

Designation: D 4490 – 96 (Reapproved 2001)^{∈1}

Standard Practice for Measuring the Concentration of Toxic Gases or Vapors Using Detector Tubes¹

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 ϵ^1 Note—Footnote 5 was removed editorially in May 2001.

1. Scope

1.1 This practice covers the detection and measurement of concentrations of toxic gases or vapors using detector tubes (1, 2).² A list of some of the gases and vapors that can be detected by this practice, their 1994–95 TLV values recommended by the ACGIH, and their measurement ranges are provided in Annex A1. This list is given as a guide and should be considered neither absolute nor complete.

1.2 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.

2. Referenced Documents

2.1 ASTM Standards:

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres³

2.2 Other Document:

29 CFR 1910 Federal Occupational Safety and Health Standard Title 29^4

3. Terminology

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

4. Summary of Practice (3)

4.1 Detector tubes may be used for either short-term sampling (grab sampling; 1 to 10 min typically) or long term sampling (actively or passively; 1 to 8 h) of atmospheres containing toxic gases or vapors.

4.1.1 Short-Term Sampling (Grab Sampling) (4-18)—A given volume of air is pulled through the tube by a mechanical pump. If the substance for which the detector tube was designed is present, the indicator chemical in the tube will change color (stain). The concentration of the gas or vapor may be estimated by either (a) the length-of-stain compared to a calibration chart, or (b) the intensity of the color change compared to a set of standards.

4.1.2 Long-Term Active Sampling (Long-Term Tubes) (19-22)—A sample is pulled through the detector tube at a slow, constant flow rate by an electrical pump. The time-weighted average concentration of the gas or vapor is determined by correlating the time of sampling either with (a) the length-of-stain read directly from the calibration curve imprinted on the tube or (b) the intensity of the color change compared to a set of standards.

4.1.3 Long-Term Passive Sampling (Diffusion or Dosimeter Tubes) (25)—The contaminant molecules move into the tube according to Fick's First Law of Diffusion. The driving force is the concentration differential between the ambient air and the inside of the tube. The time-weighted average concentration of the gas or vapor is determined by dividing the indication on the tube by the number of hours sampled (1 to 10 h according to the manufacturers' instructions).

4.2 Instructions are given for the calibration of the sampling pumps required in this practice.

4.3 Information on the correct use of the detector tubes is presented.

5. Significance and Use

5.1 The Federal Occupational Safety and Health Administration, in 29 CFR 1910, designates that certain gases and vapors must not be present in workplace atmospheres at concentrations above specific values.

5.2 This practice will provide a means for the determination of airborne concentrations of certain gases and vapors given in 29 CFR 1910.

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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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 $^{^{2}}$ The boldface numbers in parentheses refer to the list of references at the end of this practice.

³ Annual Book of ASTM Standards, Vol 11.03.

 $^{^4}$ Code of Federal Regulations, Part 1910.1000 Subpart 2 and Part 1926.55 Subpart D.

5.3 A partial list of chemicals for which this practice is applicable is presented in Annex A1.

5.4 This practice also provides for the sampling of gaseous atmospheres to be used for process control or other purposes (2, 23-25).

6. Interferences (26, 27)

6.1 Some common interferences for the various tubes are listed in the instruction sheets provided by the manufacturers.

7. Apparatus (28-31)

7.1 *Detector Tube*—A detector tube consists of a glass tube containing an inert granular material that has been impregnated with a chemical system which reacts with the gas or vapor of interest. As a result of this reaction, the impregnated chemical changes color. The granular material is held in place within the glass tube by porous plugs of a suitable inert material. The ends of the glass tube are flame-sealed to protect the contents during storage.

7.2 Pump (32):

7.2.1 Short-Term Sampling—A mechanical, hand-operated, aspirating pump is used to draw the sample through the detector tube during the short-term sampling. Two types of pumps are commercially available: piston-operated and bellows-operated. The pumps have a capacity of 100 mL for a full pump stroke. By varying the number of pump strokes, the sample volume is controlled. Sampling pumps should be maintained and calibration checked periodically according to the manufacturer's instructions. The pumps shall be accurate to ± 5 % of the volume stated.

7.2.2 Long-Term Sampling—Small electrical pumps having stable low flow rates (2 to 50 mL/min), are required for long-term sampling (2 to 8 h). Flow rates to be used with each detector tube are given by the manufacturers. As with the mechanical pumps, the electrical pumps must be maintained and calibrated regularly. Maintenance and calibration are performed using the instructions supplied by the manufacturer of the pump. The pump flow rate, and, therefore, the sampled volume, shall be accurate to ± 5 % of the stated flow rate. With this system either area or personal monitoring can be accomplished.

7.3 *Accessories*—Several accessories are provided with detector tubes for special applications:

7.3.1 *Reactor Tubes*—These are tubes that are used in conjunction with detector tubes. Some gases and vapors, because of their low reactivity, are not easily detected by detector tubes alone. The reactor tubes consist of very powerful chemical reactants, which break down the unreactive compound into other more readily detectable substances, which standard detector tubes can detect. Thus, the reactor tube is placed upstream of the detector tube and the combination must be used for certain compounds as a detector tube system.

7.3.2 *Dryer Tubes*—Water vapor interferes with the detection of certain substances; therefore, dryer tubes are used upstream of the detector tube in these cases to remove the water vapor.

7.3.3 *Pyrolyzer*—A pyrolyzer is a hot wire instrument operated by batteries. Instructions for its use and maintenance are given in the manufacturers' instruction manuals. The

purpose of the pyrolyzer, as with reactor tubes, is to break down difficult-to-detect compounds into other compounds more easily detected. The breakdown in this case is caused by heat. The pyrolzyer is particularly useful for organic nitrogen compounds, one of the products of breakdown being nitrogen dioxide, which is easily monitored.

7.3.4 *Remote Sampling Line*—When the sampling point is remote from the pump location, a length of nonreactive tubing can be attached to the pump with the detector tube attached to the other end of the tubing. This is useful for sampling in inaccessible or dangerous places.

7.3.5 *Cooling Unit*—The cooling unit consists of a length of metal tubing through which the sampled gas is pulled. Because of the high thermal conductivity of the metal tubing, the hot sampling gas is cooled sufficiently so that it will not destroy the indicator in the detector tube. The cooling unit must be placed upstream from the detector tube. Cooling units are particularly useful when sampling flue gases.

8. Reagents

8.1 The reagents used are specific for each tube, and, to detect a specific gas, may vary from manufacturer to manufacturer. The instruction sheets supplied by the manufacturers give the principal chemical reaction(s) that occur(s) in the tube, thus showing the reagent that is used to react with the gas or vapor to produce the color change.

9. Sampling with Detector Tubes

9.1 *General*—Detector tubes made by one manufacturer must not be used with pumps made by a different manufacturer (**33**). Each lot of detector tubes is calibrated at the manufacturer's plant, using their equipment. The pumps of other manufacturers have different flow characteristics that cause different lengths-of-stain resulting in erroneous readings.

9.2 *Procedure* (34)—The detector tube program should be conducted under the supervision of a trained professional such as a chemist or an industrial hygienist. Carefully follow the instruction sheet of the manufacturer for the proper use of each detector tube. In general, the instruction sheet will include the following information.

- 9.2.1 Storage conditions.
- 9.2.2 Shelf life.
- 9.2.3 Chemical reaction and color change.
- 9.2.4 Test procedure.

9.2.5 Significant interferences.

9.2.6 Temperature and humidity correction factors, if required.

9.2.7 Correction for atmospheric pressure.

9.2.8 Measurement range.

10. Accuracy of Detector Tubes

10.1 The Safety Equipment Institute (SEI) has a certification program for certain detector tubes used in short-term sampling. This program is similar to the NIOSH program for evaluating and certifying detector tube performance (**35**, **36**). Under this program the tubes are required to meet an accuracy (95 % confidence level) of ± 25 % between one and five times the SEI test concentration and ± 35 % at one half the test concentration. The SEI test concentration is chosen as the Threshold Limit Value as defined by the American Conference of Governmental Industrial Hygienists for the test gas or vapor (**37**). The calculation of tube accuracy is based on a set of statistical procedures (**38**) and provides an estimate of accuracy under actual use conditions. The SEI Certified Equipment List should be consulted for the listing of approved units.

10.2 In general, the accuracy of any detector tube depends on the construction and chemistry of the tube along with the actual composition of the test atmosphere and the conditions under which the tube is read. For gases and vapors not covered by the SEI program, detector tubes may or may not meet the accuracy requirements of the previous paragraph (**39, 40**). There is also some variation in accuracy between manufacturers tubes designed to detect a specific compound. Therefore the user should verify the accuracy with the tube manufacturer or run his own tests to determine accuracy (**41-43**). It must be emphasized that a correct estimate of accuracy can only be done by qualified operators and with careful attention to the generation and verification of test gas or vapor concentrations (44).

10.3 Because the accuracy of a detector tube towards a specific compound depends on the cross-sensitivity of the tube to other gases or vapors present in the test atmosphere, the manufacturer should be consulted for information on cross-sensitivity effects for the specific chemistry employed in their tube. Quite frequently, several different indicating chemistries for a specific compound are available. Proper choice of indicating chemistry can minimize the effect of a co-contaminant in the test atmosphere.

11. Keywords

11.1 air monitoring; detector tubes; dosimeter sampling; grab sampling; sampling and analysis; toxic gases and vapor; workplace atmospheres

ANNEX

(Mandatory Information)

A1. SOME COMPOUNDS THAT CAN BE MEASURED BY DETECTOR TUBES

A1.1 The measurement ranges shown in Table A1.1 are not for a single tube. They are for the lowest and highest concentrations listed in manufacturer's brochures. Values are given in ppm(v) unless otherwise indicated.

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TABLE A1.1 Non-Exclusive List of Compounds Measurable by Detector Tubes

| Substance | 1994-1995 TLV ACGIH, ppm(v) | Measurement Range, ppm(v) | Substance | 1994-1995 TLV ACGIH, ppm(v) | Measurement Range, ppm(v) |
|-----------------------------------|--------------------------------|------------------------------|-----------------------------------|--------------------------------|------------------------------|
| Acetaldehyde | 25^ | 4-10 000 | N,N-Dimethylaniline | 5 | 0.5–10 |
| Acetic acid | 10 | 0.13-100 | Dimethyl ether | | 100-12 000 |
| Acetic anhydride | 5 | Qual. | Dimethyl formamide | 10 | 0.8-90 |
| Acetone | 750 | 50-20 000 | 1,1-Dimethyl hydrazine | 0.5 ^E | 0.25-3 |
| Acetonitrile | 40 | | N-Dimethyl nitrosamine | E | |
| Acetylene | B,C | 50-40 000 | Dimethyl sulfate | 0.1⋿ | 0.005-0.6 |
| Acrolein | 0.1 | 0.1-18 000 | Dimethyl sulfide | ••• | 1–100 |
| Acrylonitrile | 2 ^E | 0.13-35 000 | Dioxane | 25 | 13-25 000 |
| Allyl chloride | 1 | Qual. | Epichlorohydrin | 2 | 5-50 |
| Ammonia | 25 | 0.25-300 000 | Ethanol amine | 3 | 0.5-6 |
| myl acetate (all isomers) | 100–125 | 10-3000 | 2-Ethoxyethanol | 5 | Qual. |
| myl alcohols | | 100-2000 | Ethyl acetate | 400 | 20-15 000 |
| myl mercaptan | ••• | • • • | Ethyl acrylate | 5 ^E | 2300 |
| niline | 2 | 0.5–60 | Ethyl alcohol | 1000 | 25-75 000 |
| Intimony hydride, as Sb | 0.5 mg/m ³ | 0.05–3 | Ethyl amine | 5 | 1-60 |
| rsine | 0.05 | 0.04–160 | Ethyl benzene | 100 | 5-1800 |
| Benzene | 10 ^E | 0.13-500 | Ethyl bromide | 5 ^E | 15-400 |
| enzyl bromide | | | Ethyl chloride | 1000 | 50-8000 |
| enzyl chloride | 1 | 0.2–8 | Ethyl chloroformate | | |
| romine | 0.1 | 0.2-30 | Ethyl ether | 400 | 20-10 000 |
| romobenzene | ••• | 30-720 | Ethylene | B,C | 0.1-5000 |
| romoethane | | 15-400 | Ethylenediamine | 10 | 0.5–27 |
| romoform | 0.5 | 7-200 | Ethyleneimine | 0.5 | |
| ,3-Butadiene | 2 ^E | 1-1200 | Ethylene oxide | 1 ^E | 0.4-40 000 |
| Butane | 800 | 25-8000 | Ethyl glycol acetate | | 50-700 |
| -Butoxyethanol | 25 | Qual. | Ethyl mercaptan | 0.5 | 0.5-1000 |
| Butyl acetate (all isomers) | 150-200 | 10-10 000 | N-Ethyl morpholine | 5 | |
| lutyl alcohol (all isomers) | 50-100 ^A | 45100 | Formaldehyde | 0.3 ^{A,E} | 0.04-6400 |
| lutyl amine (all isomers) | 5^ | 2-36 | Formic acid | 5 | 1-2500 |
| utyl mercaptan | 0.5 | 0.5-15 | Furfuryl alcohol | 10 | Qual. |
| arbon dioxide | 5000 | 100-1 000 000 | Heptane | 400 | 20-2600 |
| arbon disulfide | 10 | 0.65-4000 | Hexane (all isomers) | 50-500 | 20-12 000 |
| arbon monoxide | 25 | 2-400 000 | Hydrazine | 0.1 [€] | 0.05-10 |
| Carbon tetrachloride | 5 ^F | 0.2-70 | Hydrochloric acid | 5^ | 0.1-5000 |
| Carbonyl sulfide | | 0.5–60 | Hydrocyanic acid | 4.7^ | 0.2-30 000 |
| Chlorine | 0.5 | 0.05-1000 | Hydrogen | B,C | 500-20 000 |
| Chlorine dioxide | 0.1 | 0.05-20 | Hydrogen fluoride as F | 3^ | 0.25-100 |
| hlorobenzene | 10 | 5610 | Hydrogen selenide as Se | 0.05 | 1-600 |
| Chlorobromomethane | 200 | 5-180 | Hydrogen sulfide | 10 | 0.2-400 000 |
| Chloro-1,1-difluoroethane | ••• | Qual. | isoamyl alcohol | 100 | Qual. |
| Chlorodifluoromethane | 1000 | 200-2800 | Isobutyl acetate | 150 | 10-1000 |
| Chloroform | 10 ^E | 2–500 | Isobutyl alcohol | 50 | 5-2900 |
| -Chloro-1-nitropropane | 2 | | Isooctane | | 10-1500 |
| Chloropicrin | 0.1 | 0.1–15 | Isopropyl acetate | 250 | 20-12 000 |
| -Chloroprene | 10 | 0.5-90 | Isopropyl alcohol | 400 | 20-50 000 |
| chromic acid, as Cr | 0.05 mg/m ^D | 0.1–0.5 mg/m ³ | Isopropylamine | 5 | 2-30 |
| umene | 50 | 5–400 | Mercury (inorganic) | 0.025 mg/m ^{3G} | 0.05–13.2 mg/m |
| yanogen | 10 | | Methyl acetate | 200 | 100-30 000 |
| yanides, as CN | 5 mg/m ³ | 2–15 mg/m ³ | Methyl acrylate | 10 | 5-200 |
| yanogen chloride | 0.3^ | 0.25-5 | Methylacrylonitrile | 1 | 0.2-32 |
| yclohexane | 300 | 100-6000 | Methyl alcohol | 200 | 20-60 000 |
| yclohexanol | 50 | 5-500 | Methylamine | 5 | 1-60 |
| cyclohexylamine | 10 | 2–38 | Methyl bromide | 5 | 0.5-600 |
| ecaborane | 0.05 | | Methyl chloride | 50 | |
| emeton | 0.01 | Qual. | Methyl chloroformate | | 5 1500 |
| emetonmethyl | 0.5 mg/m ³ | Qual. | Methyl chloroform | 350 | 5-1500 |
| Diborane | 0.1 | 0.02-5 | Methyl cyclohexanol (all isomers) | 50 | 5-100 |
| ,2-Dibromoethane | E | 5-700 | Methylene chloride | 50 ^E | 25-3000 |
| -Dichlorobenzene | 25 | 2.5-300 | Methyl ethyl ketone (MEK) | 200 0.04 F | 20-50 000 |
| | 10 ^F | 2.5–300 | Methyl hydrazine | 0.2 ^{A,E} | 0.2-10 |
| ichlorodifluoromethane (R-12) | 1000 | | Methyl iodide | 2 ^E | 5–40 |
| ,1-Dichloroethane | 100 | 8-300 | Methyl isobutyl carbinol | 25 50 | E 15 000 |
| ,2-Dichloroethane | 10 | 5-720 | Methyl isobutyl ketone (MIBK) | 50 0.5 | 5-15 600 |
| ,2-Dichloroethylene | 200 | 5–500 | Methyl mercaptan | 0.5 | 0.25-1650 |
| ichloroethyl ether | 5 | | Methyl methacrylate | 100 | 5-700 |
| ,1-Dichloro-1-nitroethane | 2 | | Methyl styrene | 50 | 10-500 |
| ichlorotetra fluoroethane (R-114) | 1000 | 200-2800 | Morpholine | 20 | Qual. |
| ichlorovos | 0.1 | 0.05 | Nickel carbonyl | 0.05 | 0.1-800 |
| iethylamine | 5 ^G | 160 | Nitric acid | 2 | 0.1-700 |
| Diethyl benzene | ••• | | Nitric oxide | 25 | 0.55000 |
| Nisobutyl ketone | 25 | | Nitroethane | 100 | |
| Diisopropylamine | 5 | Qual. | Nitrogen dioxide | 3 | 0.5-1000 |
| Dimethyl acetamide | 10 | 1.5-400 | Nitroglycerine | 0.05 | Qual. |
| Dimethylamine | 5 | 1-60 | Nitroglycol | | 0.25 |

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TABLE A1.1 Continued

| Substance | 1994–1995 TLV ACGIH, ppm(v) | Measurement Range, ppm(v) | Substance | 1994–1995 TLV ACGIH, ppm(v) | Measurement Range, ppm(v) |
|-------------------------------|--------------------------------|------------------------------|-------------------------------|--------------------------------|------------------------------|
| Nitromethane | 20 | | Sulfuric acid | 1 mg/m ³ | 1–5 mg/m ³ |
| 1-Nitropropane | 25 | | 1,1,2,2-Tetrabromoethane | | 25-200 |
| 2-Nitropropane | 10 ^E | | 1,1,2,2-Tetrachloroethane | 1 | 50-1000 |
| Octane | 300 | 10-5000 | Tetrachloropropene | | |
| Oil (mist and vapour) | 5 mg/m ³ | 1–10 mg/m ³ | Tetrahydrofuran | 200 | 20-50 000 |
| Oxygen | | 1.5-24 vol. % | Tetrahydrothiophene | | 1–16 |
| Ozone | 0.1 ^A | 0.025-1000 | Toluene | 50 | 1-1800 |
| Pentane | 600 | 50-3900 | 2,4-Toluene diisocyanate | 0.005 | 0.02-0.2 |
| Perchloroethylene | 25F | 0.1-10 000 | 2,6-Toluene diisocyanate | | 0.02-0.2 |
| Phenol | 5 | 0.4-63 | 1,1,2-Trichloroethane | 10 | 10–170 |
| Phosgene | 0.1 | 0.02-75 | Trichloroethylene | 50 ^H | 0.125-10 000 |
| Phosphine | 0.3 | 0.01-3000 | Trichlorofluoromethane (R 11) | 1000^ | 100-1400 |
| Propane | B,C | 100-20 000 | 1,2,3-Trichloropropane | 10 | 10-1200 |
| Propyl acetate (both isomers) | 200 | 20-14 000 | 1,1,2-Trichloro-1,2,2-tri- | 1000 | 2002800 |
| Propylene | B,C | 20-31 400 | fluoroethane (R 113) | | |
| Propylene dichloride | 75 | 5-440 | Triethylamine | 1 | 1-60 |
| Propylene imine | 2 ^E | 0.25-3 | Trifluorobromomethane | 1000 | Qual. |
| Propylene oxide | 20 | 500-50 000 | Trimethylamine | 5 | 1–30 |
| Propyl mercaptan | | 0.5–10 | Vinyl chloride | 5 ^D | 0.025-10 000 |
| n-Propyl nitrate | 25 | | Vinylidene chloride | 5 | 0.4-600 |
| Pyridine | 5 | 0.2-35 | Vinyl pyridine | | |
| Stibine | 0.1 | 0.05-3 | Water vapour | | 0.05-40 mg/L |
| Styrene, monomer | 50 | 2-1000 | Xylene (all isomers) | 100 | 5-2500 |
| Sulfur dioxide | 2 | 0.1-80 000 | | | |

A Denotes ceiling limit.

^B Simple asphyxiant.

^c Explosive.

^D Confirmed human carcinogen.

E Suspected human carcinogen.

F Animal carcinogen.

^G Not classifiable as a human carcinogen.

^H Not suspected as a human carcinogen.

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