METRIC

MIL-DTL-15000M(NAVY)
29 February 2024
SUPERSEDING
MIL-DTL-15000L(NAVY)
8 August 2017

DETAIL SPECIFICATION

WATER-TESTING CHEMICALS, BOILER, SHIPBOARD USE

This specification is approved for use by the Department of the Navy and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers the composition and quality of chemicals used in testing shipboard boiler water and feedwater.

1.2 Classification. Boiler-water testing chemicals are of the following classes, as specified (see 6.2):

a. Class A - Nitric acid, reagent.

b. Class B - Mercuric nitrate solution.

c. Class F - Chloride indicator, powder.

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3 and 4 of this specification, whether or not they are listed.

2.2 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

AMERICAN CHEMICAL SOCIETY (ACS)

ACS Reagent Chemicals Specifications and Procedures for Reagents and Standard-Grade Reference Materials

(Copies of this document are available online from https://pubs.acs.org/isbn/9780841230460#.)

ASTM INTERNATIONAL

ASTM D1193 - Standard Specification for Reagent Water

ASTM D1293 - Standard Test Methods for pH of Water

ASTM E169 - Standard Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

ASTM E287 - Standard Specification for Laboratory Glass Graduated Burets

Comments, suggestions, or questions on this document should be addressed to Commander, Naval Sea Systems Command, ATTN: SEA 05S, 1333 Isaac Hull Avenue, SE, Stop 5160, Washington Navy Yard, DC 20376-5160 or emailed to usn.ncr.comnavseasyscomdc.mbx.command-standards@us.navy.mil, with the subject line "Document Comment". Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at https://assist.dla.mil.

AMSC N/A FSC 6810

ASTM E288 - Standard Specification for Laboratory Glass Volumetric Flasks

ASTM E969 - Standard Specification for Glass Volumetric (Transfer) Pipets

(Copies of these documents are available online at www.astm.org.)

2.3 Order of precedence. Unless otherwise noted herein or in the contract, in the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Chemical material. Materials used in the manufacture of each of the chemicals covered by this specification shall be in accordance with the American Chemical Society (ACS) specification, ACS Reagent Chemicals: Specifications and Procedures for Reagents and Standard-Grade Reference Materials.

3.1.1 Class A nitric acid reagent. The class A nitric acid reagent shall be a 1.0 normal (N) solution of ACS reagent grade nitric acid in ASTM D1193, type II reagent water and shall conform to the characteristics in table I when tested as specified herein.

TABLE I. Class A nitric acid reagent characteristics.

Characteristic	Minimum	Maximum	Test Paragraph
Volume, U.S. liquid quart	1.0	—	4.1.4
Normality, N	0.98	1.02	4.2.1

3.1.2 Class B mercuric nitrate solution. The class B mercuric nitrate solution shall be a 0.5 N solution of ACS reagent grade mercuric nitrate in ASTM D1193, type II reagent water and the minimum amount of nitric acid necessary to dissolve the mercuric nitrate, and shall conform to the characteristics in table II when tested as specified herein.

TABLE II. Class B mercuric nitrate solution characteristics.

Characteristic	Minimum	Maximum	Test Paragraph
Volume, U.S. liquid ounces	4.0	—	4.1.4
Insoluble material	None	Faint trace	4.2.2.1
Normality, N	0.475	0.525	4.2.2.2
pH	2.2	—	4.2.2.3

3.1.3 Class F chloride indicator powder. The class F chloride indicator powder shall be a dry mixture of 0.25-gram ACS reagent grade diphenylcarbazone and 0.02-gram ACS reagent grade bromophenol blue in a number 00 gelatin capsule, and shall conform to the characteristics in table III when tested as specified herein.

TABLE III. Class F chloride indicator powder characteristics.

Characteristic	Minimum	Maximum	Test Paragraph
Diphenylcarbazone, grams	0.212	0.288	4.2.3.2
Bromophenol blue, grams	0.017	0.023	4.2.3.3
Performance, milliliters	24.85	25.15	4.2.3.4

4. VERIFICATION

4.1 Conformance inspection. Conformance inspection shall consist of the samples specified in table IV for the tests of 4.2.1 through 4.2.3.

4.1.1 Lot. A lot shall consist of all solutions or chemicals from one batch. In cases in which it is not practicable to define the sampling lot in this manner, the lot shall consist of all solutions or chemicals of one class for delivery at one time.

4.1.2 Sampling for tests. Sampling of a lot for test purposes shall be as specified in table IV.

TABLE IV. Required samples for government acceptance testing.

Class	Description	Samples per Lot	Test Paragraph
Class A	Nitric acid, reagent	3 bottles	4.2.1
Class B	Mercuric nitrate solution	3 bottles	4.2.2
Class F	Chloride indicator, powder	6 capsules in one bottle	4.2.3

4.1.3 Examination of filled containers. Each filled container in the sample selected as specified in 4.1.2 shall be examined to verify compliance with all requirements of this specification regarding fill, marking, and other requirements not involving tests. Containers shall be examined for defects of the container, including leakage. Each sample-filled container shall also be weighed to determine the amount of the contents. Any container in the sample having one or more defects or containing less than the required amount of chemical shall be cause for rejection of that container.

4.1.4 Test glassware, reagents, and conditions. Volumetric glassware used in the test procedures (see 4.2) shall be class A glassware in accordance with ASTM E287, ASTM E288, or ASTM E969, as applicable. Water used in the test procedures shall conform with ASTM D1193, type II reagent water. Ethanol used in the test procedures shall be ACS spectrophotometric grade 95 percent. Test solutions shall be prepared from reagent grade chemicals in accordance with ACS specification, ACS Reagent Chemicals: Specifications and Procedures for Reagents and Standard-Grade Reference Materials. Titrations shall be conducted at 20 to 25 °C. Ultraviolet-Visible (UV-Vis) Quantitative Analysis shall be conducted in accordance with ASTM E169 using either a double-beam or single-beam instrument.

4.1.5 Rejection of lots. If a sample fails to pass any of the tests specified in table IV, the production lot represented by that sample shall be rejected. As specified (see 6.2), delivery of the material shall be withheld pending notification of the results of tests.

4.1.6 Test reports. When specified (see 6.2), test reports shall be prepared.

4.2 Test procedures. The following test procedures shall be followed:

4.2.1 Class A nitric acid reagent. Prepare 0.050 N sodium carbonate (2.650 grams Na_2CO_3 per liter) from ACS reagent grade sodium carbonate that has been dried at 250 °C for a minimum of 4 hours and then cooled in a desiccator. Transfer 50 milliliters (mL) of the sample nitric acid reagent to a 500-mL class A volumetric flask using a class A volumetric pipet. Fill the volumetric flask to the line using reagent water, stopper, and invert three times to mix. Use the diluted nitric acid sample solution to titrate 40 mL of the 0.05 N sodium carbonate solution to which four drops of methyl purple indicator (see appendix A) have been added. Continue the titration until the solution passes through the grey transition phase and reaches the final purple end point (see appendix A). Do not boil to remove carbon dioxide. Record the volume in mL of diluted nitric acid required to reach the end point. Calculate the normality of the nitric acid reagent using the following formula:

$$N_1 = 10 \times \frac{V_2 N_2}{V_1}$$

Where:

N_1 = Normality of nitric acid reagent sample.

V_1 = Volume in mL of diluted nitric acid sample required to reach the methyl purple end point.

N_2 = Normality of standard sodium carbonate solution (0.05 N).

V_2 = Volume in mL of standard sodium carbonate solution (40 mL).

4.2.2 Class B mercuric nitrate solution.

4.2.2.1 Stability. Allow the submitted bottles of mercuric nitrate solution to stand a minimum of 12 hours. Examine the mercuric nitrate solution in the bottles for insoluble material.

4.2.2.2 Normality. Prepare 0.025 N sodium chloride solution using ACS reagent grade sodium chloride that has been dried at 110 °C for a minimum of 3 hours. Transfer 25 mL of the sample mercuric nitrate solution to a 500-mL class A volumetric flask using a class A volumetric pipet. Fill the volumetric flask to the line using reagent water, stopper, and invert three times to mix. Use the diluted mercuric nitrate solution to titrate 20 mL of the 0.025 N sodium chloride solution in a clear glass beaker or Erlenmeyer flask to which four drops of 0.2 N nitric acid and four drops of chloride indicator (see appendix A) have been added. Continue the titration until a blue-violet color, as viewed by transmitted light, persists throughout the solution. Record the volume in mL of mercuric nitrate required to reach the end point. Calculate the normality of the mercuric nitrate using the following formula:

$$N_1 = 20 \times \frac{V_2 N_2}{V_1}$$

Where:

N_1 = Normality of mercuric nitrate solution.

V_1 = Volume in mL of diluted mercuric nitrate sample required to reach the chloride indicator end point.

N_2 = Normality of standard sodium chloride solution (0.025 N).

V_2 = Volume in mL of standard sodium chloride solution (20 mL).

4.2.2.3 pH. Determine pH of the diluted mercuric nitrate solution prepared in 4.2.2.1 by means of a standard pH meter using the glass electrode in accordance with ASTM D1293. Record the pH value to the nearest 0.1.

4.2.3 Class F chloride indicator powder.

4.2.3.1 Preparation of test chloride indicator solution. Transfer the contents of one capsule into a clean, dry 50-mL volumetric flask. Tap the capsule gently to remove contents completely. Add approximately 25 mL of ACS spectrophotometric grade 95 percent ethanol, stopper, and shake to thoroughly dissolve. Add 95 percent ethanol to the mark and stopper the flask. Invert the flask to mix.

4.2.3.2 Diphenylcarbazone. Transfer 100 mL of reagent water to a 150-mL beaker using a volumetric pipet. Add the following to this water in the following order: 0.20 mL of the chloride indicator solution prepared as specified in 4.2.3.1, six drops of 0.2 N nitric acid, and 1.0 mL of 0.025 N mercuric nitrate. Stir with a stirring rod until color appears uniform throughout the solution. Wait at least 2 minutes but no more than 5 minutes for the color to develop. Measure the absorbance of the solution against a water reference at 520 nanometers' wavelength, using a Visible (Vis) or UV-Vis spectrophotometer. For a double beam instrument, use matched optical 10-nanometer absorption cells. For a single beam instrument, either use the same 10-nanometer cell for the water reference and sample measurements, or use matched 10-nanometer cells. Record the absorbance. Calculate the quantity of diphenylcarbazone in the capsule as follows:

$$DPC = 0.309 \times A_{520\,nm}$$

Where:

DPC = Diphenylcarbazone, grams/capsule.

$A_{520\,nm}$ = Absorbance at 520 nanometers.

4.2.3.3 Bromophenol blue. Transfer a 1.0-mL aliquot of the chloride indicator solution prepared as specified in 4.2.3.1 to a 100-mL volumetric flask containing 25 mL of 95 percent ethyl alcohol. Add approximately 60 mL of reagent water and 1 mL of 0.2 N nitric acid and dilute to 100 mL with reagent water. Stopper and mix thoroughly. Prepare a reference blank containing 25 mL of 95 percent ethyl alcohol and 1 mL of 0.2 N nitric acid diluted to 100 mL with reagent water. Stopper and mix thoroughly. Measure the absorbance of the sample against the reference blank at 438 nanometers' wavelength using a Vis or UV-Vis spectrophotometer. For a double beam instrument, use matched optical 10-nanometer absorption cells. For a single beam instrument, either use the same 10-nanometer cell for the reference blank and sample measurements, or use matched 10-nanometer cells. Record the absorbance. Calculate the quantity of bromophenol blue in the capsule as follows:

$$BPB = (0.15 \times A_{438\,nm}) - (0.04 \times DPC)$$

4.2.3.4 Indicator performance. Add five drops of the chloride indicator solution prepared as specified in 4.2.3.1 to 25 mL of the 0.025 N sodium chloride solution containing four drops of 0.2 N nitric acid. The sodium chloride solution shall be prepared using ACS reagent grade sodium chloride that has been dried at 110 °C for a minimum of 3 hours. Titrate with standard 0.025 N mercuric nitrate solution until a blue-violet color, as viewed by transmitted light, persists throughout the solution. Record the volume in mL of mercuric nitrate required to reach the end point.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Service or Defense Agency, or within the military service's system commands. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. The reagent chemicals covered by this specification are intended to be used in testing shipboard boiler water and feedwater in accordance with the procedure requirements of Naval Ships' Technical Manual S9086-GX-STM-020/220, Boiler Water/Feedwater Test and Treatment. These chemicals are used in conjunction with MIL-W-16642. Current stocks of materials may be used until depleted, future requisitions for materials should refer to the current version of this specification.

6.2 Acquisition requirements. Acquisition documents should specify the following:

a. Title, number, and date of this specification.

b. Class or classes of chemicals required (see 1.2).

c. Test samples required (see 4.1.2).

d. Requirements for delayed delivery of the material pending results of tests (see 4.1.5).

e. When test reports are required (see 4.1.6).

f. Packaging and marking requirements (see 5.1, 6.3, and 6.4).

g. Safety Data Sheet (SDS), when required (see 6.5).

6.3 Suggested marking. In addition to any special marking specified (see 6.2), unit, interior, and exterior containers should be marked in accordance with MIL-STD-129 and should include the following:

a. National stock number.

b. Name and class of item.

c. Manufacturer's or contractor's name, or both.

d. Contract number.

e. Quantity/net content.

f. Date of manufacture, month, and year.

g. Expiration date of 36 months from date of manufacture.

h. Note: "Mandatory shelf-life material. Discard material after expiration date."

i. Lot or batch number.

6.4 Hazardous chemicals. As specified (see 6.2), each package unit of hazardous chemicals should have affixed thereto warning labels in accordance with Code of Federal Regulations (CFR) 49 CFR 171-199, 29 CFR 1910.1200, or appropriate Department of Defense instructions.

6.5 Safety data sheets (SDS). When required (see 6.2), contracting officers will identify those activities requiring copies of completed SDSs prepared in accordance with FED-STD-313. In order to obtain the SDS, FAR clause 52.223-3 must be in the contract.

6.6 Shelf-life. This specification covers items where the assignment of a Federal shelf-life code is a consideration. Specific shelf-life requirements should be specified in the contract or purchase order, and should include, as a minimum, shelf-life code, shelf-life package markings in accordance with MIL-STD-129 or FED-STD-123, preparation of a materiel quality storage standard for type II (extendible) shelf-life items, and a minimum of 85 percent shelf-life remaining at time of receipt by the Government. These and other requirements, if necessary, are in DoDM 4140.27, *DoD Shelf-Life Management Program*. The shelf-life codes are in the Federal Logistics Information System Total Item Record. Additive information for shelf-life management may be obtained from DoDM 4140.27, or the designated shelf-life Points of Contact (POC). The POC should be contacted in the following order: (1) the Inventory Control Points that manage the item and (2) the DoD Service and Agency administrators for the DoD Shelf-Life Program. Appropriate POCs for the DoD Shelf-Life Program can be contacted through the DoD Shelf-Life Management website: https://www.shelflife.dla.mil.

6.7 National stock numbers (NSN). Table V provides a list of NSNs assigned which correspond to this specification. This list may not include all possible NSNs associated with this document.

TABLE V. List of applicable NSNs.

Class	Description	NSN	SLC [1/]	Unit of Issue
Class A	Nitric acid reagent, 1.0 N	6810-00-270-9978	Q [2/]	QT [3/]
Class B	Mercuric nitrate, 0.5 N	6810-00-281-4163	Q	BT [4/] (4 oz)
Class F	Chloride indicator	6810-00-753-4907	Q	BT (12 capsules)

NOTES:
[1/] SLC: Shelf-Life Code.
[2/] Q: 3 years (36 months).
[3/] QT: Quart.
[4/] BT: Bottle.

6.8 Subject term (key word) listing.

Indicator, chloride
Indicator, methyl purple
Mercuric nitrate
Nitric acid
Reagent

6.9 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

TITRATION INDICATORS

A.1 SCOPE

A.1.1 Scope. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance. This appendix covers the composition and preparation of indicators used in the titration tests for nitric acid (see 4.2.1) and mercuric nitrate (see 4.2.2.2).

A.2 APPLICABLE DOCUMENTS

A.2.1 General. The documents listed in this section are specified in this appendix. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in this appendix, whether or not they are listed.

A.2.2 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

SOCIETY OF DYERS AND COLOURISTS (SDC)

Colour Index

(Copies of this document are available online from http://colour-index.com.)

A.3 TITRATION INDICATORS USED IN VERIFICATION TESTS

A.3.1 Methyl purple indicator.

A.3.1.1 Description. The methyl purple indicator used to detect the titration end point in the test for class A nitric acid reagent (see 4.2.1) shall consist of a mixture of methyl red sodium salt (Chemical Abstracts Service [CAS] Registry Number 845-10-3) and a blue dye dissolved in reagent grade water or other solvent such that the pH of the titrand will be green at pH 5.5, have a grey or clear transition phase, and will change to purple at a pH of 4.8±0.2. This indicator was originally described by now-expired U.S. Patent No. 2,416,619.

A.3.1.2 Preparation. Table A-I provides a list of some blue dyes that have been successfully used to prepare methyl purple indicator, including the CAS registry number and a cross reference to the Colour Index. U.S. Patent No. 2,416,619 provides specific preparation instructions for several of these dyes.

TABLE A-I: Blue dyes that have been successfully used to prepare methyl purple indicator.

Blue dye	CAS registry number	Color index number	
		1st edition 1/	2nd edition 2/
Acid Blue 5	3374-30-9	714	42,052
Acid Blue 7	3486-30-4	673	42,080
Acid Blue 22	28631-66-5	707	42,755
Acid Blue 102	6856-08-2	833	50,320
Direct Blue 1	2610-05-1	518	24,410

NOTES:

1/ Colour Index, Society of Dyers and Colourists, Yorkshire England, 1st ed., 1924.

2/ Colour Index, Society of Dyers and Colourists, Yorkshire England, 2nd ed., 1957, and later editions.

A.3.1.3 Unacceptable substitutes. Neither methyl red without a blue dye, nor methyl orange, nor methyl violet, shall be considered acceptable substitutes in this application.

A.3.2 Chloride indicator.

A.3.2.1 Description. The chloride indicator used to detect the titration end point in the test for class B mercuric nitrate solution (see 4.2.2.2) shall consist of a mixture of diphenylcarbazone (CAS Registry Number 538-62-5) and bromophenol blue (CAS Registry Number 115-39-9), dissolved in ACS reagent grade ethanol, methanol, or isopropanol.

A.3.2.2 Preparation. Preparation instructions are provided by now-expired U.S. Patent No. 2,784,064. Pre-mixed chloride indicator is available commercially. Class F chloride indicator powder prepared in accordance with 4.2.3.1 is acceptable for this application.

CONCLUDING MATERIAL

Custodian:
Navy – SH

Preparing activity:
Navy – SH
(Project 6810-2024-005)

Review activities:
Navy – OS
DLA – GS

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at https://assist.dla.mil.