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DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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

FEDERAL SPECIFICATIONS BOARD, SPECIFICATION No. 36a

[Revision adopted June 1, 1924]

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 fire-extinguishing liquid (carbon tetrachloride base).

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I. TYPE

The material covered by this specification is intended for use with hand-chemical fire extinguisher (1-quart-carbon-tetrachloride type), Federal Specifications Board Specification No. 124.

### 1. PRECAUTION

The fumes generated in extinguishing fire with this liquid are intensely irritating and are also poisonous. Care should, therefore, be exercised when using it in confined spaces. On account of the rapid generation of such fumes, a fire can not be fought for any considerable length of time in confined spaces. 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.

### II. MATERIAL AND WORKMANSHIP

No details.

### III. GENERAL REQUIREMENTS

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.

### IV. DETAIL REQUIREMENTS

#### 1. APPEARANCE

It shall be a clear, homogeneous, mobile liquid.

#### 2. SPECIFIC GRAVITY

The specific gravity at 15.5/15.5° C. shall be not less than 1.50.

#### 3. COLD TEST

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

#### 4. DISTILLATION

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

#### 5. 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 brass, lead, or other material of equal corrosion-resisting properties.

## V. METHOD OF INSPECTION AND TESTING

### 1. 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.

### 2. METHODS OF TESTING

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

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

(c) **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  $60^{\circ}$  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 temperature of the cooling bath should not be higher than minus  $60^{\circ}$  C. and for this purpose liquid air is recommended. However, a suitable bath can be obtained by mixing carbon dioxide snow with gasoline to form a mixture of a thick mush 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.

(d) **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  $0^{\circ}$  C. to not less than  $120^{\circ}$  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.

(e) *IMPURITIES.*—(1) *Water.*—Five cubic centimeters of the well-shaken sample shall be transferred to a test tube and a small piece of *clean*, metallic sodium immediately 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).

(2) *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.

(3) *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.

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

(5) *Carbon disulphide*.—Radcliffe's method:<sup>1</sup> 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, to obtain 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 CS<sub>2</sub> calculated (1 atom I = 1 molecule CS<sub>2</sub>).

(6) *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.

(7) *Conductivity test*.—A voltmeter having approximately 100 ohms per volt of its scale (about 150-volt 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.

## VI. PACKING AND MARKING

No details.

## VII. ADDITIONAL INFORMATION

### 1. BASIS OF PURCHASE

The material shall be purchased by weight or volume. The volume will be corrected to a standard temperature of 15.5° C. (60° F.), it being mutually agreed that this correction shall be based on a coefficient of expansion of 0.0012 per degree centigrade (or 0.00067 per degree Fahrenheit). A gallon of fire-extinguishing liquid shall mean 231 cubic inches at 15.5° C. (60° F.). The contract shall state the unit on which purchases are to be based; for example, quart, gallon, pound, 100 pounds, etc.

### 2. CALCULATION OF WEIGHT AND VOLUME

(a) *WEIGHT*.—A gallon of fire-extinguishing liquid at 15.5° C. (60° F.) weighs not less than 12.49 pounds. The exact weight in pounds per gallon of any sample can be determined by multiplying the specific gravity at 15.5/15.5° C. (60/60° F.) by 8.33. Example: If the specific gravity at 15.5° C. is 1.5832, the weight per gallon at this temperature will be  $1.5832 \times 8.33 = 13.188$  pounds.

<sup>1</sup> J. Soc. Chem. Ind., 28, p. 229; 1909.

(b) VOLUME.—The volume paid for shall be the volume corrected to a standard temperature of 15.5° C. (60° F.). The correction shall be made by deducting from (when the temperature of gauging is above 15.5° C.) or adding to (when the temperature of gauging is below 15.5° C.) the volume as gauged. Such deduction or addition shall be computed on the basis of a coefficient of expansion of 0.0012 per degree centigrade (or 0.00067 per degree Fahrenheit). Example: If the temperature at which the fire-extinguishing liquid is gauged is 75° F. and the volume delivered (at that temperature) is 100 quarts, then  $0.00067 \times 15 \times 100$  equals the quantity in quarts which must be subtracted from 100 quarts to give the true volume in quarts at 60° F. If the temperature at which the fire-extinguishing liquid is gauged is 10° C., then  $0.0012 \times 5.5 \times 100$  equals the quantity in quarts which must be added to the gauged volume of 100 quarts to give the true volume in quarts at 15.5° C.

### VIII. GENERAL SPECIFICATIONS

No details.

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