Designation: D 3686 – 95 (Reapproved 2001)$^{\epsilon 1}$

Standard Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)[1]

This standard is issued under the fixed designation D 3686; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

ϵ^1 NOTE—SI statement was added and part of Footnote F of Table A1.1 was deleted editorially in October 2001.

1. Scope

1.1 This practice covers a method for the sampling of atmospheres for determining the presence of certain organic vapors by means of adsorption on activated charcoal using a charcoal tube and a small portable sampling pump worn by a worker. A list of some of the organic chemical vapors that can be sampled by this practice is provided in Annex A1. This list is presented as a guide and should not be considered as absolute or complete.

1.2 This practice does not cover any method of sampling that requires special impregnation of activated charcoal or other adsorption media.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* A specific safety precaution is given in 9.4.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[2]

D 3687 Practice for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption Method[2]

2.2 *NIOSH Standard:*

CDC-99-74-45 Documentation of NIOSH Validation Tests[3]

HSM-99-71-31 Personnel Sampler Pump for Charcoal Tubes; Final Report[3]

2.3 *OSHA Standard:*

CFR 1910 General Industrial OSHA Safety and Health Standard[4]

3. Terminology

3.1 For definitions of terms used in this method, refer to Terminology D 1356.

3.2 Activated charcoal refers to properly conditioned coconut-shell charcoal.

4. Summary of Practice

4.1 Air samples are collected for organic vapor analysis by aspirating air at a known rate through sampling tubes containing activated charcoal, which adsorbs the vapors.

4.2 Instructions are given to enable the laboratory personnel to assemble charcoal tubes suitable for sampling purposes.

4.3 Instructions are given for calibration of the low flow-rate sampling pumps required in this practice.

4.4 Information on the correct use of sampling devices is presented.

4.5 Practice D 3687 describes a practice for the analysis of these samples.

5. Significance and Use

5.1 Promulgations by the Federal Occupational Safety and Health Administration (OSHA) in 29 CFR 1910.1000 designate that certain organic compounds must not be present in workplace atmospheres at concentrations above specific values.

5.2 This practice, when used in conjunction with Practice D 3687, will provide the needed accuracy and precision in the determination of airborne time-weighted average concentrations of many of the organic chemicals given in 29 CFR 1910.1000, CDC-99-74-45 and HSM-99-71-31.

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[1] This practice is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.04 on Workplace Atmospheres.

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[2] *Annual Book of ASTM Standards*, Vol 11.03.

[3] Available from the U.S. Department of Commerce, National Technical Information Service, Port Royal Road, Springfield, VA 22161.

[4] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

5.3 A partial list of chemicals for which this method is applicable is given in Annex A1, along with their OSHA permissible exposure limits.

6. Interferences

6.1 Water mist and vapor can interfere with the collection of organic compound vapors. Humidity greater than 60 % can reduce the adsorptive capacity of activated charcoal to 50 % for some chemicals **(1)**.[5] Presence of condensed water droplets in the sample tube will indicate a suspect sample.

7. Apparatus

7.1 *Charcoal Tube*:

7.1.1 A sampling tube consists of a length of glass tubing containing two sections of activated charcoal which are held in place by nonadsorbent material and sealed at each end.

7.1.1.1 Sampling tubes are commercially available. The tubes range in size from 100/50 to 800/400 mg, which means the tubes are divided into two sections with the front section containing 100 to 800 mg of activated charcoal and the back section containing 50 to 400 mg of activated charcoal. The 100/50-mg tube (**(2-4)** and Fig. 1) which is the one most frequently used, consists of a glass tube, 70-mm long, 6-mm outside diameter, 4-mm inside diameter, and contains two sections of 20/40 mesh-activated charcoal but separated by a 2-mm section of urethane foam. The front section of 100 mg is retained by a plug of glass wool, and the back section of 50 mg is retained by either a second 2-mm portion of urethane foam or a plug of glass wool. Both ends of the tube are flame-sealed.

NOTE 1—Urethane foam is known to adsorb certain pesticides **(5)**, for which this practice is contraindicated.

7.1.1.2 When it is desirable to sample highly volatile compounds for extended periods, or at a high volume flow rate, a larger device capable of efficient collection can be used, provided the proportions of the tube and its charcoal contents are scaled similarly to the base dimensions, to provide nominally the same linear flow rate and contact time with the charcoal bed.

7.1.2 The back portion of the sampler tube, which may contain between 25 and 100 % of the mass of activated charcoal present in the front section, adsorbs vapors that penetrate the front section and serves as a warning that breakthrough may have occurred. (Annex A1 gives recommended maximum tube loading information for many chemicals.)

7.1.2.1 Should analysis of the back portion show it to contain more than 10 % of the amount found in the front section, the possibility exists that solvent vapor penetrated both sections of charcoal, and the sample must be considered suspect. These percentages apply to 100/50-mg tubes. For other size tubes having disproportionate amounts of charcoal in the front and back sections, the percentages used to indicate potential breakthrough must be adjusted to take into account different ratios of charcoal. If results from the analysis of suspect samples are used to calculate vapor concentrations, the results must be reported as equal to or greater than the calculated concentrations. In such cases, the test must be repeated for confirmation of vapor concentration.

NOTE 2—Reportings from suspect samples would have significance when health standards are clearly exceeded and the amount by which they are exceeded is academic. (See 9.5.)

7.1.3 The adsorptive capacity and desorption efficiency of different batches of activated charcoal may vary. Commercial tubes, if used, should be purchased from the same batch and in sufficient number to provide sampling capacity for a definite period of time. *Care must be taken to have enough tubes from the same batch for a given study.*

7.1.3.1 The desorption efficiency and contamination level of a batch of tubes should be determined, following the procedure outlined in Practice D 3687 for activated charcoal. A random selection of at least five charcoal tubes from a specified lot should be taken for these checks.

7.1.4 Pressure drop across the sampling tube should be less than 25 mm Hg (3.3 kPa) at a flow rate of 1000 mL/min and less than 4.6 mm Hg (0.61 kPa) at a flow rate of 200 mL/min.

7.1.5 Charcoal sampling tubes prepared in accordance with this practice and with sealed glass ends may be stored indefinitely.

7.2 *Sampling Pumps*:

7.2.1 Any pump whose flow rate can be accurately determined and be set at the desired sampling rate is suitable. Primarily though, this practice is intended for use with small personal sampling pumps.

7.2.2 Pumps having stable low flow rates (10 to 200 mL/min) are preferable for long period sampling (up to 8 h) or

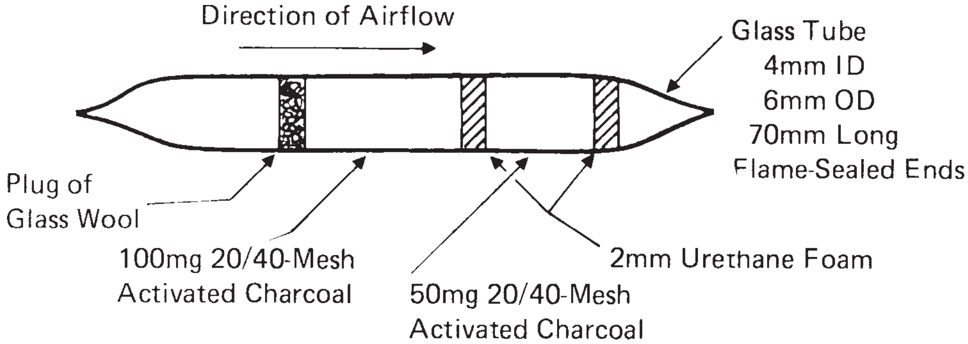

FIG. 1 Activated Charcoal Adsorption Sampling Tube

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[5] The boldface numbers in parentheses refer to the list of references at the end of this standard.

when the concentration of organic vapors is expected to be high. Reduced sample volumes will prevent exceeding the adsorptive capacity of the charcoal tubes. (Suggested flow rates and sampling times are given in Annex A1 for anticipated concentration ranges. Sample volumes are also discussed in 9.5.)

7.2.3 Pumps are available that will provide stable flow rates between ± 5 %. Pumps should be calibrated before and after sampling. If possible, flow rates should be checked during the course of the sampling procedure.

7.2.4 All sampling pumps must be carefully calibrated with the charcoal tube device in the proper sampling position. (See Annex A2 for calibration procedure.)

8. Reagents

8.1 *Activated Coconut-Shell Charcoal*—Prior to being used to make sampling devices the charcoal should be heated in an inert gas to 600°C and held there for 1 h. Commercially available coconut charcoal (20/40 mesh) has been found to have adequate adsorption capacity. Other charcoals can be used for special applications.

9. Sampling with Activated Charcoal Samplers

9.1 *Calibration of the Sampling System*—Calibrate the sampling system, including pump, flow regulator, tubing to be used, and a representative charcoal tube (or an equivalent induced resistance) with a primary or calibrated secondary flow-rate standard to ± 5%.

9.1.1 A primary standard practice is given for the calibration of low flow-rate pumps in Annex A2 and Fig. A2.1.

9.2 Break open both ends of the charcoal tube to be used for sampling, ensuring that each opening is at least one half the inside diameter of the tube.

9.3 Insert the charcoal tube into the sampling line, placing the back-up section nearest to the pump. At no time should there be any tubing ahead of the sampling tubes.

9.4 For a breathing zone sample, fasten the sampling pump to the worker, and attach the sampling tube as close to the worker's breathing zone as possible. Position the tube in a vertical position to avoid channeling of air through the adsorber sections.

NOTE 3—**Warning:** Assure that the presence of the sampling equipment is not a safety hazard to the worker.

9.4.1 Turn on the pump and adjust the flow rate to the recommended sampling rate.

9.4.2 Record the flow rate and starting time or, depending on the make of pump used, the register reading.

9.5 *Sampling Volumes*—The minimum sample volume will be governed by the detection limit of the analytical method, and the maximum sample volume will be determined by either the adsorptive capacity of the charcoal or limitations of the pump battery.

9.5.1 One method of calculating required sample volumes is to determine first the concentration range, over which it is important to report an exact number, for example from 0.2 to 2 times the permissible exposure concentration, and then calculate the sample volumes as follows:

$$\text{Minimum sample volume, m}^3 = \frac{\text{minimum detection limit, mg}}{0.2 \times \text{permissible exposure limit, mg/m}^3} \quad (1)$$

$$\text{Maximum sample volume, m}^3 = \frac{\text{tube capacity for vapors, mg}}{2 \times \text{permissible exposure limit, mg/m}^3} \quad (2)$$

9.5.2 Select a sampling rate that, in the sampling time desired, will result in a sample volume between the minimum and maximum calculated in 9.5.1.

9.5.2.1 Generally a long sampling time at a low flow rate is preferable to short-term high-volume sampling. This is consistent with the fact that most health standards are based on 8-h/day time-weighted averages of exposure concentrations.

9.5.2.2 A sample flow rate of less than 10 mL/min, however, should not be used. Calculations based upon diffusion coefficients for several representative compounds indicate that sampling at less than 10 mL/min may not give accurate results.[6]

9.5.2.3 Approximate sample volumes and sample times are given in Annex A1.

9.5.3 When spot checks are being made of an environment, a sample volume of 10 L is adequate for determining vapor concentrations in accordance with exposure guidelines.

9.6 At the end of the sampling period recheck the flow rate, turn off the pump, and record all pertinent information: time, register reading, and if pertinent, temperature, barometric pressure, and relative humidity.

9.6.1 Seal the charcoal tube with the plastic caps provided.

9.6.2 Label the tube with the appropriate information to identify it.

9.7 At least one charcoal sampling tube should be presented for analysis as a field blank with every 10 or 15 samples, or for each specific inspection or field study.

9.7.1 Break the sealed ends off the tube and cap it with the plastic caps. Do not draw air through the tube, but in all other ways treat it as an air sample.

9.7.2 The purpose of the field blank is to assure that if the sampling tubes adsorb vapors extraneous to the sampling atmosphere, the presence of the contaminant will be detected.

9.7.3 Results from the field blanks shall not be used to correct sample results. If a field blank shows contamination, the samples taken during the test must be assumed to be contaminated.

9.8 *Calculation of Sample Volume*:

9.8.1 For sample pumps with flow-rate meters:

$$\text{Sample volume, mL} = f \times t \left(\sqrt{\frac{P_1}{P_2} \times \frac{T_2}{T_1}} \right) \quad (3)$$

where:

f = flow rate sampled, mL/min,
t = sample time, min,
P_1 = pressure during calibration of sampling pump (mm Hg or kPa)
P_2 = pressure of air sampled (mm Hg or kPa)

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[6] Heitbrink, W. A., "Diffusion Effects Under Low Flow Conditions," American Industrial Hygiene Association Journal, Vol 44, No. 6, 1983, pp. 453–462.

T_1 = temperature during calibration of sampling pump (K), and
T_2 = temperature of air sampled (K).

9.8.2 For sample pumps with counters:

$$V = \frac{(R_2 - R_1) \times V}{I} \times \frac{P_1}{760} \times \frac{298}{T_1 + 273} \quad (4)$$

where:
R_2 = final counter reading,
R_1 = beginning counter reading,
V = volume, (1) mL-count (1)
P_1 = barometric pressure, mm Hg,
T_1 = temperature, °C, and
V = total sample volume, mL.

10. Handling and Shipping of Samples Collected on Charcoal Sampling Tubes

10.1 There is a paucity of information on the possible fate of the many different chemical species that can be collected in activated charcoal and the variety of conditions to which these samples may be exposed. Good practice suggests the following:[7]

10.1.1 Samples should be capped securely and identified clearly.

10.1.2 Samples collected in charcoal tubes should not be kept in warm places or exposed to direct sunlight.

10.1.3 Samples of highly vaporous or low-boiling materials, such as vinyl chloride, should be stored and transported in dry ice.

10.1.4 At present there are no published test data on the effect of conditions in aircraft cargo holds on capped samples. The preferred procedure is to carry the samples on board.

10.1.5 Samples should be shipped as soon as possible, stored under refrigeration until they are analyzed, and analyzed if possible within five working days.

10.1.6 Migration or equilibration of the sampled material within the sampling tube during prolonged or adverse storage or handling could be interpreted as break-through. This can be prevented by separating the front and back sections immediately after sampling, by having each section in a separate tube and capping them separately.

10.1.7 In some situations, circumstances and facilities may permit making up calibration standards at the facility where the study is being made and submitting these standards as quality control checks. (See Practice D 3687 for recommended procedure for making up standards.)

10.1.8 Bulk solvent samples should never be shipped or stored with the collected air samples.

11. Keywords

11.1 activated charcoal tube; air monitoring; charcoal tube; organic vapors; sampling and analysis; workplace atmospheres

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[7] Two studies that present information pertinent to this section are:
Saalwaechter, A. T., et al, "Performance Testing of the NIOSH Charcoal Tube Technique for the Determination of Air Concentrations of Organic Vapors," *American Industrial Hygiene Association Journal,* Vol 38, No. 9, September 1977, pp. 476–486.
Hill, R. H., Jr., et al, "Gas Chromatographic Determination of Vinyl Chloride in Air Samples Collected on Charcoal," *Analytical Chemistry,* Vol 48, No. 9, August 1976, pp. 1395–1398.

ANNEXES

(Mandatory Information)

A1. INFORMATION OF SOME ORGANIC COMPOUND VAPORS THAT CAN BE COLLECTED ON COCONUT-SHELL CHARCOAL (100/50 mg tubes)

TABLE A1.1 INFORMATION OF SOME ORGANIC COMPOUND VAPORS THAT CAN BE COLLECTED ON COCONUT-SHELL CHARCOAL (100/50 mg tubes)

Substance PEL ppm-mg/m^{3A}	Recommended Sampling Rate, mL/min to Detect Approximately 15 to 200 % of PEL in Time Given^B			Recommended Maximum Tube Loading, mg^C	Approximate Desorption Efficiency %^{D}	Eluent	GC Column^E	$CV_T{}^F$
	2h	4h	8h					
Acetone, 1000–2400	10	^{G}	^{G}	9	86 ± 10	CS_2	3	0.082
Acetonitrile, 40–70	50	25	25	2.7				0.072
Allyl alcohol, 2–4.8	200	100	50	<0.4	89 ± 5	CS_2 + 5 % 2-propanol	2	0.11
n-Amyl acetate, 100–525	50	25	10	15	86 ± 5	CS_2	4	0.051
sec-Amyl acetate, 125–650	50	25	10	15.5	91 ± 10	CS_2	4	0.071
Isoamyl alcohol, 100–360	50	25	10	10		CS_2 + 5 % 2-propanol	2	0.077
Benzene, 10–31.3	100	100	50		96	CS_2	1	0.060
Benzyl chloride, 1–5	—	200	200	<0.4	90 ± 5	CS_2	2	0.096
Butadiene, 1000–2200	10	^{G}	^{G}	4		CS_2	1	0.058

TABLE A1.1 *Continued*

Substance PEL ppm-mg/m^{3A}	Recommended Sampling Rate, mL/min to Detect Approximately 15 to 200 % of PEL in Time Given^B			Recommended Maximum Tube Loading, mg^C	Approximate Desorption Efficiency %^{D}	Eluent	GC Column^E	CV_T^F
	2h	4h	8h					
2-Butoxy ethanol, 50–240	100	50	25		99 ± 5	methylene chloride + 5 % methanol	2	0.060
n-Butyl acetate, 150–710	50	25	10	15	95	CS_2	4	0.069
sec-Butyl acetate, 200–950	50	25	10	15	91 ± 5	CS_2	4	0.054
tert-Butyl acetate, 200–950	50	25	10	12.5	94 ± 5	CS_2	4	0.091
Butyl alcohol, 100–300	100	50	25	10.5	88 ± 5	CS_2 + 1 % 2-propanol	2	0.065
sec-Butyl alcohol, 150–450	50	25	10	6	93 ± 5	CS_2 + 1 % 2-propanol	2	0.066
tert-Butyl alcohol, 100–300	50	25	10	5	90 ± 5	CS_2 + 1 % 2-propanol	2	0.075
Butyl glycidyl ether, 50–270	100	50	25	11.5	86 ± 10	CS_2		0.074
p-tert-Butyl toluene, 10–60	100	50	25	2.5	100+	CS_2	2	0.067
Camphor, 0.32–2	200	100	50	13.4	98 ± 5	CS_2 + 1 % methanol	2	0.074
Carbon disulfide, 20–60	200	100	50		95	benzene	8	0.059
Carbon tetrachloride, 10–65	200	100	50	7.5	97 ± 5	CS_2	1	0.092
Chlorobenzene, 75–350	50	25	10	15.5	90 ± 5	CS_2	2	0.056
Chlorobromomethane 200–1050	25	10	^{G}	9.3	94 ± 5	CS_2	2	0.061
Chloroform, 50–240	100	50	25	11	96 ± 5	CS_2	1	0.057
Cumene, 50–245	50	25	10	11	100+	CS_2	2	0.059
Cyclohexane, 300–1050	25	10	^{G}	6.3	100+	CS_2	3	0.066
Cyclohexanol, 50–200	100	50	25	10	99 ± 5	CS_2 + 5 % 2-propanol	2	0.080
Cyclohexanone, 50–200	100	50	25	13	78 ± 5	CS_2	2	0.062
Cyclohexene, 300–1015	25	10	^{G}		100+	CS_2	3	0.073
Diacetone alcohol, 50–240	100	50	25	12	77 ± 10	CS_2 + 5 % 2-propanol	2	0.101
o-Dichlorobenzene 50–300	50	25	10	15	85 ± 5	CS_2	6	0.067
1,1-Dichloroethane, 100–405	50	25	10	7.5	100+	CS_2	2	0.057
1,2-Dichloroethylene, 200–790	25	10	^{G}	5.1	100+	CS_2	2	0.052
p-Dioxane, 100–360	100	50	25	13	91 ± 5	CS_2	1	0.054
Dipropylene glycol methyl ether, 100–600	25	10	^{G}		75 ± 15	CS_2	2	0.064
2-Ethoxyethyl acetate, 100–500	50	25	10	19	74 ± 10	CS_2	4	0.062
Ethyl acetate, 400–1400	25	10	^{G}	12.5	89 ± 5	CS_2	4	0.058
Ethyl acrylate, 25–100	200	100	50	<5	95 ± 5	CS_2	4	0.054
Ethyl alcohol, 1000–1885	^{G}	^{G}	^{G}	2.6	77 ± 10	CS_2 + 1 % 2-butanol	2	0.065
Ethyl benzene, 100–435	200	100	50	16	100+	CS_2	2	0.041
Ethyl bromide, 200–890	100	50	25	7.1	83 ± 5	isopropanol	2	0.054
Ethyl butyl ketone, 50–230	50	25	10	<5.5	93 ± 5	CS_2 + 1 % methanol	2	0.086
Ethyl ether 400–1210	10	^{G}	^{G}	7.5	98 ± 5	ethyl acetate	3	0.053
Ethyl formate, 100–300	50	25	10	4.8	80 ± 10	CS_2	1	0.074
Ethylene dibromide, 20–155	100	50	25	<10.7	93 ± 5	CS_2	2	0.077
Ethylene dichloride, 50–202.5	100	50	25	12	95 ± 5	CS_2	6	0.079
Glycidol, 50–150	100	50	25	22.5	90 ± 5	tetrahydrofuran	2	0.080
Heptane, 500–2000	10	^{G}	^{G}	12.5	96 ± 5	CS_2	6	0.056
Hexane, 500–1800	10	^{G}	^{G}	11	94 ± 5	CS_2	1	0.062
Isoamyl acetate, 100–525	50	25	10	16.5	90 ± 5	CS_2	4	0.056
Isoamyl alcohol, 100–360	50	25	10	10	99 ± 5	CS_2 + 5 % 2-propanol	2	0.065
Isobutyl acetate, 150–700	50	25	10	14	92 ± 5	CS_2	4	0.065
Isobutyl alcohol, 100–305	50	25	10	10.5	84 ± 10	CS_2 + 1 % 2-propanol	2	0.073
Isopropyl acetate, 250–950	25	10	^{G}	13	85 ± 5	CS_2	4	0.067
Isopropyl alcohol 400–985	25	10	^{G}	5.6	94 ± 5	CS_2 + 1 % 2-butanol	2	0.064
Isopropyl glycidyl ether, 50–240	100	50	25	10.5	80 ± 10	CS_2	2	0.067
Mesityl oxide, 25–100	100	50	25	4.8	79 ± 5	CS_2 + 1 % methanol	2	0.071
Methyl acetate, 200–610	25	10	^{G}	7	88 ± 5	CS_2	1	0.055
Methyl acrylate, 10–35	200	100	50	<1.5	80 ± 10	CS_2	4	0.066
Methylal, 1000–3110	10	^{G}	^{G}	11.5	78 ± 10	hexane	3	0.06
Methyl amyl ketone, 100–465	50	25	10	7.5	80 ± 10	CS_2 + 1 % methanol	2	0.061
Methyl butyl ketone, 100–410	50	25	10	2.0	79 ± 10	CS_2	2	0.053
Methyl cellosolve, 25–80	100	50	25	10	97 ± 5	methylene chloride + 5 % methanol	2	0.068
Methyl cellosolve acetate, 25–120	100	50	25	5	76 ± 10	CS_2	4	0.068
Methyl chloroform, 350–1900	25	10	^{G}	18	98+	CS_2	6	0.054
Methyl cyclohexane, 500–2000	10	^{G}	^{G}		95 ± 5	CS_2	1	0.052
Methyl ethyl ketone, 200–590	50	25	10	9.5	89 ± 10	CS_2	2	0.072
Methyl isobutyl carbinol, 25–105	200	100	50	5.7	99 ± 5	CS_2 + 5 % 2-propanol	2	0.080
a-Methyl styrene, 100–480	100	50	25	21	91 ± 5	CS_2	2	0.054
Methylene chloride, 500–1740	10	^{G}	^{G}	9.3	95 ± 5	CS_2	1	0.073
Naphtha (coal tar), 100–400	100	50	25	14.8	88 ± 5	CS_2	7	0.051
n-octane, 500–2350	10	^{G}	^{G}	15	93 ± 5	CS_2	1	0.060
Pentane, 1000–2950	10	^{G}	^{G}	9	96 ± 5	CS_2	1	0.055
2-Pentanone, 200–700	25	10	^{G}		88 ± 5	CS_2	2	0.063

TABLE A1.1 *Continued*

Substance PEL ppm-mg/m^3[A]	Recommended Sampling Rate, mL/min to Detect Approximately 15 to 200 % of PEL in Time Given[B]			Recommended Maximum Tube Loading, mg[C]	Approximate Desorption Efficiency %[D]	Eluent	GC Column[E]	CV_T[F]
	2h	4h	8h					
Perchloroethylene, 100–680	50	25	10	29	95 ± 5	CS_2	6	0.052
Petroleum distillates, 500–2000	10	[G]	[G]	12.3	96 ± 5	OS_2	6	0.052
Phenyl ether vapor, 1–7		200	200	0.6	90 ± 5	CS_2	2	0.070
Phenyl glycidyl ether, 10–59	100	50	25	12.5	97 ± 5	CS_2	2	0.057
n-Propyl acetate, 200–840	50	25	10	14.5	93 ± 5	CS_2	4	0.056
n-Propyl alcohol, 200–490	50	25	10	9	87 ± 5	CS_2 + 1 % 2-propanol	2	0.075
Propylene dichloride, 75–350	50	25	10	5	97 ± 5	CS_2	2	0.056
Propylene oxide, 100–240	25	10	[G]	2	90 ± 5	CS_2	3	0.085
Pyridine, 5–15	200	100	50	<7.3	70 ± 10	CS_2		0.059
Stoddard solvent, 500–2950	10	[G]	[G]	13	96 ± 5	CS_2	7	0.052
Styrene (monomer), 100–425	100	50	25	18	87 ± 5	CS_2	2	0.057
1,1,1,2-Tetrachloro-2,2-difluoroethane, 500–4170	10	[G]	[G]	19.5	100+	CS_2	2	0.069
1,1,1,2-Tetrachloro-1,2-difluoroethane, 500–4170	10	[G]	[G]	26	96 ± 5	CS_2	2	0.054
Tetrahydrofuran, 200–590	25	10	[G]	7.5	92 ± 5	CS_2	3	0.055
1,1,2-Trichloroethane, 10–55	100	50	25	5	96 ± 5	CS_2	6	0.057
Trichloroethylene, 100–535	100	50	25	21	96 ± 5	CS_2	6	0.082
1,1,2-Trichloro-1,2,2-trifluoroethane, 1000–7660	10	[G]	[G]	20	100+	CS_2	5	0.07
Turpentine, 100–560	50	25	10	13	96 ± 5	CS_2	7	0.055
Vinyl toluene, 100–480	100	50	25	17	85 ± 10	CS_2	2	0.058

[A] *Substances*—The list does not contain all compounds for which the method is applicable. It lists only those for which reliable data could be obtained. PEL-Federal Permissible Exposure Limits, as given in the *Federal Register,* June 1974, and updated May 1976. These values, which may be either ceiling limits or 8-h/day average exposure limits, depending on the compound, are presented to give guidance in selecting sampling rates and times. These values are subject to change by the Federal Occupational Safety and Health Administration.

[B] *Recommended Sampling Rate*—The suggested sampling rates for the different sampling periods are sufficient to provide a tube loading of at least 0.01 mg when concentrations are 15 % of the PEL., but will not exceed the recommended tube loading when atmosphere are 200 % of the PEL. These figures are based on the 100-mg coconut-shell charcoal tubes described in this practice.

[C] *Recommended Maximum Tube Loading*—These values are conservative, to allow for high humidity or the presence of other substances which reduce the normal tube capacity.

[D] *Approximate Desorption Efficiency*—These figures are given only as guides for carrying out system calibrations. Actual desorption efficiencies should always be determined at the time of analysis, and any significant deviation should be regarded as a possible indication of a systematic error in the analytical technique. The figure given for desorption efficiency is an average figure. The desorption efficiency for a compound will vary with the amount; in most cases, the desorption efficiency will be lower for reduced tube loadings.

[E] *Gas Chromatographic Columns*—key:

1—20-ft × 1/8 in: ss packed with 10 % FFAP on Chromosorb W AW
2—10-ft × 1/8 in: ss packed with 10 % FFAP on Chromosorb W AW
3—4-ft × 1/4 in: ss packed with 60/80 Porapak Q
4—10-ft × 1/8 in: ss packed with 5 % FFAP on Supelcoport
5—6-ft × 1/4 in: ss packed with 60/80 Porapak Q
6—10-ft × 1/8 in: ss packed with 10 % OV-101 on Supelcoport
7—6-ft × 1/8 in: ss packed with 1.5 % OV-101 on Chromosorb W AW
8—6-ft × 1/4 in: Glass column packed with 5 % OV-17 on Supelcoport

[F] CV_T—Coefficient of variation (that is, relative standard deviation) of the total (net) error in the method (including variability of the pump).

$$CV_T = (CV^2_{A+\bar{D}\bar{E}} + CV_S^{\ 2} + CV_P^{\ 2})^{1/2}$$

where:

CV_{A+DE} = coefficient of variation of a single future assay including error in the desorption efficiency factor $\bar{D}\bar{E}$
CV_S = coefficient of variation due to sampling errors (not including variable of the pump) along with variability in true desorption efficiency from tube-to-tube, and
CV_P = coefficient of variation due to pump (CV_P = 0.05 assumed).

Acknowledgements: The information in this table comes from NIOSH Standards Completion Program.[8] We gratefully acknowledge NIOSH's contribution to this table, by making available previously unpublished CV_T data.

[G] Sample rates of less than 10 mL/min are not recommended. Shorter sampling periods are required.

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[8] Taylor, D. G., Kupel, R. E., and Bryant, J. M., "Documentation of NIOSH Validation Tests," DHEW (NIOSH), Pub. No. 77-185. Available from National Technical Information Service, Springfield, VA 22161 (PB274-248).

A2. METHOD FOR CALIBRATION OF SMALL VOLUME AIR PUMPS

A2.1 Using a buret that approximately represents a 1-min sampling volume, assemble the apparatus as shown in Fig. A2.1 using any good soap bubble solution as a source of the film. Make sure all connections are tight.

A2.1.1 It is advisable to check the volume of burets used for calibrating sampling pumps by weighing the volume of water contained in the buret and calculating the true volume.

A2.1.2 Make sure the batteries of the pump are charged.

A2.2 Prime the surface of the cylinder with bubble solution by drawing repeated films up the tube until a single film travels to the desired mark.

A2.3 With a stop watch, time the travel of a single film from an initial zero mark to a selected volume mark. Note the time and repeat this procedure at least three times.

A2.4 Calculate the sampling rate of the pump, correcting the air volume to 25°C and 760 mm Hg (101.3 kPa), using the ambient barometric pressure.

A2.5 Replace the charcoal tube sampler with another one selected at random, and repeat the calibration sequence.

A2.5.1 Sampling tubes should consistently meet the pressure drop criterion given in 7.1.4.

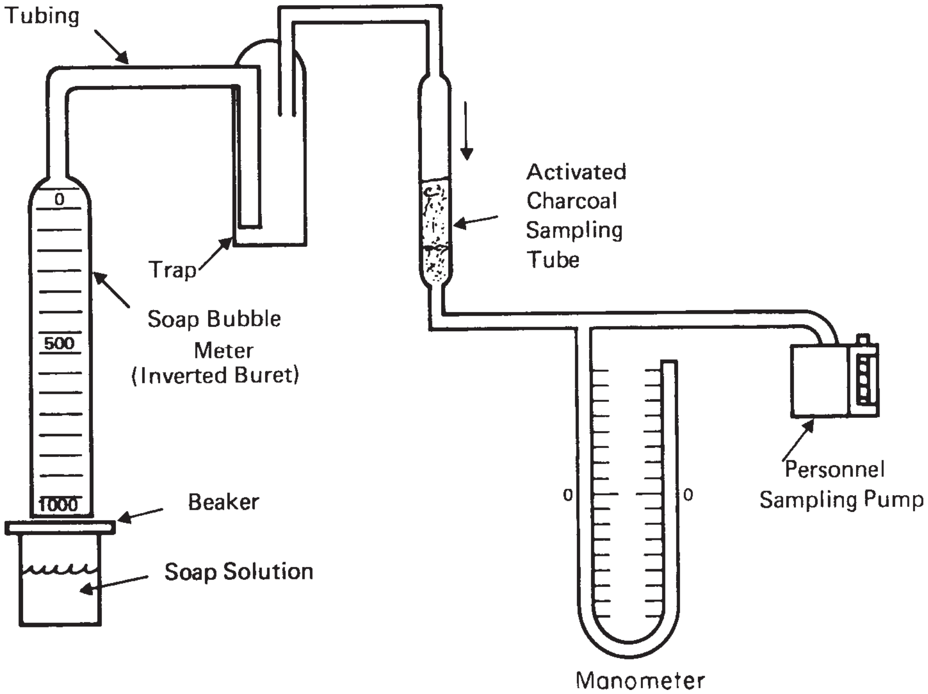

FIG. A2.1 Calibration Setup for Personnel Sampling Pump with Activated Charcoal Sampling Tube

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(**4**) Reid, F. H., and Halpin, W. R.," Determination of Halogenated and Aromatic Hydrocarbons in Air by Charcoal Tube and Gas Chromatography," *American Industrial Hygiene Association Journal*, July-August 1968, pp. 390–396.

(**5**) Turner, B. C., and Glotfelty, D. E., "Field Sampling of Pesticide Vapors with Polyurethane Foam," *Analytical Chemistry*, Vol 49, 1977, pp. 7–10.

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