling to minimize channeling through the Porapak Q.

(4) Air being sampled should not be passed through any hose or tubing before entering the Porapak Q tube.

(5) A sample size of 6 liters is recommended. A flowrate between 0.01 and 0.05 liter/minute is used for sampling; a flowrate less than 0.010 liter/minute should not be used. The sampling time, flowrate, and type of sampling pump used should be recorded.

(6) The temperature, pressure, and relative humidity of the atmosphere being sampled should be recorded. If pressure reading is not available, the elevation should be recorded.

(7) The Porapak Q tube should be capped with plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

(8) With each batch of 10 samples, 1 tube from the same lot of tubes used for sample collection should be submitted. This tube must be

subjected to exactly the same handling as the samples except that no air is drawn through it. This tube should be labeled as the blank. A minimum of 18 extra Porapak Q tubes should be provided for desorption efficiency determinations.

(9) Capped tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

(10) A sample of the bulk material should be submitted to the laboratory in a glass container with a Polyseal cap. This sample should not be transported in the same container as the Porapak Q tubes.

(d) Analysis of Samples

(1) Preparation of Samples

Remove the plastic cap from the inlet end of the Porapak Q tube. Remove the glass wool plug and transfer the first (larger) section of Porapak Q to a 2-ml stoppered sample container. Remove the separating section of urethane foam and transfer the backup section of Porapak Q to another stoppered container. Analyze these two sections separately. Firm tapping of the tube may be necessary to affect complete transfer of the Porapak Q.

(2) Desorption of Samples

Prior to analysis, pipet 1.0 ml of acetone into each sample container. Cap and shake the sample vigorously. Desorption is complete in 15 minutes. Complete analysis within 1 day after the furfuryl alcohol is desorbed.

(3) Gas Chromatographic Conditions

The typical operating conditions for the gas chromatograph are:

- 50 ml/minute (60 psig) nitrogen carrier gas flow.
- 65 ml/minute (24 psig) hydrogen gas flow to detector.
- 500 ml/minute (50 psig) airflow to detector.
- 225 C injector manifold temperature.
- 225 C detector manifold temperature.
- 200 C column temperature.

A retention time of approximately 11 minutes is to be expected for furfuryl alcohol under these conditions and using the column recommended in Apparatus (d). The acetone will elute from the column before the furfuryl alcohol.

(4) Injection

The first step in the analysis is the injection of the sample into the gas chromatograph. To eliminate difficulties arising from blowback or evaporation of solvent within the syringe needle, one should employ the solvent flush injection technique. The 10- μ l syringe is first flushed with solvent several times to wet the barrel and plunger. Three microliters of solvent are drawn into the syringe to increase the accuracy and reproducibility of the injected sample volume. The needle is removed from the solvent, and the plunger is pulled back about 0.2 μ l to separate the solvent flush from the sample with a pocket of air to be used as a marker. The needle is then immersed in the sample, and a 5- μ l aliquot is withdrawn, taking into consideration the volume of the needle, since the sample in the needle will be completely injected. After the needle is removed from the sample and prior to injection, the plunger is pulled back 1.2 μ l to minimize evaporation of the sample from the tip of the needle. It should be observed that the sample occupies 4.9-5.0 μ l in the barrel of the syringe. Duplicate injections of each sample and standard should be made. No more than a 3% difference in area is to be expected. It is not advisable to use an automatic sample injector because of possible plugging of the syringe needle with Porapak Q.

(5) The area of the sample peak is measured by an electronic integrator or some other suitable form of area measurement, and results are read from a standard curve prepared as discussed below.

(e) Determination of Desorption Efficiency

(1) The desorption efficiency of a particular compound can vary from one laboratory to another and also from one batch of Porapak Q to another. Thus, it is necessary to determine the fraction of the specific compound that is removed in the desorption process for a particular batch of Porapak Q.

(2) Porapak Q, equivalent to the amount in the first section of the sampling tube (150 mg), is measured into a 64-mm, 4-mm ID glass tube, flame sealed at one end. This Porapak Q must be from the same batch as that used in obtaining the samples. The open end is capped with Parafilm. A known amount of a benzene solution of furfuryl alcohol containing 300 mg/ml is injected directly into the Porapak Q with a microliter syringe, and the tube is capped with more Parafilm. The amount injected is equivalent to that present in a 6-liter air sample at the selected level. It is not practical to inject the neat liquid directly onto the Porapak Q because the amounts to be added would be too small to measure accurately.

Six tubes at each of three levels, equivalent to 100, 200, and 400 mg/cu m, are prepared in this manner and allowed to stand for at least overnight to assure complete adsorption of the furfuryl alcohol onto the

Porapak Q. These tubes are referred to as the samples. A parallel blank tube should be treated in the same manner except that no sample is added to it. The sample and blank tubes are desorbed and analyzed in exactly the same manner as the sampling tube described in Analysis of Samples.

Two or three standards are prepared by injecting the same volume of furfuryl alcohol into 1.0 ml of acetone with the same syringe used in the preparation of the samples. These are analyzed with the samples.

The desorption efficiency (DE) equals the average weight in mg recovered from the tube divided by the weight in mg added to the tube, or

$$DE = \frac{\text{average weight recovered (mg)}}{\text{weight added (mg)}}$$

The desorption efficiency is dependent on the amount of furfuryl alcohol collected on the Porapak Q. Plot the desorption efficiency vs weight of furfuryl alcohol found. This curve is used in Calculations (d) to correct for adsorption losses.

In any handling of benzene, stringent precautions must be exerted to limit occupational exposure and to prevent discharges to community air and water. The standard promulgated by the Occupational Safety and Health Administration (29 CFR 1910.1028) should be adhered to in any handling of benzene. It would be desirable if a solvent less toxic than benzene were used in the preparation of samples for the determination of the desorption efficiency of furfuryl alcohol from Porapak Q, but the necessary experimental work has not been performed. For guidance of those who prefer to use a solvent other than benzene, such a solvent should meet the following criteria: (1) it should be a good solvent for furfuryl alcohol, so that concentrated solutions of furfuryl alcohol can be prepared, (2) it should not dissolve the Porapak Q, and (3) it should be less strongly adsorbed by Porapak Q than is furfuryl alcohol. It seems likely that cyclohexane would meet these requirements, but necessary tests to prove its utility have not yet been conducted.

Calibration and Standards

A series of standards, varying in concentration over the range corresponding to about 0.1-3 times the permissible exposure limit is prepared and analyzed under the same gas chromatographic conditions and during the same time period as the unknown samples. Curves are established by plotting concentration in mg/1.0 ml vs peak area.

Note: Since no internal standard is used in this method, standard solutions must be analyzed at the same time that the sample analysis is done. This will minimize the effect of known day-to-day variations and variations during the same day of the flame-ionization detector response.

(a) Prepare a stock standard solution containing 120 mg/ml of furfuryl alcohol in acetone.

(b) From the above stock solution, appropriate aliquots are withdrawn and dilutions are made in acetone. Prepare at least five working standards to cover the range of 0.12-3.6 mg/1.0 ml. This range is based on a 6-liter sample.

(c) Prepare a standard calibration curve by plotting concentration of furfuryl alcohol in mg/1.0 ml vs peak area.

Calculations

(a) Read the weight, in mg, corresponding to each peak area from the standard curve. No volume corrections are needed because the standard curve is based on mg/1.0 ml acetone and the volume of sample injected is identical to the volume of the standards injected.

(b) Corrections for the blank must be made for each sample.

$$\text{mg} = \text{mg sample} - \text{mg blank}$$

where:

$$\begin{aligned} \text{mg sample} &= \text{mg found in front section of sample tube} \\ \text{mg blank} &= \text{mg found in front section of blank tube} \end{aligned}$$

A similar procedure is followed for the backup sections.

(c) Add the weights found in the front and backup sections to determine the total weight of the sample.

(d) Read the desorption efficiency from the curve (see Procedure (e)(2)) for the amount found in the front section. Divide the total weight by this desorption efficiency to obtain the corrected mg/sample.

$$\text{corrected mg/sample} = \frac{\text{total weight}}{\text{DE}}$$

(e) For personal sampling pumps with rotameters only, the following correction should be made:

$$\text{corrected volume} = f \times t \left(\sqrt{\frac{P_1}{P_2} \times \frac{T_2}{T_1}} \right)$$

where:

f = flowrate sampled
t = sampling time
P₁ = pressure during calibration of sampling pump (mmHg)
P₂ = pressure of air sampled (mmHg)
T₁ = temperature during calibration of sampling pump (K)
T₂ = temperature of air sampled (K)

(f) The concentration of furfuryl alcohol in the air sampled can be expressed in mg/cu m.

$$\text{mg/cu m} = \frac{\text{corrected mg} \times 1,000 \text{ (liters/cu m)}}{\text{corrected air volume sampled (liters)}}$$

(g) Another method of expressing concentration is in ppm.

$$\text{ppm} = \text{mg/cu m} \times \frac{24.45}{98.10} \times \frac{760}{P} \times \frac{T + 273}{298}$$

where:

P = pressure (mmHg) of air sampled
T = temperature (C) of air sampled
24.45 = molar volume (liter/mole) at 25 C and 760 mmHg
760 = standard pressure (mmHg)
298 = standard temperature (K)

IX. TABLES AND FIGURES

TABLE IX-1

PHYSICAL AND CHEMICAL PROPERTIES OF FURFURYL ALCOHOL

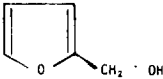
Properties		Reference
Molecular formula		
Formula weight	98.10	2
Physical state	Colorless syrupy liquid	2
Melting point (stable form)	-14.63 C	2
Freezing point	-20 C	56
Boiling point, at 760 mmHg	170 C	2
Flashpoint:		
Open cup	75 C	56
Closed cup	65 C	2
Ignition temperature in air	391	2
Flammability limits (in dry air at 72.5-122 C):		
Lower limit	1.8% (by vol)	2
Upper limit	16.3% (by vol)	2
Density (g/ml at 20 C)	1.1296	67
Vapor density (air = 1)	3.38	68
Percent in saturated air (at 31.8 C)	0.13	68
Density of saturated air (air = 1)	1.003	68
Viscosity (centipoise at 25 C)	4.62	2
Refractive index:		
At 20 C	1.4868	67
At 25 C	1.484	69
Surface tension (dynes/cm) at 25 C	38.2	56
Specific heat of liquid (cal/g/C):		
At 0 C	0.472	2
At 25 C	0.502	2
Solubility:		
In water	Miscible	2
In ethyl ether	"	2
In ethanol	"	2
In chloroform	"	69
In benzene	"	69
In petroleum hydrocarbons and most oils	Insoluble	69
Conversion factors (760 mmHg at 25 C)	1 mg/cu m = 0.25 ppm	
	1 ppm = 4.01 mg/cu m	

TABLE IX-2

OCCUPATIONS WITH POTENTIAL EXPOSURE TO FURFURYL ALCOHOL

Asbestos-reinforced equipment manufacturing workers	Grinding-wheel makers
Carbon and graphite binders production workers	Laboratory workers using furfuryl alcohol
Cellulose ester production workers	Oil-drilling workers
Chemical distributors of furfuryl alcohol	Paint-remover makers
Coumarone resin production workers	Paper and pulp industry workers
Foundry core binders manufacturing workers	Phenol-furfuryl alcohol production workers
Furan resin manufacturing workers	Thermoplastic cement, mortar, and grout manufacturing workers
Furfuryl alcohol manufacturing workers	Thermosetting resin workers
Furfuryl alcohol-formaldehyde workers	Tetrahydrofurfuryl alcohol production workers
Furfuryl alcohol-formaldehyde-urea production workers	Workers using furfuryl alcohol as a solvent to remove automotive acrylic lacquer

Adapted from references 68,70-72

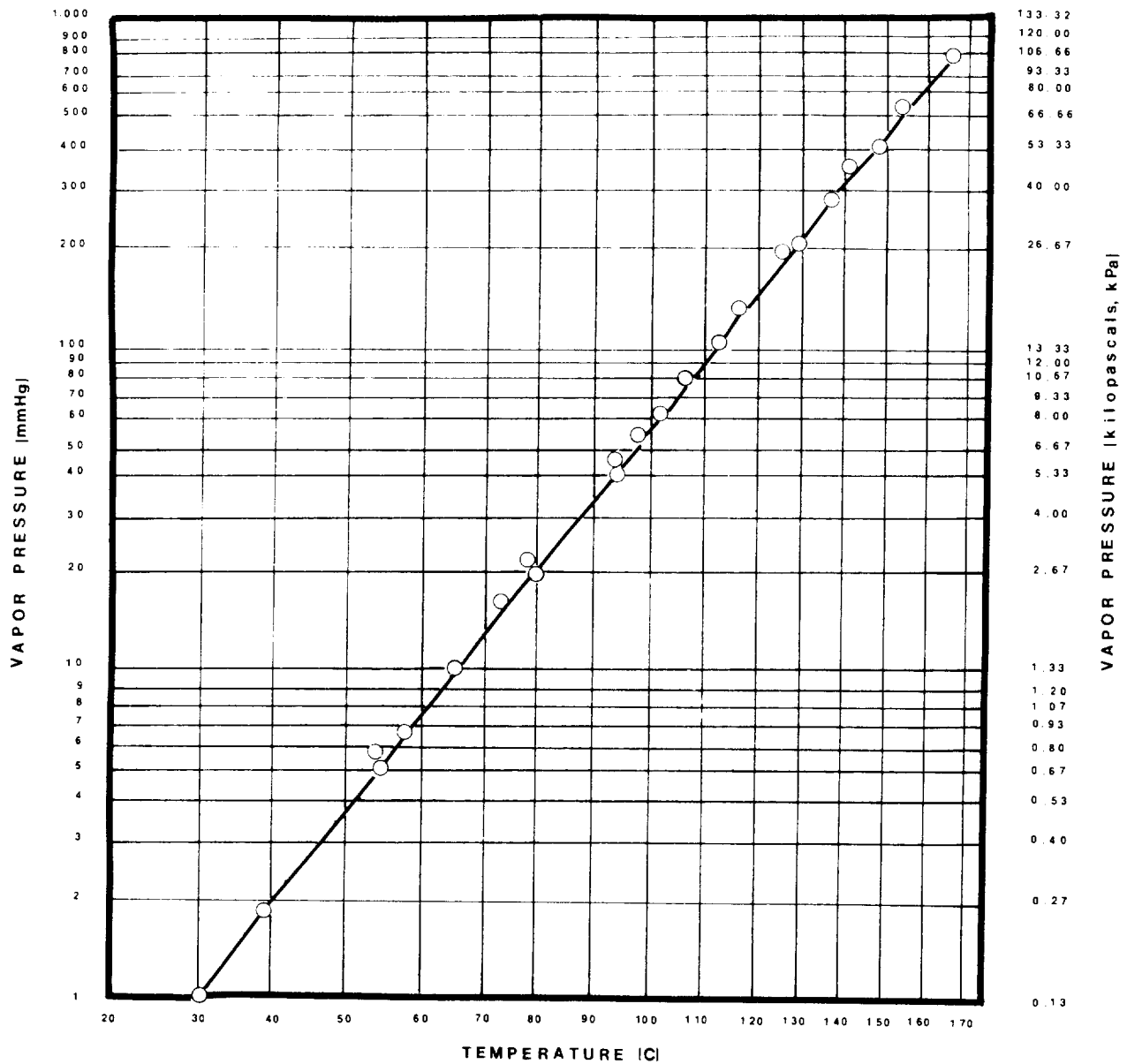


FIGURE IX-1
 VAPOR PRESSURE OF FURFURYL ALCOHOL

Adapted from reference 2

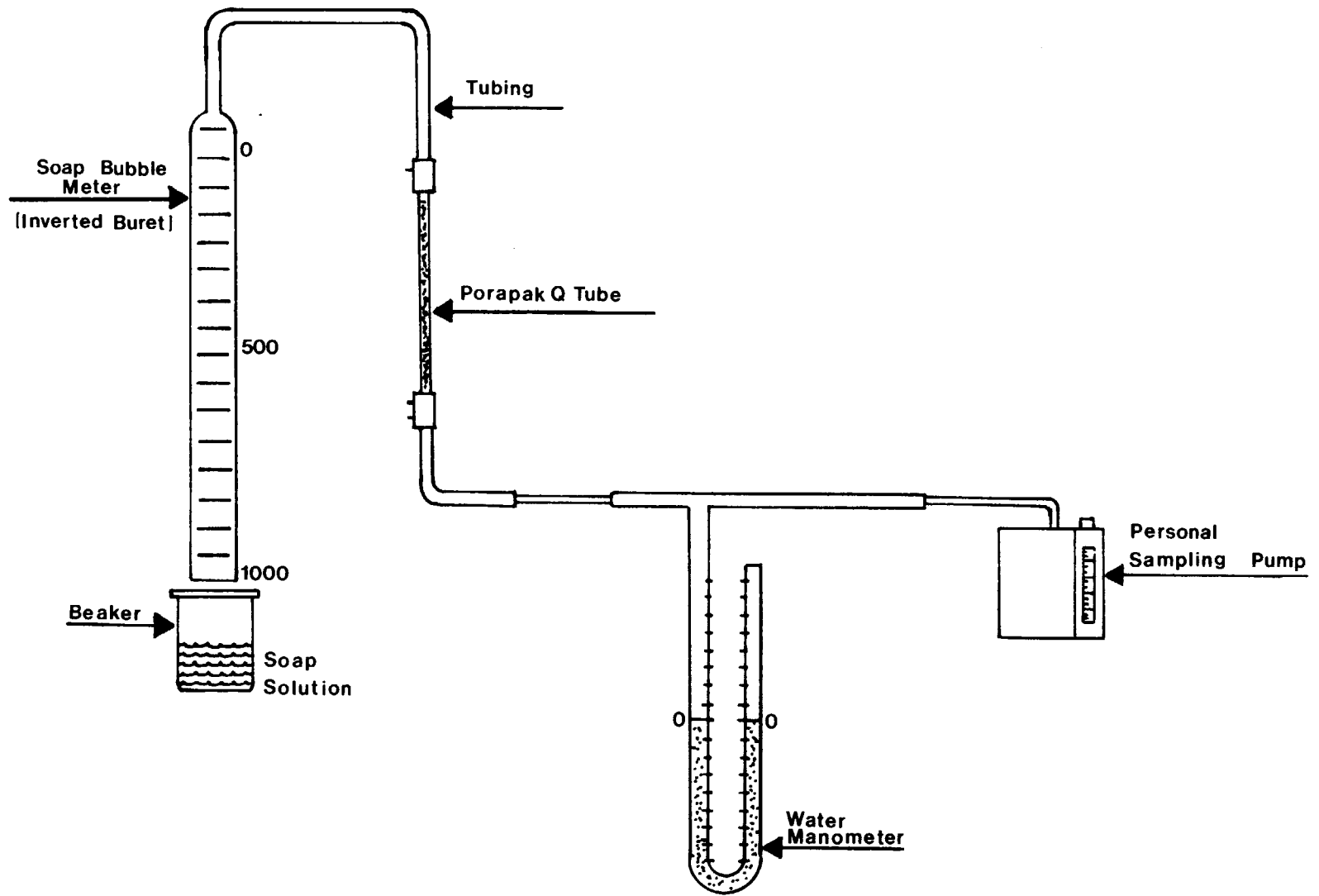


FIGURE IX-2

CALIBRATION SETUP FOR PERSONAL SAMPLING PUMP WITH PORAPAK Q TUBE

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