Designation: ~~D3686 – 13~~ D3686 – 20

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

This standard is issued under the fixed designation D3686; 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.

1. Scope

1.1 This practice covers a method for the sampling of atmospheres to determine 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 an information 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. The values given in parentheses after SI units are provided for information only and are not considered 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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* A specific safety precaution is given in 9.4.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D3687 Test Method for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption Method

D4840 Guide for Sample Chain-of-Custody Procedures

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

2.2 *NIOSH Standards:*

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

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

NIOSH Manual of Analytical ~~Methods,~~Methods ~~Fourth Edition~~[4]

2.3 *OSHA Standards:*

29 CFR 1910 U.S. Code of Federal Regulations Relating to Labor, U.S. Occupational Safety and Health Administration, Department of Labor[5]

OSHA Chemical Sampling Information [6]

OSHA Sampling and Analytical Methods [7]

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from National Technical Information Service (NTIS), 5285 Port Royal Rd., Springfield, VA 22161, http://www.ntis.gov.

[4] NIOSH Manual of Analytical ~~Methods.~~Methods, http://www.cdc.gov/niosh/nmam.

[5] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

[6] OSHA Chemical Sampling Information, http://osha.gov/dts/chemicalsampling/toc/toc_chemsamp.html.

[7] OSHA Sampling and Analytical Methods, http://osha.gov/dts/sltc/methods/toc.html.

2.4 ~~*UK Health and Safety Executive:*~~*Other Documents:*

ISO/IEC 17025 ~~Methods for Determination of Hazardous Substances (MDHS)~~General Requirements for the Competence of Testing and Calibration Laboratories[8]

~~2.5 *Berufsgenossenschaftliches Institut für Arbeitsschulz (BGIA):*~~[9]

~~GESTIS Analytical Methods~~

3. Terminology

3.1 *Definitions*—For definitions of terms used in this method, refer to Terminology D1356.

~~3.2 Activated charcoal refers to properly conditioned charcoal.~~

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *activated charcoal, n*—charcoal that has been heated or otherwise treated to increase its adsorptive power.

[8] ~~Health Safety Executive. Methods for the Determination of Hazardous Substances (MDHS) guidance. http://www.hse.gov.uk/pubns/mdhs/index.htm#~~ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

3.2.1.1 *Discussion*—

A common procedure is to increase the inherent porosity of charcoal derived from biological precursors (for example, nut shells) by steam activation. Prior to use in this application, activated charcoals may need to be further treated to remove remnant organic compounds.

4. Summary of Practice

4.1 Air samples are collected for organic vapor analysis by aspirating air at a known rate and for an appropriate time through sampling tubes containing activated charcoal.

4.2 Instructions are given to enable assembly of charcoal tubes suitable for sampling purposes.

4.3 Information on the correct use of the charcoal tube sampling device is presented.

4.4 ~~Practice~~ Test Method D3687 describes a ~~practice~~method for the analysis of these samples.

5. Significance and Use

5.1 Promulgations by the U.S. 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~~ Test Method D3687, will provide the needed accuracy and precision for the determination of airborne time-weighted average concentrations of many of the organic chemicals ~~cited in~~ including but not limited to CDC-99-74-45, HSM-99-71-31, NIOSH Manual of Analytical Methods, 29 CFR 1910.1000, OSHA Chemical Sampling Information, OSHA Sampling and Analytical Methods, and HSE Methods for the Determination of Hazardous ~~Substances, and BGIA GETIS Analytical Methods.~~Substances.

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 %~~ 60 % can reduce the adsorptive capacity of activated charcoal up to ~~50 %~~ 50 % for some chemicals (**1**).[9] Presence of condensed water droplets in the sample tube will indicate a suspect sample. Water vapor co-collected by the sorbent can lead to poor recovery of polar and reactive chemicals, for example, acetone, although it should not affect most chemicals listed in Annex A1. Alternative sorbents are available for sampling polar and reactive compounds at high humidity. Users are advised to consult their analytical laboratory, method documentation, or associated literature for more information.

6.2 High levels of organic vapors can interfere by reducing the amount of time a workplace can be sampled before the charcoal sampler becomes saturated.

7. Apparatus

7.1 *Charcoal Tube:*

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

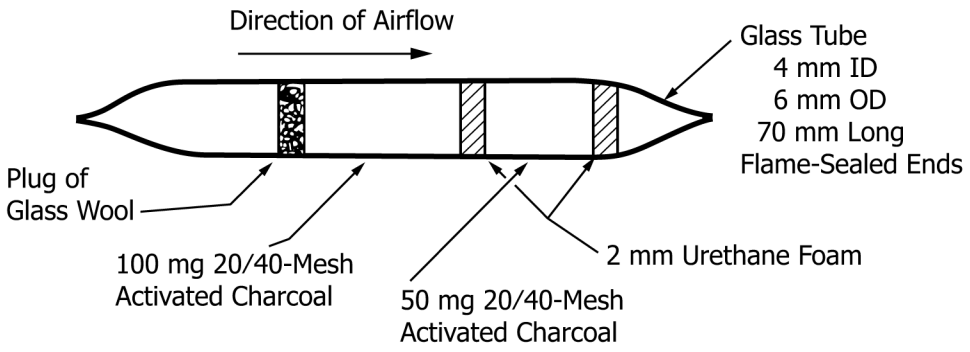

FIG. 1 Activated Charcoal Adsorption Sampling Tube

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

7.1.1.1 Sampling tubes are commercially available. The tubes are usually 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) is the one most frequently used, it consists of a glass tube that is 70-mm long, 6-mm outside diameter, 4-mm inside diameter, and contains two sections of 20/40 mesh-activated coconut-shell charcoal separated by a 2-mm section of urethane foam. The front section of 100 mg is retained by a plug of clean glass wool, and the back section of 50 mg is retained by either a second 2-mm portion of urethane foam or by a plug of clean glass wool. Both ends of the tube are usually flame-sealed.

NOTE 1—Urethane foam is known to adsorb certain pesticides (**5**). Contaminated urethane foam should not be used for this practice.

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 usually contains 50 % of the mass of activated charcoal present in the front section. The back 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.)

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

7.1.4 Pressure drop across the sampling tube should be less than ~~25 mm Hg (3.3 kPa)~~3.3 kPa (25 mm Hg) at a flow rate of 1000 mL/min and less than ~~4.6 mm Hg (0.61 kPa)~~0.61 kPa (4.6 mm Hg) 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 can be stored indefinitely. Accrediting bodies, such as American Industrial Hygiene Association, may require that sampling media have an expiration date. Accredited facilities must abide by this date.

7.2 *Sampling Pumps:*

7.2.1 Any pump with a flow rate that can be accurately determined, that can be set at the desired sampling rate, and that can maintain the desired sampling rate for a sufficient time 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 when the concentration of organic vapors is expected to be high. Reduced sample volumes will avoid 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 discussed in 9.5.)

7.2.3 Pumps are available that will provide stable flow rates between ±5 % of the desired flow rate. ~~Pumps~~ Flow rate through the charcoal tubes should be ~~calibrated~~measured before and after ~~sampling.~~sampling with an instrument calibrated for the purpose.

~~7.2.4 All sampling pumps must be carefully calibrated with the charcoal tube device in the proper sampling position.~~

7.2.4 A sampling tube holder with flexible tubing is used to connect the sampling tube to the sampling pump. The sampling tube holder is used to protect the worker from the sharp end of the samping tube.

7.3 *Flowmeter:*

7.3.1 Flowmeter, portable, with an accuracy that is sufficient to enable the volumetric flow rate to be measured to within ±5 %. The flowmeter calibration by a provider accredited to ISO/IEC 17025 for such calibrations shall be traceable to national or international standards. Retain the calibration certificate, including the pressure and temperature at which the calibration was performed, and identifying and performance documentation for the flowmeter.

8. Reagents

8.1 *Activated Charcoal*—Prior to being used to make sampling devices the charcoal should be heated in an inert gas at an appropriate temperature for a sufficient ~~time.~~ time to ensure background of organic compounds sufficiently low as to not interfere with the analysis of low concentrations of volatile organic compounds (VOC). Commercially available coconut-shell charcoal (20/40 mesh) has been found to have adequate adsorption capacity for many volatile chemicals. Other charcoals, such as petroleum-based charcoal and proprietary charcoals, can be used for appropriate applications.

9. Sampling with Activated Charcoal Samplers

9.1 ~~*Calibration*~~ *Setting the Flow-rate of the Sampling System*—~~Calibrate the sampling system, including~~ The sampling system consists of a pump, flow regulator, tubing to be used, and a representative charcoal tube (or an equivalent induced ~~resistance) with a primary flow-rate standard or with a calibrated secondary standard to within ±5 % of the desired~~resistance). The air flow through the sampling system should be adjusted and set at the desired rate by means of a flowmeter calibrated traceably to national or international standards (see D5337~~flow rate as described in Practice~~). The calibration of the flow ~~D5337. Calibrate the sampling pump~~ meter must be performed by an organization accredited for the purpose according to ISO 17205. The flow through the sampling system shall be measured in a clean location with similar temperature and barometric pressure as the sampling site. Normally, ~~calibrated~~ pump flow rates or sample air volumes are not corrected for temperature or barometric pressure where concentrations are calculated for normal temperature and pressure.

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 connective flexible tubing, placing the back-up section nearest to the pump. At no time should there be any tubing ahead of the sampling tubes. Use a sampling tube holder to protect the worker from the sharp end of the sampling tube.

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 charcoal sections. (**Warning—**Assure that the presence of the sampling equipment is not a safety hazard to the worker and that the equipment will not interfere with the worker's duties.)

9.4.1 Turn on the pump.

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

9.5 *Sampling Volumes*—The minimum sample volume will be ~~governed by~~based on the detection limit of the analytical method, and the maximum sample volume will be ~~determined by~~based on either the adsorptive capacity of the charcoal or limitations of the pump and 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. Often, two 4-h samples are preferable to a single 8-h sample so that if one is lost then the other can be used to partially document exposure. Work practices may change during the day and be better documented with two samples.

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.~~results **(6).**

9.5.2.3 Sampling information for a large number of organic chemicals is given in Annex A1, in the NIOSH Manual of Analytical Methods, OSHA Chemical Sampling Information file and methods, UK HSE MDHS database, and German BGIA GESTIS Analytical Methods database. Other appropriate sources of information and guidance, including Annex A1, can also be used.

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. Particularly volatile organic chemicals may require a lesser sample volume to prevent sampler saturation. Consult the above sources for sampling information before sampling.

9.5.4 Periodically check the sampling system to ensure the flow-rate remains within 5 % of the set flow. Some pumps trigger alarms or shut-down if this occurs, while others may not.

9.6 At the end of the sampling period, turn off the pump, and record all pertinent information: time, register reading, and if pertinent, sampling site 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.

9.6.3 Re-check the flow-rate through the sampling system a representative charcoal tube (or an equivalent induced resistance) using the calibrated measurement instrument. Record the flow-rate through the sampler as the average of the pre- and post-sampling flow-rate readings. If the flow-rate readings differ by more than 5 %, both measurements should be provided to the analytical laboratory for calculation of maximum and minimum bounds to the concentration. Alternatively, the sample can be discarded.

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 field blank tube and cap it with the plastic caps. Do not draw air through the blank 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 on the field blank.

9.8 *Calculation of Sample Volume:*

9.8.1

~~Sample volume, mL = *calibrated pump flow rate (mL/min)* × *sampling time (min)*~~ (3)

Sample volume, mL = pump flow rate (mL/min) × sampling time (min) (3)

Typically, ~~sampling pumps~~flow rate should be ~~calibrated~~measured at or near the sampling site. Sample air volumes should not be corrected for sampling site temperature or barometric pressure.

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

10.1 There is a lack of information on the stability of the many different chemical species that can be collected on activated charcoal and the variety of conditions to which these samples may be exposed. Good practice suggests the following:[10]

10.1.1 Use validated ~~NIOSH and OSHA methods (or other validated methods) whenever~~ methods (for example NIOSH, OSHA, IFA or HSE)) when possible.

10.1.2 Samples should be securely capped and clearly identified.

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

10.1.4 Samples of highly vaporous or low-boiling materials, such as vinyl chloride, should be stored and transported in dry ice or in another acceptable material. These samples should be shipped using an overnight delivery service.

10.1.5 At present there are no published test data on the effect of conditions in aircraft cargo holds on capped samples. However, it is generally advisable to avoid shipping in unpressurized aircraft cargo compartments.

10.1.6 Samples should be shipped to the analytical laboratory as soon as possible, ~~stored under refrigeration until they are analyzed, and analyzed if possible within five working days.~~under refrigeration where the method indicates a preference for storage under refrigeration, and analyzed within the maximum period provided in the method. If no storage requirements are indicated, store all tubes under refrigeration and analyze as soon after sampling as possible (preferably within 5 days).

10.1.7 Migration or equilibration of the sampled material within the sampling tube during prolonged or adverse storage or handling could be interpreted as break-through.

10.1.8 Bulk solvent samples should never be shipped or stored with air samples or with sampling media.

10.1.9 Follow sampling chain of custody procedures in accordance with Guide D4840 to ensure sample traceability. Ensure that the documentation that accompanies the samples is suitable for a “chain of custody” to be established.

11. Keywords

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

[10] Two studies that present information pertinent to this section ~~are:~~are
~~Saalwaechter, A. T., et al., “Performance Testing of the NIOSH Charcoal Tube Technique for the Determination of Air Concentrations of Organic Vapors,” Saalwaechter American (7Industrial Hygiene Association Journal,) Vol 38, No. 9, September 1977, pp. 476–486.~~and Hill
~~Hill, R. H., Jr., et al., “Gas Chromatographic Determination of Vinyl Chloride in Air Samples Collected on Charcoal,” Analytical (8Chemistry,). Vol 48, No. 9, August 1976, pp. 1395–1398.~~

ANNEX

(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 ficier
	2h	4h	8h		
Acetonitrile, 40–70	50	25	25	2.7	
Allyl alcohol, 2–5	200	100	50	<0.4	89 ± 5
n-Amyl acetate, 100–525	50	25	10	15	86 ± 5
sec-Amyl acetate, 125–650	50	25	10	15.5	91 ± 10
Isoamyl alcohol, 100–360	50	25	10	10	
Benzene, 10–31.3	100	100	50		96
Benzyl chloride, 1–5	—	200	200	<0.4	90 ± 5
2-Butoxy ethanol, 50–240	100	50	25		99 ± 5
n-Butyl acetate, 150–710	50	25	10	15	95
sec-Butyl acetate, 200–950	50	25	10	15	91 ± 5
tert-Butyl acetate, 200–950	50	25	10	12.5	94 ± 5
Butyl alcohol, 100–300	100	50	25	10.5	88 ± 5
sec-Butyl alcohol, 150–450	50	25	10	6	93 ± 5
tert-Butyl alcohol, 100–300	50	25	10	5	90 ± 5
Butyl glycidyl ether, 50–270	100	50	25	11.5	86 ± 10
p-tert-Butyl toluene, 10–60	100	50	25	2.5	100+
Camphor, 0.32–2	200	100	50	13.4	98 ± 5
Carbon disulfide, 20–60	200	100	50		95
Carbon tetrachloride, 10–55	200	100	50	7.5	97 ± 5
Chlorobenzene, 75–350	50	25	10	15.5	90 ± 5
Chlorobromomethane 200–1050	25	10	G	9.3	94 ± 5
Chloroform, 50–240	100	50	25	11	96 ± 5
Cumene, 50–245	50	25	10	11	100+
Cyclohexane, 300–1050	25	10	G	6.3	100+
Cyclohexanol, 50–200	100	50	25	10	99 ± 5
Cyclohexene, 300–1015	25	10	G		100+
Diacetone alcohol, 50–240	100	50	25	12	77 ± 10
o-Dichlorobenzene 50–300	50	25	10	15	85 ± 5
1,1-Dichloroethane, 100–400	50	25	10	7.5	100+
1,2-Dichloroethylene, 200–790	25	10	G	5.1	100+
p-Dioxane, 100–360	100	50	25	13	91 ± 5
Dipropylene glycol methyl ether, 100–600	25	10	G		99 ± 5
2-Ethoxyethyl acetate, 100–540	50	25	10	19	99 ± 5
Ethyl acetate, 400–1400	25	10	G	12.5	89 ± 5
Ethyl alcohol, 1000–1900	G	G	G	2.6	77 ± 10
Ethyl benzene, 100–435	200	100	50	16	100+
Ethyl bromide, 200–890	100	50	25	7.1	83 ± 5
Ethyl butyl ketone, 50–230	50	25	10	<5.5	93 ± 5
Ethyl ether 400–1200	10	G	G	7.5	98 ± 5
Ethyl formate, 100–300	50	25	10	4.8	80 ± 10
Ethylene dibromide, 20–155	100	50	25	<10.7	86 ± 5
Ethylene dichloride, 50–200	100	50	25	12	95 ± 5
Glycidol, 50–150	100	50	25	22.5	90 ± 5
Heptane, 500–2000	10	G	G	12.5	96 ± 5
Hexane, 500–1800	10	G	G	11	94 ± 5
Isoamyl acetate, 100–525	50	25	10	16.5	90 ± 5
Isoamyl alcohol, 100–360	50	25	10	10	99 ± 5
Isobutyl acetate, 150–700	50	25	10	14	92 ± 5
Isobutyl alcohol, 100–300	50	25	10	10.5	84 ± 10
Isopropyl acetate, 250–950	25	10	G	13	85 ± 5
Isopropyl alcohol 400–980	25	10	G	5.6	94 ± 5
Isopropyl glycidyl ether, 50–240	100	50	25	10.5	80 ± 10
Mesityl oxide, 25–100	100	50	25	4.8	79 ± 5
Methyl acetate, 200–610	25	10	G	7	88 ± 5