EXHIBIT 150
PART 13

CERTIFICATE

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By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

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Document Name: ASTM E776: Standard Test Method for Forms of Chlorine in Refuse-Derived Fuel

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Standards Body: American Society for Testing and Materials

APPROVED

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Standard Test Method for Forms of Chlorine in Refuse-Derived Fuel[1]

This standard is issued under the fixed designation E 776; 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 test method covers the determination of the forms of chlorine in refuse-derived fuel-three (RDF): total chlorine, water-soluble chloride, and water-insoluble chlorine.

1.2 This test method may be applicable to any waste material from which a laboratory analysis sample can be prepared.

1.3 *This standard does not purport to address all of the safety problems, 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.* For specific precautionary statements see Section 6 and 11.2.1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]
E 144 Practice for Safe Use of Oxygen Combustion Bombs[3]
E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[4]
E 287 Specification for Burets[3]
E 711 Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter[5]
E 829 Practice for Preparing Refuse-Derived Fuel (RDF) Laboratory Samples for Analysis[5]

3. Descriptions of Terms Specific to This Standard

3.1 *refuse-derived fuels*—solid forms of refuse-derived fuels from which appropriate analytical samples may be prepared are defined as follows in *ASTM STP 832*:[6]

RDF-1—Wastes used as a fuel in as-discarded form with only bulky wastes removed.

RDF-2—Wastes processed to coarse particle size with or without ferrous metal separation.

RDF-3—Combustible waste fraction processed to particle sizes, 95 % passing 2-in. square screening.

RDF-4—Combustible waste fraction processed into powder form, 95 % passing 10-mesh screening.

RDF-5—Combustible waste fraction densified (compressed) into the form of pellets, slugs, cubettes, or briquettes.

3.2 *total chlorine*—all chlorine as determined in the refuse-derived fuel.

3.3 *water-insoluble chlorine*—water-insoluble chlorides and chlorine in the refuse-derived fuel.

3.4 *water-soluble chloride*—those chlorides which are water-solubilized by water extraction as determined in the refuse-derived fuel.

4. Summary of Test Method

4.1 The forms of chloride and chlorine are determined. The various procedures in the method convert the forms of chlorine into a water-soluble chloride form that can be quantitated by titration.

4.1.1 *Total Chlorine*—The sample is combusted in an oxygen atmosphere. The chlorine is converted to chloride and absorbed in an alkaline solution.

4.1.2 *Water-Soluble Chlorides*—A portion of the analysis sample is successively extracted with hot chloride-free water.

4.1.3 *Water-Insoluble Chlorine*—Water-insoluble chlorine is calculated from the results of the total chlorine and the water-soluble chloride determination where:

water-insoluble chlorine = total chlorine – water-soluble chlorides

4.2 The chlorides contained in the alkaline solution (4.1.1) and the extraction solution (4.1.2) are determined by potentiometric (see Section 13) or modified Volhard titration (see Section 14).

5. Significance and Use

5.1 The standard is available to producers and users of RDF for determining the content and forms of chlorine present in the fuel.

6. Precautions

6.1 Due to the origins of RDF in municipal waste, common sense dictates that some precautions should be observed when conducting tests on the samples. Recommended hygienic practices include use of gloves when handling RDF; wearing dust masks (NIOSH-approved type), especially while milling RDF samples; conducting tests under negative pressure hood when possible; and washing hands before eating or smoking.

7. Interferences

7.1 *Potentiometric Titration Method A:*

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[1] This test method is under the jurisdiction of ASTM Committee D-34 on Waste Management and is the direct responsibility of Subcommittee D34.08 on Thermal Treatment.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[5] *Annual Book of ASTM Standards*, Vol 11.04.

[6] *Thesaurus on Resource Recovery Terminology, ASTM STP 832*, ASTM, 1983, p. 72.

TABLE 1 Typical Data Sheet and Calculation of End Point from a Potentiometric Titration of Chloride

Volume of 0.025 *N* $AgNO_3$ solution, mL	Millivolt Reading	Volume of 0.025 *N* $AgNO_3$ solution, mL	Millivolt Reading	Volume of 0.025*N* $AgNO_3$ solution, mL	Millivolt Reading
0.0	−1.38	18.4	−0.65	20.2	+0.26
1.0	−1.29	18.6	−0.63	20.3	+0.38
2.0	−1.26	18.8	−0.59	20.4	+0.47
4.0	−1.21	19.0	−0.55	20.5	+0.55
6.0	−1.18	19.1	−0.53	20.6	+0.60
8.0	−1.14	19.2	−0.51	20.7	+0.65
10.0	−1.10	19.3	−0.48	20.8	+0.69
12.0	−1.04	19.4	−0.46	20.9	+0.72
14.0	−0.97	19.5	−0.42	21.0	+0.75
15.0	−0.95	19.6	−0.38	21.5	+0.90
16.0	−0.86	19.7	−0.33	22.0	+0.98
17.0	−0.79	19.8	−0.27	22.5	+1.04
17.5	−0.75	19.9	−0.19	23.0	+1.09
18.0	−0.70	20.0	−0.06	24.0	+1.17
18.2	−0.67	20.1	+0.08	25.0	+1.24

The greatest change of voltage per unit increment addition of $AgNO_3$ solution occurs between 19.8 and 20.5 mL. Determine the end point and titer as follows:

mL	mV	1st Derivative	2nd Derivative
19.8	−0.27		
19.9	−0.19	+0.08	+0.05
20.0	−0.06	+0.13	+0.01
20.1	+0.08	+0.14	+0.04
20.2	+0.26	+0.18	−0.06
20.3	+0.38	+0.12	−0.03
20.4	+0.47	+0.09	−0.01
20.5	+0.55	+0.08	

Interpolation:

$$20.1 \text{ mL} + \left(\frac{0.04}{0.10} \times 0.1\right) = 20.14 \text{ mL}$$

7.1.1 Iodide and bromide are also titrated as chloride. Ferricyanide causes high results and must be removed. Chromate and dichromate interfere and should be reduced to the chromic state or be removed. Ferric iron interferes if present in an amount substantially higher than the amount of chloride. Chromic ions, ferrous ions, and phosphates do not interfere.

7.1.2 Grossly contaminated sample solutions usually require pretreatment. Where contamination is minor, some contaminants can be destroyed simply by the addition of nitric acid.

7.2 *Volhard Titration Method B:*

7.2.1 Compounds that have a strong oxidizing action interfere by reacting with thiocyanate. These compounds should be reduced beforehand by treatment with ferrous sulfate or a similar reducing agent.

7.2.2 Salts of mercury and palladium interfere by reacting with thiocyanate. They may be removed by precipitation with hydrogen sulfide before the addition of silver nitrate. The excess of sulfide is easily removed by gently boiling the acid solution for a few minutes. Sulfite can be eliminated in the same way.

7.2.3 Cyanide is also precipitated by silver nitrate. It is usually determined separately by the Liebig-Deniges method and a correction is applied to the results of the Volhard titration.[7]

7.2.4 The Volhard method, as with the potentiometric method, directly applied to a mixture of halides can determine only total halide content excluding fluoride. Preliminary treatment is necessary for the determination of chloride alone in a mixture.[8]

8. Apparatus

8.1 *Balance*, having a sensitivity of 0.1 mg.

8.2 *Apparatus for Bomb Combustion of the Sample*.

8.2.1 *Oxygen Bomb*, similar to that used in the determination of the calorific value of refuse-derived fuels as described in Test Method E 711.

8.2.2 *Capsule*, for holding the sample, approximately 25 mm in diameter at the top, approximately 12 mm deep, and conforming to Test Method E 711.

8.2.3 *Firing Wire*, as specified in Test Method E 711.

8.2.4 *Firing Circuit*, as specified in Test Method E 711.

8.2.5 *Metal Vessel*, cylindrical, such that the bomb will be fully immersed when approximately 2 L of water are added.

8.3 *Magnetic Stirrer and Stirring Bars*.

8.4 *Apparatus for Potentiometric Titration:*

8.4.1 *Potentiometric Titration Assembly*, using a silver indicator electrode and a calomel reference electrode containing a saturated sodium nitrate solution as a bridge.

NOTE 1—All glassware and graduated apparatus should be Class A or equivalent as described in Specification E 287.

9. Reagents

9.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the

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[7] Scott's *Standard Method of Chemical Analysis*, edited by M. H. Furman, D. Van Nostrand Co., Inc., New York, NY.

[8] Koltoff, I. M., and Stenger, V. A., *Volumetric Analysis II*, Interscience Publishers, Inc., New York, NY.

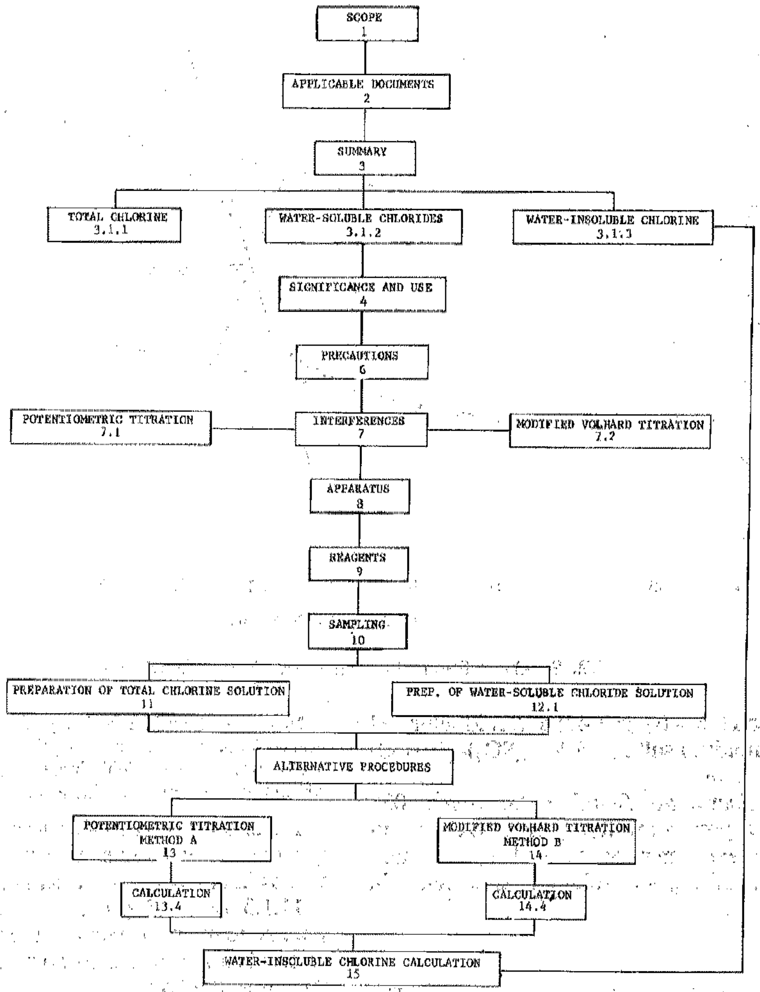

FIG. 1 Forms of Chlorine in Refused-Derived Fuel

Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[9] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

9.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean at least Type III reagent water conforming to Specification D 1193.

9.3 *Potassium Hydroxide Solution* (0.2 *N*)—Dissolve 13.2 g of potassium hydroxide (KOH) in water and dilute to 1 L with water.

9.4 *Oxygen*—The oxygen used for combustion shall be free of combustible matter. Oxygen manufactured from liquid air, guaranteed to be greater than 99.5 % pure, will meet this requirement.

9.5 *Sodium Chloride* (NaCl)—Primary standard quality (purity of 100 ± 0.02 %).

9.6 *Sodium Chloride, Primary Standard Solution* (0.025 *N*)—Crush 10 to 20 g of primary standard sodium chloride (NaCl) to 100-mesh fineness and dry in a glass container at 120°C for 2 h. Stopper and keep desiccated. Dissolve 5.844 g ± 0.1 mg of dried primary standard NaCl in water and dilute to 1 L. Dilute 25.00 mL of this solution to 100.0 mL.

9.7 *Methanol.*

9.8 *Nitric Acid* (1+1)—Mix equal volumes of concentrated nitric acid (HNO_3, sp gr, 1.42) and water.

9.9 *Silver Nitrate, Standard Solution* (0.025 *N*)—Dissolve 4.247 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L. Store in an amber glass bottle. Standardize against 0.025 *N* sodium chloride solution as directed in 13.1.1 and 14.1.1.

9.10 *Potassium Chromate – Potassium Dichromate Indicator* (K_2CrO_4 – $K_2Cr_2O_7$)—Dissolve 4.2 g of K_2CrO_4 and 0.7 g of $K_2Cr_2O_7$ in 100 mL of water.

9.11 *Nitrobenzene.*

9.12 *Ferric Ammonium Sulfate Indicator Solution*—Add

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[9] Reagent Chemicals, American Chemical Society Specifications, Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

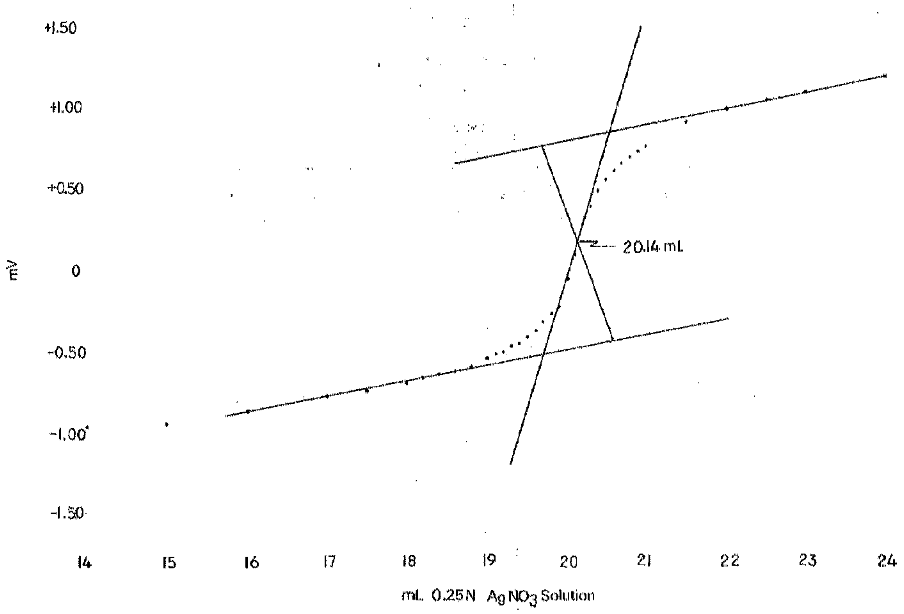

FIG. 2 Graph From a Potentiometric Titration of Chloride

sufficient concentrated HNO_3 (sp gr 1.42) to a cold saturated solution of ferric ammonium sulfate $[FeNH_4(SO_4)_2 \cdot 12H_2O]$ to remove the brown color.

9.13 *Potassium Thiocyanate, Standard Solution* (0.025 *N*)—Dissolve 3 g of potassium thiocyanate (KCNS) in freshly distilled or boiled water, dilute to 1 L, and standardize against 0.025 *N* $AgNO_3$ solution as directed in 14.1.2.

10. Sampling

10.1 RDF products are frequently nonhomogeneous. For this reason significant care should be exercised to obtain a representative laboratory sample from the RDF lot to be characterized.

10.2 The sampling method for this procedure should be based on agreement between the involved parties.

10.3 The laboratory sample must be air-dried and the particle size reduced to pass a 0.5-mm screen as described in Practice E 829. This procedure must be performed carefully to preserve the sample's representative characteristics (other than particle size), while preparing the analysis sample to be used in the procedures.

PREPARATION OF CHLORINE SOLUTIONS FOR ANALYSIS

11. Total Chlorine (Oxygen Bomb Method)

11.1 *Preparation of Sample and Bomb* (see Note 3):

11.1.1 *Sample Weight*—Weigh to the nearest 0.1 mg about 1 g of thoroughly mixed air-dried analysis RDF sample into the bomb capsule. A pellet may be made from the air-dried analysis RDF sample, accurately weighed, and placed into the bomb capsule. Place the capsule containing the sample into the capsule holder.

NOTE 2—There is a tendency for chlorine to adhere to the bomb walls, especially if the bomb is pitted or has been used previously to determine high levels of chlorine. Unless the bomb is thoroughly cleaned before use, the blanks may have values in excess of reality.

11.1.2 *Firing Wire*—Connect a length of firing wire to the ignition terminals in such a manner that the loop of firing wire is in contact with the sample.

11.1.3 *Bomb Preparation*—Add 20 to 25 mL of 0.02 *N* potassium hydroxide solution to the bomb and wet the entire internal surface of the bomb with this solution (see Note 3). Assemble the bomb.

NOTE 3—Sodium hydroxide solution at appropriate concentration may be used.

11.2 *Addition of Oxygen*—Admit oxygen to the bomb slowly to avoid blowing the sample from the capsule until a pressure of 25 atm is reached.

11.2.1 **Caution**—The following precautions are recommended for safe calorimeter operation. Additional precautions are given in Practice E 144.

11.2.1.1 The weight of RDF sample and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendations.

11.2.1.2 Bomb parts should be inspected carefully after each use. Threads on the main closure should be checked frequently for wear. Cracked or significantly worn parts should be replaced. The bomb should be returned to the manufacturer occasionally for inspection and possibly proof firing.

11.2.1.3 The oxygen supply cylinder should be equipped with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety codes. Suitable reducing valves and adaptors for 300 to 500 psi (2070 to 3450 kPa) discharge pressures are obtainable from commercial sources of compressed gas equipment. The pressure gage shall be checked periodically for accuracy.

11.2.1.4 During ignition of a sample, the operator must not permit any portion of his body to extend over the calorimeter.

11.3 *Preparation of the Chlorine Solution*—Immerse the bomb in a cold-water bath, connect it to the firing circuit, and close the circuit to ignite the sample. Allow the bomb to stand in the water bath for not less than 10 min after firing. Remove the bomb from the water bath, invert the bomb and shake for about 10 min (Note 4). Release the pressure at a slow, uniform rate so that the pressure is reduced to atmospheric in not less than 1 min. Open the bomb and examine the inside for traces of unburned material or sooty deposits. If any are found, discard the determination and thoroughly wash all parts of the bomb interior before using it again. If no unburned material or sooty deposits are present, rinse the interior of the bomb, the sample capsule, and the interior surface of the bomb cover with a fine jet of hot water and collect the contents and washings in a beaker or flask.

NOTE 4—Inverting and shaking a heavy, wet, and possibly slippery bomb may present a hazard. Precautions should be taken when performing this function.

12. Extraction of Water-Soluble Chlorides

12.1 *Preparation of the Chloride Solution*—Weigh to the nearest 0.1 mg about 5 g of thoroughly mixed, air-dried RDF analysis sample. Transfer to a 500-mL beaker and add 100 mL of hot water. Heat to close to boiling for ½ h with occasional stirring to ensure thorough wetting of the sample. Decant the supernatant liquid through a fast, qualitative filter paper collecting the filtrate in a beaker. Repeat the extraction two more times, each time using 100 mL of water and pooling the filtrates. After the third extraction, wash the sample thoroughly using 100 mL of hot water, adding the wash water to the pooled filtrates.

12.2 *Preparation of the Chloride Solution* (Alternative Method)—Weigh to the nearest 0.1 mg about 5 g of thoroughly mixed air-dried analysis sample (Note 5). Transfer to a 500-mL beaker and add 100 mL of hot water. Heat to close-to-boiling for ½ h with occasional stirring to ensure thorough wetting of the sample. Remove the beaker from the hot plate and allow the sample to settle. Decant the supernatant to a 100-mL centrifuge tube and centrifuge for ½ h at approximately 1200 rpm. Decant the supernatant through a fast qualitative filter paper. Repeat the extraction and centrifuging two more times, each time using 60 mL of water and pooling the filtrates. After the third extraction, wash the centrifuge tube and filter paper thoroughly using 60 mL of hot water, adding the wash water to the pooled filtrates (Notes 6 and 7).

NOTE 5—The weight of the sample used can be adjusted when higher or lower concentrations of water-soluble chlorides are anticipated.

NOTE 6—When high concentrations of chlorides are anticipated, dilute the cooled extraction solution to volume in a suitably sized volumetric flask and continue the analysis with a suitably sized aliquot.

NOTE 7—If the chloride solution is highly colored or turbid the potentiometric method (Section 13) should be used. If the Volhard method is to be used (Section 14), the color and turbidity must first be removed by aluminum hydroxide [$Al(OH)_3$] coagulation or by some other suitable method of turbidity or color removal without interfering with the chloride content of the solution.[10]

ALTERNATIVE PROCEDURES

13. Potentiometric Titration (Method A)

13.1 *Standardization of Reagents*—Prepare a standard solution of silver nitrate by adding 10.0 mL of standard 0.025 *N* NaCl solution to a 250-mL beaker. Add 40 to 50 mL of water and 50 mL of methanol (Note 9). Add 2 mL of HNO_3 (1 + 1) and titrate potentiometrically with the $AgNO_3$ solution as directed in 13.3. Calculate the normality of the $AgNO_3$ solution as follows:

$$\text{Normality of } AgNO_3 = \text{mL of NaCl solution used} \times \text{normality of NaCl solution/mL of } AgNO_3 \text{ solution required}$$

NOTE 8—Methanol increases the sensitivity of the inflection point. However, it may be omitted if it is found to interfere with or damage certain electrode systems.

13.2 *Blank Determination*—Prepare a solution for blank determination in the same manner as the preparation of chlorine solutions for the respective type of chlorine desired as described in Sections 11 or 12 using all the reagents in the same quantities and eliminating the RDF sample. Add to that blank solution 0.025 N NaCl solution as used in the standardization of $AgNO_3$ solution (13.1.1). Proceed as directed in 13.3. The difference in the volume of $AgNO_3$ solution required in this titration and that volume required for the standardization titration (13.1.1) is the true titer blank.

$$A - B = \text{true titer blank}$$

where:

A = millilitres of $AgNO_3$ required for the blank determination and

B = millilitres of $AgNO_3$ required for the standardization determination (13.1).

13.3 *Procedure:*

13.3.1 Concentrate the chlorides in the prepared solution (11.3, 12.1, or 12.2, respectively) for the type of chlorine desired by evaporation to about 50 mL. Add 50 mL of methanol and neutralize with HNO_3 (1+1) using phenolphthalein as an indicator. Add 2 mL of HNO_3 (1+1) in excess. Immerse the electrodes into the sample solution. While gently stirring on a magnetic stirrer, titrate by adding 0.10-mL increments of standard $AgNO_3$ solution and record the millivolt reading after each addition (Notes 9 and 10). Titrate well beyond the end point.

NOTE 9—Larger increments of $AgNO_3$ solution may be added before and after the end point. However, as the end point is reached, the 0.10-mL increment will give the most accurate end point.

NOTE 10—The end point is reached when the change of voltage is greatest upon the addition of the given increment of standard $AgNO_3$.

13.3.2 Plot the millivolt readings versus the millilitres of standard $AgNO_3$ solution added. Determine the end point (point of inflection) graphically from the titration curve or mathematically (second derivative) from the tabulated data. (See Table 1 and Fig. 1 for examples.)

13.4 *Calculations*—Calculate the weight percent of chloride as follows:

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[10] APHA, 14th ed., Method 408-A, Section 3a.

$$\text{Chloride, weight \%} = [C - (A - B)] \times N \times 0.03545 \times 100/W$$

where:

A = millilitres of $AgNO_3$ solution required for the titration of the blank (13.2),
B = millilitres of $AgNO_3$ solution required for the titration of the NaCl standard (13.1.1),
C = millilitres of $AgNO_3$ solution required for the titration of the sample (13.3.2),
N = normality of the $AgNO_3$ solution (13.1),
W = grams of the analysis sample used, and
0.03545 = milliequivalent weight of chloride.

14. Volhard Titration (Method B)

14.1 *Standardization of Reagents:*

14.1.1 *Silver Nitrate, Standard Solution*—Add 20.0 mL of standard 0.025 *N* NaCl solution to a 250-mL Erlenmeyer flask. Add 1 mL of $K_2CrO_4 - K_2Cr_2O_7$ indicator. Dilute to about 100 mL with water. Titrate with $AgNO_3$ solution to a faint red-brown end point. Calculate the normality of the $AgNO_3$ solution as follows:

$$\text{Normality of } AgNO_3 = \text{mL of NaCl solution used} \times \text{normality of NaCl solution/mL of } AgNO_3 \text{ solution required}$$

14.1.2 *Potassium Thiocyanate, Standard Solution*—Add 20.0 mL of 0.025 *N* $AgNO_3$ solution to a 250-mL glass-stoppered Erlenmeyer flask. Add 10 mL of HNO_3 (1+1) and make up to approximately 100 mL with water. Add 5 to 10 mL of nitrobenzene, stopper, and shake for 1 min. Add 8 to 10 drops of $FeNH_4(SO_4)_2$ indicator solution and titrate with KCNS solution. The end point is reached when the solution becomes faintly orange-pink in color. Calculate the normality of the KCNS solution as follows:

$$\text{Normality of KCNS} = \text{mL of } AgNO_3 \text{ solution used} \times \text{normality of } AgNO_3 \text{ solution/mL of KCNS solution required}$$

14.2 *Blank Determination*—Prepare a solution for the blank determination in the same manner as the preparation of chlorine solutions for the respective type of chlorine desired as described in Sections 11 and 12, using all the reagents in the same quantities and eliminating the RDF sample. Proceed as described in 14.3.

14.3 *Procedure*—Transfer quantitatively the chloride solution (11.3, 12.1, or 12.2, respectively) for the type of chlorine desired into a 250-mL glass-stoppered Erlenmeyer flask. Add 10 mL of HNO_3 (1+1), stirring gently during the acid addition. Add 20.0 mL of standard $AgNO_3$ solution. Stopper, mix, and let stand in the dark for 15 min. Cool to room temperature. Add 5 to 10 mL of nitrobenzene and shake for 1 min. Add 8 to 10 drops of $FeNH_4(SO_4)_2$ solution and titrate with standard KCNS solution against a white background. The end point of the determination is reached when the solution becomes faintly orange-pink in color.

14.4 *Calculations*—Calculate the weight percent of chloride as follows:

$$\text{Chloride (chlorine), weight \%} = \frac{(B - A) \times N \times 0.03545 \times 100}{W}$$

where:

A = millilitres of KCNS solution required for the titration of the sample (14.3),
B = millilitres of KCNS solution required for the titration of the blank (14.2),
N = normality of the KCNS solution (14.1.2),
W = grams of the analysis sample used, and
0.03545 = milliequivalent weight of chloride.

15. Water-Insoluble Chlorine

15.1 Calculate the percent of water-insoluble chlorine as follows: percent of total chlorine minus percent of water-insoluble chloride.

16. Precision and Bias[11]

16.1 *Precision:*

16.1.1 The standard deviations of individual determinations, in percent absolute, are as follows:

Typical Average Value, %	Within-Laboratory, %	Between Laboratories, %
Total Chlorine:		
0.49	0.03	0.11
Water-Soluble Chlorides:		
0.37	0.02	0.05

16.1.2 These precision estimates are based on an interlaboratory study conducted in accordance with Practice E 180.

16.2 *Bias*—The bias of this test has not been determined because of the lack of a recognized standard reference material.

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[11] Supporting data are available on loan from ASTM Headquarters. Request RR: E38-1000.

CERTIFICATE

By Authority Of
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WASHINGTON, D.C.

Designation: E 885 – 88

Standard Test Methods for Analyses of Metals in Refuse-Derived Fuel by Atomic Absorption Spectroscopy[1]

This standard is issued under the fixed designation E 885; 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 These test methods cover the determination of metals in solution by atomic absorption spectroscopy (AAS).

1.2 The following sections outline the operating parameters for the individual metals:

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Silver, Furnace Technique	50
Sodium, Direct Aspiration	51
Tin, Direct Aspiration	52
Tin, Furnace Technique	53
Titanium, Direct Aspiration	54
Titanium, Furnace Technique	55
Vanadium, Direct Aspiration	56
Vanadium, Furnace Technique	57
Zinc, Direct Aspiration	58
Zinc, Furnace Technique	59

1.3 Detection limits, sensitivity, and optimum ranges of the test methods will vary with the various makes and models of atomic absorption spectrophotometers. The data shown in Table 1 provide some indication of the actual concentration ranges measurable by direct aspiration and using furnace techniques. In the majority of instances, the concentration range shown in the table by direct aspiration may be extended much lower with scale expansion and conversely extended upwards by using a less sensitive wavelength or by rotating the burner head. Detection limits by direct aspiration may also be extended through concentration of the sample or through solvent extraction techniques, or both. Lower concentrations may also be determined using the furnace techniques. The concentration ranges given in Table 1 are somewhat dependent on equipment such as the type of spectrophotometer and furnace accessory, the energy source, and the degree of electrical expansion of the output signal.

1.4 When using the furnace techniques, the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures that may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects (see 6.2) and if detected, treat accordingly using either successive dilution, matrix modification or method of standard additions (see 10.5).

1.5 Where direct aspiration atomic absorption techniques do not provide adequate sensitivity, in addition to the furnace procedure, reference is made to specialized procedures such as gaseous hydride method for arsenic and selenium, the cold-vapor technique for mercury and the chelation-extraction procedure for selected metals.

1.6 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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.* For hazard statement, see 8.4 and 17.2.3.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3223 Test Method for Total Mercury in Water[2]

E 926 Test Methods of Preparing Refuse-Derived Fuel (RDF) Samples for Analyses of Metals[3]

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[1] These test methods are under the jurisdiction of ASTM Committee D-34 on Waste Management and are the direct responsibility of Subcommittee D34.08 on Thermal Treatment.

Current edition approved March 25, 1988. Published May 1988. Originally published as E 885 – 82. Last previous edition E 885 – 82.

[2] *Annual Book of ASTM Standards,* Vol 11.01.

[3] *Annual Book of ASTM Standards,* Vol 11.04.

TABLE 1 Atomic Absorption Concentrations[A]

Metal	Direct Aspiration			Furnace Technique[B,C]	
	Detection Limit, mg/L	Sensitivity, mg/L	Optimum Concentration Range, mg/L	Detection Limit, µg/L	Optimum Concentration Range, µg/L
Aluminum	0.1	1	5 to 50	3	20 to 200
Antimony	0.2	0.5	1 to 40	3	20 to 300
Arsenic[D]	0.002	...	0.002 to 0.02	1	5 to 100
Barium (*P*)	0.1	0.4	1 to 20	2	10 to 200
Beryllium	0.005	0.025	0.05 to 2	0.2	1 to 30
Cadmium	0.005	0.025	0.05 to 2	0.1	0.5 to 10
Calcium	0.01	0.08	0.2 to 7	...	...
Chromium	0.05	0.25	0.5 to 10	1	5 to 100
Cobalt	0.05	0.2	0.5 to 5	1	5 to 100
Copper	0.02	0.1	0.2 to 5	1	5 to 100
Iron	0.03	0.12	0.3 to 5	1	5 to 100
Lead	0.1	0.5	1 to 20	1	5 to 100
Lithium	...	0.035	...	...	...
Magnesium	0.001	0.007	0.02 to 0.5	...	...
Manganese	0.01	0.05	0.1 to 3	0.2	1 to 30
Mercury[E]	0.0002	...	0.0002 to 0.01	...	...
Molybdenum (*P*)	0.1	0.4	1 to 40	1	3 to 60
Nickel (*P*)	0.04	0.15	0.3 to 5	1	5 to 100
Potassium	0.01	0.04	0.1 to 2	...	...
Selenium[D]	0.002	...	0.002 to 0.02	2	5 to 100
Silver	0.01	0.06	0.1 to 4	0.2	1 to 25
Sodium	0.002	0.015	0.03 to 1	...	...
Tin	0.8	4	10 to 300	5	20 to 300
Titanium (*P*)	0.4	2	5 to 100	10	50 to 500
Vanadium (*P*)	0.2	0.8	2 to 100	4	10 to 200
Zinc	0.005	0.02	0.05 to 1	0.05	0.2 to 4

[A] The concentrations shown are not contrived values and should be obtainable with any satisfactory atomic absorption spectrophotometer.

[B] For furnace sensitivity values consult instrument operating manual.

[C] The listed furnace values are those expected when using a 20 µL injection and normal gas flow except in the case of arsenic and selenium where gas interrupt is used. The symbol (*p*) indicates the use of pyrolytic graphite with the furnace procedure.

[D] Gaseous hydride method.

[E] Cold vapor technique.

3. Descriptions of Terms Specific to This Standard

3.1 *detection limit*—detection limits can be expressed as either an instrumental or method parameter. The limiting factor of the former using acid water standards would be the signal to noise ratio and degree of scale expansion used; while the latter would be more affected by the sample matrix and preparation procedure used.

3.1.1 The Scientific Apparatus Makers Association (SAMA) has approved the following definition: The detection limit is that concentration of an element which would yield an absorbance equal to twice the standard deviation of a series of measurements of a solution, the concentration of which is distinctly detectable above, but close to blank absorbance measurement.

3.1.2 The detection limit values listed in Table 1 and on individual metal methods are to be considered minimum working limits achievable with the procedures outlined in these test methods.

3.2 *optimum concentration range*—a range defined by limits expressed in concentration, below which scale expansion must be used and above which curve correction should be considered. The range will vary with the sensitivity of the instrument and the operating condition employed.

3.3 *sensitivity*—the concentration in milligrams of metal per litre that produces an absorption of 1 %.

4. Summary of Test Methods

4.1 In direct aspiration atomic absorption spectroscopy, a sample is aspirated and atomized in a flame. The light beam from a hollow cathode lamp whose cathode is made of the element to be determined is directed through the flame into a monochromator, and into a detector that measures the amount of light absorbed. Absorption depends upon the presence of free unexcited ground state atoms in the flame. Since the wavelength of the light beam is characteristic of only the metal being determined, the light energy absorbed by the flame is a measure of the concentration of that metal in the sample. This principle is the basis of atomic absorption spectroscopy.

4.2 Pretreatment of a solid sample is necessary for complete dissolution of the metals and complete breakdown of organic material prior to analysis (see Methods E 926). This process may vary because of the metals to be determined and the nature of the sample being analyzed.

4.3 When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of the sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. As a greater percentage of available atoms are vaporized and dissociated for absorption in the tube than the flame, the use of small sample volumes or detection of low concentrations of elements is possible. The principle is essentially the same as with direct aspiration atomic absorption except a furnace, rather than a flame, is used to atomize the sample. Radiation from a given excited element is passed through the vapor containing ground state atoms of that element. The intensity of the transmitted radiation decreases in proportion to the amount of the ground state element in the vapor. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the characteristic radiation from the hollow cathode lamp, and a photosensitive device measures the attenuated transmittal radiation.

5. Significance and Use

5.1 Metals in solution may be readily determined by atomic absorption spectroscopy (AAS). The method is simple, rapid, and applicable to a large number of metals in solution. Solid type samples may be analyzed after proper treatment.

6. Interferences

6.1 *Direct Aspiration:*

6.1.1 The most troublesome type of interference in atomic absorption spectrophotometry is usually termed "chemical" and is caused by lack of absorption of atoms bound in molecular combination to the flame. This phenomenon can occur when the flame is not sufficiently hot to dissociate the molecule, as in the case of phosphate interference with magnesium, or because the dissociated atom is immediately oxidized to a compound that will not dissociate further at the temperature of the flame. The addition of lanthanum will overcome the phosphate interference in the magnesium, calcium, and barium determinations. Similarly, silica interference in the determination of manganese can be eliminated by the addition of calcium.

6.1.2 Chemical interferences may also be eliminated by separating the metal from the interfering material. While complexing agents are primarily employed to increase the sensitivity of the analysis, they may also be used to eliminate or reduce interferences.

6.1.3 Highly dissolved solids in the sample being aspirated may result in an interference from nonatomic absorbance such as light scattering. If background correction is not available, a nonabsorbing wavelength should be checked. Preferably, high solid content solutions should be extracted (see 6.1.1 and 11.2).

6.1.4 Ionization interferences occur where the flame temperature is sufficiently high to generate the removal of an electron from a neutral atom, giving a positive charged ion. This type of interference can generally be controlled by the addition, to both standard and sample solutions, of a large excess of an easily ionized element.

6.1.5 Although quite rare, spectral interference can occur when an absorbing wavelength of an element present in the sample but not being determined falls within the width of the absorption line of the element of interest. The results of the determination will then be erroneously high, due to the contribution of the interfering element to the atomic absorption signal. Also, interference can occur when resonant energy from another element in a multi-element lamp or a metal impurity in the lamp cathode falls within the bandpass of the slit setting and that metal is present in the sample. This type of interference may sometimes be reduced by narrowing the slit width.

6.2 *Flameless Atomization:*

6.2.1 Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical and matrix interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects that must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, use the following procedure. Withdraw from the sample two equal aliquots. To one of the aliquots, add a known amount of analyte and dilute both aliquots to the same predetermined volume. (The dilution volume should be based on the analysis of the undiluted sample. Preferably, the dilution should be 1:4 while keeping in mind the optimum concentration range of the analysis. Under no circumstances should the dilution be less than 1:1). The diluted aliquots should then be analyzed and the unspiked results multiplied by the dilution factor should be compared to the original determination. Agreement of the results (within ± 10 %) indicates the absence of interference. Comparison of the actual signal from the spike to the expected response from the analyte in an aqueous standard should help confirm the finding from the dilution analysis. Those samples that indicate the presence of interference should be treated in one or more of the following ways.

6.2.1.1 The samples should be successively diluted and reanalyzed to determine if the interference can be eliminated.

6.2.1.2 The matrix of the sample should be modified in the furnace. Examples are the addition of ammonium nitrate to remove alkali chlorides, ammonium phosphate to retain cadmium, and nickel nitrate for arsenic and selenium analysis (**1**).[4] The mixing of hydrogen with the inert purge gas has also been used to suppress chemical interference. The hydrogen acts as a reducing agent and aids in molecular dissociation.

6.2.1.3 Analyze the sample by method of standard additions while noting the precautions and limitations of its use (see 10.5).

6.2.2 Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. When this occurs, either the use of background correction or choosing an alternate wavelength outside the absorption band should eliminate this interference. Background correction can also compensate for nonspecific broad band absorption interference.

6.2.3 Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or using an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analysis element.

6.2.4 Samples containing large amounts of organic materials should be oxidized by conventional acid digestion prior to being placed in the furnace. In this way, broad-band absorption will be minimized.

6.2.5 From anion-interference studies in the graphite furnace it is generally accepted that nitrate is the preferred anion. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to HNO_3 is required, a minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

6.2.6 Carbide formation resulting from the chemical environment of the furnace has been observed with certain

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[4] The boldface numbers in parentheses refer to the list of references at the end of these test methods.

elements that form carbides at high temperatures. Molybdenum may be cited as an example. When this takes place, the metal will be released very slowly from the carbide as atomization continues. For molybdenum, one may be required to atomize for 30 s or more before the signal returns to baseline levels. This problem is greatly reduced, and the sensitivity increased with the use of pyrolytically-coated graphite.

6.2.7 Ionization interferences have to date not been reported with furnace techniques.

6.2.8 For comments on spectral interference see 6.1.5.

6.2.9 Contamination of the sample can be a major source of error because of the extreme sensitivities achieved with the furnace. The sample-preparation work area should be kept scrupulously clean. All glassware should be cleaned as directed. Pipet tips have been known to be a source of contamination. If suspected, they should be acid soaked with 1:5 HNO_3 and rinsed thoroughly with tap and deionized water. The use of a better grade pipet tip can greatly reduce this problem. It is very important that special attention be given to reagent blanks in both analysis and the correction of analytical results. Lastly, pyrolytic graphite, because of the production process and handling, can become contaminated. As many as five to possibly ten high temperature burns may be required to clean the tube before use.

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer*—Single or dual channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range from 190 to 800 nm, and provisions for interfacing with a strip-chart recorder.

7.2 *Burner*—The burner recommended by the particular instrument manufacturer should be used. For certain elements the nitrous oxide burner is required.

7.3 *Hollow Cathode Lamps*—Single-element lamps are to be preferred but multi-element lamps may be used. Electrodeless discharge lamps may also be used when available.

7.4 *Graphite Furnace*—Any furnace device capable of reaching the specified temperatures is satisfactory.

7.5 *Strip Chart Recorder*—A recorder is strongly recommended for furnace work so that there will be a permanent record, and any problems with the analysis such as drift, incomplete atomization, losses during charring, changes in sensitivity, etc., can be easily recognized.

7.6 *Pipets*—Microliter with disposable tips. Sizes can range from 5 to 100 µL as required.

7.7 *Pressure-reducing Valves*—The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

7.8 *Separatory Flasks*—250 mL, or larger, for extraction with organic solvents.

7.9 *Glassware*—All glassware, linear polyethylene, polypropylene or Teflon containers, including sampling bottles, should be washed and rinsed in the following order: washed with detergent; rinsed with tap water, 1:1 nitric acid, tap water, 1:1 hydrochloride acid, tap water, and deionized distilled water.

7.10 *Borosilicate Glass Distillation Apparatus.*

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

8.3 *Deionized Distilled Water*—Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized distilled water for the preparation of all reagents, calibration standards, and as dilution water.

8.4 *Nitric Acid* (concentrated)—If metal impurities are found to be present, distill reagent grade nitric acid in a borosilicate glass distillation apparatus, or use a spectrograde acid.

NOTE 1: **Precaution**—Perform distillation in hood with protective sash in place.

8.4.1 *Nitric Acid* (1:1)—Prepare a 1:1 dilution with deionized, distilled water by adding the concentrated acid to an equal volume of water.

8.5 *Hydrochloric Acid* (1:1)—Prepare a 1:1 solution of reagent grade hydrochloric acid and deionized distilled water. If metal impurities are found to be present, distill this mixture from a borosilicate glass distillation apparatus or use a spectrograde acid.

8.6 *Stock Standard Metal Solutions*—Prepare as directed in 10.1 and under the individual metal procedures. Commercially available stock standard solutions may be used.

8.7 *Calibration Standards*—Prepare a series of standards of the metal by dilution of the appropriate stock metal solution to cover the concentration range desired.

8.8 *Fuel and Oxidant*—Commercial grade acetylene is generally acceptable. Air may be supplied from a compressed air line, a laboratory compressor, or from a cylinder of compressed air. Reagent grade nitrous oxide is also required for certain determinations. Standard, commercially available argon and nitrogen are required for furnace work.

8.9 *Special Reagents for the Extraction Procedure:*

8.9.1 *Pyrrolidine Dithiocarbamic Acid (PDCA)*[6]—Prepare by adding 18 mL of analytical reagent grade pyrrolidine to 500 mL of chloroform in a litre flask.[7] Cool and add 15 mL of carbon disulfide in small portions and with swirling. Dilute to 1 L with chloroform. The solution can be used for several months if stored in a brown bottle in a refrigerator.

8.9.2 *Ammonium Hydroxide, 2N*—Dilute 3 mL concen-

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[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[6] The name pyrrolidine dithiocarbamic acid (PDCA), although commonly referenced in the scientific literature is ambiguous. From the chemical reaction of pyrrolidine and carbon disulfide a more proper name would be 1-pyrrolidine carbodithioic acid, PCDA (CAS Registry No. 25769-03-3).

[7] An acceptable grade of pyrrolidine may be obtained from the Aldrich Chemical Co., 940 West St. Paul Ave., Milwaukee, WI 53233.

trated NH_4OH to 100 mL with deionized distilled water.

8.9.3 *Bromphenol Blue Indicator* (1 g/L)—Dissolve 0.1 g bromphenol blue in 100 mL of 500 % ethanol or isopropanol.

8.9.4 *HCL* (2.5 % v/v)—Dilute 2 mL redistilled HCl to 40 mL with deionized distilled water.

9. Sample Handling and Preservation

9.1 See Methods E 926 for sample handling and preservation procedures.

10. Preparation of Standards and Calibration

10.1 *Stock Standard Solutions,* are prepared from high purity metals, oxides, or nonhygroscopic reagent grade salts using deionized distilled water and redistilled nitric or hydrochloric acids. (See individual analysis sheets for specific instruction.) Sulfuric or phosphoric acids should be avoided as they produce an adverse effect on many elements. The stock solutions are prepared at concentrations of 1000 mg of the metal per litre. Commercially available standard solutions may also be used.

10.2 *Calibration Standards,* are prepared by diluting the stock metal solutions at the time of analysis. For best results, calibration standards should be prepared fresh each time an analysis is to be made and discarded after use. Prepare a blank and at least four calibration standards in graduated amounts in the appropriate range. The *calibration standards* should be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following processing. Beginning with the blank and working toward the highest standard, aspirate the solutions and record the readings. Repeat the operation with both the calibration standards and the samples a sufficient number of times to secure a reliable average reading for each solution. Calibration standards for furnace procedures should be prepared as described on the individual sheets for that metal.

10.3 Where the sample matrix is so complex that viscosity, surface tension, and components cannot be accurately matched with standards, the method of standard addition must be used. This technique relies on the addition of small, known amounts of the analysis element to portions of the sample, the absorbance difference between those, and the original solution giving the slope of the calibration curve. The method of standard addition is described in greater detail in 10.5.

10.4 For those instruments that do not read out directly in concentration, a calibration curve is prepared to cover the appropriate concentration range. Usually, this means the preparation of standards that produce an absorption of 0 to 80 %. The correct method is to convert the percent absorption readings to absorbance and plot that value against concentration. The following relationship is used to convert absorption values to absorbance:

$$\text{absorbance} = \log(100/\%\,T) = 2 - \log(\%\,T)$$

where:

$\%\,T = 100 - \%$ absorption

As the curves are frequently nonlinear, especially at high absorption values, the number of standards should be increased in that portion of the curve.

10.5 *Method of Standard Additions:*

10.5.1 In this test method, equal volumes of sample are added to a deionized distilled water blank and to three standards containing different known amounts of the test element. The volume of the blank and the standards must be the same. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Fig. 1.

10.5.2 The method of standard additions can be very useful. For the results to be valid, the following limitations must be taken into consideration:

10.5.2.1 The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20 %) caution should be exercised.

10.5.2.2 The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

10.5.2.3 The determination must be free of spectral interference and corrected for nonspecific background interference.

11. General Procedure for Analysis by Atomic Absorption

11.1 *Direct Aspiration*—Differences between the various makes and models of satisfactory atomic absorption spectrophotometers prevent the formulation of detailed instructions applicable to every instrument. The analyst should follow the manufacturer's operating instructions for his particular instrument. In general, after choosing the proper hollow cathode lamp for the analysis, allow the lamp to warm up for a minimum of 15 min unless operated in a double beam mode. During this period, align the instrument, position the monochromator at the correct wavelength, select the proper monochromator slit width, and adjust the hollow cathode current according to the manufacturer's recommendation. Subsequently, light the flame and regulate the flow of fuel and oxidant, adjust the burner and nebulizer flow rate for maximum percent absorption and stability, and balance the photometer. Run a series of standards of the element under analysis and construct a calibration curve by plotting the concentrations of the standards against the absorbance. For those instruments which read directly in concentration set the curve corrector to read out the proper concentration. Aspirate the samples and determine the concentrations either directly or from the calibration curve. Standards must be run each time a sample or series of samples are run.

11.1.1 *Calculation for Direct Determination of Liquid Samples*—Read the metal value in mg/L from the calibration curve or directly from the readout system of the instrument.

11.1.1.1 If dilution of sample was required:

$$\text{mg/L metal in sample} = A\left(\frac{C + B}{C}\right)$$

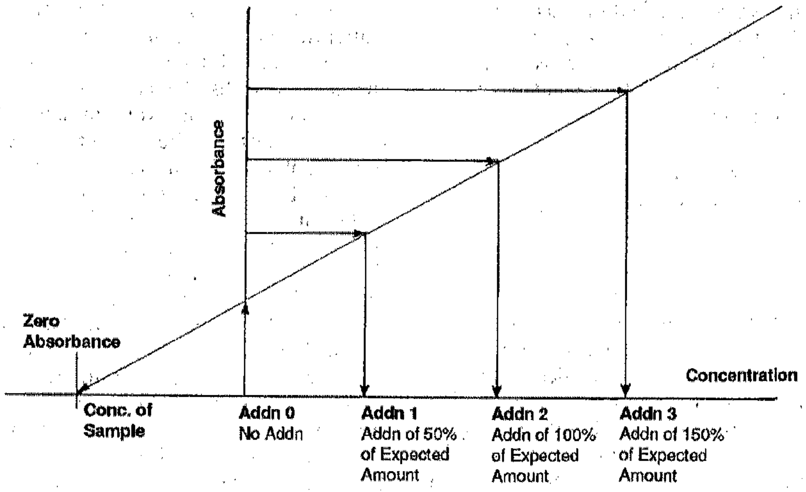

FIG. 1 Standard Addition Plot

where:
A = mg/L of metal in diluted aliquot from calibration curve,
B = mL of deionized distilled water used for dilution, and
C = mL of sample aliquot.

11.1.2 For solid samples: report all concentrations as mg/kg dry weight.

11.1.2.1 Dry sample:

$$\text{mg metal/kg sample} = \frac{A \times V}{D}$$

where:
A = mg/L of metal in processed sample from calibration curve,
V = final volume of the processed sample in mL, and
D = weight of dry sample in grams.

11.1.2.2 Wet sample:

$$\text{mg metal/kg sample} = \frac{A \times V}{W \times P}$$

where:
A = mg/L of metal in processed sample from calibration curve,
V = final volume of the processed sample in mL,
W = weight of wet sample in grams, and
P = percent solids.

11.2 *Special Extraction Procedure*—When the concentration of the metal is not sufficiently high to determine directly, or when considerable dissolved solids are present in the sample, certain metals may be chelated and extracted with organic solvents. Ammonium pyrrolidine dithiocarbamate (APDC)[8] in methyl isobutyl ketone (MIBK) is widely used for this purpose and is particularly useful for zinc, cadmium, iron, manganese, copper, silver, lead and chromium^{+6}. Trivalent chromium does not react with APDC unless it has first been converted to the hexavalent form (2). This procedure is described under method for chromium (chelation extraction). Aluminum, beryllium, barium and strontium also do not react with APDC. While the APDC-MIBK chelating-solvent system can be used satisfactorily, it is possible to experience difficulties.

NOTE 2—Certain metal chelates, manganese-APDC in particular, are not stable in MIBK and will redissolve into the aqueous phase on standing. The extraction of other metals is sensitive to both shaking rate and time. As with cadmium, prolonged extraction beyond 1 min, will reduce the extraction efficiency, whereas 3 min of vigorous shaking is required for chromium. Also, when multiple metals are to be determined either larger sample volumes must be extracted or individual extractions made for each metal being determined. The acid form of APDC-pyrrolidine dithiocarbamic acid prepared directly in chloroform as described by Lakanen has been found to be most advantageous (3). In this procedure the more dense chloroform layer allows for easy combination of multiple extractions which are carried out over a broader pH range favorable to multielement extraction. Pyrrolidine dithiocarbamic acid in chloroform is very stable and may be stored in a brown bottle in the refrigerator for months. Because chloroform is used as the solvent, it may not be aspirated into the flame. The procedure described in 11.2.1 is suggested.

11.2.1 *Extraction Procedure with Pyrrolidine Dithiocarbamic Acid (PDCA) in Chloroform:*

11.2.1.1 Transfer 200 mL of sample into a 250-mL separatory funnel, add 2 drops bromphenol blue indicator solution (8.9.3) and mix.

11.2.1.2 Prepare a blank and sufficient standards in the same manner and adjust the volume of each to approximately 200 mL with deionized distilled water. All of the metals to be determined may be combined into single solutions at the appropriate concentration levels.

11.2.1.3 Adjust the pH by addition of 2N NH_4OH solution (8.9.2) until a blue color persists. Add HCl (8.9.4) dropwise until the blue color just disappears; then add 2.0 mL HCl (8.9.4) in excess. The pH at this point should be 2.3. (The pH adjustment may be made with a pH meter instead of using indicator.)

11.2.1.4 Add 5 mL of PDCA-chloroform reagent (8.9.1) and shake vigorously for 2 min. Allow the phases to separate and drain the chloroform layer into a 100-mL beaker.

NOTE 3—If hexavalent chromium is to be extracted, the aqueous phase must be readjusted back to a pH of 2.3 after the addition of

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[8] The name ammonium pyrrolidine dithiocarbamate (APDC) is somewhat ambiguous and should more properly be called ammonium, 1-pyrollidine carbodithioate (APCD), CAS Registry No. 5108-96-3.

PDCA-chloroform and maintained at that pH throughout the extraction. For multielement extraction, the pH may be adjusted upward after the chromium has been extracted.

11.2.1.5 Add a second portion of 5 mL PDCA-chloroform reagent (8.7.1) and shake vigorously for 2 min. Allow the phases to separate and combine the chloroform phase with that obtained in 11.2.1.4.

11.2.1.6 Determine the pH of the aqueous phase and adjust to 4.5.

11.2.1.7 Repeat 11.2.1.4 again combining the solvent extracts.

11.2.1.8 Readjust the pH to 5.5, and extract a fourth time. Combine all extracts and evaporate to dryness on a steam bath.

11.2.1.9 Hold the beaker at a 45° angle, and slowly add 2 mL of concentrated distilled nitric acid, rotating the beaker to effect thorough contact of the acid with the residue.

11.2.1.10 Place the beaker on a low temperature hotplate or steam bath and evaporate just to dryness.

11.2.1.11 Add 2 mL of nitric acid (1;1) to the beaker and heat for 1 min. Cool, quantitatively transfer the solution to a 10-mL volumetric flask and bring to volume with distilled water. The sample is now ready for analysis.

11.2.2 Prepare a calibration curve by plotting absorbance versus the concentration of the metal standard (μg/L) in the 200-mL extracted standard solution. To calculate sample concentration read the metal value in μg/L from the calibration curve or directly from the readout system of the instrument. If dilution of the sample was required use the following equation:

$$\text{mg/L metal in sample} = Z\left(\frac{C+B}{C}\right)$$

where:

Z = μg/L of metal in diluted aliquot from calibration curve,

B = mL of deionized distilled water used for dilution, and

C = mL of sample aliquot.

11.3 *Furnace Procedure*—Furnace devices (flameless atomization) are a most useful means of extending detection limits. Because of differences between various makes and models of satisfactory instruments, no detailed operating instructions can be given for each instrument. Instead, the analyst should follow the instructions provided by the manufacturer of his particular instrument and use as a guide the temperature settings and other instrument conditions listed on the individual analysis sheets which are recommended for the Perkin-Elmer HGA-2100.[9] In addition, the following points may be helpful.

11.3.1 With flameless atomization, background correction becomes of high importance especially below 350 nm. This is because certain samples, when atomized, may absorb or scatter light from the hollow cathode lamp. It can be caused by the presence of gaseous molecular species, salt particles, or smoke in the sample beam. If no correction is made, sample absorbance will be greater than it should be, and the analytical result will be erroneously high.

11.3.2 If during atomization all the analyte is not volatilized and removed from the furnace, memory effects will occur. This condition is dependent on several factors such as the volatility of the element and its chemical form, whether pyrolytic graphite is used, the rate of atomization and furnace design. If this situation is detected through blank burns, the tube should be cleaned by operating the furnace at full power for the required time period as needed at regular intervals in the analytical scheme.

11.3.3 Some of the smaller size furnace devices, or newer furnaces equipped with feedback temperature control employing faster rates of atomization, can be operated using lower atomization temperatures for shorter time periods than those listed in this manual.[10]

11.3.4 Although prior digestion of the sample in many cases is not required providing a representative aliquot of sample can be pipeted into the furnace, it will provide for a more uniform matrix and possibly lessen matrix effects.

11.3.5 Inject a measured microlitre aliquot of sample into the furnace and atomize. If the concentration found is greater than the highest standard, the sample should be diluted in the same acid matrix and reanalyzed. The use of multiple injections can improve accuracy and help detect furnace pipetting errors.

11.3.6 To verify the absence of interference, follow the procedure as given in part 6.2.1.

11.3.7 A check standard should be run approximately after every 10 sample injections. Standards are run in part to monitor the life and performance of the graphite tube. Lack of reproducibility or significant change in the signal for the standard indicates that the tube should be replaced. Even though tube life depends on sample matrix and atomization temperature, a conservative estimate would be that a tube will last at least 50 firings. A pyrolytic-coating would extend that estimate by a factor of 3.

11.3.8 *Calculation*—For determination of metal concentration by the furnace: Read the metal value in μg/L from the calibration curve or directly from the readout system of the instrument.

11.3.8.1 If different size furnace injection volumes are used for samples rather than for standards, calculate as follows:

$$\text{μg/L of metal in sample} = Z\left(\frac{S}{U}\right)$$

where:

Z = μg/L of metal read from calibration curve or readout system,

S = μL volume standard injected into furnace for calibration curve, and

U = μL volume of sample injected for analysis.

11.3.8.2 If dilution of sample was required but sample injection volume was the same as for the following standard:

$$\text{μg/L of metal in sample} = Z\left(\frac{C+B}{C}\right)$$

where:

Z = μg/L metal in diluted aliquot from calibration curve,

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[9] The Perkin-Elmer HGA-2100 available from Perkin-Elmer Corp., Instruments Division, Main Ave., Norwalk, CT 06858 has been found suitable.

[10] Instrumentation Laboratories Model 555 available from Instrumentation Laboratory, Inc., Analytical Instrumentation Division, Jonspin Road, Wilmington, MA 01887; Perkin-Elmer Models HGA2200 and HGA7613; and Varian Model CRA-90 available from Varian Associates, Inc., 611 Hansen Way, Palo Alto, CA 94303 have been found suitable.

B = mL of deionized distilled water used for dilution, and
C = mL of sample aliquot.

11.3.9 For solid samples, report all concentrations as mg/kg dry weight.

11.3.9.1 *Dry sample:*

$$\text{mg metal/kg sample} = \frac{\left(\frac{Z}{1\,000}\right)V}{D}$$

where:
Z = µg/L of metal in processed sample from calibration curve (see 11.3.8.1),
V = final volume of processed sample in millilitres, and
D = weight of dry sample in grams.

11.3.10 *Wet sample:*

$$\text{mg metal/kg sample} = \frac{\left(\frac{Z}{1\,000}\right)V}{W \times P}$$

where:
Z = µg/L of metal in processed sample from calibration curve (see 11.3.8.1),
V = final volume of processed sample in millilitres,
W = weight of wet sample in grams, and
P = percent solids.

12. Aluminum—Direct Aspiration

12.1 *Requirements:*

12.1.1 *Optimum Concentration Range*, 5 to 50 mg/L using a wavelength of 309.3 nm (see Notes 4 and 5).

12.1.2 *Sensitivity*, 1 mg/L.

12.1.3 *Detection Limit*, 0.1 mg/L.

Note 4—The following lines may also be used:
308.2 nm Relative Sensitivity 1
396.2 nm Relative Sensitivity 2
394.4 nm Relative Sensitivity 2.5

Note 5—For concentrations of aluminum below 0.3 mg/L, the furnace procedure is recommended.

12.2 *Preparation of Standard Solution:*

12.2.1 *Stock Solution*—Carefully weigh 1.000 g of aluminum metal (analytical reagent grade). Add 15 mL of concentrated HCl to the metal, cover the beaker, and warm gently. When solution is complete, transfer quantitatively to a 1 L-volumetric flask and make up to volume with deionized distilled water. 1 mL = 1 mg Al (1000 mg/L).

12.2.2 *Potassium Chloride Solution*—Dissolve 95 g potassium chloride (KCl) in deionized distilled water and make up to 1 L.

12.2.3 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing. To each 100 mL of standard and sample alike add 2.0 mL potassium chloride solution.

12.3 *General Instrumental Parameters:*

12.3.1 *Aluminum Hollow Cathode Lamp.*

12.3.2 *Wavelength*—309.3 nm.

12.3.3 *Fuel*—Acetylene.

12.3.4 *Oxidant*—Nitrous oxide.

12.3.5 *Type of flame*—Fuel rich.

12.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

12.5 *Interferences*—Aluminum is partially ionized in the nitrous oxide-acetylene flame. This problem may be controlled by the addition of an alkali metal (potassium, 1000 µg/mL) to both sample and standard solutions.

13. Aluminum—Furnace Technique

13.1 *Requirements:*

13.1.1 *Optimum Concentration Range*, 20–200 µg/L (see Note 6).

13.1.2 *Detection Limit*, 3 µg/L.

Note 6—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-µL injection, continuous flow purge gas and nonpyrolytic graphite.

13.2 *Preparation of Standard Solution:*

13.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

13.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Also use these solutions for "standard additions."

13.2.3 Dilute the calibration standard to contain 0.5 % (v/v) HNO_3.

13.3 *General Instrument Parameters:*

13.3.1 *Drying Time and Temperature*—30 s at 125°C.

13.3.2 *Ashing Time and Temperature*—30 s at 1300°C.

13.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

13.3.4 *Purge Gas Atmosphere*—Argon.

13.3.5 *Wavelength*—309.3 nm.

13.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer (see Notes 7 and 8).

Note 7—Background correction may be required if the sample contains high dissolved solids.

Note 8—It has been reported that chloride ion and that nitrogen used as a purge gas suppress the aluminum signal. Therefore, the use of halide acids and nitrogen as a purge gas should be avoided.

13.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure" 11.3 (see Notes 9 and 10).

Note 9—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

Note 10—If the method of standard additions is required, follow the procedure given earlier in 10.5.

14. Antimony—Direct Aspiration

14.1 *Requirements:*

14.1.1 *Optimum Concentration Range*, 1 to 40 mg/L using a wavelength of 217.6 nm (see Note 11).

14.1.2 *Sensitivity*, 0.5 mg/L.

14.1.3 *Detection Limit*, 0.2 mg/L.

Note 11—For concentrations of antimony below 0.35 mg/L, the furnace procedure is recommended.

14.2 *Preparation of Standard Solution:*

14.2.1 *Stock Solution*—Carefully weigh 2.7426 g of antimony potassium tartrate (analytical reagent grade) and dissolve in deionized distilled water. Dilute to 1 L with deionized distilled water. 1 mL = 1 mg Sb (1000 mg/L).

14.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration*

standards should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

14.3 *General Instrumental Parameters:*

14.3.1 *Antimony Hollow Cathode Lamp.*

14.3.2 *Wavelength*—217.6 nm.

14.3.3 *Fuel*—Acetylene.

14.3.4 *Oxidant*—Air.

14.3.5 *Type of Flame*—Fuel Lean.

14.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

14.5 *Interferences:*

14.5.1 In the presence of lead (1000 mg/L), a spectral interference may occur at the 217.6-nm resonance line. In this case the 231.1-nm antimony line should be used.

14.5.2 Increasing acid concentrations decrease antimony absorption. To avoid this effect, the acid concentration in the samples and in the standards should be matched.

15. Antimony—Furnace Technique

15.1 *Requirements:*

15.1.1 *Optimum Concentration Range*, 20–300 µg/L (see Note 12).

15.1.2 *Detection Limit*, 3 µg/L.

NOTE 12—The concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-µL injection, continuous flow purge gas and non-pyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the recommended settings.

15.2 *Preparation of Standard Solution:*

15.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

15.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Also use these solutions for "standard additions."

15.2.3 Dilute the calibration standard to contain 0.2 % (v/v) HNO_3.

15.3 *General Instrument Parameters:*

15.3.1 *Drying Time and Temperature*—30 s at 125°C.

15.3.2 *Ashing Time and Temperature*—30 s at 800°C.

15.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

15.3.4 *Purge Gas Atmosphere*—Argon.

15.3.5 *Wavelength*—217.6 nm.

15.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer (see Notes 13 and 14).

NOTE 13—The use of background correction is recommended.

NOTE 14—Nitrogen may also be used as the purge gas.

15.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure" 11.3 (see Notes 15, 16, and 17).

NOTE 15—If chloride concentration presents a matrix problem or causes a loss previous to atomization, add an excess of 5 mg of ammonium nitrate to the furnace and ash using a ramp accessory or with incremental steps until the recommended ashing temperature is reached.

NOTE 16—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 17—If the method of standard additions is required, follow the procedure given in 10.5.

16. Arsenic—Furnace Technique

16.1 *Requirements:*

16.1.1 *Optimum Concentration Range*, 5–100 µg/L (see Note 18).

16.1.2 *Detection Limit*, 1 µg/L.

NOTE 18—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-µL injection, continuous flow purge gas and non-pyrolytic graphite. Smaller size furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

16.2 *Preparation of Standard Solution:*

16.2.1 *Stock Solution*—Dissolve 1.320 g of arsenic trioxide, As_2O_3 (analytical reagent grade) in 100 mL of deionized distilled water containing 4 g NaOH. Acidify the solution with 20 mL concentrated HNO_3 and dilute to 1 L. 1 mL = 1 mg As (1000 mg/L).

16.2.2 *Nickel Nitrate Solution*, 5 %—Dissolve 24.780 g of ACS reagent grade $Ni(NO_3)_2 \cdot 6H_2O$ in deionized distilled water and make up to 100 mL.

16.2.3 *Nickel Nitrate Solution*, 1 %—Dilute 20 mL of the 5 % nickel nitrate to 100 mL with deionized distilled water.

16.2.4 *Working Arsenic Solutions*—Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Withdraw appropriate aliquots of the stock solution, add 1 mL of concentrated HNO_3, 2 mL of 30 % H_2O_2 and 2 mL of the 5 % nickel nitrate solution. Dilute to 100 mL with deionized distilled water.

16.3 *Sample Preparation:*

16.3.1 Transfer 100 mL of well-mixed sample to a 250-mL Griffin beaker. Add 2 mL of 30 % H_2O_2 and sufficient concentrated HNO_3 to result in an acid concentration of 1 % (v/v). Heat for 1 h at 95°C or until the volume is slightly less than 50 mL.

16.3.2 Cool and bring back to 50 mL with deionized distilled water.

16.3.3 Pipet 5 mL of this digested solution into a 10 mL volumetric flask, add 1 mL of the 1 % nickel nitrate solution and dilute to 10-mL with deionized distilled water. The sample is now ready for injection into the furnace.

NOTE 19—If solubilization or digestion is not required, adjust the HNO_3 concentration of the sample to 1 % (v/v) and add 2 mL of 30 % H_2O_2 and 2 mL of 5 % nickel nitrate to each 100 mL of sample. The volume of the calibration standard should be adjusted with deionized distilled water to match the volume change of the sample.

16.4 *General Instrument Parameters:*

16.4.1 *Drying Time and Temperature*—30 s at 125°C.

16.4.2 *Ashing Time and Temperature*—30 s at 1100°C.

16.4.3 *Atomizing Time and Temperature*—10 s at 2700°C.

16.4.4 *Purge Gas Atmosphere*—Argon.

16.4.5 *Wavelength*—193.7 nm.

16.4.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 20—The use of background correction is recommended.

16.5 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure" 11.3.

NOTE 21—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 22—If the method of standard additions is required, follow the procedure given in 10.5.

17. Arsenic Gaseous Hydride Method

17.1 *Scope and Application:* The gaseous hydride method determines inorganic arsenic when present in concentrations at or about 2 μg/L. The method is applicable to drinking water and most fresh and saline waters in the absence of high concentrations of chromium, cobalt, copper, mercury, molybdenum, nickel, and silver.

17.2 *Summary of Test Method:*

17.2.1 Arsenic in the sample is first reduced to the trivalent form using $SnCl_2$ and converted to arsine, AsH_3, using zinc metal. The gaseous hydride is swept into an argon-hydrogen flame of an atomic absorption spectrophotometer. The working range of the method is 2 to 20 μg/L. The 193.7 nm wavelength is used.

17.2.2 Organic arsenic must be converted to inorganic compounds.

NOTE 23: **Precaution**—Arsine is a toxic gas. Precautions should be made to keep the system closed to the atmosphere.

17.3 Except for the perchloric acid step, the procedure to be used for this determination is found in Standard Methods for the Examination of Water and Wastewater **(4)**.

18. Barium—Direct Aspiration

18.1 *Requirements:*

18.1.1 *Optimum Concentration Range,* 1–20 mg/L using a wavelength of 553.6 nm (see Note 24).

18.1.2 *Sensitivity,* 0.4 mg/L.

18.1.3 *Detection Limit,* 0.1 mg/L.

NOTE 24—For concentrations of barium below 0.2 mg/L, the furnace procedure is recommended.

18.2 *Preparation of Standard Solution:*

18.2.1 *Stock Solution*—Dissolve 1.7787 g barium chloride ($BaCl_2 \cdot 2H_2O$, analytical reagent grade) in deionized distilled water and dilute to 1 L. 1 mL = 1 mg Ba (1000 mg/L).

18.2.2 *Potassium Chloride Solution*—Dissolve 95 g potassium chloride, KCl, in deionized distilled water and make up to 1 L.

18.2.3 Prepare dilutions of the stock barium solution to be used as calibration standards at the time of analysis. To each 100 mL of standard and sample alike, add 2.0 mL potassium chloride solution. The *calibration standards* should be prepared using the same type of acid and the same concentration as that of the sample being analyzed either directly or after processing.

18.3 *General Instrumental Parameters:*

18.3.1 *Barium hollow cathode lamp.*

18.3.2 *Wavelength*—553.6 nm.

18.3.3 *Fuel*—Acetylene.

18.3.4 *Oxidant*—Nitrous oxide.

18.3.5 *Type of Flame*—Fuel rich.

18.4 *Analysis of Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

18.5 *Interferences:*

18.5.1 The use of nitrous oxide-acetylene flame virtually eliminates chemical interference. However, barium is easily ionized in this flame, and potassium must be added (1000 mg/L) to standards and samples alike to control this effect.

18.5.2 If the nitrous oxide flame is not available and acetylene-air is used, phosphate, silicon and aluminum will severely depress the barium absorbance. This may be overcome by the addition of 2000 mg/L lanthanum.

19. Barium—Furnace Technique

19.1 *Requirements:*

19.1.1 *Optimum Concentration Range,* 10 to 200 μg/L (see Note 25).

19.1.2 *Detection Limit,* 2 μg/L.

NOTE 25—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-μL injection, continuous flow purge gas and pyrolytic graphite.

19.2 *Preparation of Standard Solution:*

19.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

19.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions."

19.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

NOTE 26—The use of halide acid should be avoided.

19.3 *General Instrument Parameters:*

19.3.1 *Drying Time and Temperature*—30 s at 125°C.

19.3.2 *Ashing Time and Temperature*—30 s at 1200°C.

19.3.3 *Atomizing Time and Temperature*—10 s at 2800°C.

19.3.4 *Purge Gas Atmosphere*—Argon.

NOTE 27—Because of possible chemical interaction, nitrogen should not be used as a purge gas.

19.3.5 *Wavelength*—553.6 nm.

19.3.6 *Other Operating Parameters,* should be set as specified by the particular instrument manufacturer.

19.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure" 11.3.

NOTE 28—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 29—If the method of standard additions is required, follow the procedure given in 10.5.

20. Beryllium—Direct Aspiration

20.1 *Requirements:*

20.1.1 *Optimum Concentration Range,* 0.05 to 2 mg/L using a wavelength of 234.9 nm (see Notes 30 and 31).

20.1.2 *Sensitivity,* 0.025 mg/L.

20.1.3 *Detection Limit,* 0.005 mg/L.

NOTE 30—The "aluminon colorimetric method" may also be used **(6)**. The minimum detectable concentration by this method is 5 μg/L.

NOTE 31—For concentrations of beryllium below 0.02 mg/L, the furnace procedure is recommended.

20.2 *Preparation of Standard Solution:*

20.2.1 *Stock solution*—Dissolve 11.6586 g beryllium sulfate, $BeSO_4$, in deionized distilled water containing 2 mL concentrated nitric acid and dilute to 1 L. 1 mL = 1 mg Be (1000 mg/L).

20.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration*

standards should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

20.3 *General Instrumental Parameters:*

20.3.1 *Beryllium Hollow Cathode Lamp.*

20.3.2 *Wavelength*—234.9 nm.

20.3.3 *Fuel*—Acetylene.

20.3.4 *Oxidant*—Nitrous oxide.

20.3.5 *Type of Flame*—Fuel-rich.

20.4 *Analysis Procedure:*

20.4.1 For analysis procedure and calculation, see "Direct Aspiration," 11.3.

20.5 *Interferences:*

20.5.1 Sodium and silicon at concentrations in excess of 1000 mg/L have been found to severely depress the beryllium absorbance.

20.5.2 Bicarbonate ion is reported to interfere; however, its effect is eliminated when samples are acidified to a pH of 1.5.

20.5.3 Aluminum at concentrations of 500 μg/L is reported to depress the sensitivity of beryllium **(5)**.

21. Beryllium—Furnace Technique

21.1 *Requirements:*

21.1.1 *Optimum Concentration Range*, 1 to 30 μg/L (see Note 32).

21.1.2 *Detection Limit*, 0.2 μg/L.

NOTE 32—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-μL injection, continuous flow purge gas and non-pyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

21.2 *Preparation of Standard Solution:*

21.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

21.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Also use these solutions for "standard additions."

21.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

21.3 *General Instrumental Parameters:*

21.3.1 *Drying Time and Temperature*—30 s at 125°C.

21.3.2 *Ashing Time and Temperature*—30 s at 1000°C.

21.3.3 *Atomizing Time and Temperature*—10 s at 2800°C.

21.3.4 *Purge Gas Atmosphere*—Argon.

21.3.5 *Wavelength*—234.9 nm.

21.3.6 The operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 33—The use of background correction is recommended.

NOTE 34—Because of possible chemical interaction and reported lower sensitivity, nitrogen should not be used as the purge gas.

21.4 *Analysis Procedure*—For the analysis procedure and the calculation see "Furnace Procedure," 11.3.

NOTE 35—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 36—If the method of standard additions is required, follow the procedure given in 10.5.

22. Cadmium—Direct Aspiration

22.1 *Requirements:*

22.1.1 *Optimum Concentration Range*, 0.05 to 2 mg/L using a wavelength of 228.8 nm (see Note 37).

22.1.2 *Sensitivity*, 0.025 mg/L.

22.1.3 *Detection Limit*, 0.005 mg/L.

NOTE 37—For levels of cadmium below 20 μg/L, either the Special Extraction Procedure given in 11.2 or the furnace technique is recommended.

22.2 *Preparation of Standard Solution:*

22.2.1 *Stock Solution*—Carefully weigh 2.282 g of cadmium sulfate ($3CdSO_4 \cdot 8H_2O$, analytical reagent grade) and dissolve in deionized distilled water. 1 mL = 1 mg Cd (1000 mg/L).

22.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

22.3 *General Instrumental Parameters:*

22.3.1 *Cadmium Hollow Cathode Lamp.*

22.3.2 *Wavelength*—228.8 nm.

22.3.3 *Fuel*—Acetylene.

22.3.4 *Oxidant*—Air.

22.3.5 *Type of Flame*—Oxidizing.

22.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

23. Cadmium—Furnace Technique

23.1 *Requirements:*

23.1.1 *Optimum Concentration Range*, 0.5 to 10 μg/L (see Note 38).

23.1.2 *Detection Limit*, 0.1 μg/L.

NOTE 38—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-μL injection, continuous flow purge gas and non-pyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

23.2 *Preparation of Standard Solution:*

23.2.1 *Stock Solution*: Prepare as described under "direct aspiration method."

23.2.2 *Ammonium Phosphate Solution* (40 %)—Dissolve 40 g of ammonium phosphate, $(NH_4)2HPO_4$ (analytical reagent grade) in deionized distilled water and dilute to 100 mL.

23.2.3 Prepare dilutions of the stock cadmium solution to be used as calibration standards at the time of analysis. To each 100 mL of standard and sample alike add 2.0 mL of the ammonium phosphate solution. The calibration standards should be prepared to contain 0.5 % (v/v) HNO_3.

23.3 *General Instrument Parameters:*

23.3.1 *Drying Time and Temperature*—30 s at 125°C.

23.3.2 *Ashing Time and Temperature*—30 s at 500°C.

23.3.3 *Atomizing Time and Temperature*—10 s at 1900°C.

23.3.4 *Purge Gas Atmosphere*—Argon.

23.3.5 *Wavelength*—228.8 nm.

23.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 39—The use of background correction is recommended.

23.4 *Analysis Procedure*—For the analysis procedure and the calculation see "Furnace Procedure," 11.3.

NOTE 40—Contamination from the work area is critical in cadmium analysis. Use of pipet tips which are free of cadmium is of particular importance.

NOTE 41—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 42—If the method of standard additions is required, follow the procedure given in 10.5.

24. Cadmium—Direct Aspiration

24.1 *Requirements:*

24.1.1 *Optimum Concentration Range,* 0.2 to 7 mg/L using a wavelength of 422.7 nm (see Notes 43 and 44).

24.1.2 *Sensitivity,* 0.08 mg/L.

24.1.3 *Detection Limit,* 0.01 mg/L.

NOTE 43—Phosphate, sulfate and aluminum interfere but are masked by the addition of lanthanum. Since low calcium values result if the pH of the sample is above 7, both standards and samples are prepared in dilute hydrochloric acid solution. Concentrations of magnesium greater than 1000 mg/L also cause low calcium values. Concentrations of up to 500 mg/L each of sodium, potassium and nitrate cause no interference.

NOTE 44—The 239.9 nm line may also be used. This line has a relative sensitivity of 120.

24.2 *Preparation of Standard Solution:*

24.2.1 *Stock Solution*—Suspend 1.250 g of $CaCO_3$ (analytical reagent grade) dried at 180°C for 1 h before weighing, in deionized distilled water, and dissolve cautiously with a minimum of dilute HCl. Dilute to 1000 mL with deionized distilled water. 1 mL = 0.5 mg Ca (500 mg/L).

24.2.2 *Lanthanum Chloride Solution*—Dissolve 29 g of La_2O_3, slowly and in small portions, in 250 mL concentrated HCl (**Caution**—Reaction is violent). Dilute to 500 mL with deionized distilled water.

24.2.3 Prepare dilutions of the stock calcium solutions to be used as calibration standards at the time of analysis. To each 10 mL volume of calibration standard and sample alike, add 1.0 mL of the lanthanum chloride solution, that is, 20 mL of standard or sample + 2 mL $LaCl_3$ = 22 mL.

24.3 *General Instrumental Parameters:*

24.3.1 *Cadmium Hollow Cathode Lamp.*

24.3.2 *Wavelength*—422.7 nm.

24.3.3 *Fuel*—Acetylene.

24.3.4 *Oxidant*—Air.

24.3.5 *Type of Flame*—Reducing.

24.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

NOTE 45—Anionic chemical interferences can be expected if lanthanum is not used in samples and standards.

NOTE 46—The nitrous oxide-acetylene flame will provide two to five times greater sensitivity and freedom from chemical interferences. Ionization interferences should be controlled by adding a large amount of alkali to the sample and standards. The analysis appears to be free from chemical suppressions in the nitrous oxide-acetylene flame (7).

25. Chromium—Direct Aspiration

25.1 *Requirements:*

25.1.1 *Optimum Concentration Range:* 0.5 to 10 mg/L using a wavelength of 357.9 nm (see Notes 47 and 48).

25.1.2 *Sensitivity*—0.25 mg/L.

25.1.3 *Detection Limit*—0.05 mg/L.

NOTE 47—The following wavelengths may also be used:

359.3 nm Relative Sensitivity 1.4,
425.4 nm Relative Sensitivity 2,
427.5 nm Relative Sensitivity 3, and
428.9 nm Relative Sensitivity 4.

NOTE 48—For levels of chromium between 50 and 200 µg/L, where the air-acetylene flame cannot be used or for levels below 50 µg/L, either the furnace procedure or the extraction procedure is recommended.

25.2 *Preparation of Standard Solution:*

25.2.1 *Stock Solution:* Dissolve 1.923 g of chromium trioxide (CrO_3, reagent grade) in deionized distilled water. When solution is complete, acidify with redistilled HNO_3 and dilute to 1 L with deionized distilled water. 1 mL = 1 mg Cr (1000 mg/L).

25.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

25.3 *General Instrumental Parameters:*

25.3.1 *Chromium Hollow Cathode Lamp.*

25.3.2 *Wavelength*—357.9 nm.

25.3.3 *Fuel*—Acetylene.

NOTE 49—The fuel-rich air-acetylene flame provides greater sensitivity but is subject to chemical and matrix interference from iron, nickel, and other metals. If the analysis is performed in a lean flame the interference can be lessened but the sensitivity will also be reduced.

NOTE 50—The suppression of both Cr (III) and Cr (VI) absorption by most interfering ions in fuel rich air-acetylene flames is reportedly controlled by the addition of 1 % ammonium bifluoride in 0.2 % sodium sulfate (8). A 1 % oxine solution is also reported to be useful.

25.3.4 *Oxidant*—Nitrous oxide.

25.3.5 *Type of Flame*—Fuel rich.

25.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

26. Chromium—Furnace Technique

26.1 *Requirements:*

26.1.1 *Optimum Concentration Range,* 5 to 100 µg/L (see Note 51).

26.1.2 *Detection Limit,* 1 µg/L.

NOTE 51—The concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of 20 µL injection, continuous flow purge gas and non-pyrolytic graphite.

26.2 *Preparation of Standard Solution:*

26.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

26.2.2 *Calcium Nitrate Solution*—Dissolve 11.8 g of calcium nitrate, $Ca(NO_3)_2 \cdot 4H_2O$ (analytical reagent grade) in deionized distilled water and dilute to 100 mL. 1 mL = 20 mg Ca.

26.2.3 Prepare dilutions of the stock chromium solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared to contain 0.5 % (v/v) HNO_3. To each 100 mL of standard and sample alike, add 1 mL of 30 % H_2O_2 and 1 mL of the calcium nitrate solution.

NOTE 52—Hydrogen peroxide is added to the acidified solution to convert all chromium to the trivalent state. Calcium is added to a level above 200 mg/L where its suppressive effect becomes constant up to 1000 mg/L.

26.3 *General Instrument Parameters:*

26.3.1 *Drying Time and Temperature*—30 s at 125°C.

26.3.2 *Ashing Time and Temperature*—30 s at 1000°C.

26.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

26.3.4 *Purge Gas Atmosphere*—Argon.

26.3.5 *Wavelength*—357.9 nm.

26.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 53—Background correction may be required if the sample contains high dissolved solids.

NOTE 54—Nitrogen should not be used as a purge gas because of possible CN band interference.

26.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure" 11.3.

NOTE 55—Pipet tips have been reported to be a possible source of contamination.

NOTE 56—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 57—If the method of standard additions is required, follow the procedure given in 10.5.

27. Chromium—Chelation-Extraction

27.1 *Scope*—This test method may be used to analyze samples containing from 1.0 to 25 µg of chromium per litre of solution.

27.2 *Summary of the Test Method:*

27.2.1 This test method is based on the chelation of hexavalent chromium with ammonium pyrrolidine dithiocarbamate (APDC) following oxidation of trivalent chromium. The chelate is extracted with methyl isobutyl ketone (MIBK) and aspirated into the flame of the atomic absorption spectrophotometer.

27.2.2 Hexavalent chromium may also be chelated with pyrrolidine dithiocarbamic acid in chloroform as described in 11.2.

27.3 *Interferences*—High concentrations of other reactive metals, as may be found in wastewaters, may interfere. The method is free from interferences from elements normally occurring in fresh water.

27.4 *General Instrumental Parameters:*

27.4.1 *Chromium Hollow Cathode Lamp.*

27.4.2 *Wavelength*—357.9 nm.

27.4.3 *Fuel*—Acetylene.

27.4.4 *Oxidant*—Air.

27.4.5 *Type of Flame*—Fuel rich (adjust for organic solvent).

27.5 *Reagents:*

27.5.1 *Ammonium Pyrrolidine Dithiocarbamate* (APDC) *Solution*—Dissolve 1.0 g APDC in dimineralized water and dilute to 100 mL. Prepare fresh daily.

27.5.2 *Bromphenol Blue Indicator Solution*—Dissolve 0.1 g bromphenol blue in 100 mL 50 % ethanol.

27.5.3 *Potassium Dichromate Standard Solution* (1.0 mL = 0.08 mg Cr)—Dissolve 0.2263 g dried analytical reagent grade $K_2Cr_2O_7$ in demineralized water, and make up to 1000 mL.

27.5.4 *Trivalent Chromium Stock Solution* (1.0 mL = 0.002 mg $Cr^{+}3$)—Pipet 5.00 mL of the potassium dichromate standard solution (5.3) into an Erlenmeyer flask. Add approximately 15 mg Na_2SO_3 and 0.5 mL concentrated HNO_3. Gently evaporate to dryness; strong heating reoxidizes the chromium. Add 0.5 mL concentrated HNO_3 and again evaporate to dryness to destroy any excess sulfite. Take up in 1 mL concentrated HNO_3 with warming and dilute to 200.00 mL with demineralized water.

27.5.5 *Trivalent Chromium Working Solution* (1.0 mL = 0.005 mg Cr^{+3})—Immediately before use, dilute 25.0 mL of trivalent chromium stock solution (27.5.4) to 100.0 mL with demineralized water.

27.5.6 *Potassium Permanganate* (0.1 N)—Dissolve 0.32 g potassium permanganate in 100 mL demineralized water.

27.5.7 *Sodium Azide* (0.1 %)—Dissolve 100 mg sodium azide in demineralized water and dilute to 100 mL.

27.5.8 *Methyl Isobutyl Ketone* (MIBK).

27.5.9 *Sodium Hydroxide Solution* (1 M)—Dissolve 40 g NaOH in demineralized water and dilute to 1 L.

27.5.10 *Sulfuric Acid* (0.12 M)—Slowly add 6.5 mL concentrated H_2SO_4 (sp gr 1.84) to demineralized water and dilute to 1 L.

27.6 *Procedure:*

27.6.1 Pipet a volume of sample containing less than 2.5 µg chromium (100 mL maximum) into a 200 mL volumetric flask, and adjust the volume to approximately 100 mL. The pH must be 2.0 or less. Add concentrated HNO_3 if necessary.

27.6.2 Acidify a litre of demineralized water with 1.5 mL concentrated HNO_3. Prepare a blank and sufficient standards using trivalent chromium, and adjust volumes to approximately 100 mL with the acidified demineralized water.

27.6.3 Add 0.1 N $KMnO_4$ dropwise to both standards and samples until a faint pink color persists.

27.6.4 Heat on a steam bath for 20 min. If the color disappears, add additional $KMnO_4$ solution dropwise to maintain a slight excess.

27.6.5 While still on the steam bath, add sodium azide solution dropwise until the $KMnO_4$ color just disappears. Heat for about 2 min between each addition and avoid adding any excess. Continue heating for 5 min after adding the last drop of sodium azide solution.

27.6.6 Transfer the flasks to a water bath and cool to room temperature.

27.6.7 Remove from the water bath and filter (through Whatman No. 40 filter paper or equivalent) any sample that has a brownish precipitate or coloration which may interfere with the pH adjustment.

27.6.8 Add 2.0 mL of 1 M NaOH and 2 drops bromphenol blue indicator solution. Continue the addition of 1 M NaOH dropwise to all samples and standards in which the indicator change from yellow to blue has not occurred. Add 0.12 M H_2SO_4 dropwise until the blue color just disappears, then add 2.0 mL in excess. The pH at this point will be 2.4.

27.6.9 The pH adjustment to 2.4 may also be made with a pH meter instead of using an indicator.

27.6.10 Add 5.0 mL APDC solution and mix. The pH should then be approximately 2.8.

27.6.11 Add 10.0 mL MIBK and shake vigorously for 3 min.

27.6.12 Allow the layers to separate and add demineralized water until the ketone layer is completely in the neck of the flask.

27.6.13 Aspirate the ketone layer, record the instrument reading for each sample and standard against the blank. Repeat, and average the duplicate results.

27.7 *Calculations:*

27.7.1 Determine the µg/L Cr in each sample from a plot of the instrument readings of standards. A working curve must be prepared with each set of samples. Report Cr concentrations as follows:

27.7.1.1 Less than 10 µg/L, nearest µg/L, and

27.7.1.2 10 µg/L and above, two significant figures.

27.7.2 Calculate the mg metal per kg of samples as outlined in 11.2.

28. Chromium, Hexavalent—Chelation-Extraction

28.1 *Scope:*

28.1.1 This test method may be used to analyze samples containing from 1.0 to 25 µg of chromium per litre of solution.

28.2 *Summary of Test Method:*

28.2.1 This method is based on the chelation of hexavalent chromium with ammonium pyrrolidine dithiocarbamate (APDC) and extraction with methyl isobutyl ketone (MIBK). The extract is aspirated into the flame of the atomic absorption spectrophotometer.

28.2.2 Hexavalent chromium may also be chelated with pyrrolidine dithiocarbamic acid in chloroform as described in 11.2. A pH of 2.3 must be maintained throughout the extraction.

28.2.3 The diphenylcarbazide colorimetric procedure as found in "Standard Methods for the Examination of Water and Wastewater" may also be used (9).

28.3 *Sample Handling and Preservation:*

28.3.1 Stability of hexavalent chromium is not completely understood at this time. Therefore, the chelation and extraction should be carried out as soon as possible.

28.3.2 To retard the chemical activity of hexavalent chromium, the sample should be transported and stored until time of analysis at 4°C.

28.4 *Interferences:*

28.4.1 High concentrations of other reactive metals, as may be found in wastewaters, may interfere. The method is free from interferences from elements normally occurring in fresh water.

28.5 *General Instrumental Parameters:*

28.5.1 *Chromium hollow cathode lamp.*

28.5.2 *Wavelength*—357.9 nm.

28.5.3 *Fuel*—Acetylene.

28.5.4 *Oxidant*—Air.

28.5.5 *Type of Flame:* Fuel-rich (adjust for organic solvent).

28.6 *Reagents:*

28.6.1 *Ammonium Pyrrolidine Dithiocarbamate (APDC) Solution*—Dissolve 1.0 g APDC in demineralized water and dilute to 100 mL. Prepare fresh daily.

28.6.2 *Bromphenol Blue Indicator Solution*—Dissolve 0.1 g bromphenol blue in 100 mL 50 % ethanol.

28.6.3 *Chromium Standard Solution I* (1.0 mL = 100 µg Cr)—Dissolve 0.2829 g pure, dried $K_2Cr_2O_7$ in demineralized water and dilute to 1000 mL.

28.6.4 *Chromium Standard Solution II* (1.0 mL = 10.0 µg Cr)—Dilute 100 mL chromium standard solution I to 1000 mL with demineralized water.

28.6.5 *Chromium Standard Solution III* (1.0 mL = 0.10 µg Cr)—Dilute 10.0 mL chromium standard solution II to 1000 mL with demineralized water.

28.6.6 *Methyl Isobutyl Ketone* (MIBK).

28.6.7 *Sodium Hydroxide Solution* (1 M)—Dissolve 40 g NaOH in demineralized water and dilute to 1 L.

28.6.8 *Sulfuric Acid* (0.12 M)—Slowly add 6.5 mL concentrated H_2SO_4 (sp gr 1.84) to demineralized water and dilute to 1 L.

28.7 *Procedure:*

28.7.1 Pipet a volume of sample containing less than 2.5 µg chromium (100 mL maximum) into a 200-mL volumetric flask, and adjust the volume to approximately 100 mL.

28.7.2 Prepare a blank and sufficient standards, and adjust the volume of each to approximately 100 mL.

28.7.3 Add 2 drops bromphenol blue indicator solution. (The pH adjustment to 2.4 may also be made with a pH meter instead of using an indicator.)

28.7.4 Adjust the pH by addition of 1 M NaOH solution dropwise until a blue color persists. Add 0.12 M H_2SO_4 dropwise until the blue color just disappears in both the standards and sample. Then add 2.0 mL of 0.12 M H_2SO_4 in excess. The pH at this point should be 2.4.

28.7.5 Add 5.0 mL APDC solution and mix. The pH should then be approximately 2.8.

28.7.6 Add 10.0 mL MIBK and shake vigorously for 3 min.

28.7.7 Allow the layers to separate and add demineralized water until the ketone layer is completely in the neck of the flask.

28.7.8 Aspirate the ketone layer, and record the scale reading for each sample and standard against the blank. Repeat and average the duplicate results.

28.8 *Calculations:*

28.8.1 Determine the µg/L Cr^{+6} in each sample from a plot of scale readings of standards. A working curve must be prepared with each set of samples. Report Cr^{+6} concentrations as follows: Less than 10 µg/L, nearest µg/L; 10 µg/L and above, two significant figures.

28.8.2 Calculate the mg metal per kg of sample as outlined in 11.2.

29. Cobalt—Direct Aspiration

29.1 *Requirements:*

29.1.1 *Optimum Concentration Range*—0.5 to 5 mg/L using a wavelength of 240.7 nm (see Note 58).

29.1.2 *Sensitivity*—0.2 mg/L.

29.1.3 *Detection Limit*—0.05 mg/L.

NOTE 58—For levels of cobalt below 100 µg/L, either the special extraction procedure (11.2), or the furnace technique is recommended.

29.2 *Preparation of Standard Solution:*

29.2.1 *Stock Solution*—Dissolve 4.307 g of cobaltous chloride, $CoCl_2 \cdot 6H_2O$ (analytical reagent grade), in deionized distilled water. Add 10 mL of concentrated nitric acid and dilute to 1 L with deionized distilled water. 1 mL = 1 mg Co (1000 mg/L).

29.2.2 Prepare dilutions of the stock cobalt solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

29.3 *General Instrumental Parameters:*

29.3.1 *Cobalt Hollow Cathode Lamp.*

29.3.2 *Wavelength*—240.7 nm.

29.3.3 *Fuel*—Acetylene.

29.3.4 *Oxidant*—Air.

29.3.5 *Type of Flame*—Oxidizing.

29.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

30. Cobalt—Furnace Technique

30.1 *Requirements:*

30.1.1 *Optimum Concentration Range,* 5–100 µg/L (see Note 59).

30.1.2 *Detection Limit,* 1 µg/L.

NOTE 59—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 µL injection, continuous flow purge gas and nonpyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

30.2 *Preparation of Standard Solution:*

30.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

30.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions."

30.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

30.3 *General Instrument Parameters:*

30.3.1 *Drying Time and Temperature*—30 s at 125°C.

30.3.2 *Ashing Time and Temperature*—30 s at 900°C.

30.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

30.3.4 *Purge Gas Atmosphere*—Argon.

30.3.5 *Wavelength*—240.7 nm.

30.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 60—The use of background correction is recommended.

NOTE 61—Nitrogen may also be used as the purge gas but with reported lower sensitivity.

30.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure" 11.3.

NOTE 62—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 63—If the method of standard additions is required, follow the procedure given earlier in 10.5.

31. Copper—Direct Aspiration

31.1 *Requirements:*

31.1.1 *Optimum Concentration Range,* 0.2–5 mg/L using a wavelength of 324.7 nm (see Notes 64 and 65).

31.1.2 *Sensitivity,* 0.1 mg/L.

31.1.3 *Detection Limit,* 0.02 mg/L.

NOTE 64—For levels of copper below 50 µg/L, either the Special Extraction Procedure, given in 11.2 or the furnace technique is recommended.

NOTE 65—Numerous absorption lines are available for the determination of copper. By selecting a suitable absorption wavelength, copper samples may be analyzed over a very wide range of concentration. The following lines may be used:

327.4 nm Relative Sensitivity 2,
216.5 nm Relative Sensitivity 7, and
222.5 nm Relative Sensitivity 20.

31.2 *Preparation of Standard Solution:*

31.2.1 *Stock Solution*—Carefully weigh 1.00 g of electrolyte copper (analytical reagent grade). Dissolve in 5 mL redistilled HNO_3, and make up to 1 L with deionized distilled water. Final concentration is 1 mg Cu per mL (1000 mg/L).

31.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

31.3 *General Instrumental Parameters:*

31.3.1 *Copper Hollow Cathode Lamp.*

31.3.2 *Wavelength*—324.7 nm.

31.3.3 *Fuel*—Acetylene.

31.3.4 *Oxidant*—Air.

31.3.5 *Type of Flame*—Oxidizing.

31.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration", 11.1.

32. Copper—Furnace Technique

32.1 *Requirements:*

32.1.1 *Optimum Concentration Range,* 5 to 100 µg/L (see Note 66).

32.1.2 *Detection Limit,* 1 µg/L.

NOTE 66—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-µL injection, continuous flow purge gas and non-pyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

32.2 *Preparation of Standard Solution:*

32.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

32.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions."

32.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

32.3 *General Instrument Parameters:*

32.3.1 *Drying Time and Temperature*—30 s at 125°C.

32.3.2 *Ashing Time and Temperature*—30 s at 900°C.

32.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

32.3.4 *Purge Gas Atmosphere*—Argon.

32.3.5 *Wavelength*—324.7 nm.

32.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 67—Background correction may be required if the sample contains high dissolved solids.

NOTE 68—Nitrogen may also be used as the purge gas.

32.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure" 11.3.

NOTE 69—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 70—If the method of standard additions is required, follow the procedure given in 10.5.

33. Iron—Direct Aspiration

33.1 *Requirements:*

33.1.1 *Optimum Concentration Range,* 0.3 to 5 mg/L using a wavelength of 248.3 nm (see Notes 71 and 72).

NOTE 71—The following lines may also be used:

248.8 nm relative sensitivity 2,
271.9 nm relative sensitivity 4,
302.1 nm relative sensitivity 5,
252.7 nm relative sensitivity 6, and
372.0 nm relative sensitivity 10.

NOTE 72—For concentrations of iron below 0.05 mg/L, either the Special Extraction Procedure given in 11.2 or the furnace procedure, is recommended.

33.1.2 *Sensitivity*, 0.12 mg/L.

33.1.3 *Detection Limit*, 0.03 mg/L.

33.2 *Preparation of Standard Solution:*

33.2.1 *Stock Solution*—Carefully weigh 1.000 g of pure iron wire (analytical reagent grade) and dissolve in 5 mL redistilled HNO_3, warming if necessary. When solution is complete, make up to 1 L with deionized distilled water. 1 mL = 1 mg Fe (1000 mg/L).

33.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as will result in the sample to be analyzed either directly or after processing.

33.3 *General Instrumental Parameters:*

33.3.1 *Iron Hollow Cathode Lamp.*

33.3.2 *Wavelength*—248.3 nm.

33.3.3 *Fuel*—Acetylene.

33.3.4 *Oxidant*—Air.

33.3.5 *Type of Flame*—Oxidizing.

33.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

34. Iron—Furnace Technique

34.1 *Requirements:*

34.1.1 *Optimum Concentration Range*—5 to 100 μg/L (see Note 73).

34.1.2 *Detection Limit*—1 μg/L.

NOTE 73—The concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 μL-injection, continuous flow purge gas, and nonpyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the recommended settings.

34.2 *Preparation of Standard Solution:*

34.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

34.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions."

34.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

34.3 *General Instrument Parameters:*

34.3.1 *Drying Time and Temperature*—30 s at 125°C.

34.3.2 *Ashing Time and Temperature*—30 s at 1000°C.

34.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

34.3.4 *Purge Gas Atmosphere*—Argon.

34.3.5 *Wavelength*—248.3 nm.

34.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 74—The use of background correction is recommended.

NOTE 75—Nitrogen may also be used as the purge gas.

34.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure," 11.3.

NOTE 76—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 77—If the method of standard additions is required, follow the procedure given in 10.5.

35. Lead—Direct Aspiration

35.1 *Requirements:*

35.1.1 *Optimum Concentration Range*, 1–20 mg/L using a wavelength of 283.3 nm (see Notes 78 and 79).

35.1.2 *Sensitivity*, 0.5 mg/L.

35.1.3 *Detection Limit*, 0.1 mg/L.

NOTE 78—For levels of lead below 200 μg/L, either the Special Extraction Procedure given in 11.2 or the furnace technique is recommended.

NOTE 79—The following lines may also be used:
217.0 nm Relative Sensitivity 0.4, and
261.4 nm Relative Sensitivity 10.

35.2 *Preparation of Standard Solution:*

35.2.1 *Stock Solution*—Carefully weigh 1.599 g of lead nitrate, $Pb(NO_3)_2$ (analytical reagent grade), and dissolve in deionized distilled water. When solution is complete, acidify with 10 mL redistilled HNO_3 and dilute to 1 L with deionized distilled water. 1 mL = 1 mg Pb (1000 mg/L).

35.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

35.3 *General Instrumental Parameters:*

35.3.1 *Iron Hollow Cathode Lamp.*

35.3.2 *Wavelength*—283.3 nm.

35.3.3 *Fuel*—Acetylene.

35.3.4 *Oxidant*—Air.

35.3.5 *Type of Flame*—Oxidizing.

35.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

NOTE 80—The analysis of this metal is exceptionally sensitive to turbulence and absorption bands in the flame. Therefore, some care should be taken to position the light beam in the most stable, center portion of the flame. To do this, first adjust the burner to maximize the absorbance reading with a lead standard. Then, aspirate a water blank and make minute adjustments in the burner alignment to minimize the signal.

36. Lead—Furnace Technique

36.1 *Requirements:*

36.1.1 *Optimum Concentration Range*, 5 to 100 μg/L (see Note 81).

36.1.2 *Detection Limit*, 1 μg/L.

NOTE 81—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-μL injection, continuous flow purge gas and nonpyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

36.2 *Preparation of Standard Solution:*

36.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

36.2.2 *Lanthanum Nitrate Solution*—Dissolve 58.64 g of ACS reagent grade La_2O_3 in 100 mL concentrated HNO_3, and dilute to 1000 mL with deionized distilled water. 1 mL = 50 mg La.

36.2.3 *Working Lead Solution*—Prepare dilutions of the stock lead solution to be used as calibration standards at the time of analysis. Each calibration standard should contain 0.5 % (v/v) HNO_3. To each 100 mL of diluted standard, add 10 mL of the lanthanum nitrate solution.

36.3 *General Instrument Parameters:*

36.3.1 *Drying Time and Temperature*—30 s at 125°C.

36.3.2 *Ashing Time and Temperature*—30 s at 500°C.

36.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

36.3.4 *Purge Gas Atmosphere*—Argon.

36.3.5 *Wavelength*—283.3 nm.

NOTE 82—Greater sensitivity can be achieved using the 217.0-nm line, but the optimum concentration range is reduced. The use of a lead electrodeless discharge lamp at this lower wavelength has been found to be advantageous. Also a lower atomization temperature (2400°C) may be preferred.

36.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 83—The use of background correction is recommended.

36.4 *Analysis Procedure*—For the analysis procedure in the calculation see "Furnace Procedure," 11.3.

NOTE 84—To suppress sulfate interference (up to 1500 ppm) lanthanum is added as the nitrate to both samples and calibration standards (**10**).

NOTE 85—Since glassware contamination is a severe problem in lead analysis, all glassware should be cleaned immediately prior to use, and once cleaned, should not be open to the atmosphere except when necessary.

NOTE 86—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 87—If the method of standard additions is required, follow the procedure given in 10.5.

37. Lithium—Direct Aspiration

37.1 *Requirements:*

37.1.1 *Optimum Concentration Range*—to 0.2 mg/L using a wavelength of 670.8 nm (see Note 88).

37.1.2 *Sensitivity*—0.035 mg/L.

NOTE 88—The following lines may also be used:
323.3 nm relative sensitivity 235, and
610.4 nm relative sensitivity 3600.

37.2 *Preparation of Standard Solution:*

37.2.1 *Stock Solution*—Dissolve 5.324 g of lithium carbonate, Li_2CO_3 in a minimum volume of (1+1) HCl and dilute to 1 L with deionized water. 1 mL = 1.00 mg Li (1000 mg/L).

37.2.2 Prepare dilutions of the stock lithium solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

37.3 *General Instrumental Parameters:*

37.3.1 *Lithium Hollow Cathode Lamp.*

37.3.2 *Wavelength*—670.8 nm.

37.3.3 *Fuel*—Acetylene.

37.3.4 *Oxidant*—Air.

37.4 *Analysis Procedure*—For analysis procedure and calculations, see "Direct Aspiration," 11.1.

38. Magnesium—Direct Aspiration

38.1 *Requirements:*

38.1.1 *Optimum Concentration Range*, 0.02 to 0.5 mg/L using a wavelength of 285.2 nm (see Notes 89 and 90).

38.1.2 *Sensitivity*, 0.007 mg/L.

38.1.3 *Detection Limit*, 0.001 mg/L.

NOTE 89—The following line may also be used: 202.5 nm relative sensitivity 25.

NOTE 90—To cover the range of magnesium values normally observed in surface waters (0.1 to 20 mg/L), it is suggested that either the 202.5 nm line be used or the burner head be rotated. A 90° rotation of the burner head will produce approximately one-eighth the normal sensitivity.

38.2 *Preparation of Standard Solution:*

38.2.1 *Stock Solution*—Dissolve 0.829 g of magnesium oxide, MgO (analytical reagent grade), in 10 mL of redistilled HNO_3, and dilute to 1 L with deionized distilled water. 1 mL = 0.50 mg Mg (500 mg/L).

38.2.2 *Lanthanum Chloride Solution*—Dissolve 29 g of La_2O_3, slowly and in small portions in 250 mL concentrated HCl, (**Caution**—Reaction is violent), and dilute to 500 mL with deionized distilled water.

38.2.3 Prepare dilutions of the stock magnesium solution to be used as calibration standards at the time of analysis. These *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed directly or after processing. To each 10-mL volume of *calibration standard* and sample alike add 1.0 mL of the lanthanum chloride solution, that is, 20 mL of standard or sample +2 mL $LaCl_3$ = 22 mL.

38.3 *General Instrumental Parameters:*

38.3.1 *Magnesium Hollow Cathode Lamp.*

38.3.2 *Wavelength*—285.2 nm.

38.3.3 *Fuel*—Acetylene.

38.3.4 *Oxidant*—Air.

38.3.5 *Type of Flame*—Oxidizing.

38.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

NOTE 91—The interference caused by aluminum at concentrations greater than 2 mg/L is masked by addition of lanthanum. Sodium, potassium and calcium cause no interference at concentrations less than 400 mg/L.

39. Manganese—Direct Aspiration

39.1 *Requirements:*

39.1.1 *Optimum Concentration Range*, 0.1 to 3 mg/L using a wavelength of 279.5 nm (see Note 92).

39.1.2 *Sensitivity*, 0.05 mg/L.

39.1.3 *Detection Limit*, 0.01 mg/L.

NOTE 92—The following line may also be used: 403.1 nm Relative Sensitivity 10.

39.2 *Preparation of Standard Solution:*

39.2.1 *Stock Solution*—Carefully weigh 1.000 g of manganese metal (analytical reagent grade), and dissolve in 10 mL of redistilled HNO_3. When solution is complete, dilute to 1 L with 1 % (v/v) HCl. 1 mL = 1 mg Mn (1000 mg/L).

39.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

39.3 *General Instrumental Parameters:*

39.3.1 *Manganese hollow cathode lamp.*

39.3.2 *Wavelength*—279.5 nm.

39.3.3 *Fuel*—Acetylene.

39.3.4 *Oxidant*—air.

39.3.5 *Type of Flame*—oxidizing.

39.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

NOTE 93—For levels of manganese below 25 µg/L, either the furnace procedure or the Special Extraction Procedure given in 10.2 is recommended. The extraction is carried out at a pH of 4.5 to 5. The manganese chelate is very unstable and the analysis must be made without delay to prevent its solution in the aqueous phase.

40. Manganese—Furnace Technique

40.1 *Requirements:*

40.1.1 *Optimum Concentration Range,* 1 to 30 µg/L (see Note 94).

40.1.2 *Detection Limit,* 0.2 µg/L.

NOTE 94—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-uL injection, continuous flow purge gas and nonpyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

40.2 *Preparation of Standard Solution:*

40.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

40.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions."

40.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

40.3 *General Instrument Parameters:*

40.3.1 *Drying Time and Temperature*—30 s at 125°C.

40.3.2 *Ashing Time and Temperature*—30 s at 1000°C.

40.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

40.3.4 *Purge Gas Atmosphere*—Argon.

40.3.5 *Wavelength*—279.5 nm.

40.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 95—The use of background correction is recommended.

NOTE 96—Nitrogen may also be used as the purge gas.

40.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure" 11.3.

NOTE 97—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 98—If the method of standard additions is required, follow the procedure given earlier in 10.5.

41. Mercury Cold Vapor Technique

41.1 *Scope and Application* (**11**):

41.1.1 In addition to inorganic forms of mercury, organic mercurials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenyl mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100 % recovery when used as the oxidant with these compounds. Therefore, a persulfate oxidation step following the addition of the permanganate has been included to ensure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. For distilled water the heat step is not necessary.

41.1.2 The range of the test method may be varied through instrument or recorder expansion, or both. Using a 100-mL sample, a detection limit of 0.2 µg Hg/L can be achieved. Concentrations below this level should be reported as <0.2.

NOTE 99—If additional sensitivity is required, a 200 mL sample with recorder expansion may be used provided the instrument does not produce undue noise. Using a Coleman MAS-50 with a drying tube of magnesium perchlorate and a variable recorder, 2 mv was set to read full scale. With these conditions, and distilled water solutions of mercuric chloride at concentrations of 0.15, 0.10, 0.05 and 0.025 µg/L, the standard deviations were ±0.027, ±0.006, ±0.01, and ±0.004. Percent recoveries at these levels were 107, 83, 84, and 96 %, respectively.

41.2 *Summary of Test Method:*

41.2.1 The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

41.3 *Sample Handling and Preservation:*

41.3.1 Until more conclusive data are obtained, samples should be preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection. If only dissolved mercury is to be determined, the sample should be filtered through an all glass apparatus before the acid is added. For total mercury, the filtration is omitted.

41.4 *Interference:*

41.4.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

41.4.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

41.4.3 High chloride concentrations require additional permanganate (as much as 25 mL). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation of 253 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). In addition, the dead air space in the BOD bottle must be purged before the addition of stannous sulfate. Both inorganic and organic mercury spikes have been quantitatively recovered from sea water using this technique.

41.4.4 Interference from certain volatile organic materials that will absorb at this wavelength is also possible. A preliminary run without reagents should determine if this type of interference is present.

NOTE 100—The possibility of absorption from certain organic substances actually being present in the sample does exist. This is

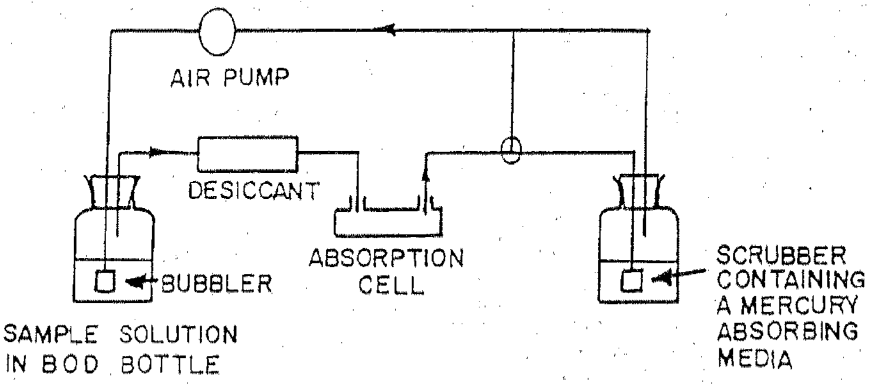

FIG. 2 Apparatus for Flameless Mercury Determination

mentioned only to caution the analyst of the possibility. A simple correction that may be used is as follows: If an interference has been found to be present, the sample should be analyzed both by using the regular procedure and again under oxidizing conditions only, that is without the reducing reagents. The true mercury value can then be obtained by subtracting the two values.

41.5 *Apparatus:*

41.5.1 Atomic Absorption Spectrophotometer; (See Note 101) Any atomic absorption unit having an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed.

NOTE 101—Instruments designated specifically for the measurement of mercury using the cold vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

41.5.2 *Mercury Hollow Cathode Lamp*—argon filled, or equivalent.[11]

41.5.3 *Recorder*—Any multi-range variable speed recorder that is compatible with the UV detection system is suitable.

41.5.4 *Absorption Cell*—Standard spectrophotometer cells 10 cm long, having quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 1 outside diameter by 4½ in. The ends are ground perpendicular to the longitudinal axis and quartz windows (1-in. diameter by 1/16-in. thickness) are cemented in place. The cell is strapped to a burner for support and aligned in the light beam by use of two 2 by 2 in. cards. One-inch diameter holes are cut in the middle of each card; the cards are then placed over each end of the cell. The cell is then positioned and adjusted vertically and horizontally to give the maximum transmittance.

41.5.5 *Air Pump*—Any peristaltic pump capable of delivering 1 L of air per minute may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

41.5.6 *Flowmeter*—Capable of measuring an air flow of 1 L per minute.

41.5.7 *Aeration Tubing*—A straight-glass frit having a coarse porosity. Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return.

41.5.8 *Drying Tube*—6 by ¾ in. diameter tube containing 20 g of magnesium perchlorate (see Note 102). The apparatus is assembled as shown in Fig. 2.

NOTE 102—In place of the magnesium perchlorate drying tube, a small reading lamp with 60 W bulb may be used to prevent condensation of moisture inside the cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10.25°C above ambient.

41.6 *Reagents:*

41.6.1 *Sulfuric Acid, Concentrated*—Reagent grade.

41.6.1.1 *Sulfuric Acid,* 0.5 N—Dilute 14.0 mL of concentrated sulfuric acid to 1.0 L.

41.6.2 *Nitric Acid,* Concentrate—Reagent grade of low mercury content.

NOTE 103—If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

41.6.3 *Stannous Sulfate*—Add 25 g stannous sulfate to 250 mL of 0.5 *N* sulfuric acid. This mixture is a suspension and should be stirred continuously during use. (Stannous chloride may be used in place of stannous sulfate.)

41.6.4 *Sodium Chloride-Hydroxylamine Sulfate Solution*—Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in distilled water and dilute to 100 mL. (Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.)

41.6.5 *Potassium Permanganate* (5 % solution, w/v)—Dissolve 5 g of potassium permanganate in 100 mL of distilled water.

41.6.6 *Potassium Persulfate* (5 % solution, w/v)—Dissolve 5 g of potassium persulfate in 100 mL of distilled water.

41.6.7 *Stock Mercury Solution*—Dissolve 0.1354 g of mercuric chloride in 75 mL of distilled water. Add 10 mL of concentrated nitric acid and adjust the volume to 100.0 mL. 1 mL = 1 mg Hg.

41.6.8 *Working Mercury Solution*—Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 µg per mL. This working standard and the dilutions of the stock mercury solution should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15 % nitric acid. This acid should be added to the flask as needed before the addition of the aliquot.

41.7 *Calibration:*

41.7.1 Transfer 0, 0.5, 1.0, 2.0, 5.0, and 10.0 mL aliquots of the working mercury solution containing 0 to 1.0 µg of mercury to a series of 300 mL BOD bottles. Add enough

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[11] Westinghouse WL-22847 available from any laboratory supply house has been found suitable.

distilled water to each bottle to make a total volume of 100 mL. Mix thoroughly and add 5 mL of concentrated sulfuric acid and 2.5 mL of concentrated nitric acid to each bottle. Add 15 mL of $KMnO_4$ solution to each bottle, and allow to stand at least 15 min. Add 8 mL of potassium persulfate to each bottle and heat for 2 h in a water bath maintained at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. When the solution has been decolorized wait 30 s, add 5 mL of the stannous sulfate solution and immediately attach the bottle to the aeration apparatus forming a closed system. At this point the sample is allowed to stand quietly without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 L per minute, is allowed to run continuously. (An open system where the mercury vapor is passed through the absorption cell only once may be used instead of the closed system.)

41.7.2 The absorbance will increase and reach maximum within 30 s. As soon as the recorder pen levels off, approximately 1 min, open the bypass valve and continue the aeration until the absorbance returns to its minimum value. Because of the toxic nature of mercury vapor, precaution must be taken to avoid its inhalation. Therefore, a bypass has been included in the system to either vent the mercury vapor into an exhaust hood or pass the vapor through some absorbing media such as follows:

(*a*) equal volumes of 0.1 M $KMnO_4$ and 10 % H_2SO_4, and

(*b*) 0.25 % iodine in a 3 % KI solution.

NOTE 104—Directions for the disposal of mercury-containing wastes are given in Test Method D 3223.[12]

41.7.3 Close the bypass valve, remove the stopper and frit from the BOD bottle and continue the aeration. Proceed with the standards and construct a standard curve by plotting peak height versus micrograms of mercury.

41.8 *Procedure:*

41.8.1 Transfer 100 mL, or an aliquot diluted to 100 mL, containing not more than 1.0 µg of mercury, to a 300-mL BOD bottle. Add 5 mL of sulfuric acid and 2.5 mL of concentrated nitric acid mixing after each addition. Add 15 mL of potassium permanganate solution to each sample bottle. For sewage, sample additional permanganate solution, if necessary, until the purple color persists for at least 15 min. Add 8 mL of potassium persulfate to each bottle and heat for 2 h in a water bath at 95°C. Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. After a delay of at least 30 s, add 5 mL of stannous sulfate, and immediately attach the bottle to the aeration apparatus. Continue as described in 41.7.

41.9 *Calculation:*

41.9.1 Determine the peak height of the unknown from the chart, and read the mercury value from the standard curve.

41.9.2 Calculate the mercury concentration in the sample by the formula:

$$\mu g\ Hg/L = (\mu g\ Hg\ in\ aliquot)\left(\frac{1{,}000}{\text{volume of aliquot in mL}}\right)$$

41.9.3 Report mercury concentrations as follows: Below 0.2 µg/L, <0.2; between 1 and 10 µg/L, one decimal; above 10 µg/L, whole numbers.

42. Molybdenum—Direct Aspiration

42.1 *Requirements:*

42.1.1 *Optimum Concentration Range,* 1 to 40 mg/L using a wavelength of 313.3 nm (see Note 105).

42.1.2 *Sensitivity,* 0.4 mg/L.

42.1.3 *Detection Limit,* 0.1 mg/L.

NOTE 105—For concentrations of molybdenum below 0.2 mg/L, the furnace procedure is recommended.

42.2 *Preparation of Standard Solution:*

42.2.1 *Stock Solution*—Dissolve 1.840 g of ammonium molybdate $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ (analytical reagent grade) in deionized distilled water and dilute to 1 L. 1 mL = 1 mg Mo (1000 mg/L).

42.2.2 *Aluminum Nitrate Solution*—Dissolve 139 g aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$, in 150 mL of deionized distilled water; heat to effect solution. Allow to cool and make up to 200 mL.

42.2.3 Prepare dilutions of the stock molybdenum solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing. To each 100 mL of standard and sample alike, add 2 mL of the aluminum nitrate solution.

42.3 *General Instrumental Parameters:*

42.3.1 *Molybdenum Hollow Cathode Lamp.*

42.3.2 *Wavelength*—313.3 nm.

42.3.3 *Fuel*—Acetylene.

42.3.4 *Oxidant*—Nitrous Oxide.

42.3.5 *Type of Flame*—Fuel rich.

42.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

42.5 *Interferences:*

42.5.1 With the recommended nitrous oxide-acetylene flame, interferences of calcium and other ions may be controlled by adding 1000 mg/L of a refractory metal such as aluminum **(12)**. This should be done to both samples and standards alike.

43. Molybdenum—Furnace Technique

43.1 *Requirements:*

43.1.1 *Optimum Concentration Range,* 3 to 60 µg/L (see Note 106).

43.1.2 *Detection Limit,* 1 µg/L.

NOTE 106—The concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-µL injection, continuous flow purge gas and pyrolytic graphite.

43.2 *Preparation of Standard Solution:*

43.2.1 *Stock Solution*—Prepare as described under "direct aspiration method".

43.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions".

43.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

43.3 *General Instrument Parameters:*

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[12] A specially treated charcoal that will absorb mercury vapor is also available from Barneby and Cheney, East 8th Ave. and North Cassidy St., Columbus, OH 43219, Catalog No. E580-13 or 580-22.

43.3.1 *Drying Time and Temperature*—30 s at 125°C.

43.3.2 *Ashing Time and Temperature*—30 s at 1400°C.

43.3.3 *Atomizing Time and Temperature*—15 s at 2800°C.

43.3.4 *Purge Gas Atmosphere*—Argon.

43.3.5 *Wavelength*—313.3 nm.

43.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 107—Background correction may be required if the sample contains high dissolved solids.

NOTE 108—The use of nitrogen as a purge gas is not recommended.

43.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure," 11.3.

NOTE 109—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 110—If the method of standard additions is required, follow the procedure given in 10.5.

44. Nickel—Direct Aspiration

44.1 *Requirements:*

44.1.1 *Optimum Concentration Range,* 0.3 to 5 mg/L using a wavelength of 232.0 nm (see Note 111).

44.1.2 *Sensitivity,* 0.15 mg/L.

44.1.3 *Detection Limit,* 0.04 mg/L.

NOTE 111—For levels of nickel below 100 µg/L, either the Special Extraction Procedure, given in 11.2, or the furnace technique is recommended.

44.2 *Preparation of Standard Solution:*

44.2.1 *Stock Solution*—Dissolve 4.953 g of nickel nitrate, $Ni(NO_3)_2 \cdot 6H_2O$ (analytical reagent grade) in deionized distilled water. Add 10 mL of concentrated nitric acid and dilute to 1 L with deionized distilled water. 1 mL = 1 mg Ni (1000 mg/L).

44.2.2 Prepare dilutions of the stock nickel solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

44.3 *General Instrumental Parameters:*

44.3.1 *Nickel Hollow Cathode Lamp.*

44.3.2 *Wavelength*—232.0 nm.

44.3.3 *Fuel*—Acetylene.

44.3.4 *Oxidant*—Air.

44.3.5 *Type of Flame*—Oxidizing.

44.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration", 11.1.

44.5 *Interferences*—The 352.4-nm wavelength is less susceptible to interference and may be used. The calibration curve is more linear at this wavelength; however, there is some loss of sensitivity.

45. Nickel—Furnace Technique

45.1 *Requirements:*

45.1.1 *Optimum Concentration Range,* 5 to 100 µg/L (see Note 112).

45.1.2 *Detection Limit,* 1 µg/L.

NOTE 112—The concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-µL injection, continuous flow purge gas and pyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the recommended settings.

45.2 *Preparation of Standard Solution:*

45.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

45.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions."

45.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

45.3 *General Instrument Parameters:*

45.3.1 *Drying Time and Temperature*—30 s at 125°C.

45.3.2 *Ashing Time and Temperature*—30 s at 900°C.

45.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

45.3.4 *Purge Gas Atmosphere*—Argon.

45.3.5 *Wavelength*—232.0 nm.

45.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 113—The use of background correction is recommended.

NOTE 114—Nitrogen may also be used as the purge gas.

45.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure", 11.3.

NOTE 115—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 116—If the method of standard additions is required, follow the procedure given in 10.5.

46. Potassium—Direct Aspiration

46.1 *Requirements:*

46.1.1 *Optimum Concentration Range*—0.1 to 2 mg/L using a wavelength of 766.5 nm (see Note 117).

46.1.2 *Sensitivity*—0.04 mg/L.

46.1.3 *Detection Limit*—0.01 mg/L.

NOTE 117—The 404.4-nm line may also be used. This line has a relative sensitivity of 500.

46.2 *Preparation of Standard Solution:*

46.2.1 *Stock Solution*—Dissolve 0.1907 g of KCl (analytical reagent grade), dried at 110°C, in deionized distilled water and make up to 1 L. 1 mL = 0.10 mg K (100 mg/L).

46.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

46.3 *General Instrumental Parameters:*

46.3.1 *Potassium Hollow Cathode Lamp.*

46.3.2 *Wavelength*—766.5 nm.

46.3.3 *Fuel*—Acetylene.

46.3.4 *Oxidant*—Air.

46.3.5 *Type of Flame*—Slightly oxidizing.

46.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration", 11.1.

NOTE 118—In air-acetylene or other high temperature flames (>2800°C), potassium can experience partial ionization which indirectly affects absorption sensitivity. The presence of other alkali salts in the sample can reduce this ionization and thereby enhance analytical results. The ionization suppressive effect of sodium is small if the ratio of Na to K is under 10. Any enhancement due to sodium can be stabilized by adding excess sodium (1000 µg/mL) to both sample and standard solutions. If more stringent control of ionization is required, the addition of cesium should be considered. Reagent blanks should be analyzed to correct for potassium impurities in the buffer stock.

NOTE 119—To cover the range of potassium values normally observed in surface waters (0.1 to 20 mg/L), it is suggested that the burner head be rotated. A 90° rotation of the burner head provides approximately one-eighth the normal sensitivity.

47. Selenium—Furnace Technique

47.1 *Requirements:*

47.1.1 *Optimum Concentration Range*, 5 to 100 µg/L (see Note 120).

47.1.2 *Detection Limit*, 2 µg/L.

NOTE 120—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-µL injection, continuous flow purge gas and non-pyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

47.2 *Preparation of Standard Solution:*

47.2.1 *Stock Selenium Solution*—Dissolve 0.3453 g of selenous acid (actual assay 94.6 % H_2SeO_3) in deionized distilled water and make up to 200 mL. 1 mL = 1 mg Se (1000 mg/L).

47.2.2 *Nickel Nitrate Solution, 5 %*—Dissolve 24.780 g of ACS reagent grade $Ni(NO_3)_2 \cdot 6H_2O$ in deionized distilled water and make up to 100 mL.

47.2.3 *Nickel Nitrate Solution, 1 %*—Dilute 20 mL of the 5 % nickel nitrate to 100 mL with deionized distilled water.

47.2.4 *Working Selenium Solution*—Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Withdraw appropriate aliquots of the stock solution, add 1 mL of concentrated HNO_3, 2 mL of 30 % H_2O_2 and 2 mL of the 5 % nickel nitrate solution. Dilute to 100 mL with deionized distilled water.

47.3 *Sample Preparation:*

47.3.1 Transfer 100 mL of well-mixed sample to a 250-mL Griffin beaker, add 2 mL of 30 % H_2O_2 and sufficient concentrated HNO_3 to result in an acid concentration of 1 % (v/v). Heat for 1 h at 95°C or until the volume is slightly less than 50 mL.

47.3.2 Cool and bring back to 50 mL with deionized distilled water.

47.3.3 Pipet 5 mL of this digested solution into a 10 mL volumetric flask, add 1 mL of the 1 % nickel nitrate solution and dilute to 10 mL with deionized distilled water. The sample is now ready for injection into the furnace.

NOTE 121—If solubilization or digestion is not required, adjust the HNO_3 concentration of the sample to 1 % (v/v) and add 2 mL of 30 % H_2O_2 and 2 mL of 5 % nickel nitrate to each 100 mL of sample. The volume of the calibration standard should be adjusted with deionized distilled water to match the volume change of the sample.

47.4 *Instrument Parameters:*

47.4.1 *Drying Time and Temperature*—30 s at 125°C.

47.4.2 *Charring Time and Temperature*—30 s at 1200°C.

47.4.3 *Atomizing Time and Temperature*—10 s at 2700°C.

47.4.4 *Purge Gas Atmosphere*—Argon.

47.4.5 *Wavelength*—196.0 nm.

47.4.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 122—The use of background correction is recommended.

47.5 *Analysis Procedure*—For the analysis procedure and the calculation see "Furnace Procedure," 11.3.

NOTE 123—Selenium analysis suffers interference from chlorides (>800 mg/L) and sulfate (>200 mg/L). For the analysis of industrial effluents and samples with concentrations of sulfate from 200 to 2000 mg/L, both samples and standards should be prepared to contain 1 % nickel.

NOTE 124—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 125—If the method of standard additions is required, follow the procedure given in 10.5.

48. Selenium—Gaseous Hydride

48.1 *Scope and Application*—The gaseous hydride method determines inorganic selenium when present in concentrations at or above 2 µg/L. The test method is applicable in the absence of high concentrations of chromium, cobalt, copper, mercury, molybdenum, nickel, and silver.

48.2 *Summary of Test Method*—Selenium in the sample is reduced from the +6 oxidation state to the +4 oxidation state by the addition of $SnCl_2$. Zinc is added to the acidified sample, producing hydrogen and converting the selenium to the hydride, SeH_2. The gaseous selenium hydride is swept into an argon-hydrogen flame of an atomic absorption spectrophotometer. The working range of the test method is 2 to 20 µg/L using the 196.0-nm wavelength.

48.3 *Discussion:*

48.3.1 In analyzing drinking water and most surface and ground waters, interferences are rarely encountered. Industrial waste samples should be spiked with a known amount of selenium to establish adequate recovery.

48.3.2 Organic forms of selenium must be converted to an inorganic form and organic matter must be oxidized before beginning the analysis. The oxidation procedure given in Procedure 4.1 of Method 206.5 should be used **(13)**.

48.4 References

48.4.1 Except for the perchloric acid step, the procedure to be used for this determination is found in *Standard Methods for the Examination of Water and Wastewater* **(14)**.

49. Silver—Direct Aspiration

49.1 *Requirements:*

49.1.1 *Optimum Concentration Range*, 0.1 to 4 mg/L using a wavelength of 328.1 nm (see Notes 126 and 127).

49.1.2 *Sensitivity*, 0.06 mg/L.

49.1.3 *Detection Limit*, 0.01 mg/L.

NOTE 126—For levels of silver below 30 µg/L, either the Special Extraction Procedure, given in 11.2 or the furnace procedure is recommended.

NOTE 127—The 338.2-nm wavelength may also be used. This has a relative sensitivity of 2.

49.2 *Preparation of Standard Solution:*

49.2.1 *Stock Solution*—Dissolve 1.575 g of $AgNO_3$ (analytical reagent grade) in deionized distilled water, add 10 mL concentrated HNO_3 and make up to 1 L. 1 mL = 1 mg Ag (1000 mg/L).

NOTE 128—Silver nitrate standards are light sensitive. Dilutions of the stock should be discarded after use as concentrations below 10 mg/L are not stable over long periods of time.

49.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration*

standards should be prepared using nitric acid and at the same concentration as that of the sample being analyzed either directly or after processing.

49.2.3 *Iodine Solution* (1 *N*)—Dissolve 20 g of potassium iodide, KI (analytical reagent grade) in 50 mL of deionized distilled water, add 12.7 g of iodine, I_2 (analytical reagent grade) and dilute to 100 mL. Store in a brown bottle.

49.2.4 *Cyanogen Iodide* (CNI) *Solution*—To 50 mL of deionized distilled water add 4.0 mL concentrated NH_4OH, 6.5 g KCN, and 5.0 mL of 1.0 *N* I_2 solution. Mix and dilute to 100 mL with deionized distilled water. Fresh solution should be prepared every 2 weeks.

49.3 *General Instrumental Parameters:*

49.3.1 *Silver Hollow Cathode Lamp.*

49.3.2 Wavelength—328.1 nm.

49.3.3 *Fuel*—Acetylene.

49.3.4 *Oxidant*—Air.

49.3.5 *Type of Flame*—Oxidizing.

49.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Direct Aspiration," 11.1.

NOTE 129—If absorption to container walls or the formation of AgCl is suspected, make the sample basic using concentrated NH_4OH and add 1 mL of (CNI) solution per 100 mL of sample. Mix the sample and allow to stand for 1 h before proceeding with the analysis.

50. Silver—Furnace Technique

50.1 *Requirements:*

50.1.1 *Optimum Concentration Range*—1 to 25 μg/L (see Note 130).

50.1.2 *Detection Limit*—0.2 μg/L.

NOTE 130—The concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-μL injection, continuous flow purge gas and nonpyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

50.2 *Preparation of Standard Solution:*

50.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

50.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions."

50.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

NOTE 131—The use of halide acids should be avoided.

50.3 *General Instrument Parameters:*

50.3.1 *Drying Time and Temperature*—30 s at 125°C.

50.3.2 *Ashing Time and Temperature*—30 s at 400°C.

50.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

50.3.4 *Purge Gas Atmosphere*—Argon.

50.3.5 *Wavelength*—328.1 nm.

50.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 132—Background correction may be required if the sample contains high dissolved solids.

50.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure," 11.3 (see Notes 3, 5, 6, and 7).

NOTE 133—If adsorption to container walls or formation of AgCl is suspected, see the Direct Aspiration Method.

NOTE 134—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 135—If the method of standard additions is required, follow the procedure given in 10.5.

51. Sodium—Direct Aspiration

51.1 *Requirements:*

51.1.1 *Optimum Concentration Range,* 0.03 to 1 mg/L using a wavelength of 589.6 nm (see Note 136).

51.1.2 *Sensitivity,* 0.015 mg/L.

51.1.3 *Detection Limit,* 0.002 mg/L.

NOTE 136—The 330.2 nm resonance line of sodium, which has a relative sensitivity of 185, provides a convenient way to avoid the need to dilute more concentrated solutions of sodium.

51.2 *Preparation of Standard Solution:*

51.2.1 *Stock Solution*—Dissolve 2.542 g of NaCl (analytical reagent grade), dried at 140°C, in deionized distilled water and make up to 1 L. 1 mL = 1 mg Na (1000 mg/L).

51.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

51.3 *General Instrumental Parameters:*

51.3.1 *Sodium Hollow Cathode Lamp.*

51.3.2 *Wavelength*—589.6 nm.

51.3.3 *Fuel*—Acetylene.

51.3.4 *Oxidant*—Air.

51.3.5 *Type of Flame*—Oxidizing.

NOTE 137—Low-temperature flames increase sensitivity by reducing the extent of ionization of this easily ionized metal. Ionization may also be controlled by adding potassium (1000 mg/L) to both standards and samples.

51.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

52. Tin—Direct Aspiration

52.1 *Requirements:*

52.1.1 *Optimum Concentration Range,* 10 to 300 mg/L using a wavelength of 286.3 nm (see Note 138).

52.1.2 *Sensitivity,* 4 mg/L.

52.1.3 *Detection Limit,* 0.8 mg/L.

NOTE 138—For concentrations of tin below 2 mg/L, the furnace procedure is recommended.

52.2 *Preparation of Standard Solution:*

52.2.1 *Stock Solution*—Dissolve 1.000 g of tin metal (analytical reagent grade) in 100 mL of concentrated HCl and dilute to 1 L with deionized distilled water. 1 mL = 1 mg Sn (1000 mg/L).

52.2.2 Prepare dilutions of the stock tin solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

52.3 *General Instrumental Parameters:*

52.3.1 *Tin Hollow Cathode Lamp.*

52.3.2 *Wavelength*—286.3 nm.

52.3.3 *Fuel*—Acetylene.

52.3.4 *Oxidant*—Nitrous oxide.

52.3.5 *Type of Flame*—Fuel rich.

52.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Direct Aspiration," 11.1.

53. Tin—Furnace Technique

53.1 *Requirements:*

53.1.1 *Optimum Concentration Range,* 20 to 300 μg/L (see Note 139).

53.1.2 *Detection Limit,* 5 μg/L.

NOTE 139—The concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 μL injection, continuous flow purge gas and non-pyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the recommended settings.

53.2 *Preparation of Standard Solution:*

53.2.1 *Stock Solution*—Prepare as described under "direct aspiration method".

53.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions".

53.2.3 The calibration standard should be diluted to contain 2 % (v/v) HNO_3.

53.3 *Instrument Parameters:*

53.3.1 *Drying Time and Temperature*—30 s at 125°C.

53.3.2 *Ashing Time and Temperature*—30 s at 600°C.

53.3.3 *Atomizing Time and Temperature*—10 s at 2700°C.

53.3.4 *Purge Gas Atmosphere*—Argon.

53.3.5 *Wavelength*—224.6 nm.

53.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 140—The use of background correction is recommended.

NOTE 141—Nitrogen may also be used as the purge gas.

53.4 *Analysis Procedure*—For the analysis procedure and the calculation see "Furnace Procedure," 11.3.

NOTE 142—Tin analysis is sensitive to chloride concentration. If chloride concentration presents a matrix problem or causes a loss previous to atomization, add an excess of 5 mg of ammonium nitrate to the furnace and ash using a ramp necessary or with incremental steps until the recommended ashing temperature is reached. Extended ashing times have been reported to improve precision.

NOTE 143—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 144—If the method of standard additions is required, follow the procedure given in 10.5.

54. Titanium—Direct Aspiration

54.1 *Requirements:*

54.1.1 *Optimum Concentration Range,* 5 to 100 mg/L using a wavelength of 365.3 nm (see Note 145).

54.1.2 *Sensitivity,* 2 mg/L.

54.1.3 *Detection Limit,* 0.4 mg/L.

NOTE 145—For concentrations of titanium below 1.0 mg/L, the furnace procedure is recommended.

54.2 *Preparation of Standard Solution:*

54.2.1 *Stock Solution*—Dissolve 4.008 g of titanium sulfate, $Ti_2(SO_4)_3$, in dilute HCl and make up to 1 L with deionized distilled water. 1 mL = 1 mg Ti (1000 mg/L).

54.2.2 *Potassium Chloride Solution*—Dissolve 95 g potassium chloride, KCl, in distilled water and make up to 1 L.

54.2.3 Prepare dilutions of the stock titanium solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing. To each 100 mL of standard and sample alike, add 2 mL of potassium chloride solution.

54.3 *General Instrumental Parameters:*

54.3.1 *Titanium Hollow Cathode Lamp.*

54.3.2 *Wavelength*—365.3 nm.

54.3.3 *Fuel*—Acetylene.

54.3.4 *Oxidant*—Nitrous oxide.

54.3.5 *Type of Flame*—Fuel rich.

54.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Direct Aspiration," 11.1.

54.5 *Interferences*—A number of elements increase the sensitivity of titanium. To control this problem, potassium (1000 mg/L) must be added to standards and samples alike **(15)**.

55. Titanium Furnace Technique

55.1 *Requirements:*

55.1.1 *Optimum Concentration Range,* 50 to 500 μg/L (see Note 146).

55.1.2 *Detection Limit,* 10 μg/L.

NOTE 146—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-μL injection, continuous flow purge gas and pyrolytic graphite.

55.2 *Preparation of Standard Solution:*

55.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

55.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis.

55.2.3 The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

55.3 *General Instrument Parameters:*

55.3.1 *Drying Time and Temperature*—30 s at 125°C.

55.3.2 *Ashing Time and Temperature*—30 s at 1400°C.

55.3.3 *Atomizing Time and Temperature*—15 s at 2800°C.

55.3.4 *Purge Gas Atmosphere*—Argon.

55.3.5 *Wavelength*—365.4 nm.

55.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 147—Background correction may be required if the sample contains high dissolved solids.

NOTE 148—Because of possible chemical interactions, nitrogen should not be used as the purge gas.

55.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure," 11.3.

NOTE 149—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 150—If the method of standard additions is required, follow the procedure given in 10.5.

56. Vanadium—Direct Aspiration

56.1 *Requirements:*

56.1.1 *Optimum Concentration Range*—2 to 100 mg/L using a wavelength of 318.4 nm (see Note 151).

56.1.2 *Sensitivity*—0.8 mg/L.

56.1.3 *Detection Limit*—0.2 mg/L.

NOTE 151—For concentrations of vanadium below 0.5 mg/L, the furnace procedure is recommended.

56.2 *Preparation of Standard Solution:*

56.2.1 *Stock Solution*—Dissolve 1.7854 g of vanadium pentoxide, V_2O_5 (analytical reagent grade) in 10 mL of concentrated nitric acid and dilute to 1 L with deionized distilled water. 1 mL = 1 mg V (1000 mg/L).

56.2.2 *Aluminum Nitrate Solution*—Dissolve 139 g aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$, in 150 mL of deionized distilled water; heat to effect solution. Allow to cool and make up to 200 mL.

56.2.3 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing. To each 100 mL of standard and sample alike, add 2 mL of the aluminum nitrate solution.

56.3 *General Instrumental Parameters:*

56.3.1 *Vanadium Hollow Cathode Lamp.*

56.3.2 *Wavelength*—318.4 nm.

56.3.3 *Fuel*—Acetylene.

56.3.4 *Oxidant*—Nitrous oxide.

56.3.5 *Type of Flame*—Fuel rich.

56.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Direct Aspiration," 11.1.

56.5 *Interferences*—It has been reported that high concentrations of aluminum and titanium increase the sensitivity of vanadium. This interference can be controlled by adding excess aluminum (1000 ppm) to both samples and standards **(16)**.

57. Vanadium—Furnace Technique

57.1 *Requirements:*

57.1.1 *Optimum Concentration Range,* 10 to 200 μg/L (see Note 152).

57.1.2 *Detection Limit,* 4 μg/L.

NOTE 152—The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-μL injection, continuous flow purge gas and pyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

57.2 *Preparation of Standard Solution:*

57.2.1 *Stock Solution*—Prepare as described under "direct aspiration method."

57.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions."

57.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

57.3 *General Instrument Parameters:*

57.3.1 *Drying Time and Temperature*—30 s at 125°C.

57.3.2 *Ashing Time and Temperature*—30 s at 1400°C.

57.3.3 *Atomizing Time and Temperature*—15 s at 2800°C.

57.3.4 *Purge Gas Atmosphere*—Argon.

57.3.5 *Wavelength*—318.4 nm.

57.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 153—Background correction may be required if the sample contains high dissolved solids.

NOTE 154—Because of possible chemical interaction, nitrogen should not be used as the purge gas.

57.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure," 11.3.

NOTE 155—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 156—If the method of standard additions is required, follow the procedure given in 10.5.

58. Zinc—Direct Aspiration

58.1 *Requirements:*

58.1.1 *Optimum Concentration Range,* 0.05 to 1 mg/L using a wavelength of 213.9 nm (see Note 157).

58.1.2 *Sensitivity,* 0.02 mg/L.

58.1.3 *Detection Limit,* 0.005 mg/L.

NOTE 157—For concentrations of zinc below 0.01 mg/L, either the Special Extraction Procedure or the Furnace Procedure, is recommended.

58.2 *Preparation of Standard Solution:*

58.2.1 *Stock Solution*—Carefully weigh 1.00 g of zinc metal (analytical reagent grade) and dissolve cautiously in 10 mL HNO_3. When solution is complete make up to 1 L with deionized distilled water. 1 mL = 1 mg Zn (1000 mg/L).

58.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The *calibration standards* should be prepared using the same type of acid and at the same concentration as that of the sample being analyzed either directly or after processing.

58.3 *Instrumental Parameters:*

58.3.1 *Zinc Hollow Cathode Lamp.*

58.3.2 *Wavelength*—213.9 nm.

58.3.3 *Fuel*—Acetylene.

58.3.4 *Oxidant*—Air.

58.3.5 *Type of Flame*—Oxidizing.

58.4 *Analysis Procedure*—For analysis procedure and calculation, see "Direct Aspiration," 11.1.

NOTE 158—High levels of silicon may interfere.

NOTE 159—The air-acetylene flame absorbs about 25 % of the energy at the 213.9 nm line.

NOTE 160—The sensitivity may be increased by the use of low-temperature flames.

NOTE 161—Some sample container cap liners can be a source of zinc contamination. To circumvent or avoid this problem, the use of polypropylene caps is recommended.

59. Zinc—Furnace Technique

59.1 *Requirements:*

59.1.1 *Optimum Concentration Range*—0.2 to 4 μg/L (see Note 162).

59.1.2 *Detection Limit*—0.05 μg/L.

NOTE 162—The concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20-μL injection, continuous flow purge gas and non-pyrolytic graphite. Smaller sized furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the recommended settings.

59.2 *Preparation of Standard Solution:*

59.2.1 *Stock Solution*—Prepare as described under "Direct Aspiration Method."

59.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions."

59.2.3 The calibration standard should be diluted to contain 0.5 % (v/v) HNO_3.

59.3 *General Instrument Parameters:*

59.3.1 *Drying Time and Temperature*—30 s at 125°C.

59.3.2 *Ashing Time and Temperature*—30 s at 400°C.

59.3.3 *Atomizing Time and Temperature*—10 s at 2500°C.

59.3.4 *Purge Gas Atmosphere*—Argon.

59.3.5 *Wavelength*—213.9 nm.

59.3.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE 163—The use of background correction is recommended.

NOTE 164—Nitrogen may also be used as a purge gas.

59.4 *Analysis Procedure*—For the analysis procedure and the calculation, see "Furnace Procedure," 11.3.

NOTE 165—The analysis of zinc by the graphite furnace is extremely sensitive and very subject to contamination from the work area, reagents, and pipet tips. Since all these factors affect the precision and accuracy, zinc should be analyzed by the direct aspiration procedure whenever possible.

NOTE 166—For every sample matrix analyzed, verification is necessary to determine that the method of standard additions is not required (see 6.2.1).

NOTE 167—If the method of standard additions is required, follow the procedure given in 10.5.

60. Precision and Bias

60.1 Precision and bias data for the methods described on RDF are not available at this time.

REFERENCES

(1) *Atomic Absorption Newsletter*, Vol 14, No. 5, Sept. and Oct., Perkin-Elmer Corp., Norwalk, CT, 1975, p. 127.

(2) *Atomic Absorption Newsletter*, Vol 6, 1967, p. 128.

(3) *Atomic Absorption Newsletter*, Vol 5, 1966, p. 17.

(4) *Standard Methods for the Examination of Water and Wastewater*, 14th Ed. Method 301A (VII), American Public Health Assn., American Water Works Assn. and Water Pollution Control Federation, 1975, p. 159.

(5) *Spectrochimica Acta*, Vol 22, Pergamon Press, 1966, p. 1325.

(6) *Standard Methods for the Examination of Water and Wastewater*, 14th Ed., 1975, p. 177.

(7) *Atomic Absorption Newsletter*, Vol 14, 1975, p. 29.

(8) *Talanta*, Vol 20, An International Journal of Analytical Chemistry, Pergamon Press, Inc. Journals Division, Maxwell House, Fairview Park, Elmford, NY 10523, 1973, p. 631.

(9) *Standard Methods for the Examination of Water and Wastewater*, 14th Ed., 1975, p. 192.

(10) *Atomic Absorption Newsletter*, Vol 15, No. 3, May and June 1976, p. 71.

(11) Kopp, J. F., Longbottom, M. C., and Lobring, L. B., "Cold Vapor Method for Determining Mercury," American Water Works Assoc., Vol 64, Jan. 1971, p. 20.

(12) *Analytical Chimica Acta*, Vol 44, Elsevier Scientific Publishing Co., Box 211, 1000 AE, Amsterdam, Netherlands, 1969, p. 437.

(13) *Standard Methods for the Examination of Water and Wastewater*, 14th Ed., 404B, 1975, p. 285.

(14) *Standard Methods for the Examination of Water and Wastewater*, 14th Ed., Method 301A (VII), 1975, p. 159.

(15) *Atomic Absorption Newsletter*, Vol 6, 1967, p. 86.

(16) *Talanta*, Oxford/NY, 1968, p. 871.

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Standard Test Methods for Water Vapor Transmission of Materials[1]

This standard is issued under the fixed designation E 96; 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.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

NOTE—Table 1 was corrected editorially and the year date was changed on March 6, 1995.

1. Scope

1.1 These test methods cover the determination of water vapor transmission (WVT) of materials through which the passage of water vapor may be of importance, such as paper, plastic films, other sheet materials, fiberboards, gypsum and plaster products, wood products, and plastics. The test methods are limited to specimens not over 1¼ in. (32 mm) in thickness except as provided in Section 9. Two basic methods, the Desiccant Method and the Water Method, are provided for the measurement of permeance, and two variations include service conditions with one side wetted and service conditions with low humidity on one side and high humidity on the other. Agreement should not be expected between results obtained by different methods. That method should be selected which more nearly approaches the conditions of use.

1.2 The values stated in inch-pound units are to be regarded as the standard. Metric inch-pound conversion factors for WVT, permeance, and permeability are stated in Table 1. All conversions of mm Hg to Pa are made at a temperature of 0°C.

1.3 *This standard does not purport to address all of the safety problems, 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:*

C 168 Terminology Relating to Thermal Insulating Materials[2]

D 449 Specification for Asphalt Used in Dampproofing and Waterproofing[3]

D 2301 Specification for Vinyl Chloride Plastic Pressure-Sensitive Electrical Insulating Tape[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

TABLE 1 Metric Units and Conversion Factors[A,B]

Multiply	by	To Obtain (for the same test condition)
	WVT	
g/h·m²	1.43	grains/h·ft²
grains/h·ft²	0.697	g/h·m²
	Permeance	
g/Pa·s·m²	1.75×10^{7}	1 Perm (inch-pound)
1 Perm (inch-pound)	5.72×10^{-8}	g/Pa·s·m²
	Permeability	
g/Pa·s·m	6.88×10^{8}	1 Perm inch
1 Perm inch	1.45×10^{-9}	g/Pa·s·m

[A] These units are used in the construction trade. Other units may be used in other standards.

[B] All conversions of mm Hg to Pa are made at a temperature of 0°C.

3. Terminology

3.1 Definitions of terms used in this standard will be found in Terminology C 168, from which the following is quoted:

"*water vapor permeability*—the time rate of water vapor transmission through unit area of flat material of unit thickness induced by unit vapor pressure difference between two specific surfaces, under specified temperature and humidity conditions.

DISCUSSION—Permeability is a property of a material, but the permeability of a body that performs like a material may be used. Permeability is the arithmetic product of permeance and thickness.

water vapor permeance—the time rate of water vapor transmission through unit area of flat material or construction induced by unit vapor pressure difference between two specific surfaces, under specified temperature and humidity conditions.

DISCUSSION—Permeance is a performance evaluation and not a property of a material.

water vapor transmission rate—the steady water vapor flow in unit time through unit area of a body, normal to specific parallel surfaces, under specific conditions of temperature and humidity at each surface."

4. Summary of Test Methods

4.1 In the Desiccant Method the test specimen is sealed to the open mouth of a test dish containing a desiccant, and the assembly placed in a controlled atmosphere. Periodic weighings determine the rate of water vapor movement

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[1] These test methods are under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and are the direct responsibility of Subcommittee C16.33 on Thermal Insulation Finishes and Vapor Transmission.

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[2] *Annual Book of ASTM Standards*, Vol 04.06.

[3] *Annual Book of ASTM Standards*, Vol 04.04.

[4] *Annual Book of ASTM Standards*, Vol 10.01.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

through the specimen into the desiccant.

4.2 In the Water Method, the dish contains distilled water, and the weighings determine the rate of vapor movement through the specimen from the water to the controlled atmosphere. The vapor pressure difference is nominally the same in both methods except in the variation, with extremes of humidity on opposite sides.

5. Significance and Use

5.1 The purpose of these tests is to obtain, by means of simple apparatus, reliable values of water vapor transfer through permeable and semipermeable materials, expressed in suitable units. These values are for use in design, manufacture, and marketing. A permeance value obtained under one set of test conditions may not indicate the value under a different set of conditions. For this reason, the test conditions should be selected that most closely approach the conditions of use. While any set of conditions may be used and those conditions reported, standard conditions that have been useful are shown in Appendix X1.

6. Apparatus

6.1 *Test Dish*—The test dish shall be of any noncorroding material, impermeable to water or water vapor. It may be of any shape. Light weight is desirable. A large, shallow dish is preferred, but its size and weight are limited when an analytical balance is chosen to detect small weight changes. The mouth of the dish shall be as large as practical and at least 4.65 in.^{2} (3000 mm^2). The desiccant or water area shall be not less than the mouth area except if a grid is used, as provided in 12.1, its effective area shall not exceed 10 % of the mouth area. An external flange or ledge around the mouth, to which the specimen may be attached, is useful when shrinking or warping occurs. When the specimen area is larger than the mouth area, this overlay upon the ledge is a source of error, particularly for thick specimens. This overlay material should be masked as described in 10.1 so that the mouth area defines the test area. The overlay material results in a positive error, indicating excessive water vapor transmission. The magnitude of the error is a complex function of the thickness, ledge width, mouth area, and possibly the permeability. This error is discussed by Joy and Wilson.[6] This type of error should be limited to about 10 to 12 %. For a thick specimen the ledge should not exceed ¾ in. (19 mm) for a 10-in. (254-mm) or larger mouth (square or circular) or ⅛ in. (3 mm) for a 5-in. (127-mm) mouth (square or circular). For a 3-in. (76-mm) mouth (square or circular) the ledge should not exceed 0.11 in. (2.8 mm) wide. An allowable ledge may be interpolated for intermediate sizes or calculated according to Joy and Wilson.[6] A rim around the ledge (Fig. X2.1) may be useful. If a rim is provided, it shall be not more than ¼ in. (6 mm) higher than the specimen as attached. Different depths may be used for the Desiccant Method and Water Method, but a ¾-in. (19-mm) depth (below the mouth) is satisfactory for either method.

6.2 *Test Chamber*—The room or cabinet where the assembled test dishes are to be placed shall have a controlled temperature and relative humidity. The temperature chosen shall be between 70 and 90°F (21 and 32°C), and shall be maintained constant within 1°F (0.6°C). A temperature of 90°F (32°C) is recommended (Note 1). The relative humidity shall be maintained at 50 ± 2 %, except where extremes of humidities are desired, when the conditions shall be 100 ± 1°F (38 ± 0.6°C) and 90 ± 2 % relative humidity. Both temperature and relative humidity shall be measured frequently, or preferably recorded continuously. Air shall be continuously circulated throughout the chamber, with a velocity sufficient to maintain uniform conditions at all test locations. The air velocity over the specimen in feet per minute shall be, numerically, not less than ten times the permeance of the specimen expressed in perms, not exceeding a maximum of 600 ft/min (3.05 m/s). However, for designed materials with a permeance of greater than 55 perms the velocity shall be 550 ± 50 ft/min (2.80 ± 25 m/s). Suitable racks shall be provided on which to place the test dishes within the test chamber.

NOTE 1—Simple temperature control by heating alone is usually made possible at 90°F (32°C). However, it is very desirable to enter the controlled space, and a comfortable temperature is more satisfactory for that arrangement. Temperatures of 73.4°F (23°C) and 80°F (26.7°C) are in use and are satisfactory for this purpose. With cyclic control, the average test temperature may be obtained from a sensitive thermometer in a mass of dry sand. The temperature of the chamber walls facing a specimen over water should not be cooler than the water to avoid condensation on the test specimen.

6.3 *Balance and Weights*—The balance shall be sensitive to a change smaller than 1 % of the weight change during the period when a steady state is considered to exist. The weights used shall be accurate to 1 % of the weight change during the steady-state period. For example: A 1-perm (5.7×10^{-11} $kg \cdot Pa^{-1} \cdot s^{-1} \cdot m^{-2}$) specimen 10 in. (254 mm) square at 80°F (26.7°C) passes 8.6 grains or 0.56 g/day. In 18 days of steady state, the transfer is 10 g. For this usage, the balance must have a sensitivity of 1 % of 10 g or 0.1 g and the weights must be accurate to 0.1 g. If, however, the balance has a sensitivity of 0.2 g or the weights are no better than 0.2 g, the requirements of this paragraph can be met by continuing the steady state for 36 days. An analytical balance that is much more sensitive will permit more rapid results on specimens below 1 perm (5.7×10^{-11} $kg \cdot Pa^{-1} \cdot s^{-1} \cdot m^{-2}$) when the assembled dish is not excessively heavy. A light wire sling may be substituted for the usual pan to accommodate a larger and heavier load.

7. Materials

7.1 *Desiccant and Water:*

7.1.1 For the Desiccant Method, anhydrous calcium chloride in the form of small lumps that will pass a No. 8 (2.36-mm) sieve, and free of fines that will pass a No. 30 (600-µm) sieve, shall be used (Note 2). It shall be dried at 400°F (200°C) before use.

NOTE 2—If $CaCl_2$ will react chemically on the specimen, an adsorbing desiccant such as silica gel, activated at 400°F (200°C), may be used; but the moisture gain by this desiccant during the test must be limited to 4 %.

7.1.2 For the Water Method, distilled water shall be used in the test dish.

7.2 *Sealant*—The sealant used for attaching the specimen

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[6] Joy, F. A., and Wilson, H. G., "Standardization of the Dish Method for Measuring Water Vapor Transmissions," National Research Council of Canada, Research Paper 279, January 1966, p. 263.

to the dish, in order to be suitable for this purpose, must be highly resistant to the passage of water vapor (and water). It must not lose weight to, or gain weight from, the atmosphere in an amount, over the required period of time, that would affect the test result by more than 2 %. It must not affect the vapor pressure in a water-filled dish. Molten asphalt or wax is required for permeance tests below 4 perms (2.3×10^{-10} $kg \cdot Pa^{-1} \cdot s^{-1} \cdot m^{-2}$). Sealing methods are discussed in Appendix X2.

8. Sampling

8.1 The material shall be sampled in accordance with standard methods of sampling applicable to the material under test. The sample shall be of uniform thickness. If the material is of nonsymmetrical construction, the two faces shall be designated by distinguishing marks (for example, on a one-side-coated sample, "I" for the coated side and "II" for the uncoated side).

9. Test Specimens

9.1 Test specimens shall be representative of the material tested. When a product is designed for use in only one position, three specimens shall be tested by the same method with the vapor flow in the designated direction. When the sides of a product are indistinguishable, three specimens shall be tested by the same method. When the sides of a product are different and either side may face the vapor source, four specimens shall be tested by the same method, two being tested with the vapor flow in each direction and so reported.

9.2 A slab, produced and used as a laminate (such as a foamed plastic with natural "skins") may be tested in the thickness of use. Alternatively, it may be sliced into two or more sheets, each being separately tested and so reported as provided in 9.4, provided also, that the "overlay upon the cup ledge" (6.1) of any laminate shall not exceed ⅛ in. (3 mm).

9.3 When the material as used has a pitted or textured surface, the tested thickness shall be that of use. When it is homogeneous, however, a thinner slice of the slab may be tested as provided in 9.4.

9.4 In either case (9.2 or 9.3), the tested overall thickness, if less than that of use, shall be at least five times the sum of the maximum pit depths in both its faces, and its tested permeance shall be not greater than 5 perms (3.3 metric perms).

9.5 The overall thickness of each specimen shall be measured at the center of each quadrant and the results averaged. Measurement of specimens of 0.125 in. or less in thickness shall be made to the nearest 0.0001 in. Measurement of specimens greater than 0.125 in. in thickness shall be made to the nearest 0.001 in.

9.6 When testing any material with a permeance less than 0.05 perms or when testing a low permeance material that may be expected to lose or gain weight throughout the test (because of evaporation or oxidation), it is strongly recommended that an additional specimen, or "dummy," be tested exactly like the others, except that no desiccant or water is put in the dish. Failure to use this dummy specimen to establish modified dish weights may significantly increase the time required to complete the test. Because time to reach equilibrium of water permeance increases as the square of thickness, thick, particularly hygroscopic, materials may take as long as 60 days to reach equilibrium conditions.

10. Attachment of Specimen to Test Dish

10.1 Attach the specimen to the dish by sealing (and clamping if desired) in such a manner that the dish mouth defines the area of the specimen exposed to the vapor pressure in the dish. If necessary, mask the specimen top surface, exposed to conditioned air so that its exposure duplicates the mouth shape and size and is directly above it. A template is recommended for locating the mask. Thoroughly seal the edges of the specimen to prevent the passage of vapor into, or out of, or around the specimen edges or any portion thereof. The same assurance must apply to any part of the specimen faces outside their defined areas. Suggested methods of attachment are described in Appendix X2.

NOTE 3—In order to minimize the risk of condensation on the interior surface of the sample when it is placed in the chamber, the temperature of the water prior to preparation of the test specimen should be within ±2°F (±1.1°C) of the test condition.

11. Procedure for Desiccant Method

11.1 Fill the test dish with desiccant within ¼ in. (6 mm) of the specimen. Leave enough space so that shaking of the dish, which must be done at each weighing, will mix the desiccant.

11.2 Attach the specimen to the dish (see 10.1) and place it in the controlled chamber, specimen up, weighing it at once. (This weight may be helpful to an understanding of the initial moisture in the specimen.)

11.3 Weigh the dish assembly periodically, often enough to provide eight or ten data points during the test. A data point is the weight at a particular time. The time that the weight is made should be recorded to a precision of approximately 1 % of the time span between successive weighing. Thus, if weighings are made every hour, record the time to the nearest 30 s; if recordings are made every day, a time to the nearest 15 min would be allowed. At first the weight may change rapidly; later a steady state will be reached where the rate of change is substantially constant. Weighings should be accomplished without removal of the test dishes from the controlled atmosphere, but if removal is prescribed necessary, the time the specimens are kept at different conditions, temperature or relative humidity, or both, should be kept to a minimum. When results of water vapor transmission are expected to be less than .05 perm, a dummy specimen is strongly recommended. Such a dummy specimen should be attached to an empty cup in the normal manner. The environmental effects of temperature variation and buoyancy variability due to barometric pressure fluctuation can be arithmetically tared out of the weighing values. This precaution permits earlier and more reliable achievement of equilibrium conditions. Analyze the results as prescribed in 13.1.

11.4 Terminate the test or change the desiccant before the water added to the desiccant exceeds 10 % of its starting weight (Notes 1 and 3). This limit cannot be exactly determined and judgement is required. The desiccant gain may be more or less than the dish weight-gain when the moisture content of the specimen has changed.

NOTE 4—The WVT of some materials (especially wood) may depend on the ambient relative humidity immediately before the test. An apparent hysteresis results in higher WVT if the prior relative humidity was above the test condition and vice versa. It is therefore recommended that specimens of wood and paper products be conditioned to constant weight in a 50 % relative humidity atmosphere before they are tested. Some specimens may be advantageously preconditioned to minimize the moisture that the specimen will give up to the desiccant. This applies when the specimen is likely to have high moisture content or when it is coated on the top (vapor source) side.

12. Procedure for Water Method

12.1 Fill the test dish with distilled water to a level ¾ ± ¼ in. (19 ± 6 mm) from the specimen. The air space thus allowed has a small vapor resistance, but it is necessary in order to reduce the risk of water touching the specimen when the dish is handled. Such contact invalidates a test on some materials such as paper, wood, or other hygroscopic materials. The water depth shall be not less than ⅛ in. (3 mm) to ensure coverage of the dish bottom throughout the test. However, if the dish is of glass, its bottom must be visibly covered at all times but no specific depth is required. Water surges may be reduced in placing a grid of light noncorroding material in the dish to break the water surface. This grid shall be at least ¼ in. (6 mm) below the specimen, and it shall not reduce the water surface by more than 10 % (Note 4).

NOTE 5—For the Water Method, baking the empty dish and promptly coating its mouth with sealant before assembly is recommended. The water may be added most conveniently after the specimen is attached, through a small sealable hole in the dish above the water line.

12.2 Attach the specimen to the dish (see 10.1). Some specimens are likely to warp and break the seal during the test. The risk is reduced by preconditioning the specimen, and by clamping it to the dish ledge (if one is provided).

12.3 Weigh the dish assembly and place it in the controlled chamber on a true horizontal surface. Follow the procedure given in 11.3. If the test specimen cannot tolerate condensation on the surface, the dish assembly shall not be exposed to a temperature that differs by more than 5°F (2.8°C) from the control atmosphere to minimize the risk of condensation on the specimen. When results of water vapor transmission are expected to be less than .05 perm, a dummy specimen is strongly recommended. Such a dummy specimen should be attached to an empty cup in the normal manner. The environment effects of temperature variation and buoyancy variability due to barometric pressure fluctuation can be arithmetically tared out of the weighing values. This precaution permits earlier and more reliable achievement of equilibrium conditions. Analyze the results as prescribed in 13.1.

12.4 Where water is expected to be in contact with the barrier in service, proceed as in 11.3 except place the dish in an inverted position. The dish must be sufficiently level so that water covers the inner surface of the specimen despite any distortion of the specimen due to the weight of the water. With highly permeable specimens it is especially important to locate the test dish so that air circulates over the exposed surface at the specified velocity. The test dishes may be placed on the balance in the upright position for weighing, but the period during which the wetted surface of the specimen is not covered with water must be kept to a minimum.

13. Calculation and Analysis of Results

13.1 The results of the rate of water vapor transmission may be determined either graphically or numerically.

13.1.1 *Dummy Specimen*—If a dummy specimen has been used to compensate for variability in test conditions, due to temperature or barometric pressure, or both, the daily recorded weights can be adjusted by calculating the weight change from initial to time of weighing. This adjustment is made by reversing the direction of the dummy's weight change, relative to its initial weight, and modifying all the appropriate specimen weight(s) recorded at this time. This permits earlier achievement of equilibrium conditions. An alternate procedure, particulary for tests of long duration and more than six weighings, is to subtract the arithmetic mean slope of the rate of weight change of the dummy specimen from the arithmetic mean slope of each similar specimen to get an effective rate of weight change. These procedures are also desirable if the specimen is changing weight due to a curing process while under test.

13.1.2 *Graphic Analysis*—Plot the weight, modified by the dummy specimen when used, against elapsed time, and inscribe a curve which tends to become straight. Judgment here is required and numerous points are helpful. When a straight line adequately fits the plot of at least six properly spaced points (periodic weight changes matching, or exceeding 20 % of the multiple of 100 times the scale sensitivity), a nominally steady state is assumed, and the slope of the straight line is the rate of water vapor transmission.

13.1.3 *Numerical Analysis*—A mathematical least squares regression analysis of the weight, modified by the dummy specimen when used, as a function of time will give the rate of water vapor transmission. An uncertainty, or standard deviation of this rate, can also be calculated to define the confidence band. For very low permeability materials, this method can be used to determine the results after 30 to 60 days when using an analytical balance, with a sensitivity of ±1 mg, even if the weight change does not meet the 100 times the sensitivity requirement of 6.3. Specimens analyzed in this manner must be clearly identified in the report.

13.2 Calculate the water vapor transmission, WVT, and permeance as follows:

13.2.1 *Water Vapor Transmission:*

$$\mathrm{WVT} = G/tA = (G/t)/A$$

where:

In inch-pound units:

G = weight change, grains (from the straight line),
t = time during which G occurred, h,
G/t = slope of the straight line, grains/h,
A = test area (cup mouth area), ft^2, and
WVT = rate of water vapor transmission, grains/h·ft^2.

In metric units:

G = weight change (from the straight line), g,
t = time, h,
G/t = slope of the straight line, g/h,
A = test area (cup mouth area), m^2, and
WVT = rate of water vapor transmission, g/h·m^2.

13.2.2 *Permeance:*

$$\text{Permeance} = \text{WVT}/\Delta p = \text{WVT}/S(R_1 - R_2)$$

where:

In inch-pound units:

Δp = vapor pressure difference, in. Hg,

S = saturation vapor pressure at test temperature, in. Hg,

R_1 = relative humidity at the source expressed as a fraction (the test chamber for desiccant method; in the dish for water method), and

R_2 = relative humidity at the vapor sink expressed as a fraction

In metric units:

Δp = vapor pressure difference, mm Hg (1.333×10^2 Pa),

S = saturation vapor pressure at test temperature, mm Hg (1.333×10^2 Pa),

R_1 = relative humidity at the source expressed as a fraction (the test chamber for desiccant method; in the dish for water method), and

R_2 = relative humidity at the vapor sink expressed as a fraction.

13.2.3 In the controlled chamber the relative humidity and temperature are the average values actually measured during the test and (unless continuously recorded) these measurements shall be made as frequently as the weight measurements. In the dish the relative humidity is nominally 0 % for the desiccant and 100 % for the water. These values are usually within 3 % relative humidity of the actual relative humidity for specimens below 4 perms (2.3×10^{-7} $\text{g}\cdot\text{Pa}^{-1}\cdot\text{s}^{-1}\cdot\text{m}^{-2}$) when the required conditions are maintained (no more than 10 % moisture in $CaCl_2$ and no more than 1 in. (25 mm) air space above water).

13.3 Only when the test specimen is homogeneous (not laminated) and not less than ½ in. (12.5 mm) thick, calculate its average permeability (perm in.) (metric perm-cm) as follows:

$$\text{Average permeability} = \text{permeance} \times \text{thickness}$$

NOTE 6: *Example*—In a desiccant test that ran 288 h (12 days) on an exposed area of 100 in.² (0.0645 m²), it was found that the rate of gain was substantially constant after 48 h and during the subsequent 240 h, the weight gain was 12 g. The controlled chamber conditions were measured at 89.0°F (31.7°C) and 49 % relative humidity.

Required: WVT and permeance

Calculation (inch-pound units):

$G/t = 12\text{ g} \times \dfrac{15.43\text{ grains}}{\text{g}} \div 240\text{ h}$

$= 0.771$ grains/h,

$A = 100\text{ in.}^2 \times \dfrac{1\text{ ft}^2}{144\text{ in.}^2} = 0.695\text{ ft}^2,$

S = 1.378 in. Hg (from standard references tables),

R_1 = 49 % (in chamber),

R_2 = 0 % (vapor sink), and

WVT $= 0.771\text{ grains/h} \div 0.694\text{ ft}^2 = 1.11\text{ grains/ft}^2\cdot\text{h}.$

Permeance $= \text{WVT}/\Delta P = \text{WVT}/S\,(R_1 - R_2)$

$= 1.11\text{ grains/ft}^2\cdot\text{h} \div 1.378\text{ in. Hg}\,(0.49 - 0)$

$= 1.64\text{ grains/ft}^2\cdot\text{h}\cdot\text{in. Hg} = 1.64\text{ perms}$

Calculation (metric units):

$G/t = 12\text{ g}/240\text{ h} = 0.05\text{ g/h},$

$A = 0.0645\text{ m}^2,$

S = 35 mm Hg (from reference tables),

$= 35\text{ mm Hg} \times 1.333 \times 10^2\text{ Pa/mm Hg} = 46.66 \times 10^2\text{ Pa},$

R_1 = 49 % (in chamber),

R_2 = 0 % (vapor sink), and

WVT $= 0.05\text{ g/h} \div 0.0645\text{ m}^2 = 0.775\text{ g/h}\cdot\text{m}^2.$

Permeance $= \text{WVT}/\Delta P - \text{WVT}/S\,(R_1 - R_2)$

$= 0.775\text{ g/h}\cdot\text{m}^2 \times 1\text{ h}/3600\text{s} \div 46.66 \times 10^2\text{ Pa} \times (0.49 - 0)$

$= 9.42 \times 10^{-8}\text{ g/Pa}\cdot\text{s}\cdot\text{m}^2$

13.4 Metric units and conversion factor are given in Table 1.

14. Report

14.1 The report shall include the following:

14.1.1 Identification of the material tested, including its thickness.

14.1.2 Test method used (desiccant or water).

14.1.3 Test temperature.

14.1.4 Relative humidity in the test chamber.

14.1.5 Permeance of each specimen in perms (to two significant figures).

14.1.6 The side of each specimen on which the higher vapor pressure was applied. (The sides shall be distinguished as "side A" and "side B" when there is no obvious difference between them. When there is an obvious difference, this difference shall also be stated, such as "side A waxed" and "side B unwaxed.").

14.1.7 The average permeance of all specimens tested in each position.

14.1.8 The permeability of each specimen (as limited by 13.3), and the average permeability of all specimens tested.

14.1.9 Include a portion of the plot indicating the section of the curve used to calculate permeability.

14.1.10 State design of cup and type or composition of sealant.

15. Precision and Bias[7]

15.1 *Precision*—Table 2 is based on an interlaboratory tests conducted in 1988 and 1991.[7] In 1988 four materials (A, B, C, D) were tested using the dessicant method and the water method in triplicate. Fifteen laboratories contributed data, with full results secured from four laboratories. In 1991 ten laboratories contributed data for material E, using triplicate specimens, again using both the dessicant method and the water method.

15.1.1 Test results were analyzed using Practice E 691.

15.2 *Bias*—This test method has no bias because water vapor transmission of materials is defined in terms of this test method.

16. Keywords

16.1 permeability; plastics (general), plastic sheet and film; sheet material; thermal-insulating materials; thermal insulation permeability films; water vapor transmission (WVT).

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[7] Supporting data have been filed at ASTM Headquarters. Request RR: C-16-1014.

TABLE 2 Precision Results from Interlaboratory Testing

For Desiccant Method at 73.4°F:

Material	Thickness, in.	WVT (mean), perm	Repeatability[A]			Reproducibility[A]		
			S	*CV* %	*LSD*	*S*	*CV* %	*LSD*
A	0.001	0.606	0.0166	2.70	0.047	0.098	15.0	0.278
B	0.0055	0.0129	0.0028	22.1	0.008	0.0055	42.6	0.016
C	0.5	0.0613	0.0044	7.22	0.012	0.0185	30.6	0.052
D	1.0	0.783	0.0259	3.30	0.073	0.0613	7.8	0.174
E	0.014	0.0461	0.0023	4.99	0.007	0.0054	11.7	0.015

For Water Method at 73.4°F:

Material	Thickness, in.	WVT (mean), perm	Repeatability[A]			Reproducibility[A]		
			S	*CV* %	*LSD*	*S*	*CV* %	*LSD*
A	0.001	0.715	0.0134	1.95	0.039	0.156	21.9	0.44
B	0.0055	0.0157	0.0022	13.8	0.0062	0.0021	19.4	0.006
C	0.5	0.097	0.0055	5.7	0.016	0.0195	20.9	0.055
D	1.0	1.04	0.0192	1.8	0.054	0.217	20.9	0.62
E	0.014	0.0594	0.0034	5.7	0.010	0.0082	13.8	0.023

[A] For this data,

S = standard deviation,
CV = percent coefficient of variation (*S* × 100/mean), and
LSD = least significant difference between two individual test results based on a 95 % confidence level = $2\sqrt{2}S$.

[B] Material B was Teflon[8] PTFE fluorocarbon resin brand of tetrafluoroethylene. It was extremely difficult to provide a seal to this sample, which accounts for the poor repeatability.

APPENDIXES

(Nonmandatory Information)

X1. STANDARD TEST CONDITIONS

X1.1 Standard test conditons that have been useful are:

X1.1.1 *Procedure A*—Desiccant Method at 73.4°F (23°C).

X1.1.2 *Procedure B*—Water Method at 73.4°F (23°C).

X1.1.3 *Procedure BW*—Inverted Water Method at 73.4°F (23°C).

X1.1.4 *Procedure C*—Desiccant Method at 90°F (32.2°C).

X1.1.5 *Procedure D*—Water Method at 90°F (32.2°C).

X1.1.6 *Procedure E*—Desiccant Method at 100°F (37.8°C).

X2. CUP DESIGN AND SEALING METHODS

X2.1 An ideal sealing material has the following properties:

X2.1.1 Impermeability to water in either vapor or liquid form.

X2.1.2 No gain or loss of weight from or to the test chamber (evaporation, oxidation, hygroscopicity, and water solubility being undersirable).

X2.1.3 Good adhesion to any specimen and to the dish (even when wet).

X2.1.4 Complete conformity to a rough surface.

X2.1.5 Compatibility with the specimen and no excessive penetration into it.

X2.1.6 Strength or pliability (or both).

X2.1.7 Easy handleability (including desirable viscosity and thermal of molten sealant).

X2.1.8 Satisfactory sealants possess these properties in varying degrees and the choice is a compromise, with more tolerance in items at the beginning of this list for the sake of those at the latter part of the list when the requirements of 7.2 are met (Note A2). Molten asphalt or wax is required for permeance tests below 4 perms (2.6 metric perms). Tests to determine sealant behavior should include:

X2.1.8.1 An impervious specimen (metal) normally sealed to the dish and so tested, and

X2.1.8.2 The seal normally assembled to an empty dish with no specimen and so tested.

X2.2 The following materials are recommended for general use when the test specimen will not be affected by the temperature of the sealant:

X2.2.1 Asphalt, 180 to 200°F (82 to 93°C) softening point, meeting the requirements of Specification D 449, Type C. Apply by pouring.

X2.2.2 Beeswax and rosin (equal weights). A temperature of 275°F (135°C) is desirable for brush application. Pour at lower temperature.

X2.2.3 Microcrystalline wax[9] (60 %), mixed with refined crystalline paraffin wax (40 %).

X2.3 The materials listed in X2.3.1 are recommended for particular uses such as those shown in Figure X2.1. The suggested procedure described in X2.3.2 applies to an 11-3⁄8-in. (289-mm) square specimen if its permeance exceeds 4 perms (2.6 metric perms) (limited by evaporation of sealants).

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[8] Available from E. I. DuPont de Nemours & Co., Inc., Polymer Products Dept., Wilmington, DE 19898.

[9] Grade Nos. 2305 or 2310 of the Mobil Oil Corp., or their equivalent, have been found satisfactory for this purpose.

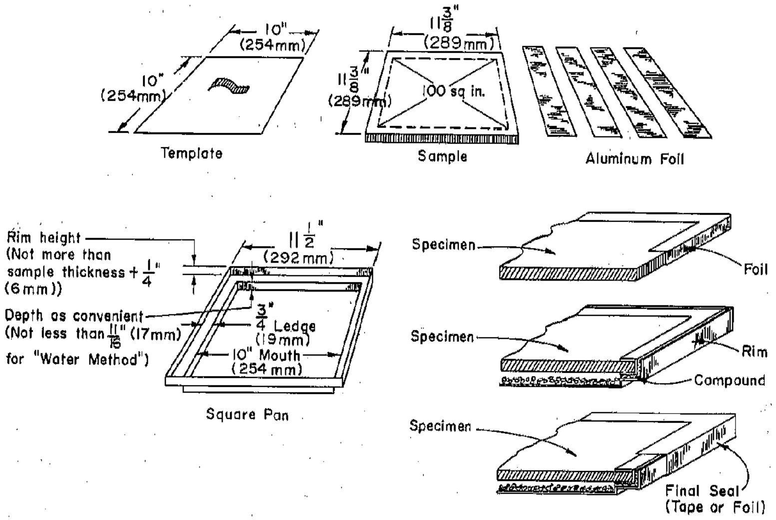

FIG. X2.1 Apparatus for Water Vapor Transmission Tests of Large Thick Specimens

X2.3.1 *Materials:*

X2.3.1.1 Aluminum foil, 0.005 in. (0.125 mm) minimum thickness.

X2.3.1.2 Tape, meeting the requirements of Specification D 2301, vinyl chloride plastic pressure-sensitive, electrical insulating tape.

X2.3.1.3 Cement, contact bond, preferably rubber base.

X2.3.2 *Procedure:*

X2.3.2.1 *Step 1*—Seal aluminum foil around edges of specimen, leaving a 100-in.^{2} (0.0654-m^2) exposed test area on each side. Use contact bond cement as directed by the manufacturer.

X2.3.2.2 *Step 2*—Spread sealant on inside of rim and ledge. Place desiccant (dry), or water and surge control material (wet) in pan. Press specimen in place. Avoid squeezing compound into the test area.

X2.3.2.3 *Step 3*—Coat outside of rim and bottom of ledge with contact bond cement, and place foil strips from edge of template, around rim, and bottom of ledge.

X2.4 A method of using hot asphalt, as applied to a 10-in. (254-mm) square-mouth dish with ledge and rim, is as follows:

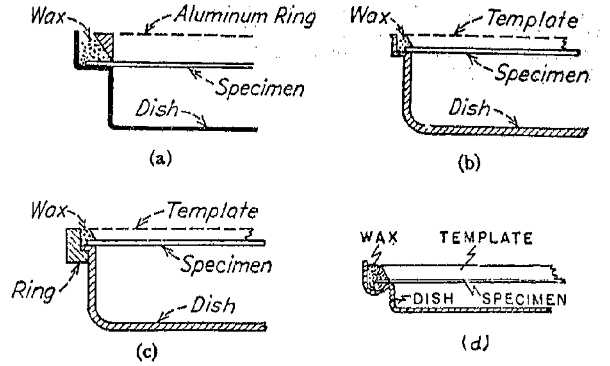

FIG. X2.2 Several Types of Dishes for Water Vapor Transmission Tests of Materials in Sheet Form

X2.4.1 *Apparatus:*

X2.4.1.1 *Template*—A square frame of brass or steel, 3/16 in. (5 mm) thick and ¾ in. (19 mm) deep. The 3/16-in. (5-mm) thickness is tapered to zero at the bottom of the frame where it will touch the test specimen and maintain a 10-in. (254-mm) square test area.

X2.4.1.2 *Sealant*—Asphalt (see X2.2.1 used at the proper pouring consistency of 375 to 450°F (179 to 232°C).

X2.4.1.3 *Melting Pot* for the asphalt, electrically heated, with one dimension greater than 11⅜ in. (289 mm).

X2.4.1.4 *Small Ladle* for pouring.

X2.4.2 *Procedure*—Mark the 11⅜-in. (289-mm) square specimen with a line at an equal distance from each edge, so that the area enclosed by the lines is as nearly as possible a 10-in. (254-mm) square. The template may be used for marking. Dip each edge of the specimen in molten asphalt up to the line, so that the test area is defined and all edges are coated with a heavy layer of asphalt. Place the specimen over the pan containing water or desiccant. Lightly oil the template or coat with petroleum jelly on its outer side, and place on the specimen. Pour molten asphalt into the space between the template and the rim of the pan. After the asphalt has cooled for a few minutes, the template should be easily removable.

X2.5 Hot wax may be applied like asphalt. It may also be applied (freely) with a small brush. Its lower working temperature may be advantageous when a specimen contains moisture.

X2.6 Several designs for dishes with supporting rings and flanges are shown in Fig. X2.2. Various modifications of these designs may be made provided that the principle of prevention of edge leakage by means of a complete seal is retained. The dishes may be constructed of any rigid, impermeable, corrosion-resistant material, provided that they can be accommodated on the available analytical balance. A lightweight metal, such as aluminum or one of its alloys, is generally used for larger-size dishes. In some cases

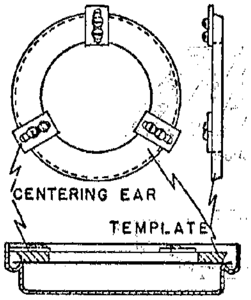

FIG. X2.3 Template Suitable for Use in Making the Wax Seals on Test Dishes

when an aluminum dish is employed and moisture is allowed to condense on its surface, there may be appreciable oxidation of the aluminum with a resulting gain in weight. Any gain in weight will ordinarily depend on the previous history of the dish and the cleanness of the surface. An empty dish carried through the test procedure as a control will help to determine whether any error may be expected from this cause. When aluminum dishes are used for the water methods, a pressure may develop inside the assembly during a test due to corrosion. This can cause seal failure or otherwise affect the result. Where this is a problem, it can be overcome by providing inside the dish a protective coating of baked-on epoxy resin or similar material. Dishes with flanges or rings that project from the inner walls of the dish are to be avoided, as such projections influence the diffusion of the water vapor. The depth of the dish for the water procedures is such that there is a 0.80 ± 0.20 in. (20 ± 5-mm) distance between the water surface and the under surface of the specimen, with a water depth of about 0.20 in. (5 mm).

X2.6.1 For the desiccant-in-dish procedures, the dishes need not be as deep as those required for the water-in-dish procedures. The desiccant is within ¼ in. (6 mm) of the under surface, and a minimum depth of only ½ in. (12 mm) of desiccant is required.

X2.6.2 The dishes shown in Fig. X2.2 require a molten seal.

X2.6.3 A template such as is shown in Fig. X2.3 is usually used for defining the test area and effecting the wax seal. It consists of a circular metal dish ⅛ in. (3.18 mm) or more in thickness with the edge beveled to an angle of about 45°. The diameter of the bottom (smaller) face of the template is approximately equal to, but not greater than, the diameter of the effective opening of the dish in contact with the specimen. Small guides may be attached to the template to center it automatically on the test specimen. A small hole through the template to admit air, and petrolatum applied to the beveled edge of the template facilitate its removal after sealing the test specimen to the dish. In use, the template is placed over the test specimen and when it is carefully centered with the dish opening, molten wax is flowed into the annular space surrounding the beveled edge of the template. As soon as the wax has solidified, the template is removed from the sheet with a twisting motion. The outside flange of the dish should be high enough to extend over the top of the specimen, thus allowing the wax to completely envelop the edge.

X2.6.4 Gasketed types of seals are also in use on appropriately designed dishes. These simplify the mounting of the specimen, but must be used with caution, since the possibility of edge leakage is greater with gasketed seals than with wax seals. Gasketed seals are not permitted for the measurement of permeance less than 44 perms (2.3×10^{-7} $g \cdot Pa^{-1} \cdot s^{-1} \cdot m^2$). As a further precaution when gasketed seals are used instead of preferred sealants, a blank test run is suggested using glass or metal as a dummy specimen.

X2.6.5 A suitable weighing cover consists of a circular disk of aluminum 1⁄32 to 3⁄32 in. (0.8 to 2.4 mm) in thickness provided with a suitable knob in the center for lifting. The cover fits over the test specimen when assembled and makes contact with the inside beveled surface of the wax seal at, or just above, the plane of the specimen. The cover is free of sharp edges which might remove the wax and is numbered or otherwise identified to facilitate its exclusive use with the same dish.

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* * *

Document Name:

CFR Section(s):

Standards Body:

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: F 1003 – 86 (Reapproved 1992)

An American National Standard

Standard Specification for Searchlights on Motor Lifeboats[1]

This standard is issued under the fixed designation F 1003; 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 specification covers searchlights for motor lifeboats.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The following precautionary caveat pertains only to the test method portion, Section 7, of this specification: *This standard does not purport to address all of the safety problems 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 Standard:*

B 117 Test Method of Salt Spray (Fog) Testing[2]

2.2 *Military Standard:*

MIL-STD-105D Sampling Procedures and Tables for Inspection by Attributes[3]

3. Descriptions of Terms Specific to This Standard

3.1 *lot*—a manufacturer's production run for a specific type of searchlight.

3.2 *order batch*—size of a specific contract or purchase order taken from the lot.

3.3 *production testing*—testing performed during a lot run of specific searchlights.

4. Materials and Manufacture

4.1 *Material:*

4.1.1 All materials used in the construction of these searchlights shall be of a quality suitable for the purpose intended and shall conform to the requirements of this specification.

4.1.2 The searchlight shall be constructed of brass, copper-alloy, an equivalent corrosion-resistant material, or a material that when tested in accordance with Method B 117 for 200 h, does not show signs of pitting, cracking, or deterioration.

4.1.3 Plastic, when used, shall be of a suitable thermoplastic or thermosetting material so molded as to produce a dense solid structure, uniform in texture, finish and mechanical properties.

5. Requirements

5.1 The height of the searchlight from the base to the top of the light shall not exceed 19 in. (483 mm).

5.2 The housing of the searchlight shall be capable of free movement of at least 60° above and 45° below the horizontal, and be able to rotate 360° in the horizontal plane. There shall be a means provided to lock the searchlight in any desired position without the use of tools (vertically and horizontally).

5.3 The searchlight shall be capable of illuminating a light colored object at night at 55 ft (180 m). The searchlight shall project a beam of light of not less than 5.5 ft (18 m) in diameter at a distance of 55 ft from the light source. The edge of the beam shall be a point where the intensity of the light is 10 % of the maximum intensity. The light source shall have a candlepower rating of no less than 350 000 cd.

5.4 The searchlight shall be capable of being operated for not less than 3 h of continuous use and 6 h of intermittent use.

5.5 The lamp utilized in the searchlight shall be of the incandescent, quartz, or other type which would allow for instant start. The lamps shall be rated for 12 V.

5.6 Each searchlight shall be watertight. The searchlight shall show no leakage of water following the test method prescribed in 7.1.

5.7 Each searchlight shall be wired with a 6-ft (2-m) length of rubber jacketed hard service flexible cord, unless otherwise specified in 8.3. The conductor size shall be no less than 16 AWG. The cable entry into the searchlight shall be sealed with a watertight bushing and packing gland. A suitable clamping device shall be installed in the area where the cables enter the gland to prevent any force being exerted on the gland or connections. The free end of this cord shall be dead-ended unless otherwise specified in 8.3.

5.8 Each searchlight shall be provided with a handle or handgrip to allow for ease of maneuvering the light into various positions.

6. Workmanship, Finish, and Appearance

6.1 Searchlights shall be of sturdy construction, and free from mechanical, electrical, or other imperfections or defects which materially affect appearance or which may affect quality, reliability, or serviceability.

6.2 The finished searchlight shall not contain rough edges, burrs, or other disfigurations and shall be clean, free from rust, tool marks, and other injurious defects.

7. Test Methods

7.1 *Watertightness*—The searchlight shall be submerged

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[1] This specification is under the jurisdiction of ASTM Committee F-25 on Shipbuilding and is the direct responsibility of Subcommittee F25.10 on Electrical, Electronics, and Automation.

Current edition approved Dec. 26, 1986. Published February 1987.

[2] *Annual Book of ASTM Standards*, Vols 02.05 and 03.02.

[3] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

in a saltwater solution (1.04 sp gr) to a depth of 3 ft (0.9 m) of 60°F (16°C) for 2 h. The light will then be subjected to the tests of 7.2.1 and be in perfect working order.

7.2 *Environmental:*

7.2.1 *Operational Test*—The searchlight shall be operated continuously for 3 h at rated voltage in an ambient temperature of 77°F (25°C) and be operational after being subjected to the watertightness test of 7.1. The searchlight shall then be operated intermittently. The intermittent time periods shall be 15 min "ON" and 5 min "OFF" for a total 6-h period. These tests shall be repeated three times.

7.2.2 *Impact:*

7.2.2.1 *Test Conditions*—The searchlight shall be placed in a cold chamber at −40 ± 5°F (−40 ± 3°C) for 2 h. With the searchlight stabilized at this temperature, it shall be immediately subjected to the low- and high-impact tests specified in 7.2.2.2 and 7.2.2.3. The point of impact shall be applied to the outside of the case at a point midway between the ends of the case at 4 points 90° apart and the back plate.

7.2.2.2 *Low Impact*—The searchlight shall be subjected to a 12 in.·lbf (1.3 J) impact using a 1-lb (0.5-kg) steel ball at each of the points of impact specified in 7.2.2.1. The searchlight shall then be subjected to the watertightness test (see 7.1). There shall be no evidence of breakage from impact and no evidence of moisture shall be found in the case.

7.2.2.3 *High Impact*—The searchlight, after passing the low-impact test, shall be again placed in the cold chamber at −40 ± 5°F (−40 ± 3°C) for 2 h and then immediately subjected to a 20 in.·lbf (2.3 J) impact using a 1-lb (0.5-kg) steel ball at each of the points of impact specified in 7.2.2.1. There shall be no evidence of damage to the case or the lens.

7.3 *Test for Light Projection*—The beam spread of the searchlight shall be shown to meet the calculations contained in the IES Lighting Handbook.[4] This test shall be conducted at rated voltage.

7.4 *Switch Endurance*—The contact switch mechanism of the searchlight shall be tested by operating the switch for 25 000 continuous cycles. A cycle shall consist of movements from "OFF" position through the full "ON" and "FLASHING" positions and back to "OFF" position. The switch shall be operated under normal electrical load conditions, and the lamp and battieres shall be replaced as often as required to ensure that the switch mechanism is operating under normal load throughout the 25 000 cycles. Burning out of bulbs and batteries during the test shall not constitute failure of this test. Failure of the switch to complete 25 000 cycles shall constitute failure of this test.

7.5 The searchlight, when mounted to a lifeboat, shall be able to withstand a vertical drop test of the lifeboat with forces of 10 *g* and a side impact test of the lifeboat with forces of 20 *g*.

8. Packaging and Package Marking

8.1 *Product Marking*—Each searchlight conforming to all the requirements in this specification shall be marked with the name, brand or trademark of the manufacturer, this ASTM specification number, and any other information as may be specified in the contract or purchase order.

8.2 *Packaging*—Unless otherwise specified by the customer in the contract or purchase order, the searchlight shall be packaged, packed, and marked in accordance with the manufacturer's commercial practice to ensure acceptance and safe delivery by the carrier for the mode of shipping and handling.

8.3 Details pertaining to the cable, cable terminations, and voltage should be provided by the procuring agency in the contract or purchase order.

9. Quality Assurance

9.1 *Responsibility for Inspection*—Unless otherwise specified in the contract or purchase order, the producer is responsible for the performance of all inspection and requirements as specified herein. Except as otherwise specified in the contract or purchase order, the producer may use his own or any other facilities suitable for the performance of the inspection requirements specified herein. The purchaser reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure the searchlights conform to the prescribed requirements.

9.2 The producer shall perform the necesary inspection and tests to assure that an S-4 inspection level, in accordance with MIL-STD-105D, is provided. An Acceptable Quality Level (AQL) of 1 % defective for the operational test and the switch leakage test, and an AQL of 4 % for any of the other requirement or test specified shall be provided. Sample testing shall be in accordance with Table 1.

TABLE 1 Sample Testing

Production	Paragraph	Order Batch	Paragraph
Watertightness	7.1	Light projection	7.3
Operation	7.2.1	Operation	7.2.1
Impact	7.2.2		
Light projection	7.3		
Switch endurance	7.4		

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[4] IES Lighting Handbook (Vol 1, Section 20), is available from Illuminating Engineering Society, 345 E. 47th St., New York, NY 10017.

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* * *

Document Name: ASTM F1006: Standard Specification for Entrainment Separators for Use in Marine Piping Applications

CFR Section(s): 46 CFR 56.60-1(b)

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: F 1006 - 86 (Reapproved 1997)

An American National Standard

Standard Specification for Entrainment Separators for Use in Marine Piping Applications[1]

This standard is issued under the fixed designation F 1006; 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 specification provides the minimum requirements for the pressure-temperature rating, testing, and making of pressure containing vessels for entrainment separators.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The following safety hazards caveat pertains only to the test methods portion, Section 6, of this specification: *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 *ANSI Standards:*

B 2.1 Pipe Threads (Except Dryseal)[2]
B 16.1 Cast Iron Pipe Flanges and Flanged Fittings[2]
B 16.3 Malleable Iron Threaded Fittings, Class 150 and 300[2]
B 16.4 Cast Iron Threaded Fittings, Class 125 and 250[2]
B 16.5 Steel Pipe Flanges and Flanged Fittings[2]
B 16.11 Forged Steel Fittings, Socket Welding and Threaded[2]
B 16.15 Cast Bronze Threaded Fittings, Class 150 and 300[2]
B 16.24 Bronze Flanges and Flanged Fittings, Class 150 and 300[2]
B 16.25 Buttwelding Ends[2]
B 16.31 Nonferrous Pipe Flanges[2]

2.2 *ASME Standards:*

SA 278 Cast Gray Iron Pressure Vessels[3]
SA 395-60 Cast Ductile Iron[3]
Boiler and Pressure Vessel Code, Section VIII[3]
Boiler and Pressure Vessel Code, Section II[3]

2.3 *Manufacturer's Standardization Society of the Valve and Fittings Industry Standard:*

MSS SP-51 150 LB Corrosion Resistant Cast Flanges and Flanged Fittings[4]

2.4 *Military Standard:*

MIL-F-1183 Fittings Tube, Bronze, Cast (Silver Brazings)[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *entrainment separator*—a mechanical device inserted in a pipeline which by centrifugal force, baffles, or other means will separate a liquid from a gas (vapor).

3.1.2 *hydrostatic test*—the act of filling an entrainment separator vessel with water and applying internal pressure to all parts of the vessel.

3.1.3 *master gage*—the calibrated gage used to verify the accuracy of the test gage. This gage shall be recalibrated traceable to the National Bureau of Standards.

3.1.4 *pressure rating*—the maximum working pressure of an entrainment separator when operated at a specific temperature.

3.1.5 *proof test*—the act of filling an entrainment separator vessel with water and applying internal pressure to all parts of the vessel for the purpose of causing yielding of the vessel and bursting of the vessel.

3.1.6 *temperature ratings*—minimum and maximum temperatures at which the entrainment separator may be operated while at specific pressures.

3.1.7 *test gage*—the pressure gage that is used to check the internal pressure of the entrainment separator. The test gage shall be calibrated at least annually or at any time it is suspected to be in error by a calibrated master gage.

4. Materials and Manufacture

4.1 The pressure-temperature ratings established under this specification are based upon the manufacturer's usage of high quality materials produced under regular control of chemical and physical properties by a recognized process. The manufacturer shall be prepared to submit certification of compliance, verifying that his product has been so produced and that it has been manufactured from material with chemical and physical properties at least equal to the requirements of the appropriate standard or specification listed in 4.3 of this specification or Section II of the ASME Boiler and Pressure Vessel Code.

4.2 For materials not having values of allowable stress tabulated in Section VIII Division I of the ASME Boiler and Pressure Vessel Code, allowable stresses shall be determined in accordance with the procedures outlined in Appendix P of Section VIII, Division 1 of the ASME Boiler and Pressure Vessel Code.

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[1] This specification is under the jurisdiction of Committee F-25 on Ships and Marine Technology and is the direct responsibility of Subcommittee F25.13 on Piping Systems.

Current edition approved July 25, 1986. Published September 1986.

[2] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[3] Available from American Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017.

[4] Available from Manufacturer's Standardization Society of the Valve and Fittings Industry, 127 Park St., N.E. Vienna, VA 22180.

[5] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

4.3 Materials of construction shall be suitable for the service intended.

4.4 Bolting materials shall be at least equal to those listed in Table 1B of ANSI B16.5. Bolting materials shall not be used beyond temperature limits specified in the governing codes.

5. Requirements

5.1 Entrainment separators covered in this specification shall be designed according to the lowest pressure-temperature rating of any individual component, or as established by proof tests.

5.2 The design pressure-temperature of entrainment separators covered in this specification will be established by the manufacturer using one of the following methods:

5.2.1 Design calculations in accordance with the requirements prescribed in the ASME Boiler and Pressure Vessel Code, Section VIII, Division 1, Part UG of Subsection A and the applicable part of Subsection C.

5.2.2 Proof test in accordance with the requirements of UG 101 (m), UCI-101, or UCD-101 of Section VIII, Division 1 of the ASME Boiler and Pressure Vessel Code.

5.2.3 Where any part of the entrainment separator vessel cannot be designed within the scope of the ASME Boiler and Pressure Vessel Code, Section VIII, Division 1, Design Section, the pressure-temperature rating must be determined through proof and hydrostatic tests using the following formulas to determine the allowable operating limits of pressure and temperature. Operator safety should be considered when conducting these tests.

$P = (P_{HT}/4) \times (S_1/S_2)$ (For Steel Vessels)

$P = (P_{HT}/4) \times (T_1/T_2)$ (For Cast Iron Vessels)

where:

P = maximum allowable working pressure (psig) at design temperature,

P_{HT} = hydrostatic test pressure (psig) at test temperature,

S_1 = stress value at design temperature (psi),

S_2 = stress value at test temperature (psi),

T_1 = specified minimum tensile strength (psi), and

T_2 = actual tensile strength test specimen (psi).

5.2.3.1 Stress values S_1 and S_2 are determined from Section VIII, of the ASME Boiler and Pressure Vessel Code.

5.2.3.2 The value of P_{HT} to be used in determining the maximum allowable working pressure shall be the maximum pressure to which the entrainment separator was subjected to without permanent deformation or rupture.

5.2.3.3 Test water temperature[6] and entrainment separator temperature must be at equilibrium before hydrostatic test pressure is applied.

5.2.3.4 All possible air pockets must be purged while the entrainment separator vessel is being filled with water. Adequate vents shall be provided at all high points of the vessel.

5.2.3.5 External equipment not to be pressurized with the entrainment separator should be isolated or disconnected before applying the hydrostatic test pressure.

5.2.3.6 Hydrostatic test pressure shall be applied gradually to the entrainment separator and held stationary at each increment for a sufficient time in order that visual inspection can be made for leaks or deformation of the vessel. The final value of hydrostatic test pressure that is not in conflict with 5.2.3.2 is called P_{HT}.

5.3 Pressure-temperature rating and construction of all pipe connections shall be in accordance with the following standards or specifications: ANSI B2.1, ANSI B16.1, ANSI B16.3, ANSI B16.4, ANSI B16.5, ANSI B16.11, ANSI B16.15, ANSI B16.24, ANSI B16.25, ASME SA-278, ANSI B16.31, MSS SP-51, and MIL-F-1183.

6. Test Methods

6.1 All entrainment separators must be pressure tested in accordance with the following:

6.1.2 Each entrainment separator shall be tested by subjecting it to an internal hydrostatic test procedure, which at every point in the separator is at least equal to 1.5 times the maximum allowable working pressure, multiplied by the lowest ratio of the stress value for the design temperature.

$$\text{Test Pressure} = 1.5 \times \text{maximum allowable pressure} \times \frac{\text{stress value at test temperature}}{\text{stress value at design temperature}}$$

6.1.2.1 The hydrostatic test pressure shall be held stationary for a suitable time necessary for observation and inspection of the separator. The minimum time of test shall be no less than 1 min.

6.1.2.2 A test gage, as defined in this specification (see Section 3) shall be connected directly to the entrainment separator.

6.1.2.3 For compliance with ASME Codes leading to ASME certification proceed with steps as outlined in Section VIII, Division 1 of the ASME Boiler and Pressure Vessel Code for Standard Hydrostatic Tests.

7. Packaging and Package Marking

7.1 Each entrainment separator shall have a securely attached nameplate or other permanent marking indicating the following:

7.1.1 Manufacturer's name and trademark,
7.1.2 Pressure-temperature rating,
7.1.3 Manufacturer's serial number,
7.1.4 Year built,
7.1.5 Size (end connection pipe size),
7.1.6 Flow direction,
7.1.7 National board number, where applicable,
7.1.8 ASME code stamp, where applicable, and
7.1.9 ASTM designation and year of issue.

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[6] Test water temperature to be no less than 60°F (10°C), but not to exceed 125°F (52°C).

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CFR Section(s):

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Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: F 1007 – 86 (Reapproved 1996)$^{\epsilon1}$

An American National Standard

Standard Specification for Pipe-Line Expansion Joints of the Packed Slip Type for Marine Application[1]

This standard is issued under the fixed designation F 1007; 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.

$^{\epsilon1}$ NOTE—Keywords were added editorially in November 1996.

1. Scope

1.1 This specification covers the design, manufacturing, and testing of packed slip type expansion joints utilized in pipe lines for accommodating axial thermal growth or contraction from the pipe-line carrying fluid.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 *ASTM Standards:*

A 53 Specification for Pipe, Steel, Black and Hot-Dipped, Zinc-Coated Welded and Seamless[2]

A 216/A 216M Specification for Steel Castings, Carbon, Suitable for Fusion Welding, for High-Temperature Service[3]

A 285/A 285M Specification for Pressure Vessel Plates, Carbon Steel, Low- and Intermediate-Tensile Strength[4]

B 650 Specification for Electrodeposited Engineering Chromium Coatings on Ferrous Substrates[5]

2.2 *ANSI Standards:*

B 16.5 Steel Pipe Flanges and Flanged Fittings[6]

B 16.25 Buttwelding Ends[6]

B 31.1 Power Piping[6]

2.3 *ASME Standards:*

Section V Nondestructive Examination[7]

Section VIII, Division 1 Pressure Vessels[7]

Section IX Welding and Brazing Qualifications[7]

2.4 *AISI Standard:*

C-1018 Carbon Steel[8]

3. Classification

3.1 The expansion joints shall be of the following types, styles, classes and forms:

3.1.1 *Type I*—Injectable semi-plastic packing type, designed for injecting packing under full line pressure.

3.1.2 *Style I*—Internally-externally guided with guides integral with stuffing box.

3.1.3 *Style II*—Internally-externally guided with guides integral with stuffing box and with low-friction inserts at the guide surfaces.

3.1.4 *Class I*—Single joint, single slip.

3.1.5 *Class II*—Double joint, double slip.

3.1.6 *Form I*—Weld end.

3.1.7 *Form II*—Flanged end.

3.1.8 *Form III*—Other.

4. Ordering Information

4.1 Expansion joints shall meet all the requirements of the latest issue of this specification. Where possible, the expansion joint shall be the manufacturer's standard commercial product. Additional or superior features that are not prohibited by this specification but which are a part of the manufacturer's standard product, shall be included with the expansion joint being offered. A standard commercial product is a product that has been sold or is currently being offered for sale on the commercial market through advertisements or manufacturer's catalogs, or brochures, and represents the latest production model.

4.2 Purchase order or inquiry for expansion joints to this specification shall specify the following:

4.2.1 Title, number, and latest revision of this specification.

4.2.2 Style, class, and form required.

4.2.3 Materials, other than standard as specified (see Section 8).

4.2.4 Service conditions shall specify the following:

4.2.4.1 Maximum and minimum operating temperature, (°F).

4.2.4.2 Maximum operating pressure, (psig).

4.2.4.3 Fluid handled.

4.2.4.4 Corrosive conditions, if applicable.

4.2.5 Total axial expansion or contraction.

4.2.6 ANSI pressure class, facing and drilling for flanged end joint and pipe schedule or wall thickness of ends for weld end joint.

4.2.7 If base is required for support or main anchor on Class I expansion joint.

4.2.8 Drain connection, if required.

4.2.9 Service connection if required, noting location and type of connection.

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[1] This specification is under the jurisdiction of Committee F-25 on Ships and Marine Technology and is the direct responsibility of Subcommittee F25.13 on Piping Systems.

Current edition approved July 25, 1986. Published September 1986.

[2] *Annual Book of ASTM Standards*, Vol 01.01.

[3] *Annual Book of ASTM Standards*, Vol 01.02.

[4] *Annual Book of ASTM Standards*, Vol 01.04.

[5] *Annual Book of ASTM Standards*, Vol 02.05.

[6] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[7] Available from American Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017.

[8] Available from American Iron and Steel Institute, 1000 N.W. 16th St., Washington, DC 20036.

4.2.10 Slip protectors, if required.

4.2.11 Adjustment rods for field extension or compression of the slip if required.

4.2.12 If hot-dip galvanizing of parts is required.

4.2.13 Spare parts, accessories, and special tools, if required.

4.2.14 Hydrostatic testing and test pressure, if required.

4.2.15 Radiographic or other nondestructive testing of weld joints if required.

5. Materials and Manufacture

5.1 *Materials:*

5.1.1 Materials of construction shall conform to the requirements as specified in this specification (see Section 8) and shall be new and free from defects that would adversely affect the performance of individual components or assemblies.

5.1.2 As specified in this specification and as required, the expansion joint shall be provided with flanged or welded end connections, limit stops, stuffing boxes with integral guides, base, drain connection, service connection, slip protectors, and adjustment rods.

5.2 *Manufacture:*

5.2.1 *General*—Unless otherwise required by this specification, the manufacturer's standard shop practices for the fabrication of the expansion joint is acceptable, provided these practices conform to the requirements and recommendations of this specification.

5.2.2 *All Welding*—Welding procedure qualifications, welder performance qualification, welding materials, preheat, and post weld heat treatment if required, shall be in accordance with ANSI B31.1 and ASME Code Section IX.

5.2.3 *Identification*—Each completed expansion joint shall have a name plate made from a corrosion resistant material permanently attached showing as a minimum the following:

5.2.3.1 Manufacturer's model number and joint size,

5.2.3.2 Design pressure and design temperature range,

5.2.3.3 Nominal traverse or movement per slip,

5.2.3.4 Type of packing and service fluid, and

5.2.3.5 Date of manufacture.

6. Joint Descriptions

6.1 *Styles:*

6.1.1 *Style I*—The slip of the expansion joint shall be guided with the body of the expansion joint by internal and external guides that are integral with the stuffing box. Semi-plastic packing shall be injected into the stuffing box and may be contained within the stuffing box chamber by ring type packing. All packing shall be of the self-lubricating type. The expansion joint and semi-plastic packing shall be suitable for the safe injection of the packing under full line pressure to stop leakage. Provisions for packing injection shall be by devices located radially about the stuffing box and designed to permit a maximum evacuation of packing at the bottom of the packing device when the injector is fully engaged. The number of devices with injectors for each stuffing box shall be in accordance with the manufacturer's standard practice. The design of the packing injection devices shall be such as to ensure no blowback of injectable packing or the service fluid when injecting packing under full line pressure.

6.1.2 *Style II*—The expansion joint shall be the same as Style I except low friction corrosion resistant material or inserts shall be used for internal and external guiding to prevent slip scoring from pipe misalignment, or vibration and to prevent corrosion of the guide surfaces.

6.2 *Classes:*

6.2.1 *Class I*—The expansion joint shall have slip at only one end of the joint. Class I expansion joints may or may not require a base.

6.2.2 *Class II*—The expansion joint shall have slip at both ends of the joint. Class II expansion joints shall require a base.

6.3 *Forms:*

6.3.1 *Form I—Welded End:* The expansion joint shall have provisions at each end of the Joint for field welding to the adjoining pipe line.

6.3.2 *Form II—Flanged End:* The expansion joint shall have flanges at each end of the joint for bolting to the mating flanges of the adjoining pipe line.

6.3.3 *Form III*—The expansion joint shall have other end connections as specified for attaching to the adjoining pipe line.

7. Design

7.1 The expansion joint shall be designed to conform to applicable sections of the latest edition of ANSI B31.1 and other applicable documents as noted in Section 2.

7.2 *Compresson Force*—Unless otherwise specified, the force to compress or extend the slip of the expansion joint shall not exceed 1000 lbf/in. (175 100 N/m) of nominal pipe diameter.

8. Construction

8.1 *Slip*—The slip shall be manufactured from steel pipe conforming to Specification A 53, Grade B, or a rolled and welded cylinder from Specification A 285/A 285M, Grade C plate, or equal with the longitudinal weld seam 100 % radiographed. The minimal wall thickness of the pipe or rolled cylinder shall be equivalent to Schematic 80 pipe for all sizes to 14 in. (356 mm), inclusive and Schematic 60 for sizes 16 to 24 in. (406 to 610 mm) to preclude slip collapse due to external loading of the injectable packing. Heavier wall pipe may be required for expansion joints subjected to pressures above 600 psig and for pipe sizes above 24 in. (610 mm) diameter.

8.1.1 *Chrome Plate*—The slip of the expansion joints shall be chrome plated with engineering chrome in accordance with Specification B 650, Class 50.

8.2 *Stuffing Box*—The stuffing box with integral internal and external guides shall be machined from Specification A 53, Grade B Pipe, or AISI C-1018 seamless tubing or cast steel in accordance with A 216/A 216M, Grade WCB. A rolled and welded cylinder from Specification A 285/-A 285M, Grade C plate, or equal may be used provided the longitudinal weld seam is 100 % radiograph examined in accordance with ANSI B31.1 and ASME Code Section V.

8.3 *Traverse Chamber*—This chamber, also referred to as the "E.J. Body," shall be machined from Specification A 53, Grade B seamless steel pipe having a wall thickness suitable for the design service conditions. A rolled and welded cylinder from Specification A 285/A 285M, Grade C, or equal steel plate of suitable thickness for the design service conditions may also be used if the longitudinal weld seam is 100 % radiograph inspected to ANSI B31.1 and ASME Code Section V.

8.4 *End Connections*—The end connections of the expansion joint (see Section 6) shall be flanged, weld end, combination of both or others as specified. Flanges shall conform to ANSI B16.5 and be of the size, material, pressure class, and facing specified. Weld ends shall be beveled for welding to conform to ANSI B16.25 and be of the size, material and nominal wall thickness as specified for the mating pipe.

8.4.1 *Body End Connection for Class I Expansion Joints:*

8.4.1.1 *Form I—Weld End:* The reduction of the body size to the pipe line size shall be accomplished by a formed reduction of the traverse chamber (body) or by a reducer butt welded to the traverse chamber (body) that meets the requirements of ANSI B31.1 and the ASME Code Section VIII, Division 1.

8.4.1.2 *Form II—Flanged End:* Forged flanges shall be attached to the body by butt welding only and shall be in accordance with the requirements of ANSI B31.1 and the ASME Code Section VIII, Division 1.

8.5 *Limit Stops*—Limit stops may be of the external or internal type manufactured from a suitable material and designed to withstand the full line pressure thrust load in the event of a pipeline anchor failure.

8.6 *Internal and External Guides*—See 8.2.

8.6.1 *Low Friction Internal and External Guides*—The low friction internal and external guides or inserts shall be made from noncorrosive materials with appropriate differential hardness to also prevent scoring or binding of the slip.

8.7 *Base:*

8.7.1 *Expansion Joint Without a Service Connection*—When a base is specified for Class I expansion joints, the base shall be designed for use as a main anchor. Class II expansion joints shall have a base that is suitable as an intermediate anchor. The base shall be of cast or fabricated steel that conforms to the applicable ASTM standard and shall be suitably attached to the joint and drilled in accordance with the manufacturer's standard practice.

8.7.2 *Expansion Joint With a Service Connection*—Class I and Class II expansion joints with a service connection shall be provided with a main anchor base and all anchor loading data (forces and moments) shall be made available to the manufacturer to assure adequate anchor design. The base shall be of cast or fabricated steel that conforms to the applicable ASTM standard and shall be suitably attached to the joint and drilled in accordance with the manufacturer's standard practice.

8.8 *Service Connection*—Unless otherwise specified, a service connection, when required shall be the manufacturer's standard design and shall meet the applicable requirements of the ASTM, ANSI, and ASME Codes. The service connection shall be supplied with end connections as specified (See 8.5).

8.9 *Drain Connection*—When required, a drain connection shall be attached to the body of the expansion joint and shall be the manufacturer's standard unless otherwise specified. The drain connection shall meet the requirements for fitting connections as specified in ANSI B31.1.

8.10 *Packing*—Unless otherwise specified, the packing shall be the manufacturer's standard type packing and shall be suitable for the specified service conditions.

8.11 *Other Materials of Construction*—Where the expansion joint is to transfer corrosive fluids, or be installed in a corrosive atmosphere, corrosive resistant materials as specified may be substituted for the carbon steel components, especially the sliding slip.

9. Drawing Requirements

9.1 *Drawings*—When specified, dimensional sketches or drawings sufficiently detailed to describe the expansion joint to be supplied, shall be submitted with bid proposals. The inch-pound system of measurements shall be used to dimension drawings. Drawings shall note that the design of the components or products is in full compliance with this specification.

10. Cleaning and Surface Preservation

10.1 *Cleaning*—The internal and external surfaces of the expansion joint shall be cleaned of dirt, oil, grease, and other foreign material using a suitable cleaning solvent. Extreme care shall be utilized to ensure the interior is free of any slag, steel chips, or other similar materials that could lodge between the slip and the body and score the slip surface.

10.2 *Surface Preservation*—Unless specified, no preservation will be required on the internal surface of the expansion joint. All external surfaces except the chrome plated slip surface, flanged faces, and weld bevel surfaces shall be painted in accordance with the manufacturer's standard practice. Weld end joint surfaces for field welding shall be coated with "deoxaluminate" preservative.

11. Packaging and Package Marking

11.1 All openings shall be suitably sealed to protect the opening connection surfaces and prevent entrance of foreign materials.

11.2 Unless otherwise specified, the exposed chrome plated surface of the slips shall be protected in accordance with the manufacturer's standard practice.

11.3 Unless otherwise specified, the completed expansion joints shall be suitably attached with steel strapping or hold down bolting, to wood skids or crates in accordance with standard commercial practice for domestic shipments.

12. Quality Assurance

12.1 *Inspection*—Unless otherwise specified the contractor shall perform inspections as required to assure compliance with this specification. The procuring agency may establish inspection requirements, and it is the contractor's responsibility to provide access to his facilities for the procuring agency's inspection of material, work in process, and quality assurance testing or results as required.

12.2 *Material Certification*—Certified material test reports or certificates of compliance shall be required for all pressure retaining material. To maintain traceability, all pressure retaining material shall be metal stamped or otherwise suitably marked with heat treat numbers or other identification codes. If metal stamping is used, the indentations shall not exceed 1/32 in. (0.8 mm) or infringe on minimum wall thickness. A round-nose, low-impression type stamping die shall be used.

12.3 *Nondestructive Testing*—When required all nondestructive testing shall be performed by qualified personnel in accordance with qualified acceptable procedures.

12.3.1 *Radiography*—Radiographic examination of welds shall be in accordance with ANSI B31.1 and Secton V of the ASME Code.

12.3.2 *Visual Examination, Magnetic Particle, and Liquid Penetrant Examination*—Whenever required, these examinations shall be in accordance with ANSI B31.1 and Section V of the ASME Code.

13. Keywords

13.1 axial thermal growth; expansion joint; fluid pipe line; marine technology; packed slip expansion joint; pipe line; ship

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

Document Name:	ASTM F1014: Standard Specification for Flashlights on Vessels
CFR Section(s):	46 CFR 35.30-20(c)(3)
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Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: F 1014 – 92

An American National Standard

Standard Specification for Flashlights on Vessels[1]

This standard is issued under the fixed designation F 1014; 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.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This specification covers three types of flashlights (see Section 4).

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The following precautionary caveat pertains only to the test method portion, Section 9, of this specification: *This standard does not purport to address all of the safety problems, 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 *Military Standard:*

MIL-STD-105D Department of Defense Standard; Sampling Procedures and Tables for Inspection by Attributes[2]

2.2 *UL Standard:*

UL Standard No. 783[3]

3. Terminology

3.1 *Descriptions of Terms Specific to This Standard:*

3.1.1 *lot*—a manufacturers production run for a specific type of flashlight.

3.1.2 *order batch*—the size of a specific contract or purchase order taken from the lot.

3.1.3 *production testing*—the testing performed during a lot run of specific flashlights.

4. Classification

4.1 The three types of flashlights covered in this specification are classified as follows:

4.1.1 *Type I*—Flashlights for use in lifeboats and liferafts.

4.1.2 *Type II*—Flashlights for use in hazardous locations where fire or explosion hazards may exist due to the presence of flammable gases or vapors, flammable liquids, combustible dust, or ignitable fibers or flyings.

4.1.3 *Type III*—Flashlights for use in lifeboats and liferafts and suitable for hazardous locations.

4.2 Type I flashlights shall be manufactured in accordance with Sections 2 through 11. Type II flashlights shall meet the requirements in Sections 11 and 12. Type III flashlights shall meet the requirements in Sections 2 through 12.

5. Materials and Manufacture

5.1 *Materials*—All materials used in the construction of these flashlights shall be of a quality suitable for the purpose intended and shall conform to the requirements of this specification.

5.2 *Manufacture*—Plastic, when used, shall be a suitable thermoplastic or thermosetting material so molded as to produce a dense solid structure, uniform in texture, finish, and mechanical properties.

6. General Requirements

6.1 Each flashlight shall be a three-cell light.

6.2 Each flashlight shall provide a concentrated beam of light. When used in this specification, a concentrated beam of light is light projected in a nearly parallel beam and is used to illuminate objects at considerable distances.

6.3 The flashlight shall show no leakage of water and shall be in perfect working order following the test prescribed in 9.2.

6.4 The proportions and design of each flashlight shall be such that the assembled unit complete with lamp and cells shall be capable of withstanding, without breakage or material distortion of any part and without upsetting the lamp focus, the test specified in 9.6. Damage of lamp filament or shifting of filament within the lamp as a result of this test shall not be cause for rejection of the flashlight if the flashlight operates when the spare lamp is used.

6.5 Each flashlight must be furnished with two lamps. One of these lamps is a spare and must be contained within the body of the flashlight, either in the end cap or reflector head area.

6.6 All metal parts of each flashlight shall be made of corrosion-resistant material. All copper or copper-alloy parts coming in contact with rubber shall not corrode or disintegrate the rubber.

6.7 The construction of each flashlight shall be such that a metallic contact outside the case cannot close the battery circuit and cause the light to come on.

6.8 Each flashlight shall be provided with means to prevent it from rolling.

7. Physical Requirements

7.1 *Dimensions*—Each flashlight shall not exceed 11½ in.

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[1] This specification is under the jurisdiction of ASTM Committee F-25 on Shipbuilding and is the direct responsibility of Subcommittee F25.10 on Electrical, Electronics, and Automation.

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[2] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

[3] Available from Underwriters Laboratories, 333 Pfingsten Rd., Northbrook, IL 60062.

(292 mm) in length and shall have a lens ring diameter of no more than 2½ in. (63.5 mm).

7.2 *Case, Lens Head Ring, and End Cap:*

7.2.1 The flashlight case must be suitable to receive three commercial D-size dry cells. The exterior of the flashlight shall have molded flutes or ribs to provide a satisfactory gripping surface when wet. Cells must be held in the case under sufficient pressure to ensure good contact and to prevent breaking of the circuit when the flashlight is tested in accordance with 9.1.2. The flashlight must be constructed so that the cells are readily replaceable.

7.2.2 The case, ring, and cap, need not be made of the same material but cooperating threaded surfaces should be of the same type material.

7.3 *Lens*—The lens shall be of good quality, laminated, shatter-proof glass or clear plastic, free from bubbles, striae, wrinkles, or other defects and blemishes that would affect the light distribution. The lens shall be secured to the lens head ring by a suitable retaining ring or other device so as to prevent it from being dropped when the lens head ring is unscrewed.

7.4 *Reflector*—The reflector may be of metal or plastic and must have sufficient rigidity so as not to become distorted when the flashlight is completely assembled with cells and lamp. It must have an essentially parabolic reflecting surface, highly polished and resistant to corrosion and discoloration. The reflector shall be 1¾ ± ¼ in. (44 ± 6 mm) in diameter.

7.5 *Lamps*—The lamps shall be appropriate for use with alkaline or carbon-zinc batteries. Each lamp must have a lamp life of at least 15 h at the rated battery voltage.

7.6 *Lamp Holder*—The lamp shall be firmly positioned in the reflector. It shall be so positioned that the lamp, when assembled in the holder, shall have its filament located at the focus of the reflector, with the accuracy necessary to produce a concentrated beam of light as specified in 9.7. It shall have sufficient rigidity so as not to become distorted when the flashlight is completely assembled with cells and lamp. Means shall be provided to protect the lamp from damage by battery impact and for conveniently removing and replacing the lamp. The mounting adjustment shall have sufficient stability to retain the focal adjustment of the lamp under a condition of vibration, and when the flashlight is tested in accordance with 9.1.2.

7.7 *Switch*—The switch shall be conveniently located and securely attached to the flashlight. It shall be provided with a permanent "ON" position, a manually operable signaling or "FLASHING" position, and a locked "OFF" position. It shall be provided with means to prevent accidental closing of the lamp circuit. The switch parts and switch contact strip shall be adequately insulated so that it will be impossible for metal-clad cells to close a circuit when the switch is on the "OFF" position. The flashlight shall be designed with a switch guard. Contact springs shall be of phosphor bronze, spring brass, or other corrosion-resistant equivalent material.

7.8 *Suspension Member*—Each flashlight shall be equipped with a suitable suspension ring or clip. The ring or clip shall be tested as specified in 9.8.

8. Workmanship

8.1 Flashlights shall be of sturdy construction and free of mechanical, electrical, or other imperfections or defects that materially affect appearance or that may affect quality, reliability, or serviceability. The finished flashlight shall not contain rough edges, burrs, blemishes, or other disfigurements and shall be clean, free from rust, toolmarks, pits, and other injurious defects.

8.1.1 Threaded parts shall be smooth and close fitting and shall be capable of being easily moved by hand relative to each other. Threaded parts shall not jump or change adjustment when being put together or when subjected to the tests in Section 9.

9. Test Methods

9.1 *Switches:*

9.1.1 *Switch Leakage*—With the lamp and reflector removed, insert fresh batteries into the flashlight. Connect a voltmeter across the switch and battery or the switch and battery contacts, as appropriate, in such a way as to read the battery voltage through the switch. With the switch in the "OFF" position, read the voltage. Any distinguishable deflection of the meter hand when the meter is set in the voltage range, nearest the battery voltage, shall constitute failure of this test.

9.1.2 *Operation*—Insert batteries into the flashlight and operate the switch five times in each of the three switch positions. Shake the flashlight vigorously in each "ON" and "FLASHING" switch position. The flashlight must not go off when shaken vigorously while the switch is in the "ON" or "FLASHING" position.

9.1.3 *Switch Endurance*—Test the contact switch mechanism of the flashlight by operating the switch for 25 000 continuous cycles. A cycle shall consist of movements from "OFF" position through the full "ON" and "FLASHING" positions and back to "OFF" position. Operate the switch under normal electrical load conditions, and replace the lamp and batteries as often as required to ensure that the switch mechanism is operating under normal load throughout the 25 000 cycles. Burning out of bulbs and batteries during the test shall not constitute failure of this test. Failure of the switch to complete 25 000 cycles shall constitute failure of this test.

9.2 *Watertightness*—Test the flashlight as follows:

9.2.1 Submerge the assembled flashlight in a salt water solution (1.04 sp gr) under a head of 1 ft (0.3 m) for a period of 24 h at a water temperature of 65°F (18°C). Remove the flashlight and wipe off the excess water. The total water absorption shall not exceed 5 % weight. The flashlight shall be capable of being disassembled and reassembled without undue difficulty upon completion of the test and shall be in perfect working order.

9.3 *Impact:*

9.3.1 *Test Conditions*—Place the flashlight, without batteries installed, in a cold chamber at −40 ± 5°F (−40 ± 3°C) for 2 h. With the flashlight stabilized at this temperature, immediately subject it to the low- and high-impact test specified in 9.3.2 and 9.3.3. Apply the point of impact to the following:

9.3.1.1 The outside of the flashlight case at a point midway between the ends of the case on a side 90° from the switch, 9.3.1.2 The switch (in the "ON" position and in the "OFF" position),

9.3.1.3 The lens cap, and

9.3.1.4 The end cap.

9.3.2 *Low Impact*—Subject the flashlight to a 12-lbf·in. (1.4 N·m) impact using a 1-lb (0.4-kg) steel ball at each of the points of impact specified in 9.3.1. Provided the flashlight remains intact, next subject the flashlight to the watertightness test (see 9.2). There shall be no evidence of breakage from impact or moisture in the case.

9.3.3 *High Impact*—After the flashlight passes the low-impact test, again place the flashlight in the cold chamber at −40°F ± 5°F (−40 ± 3°C) for 2 h, and then immediately subject it to a 20 lbf·in. (2.3 N·m) impact using a 1-lb (0.4-kg) steel ball at each of the points of impact specified above. There shall be no evidence of damage to the case, the lens, or the end cap.

9.4 *Environmental:*

9.4.1 *Heat and Humidity*—Place the flashlight, with dry cells, on a horizontal surface and subject to dry heat at 150 ± 5°F (65.6 ± 3°C) for 16 h, followed by an 85 ± 5 % relative humidity at 100 ± 2°F (38 ± 1°C) for 6 h. The flashlight shall be compared with untested flashlights for dimensional stability, crazing of surface, and then tested to determine that the flashlight operates in accordance with 9.1.2.

9.5 *Corrosion*—Subject the flashlights, without dry cells, to salt spray for 200 h. Then wash the flashlight with fresh water, dry, and then test to determine that the flashlight operates in accordance with 9.1.2. There shall be no evidence of corrosion.

9.6 *Rough Use*—Drop the flashlight, complete with dry cells, lamps, and lens, 5 ft (1.5 m) in free fall onto a vinyl-asbestos tiled concrete floor. Drop the flashlight twice in a horizontal position upon the switch, with the switch in the "ON" position, twice in a horizontal position upon the switch, with the switch in the "OFF" position, twice in a vertical position upon the head of the flashlight, and upon the base of the flashlight. Do not tighten parts once the test has begun. There shall be no evidence of damage to any part of the flashlight. Then test the flashlight in accordance with 9.1.3.

9.7 *Light Projection*—The flashlight must project a concentrated beam of light not less than 5 in. (127 mm) nor more than 11 in. (279 mm) in diameter when located 5 ft (1.524 m) from a screen. The plane of the screen must be perpendicular to the optical axis of the flashlight.

9.8 *End Cap Ring*—The end cap ring shall support a weight of 25 lb (11.3 kg) for 1 min without evidence of distortion.

10. Marking, Packaging, and Packing

10.1 *Product Marking*—Each flashlight conforming to all the requirements in this specification shall be marked with the name, brand, or trademark of the manufacturer, this ASTM specification number, the type of flashlight (Type I, Type II, or Type III), and any other information as may be specified in the contract or purchase order.

10.2 Unless otherwise specified by the customer in the contract or purchase order, the flashlight shall be packaged, packed, and marked in accordance with the manufacturer's commercial practice to ensure acceptance and safe delivery by the carrier for the mode of shipping and handling.

TABLE 1 Sample Testing

Production	Order Batch
1. Switch leakage (9.1.1)	1. Switch leakage (9.1.1)
2. Operation (9.1.2)	2. Operation (9.1.2)
3. Switch endurance (9.1.3)	3. Rough use (9.6)
4. Watertightness (9.2)	4. Light projection (9.7)
5. Impact (9.3)	5. End cap ring (9.8)
6. Heat and humidity (9.4.1)	
7. Corrosion (9.5)	
8. Rough use (9.6)	
9. Light projection (9.7)	
10. End cap ring (9.8)	

11. Quality Assurance Provisions

11.1 Unless otherwise specified in the contract or purchase order, the producer is responsible for the performance of all inspection and requirements as specified herein. Except as otherwise specified in the contract or purchase order, the producer may use his own or any other facilities suitable for the performance of the inspection requirements specified herein. The purchaser reserves the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to ensure that the flashlights conform to the prescribed requirements.

11.2 The producer shall perform the necessary inspection and tests to ensure that an S-4 inspection level, in accordance with MIL-STD-105D, is provided. An Acceptable Quality Level (AQL) of 1 % defective for the operational test and the switch leakage test, and an AQL of 4 % for any of the other requirements or tests specified shall be provided. Sample testing shall be in accordance with Table 1.

12. Flashlights for Use in Hazardous Locations on U.S. Flag Merchant Vessels

12.1 Details such as dimensions, construction criteria, battery configuration, and voltage shall be provided by the procuring agency in the contract or purchase order.

12.2 Flashlights that are to be used in hazardous locations shall conform to the requirements of Underwriters Laboratories (UL) Standard No. 783, or equivalent standard, and to the following requirements.

12.3 Flashlights that are to be used in hazardous locations shall be listed by an independent testing laboratory that is concerned with product evaluation, that maintains periodic inspection of production of listed flashlights, and whose listing states that the flashlight has been tested and found suitable for use in the hazardous location specified (that is, Class and Group designation). The testing laboratory must be acceptable to the appropriate regulatory bodies or the procurement agency.

12.4 Flashlights that have been listed by a testing laboratory meeting the criteria in 12.3 shall affix a label, symbol, or other identifying mark to the flashlight that shall indicate that the flashlight is suitable for use in the hazardous location.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

* * *

Document Name:

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THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

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An American National Standard

Standard Specification for Line-Blind Valves for Marine Applications[1]

This standard is issued under the fixed designation F 1020; 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.

$^{\epsilon 1}$ NOTE—Keywords were added editorially in November 1996.

1. Scope

1.1 This specification provides the minimum requirements for design fabrication, pressure rating, and testing for line-blind valves.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The following safety hazards caveat pertains only to the test methods portion, Section 5, of this specification: *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 Standard:*

A 53 Specification for Pipe, Steel, Black and Hot-Dipped, Zinc-Coated Welded and Seamless[2]

2.2 *ANSI Standards:*

B16.5 Pipe Flanges and Flanged Fittings, Steel-Nickel Alloy and Other Special Alloys[3]

B31.1 Power Piping[3]

2.3 *MSS Standards:*

SP-6 Finish for Contact Faces of Pipe Flanges and Connecting End Flanges of Valves and Fittings[4]

SP-25 Marking System for Valves, Fittings, Flanges, and Unions[4]

SP-55 Quality Standard for Steel Castings for Valves, Flanges and Fittings, and Other Piping Components (Visual Method)[4]

2.4 *ASME Standard:*

ASME Boiler and Pressure Vessel Code, Sections II, VIII, IX[5]

3. Descriptions of Terms Specific to This Standard

3.1 *blank*—a solid one-piece circular unit inserted into a pipeline to prevent flow.

3.2 *line-blind valve*—an assembly consisting of a spectacle plate, bolting, and body, the purpose of which is to provide a convenient means to align a piping system to an open or positively closed configuration. The assembly is designed to provide a simplified method of changing over the flow control spectacle plate without the necessity of plate removal from the valve body.

3.3 *spectacle plate,* (*also spectacle blind*)—a figure-eight shaped unit with one end open for flow and the other solid to prevent flow.

4. Materials and Manufacture

4.1 *Materials:*

4.1.1 Materials for spectacle plates, bolting, and body shall be those contained in ASME Section II. For the purpose of stress calculations, ASME Section VIII values shall be used.

4.1.2 All welding shall be done with procedures and welders qualified in accordance with ASME Section IX; and 80 % weld efficiency factor shall be used.

4.1.3 All castings shall be visually inspected and acceptable in accordance with MSS-SP-55.

4.2 *Manufacture:*

4.2.1 The spectacle plate shall be designed in accordance with ANSI B31.1, paragraph 104.5.3.

4.2.2 The calculations of 4.2.3 and 4.2.4 shall ensure that a line blind is designed for the gasket material, of all that can be used with the line blind being designed, that imposes the most critical bolt-load conditions as a result of its gasket factor, m, and gasket or joint-contact-surface unit seating load, y.

4.2.3 The bolting shall be either of the following:

4.2.3.1 Modify the external loads in accordance with Section 6 as determined using ASME Section VIII, Division 1, Appendix 2.

4.2.3.2 The equivalent in cross-section to that of ANSI B16.5 flange bolting of equivalent nominal size and pressure.

4.2.3.3 In no case shall bolts have a nominal diameter less than ½ in. (12.7 mm).

4.2.3.4 The material class shall be an approved ANSI B16.5 material or equal in tensile strength.

4.2.3.5 Cast bolting shall be x-rayed or have an 80 % efficiency factor applied.

4.2.4 The body shall be calculated using ASME Section VIII, Division 1, Appendix 2 with consideration for the size and material of bolting in the appendix, the bolt load satisfying Note 2 of paragraph 2-5 of Appendix 2.

4.2.5 The spectacle plate and mating body facings shall be in accordance with MSS-SP-6.

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[1] This specification is under the jurisdiction of ASTM Committee F-25 on Ships and Marine Technology and is the direct responsibility of Subcommittee F25.13 on Piping Systems.

Current edition approved Sept. 26, 1986. Published November 1986.

[2] *Annual Book of ASTM Standards,* Vol 01.01.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[4] Available from Manufacturer's Standardization Society of the Valve and Fittings Industry, Inc., 127 Park St., N.E. Vienna, VA 22180.

[5] Available from American Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017.

5. Testing

5.1 Each valve body shall be hydrostatically tested to the shell test pressure in accordance with ANSI B16.5, Table 3. A valve supplied to the WOG standard shall be tested up to the nearest ANSI Class.

5.2 Each valve shall be tested for leakage at pressure 1.5 times the cold water rating of the valve rounded upward to the next higher 25 psi (170 kPa) increment. Acceptance criteria shall be that no leakage occurs during a 10-min "hold time" at pressure.

6. Calculations

6.1 Bolting calculations from ASME Section VIII, Division 1, Appendix 2 shall be modified to account for an externally applied load due to piping. Add to the minimum required bolt load for the operating conditions, W_{m1}, an external moment bolt load, H_x, such that the modified total cross-sectional area of the bolts shall be as follows:

$$A'_{m1} = (W_{m1} + H_x)/S_b$$

where:

H_x = SZ/d_b,
Z = $\Pi((D_o^4 - D_i^4)/D_o)/32$,
D_i = $D_o - 2t_p$,
t_p = $PD_o/(2(0.875)(S))$, but not less than 0.25 in. (6.4 mm), and
d_b = $C/\Pi + 2((G_o^3 - G_i^3)/(G_o^2 - G_i^2))3\Pi$.

6.1.1 All terms are identical to those defined in ASME Section VIII, Appendix 2 with the addition of the following:

6.1.1.1 A'_{m1} = modified total cross-sectional area of bolts at root of thread or section of least diameter under stress, required for the operating conditions, square inch. (square millimetre).

6.1.1.2 H_x = external moment of bolt load, lbf (N).

6.1.1.3 0.875 = assumed pipe wall tolerance factor.

6.1.1.4 S = ASME allowable stress of the pipe material at design temperature, psi (kPa), (for purposes of meeting this specification, 15 000 psi (103.4 MPa) from Specification A 53, Grade B, Type S shall be acceptable).

6.1.1.5 Z = section modulus of pipe shell, cubic inches (cubic millimetres).

6.1.1.6 D_o = nominal outside diameter of pipe, inches (millimetres).

6.1.1.7 D_i = calculated inside diameter of pipe, inches (millimetres).

6.1.1.8 t_p = calculated pipe wall thickness, inches (millimetres).

6.1.1.9 d_b = moment arm of external moment on bolts and gasket, inches (millimetres).

6.1.1.10 G_o = outside diameter of contact surface of gasket, inches (millimetres).

6.1.1.11 G_i = inside diameter of contact surface of gasket, inches (millimetres).

6.2 The selection of bolts to be used shall be made such that the actual total cross-sectional area of bolts, A_b, will not be less than A_m, where A_m is taken as the greater of A'_{m1} and A_{m2}.

7. Product Marking

7.1 Each valve must have the following markings in accordance with MSS-SP-25:

7.1.1 Manufacturer's name and trademark.
7.1.2 Appropriate pressure class.
7.1.3 Size of end connection.
7.1.4 ASTM designation of materials.
7.1.5 ASTM designation and year of issue of this specification.

8. Keywords

8.1 blind valve; line-blind valve; marine technology; piping system; ship; valve

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

* * *

Document Name:

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Standards Body:

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: F 1120 – 87 (Reapproved 1998)

An American National Standard

Standard Specification for Circular Metallic Bellows Type Expansion Joints for Piping Applications[1]

This standard is issued under the fixed designation F 1120; 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 specification establishes the minimum requirements for the mechanical design, manufacture, inspection, and testing of circular metallic bellows-type expansion joints used to absorb the dimensional changes resulting from piping thermal expansion or contraction, as well as the movement of terminal equipment and supporting structures.

1.2 Additional or better features, over and above the minimum requirements set by this specification, are not prohibited by this specification.

1.3 The layout of many piping systems provides inherent flexibility through natural changes in direction so that any displacements produce primarily bending or torsional strains, within acceptable limits. Where the system lacks this inherent flexibility the designer should then consider adding flexibility through the use of metallic bellows-type expansion joints.

1.4 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 *ANSI Standards:*

B16.5 Pipe Flanges and Flanged Fittings[2]

B16.25 Butt Welding Ends[2]

B31.1 Power Piping Code[2]

2.2 *ASME Standards:*

Section VIII, Division 1, Pressure Vessels[3]

Section IX, Welding and Brazing Qualifications[3]

2.3 *EJMA Standard:*

Standards of the Expansion Joint Manufacturer's Association[4]

2.4 *Pipe Fabrication Institute Standard:*

ES-3 Fabrication Tolerances[5]

3. Terminology Definitions

3.1 Expansion joint definitions shall be in accordance with those in the EJMA standards.

3.2 *double expansion joint*—expansion joint consisting of two bellows joined by a common connector.

3.3 *Discussion*—The common connector is anchored to some rigid part of the installation by means of an anchor base. The anchor base may be attached to the common connector either at installation or at time of manufacture. Each bellows acts as a single expansion joint and absorbs the movement of the pipe section in which it is installed independently of the other bellows.

3.4 *gimbal expansion joint*—expansion joint designed to permit angular rotation in any plane by the use of two pairs of hinges affixed to a common floating gimbal ring.

3.5 *Discussion*—The gimbal ring, hinges, and pins are designed to restrain the thrust of the expansion joint as a result of internal pressure and extraneous forces, where applicable.

3.6 *hinged expansion joint*—expansion joint containing one bellow designed to permit angular rotation in one plane only by the use of a pair of pins through hinge plates attached to the expansion joint ends.

3.7 *Discussion*—The hinges and hinge pins are designed to restrain the thrust of the expansion joint as a result of internal pressure and extraneous forces. Hinged expansion joints should be used in sets of two or three to function properly.

3.8 *pressure balanced expansion joint*—expansion joint designed to absorb axial movement or lateral deflection, or both, while restraining the pressure thrust by means of tie devices interconnecting the flow bellows with an opposed bellows also subjected to line pressure.

3.9 *Discussion*—This type of expansion joint is intended for use where a change of direction occurs in a run of piping. The flow end of a pressure balanced expansion joint sometimes contains two bellows separated by a common connector, in which case it is called a universal pressure balanced expansion joint.

3.10 *single expansion joint*—simplest form of expansion joint, consisting of single bellows construction, designed to

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[1] This specification is under the jurisdiction of ASTM Committee F-25 on Ships and Marine Technology and is the direct responsibility of Subcommittee F25.13 on Piping Systems.

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[2] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[3] Available from American Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017.

[4] Available from Expansion Joint Manufacturer's Association, 25 N. Broadway, Tarrytown, NY 10591. The Standards of the Expansion Joint Manufacturer's Association are a collection of standards developed by this industry and published in one volume, herein called EJMA Standards.

[5] Available from Pipe Fabrication Institute, 1326 Freeport Rd., Pittsburgh, PA 15238.

absorb all movement of the pipe section in which it is installed.

3.11 *swing expansion joint*—expansion joint designed to absorb lateral deflection or angular rotation, or both, in one plane.

3.12 *Discussion*—Pressure thrust and extraneous forces are restrained by the use of a pair of swing bars, each of which is pinned to the expansion joint ends.

3.13 *universal expansion joint*—expansion joint containing two bellows joined by a common connector for the purpose of absorbing any combination of axial movement, lateral deflection, and angular rotation.

3.14 *Discussion*—Universal expansion joints are usually furnished with control rods to distribute the movement between the two bellows of the expansion joint and stabilize the common connector.

4. Ordering Information

4.1 An expansion joint is a unique product and must be specifically designed for the intended service. It is the responsibility of the piping system designer to supply sufficient engineering data necessary for the complete design. The information compiled by the piping system designer must be complete and contain all pertinent data detailing the conditions under which the expansion joint is expected to operate.

4.2 Orders for each expansion joint shall include the following information:

4.2.1 Title, designation number, and latest revision of this specification.

4.2.2 *Size*—The nominal pipe diameter or specific ducting diameter.

4.2.3 *Type of Expansion Joint*—single, double, universal, guided, hinged, gimbal, swing, or pressure balanced.

4.2.4 *Flow Characteristics*:

4.2.4.1 *Flow Medium*—indicate whether the medium is gas or liquid.

4.2.4.2 Flow velocity, medium density, or viscosity, or combination thereof.

4.2.4.3 Flow direction.

4.2.5 *Pressure in psig (N/mm^2)*—design, operating, and test pressures.

4.2.6 *Temperature in °F (°C)*—design, operating, and installation temperatures.

4.2.7 *Movement*—axial (extension, compression); lateral (single plane, multiplane); angular; torsional (to be avoided). Differentiate between start-up, operational, or field installation tolerance movements.

4.2.8 *Materials*—Material types (including that for the bellows) shall be specified by the purchaser (see 5.1 for material restrictions).

4.2.9 *Internal Liner*—Liner shall be specified when needed because of flow velocity or other flow conditions. Specific criteria for liners is shown in Section C-3 of the EJMA Standards (see 6.6).

4.2.10 *External Cover*—To protect personnel having close access to the bellows, when thermal insulation is to be added in the field, or when external mechanical damage is possible (see 6.5).

4.2.11 *End Fittings*—The type of end connections such as flanged, threaded, or others to match the mating piping or terminal equipment.

4.2.12 *Accessories*—Specify what accessories are required and the conditions under which they operate. Consider items such as insulation lugs, tie, limit, or control rods, pantographic linkages, trunions, gimbals, drains, purge connections, anchor bases, and interply monitoring devices.

4.2.13 *Dimensional Limitations*—If space limitations exist, specify the maximum overall length, maximum outside diameter, minimum inside diameter, and installation tolerances.

4.2.14 *Operating Forces*—Specify calculated bellows spring forces and pressure thrust forces if they are required for subsequent anchor design or other piping systems analysis. If there are maximum allowable values, these must also be specified.

4.2.15 *Installation Position*—horizontal, vertical (flow up or down). Specify if liner drainage holes are required.

4.2.16 *Cycle Life Requirements*—Specify an anticipated number of thermal cycles over the intended life of the expansion joint.

4.2.17 *Testing Requirements*—Specify testing requirements in addition to the hydrostatic test required by 9.4 (for example, vacuum testing, testing at operating temperature).

4.2.18 *Inspection Requirements*—Specify inspection requirements in addition to the inspection required by Section 9 (that is, radiographic, fluorescent penetrant, or mass spectrometer).

4.2.19 *Piping Code Requirements*—Specify any piping or design code that must be used as the basis for design in addition to those specified in 5.2.

4.2.20 *Special Requirements*—Specify the magnitude of special system conditions such as vibration, shock, or hydraulic surge.

4.2.21 *Shipping Requirements*—Specify whether special packing is required including protection for extended outside storage, export handling, or special lifting considerations for heavy or large assemblies.

4.2.22 *Piping Drawing*—In addition to specifying the above information it would be beneficial to provide a drawing of the proposed piping system.

4.2.23 *Supplementary Requirements*—Specify any additional requirements not identified herein.

4.3 Fig. 1 and Fig. 2 should be used as a guide in ordering expansion joints to this specification.

5. Materials and Manufacture

5.1 *Materials*:

5.1.1 Pressure-containing parts shall be manufactured from material specifications and grades listed in Section VIII, Division 1, of the ASME Code or ANSI B31.1. End connection material shall have in service properties similar to the bellows material. Flanges shall meet ANSI B16.5.

5.1.2 All other materials of construction shall be of the type specified by the user and shall conform to an ASTM or ASME material specification. Materials not identified by the ordering data shall be of the manufacturer's standard and of the same quality used for the intended purpose in commercial practice.

5.1.3 Materials used shall be free from defects that would adversely affect the performance of the expansion joint.

COMPANY:

DATE / /

SHEET OF

PROJECT:

INQUIRY NO.

JOB NO.

	ITEM NO./EJ TAG NO.					
1	QUANTITY		EJMA PAGE REFERENCE			
2	NOMINAL SIZE/I.D./O.D. (IN.)					
3	EXPANSION JOINT TYPE		1			
4a	FLUID INFORMATION	MEDIUM GAS/LIQUID	5, 6, 147 77			
4b		VELOCITY (FT./SEC)				
4c		FLOW DIRECTION				
5	DESIGN PRESSURE, PSIG		6, 19, 83, 135			
6	TEST PRESSURE, PSIG					
7a	TEMPERATURE	DESIGN (°F)	6, 13			
7b		MAXIMUM/MINIMUM (°F)				
7c		INSTALLATION (°F)				
8a	MAXIMUM INSTALLATION MOVEMENT	AXIAL COMPRESSION (IN.)	6, 7, 8, 141			
8b		AXIAL EXTENSION (IN.)				
8c		LATERAL (IN.)				
8d		ANGULAR (DEG.)				
9a	MAXIMUM DESIGN MOVEMENTS	AXIAL COMPRESSION (IN.)	6, 7, 13, 47			
9b		AXIAL EXTENSION (IN.)				
9c		LATERAL (IN.)				
9d		ANGULAR (DEG.)				
9e		NO. OF CYCLES				
10a	OPERATING FLUCTUATIONS	AXIAL COMPRESSION (IN.)	84			
10b		AXIAL EXTENSION (IN.)				
10c		LATERAL (IN.)				
10d		ANGULAR (DEG.)				
10e		NO. OF CYCLES				
11a	MATERIALS OF CONSTRUCTION	BELLOWS	5, 6, 45 77, 78			
11b		LINERS				
11c		COVER	3, 7, 72			
11d		PIPE SPECIFICATION				
11e		FLANGE SPECIFICATION	3, 43			
12	RODS (TIE/LIMIT/CONTROL)		3, 4, 41			
13	PANTOGRAPHIC LINKAGE		4			
14	ANCHOR BASE (MAIN/INTERMEDIATE)		1, 2, 17			
15a	DIMENSIONAL LIMITATIONS	OVERALL LENGTH (IN.)				
15b		OUTSIDE DIAMETER (IN.)				
15c		INSIDE DIAMETER (IN.)				
16a	SPRING RATE LIMITATIONS	AXIAL (LBS./IN.)	54			
16b		LATERAL (LBS./IN.)				
16c		ANGULAR (IN.-LBS./DEG.)				
17	INSTALLATION POSITION HORIZ./VERT.		8, 141			
18a	QUALITY ASSURANCE REQUIREMENTS	BELLOWS WELD NDE LONG. SEAM	133			
18b		BELLOWS WELD NDE ATTACH.				
18c		PIPING NDE				
18d		DESIGN CODE REQRD.				
18e		PARTIAL DATA REQRD.				
18f						
18g						
19	VIBRATION AMPLITUDE/FREQUENCY					

FIG. 1 Standard Expansion Joint Specification Sheet

Company ______________________ Date ______________

______________________ Proposal No. ______________

Project ______________________ Inquiry/Job No. ______________

______________________ Sheet ________ of ________

	ITEM NO.				
20.	PURGE, INSTRUMENTATION CONNECTION				
21a.	SPECIAL FLANGE DESIGN	FACING			
21b.		O.D. (IN.)			
21c.		I.D. (IN.)			
21d.		THICKNESS (IN.)			
21e.		B.C. DIAMETER (IN.)			
21f.		NO. HOLES			
21g.		SIZE HOLES			
21h.		HOLE ORIENTATION			

ISOMETRIC PIPING SKETCH:

FIG. 2 Supplemental Specification Sheet (To Be Used With Standard Expansion Joint Specification Sheet)

5.1.4 All material incorporated in the work covered by this specification shall be new. The use of rebuilt or used products is not allowed under this specification.

5.1.5 Materials for hinge or gimbal hardware, or other sliding parts, shall be chosen to minimize galling of the contacting parts.

5.2 *Manufacture*:

5.2.1 Expansion joints shall be designed and fabricated in accordance with requirements set forth in the ordering data and the EJMA Standards.

5.2.2 Nonstandard flanges shall be designed and fabricated in accordance with Appendix 2 of Section VIII, Division 1, of the ASME Code. Flanges machined from plate shall not be used at pressures exceeding 150 psi (1034 kPa) and temperatures exceeding 450°F (232°C). Hubbed flanges machined from plate or bar stock shall meet the requirements of Appendix 2, Paragraph 2-2(*d*) of Section VIII, Division 1, of the ASME Code.

5.2.3 All welding shall be accomplished in accordance with ANSI B 31.1.

5.2.4 Welding personnel and welding procedures shall be qualified in accordance with the applicable sections of Section IX of the ASME Code.

5.2.5 All fabrication details not covered by the referenced codes and standards shall be taken from the appropriate ANSI standard. If no standard applies, accepted industry practice shall govern.

5.2.6 The bellows shall be of tested and proven convolution geometry.

6. Other Requirements

6.1 The details of design, material supply, fabrication, and testing of the complete product are the responsibility of the manufacturer unless specific details are requested by the purchaser.

6.2 The specified normal operating movements (axial, lateral, and angular) shall be available concurrently. The specified lateral and angular movements shall be available on either side of the expansion joint centerline.

6.3 Internal sleeves, external covers, and all attached hardware shall be constructed so as not to interfere with adjacent parts when the joint is in the fully deflected position.

6.4 Universal expansion joints shall be designed and fabricated to be self-supporting and not require any external structure for the support of the center pipe spool piece and its contents.

6.5 Expansion joints to be installed in systems above 150°F (66°C) shall have an external cover. When external mechanical damage is possible, a cover shall be fabricated to protect the joint and personnel.

6.6 Internal sleeves shall be installed in expansion joints when the fluid velocity of the system, where the expansion joint is to be installed, is greater than the values listed in Section C-3.1 of the EJMA Standards and where the flow velocity exceeds 75 % of the velocity calculated using Section C-3.1.4 of the EJMA Standards.

7. Dimensions and Permissible Variations

7.1 Dimensional tolerances on completed expansion joint assemblies shall be in accordance with Section D-2.9 of the EJMA Standards and Standard ES-3 of the Pipe Fabricating Institute.

8. Workmanship, Finish, and Appearance

8.1 The quality of workmanship shall be such as to produce a product that is in accordance with the requirements of this specification and ensures the proper functioning of all parts of the unit.

8.2 The bellows shall be manufactured and carefully handled to prevent surface flaws or deep scratches from being generated. The surface condition of the completed joint assembly shall be free from injurious surface discontinuities and any contaminants that would affect the operation of the assembly.

8.3 On completion of fabrication, and before shipment, the manufacturer shall clean the inside and outside of the completed assembly of all loose scale, grease, dirt, sand, rust, weld spatter, cutting chips, and any other foreign matter by any suitable means. The inside of the assembly shall then be inspected for cleanliness. All openings where practicable shall be suitably closed to prevent the entrance of foreign matter after cleaning and during shipment. The use of chlorinated solvents is prohibited.

9. Inspection

9.1 The responsibility for quality control rests with the manufacturer. However, all phases of fabrication may be subject to review by a representative of the purchaser.

9.2 The inspector representing the purchaser shall have access at all times, while work on the contract of the purchaser is being performed, to all parts of the manufacturer's plant that concern the manufacture of the product ordered. The manufacturer shall afford the inspector all reasonable facilities to satisfy the inspector that the product is being furnished in accordance with this specification. Inspection shall be made at the place of manufacture before shipment, unless otherwise specified, and shall be scheduled not to interfere unnecessarily with the operations of the manufacturer. This requirement applies to all subcontractors.

9.2.1 Acceptance of a particular phase of manufacturer of an assembly by a purchaser's representative shall not be considered a waiver of any of the requirements of this specification and shall not relieve the manufacturer of the responsibility of furnishing a satisfactory product.

9.3 When the bellows is formed from a longitudinally butt-welded cylinder, the longitudinal weld(s) shall be 100 % liquid penetrant examined on the outside and inside surfaces (if accessible) before forming. Liquid penetrant examination on all accessible inside and outside weld surfaces shall be repeated after forming. All other welds essential to pressure containing or restraining shall be liquid penetrant examined. Ferromagnetic materials may be examined by magnetic particle inspection instead of liquid penetrant at the manufacturer's option. Liquid penetrant and magnetic particle inspection shall be in accordance with Section D-3.1.2 of the EJMA Standards.

9.4 All pressure retaining components shall be hydrostatically tested to 1.5 times their design pressure as outlined in Section D-3.1.6 of the EJMA Standards. Moment restraint, simulating piping rigidity, shall be used if necessary. The expansion joint shall be vented before hydrotest. Test pressure shall be held for 10 min.

9.4.1 Unless otherwise specified, potable water is acceptable for hydrotesting.

9.4.2 All piping and bellows shall be thoroughly drained after hydrotesting.

9.4.3 Pneumatic testing may be substituted for hydrostatic testing at the manufacturer's option. When substituted, pneumatic testing shall be accomplished in accordance with Section 137.5 of ANSI B31.1.

9.5 A dimensional inspection of the completed expansion joint assemblies shall be performed in accordance with Section D-2.9 of the EJMA Standards and Standard ES-3 of the Pipe Fabricating Institute.

9.6 A visual examination of the completed expansion joint shall be made.

10. Rejection and Rehearing

10.1 Expansion joint assemblies or parts thereof indicating fabrication not in accordance with the manufacturing drawings and procedures, or this specification, shall be subject to rejection and shall be resolved in accordance with the manufacturer's quality assurance program (see Section 14).

10.2 All repairs shall be in accordance with the specified code and other applicable specifications.

10.3 Expansion joint assemblies or parts thereof accepted by the purchaser's representative at the place of manufacture that subsequently reveal imperfections not previously detected or which by subsequent tests or analysis show nonconformance with this specification are subject to rejection.

11. Certification

11.1 When specified in the purchase order or contract, the manufacturer's certification shall be furnished to the purchaser. It shall state that each expansion joint has been manufactured, tested, and inspected in accordance with this specification and the requirements have been complied with. When specified, a report of any test results shall be furnished.

11.2 When specified, certification of the conformance to the requirements of this specification may be made by a third party.

11.3 ASTM/ASME mill test reports are required for pressure retaining and containing components.

11.4 No records are required for pipe fittings or flanges provided they are made and marked in accordance with an acceptable standard (such as ANSI). Certificates of conformance are required when the markings are missing or are removed during fabrication.

12. Product Marking

12.1 Each expansion joint shall be provided with a permanently attached corrosion resistant nameplate indicating as a minimum the following information:

12.1.1 Manufacturer's name.

12.1.2 Manufacturer's model number.

12.1.3 *Design Conditions*—pressure, temperature, movements.

12.1.4 This specification number (indicating compliance thereto).

12.1.5 Purchaser's specified component item number, if ordered.

12.2 When an expansion joint is supplied with an internal liner, a permanent arrow indicating the direction of flow shall be plainly visible on the outside of the expansion joint.

12.3 Impression stamping directly on bellows material is not permitted.

13. Packaging and Shipping

13.1 The expansion joint shall be containerized or shipped on pallets with all materials strapped down and prepared for shipment in such a manner that the quality, cleanliness, and finish shall be maintained during shipment.

13.2 Yellow painted shipping bars shall be furnished to maintain proper shipping length and alignment, and designed not to interfere with the installation of the assembly. The shipping bars shall be removed after installation and before piping system test. Expansion joints with tie rods can be provided with tie rod spacers instead of shipping bars.

13.3 Installation instructions shall be supplied in a weatherproof envelope with each expansion joint assembly.

13.4 When the expansion joint is to be transported to the job site by ship, it should preferably be sent as below deck cargo.

13.5 All external surfaces shall be treated and painted in accordance with the manufacturer's standard practices, unless otherwise specified. Paint shall be suitable for service temperatures.

14. Quality Assurance

14.1 The manufacturer shall have a current certificate of authorization to manufacture ASME Section VIII, Division 1, Pressure Vessels to assure an adequate quality assurance program that is applicable to all phases of manufacturing, including materials supplied by subcontractors.

14.2 Nothing in this specification shall relieve the manufacturer of the responsibility for performing, in addition to the requirements of this specification, such analyses, tests, inspections, or other activities that the manufacturer considers necessary to ensure that the design, material, and workmanship are satisfactory for the service intended, or as may be required by common usage or good practice.

15. Keywords

15.1 expansion joint; metallic bellows-type expansion joints; piping systems; piping thermal contraction; piping thermal expansion

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirements are for use when desired by the purchaser. Other requirements not identified in this specification may be included by agreement between the manufacturer and the purchaser.

S1. Documents for Approval and/or Records

S1.1 Manufacturing drawings.

S1.2 Welding procedures and qualifications.

S1.3 Applicable nondestructive examination procedures.

S1.4 Heat treatment procedures or temperature charts, or both.

S1.5 Complete engineering design analysis calculations for the metallic bellows or hardware, or both.

S1.6 ASME partial data forms.

S2. Qualification Testing

S2.1 When specified, the manufacturer shall furnish a first article test assembly to determine conformance with this specification. The test assembly shall consist of the bellows and appropriate end connections. Liners, covers, tie, limit or control rods, hinges, gimbal rings, and other similar devices need not be provided on the first article test unit, unless they are necessary for the performance of the specific test(s) verification.

S2.2 When specified, cyclic endurance testing (fatigue testing) shall be performed for the required number of complete cycles. The test shall be performed under pressure at ambient temperature and the assembly need be cycled in axial movement only.

S2.2.1 During the test, the pressure in the assembly shall be adjusted to simulate, as closely as possible, the maximum design pressure of the unit being qualified. The pressure may vary from this value during each cycle.

S2.2.2 A single test bellows can be used instead of a multiple bellows assembly being qualified.

S2.2.3 In determining the qualifying extension or compression, or both, the equivalent axial movement caused by lateral deflection and angular rotation shall be included. The equivalent axial movement shall be computed in accordance with the EJMA Standards and shall be algebraically added to the specified values of axial movement.

S2.3 The purchaser may require that the expansion joint be certified as passing shock requirements. The shock requirements shall be specified by the purchaser.

S2.3.1 The purchaser may require that the expansion joint be certified as passing vibration requirements. The vibration requirements shall be specified by the purchaser.

S2.3.2 When specified, other qualifying tests shall be performed on a first article test unit under the requirements of the purchaser's contract.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

Document Name:	ASTM F1121: Standard Specification for International Shore Connections for Marine Fire Applications
CFR Section(s):	33 CFR 126.15(a)(5)
Standards Body:	American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: F 1121 – 87 (Reapproved 1998)

An American National Standard

Standard Specification for International Shore Connections for Marine Fire Applications[1]

This standard is issued under the fixed designation F 1121; 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 specification covers the design and manufacture of international shore connections to be used with marine fire fighting systems during an emergency when a stricken ship has a system failure.

1.2 International shore connections are portable universal couplings that permit connection of shipboard firemain systems between one ship and another or between a shore facility and a ship when their respective system threading is mismatched. Both the ship and the facility are expected to have a fitting such that in an emergency can be attached to their respective fire hose and bolted together to permit charging the ship's system. It must be portable to accommodate hose to hose connection and allow assistance from any position.

1.3 The international shore connection is required by international treaty[2,3] to be carried onboard all passenger and cargo vessels of 500 gross tons or more, regardless of firemain size, engaged in international voyages, and is recommended for all vessels that would be expected to render assistance. It is also intended to be provided at shore facilities that would be used to supply water to a ship's firemain system.

1.4 Fabrication either on board a vessel, in a shipyard, or other shore facility is not precluded by this specification.

1.5 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

2. Ordering Information

2.1 The purchase order or inquiry for an international shore connection shall include the following as applicable:

2.1.1 Material of construction.

2.1.2 Title, number, and latest revision of this specification.

2.1.3 Maximum operating pressure (psig) (when above 150 psig, see 4.2).

2.1.4 Preservation (coating) requirements (if any, see 7.2).

2.1.5 Diameter and threading to be provided in the coupling.

3. Materials and Manufacture

3.1 The international shore connection may be machined from forgings, castings, plate or bar stock, or may be fabricated out of more than one piece.

3.2 The material shall be brass, bronze, or other suitable corrosion-resistant material. When fabricated out of more than one piece, the flange may be steel and the coupling a corrosion-resistant material. Aluminum shall not be used.

3.3 Nuts, bolts, and washers shall be a corrosion-resistant material.

4. Other Requirements

4.1 *Design*:

4.1.1 The connection shall consist of a flat face flange and a threaded coupling. Threading shall be specified by the purchaser.

4.1.2 The dimensions of the international shore connection shall be in accordance with Fig. 1.

4.2 The maximum allowable working pressure (MAWP) shall be at least 150 psig (1 N/mm^2).

4.3 The international shore connection shall be supplied with four bolts, ⅝ in. (16 mm) in diameter, at least 2 in. (50 mm) in length, and threaded at least to within 1 in. (25 mm) of the bolt head.

4.3.1 The bolts shall be supplied with four corresponding nuts and eight washers.

4.4 The international shore connection shall be supplied with a flange gasket suitable for the MAWP and seawater service.

5. Workmanship, Finish, and Appearance

5.1 The quality of workmanship shall be such as to produce a product that is in accordance with the requirements of this specification. Completed units shall be free from imperfections or defects that materially affect appearance or that may affect serviceability.

6. Inspection

6.1 Each finished international shore connection shall be visually examined and dimensionally checked to ensure it

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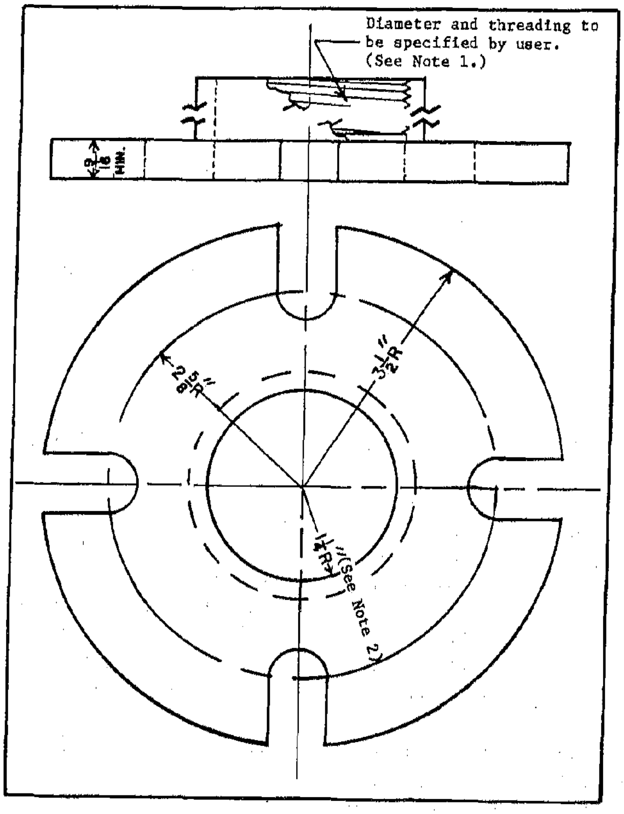
[1] This specification is under the jurisdiction of ASTM Committee F-25 on Ships and Marine Technology and is the direct responsibility of Subcommittee F25.13 on Piping Systems.

Current edition approved Dec. 31, 1987. Published February 1988.

[2] Amendments to the International Convention for the Safety of Life at Sea, Chapter II-2, Regulation 19, "International Shore Connection," 1974.

[3] International Maritime Organization Assembly Resolution A, XII 470, Jan. 4, 1987. This document is available from International Maritime Organization, 4 Albert Embankment, London, U.K. SE1 7SR.



NOTE 1—Fire hose coupling may be used.
NOTE 2—For 1½-in. fire hose coupling, the radius will be ¾ in.
NOTE 3—1 in. = 25.4 mm.

FIG. 1 International Shore Connection

corresponds to this specification.

7. Packaging and Preservation

7.1 Unless otherwise specified, the international shore connection shall be packaged for shipment in accordance with the manufacturer's standard commercial practice.

7.2 Preservation of the international shore connection, by the manufacturer or user, shall be satisfactory for preventing deterioration of the connection during long-term storage on vessels or at shore facilities.

8. Quality Assurance

8.1 The manufacturer shall use quality assurance procedures that assure manufacture of high quality international shore connections that are designed in accordance with this specification.

9. Keywords

9.1 fire fighting systems; international shore connections; hose-to-hose connection; marine fire applications; portable universal couplings; shipboard firemain systems; stricken ship

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

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◆

Document Name: ASTM F1122: Standard Specification for Quick Disconnect Couplings

CFR Section(s): 33 CFR 154.500(d)(3)

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: F 1122 – 87 (Reapproved 1998)

An American National Standard

Standard Specification for Quick Disconnect Couplings[1]

This standard is issued under the fixed designation F 1122; 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 specification covers the manufacturing data required to produce a variety of styles and sizes of quick disconnect couplings for marine use that ensure interchangeability and safety of operation.

1.2 In general, quick disconnect couplings are hose and pipe end fittings that permit quick mechanical attachment by means other than bolted or threaded fittings. The method of attachment is a male coupling half (adapter) that fits into a female coupling half (coupler) of the same size. By closing attached cam handles, the coupling halves seal, permitting fluids to be transported under pressure through the quick disconnect coupling.

1.3 The values stated in this specification are in inch-pound units with SI units given in parentheses. The values of each system may not be exact equivalents. Therefore, each system should be considered separately.

1.4 The following safety hazards caveat pertains only to the test method described in this specification: *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 *ANSI Standards:*

B2.1 Pipe Threads[2]

B16.5 Pipe Flanges and Flanged Fittings, Steel-Nickel Alloy, and Other Special Alloys[2]

B16.24 Bronze Pipe Flanges and Flanged Fittings[2]

B16.42 Ductile Iron Pipe Flanges and Flanged Fittings[2]

B31.1 Power Piping Code[2]

2.2 *MSS Standards:*

MSS-SP-6 Standard Finish for Contact Faces of Pipe Flanges and Connecting End Flanges of Valves and Fittings[3]

MSS-SP-25 Standard Marking System for Valves, Fittings, Flanges, and Unions[3]

MSS-SP-55 Quality Standard for Steel Castings for Valves, Flanges, and Fittings and Other Piping Components (Visual Method)[3]

2.3 *ASME Standards:*

Section VIII, Division 1, Pressure Vessels[4]

Section IX, Welding and Brazing Qualifications[4]

3. Definitions of Terms Specific to This Standard

3.1 *adapter*—one half of a quick disconnect coupling that fits into the coupler and seals against an elastomer gasket positioned inside the coupler.

3.2 *cam handles*—handles that are assembled to the coupler half which by closing engages the adapter sealing the coupling.

3.3 *coupler*—one half of a quick disconnect coupling that receives the adapter. This half contains the sealing gasket and cam handles (see Fig. 1).

NOTE 1—See Fig. 2 for a description of typical coupler pipe fittings.

4. Classification

4.1 Quick disconnect couplings shall consist of the following types:

4.1.1 *Standard Class*—This type is to be designed for a 4:1 burst factor of safety.

4.1.2 *Class I*—This type is to be designed for a 5:1 burst factor of safety.

4.2 Both Standard Class and Class I will be called quick disconnect couplings in the body of this specification unless otherwise specified.

5. Ordering Information

5.1 Purchase orders for quick disconnect couplings under this specification shall include the following applicable information:

5.1.1 Class.

5.1.2 Size and type of each coupling half-end connection. (Example—2- by 1½-in. NPT).

5.1.3 ASTM material designation and date including alloy specifications for the following:

5.1.3.1 Adapter and coupler halves,

5.1.3.2 Cam handle, and

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[1] This specification is under the jurisdiction of ASTM Committee F-25 on Ships and Marine Technology and is the direct responsibility of Subcommittee F25.13 on Piping Systems.

Current edition approved Dec. 31, 1987. Published February 1988.

[2] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[3] Available from Manufacturers' Standardization Society of the Valve and Fittings Industry, Inc., 1815 N. Fort Myers Dr., Arlington, VA 22209.

[4] Available from American Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017.

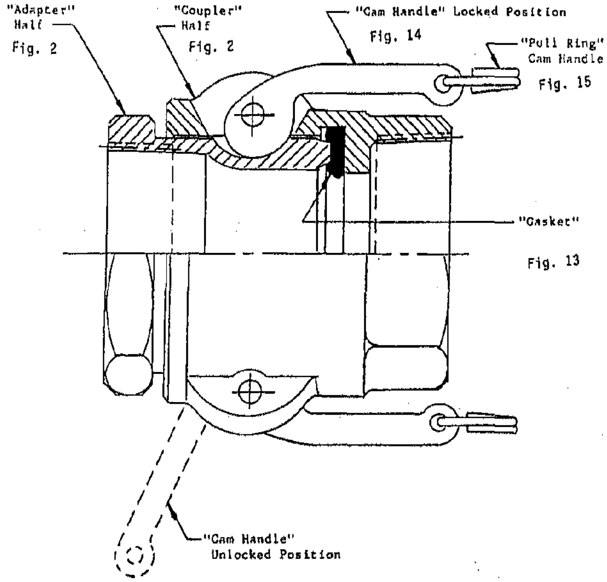


FIG. 1 Typical Coupler Assembly

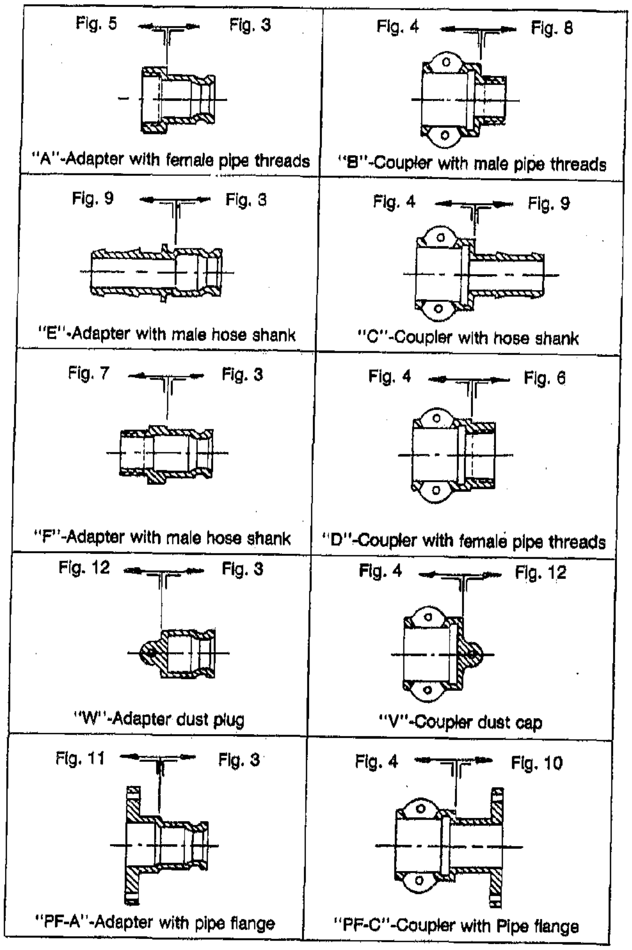


FIG. 2 Adapter/Coupler Types (End Dimensional Data Located in Tables Indicated by Arrows)

5.1.3.3 Cam handle pivot pin.

5.1.4 Product or fluid in applicable system.

5.1.5 Shipping instructions.

5.1.6 Any special requirements, such as testing, coatings, and threads.

6. Materials and Manufacture

6.1 *Materials*:

6.1.1 Pressure-retaining parts shall be manufactured from material specifications and grades listed in Section VIII, Division 1, of the ASME Boiler and Pressure Vessel Code or ANSI B31.1.

6.1.2 All other materials of construction shall be of the type specified by the user and shall conform to ASTM, ASME, or Metal Power Industry Federation material specifications. Materials not identified by the ordering data shall be of the manufacturer's standard and of the same quality used for the intended purpose in commercial practice.

6.1.3 All material incorporated in the work covered by this specification shall be new. The use of rebuilt or used products are not allowed under this specification.

6.1.4 Gaskets are to be produced from a compressible elastomeric material and shall be compatible with the fluid to be transferred (see 5.1.4).

6.2 *Manufacture*:

6.2.1 Adapters and couplers are to be produced as castings or forgings. Cam handles may be produced by casting, forging, or sintered metal processes. Established commercial processing methods are to be used to produce these parts, provided chemical and physical properties are consistent with those cataloged for the specified materials.

6.2.2 Pipe threads on the service end of couplers or adapters, when specified, shall meet ANSI B2.1.

6.2.3 Flanges on the service end of couplers or adapters, when specified, shall meet ANSI B16.5, B16.24, or B16.42.

6.2.4 Flanges shall be finish machined to specifications shown in MSS-SP-6.

6.2.5 Cam handles assembled to the coupler are to have a safety locking device to ensure against the handles being opened unintentionally or vibrating open. This locking device must require a separate, deliberate effort in the opening operation over the standard handle operation.

6.2.6 Cam handles shall be manufactured to contain the adapter within the coupler under rated pressure with no leakage occurring. The cam action shall not distort the couplers rendering the coupler unusable. The force required to close the cam handles shall be adequate to prevent leakage, but shall be easily attainable through hand operation by an average strength person. Handles should not need to be hammered closed.

6.2.7 Welding procedure qualifications, welder performance qualifications, and welding materials shall be in accordance with ANSI B 31.1 and Section IX of the ASME Code. Brazing or soldering shall not be used. Where radiography is required (see 12.1), all welds shall be butt welds.

7. Other Requirements

7.1 All couplings shall have a maximum allowable working pressure of not less than 150 psi (1034 kPa).

7.2 Maximum allowable working pressure (MAWP) for a Standard Class coupling shall be 25 % of its burst pressure.

7.3 Maximum allowable working pressure for a Class I coupling shall be 20 % of its burst pressure.

7.4 Burst pressure shall be determined in accordance with Section VIII, Division 1 of the ASME Code.

8. Dimensions

8.1 The dimensions and tolerances required to ensure interchangeability of adapter and coupler halves, of common sizes, shall be as given in Figs. 3-15.

8.2 Sizes as listed in the tables correspond to NPS for piping systems.

9. Workmanship, Finish, and Appearance

9.1 Couplings are to be produced with quality workmanship. Casting surface quality is to be in accordance with MSS-SP-55. Machined surfaces are to be finish machined to a 125-µin. (3175-µmm) (AA) finish or better with no porosity showing.

9.1.1 Couplings are to be free of burrs or sharp edges. Machined surfaces are to be free of nicks or scratches that may affect the sealing capabilities of the couplings.

9.1.2 Surfaces are to be sound with good appearance and true pattern. Internal surfaces, if not machined, shall be smooth, as expected from good quality casting, and free from any flaw that would render the part unsafe for its intended use.

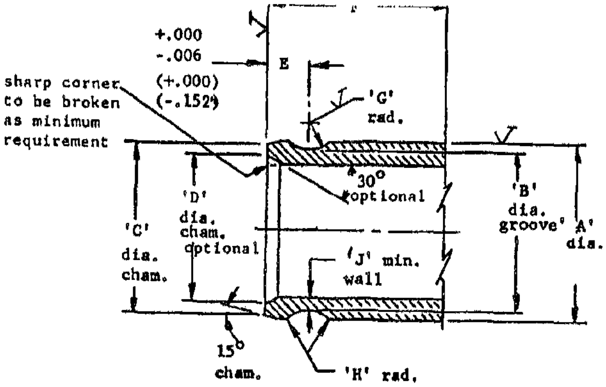
9.2 Cam handles are to be smooth, having no burrs or sharp edges. Cam closing surface is to be continuous, providing an increasing axial pull on mating adapter until proper seating against the gasket occurs.

9.3 Defective parts are not to be repaired by welding, brazing, or any other method, to fill porosity or other flaws in the casting.

9.4 Gaskets are to be produced having parallel sealing faces. Faces are not to contain any imperfections that will allow leakage to occur at working pressure.

10. Number of Tests

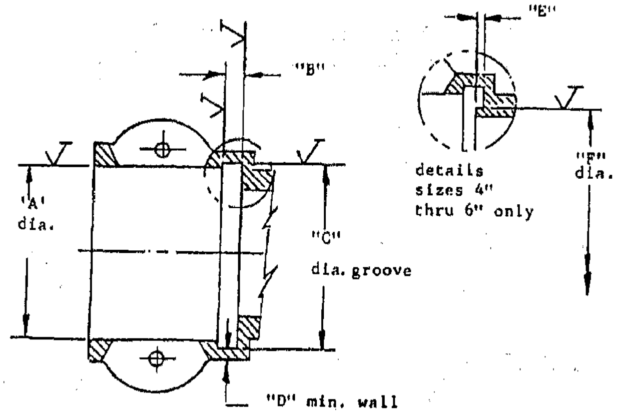
10.1 A prototype coupling of each size of each particular



NOTE 1—Unidentified tolerances; fractional = ±1/64 (±0.4), decimal = +0.000/−0.005 (+0.000/−0.127), angular = ±2° (±0.035 rad)

Size	Dimensions, in. (mm)								
	A	B	C	D	E	F	G	H	J
½	1.264 (32.1)	1.035 (26.3)	1 7/32 (31)	27/32 (21)	0.471 (12)	1 (25.4)	0.378 (9.6)	1/16 (1.6)	3/32 (2.4)
¾	1.264 (32.1)	1.035 (26.3)	1 7/32 (31)	31/32 (24.6)	0.471 (12)	1 (25.4)	0.378 (9.6)	1/16 (1.6)	3/32 (2.4)
1	1.446 (36.7)	1.144 (29)	1⅜ (34.9)	1 3/32 (27.8)	0.565 (14.3)	1 5/16 (33.3)	0.378 (9.6)	3/32 (2.4)	3/32 (2.4)
1¼	1.792 (45.5)	1.388 (35.2)	1 23/32 (43.6)	1¼ (31.7)	0.690 (17.5)	1 9/16 (39.7)	0.441 (11.2)	⅛ (3)	⅛ (3)
1½	2.105 (53.4)	1.690 (42.9)	2 1/32 (51.6)	1 9/16 (39.7)	0.690 (17.5)	1⅝ (41.3)	0.441 (11.2)	⅛ (3)	⅛ (3)
2	2.484 (63.1)	2.065 (52.4)	2 13/32 (61.1)	1 15/16 (49.2)	0.848 (21.5)	1⅞ (47.6)	0.441 (11.2)	⅛ (3)	⅛ (3)
2½	2.985 (75.8)	2.545 (64.6)	2⅞ (73)	2 7/16 (61.9)	0.848 (21.5)	1 15/16 (49.2)	0.441 (11.2)	⅛ (3)	5/32 (4)
3	3.604 (91.5)	3.202 (81.3)	3½ (88.9)	3 3/32 (78.6)	0.895 (22.7)	2 (50.8)	0.503 (12.8)	⅛ (3)	5/32 (4)
4	4.708 (119.6)	4.307 (109.4)	4 19/32 (116.7)	4⅛ (104.8)	0.895 (22.7)	2 1/16 (52.4)	0.503 (12.8)	⅛ (3)	7/32 (5.6)
5	5.728 (145.5)	5.312 (134.9)	5⅝ (142.9)	5⅛ (130.2)	0.897 (22.8)	2⅛ (54)	0.503 (12.8)	⅛ (3)	7/32 (5.6)
6	6.929 (176)	6.362 (161.6)	6 13/16 (173)	6 5/32 (156.4)	0.963 (24.5)	2¼ (57.1)	0.659 (16.7)	⅛ (3)	7/32 (5.6)

FIG. 3 Adapter End Dimensions



NOTE 1—Unidentified tolerances; fractional = ±1⁄64 (±0.4), angular = ±2° (±0.035 rad), decimal = ±0.005 (±0.127)

Size	Dimensions, in. (mm)					
	A	B	C	D	E	F
½	1.274 (32.3)	¼ (6.3)	1²⁷⁄₆₄ (36.1)	³⁄₃₂ (2.4)	. . .	. . .
¾	1.274 (32.3)	¼ (6.3)	1²⁷⁄₆₄ (36.1)	³⁄₃₂ (2.4)	. . .	. . .
1	1.464 (37.2)	⁹⁄₃₂ (7.1)	1¹⁹⁄₃₂ (40.5)	³⁄₃₂ (2.4)	. . .	. . .
1¼	1.810 (46)	⁹⁄₃₂ (7.1)	2 (50.8)	⅛ (3)	. . .	. . .
1½	2.123 (53.9)	⁹⁄₃₂ (7.1)	2¼ (57.1)	⅛ (3)	. . .	. . .
2	2.508 (63.7)	⁹⁄₃₂ (7.1)	2¹¹⁄₁₆ (68.3)	⅛ (3)	. . .	. . .
2½	3.009 (76.4)	⁹⁄₃₂ (7.1)	3³⁄₁₆ (81)	⁵⁄₃₂ (4)	. . .	. . .
3	3.628 (92.1)	⁹⁄₃₂ (7.1)	3²⁵⁄₃₂ (96)	⁵⁄₃₂ (4)	. . .	. . .
4	4.732 (120.2)	⁹⁄₃₂ (7.1)	4¹⁵⁄₁₆ (125.4)	⁷⁄₃₂ (5.6)	³⁄₃₂ (2.4)	3²⁹⁄₃₂ (99.2)
5	5.752 (146.1)	⁹⁄₃₂ (7.1)	5³¹⁄₃₂ (151.6)	⁷⁄₃₂ (5.6)	³⁄₃₂ (2.4)	4⁵¹⁄₆₄ (121.8)
6	6.948 (176.5)	⁹⁄₃₂ (7.1)	7⁹⁄₆₄ (181.4)	⁷⁄₃₂ (5.6)	³⁄₃₂ (2.4)	5²⁹⁄₃₂ (150)

FIG. 4 Coupler End Dimensions

design shall be tested to determine conformance to this specification.

10.2 Each coupling shall be tested as outlined in production testing of 11.2.

11. Test Methods

11.1 Prototype testing of each size of each particular design shall be as follows. Maintain the indicated test pressures for at least 1 min without leakage.

11.1.1 Pressure test to twice the MAWP after having been cycled (connected, closed, opened, and disconnected) three times.

11.1.2 Pressure test at MAWP while applying a moment of 1000 in.-lbs (113 N·M) to one coupling half attempting to twist it loose. Reapply MAWP after the moment is removed.

11.2 *Production Test*—Hydrostatically test each coupling to 1.5 times MAWP.

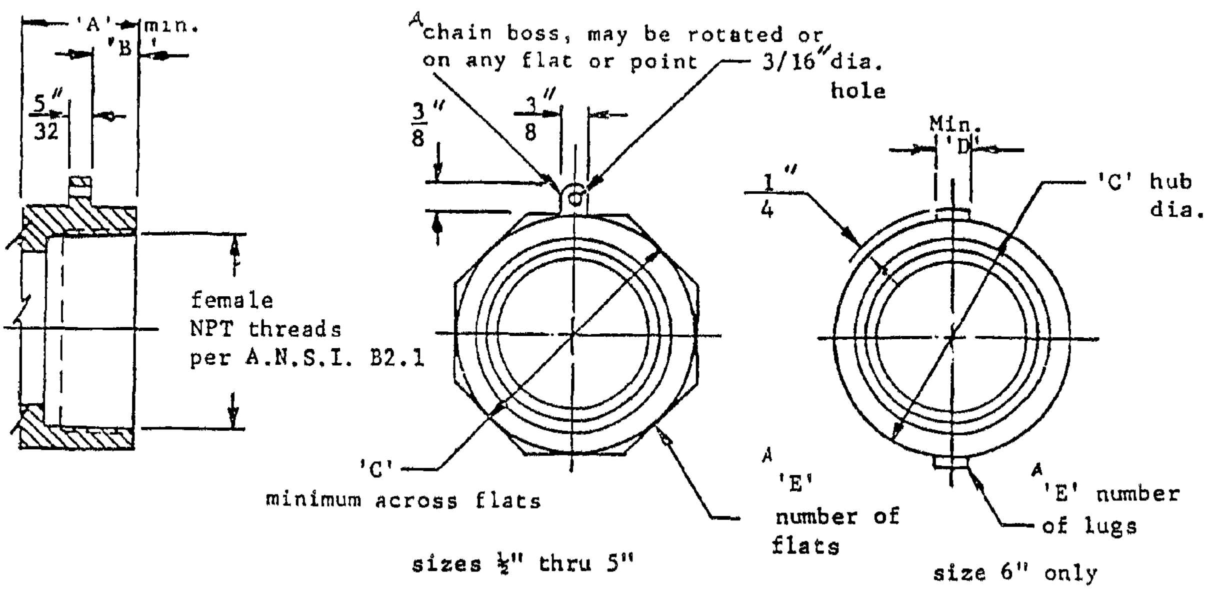
11.3 The test apparatus for 11.2 is to be a state-of-the-art device, designed to apply internal pressure to the coupling halves with gasket in place. Dye the fluid used to apply the hydrostatic pressure to facilitate observation of leakage should leakage occur.

12. Inspection

12.1 Nondestructive examination of welds shall be performed as required by ANSI B31.1. Additionally, 100 % radiography of all welds is required for couplings equal to or greater than 4-in. (100-mm) nominal diameter and when the nominal wall thickness is greater than 0.375 in. (9.4 mm).

12.2 The manufacturer is responsible for the inspection and testing of each coupling and assuring conformity to this specification. Samples selected for inspection, at least one coupling from each lot of 100 or fraction thereof, are to be dimensionally checked 100 % to verify adherence to this specification. Couplings having threads are to be inspected using proper thread gages. The entire lot of couplings is to be visually checked for porosity and other flaws that could adversely affect usability of this product.

12.3 The manufacturer shall afford the purchaser's inspector all reasonable facilities necessary to satisfy him that the material is being furnished in accordance with this specification. Inspection by the purchaser shall not interfere unnecessarily with the manufacturer's operations. All examinations and inspections shall be made at the place of manufacture,



NOTE 1—^A Chain boss to be provided at the manufacturer's discretion; number of flats or lugs provided are at the manufacturer's discretion. Unidentified tolerances; fractional ±1/32 (±0.8)

Size	Dimensions, in. (mm)				
	A	B	C (minimum)	D (minimum)	E
½	9/16 (14.3)	7/32 (5.6)	1.259 (32)	. . .	8
¾	9/16 (14.3)	7/32 (5.6)	1.259 (32)	. . .	8
1	9/16 (14.3)	7/32 (5.6)	1.441 (36.6)	. . .	8
1¼	9/16 (14.3)	7/32 (5.6)	1.787 (45.4)	. . .	8
1½	9/16 (14.3)	7/32 (5.6)	2.100 (53.3)	. . .	8
2	9/16 (14.3)	7/32 (5.6)	2.479 (63)	. . .	8
2½	¾ (19)	5/16 (7.9)	2.980 (75.7)	. . .	8
3	¾ (19)	5/16 (7.9)	3.599 (91.4)	. . .	8
4	1 (25.4)	13/32 (10.3)	4.703 (119.5)	. . .	8
5	1 (25.4)	13/32 (10.3)	5.723 (145.4)	. . .	8
6	1 1/16 (27)	NA	6.924 (175.9)	¾ (19)	2

FIG. 5 Adapter Female Threads End

unless otherwise agreed upon.

12.4 Other tests, when agreed upon, shall be made from material of the lots covered in the order.

13. Certification

13.1 When specified in the purchase order or contract, the manufacturer's certification shall be furnished to the purchaser stating that the samples representing each lot have been manufactured, tested, and inspected in accordance with this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

14. Product Marking

14.1 Each adapter and coupler shall be marked in accordance with MSS-SP-25 including the following:

14.1.1 ASTM designation of this specification.

14.1.2 "Cl I" if it is a Class I adapter or coupler.

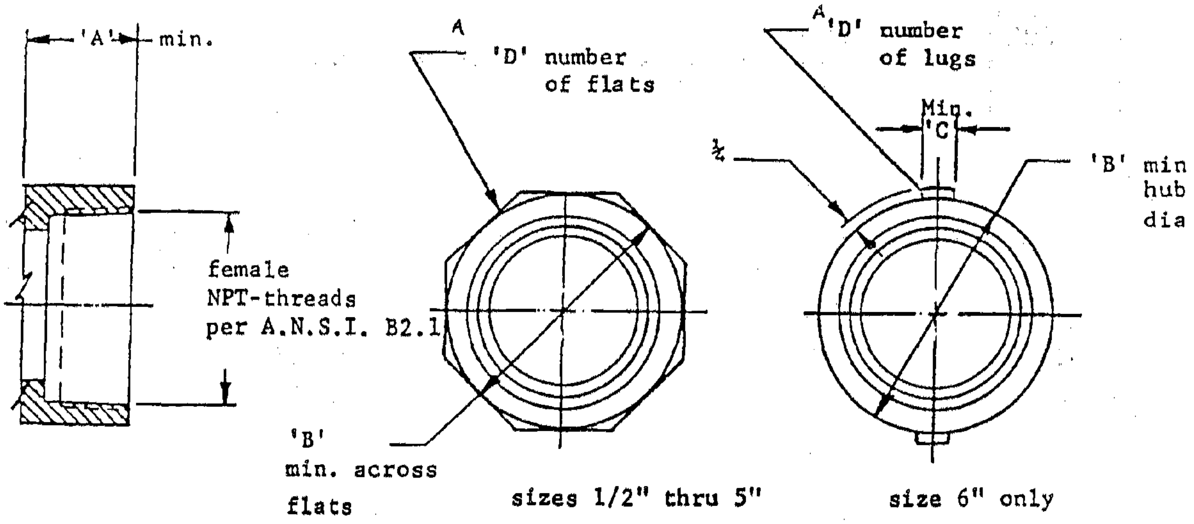
15. Packaging and Package Marking

15.1 The couplings shall be packed according to the manufacturer's standard practice, providing reasonable care to prevent lost or damaged parts in shipment.

15.2 Containers and packages shall be marked or tagged to adequately identify the contents or purchaser's order number.

16. Quality Assurance

16.1 The manufacturer of the quick disconnect couplings shall maintain the quality of the couplings that are designed,

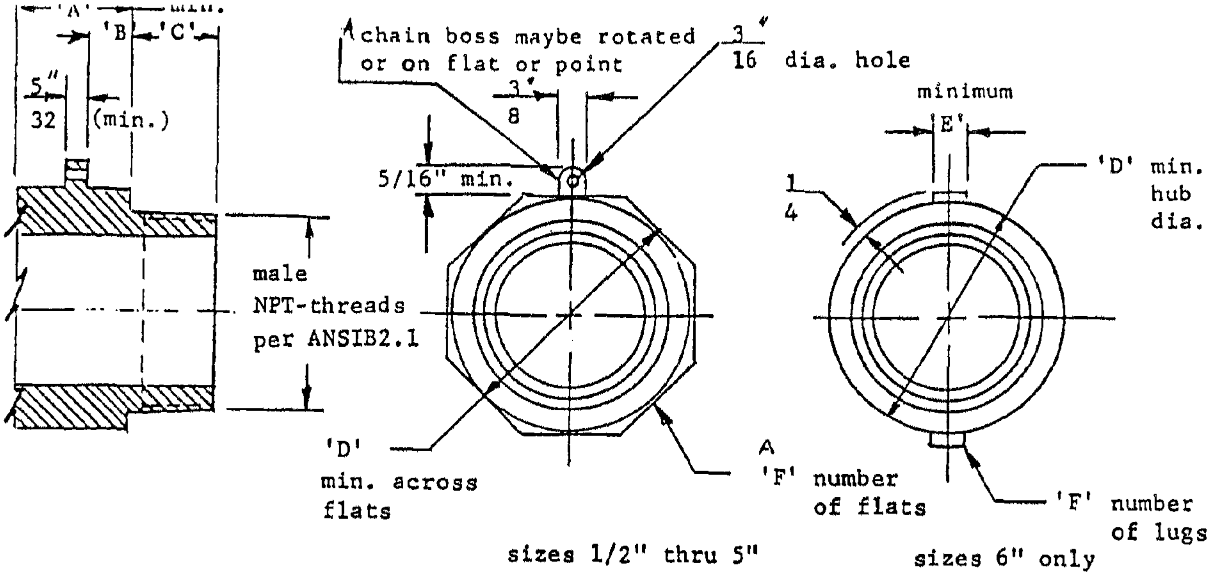


NOTE 1—[A] Number of flats or lugs provided is at the manufacturer's discretion.
Unidentified tolerance; fractional ±1⁄32 (±0.8)

Size	Dimensions, in. (mm)			
	A	B (minimum)	C (minimum)	D
½	43⁄64 (17.1)	1³⁄₁₆ (30.2)	. . .	8
¾	¾ (19)	1¼ (37.7)	. . .	8
1	¾ (19)	1½ (38.1)	. . .	8
1¼	¾ (19)	2 (50.8)	. . .	8
1½	⅞ (22.2)	2⁹⁄₃₂ (57.9)	. . .	8
2	⅞ (22.2)	2¾ (69.8)	. . .	8
2½	1⁵⁄₃₂ (29.4)	3⁵⁄₁₆ (84.1)	. . .	8
3	1⁵⁄₃₂ (29.4)	3¹⁵⁄₁₆ (100)	. . .	8
4	1⅜ (34.9)	4¹⁵⁄₁₆ (125.4)	. . .	8
5	1¾ (44.4)	6 (152.4)	. . .	8
6	1¼ (31.7)	7³⁄₁₆ (182.6)	¾ (19)	2

FIG. 6 Coupler Female Thread End

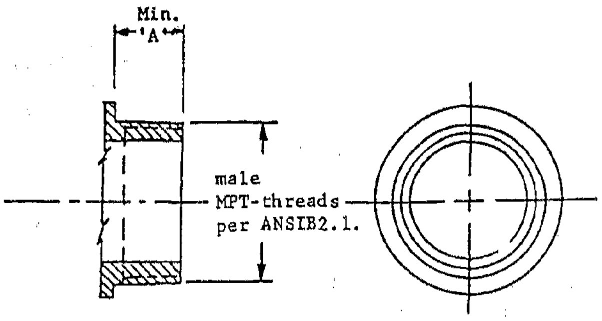
tested, and marked in accordance with this specification. At no time shall a coupling be sold with this specification designation that does not meet the requirements herein.



NOTE 1—^{A} Chain boss and number of flats or lugs provided is at the manufacturer's discretion.
Unidentified tolerance; fractional ±1/32 (±0.8)

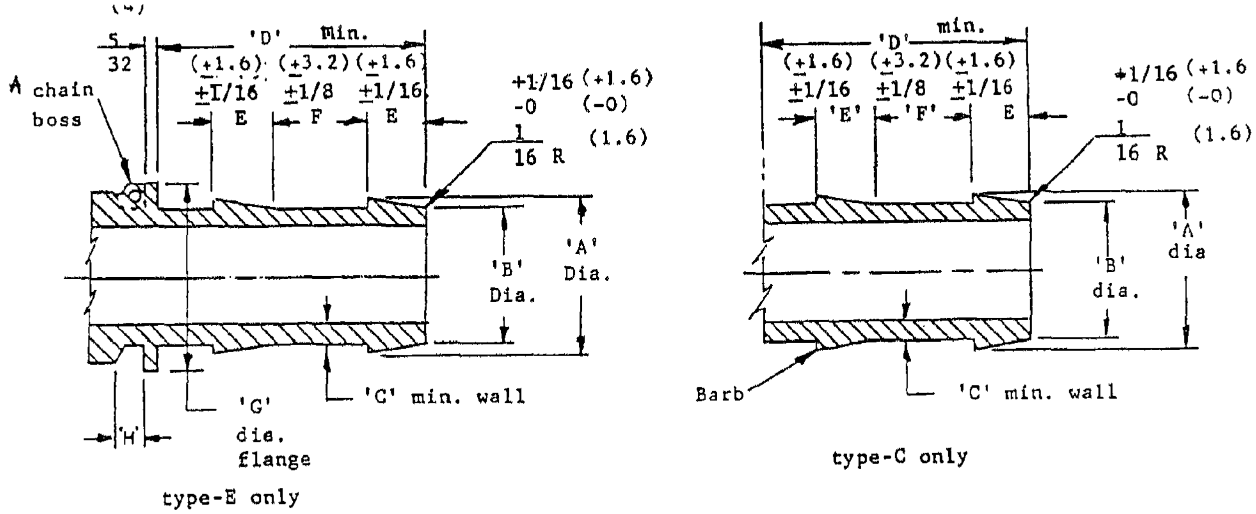
Size	Dimensions, in. (mm)					
	A	B	C (minimum)	D (minimum)	E (minimum)	F
½	9/16 (14.3)	13/64 (5.2)	5/8 (15.9)	(1.259) (32)	. . .	8
¾	9/16 (14.3)	13/64 (5.2)	5/8 (15.9)	1.259 (32)	. . .	8
1	9/16 (14.3)	13/64 (5.2)	7/8 (22.2)	1.441 (36.6)	. . .	8
1¼	(14.3)	13/64 (5.2)	7/8 (22.2)	1.787 (45.4)	. . .	8
1½	9/16 (14.3)	13/64 (5.2)	7/8 (22.2)	2.100 (53.3)	. . .	8
2	9/16 (14.3)	13/64 (5.2)	15/16 (23.8)	2.479 (63)	. . .	8
2½	¾ (19)	19/64 (7.5)	1 5/16 (33.3)	2.982 (75.7)	. . .	8
3	¾ (19)	19/64 (7.5)	1 7/16 (36.5)	3.599 (91.4)	. . .	8
4	1 (25.4)	27/64 (10.7)	1 9/16 (39.7)	4.703 (119.5)	. . .	8
5	1 (25.4)	27/64 (10.7)	1¾ (44.4)	5.723 (145.4)	. . .	8
6	1 (25.4)	. . .	1¾ (44.4)	6.924 (175.9)	¾ (19)	2

FIG. 7 Adapter Male Thread End



Size	Dimensions, in. (mm)
	A
½	⅝ (15.9)
¾	⅝ (15.9)
1	⅞ (22.2)
1¼	⅞ (22.2)
1½	⅞ (22.2)
2	15/16 (23.8)
2½	1 5/16 (33.3)
3	1 7/16 (36.5)
4	1 9/16 (39.7)
5	1¾ (44.4)
6	1¾ (44.4)

FIG. 8 Coupler Male Thread End



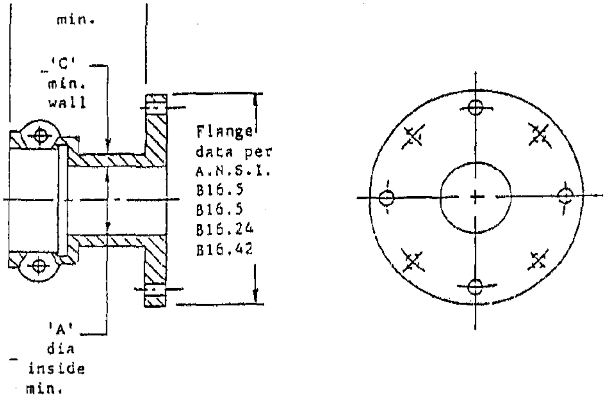
NOTE 1—[A] Chain boss provided at the manufacturer's discretion.

NOTE 2—Additional barbs may be provided at the manufacturer's discretion.

Unidentified tolerance's = +1⁄32 (±0.8)

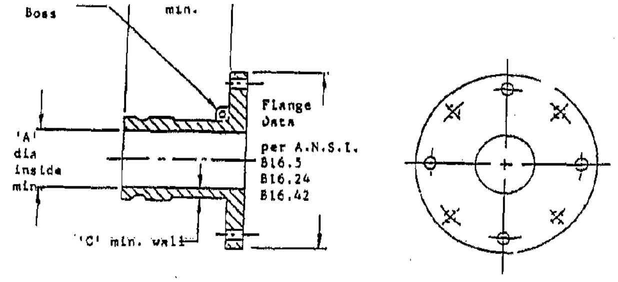
Size	Dimensions, in. (mm)							
	A	B	C	D	E	F	G	H
½	19⁄32 (15.1)	31⁄64 (12.3)	3⁄32 (2.4)	1 5⁄16 (33.3)	¼ (6.3)	¾ (19)	1¼ (31.7)	⅜ (9.5)
¾	27⁄32 (21.4)	47⁄64 (18.6)	3⁄32 (2.4)	2 (50.8)	¼ (6.3)	¾ (19)	1¼ (31.7)	⅜ (9.5)
1	1 5⁄64 (27.4)	63⁄64 (25)	3⁄32 (2.4)	2¼ (57.1)	⅜ (9.5)	¾ (19)	1⅝ (41.3)	7⁄16 (11.1)
1¼	1 11⁄32 (34.1)	1 15⁄64 (31.3)	⅛ (3)	2¼ (57.1)	⅜ (9.5)	13⁄16 (20.6)	2 (50.8)	7⁄16 (11.1)
1½	1 19⁄32 (40.5)	1 31⁄64 (37.7)	⅛ (3)	2⅜ (60.3)	⅜ (9.5)	13⁄16 (20.6)	2 5⁄16 (58.7)	7⁄16 (11.1)
2	2 3⁄32 (53.2)	1 31⁄32 (50)	⅛ (3)	2¾ (69.8)	⅜ (9.5)	1⅛ (28.6)	2 11⁄16 (68.3)	7⁄16 (11.1)
2½	2⅝ (66.7)	2 31⁄64 (63.1)	5⁄32 (4)	3⅛ (79.4)	7⁄16 (11.1)	1⅜ (34.9)	3 1⁄16 (81)	9⁄16 (14.3)
3	3⅛ (79.4)	2 31⁄32 (75.4)	5⁄32 (4)	4 (101.6)	½ (12.7)	1 9⁄16 (39.7)	3 25⁄32 (96)	9⁄16 (14.3)
4	4⅛ (104.8)	3 31⁄32 (100.8)	7⁄32 (5.6)	4¼ (107.9)	½ (12.7)	1¾ (44.4)	4⅞ (123.8)	9⁄16 (14.3)
5	5⅛ (130.2)	5 (127)	7⁄32 (5.6)	4½ (114.3)	½ (12.7)	1¾ (44.4)	6 9⁄16 (166.7)	⅝ (15.9)
6	6⅛ (155.6)	6 (152.4)	7⁄32 (5.6)	5½ (139.7)	½ (12.7)	2⅝ (66.7)	7⅛ (181)	¾ (19)

FIG. 9 Hose Shank End



Size	Dimensions, in. (mm)		
	A	B	C
½	11/16 (17.5)	2⅝ (66.7)	3/32 (2.4)
¾	13/16 (20.6)	2⅝ (66.7)	3/32 (2.4)
1	1 (25.4)	3 1/16 (77.8)	3/32 (2.4)
1¼	1 5/16 (33.3)	4 (101.6)	⅛ (3)
1½	1 9/16 (39.7)	4 (101.6)	⅛ (3)
2	1 15/16 (49.2)	4¼ (107.9)	⅛ (3)
2½	2 5/16 (58.7)	4¼ (107.9)	5/32 (4)
3	2⅞ (73)	4⅝ (117.5)	5/32 (4)
4	3½ (88.9)	4⅝ (117.5)	7/32 (5.6)
5	4⅜ (111.1)	5¼ (133.3)	7/32 (5.6)
6	5½ (139.7)	6⅝ (168.3)	7/32 (5.6)

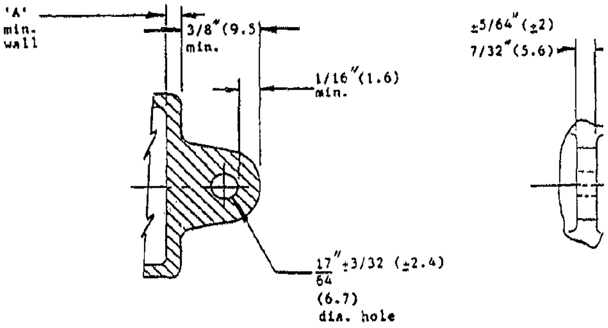
FIG. 10 Flanged Coupler ANSI Class 150



NOTE 1—Chain boss to be provided at the manufacturer's discretion.

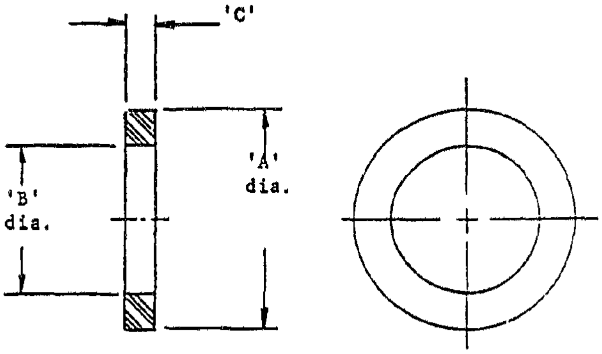
Size	Dimensions, in. (mm)		
	A	B	C
½	19/32 (15.1)	1¾ (44.4)	3/32 (2.4)
¾	11/16 (17.5)	1¾ (44.4)	3/32 (2.4)
1	13/16 (20.6)	2⅛ (54)	3/32 (2.4)
1¼	1″ (25.4)	2⅜ (60.3)	⅛ (3)
1½	1 9/32 (32.5)	2⅝ (66.7)	⅛ (3)
2	1 21/32 (42.1)	2¾ (69.8)	⅛ (3)
2½	2 1/16 (52.4)	2 13/16 (71.4)	5/32 (4)
3	2 11/16 (68.3)	2⅞ (73)	5/32 (4)
4	3 11/16 (93.4)	2⅞ (73)	7/32 (5.6)
5	4 11/16 (119.1)	3½ (88.9)	7/32 (5.6)
6	5 11/16 (144.5)	3½ (88.9)	7/32 (5.6)

FIG. 11 Flanged Adapter ANSI Class 150



Size	Dimensions, in. (mm)
	A
½	3/32 (2.4)
¾	3/32 (2.4)
1	3/32 (2.4)
1¼	⅛ (3)
1½	⅛ (3)
2	⅛ (3)
2½	5/32 (4)
3	5/32 (4)
4	7/32 (5.6)
5	7/32 (5.6)
6	7/32 (5.6)

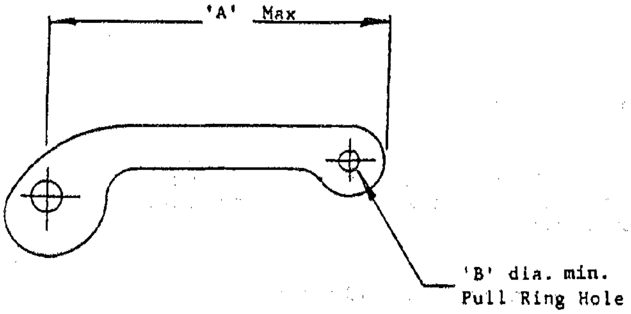
FIG. 12 Adapter Chain Boss End



NOTE 1—Unidentified Tolerance; Fractional = ±1/64 (±0.4)

Size	Dimensions, in. (mm)			Minimum Gasket Compression for Proper Sealing
	A	B	C	
½	1⅜ (34.9)	⅞ (22.2)	7/32 (5.6)	0.030 (0.762)
¾	1⅜ (34.9)	⅞ (22.2)	7/32 (5.6)	0.030 (0.762)
1	1 9/16 (39.7)	1 1/16 (27)	¼ (6.3)	0.030 (0.762)
1¼	1 15/16 (49.2)	1 23/64 (34.5)	¼ (6.3)	0.030 (0.762)
1½	2 3/16 (55.6)	1⅝ (41.3)	¼ (6.3)	0.030 (0.762)
2	2⅝ (66.7)	2 (50.8)	¼ (6.3)	0.030 (0.762)
2½	3⅛ (79.4)	2⅜ (60.3)	¼ (6.3)	0.030 (0.762)
3	3 23/32 (94.5)	3 (76.2)	¼ (6.3)	0.025 (0.635)
4	4⅞ (123.8)	4 (101.6)	¼ (6.3)	0.025 (0.635)
5	5 29/32 (150)	4⅞ (123.8)	¼ (6.3)	0.025 (0.635)
6	7 1/16 (179.4)	6 (152.4)	¼ (6.3)	0.025 (0.635)

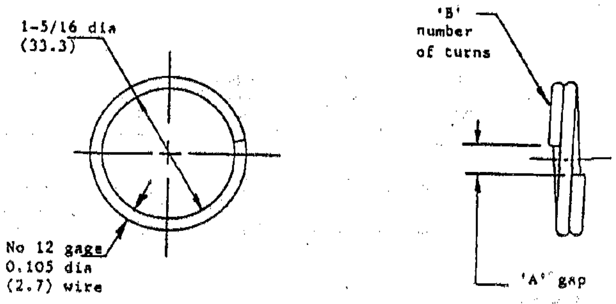
FIG. 13 Gasket



NOTE 1—Unidentified tolerance: fractional ±1⁄64 (±0.4)

Size	Dimensions, in. (mm)	
	A	B
½	1⅞ (47.6)	5⁄32 (4)
¾	1⅞ (47.6)	5⁄32 (4)
1″	1⅞ (47.6)	5⁄32 (4)
1¼	3 (76.2)	5⁄32 (4)
1½	3 (76.2)	5⁄32 (4)
2	3 (76.2)	5⁄32 (4)
2½	3 (76.2)	5⁄32 (4)
3	3¼ (82.5)	¼ (6.3)
4	3¼ (82.5)	¼ (6.3)
5	3¼ (82.5)	¼ (6.3)
6	4½ (114.3)	¼ (6.3)

FIG. 14 Cam Handle



NOTE 1—Unidentified tolerance: ±1⁄16 (±1.6); decimal ±0.002 (±0.05)

Size	Dimensions, in. (mm)	
	A	B
½	3⁄16 (4.8)	2
¾	3⁄16 (4.8)	2
1″	3⁄16 (4.8)	2
1¼	3⁄16 (4.8)	2
1½	3⁄16 (4.8)	2
2	3⁄16 (4.8)	2
2½	3⁄16 (4.8)	2
3	¼ (6.3)	3
4	¼ (6.3)	3
5	¼ (6.3)	3
6	¼ (6.3)	3

FIG. 15 Pull Ring, Cam Handle

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Designation: F 1123 – 87 (Reapproved 1998)

An American National Standard

Standard Specification for Non-Metallic Expansion Joints[1]

This standard is issued under the fixed designation F 1123; 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 specification provides the minimum requirements for construction, materials, performance, and dimensional requirements of arch-type non-metallic expansion joints.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The following safety hazards caveat pertains only to the test method described in this specification. *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:*

A 395 Specification for Ferritic Ductile Iron Pressure-Retaining Castings for Use at Elevated Temperatures[2]

D 1418 Practice for Rubber and Rubber Latices—Nomenclature[3]

2.2 *Federal Standard:*

Code of Federal Regulations, Title 30, Chapter I, Mine Safety and Health Administration[4]

2.3 *ANSI Standards:*

B16.1 Cast Iron Pipe Flanges and Flanged Fittings[5]

B16.5 Steel Pipe Flanges and Flanged Fittings[5]

B16.24 Bronze Flanges and Flanged Fittings[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *floating metallic flange type*—expansion joint having the tube, fabric plies, and cover brought up from the joint body to form a bead.

NOTE 1—This bead is molded into a groove in the metallic flange. Retaining rings are not required with this design.

3.1.2 *integral rubber flange type*—expansion joint having the tube, fabric plies, and cover brought up from the joint body to form a rubber flange.

NOTE 2—Additional plies or other reinforcement may be used in the flange to meet service conditions. Retaining rings must be used with this design.

3.1.3 *maximum allowable working pressure (MAWP)*—manufacturer's recommended maximum continuous operating pressure (lb/in.^{2} (Pa)).

3.1.4 *non-metallic flanged expansion joint*—flexible connector fabricated from natural or synthetic rubber and fabrics, usually with metal reinforcement, to isolate vibration and noise and provide stress relief in piping systems caused by thermal changes and other system movements.

4. Ordering Information

4.1 Orders for products under this specification shall include the following information:

4.1.1 Inside diameter of connecting pipes (joint ID).

4.1.2 Face-to-face dimension that is the flange-to-flange dimension into which the expansion joint is to be installed.

4.1.3 Maximum and minimum operating pressure in pounds-force per square inch gage (pascals).

4.1.4 Maximum and minimum operating temperature in °F (°C).

4.1.5 Flange drilling in accordance with the appendix titled "Common Flange Dimension/Drilling Chart" of the *Technical Handbook on Rubber Expansion Joints and Flexible Pipe Connectors*[6] (herein referred to as the *Technical Handbook*) or in accordance with special customer requirements.

4.1.6 Fluid to be handled.

4.1.7 This ASTM specification designation.

4.1.8 Movement data requirements (including shock or vibratory excursions if applicable).

4.1.9 Design certification burst test if required (see 9.1).

4.1.10 Hydrostatic or special tests if required (see 9.2).

4.1.11 Certification of expansion joint if required (see Section 12).

4.1.12 Certified detailed drawing of the expansion joint if required (see 12.2).

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[1] This specification is under the jurisdiction of ASTM Committee F-25 on Ships and Marine Technology and is the direct responsibility of Subcommittee F25.13 on Piping Systems.

Current edition approved Dec. 31, 1987. Published February 1988.

[2] *Annual Book of ASTM Standards*, Vol 01.02.

[3] *Annual Book of ASTM Standards*, Vol 09.01.

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

[5] Available from American Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017.

[6] The *Technical Handbook on Rubber Expansion Joints and Flexible Pipe Connections* is available from Fluid Sealing Association, 2017 Walnut St., Philadelphia, PA 19103.

5. Materials and Manufacture

5.1 Expansion joints shall be fabricated with an elastomeric tube reinforced with multiple plies of woven cloth or tire cord covered with synthetic rubber. The inner tube shall be a natural rubber, synthetic rubber, or blend of synthetic rubber that meets the requirements of this specification. The woven cloth or tire cord shall be nylon, polyester, fiberglass, or aramid. Cotton is not acceptable. The reinforcing fabric shall be impregnated with a compatible friction stock. Additional reinforcement to the fabric may be provided in the body of the expansion joint and may be solid metal rings or wire imbedded in the synthetic rubber. Tensile properties of the wire, if used, shall be as given in 5.2. Body rings, if used, must be welded before being installed in the expansion joint body. Welds must be 100 % penetration.

5.1.1 The list of elastomers used in expansion joints and rubber pipe in accordance with the *Technical Handbook*[6] lists acceptable natural rubber and synthetic elastomers for construction of non-metallic expansion joints.

5.2 Reinforcing wire shall have properties that allow the expansion joints to meet the requirements of this specification.

5.3 All expansion joints shall be manufactured with a cover of Hypalon or Neoprene (Chloroprene), in accordance with Practice D 1418. This cover material must consist of 100 % Hypalon or Chloroprene (not blended with any other elastomer) plus normal additives to provide for curing and a durometer between 50 and 75 on the Shore A Scale. Neoprene and Hypalon are selected as the best fire-retardant elastomer of the common types used for expansion joints. This material shall be certified flame resistant as outlined in 10.3.

5.4 *Integral Flanges*—The tube, fabric plies, and cover shall be brought up from the joint body to form an intregal flange. This rubber flange shall extend beyond the bolt holes of the retaining ring.

5.5 *Floating Metallic Flanges*—The metal flanges shall have a groove to accept the molded bead in the body at each end of the expansion joint bellows.

5.6 *Arches*—Arches may be either straight sided or long radius depending on the manufacturer's standard construction. The arch size and shape determine the movement capability of the joint. Minimum movement capability of single arch joints shall be in accordance with the *Technical Handbook*[6] table titled "Expansion Joint Movement Force/Spring Rate Capability." Movement capability information for multiple arch designs shall be available from the manufacturer.

5.7 *Metallic Flanges*:

5.7.1 Flanges shall be drilled in accordance with the *Technical Handbook*[6] appendix titled "Common Flange Dimension/Drilling Chart" or in accordance with the customer order as required, to match the mating flanges.

5.7.2 Metallic flanges shall meet the material requirements and pressure-temperature ratings in accordance with ANSI B16.1, B16.5, or B16.24.

5.8 *Retaining Rings*—Retaining rings for the integral flange type are installed behind the flanges and are drilled to match the flange drilling. The sections supplied for each flange should be split at the bolt holes to ensure a proper seal at all points when the bolts are tightened. The edge next to the rubber flange shall be broken or bevelled to prevent cutting the rubber flanges. Retaining rings must be a minimum thickness of ⅜ in. (9.5 mm) and shall be made of steel or ductile iron. Carbon steel shall be galvanized. Ductile iron shall be in accordance with Specification A 395.

6. Other Requirements

6.1 All expansion joints shall be designed for a minimum burst pressure of four times the maximum allowable working pressure. The design shall be based on analytical or experimental test of expansion joints of similar construction, class, type, and size. The design shall be certified by tests if ordered (see 4.1.9).

6.2 *Performance Requirements*—Single arch expansion joint movement shall not exceed the limits of the *Technical Handbook*[6] table referred to in 5.6 unless the manufacturer certifies that a proposed design can exceed the listed minimum movement capability to meet a special requirement greater than the minimum listed. Multiple arch-joint movement shall be of the manufacturer's certified design.

6.3 *Pressure Rating*—Expansion joints shall be limited to the pressures listed in the table "Pressure Characteristics of Rubber Expansion Joints" of the *Technical Handbook*[6].

7. Dimensions and Permissible Variations

7.1 Expansion joints shall be dimensioned in accordance with the *Technical Handbook*[6] table titled "Expansion Joint Movement/Force/Spring Rate Capability."

8. Workmanship, Finish, and Appearance

8.1 *Tube*—The tube shall be free from cuts and breaks or severe abrasions. Small depressions and indentations are acceptable as long as the surface of the elastomer is not broken.

8.2 *Integral Rubber and Fabric Flange*:

8.2.1 The face of an integral rubber and fabric flange shall be free of cuts or obvious breaks and shall be covered with rubber. The most critical portion of the flange is from the inside diameter of the joint out to the bolt holes.

8.2.2 Bolt holes shall be drilled cleanly through the rubber flange. The retaining rings shall be checked for proper alignment with bolt holes.

8.2.3 The outer edge of the flange shall be sealed with rubber, so that moisture from the atmosphere cannot attack the reinforcing fabrics.

8.2.4 The surface of the rubber flange against which the retaining rings are installed shall be covered with rubber. The surface may be irregular and building form tooling marks are not objectionable.

8.3 *Expansion Joint Cover and Body*—The cover of a joint does not contribute to the strength of the joint. Its purpose is to keep fluids from the atmosphere or surroundings from being absorbed into the body plies of fabric. Surface blemishes, such as flash, nylon wrap markings, grooves, and other indentations in the cover, over the arch, or in the body area, are not harmful as long as the body fabric is not exposed.

9. Number and Type of Tests

9.1 *Prototype Test*:

9.1.1 When required by the purchaser, an expansion joint of each size and type shall be burst tested to determine its maximum allowable working pressure (see 10.1).

9.1.2 The outer covering material of the expansion joint shall be tested to determine its self-extinguishing characteristics (see 10.3).

9.2 *Production Test*—When required by the purchaser, an expansion joint shall be hydrostatically tested to 1.5 times its maximum allowable working pressure at its rated maximum operating temperature (see 10.2).

10. Test Methods

10.1 *Burst Test*—Fill each joint to be tested with water before the application of pressure, allowing all air in the joint to escape. Apply pressure at a uniform rate until failure of the joint occurs. Consider the pressure at which any fluid leakage first occurs as the bursting pressure of the joint. The maximum allowable working pressure of the joint shall be less than or equal to ¼ the bursting pressure determined by this test.

10.2 *Hydrostatic Test*—Fill each joint to be tested with water before the application of pressure, allowing all air in the joint to escape. Apply pressure at a uniform rate up to the test pressure. The joint shall show no sign of leakage at the test pressure.

10.3 *Flame Resistance Test*—Test four specimens of the joint, ½ by 6 in. (12.7 by 152.4 mm) by the thickness of the expansion joint, in accordance with 30 CFR 18.65(*b*) through (*d*). Consider the expansion joint flame resistant if the tests of the four specimens meet the test requirements in 30 CFR 18.65(*e*).

11. Inspection

11.1 Inspection of the material shall be agreed upon between the purchaser and the supplier as part of the purchase contract.

12. Certification

12.1 When specified in the purchase order or contract, the manufacturer's certification shall be furnished to the purchaser stating that samples representing each lot have been manufactured, tested, and inspected in accordance with this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

12.2 When specified in the purchase order or contract, a certified drawing detailing the expansion joint shall be provided.

13. Product Marking

13.1 Each expansion joint shall be permanently marked or tagged with the following information:

13.1.1 Manufacturer's name or trademark.

13.1.2 Nominal diameter.

13.1.3 Manufactured date (month and year).

13.1.4 Maximum allowable working pressure and temperature (psi/°F (Pa/°C)).

13.1.5 Face-to-face dimension.

13.1.6 ASTM designation and year of issue of this specification.

14. Quality Assurance

14.1 The manufacturer of the expansion joint shall maintain the quality of the joints that are designed, tested, and marked in accordance with this specification. At no time shall a joint be sold with this specification designation that does not meet the requirements herein.

15. Keywords

15.1 arch-type non-metallic expansion joints; expansion joints

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

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An American National Standard

Standard Specification for Steam Traps and Drains[1]

This standard is issued under the fixed designation F 1139; 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 specification[2] provides the minimum requirements for the design, fabrication, pressure rating, marking, and testing of steam traps and drains.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The following safety hazards caveat pertains only to the test method portion of this specification. *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 *ANSI Standards:*

B16.1 Cast Iron Pipe Flanges and Flanged Fittings[3]

B16.3 Malleable-Iron Screwed Fittings[3]

B16.4 Cast-Iron Screwed Fittings[3]

B16.5 Steel Pipe Flanges and Flanged Fittings[3]

B16.11 Forged Steel Fittings Socket-Welding and Threaded[3]

B16.15 Cast Bronze Screwed Fittings[3]

B16.18 Cast Bronze Solder-Joint Pressure Fittings[3]

B16.22 Wrought Copper and Bronze Solder-Joint Pressure Fittings[3]

B16.24 Bronze Flanges and Flanged Fittings[3]

B16.34 Steel Valves, Flanged and Buttweld Ends[3]

B31.1 Power Piping[3]

2.2 *MSS Standards:*

SP-25 Standard Marking System for Valves, Fittings, Flanges, and Unions[4]

SP-51 150 lb Corrosion Resisting Cast Flanges and Flanged Fittings[4]

2.3 *ASME Standards:*

ANSI/ASME PTC 39.1 Condensate Removal Devices for Steam Systems[5]

ASME Boiler and Pressure Vessel Code, Section VIII, Division I, Pressure Vessels[5]

ASME Boiler and Pressure Vessel Code, Section IX, Welding and Brazing Qualifications[5]

3. Definitions of Terms Specific to This Standard

3.1 *cold condensate capacity (QC)*—maximum mass of condensate that the steam trap/drain can discharge in 1 h at a given pressure and temperature, the trap/drain being fully open (lb/h (kg/h)).

3.2 *drain*—device having no moving parts permitting the discharge of fluids at a fixed or adjustable rate.

3.3 *hot condensate capacity (QH)*—maximum mass of condensate that a steam trap/drain can discharge in 1 h at a given pressure and temperature (lb/h (kg/h)).

3.4 *hydrostatic proof test (PTHP)*—test used in determining maximum allowable pressure (PMA) and maximum allowable temperature (TMA) (lb/in.^{2}(kg/mm^2)).

3.5 *maximum allowable pressure (PMA)*—maximum pressure that the shell of the steam trap/drain can withstand permanently at a given temperature (lb/in.^{2}(kg/mm^2)).

3.6 *maximum allowable temperature (TMA)*—maximum temperature to which the shell of the steam trap/drain can be raised permanently (°F (°C)).

3.7 *maximum differential pressure* (ΔPMX)—maximum difference between operating pressure and operating back pressure (lb/in.^{2}(kg/mm^2)).

3.8 *maximum operating back pressure (PMOB)*—maximum permissible pressure measured at the outlet of the steam trap/drain allowing correct functioning (lb/in.^{2}(kg/mm^2)).

3.9 *maximum operating pressure (PMO)*—pressure for which a steam trap/drain is rated by the manufacturer.

3.9.1 *Discussion*—This pressure is normally a function of the limitations related to the internal mechanism of the steam trap/drain (lb/in.^{2}(kg/mm^2)).

3.10 *maximum test pressure (PTMX)*—maximum pressure applied to the steam trap/drain under test including its internal mechanism (lb/in.^{2}(kg/mm^2)).

3.11 *minimum differential pressure* (ΔPMN)—minimum difference between operating pressure and operating back

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[1] This specification is under the jurisdiction of ASTM Committee F-25 on Ships and Marine Technology and is the direct responsibility of Subcommittee F25.13 on Piping Systems.

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[2] This specification was developed from Fluid Controls Institute Standards, 69-1 Pressure Rating Standards for Steam Traps and 85-1 Standard Production Test for Steam Traps.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[4] Available from Manufacturer's Standardization Society of the Valve and Fittings Industry, Inc., 1815 N. Fort Myers Dr., Arlington, VA 22209.

[5] Available from American Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017.

pressure (lb/in.^{2}(kg/mm^2)).

3.12 *operating back pressure (POB)*—pressure measured at the outlet of the steam trap/drain under operating conditions (lb/in.^{2}(kg/mm^2)).

3.13 *operating differential pressure* (ΔP)—difference between the operating pressure and the operating back pressure (lb/in.^{2}(kg/mm^2)).

3.14 *operating pressure (PO)*—pressure measured at the inlet of the steam trap/drain under operating conditions (lb/in.^{2}(kg/mm^2)).

3.15 *operating temperature (TO)*—temperature measured at the inlet of the steam trap/drain under operating conditions (°F (°C)).

3.16 *performance characteristics tests*—tests carried out to determine the operational characteristics of a particular design of steam trap/drain.

3.17 *production tests*—tests carried out by the manufacturer to confirm that the steam trap/drain functions correctly.

3.17.1 *Discussion*—These tests may be witnessed by the purchaser or his representative. In this case, these tests are referred to as acceptance tests.

3.18 *steam trap*—self-contained valve that automatically drains the condensate from a steam containing enclosure while remaining tight to live steam, or if necessary, allowing steam to flow at a controlled or adjusted rate.

3.18.1 *Discussion*—Most steam traps will also pass noncondensable gases while remaining tight to live steam.

4. Ordering Information

4.1 Orders for products under this specification shall include the following information as applicable:

4.1.1 Performance characteristics required—See Section 7.

4.1.2 Certification of performance characteristics if required. See Section 7.

4.1.3 Nominal pipe size.

4.1.4 Maximum operating pressure, psig (kPa). See 3.5.

4.1.5 Capacity, lb/h (kg/h) (QC or QH). See 3.17 and 3.18.

4.1.6 Connection type (that is, threaded, socket weld, flanged). See 5.2.1.

4.1.7 *Materials*—external and internal.

4.1.8 Type of trap/drain.

4.1.9 Maximum test pressure, psig (kPa). See 3.12.

4.1.10 Maximum allowable pressure, psig (kPa). See 3.14.

4.1.11 Pressure differential (operating, maximum, or minimum, or combination thereof).

4.1.12 *Notice for Acceptance Test*—If the purchaser wishes to witness the production tests, this shall be specified in the order. See 8.2.

5. Materials and Manufacture

5.1 *Materials*:

5.1.1 The pressure ratings established under this specification are based upon materials of high quality produced under regular control of chemical and mechanical properties by a recognized process. The manufacturer shall be prepared to certify that his product has been so produced and that the mechanical and chemical properties thereof, as proved by test specimens and nondestructive testing or as documented by certifications from the producer or recognized distributor of these materials, are at least equal to the requirements of the appropriate specifications.

5.1.2 Housings of traps/drains, and other parts or bolting, or combination thereof, used for pressure retention, shall be constructed of materials in accordance with ANSI/ASME B31.1 or Section VIII, Division 1 of the ASME Boiler and Pressure Vessel Code.

5.1.3 Seals and parts, in addition to pressure containing parts and bolting used for pressure retention, shall be of materials suitable for the service.

5.1.4 Users are cautioned against applications with fluids that may react chemically with any materials used in these products.

5.1.5 For materials not having values of allowable stress tabulated in Section VIII, Division 1, allowable stresses shall be determined in accordance with the procedures outlined in Subsection C and Appendix P of Section VIII of the ASME Boiler and Pressure Vessel Code. Where it can be shown that the values of allowable stress listed for a particular material in one product form (because of similar chemistry, mechanical properties, directional properties, heat treatment, and so forth) are applicable to the same material in an unlisted product form, the listed values of allowable stress may be used.

5.2 *Manufacture*:

5.2.1 Steam traps/drains with end fittings in compliance with the following standards may be used within the pressure-temperature ranges permitted by the applicable standard provided the trap/drain housing (less end fittings) is satisfactory for these conditions:

5.2.1.1 ANSI B16.1,
5.2.1.2 ANSI B16.3,
5.2.1.3 ANSI B16.4,
5.2.1.4 ANSI B16.5,
5.2.1.5 ANSI B16.11,
5.2.1.6 ANSI B16.15,
5.2.1.7 ANSI B16.18,
5.2.1.8 ANSI B16.22,
5.2.1.9 ANSI B16.24,
5.2.1.10 ANSI B16.34, and
5.2.1.11 MSS SP-51.

5.2.2 Weld design details, welding, and nondestructive testing shall be in accordance with Section VIII, Division 1, of the ASME Boiler and Pressure Vessel Code. Welders and weld procedures shall be qualified in accordance with Section IX of the ASME Boiler and Pressure Vessel Code.

6. Requirements

6.1 *Pressure Rating and Design*:

6.1.1 The maximum allowable pressure (PMA) and maximum allowable temperature (TMA) rating for steam traps/drains conforming to this specification shall be established by at least one of the following methods:

6.1.1.1 Proof test in accordance with the requirements prescribed in paragraph UG-101 of Section VIII of the ASME Boiler and Pressure Vessel Code. If burst-type tests as outlined in UG-101(*m*) are used, it is not necessary to rupture the component. In this case, the value of "*B*" to be used in determining the maximum allowable pressure shall be the maximum pressure to which the component was subjected without rupture. Safety of personnel shall be given serious consideration when conducting hydrostatic tests. Components that have been subjected to a hydrostatic proof test shall not be offered for sale.

6.1.1.2 Design calculations in accordance with the requirements prescribed in Section VIII, Division 1 of the ASME Boiler and Pressure Vessel Code.

6.1.1.3 Hydrostatic proof test of a representative production sample to establish the maximum allowable pressure (PMA) and maximum allowable temperature (TMA) rating as described in 8.1.

6.1.1.4 Extensive and successful performance experience under comparable service conditions with similarly proportioned components of the same or similar material may be used as a basis for rating provided all other provisions of this specification are met.

6.2 *Production Tests*:

6.2.1 The manufacturer shall production test every steam trap/drain as described in Section 8 by one of the following test methods:

6.2.1.1 *Hydrostatic*—See 8.2.

6.2.1.2 *Steam*—See 8.3.

6.2.1.3 *Air*—See 8.4.

6.2.2 Samples of the traps/drains shall be visually examined and dimensionally checked to ensure that the traps/drains correspond to this specification and are marked in accordance with Section 9.

6.2.3 Sample steam traps/drains shall be given an operational check steam test to ensure that they open to discharge condensate and close satisfactorily in accordance with 8.5. This test does not apply to labyrinth (orifice) steam traps/drains.

7. Performance Characteristics

7.1 A manufacturer may describe the operation of a particular type of steam trap or drain by referring to one or more of the performance characteristics. When this is done, the associated tests described in 8.6 must be performed on a representative production sample. A brief explanation of the derivation of each characteristic is given as follows. Further details on test methods are specified in Section 8.

7.1.1 Certification of performance characteristics shall be available if required by the purchaser (see 4.1.2).

7.2 *Minimum Operating Pressure*—The steam trap shall be tested to determine the minimum pressure (atmospheric or above) at which correct opening and closing will occur.

7.3 *Maximum Operating Pressure (PMO)*—The steam trap shall be tested to determine the maximum pressure at which correct opening and closing will occur.

7.4 *Maximum Operating Back Pressure (PMOB)*—The steam trap shall be tested to determine the maximum pressure permissible at the outlet of the device that allows correct functioning.

7.5 *Air Venting Capability*—The steam trap/drain shall be tested to determine its ability to discharge air and other noncondensable gases.

7.6 *Operating Temperature (TO)*—The steam trap/drain shall be tested to determine the temperature at which the device operates and, in particular, the temperature at which it passes its specified capacity.

7.7 *Condensate Capacity (QH or QC)*—The steam trap/drain shall be flow tested to determine its condensate capacity throughout its operating pressure range.

7.8 *Live Steam Loss*—The steam trap shall be tested to determine the amount of live steam lost through the trap.

8. Test Methods

8.1 *Hydrostatic Proof Test*—Establish the maximum allowable pressure (PMA) and temperature (TMA) rating using a hydrostatic proof test of a representative production sample, chosen in accordance with 6.1.1.3 and perform as follows:

$$\text{PMA} = \frac{PTHP(f)}{5}$$

$$\times \frac{\text{specified minimum tensile strength}}{\text{average tensile of test specimens of pressure retaining components}}$$

$$\times \frac{\text{stress value at design temperature}}{\text{stress value at test temperature}}$$

8.1.1 Determine stress values in accordance with Section VIII, Division 1 of the ASME Boiler and Pressure Vessel Code or ANSI B31.1.

8.1.2 Casting quality factor (*f*) shall be 1.0 for wrought materials and 0.8 for castings.

8.1.3 Gasket leakage during test does not constitute failure unless a result of rupture of a pressure containing part.

8.1.4 Do not exceed water temperature of 125°F (50°C) during the test.

8.1.5 Retain certification of the hydrostatic proof test by the manufacturer and make available upon request.

8.2 *Hydrostatic Production Shell Test*—Give steam traps/drains a hydrostatic shell test at a pressure of 1.5 times its maximum allowable pressure rating at 68°F (20°C).

8.2.1 Do not exceed water temperature of 125°F (50°C) during the hydrostatic test.

8.2.2 The minimum duration of the shell test shall be as follows:

Nominal Pipe Size	*Duration*
⅛ through 2 (6-50 mm)	15 s
2½ through 8 (65-200 mm)	1 min
10 and over (250 mm)	3 min

8.2.2.1 Because of the complexity of product shape and size, the duration of the hydrostatic test may vary, but in no case shall this duration be less than that given in 8.2.2.

8.2.3 No visible leakage through pressure boundary walls or structural damage shall be evident during the shell tests.

8.3 *Steam Production Shell Test*—Test steam traps and drains no larger than 2-in. (50-mm) pipe size on saturated steam at their maximum operating pressure but not to exceed 250 psig (1820 kPa).

8.3.1 The minimum duration of the shell test shall be 15 s.

8.3.2 If this option is exercised, the manufacturer shall be able to certify that a prototype from each production lot of the same size steam trap/drain tested in this manner was subjected to a hydrostatic shell test in accordance with 8.2.

8.3.3 Show no visible leakage or structural damage during the shell tests.

8.4 *Air Production Shell Test*—Test traps and drains no larger than 2-in. (50-mm) pipe size for shell leaks on air at ambient temperature and 80 psi (551 kPa).

8.4.1 The minimum duration of the shell test shall be 15 s.

8.4.2 If this option is exercised, the manufacturer shall be able to certify that a prototype from each production lot of the same size steam trap/drain tested in this manner was subjected to a hydrostatic shell test in accordance with 8.2.

8.4.3 Visually detectable leakage through the pressure retaining walls is not acceptable.

8.5 *Operational Check Steam Test*—Feed the steam trap with steam and introduce condensate intermittently. When only steam is present, the steam trap shall close. On the introduction of condensate, the steam trap shall open (the time taken will vary as a function of the steam trap type). When the condensate has been discharged, the steam trap shall close again when steam enters the trap. The test is satisfied when at least one complete cycle has been performed.

8.6 *Performance Characteristics Tests*:

8.6.1 *Determination of Minimum Operating Pressure*—Carry out operational checks, as described in 8.5, while successively reducing the test pressure until the steam trap fails to open and close correctly. The minimum operating pressure is the lowest test pressure at which correct operation is observed.

8.6.2 *Determination of Maximum Operating Pressure*—Verify the maximum operating pressure of the steam trap by carrying out operational checks, as described in 8.5, while successively increasing the test pressure up to the steam trap's maximum operating pressure. The steam trap shall open and close correctly throughout the test.

8.6.3 *Determination of Maximum Operating Back Pressure*—Carry out operational checks, as described in 8.5, with the outlet from the steam trap connected to a vessel in which the pressure can be raised, independent of the test pressure upstream of the steam trap. While maintaining a reference pressure at the steam trap inlet, successively raise the pressure at its outlet until the steam trap fails to open and close correctly. The maximum operating back pressure is the highest pressure applied to the steam trap outlet at which correct operation is still observed.

8.6.4 *Determination of Air Venting Capability*—Introduce air at a specified temperature into the trap or upstream piping. Check the air venting capability by an air flow measurement carried out at minimum and maximum operating pressure. Record the temperature at the trap inlet.

8.6.5 *Determination of Operating Temperature*—Feed steam into the steam trap to effect closure. Introduce condensate, at saturation steam temperature, and, unless the steam trap opens immediately, allow to cool slowly at the steam trap's inlet. The operating temperature is the temperature of the condensate, measured at the inlet to the trap, at which the trap opens sufficiently to pass its specified capacity.

8.6.6 *Determination of Condensate Capacity*—Determine the capacity of the steam trap/drain by measuring the amount of condensate that is discharged from the device under specified conditions of pressure, pressure differential, and condensate temperature. Carry out the test with condensate at different temperatures and at different pressure within the steam trap/drain's operating range in accordance with ANSI/ASME PTC 39.1.

8.6.7 *Determination of Live Steam Loss*—Use several methods to determine the amount of live steam lost, if any, by the steam trap/drain in accordance with ANSI/ASME PTC 39.1.

9. Product Marking

9.1 Each steam trap/drain shall as a minimum be permanently marked with the following information:

9.1.1 Manufacturer's name or trademark.

9.1.2 Maximum operating pressure (PMO) or maximum differential pressure (ΔPMX) rating.

9.1.3 Maximum allowable pressure (PMA).

9.1.4 Indication of flow direction (arrow or word "inlet" or "outlet," or both).

9.1.5 ASTM designation of this specification.

9.2 Flanges, butt welding, threaded, or other ends complying with a standard listed in 5.2.1 may be marked in accordance with the applicable requirements of MSS SP-25 for dimensional identification purpose if desired.

9.3 *Omission of Markings on Trap/Drain Body*—On traps/drains whose size or shape limit the markings, markings shall be applied on an identification plate securely attached to the body. In no case shall the markings be hidden by the fixing elements of the steam trap.

9.4 *Additional Markings*—A manufacturer having complied with the requirements of Section 9 may include the following:

9.4.1 Mark any of the above mentioned items in more than one place, for example, if any item is marked on the body, it may also be repeated on an identification plate; or

9.4.2 Add any other markings, for example, catalog item numbers, providing that there is no risk of confusion between these markings and those mentioned in Section 9.

10. Quality Control

10.1 The trap/drain manufacturer shall maintain the quality of the traps/drain that are designed, tested, and marked in accordance with this specification. At no time shall a trap/drain be sold with this specification designation that does not meet the requirements herein.

11. Keywords

11.1 fluid discharge; steam drains; steam traps; valve