

DEPARTMENT OF COMMERCE.

BUREAU OF STANDARDS.

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CIRCULAR OF THE BUREAU OF STANDARDS.

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UNITED STATES GOVERNMENT SPECIFICATION FOR
FIRE-EXTINGUISHING LIQUID (CARBON TETRA-
CHLORIDE BASE).¹

FEDERAL SPECIFICATIONS BOARD.

STANDARD SPECIFICATION NO. 36.

This Specification was officially adopted by the Federal Specifications Board, on July 3, 1922, for the use of the Departments and Independent Establishments of the Government in the purchase of materials covered by it.

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1. GENERAL.

The liquid shall be capable of extinguishing fires caused by the burning of inflammable liquids and solids, shall be a nonconductor of electricity, and shall consist of carbon tetrachloride containing other suitable products necessary to produce a liquid conforming to the following requirements when tested according to the methods herein stated:

APPEARANCE.—It shall be a clear, homogeneous, mobile liquid.

SPECIFIC GRAVITY.—The specific gravity at 15.5/15.5° C. shall not be less than 1.500.

COLD TEST.—The liquid must have a cold test not higher than minus 45.5° C. (minus 50° F.).

¹ The fumes generated in extinguishing fire with this liquid are intensely irritating and are also poisonous, and it is unsafe for use in confined spaces. On account of the rapid generation of such fumes, a fire can not be fought for any considerable length of time. However, this material is the only suitable liquid known, which is a nonconductor of electricity, for use in hand-fire extinguishers in fighting incipient fires in connection with electrical apparatus. Care should be exercised in its use.

DISTILLATION.—Not over 2 per cent shall distil below 60° C. At least 90 per cent shall distil between 70 and 80° C. At least 99 per cent shall distil below 100° C.

IMPURITIES.—The liquid shall be free from nitrobenzene, water, acid or alkali, and free halogens, and shall not contain more than 1 per cent by weight of carbon disulphide. It shall not corrode metals used in fire extinguishers.

2. SAMPLING.

No samples shall be submitted with bids. It is mutually agreed by buyer and seller that a single container (sealed can of not more than 1 gallon capacity) out of each lot of not more than 1,000 containers (sealed can of not more than 1 gallon capacity) shall be taken as representative of the whole, and sent unopened to the laboratory for test. When for any reason this is not done, the inspector shall thoroughly mix the contents of the container sampled, transfer not less than 1 quart to a clean, dry glass bottle or tin can, which must be nearly filled with the sample, securely stoppered with a new clean cork or well-fitting cover or cap, sealed, and distinctly labeled by the inspector, and sent to the laboratory for test.

3. METHODS OF TESTING.

APPEARANCE.—The sample, thoroughly shaken in the original container and immediately poured into a clean, dry glass vessel, shall not show any turbidity.

SPECIFIC GRAVITY.—Specific gravity shall be determined by any convenient method accurate to the third decimal place.

COLD TEST.—The apparatus used for this test shall consist of a 1 by 8 inch test tube supported in a larger tube about $1\frac{1}{4}$ inches in diameter. The inner tube shall be provided with a stopper carrying a low-temperature thermometer graduated in degrees to minus 60° C. or below, and a wire bent into a loop at the lower end, which serves as a stirrer. Twenty-five cubic centimeters of the liquid to be tested shall be placed in the inner tube and then the whole slowly immersed in the cooling bath contained in a Dewar vacuum bottle. The liquid shall be kept in constant and vigorous agitation by means of the stirrer, taking frequent temperature readings as the sample cools. The lowest temperature at which the material flows shall be taken as the cold test of the liquid. The cooling bath should not be higher than minus 60° C. and for this purpose liquid air is recommended. However, a suitable

bath may be obtained by mixing carbon dioxide snow with gasoline to form a mixture of a thick mushy consistency. The carbon dioxide snow can be readily obtained from the ordinary cylinders in which it is furnished commercially by holding a felt sack or several layers of towel over the mouth of the valve, tilting the cylinder until the liquid carbon dioxide can flow out, and opening the valve.

DISTILLATION.—The apparatus used for this test shall consist of a standard 100 cc Engler flask, an accurately graduated thermometer having a range of from 0° C. to not less than 120° C., a suitable water-cooled condenser, and an accurately graduated 100 cc cylinder.

PROCEDURE.—(a) The condenser tube shall be swabbed to remove any liquid remaining from a previous test. A piece of soft cloth attached to a cord or copper wire may be used for this purpose.

(b) The bulb of the thermometer shall be uniformly covered with long-fiber absorbent cotton weighing not less than 3 nor more than 5 mg. A fresh portion of clean cotton shall be used for each distillation.

(c) One hundred cubic centimeters of the sample shall be transferred to the Engler flask, taking care that none of the liquid shall be permitted to flow into the vapor tube.

(d) The thermometer provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(e) The flask shall be supported on a plate of asbestos 20 cm in diameter, having an opening 4 cm in diameter in its center. The flask and burner shall be surrounded with a shield to prevent fluctuation in the temperature of the neck of the flask.

(f) The vapor outlet tube shall be connected by means of a cork to the condenser tube. The vapor tube shall extend into the condenser tube from 2 to 5 cm.

(g) When everything is in readiness, heat shall be applied with an open flame at a uniform rate, so regulated that the first drop of condensate falls from the condenser into the 100 cc cylinder in not less than 5 nor more than 10 minutes. The heat shall then be so regulated that the distillation will proceed at a uniform rate of not less than 4 nor more than 5 cc per minute. The volume of distillate shall be recorded at 60, 70, 80, and 100° C.

IMPURITIES.—(a) *Water*.—Five cubic centimeters of the well-shaken sample shall be transferred to a test tube and a small piece of *clean*, metallic sodium added; no gas should be evolved from the surface of the metal. The appearance of the solution shall also be noted. Turbidity indicates the presence of water (owing to the insolubility of sodium hydroxide in carbon tetrachloride).

(b) *Neutrality*.—Twenty-five cubic centimeters of the liquid shall be thoroughly shaken with an equal volume of water and allowed to settle. Small pieces of sensitive red and blue litmus paper put in the aqueous layer shall not indicate an acid or alkaline reaction.

(c) *Free halogens*.—No color shall be produced when 25 cc of the liquid is thoroughly shaken with an equal volume of 10 per cent potassium iodide solution containing starch as an indicator.

(d) *Nitrobenzene*.—The original sample or any fraction thereof obtained by distillation must have no odor of nitrobenzene.

(e) *Carbon disulphide*.—Radcliffe's method:² Twenty-five cubic centimeters of about 2 *N* alcoholic potassium hydroxide solution shall be transferred to a flask of suitable size, the flask loosely stoppered with a cork and weighed. One cubic centimeter of the sample shall be added by means of a pipette, the cork replaced, and the flask again weighed, obtaining the weight of the sample taken. The mixture shall stand at room temperature for five minutes and then be rendered slightly acid with dilute acetic acid, using phenolphthalein as indicator. The mixture shall be diluted with water, cooled thoroughly, and an excess of solid sodium bicarbonate added. The milky, turbid mixture shall then be titrated with 0.1 *N* iodine solution, using starch solution as indicator, and the percentage by weight of CS₂ calculated (1 atom I = 1 molecule CS₂).

(f) *Corrosion*.—Satisfactory corrosion tests can not be made in a short time, but the Government reserves the right to reject bids for material that has been found corrosive in service.

(g) *Conductivity test*.—A standard voltmeter having 100 ohms per volt of its scale shall be connected in series with a 110-volt circuit and two electrodes consisting of parallel copper wires 5 cm long and spaced 2.5 mm apart immersed in the liquid to be tested. On closing the circuit there shall be no appreciable motion of the needle.

²J. Soc. Chem. Ind., 28, p. 229: 1909.



