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MILITARY HANDBOOK

METALS AND ALLOYS, RAPID ON-SITE IDENTIFICATION OF (RECOMMENDED PROCEDURE FOR CHEMICAL SPOT TESTING AND ASSOCIATED PHYSICAL TESTS TO VERIFY METAL ALLOY CLASSIFICATION) (METRIC)

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DEPARTMENT OF THE NAVY
NAVAL SEA SYSTEMS COMMAND

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Metals and Alloys, Rapid On-Site Identification of (Recommended Procedure for Chemical Spot Testing and Associated Physical Tests to Verify Metal Alloy Classification) (Metric)

1. This standardization handbook was developed by the Department of Defense in accordance with established procedure.

2. This publication was approved on 26 December 1984 for printing and inclusion in the military standardization handbook series.

3. This document provides basic and fundamental information on the rapid on-site identification of metals and alloys which include both chemical spot testing and associated physical tests to identify metal alloy classification. The handbook is not intended to be referenced in purchase specifications except for informational purposes, nor shall it supersede any specification requirements.

4. Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Sea Systems Command, SEA 55Z3, Department of the Navy, Washington, DC 20362 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FOREWORD

The purpose of this handbook is to provide chemical spot testing and similar methods for identification of metals and coatings on metal. These test procedures are qualitative only. Quantitative results should be obtained by spectrographic or chemical analytical procedures or by one or more special testing methods which may be applicable for the purpose.

CONTENTS

FIGURES

TABLE

1. SCOPE

1.1 This handbook describes the equipment and procedures for rapid on-site identification of metals and alloys. These procedures are not intended for use when quantitative chemical analysis is required, but will provide a reliable means of identifying the most common categories of metals and alloys.

2. REFERENCED DOCUMENTS

2.1 Issues of documents. The following documents, of the issue in effect on date of invitation for bids or request for proposal, form a part of this handbook to the extent specified herein.

SPECIFICATIONS

FEDERAL

- QQ-B-626 - Brass, Leaded and Nonleaded: Rod, Shapes, Forgings, and Flat Products with Finished Edges (Bar and Strip).
- QQ-B-637 - Brass, Naval: Rod, Wire, Shapes, Forgings, and Flat Products with Finished Edges (Bar, Flat Wire, and Strip).
- QQ-B-639 - Brass, Naval: Flat Products (Plate, Bar, Sheet, and Strip).
- QQ-B-750 - Bronze, Phosphor; Bar, Plate, Rod, Sheet, Strip, Flat Wire, and Structural and Special Shaped Sections.
- QQ-C-390 - Copper Alloy Casting, (Including Cast Bar).
- QQ-C-591 - Copper-Silicon, Copper-Zinc-Silicon, and Copper-Nickel-Silicon Alloys: Rod, Wire, Shapes, Forgings, and Flat Products (Flat Wire, Strip, Sheet, Bar, and Plate).
- QQ-N-281 - Nickel-Copper Alloy Bar, Rod, Plate, Sheet, Strip, Wire, Forgings, and Structural and Special Shaped Sections.
- QQ-N-286 - Nickel-Copper-Aluminum Alloy, Wrought.
- QQ-S-763 - Steel Bars, Wire, Shapes, and Forgings, Corrosion-Resisting.
- QQ-W-390 - Wire, Nickel-Chromium-Iron Alloy.

MILITARY

- MIL-S-870 - Steel Castings, Molybdenum Alloy.
- MIL-S-872 - Steel Bars, Billets, and Forgings - Carbon-Molybdenum Alloy.
- MIL-S-1222 - Studs, Bolts, Hex Cap Screws, and Nuts.
- MIL-N-7786 - Nickel-Chromium Alloy, Sheet and Strip, Age-Hardenable Annealed.
- MIL-N-8550 - Nickel Alloy, Bars and Forgings, 1200° to 1500°F Operating Temperatures.
- MIL-S-16216 - Steel Plate, Alloy, Structural, High Yield Strength (HY-80 and HY-100).
- MIL-T-16420 - Tube, Copper-Nickel Alloy, Seamless and Welded (Copper Alloy Numbers 715 and 706).

MILITARY (Continued)

MIL-S-16974 - Steel Bars, Billets, Blooms and Slabs, Carbon and Alloy (For Reforging or Other Operations Before Heat Treatment).
MIL-R-17131 - Rods and Powders, Welding, Surfacing.
MIL-S-18410 - Steel Bars, Billets, and Forgings - Chromium-Molybdenum Alloy.
MIL-T-20155 - Tube and Pipe, Carbon-Molybdenum Alloy Steel, Seamless (ASTM A-335-55T, Grade P-1).
MIL-T-20157 - Tube and Pipe, Carbon Steel, Seamless.
MIL-C-20159 - Copper-Nickel Alloy (70-30 and 90-10): Castings.
MIL-S-22698 - Steel Plate and Shapes, Weldable Ordinary Strength and Higher Strength: Hull Strength.
MIL-S-23192 - Springs, Helical, Age-Hardenable Nickel-Chromium-Iron Alloy.
MIL-T-23227 - Tube and Pipe, Nickel-Chromium-Iron Alloy.
MIL-N-23228 - Nickel-Chromium-Iron Alloy Plate, Sheet and Strip, Air Melted or Vacuum Remelted.
MIL-N-23229 - Nickel-Chromium-Iron Alloy Bars and Forgings.
MIL-T-24107 - Tube, Copper (Seamless) (Copper numbers 102, 103, 108, 120, 122, and 142).
MIL-N-24114 - Nickel-Chromium-Iron Age-Hardenable Alloy Bars, Rods, and Forgings.
MIL-I-24137 - Iron, Castings, Nodular Graphitic (Ductile Iron) and Nodular Graphitic (Corrosion Resisting, Austenitic, Low Magnetic Permeability) (For Shipboard Application).
MIL-C-24252 - Cobalt-Chromium-Tungsten-Nickel and Cobalt-Chromium-Tungsten Alloy Bars, Rods and Forgings.
MIL-B-24480 - Bronze, Nickel-Aluminum Castings, For Seawater Service.
MIL-N-24492 - Nickel-Chromium-Iron-Columbium Age-Hardenable Alloy Bars, Rods, Reforging Stock, and Forging (Alloy 718).
MIL-C-24527 - Chromium-Nickel Alloy, Steel Bars, Rods, and Forgings, Corrosion-Resistant (Precipitation Hardening).
MIL-N-24549 - Nickel-Copper-Aluminum Alloy, Age Hardenable, Bars and Rods.

STANDARD

FEDERAL

FED-STD-66 - Steel: Chemical Composition and Hardenability.

(Copies of specifications and standards required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this handbook to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

ASTM

- A 48 - Gray Iron Castings, Standard Specification for.
- A 217 - Martensitic Stainless Steel and Alloy Steel Castings for Pressure-Containing Parts Suitable for High-Temperature Service.
- B 160 - Nickel Rod and Bar, Specification for.
- B 443 - Nickel-Chromium-Molybdenum-Columbium Alloy (UNS N06625) Plate, Sheet, and Strip, Specification for.
- B 444 - Nickel-Chromium-Molybdenum-Columbium Alloys (UNS N06625) Pipe and Tube, Specification for.
- B 446 - Nickel-Chromium-Molybdenum-Columbium Alloy (UNS N06625) Rod and Bar, Specification for.
- B 637 - Precipitation-Hardening Nickel Alloy Bars, Forgings, and Forging Stock for High-Temperature Service, Specification for.

(Application for copies should be addressed to ASTM, 1916 Race Street, Philadelphia, PA 19103.)

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

3. GENERAL REQUIREMENTS

3.1 Intended use.

3.1.1 This procedure is intended for use when it is desired to identify metallic materials by category or subcategory, i.e., alloy identification. This metal alloy classification procedure can be used for identification for welding, sorting of scrap, locating weld beads or overlays, and insuring removal of weld overlays, plating, or lead shielding.

3.1.2 This procedure is not intended as a substitute for quantitative chemical analysis. It cannot be used to determine whether or not a metal meets specification chemical requirements. This procedure is only valid for metals which meet specification requirements since a metal which is off specification may behave in this procedure as a different metal.

3.1.3 The procedure of this handbook will only confirm that the unknown reacts the same as a standard of known chemistry.

3.2 Testing conditions.

3.2.1 Chemical spot tests should be performed on materials that are at temperatures below 60 degrees Celsius (°C) or above 7°C. Normal ambient temperature (25 ± 5°C) is best. Temperature variations cause variances in reaction time and intensity. Both the unknown and the test standard must be at the same temperature.

3.2.2 To assure accurate determination and comparisons of colors and reaction rates, the best possible lighting conditions shall be provided in each testing situation. Unfiltered daylight would be preferable. The case of a flashlight may provide the lowest acceptable light intensity. In any case, lighting conditions shall be the same for both the unknown and the test standard.

3.3 Safety precautions.

3.3.1 Users of this procedure shall comply with all local regulations regarding safety, storage, and disposal with respect to the reagents involved in this procedure.

3.3.2 Most of the reagents are acids or corrosive chemicals. Normal precautions (see 4.1(f)) to avoid contact with clothing, skin, and eyes should be observed. Plastic containers shall be used and should be tightly closed when not in use to avoid spills. Adequate ventilation should be provided.

3.3.3 At the completion of testing, the specimens should be rinsed with water followed by the use of moist, absorbent cloths or paper to wipe off reaction products and the work shall be dried to prevent further chemical attack.

3.4 Labeling. Reagents should be labeled and have a hazardous material sticker affixed. The label shall include, as a minimum, the following information:

(a) Reagent name and the identifying paragraph number.
(b) Preparation date.
(c) Expiration date.
(d) Special safety precautions.
(e) Signature of preparer.

4. EQUIPMENT

4.1 The equipment to be used is as follows:

(a) Reagents and distilled water as specified herein.
(b) Plastic dropping bottles 50 milliliters (mL) capacity for storing the reagents.
(c) Cleaning materials such as a fine file or abrasive paper.
(d) Filter paper, Whatman No. 3 paper or equivalent, cut into strips about 2.5 centimeters (cm) (1-inch) wide.
(e) A flashlight because lighting is often poor under testing conditions and shades of color in both alloy and reaction products are difficult to judge without an adequate light source.
(f) Rubber or plastic gloves or finger cots (to protect skin from contact with reagents) and other safety equipment as required by local regulations. Recommended items include plastic gloves, safety glasses, and eye wash bottle.
(g) Clean, uncoated steel nails.
(h) Small magnet.
(i) A suitable carrying case for equipment, reagents, and test standards.
(j) A stopwatch or equivalent timing device.

5. REAGENTS

5.1 Fifty percent nitric acid. Fifty percent nitric acid is one volume of concentrated nitric acid, with a specific gravity of 1.42, added to one volume of distilled water. The shelf life is 6 months.

5.2 Five percent chromotropic acid solution. Five percent chromotropic acid solution is 1 gram of chromotropic acid powder dissolved in 20 mL distilled water. The shelf life is 8 hours.

NOTE: In view of the very brief shelf life of chromotropic acid solution, the powder, which has a 3-year shelf life, shall be mixed in proper amounts with distilled water, only as needed for use within 8 hours. The container should be marked with the date of preparation to avoid accidental use of deteriorated reagent.

5.3 Silver nitrate. Silver nitrate is 1 gram of silver nitrate dissolved in 100 mL of distilled water. The reagent bottle should be wrapped with opaque tape to prevent sunlight from degrading the solution. The shelf life is 6 months.

5.4 Concentrated hydrochloric acid. The specific gravity of the concentrated hydrochloric acid is 1.18. The shelf life is indefinite.

5.5 Ten percent potassium ferricyanide. Ten percent potassium ferricyanide is 10 grams of potassium ferricyanide dissolved in 100 mL of distilled water. The shelf life is 6 months.

5.6 Forty percent sodium hydroxide. Forty percent sodium hydroxide is 40 grams of sodium hydroxide dissolved in 100 mL of distilled water. The shelf life is 6 months.

5.7 Twenty percent sulfuric acid. Twenty percent sulfuric acid is 20 mL of concentrated sulfuric acid slowly added to 80 mL of distilled water. The shelf life is 6 months.

5.8 Potassium ethyl xanthogenate (paper). Potassium ethyl xanthogenate (paper) is 10 grams of potassium ethyl xanthogenate dissolved in 100 mL of distilled water. Soak whole pieces of filter paper in the solution, remove, and allow to dry completely. After drying, cut into strips approximately 0.6 cm wide by 5 cm long (1/4-inch by 2 inches). Store in an air tight container. The shelf life of the paper is 6 months.

5.9 Ten percent nitric acid. Ten percent nitric acid is 10 mL of concentrated acid added to 90 mL of distilled water. The shelf life is 6 months.

5.10 Dimethylglyoxime (paper). Dimethylglyoxime paper is prepared by soaking filter paper in a solution prepared as follows: dissolve 0.4 grams of dimethylglyoxime powder in 40 mL of ethyl alcohol. Prepare a second solution by dissolving 5 grams of ammonium acetate in 15 mL of concentrated ammonium hydroxide. Combine the two solutions and mix well. Add this solution to 30 mL of glacial acetic acid. After soaking for 1 minute, remove the paper and allow to air dry thoroughly. The paper is then cut into strips measuring approximately 0.6 cm wide by 5 cm long (1/4-inch by 2-inches). Store in an air tight container. The shelf life of the paper is 6 months.

5.11 Fifty percent sulfuric acid. Fifty percent sulfuric acid is one volume of concentrated sulfuric acid slowly added to one volume of distilled water. The shelf life is 6 months.

5.12 Five percent sodium hydroxide. Five percent sodium hydroxide is 5 grams of sodium hydroxide dissolved in 100 mL of distilled water. The shelf life is 6 months.

5.13 Five percent sodium sulfide. Five percent sodium sulfide is 5 grams of sodium sulfide dissolved in 100 mL of distilled water. The shelf life is 6 months.

5.14 Fifty percent hydrochloric acid. Fifty percent hydrochloric acid is 50 mL of concentrated hydrochloric acid slowly added to 50 mL of distilled water. The shelf life is 6 months.

5.15 Stannous chloride. Stannous chloride is 35 grams of stannous chloride dissolved in 20 mL of 50 percent hydrochloric acid and diluted to 100 mL with distilled water.

5.16 Ammonium thiocyanate. Ammonium thiocyanate is 100 grams of ammonium thiocyanate dissolved in 100 mL of distilled water.

6. TEST STANDARDS

6.1 A set of standard test specimens of known chemical composition is required. These standards shall be tested concurrently with the materials being tested for comparison of color, chemical tests, and magnetic tests. Specimens shall have an area of 6.25 cm^2 (1 square inch) or more. The following list of specifications provides examples of standards which can be used for this procedure.

6.2 Materials having a "white or silvery, steel-like metallic luster".

6.2.1 Carbon or alloy steel. A specimen from any one of the many available as AISI or SAE grades of carbon steel or low alloy steel. Compositions are as specified in MIL-S-16974 and FED-STD-66.

6.2.2 Cast iron. Any of the gray cast irons shall be in accordance with ASTM A 48. Ductile iron shall be in accordance with MIL-I-24137, class A.

6.2.3 300 series corrosion resistant steel (CRES). 300 series CRES is any one of the 300 series classes listed in QQ-S-763.

6.2.4 17-4 PH CRES. 17-4 PH CRES is precipitation hardening 17-4 PH stainless steel, type I or II of MIL-C-24527.

6.2.5 400 series CRES. 400 series CRES is any one of the 400 series classes listed in QQ-S-763.

6.2.6 Nickel-copper. Nickel-copper is class A (UNS N04400) of QQ-N-281.

6.2.7 Nickel-copper-aluminum. Nickel-copper-aluminum is UNS N05500 of QQ-N-286 or MIL-N-24549.

6.2.8 Copper-nickel. Copper-nickel is 70/30 Cu-Ni of MIL-T-16420.

6.2.9 Nickel-chromium-iron.

6.2.9.1 UNS N06600. UNS N06600 is as follows:

(a) Bars, rods and forgings shall be in accordance with MIL-N-23229.
(b) Plate, sheet and strip shall be in accordance with MIL-N-23228.
(c) Pipe and tube shall be in accordance with MIL-T-23227.
(d) Wire shall be in accordance with QQ-W-390.

6.2.9.2 UNS N06625. UNS N06625 is as follows:

(a) Rod and forging stock shall be in accordance with ASTM B 446.
(b) Plate, sheet and strip shall be in accordance with ASTM B 443.
(c) Pipe and tube shall be in accordance with ASTM B 444.

6.2.9.3 UNS N07718. UNS N07718 shall be in accordance with ASTM B 637 or MIL-N-24492.

6.2.9.4 UNS N07750. UNS N07750 is as follows:

(a) Bars, rods, and forgings shall be in accordance with MIL-N-8550, MIL-S-23192 or MIL-N-24114.
(b) Plate, sheet and strip shall be in accordance with MIL-N-7786.

6.2.10 Cobalt-chromium-tungsten alloy shall be in accordance with MIL-R-17131 or MIL-C-24252.

6.2.11 Cobalt-chromium-tungsten-nickel alloy shall be in accordance with MIL-C-24252.

6.2.12 Nickel. Nickel 200 shall be in accordance with ASTM B 160.

6.2.13 Titanium. Titanium shall be pure titanium or any alloy of at least 70 percent titanium.

6.2.14 Aluminum. Aluminum shall be pure aluminum or any alloy of at least 70 percent aluminum.

6.2.15 Magnesium. Magnesium shall be pure magnesium or any alloy of at least 70 percent magnesium.

6.2.16 Zinc. Zinc shall be pure zinc or any alloy of at least 70 percent zinc.

6.3 Materials having "reddish, red-brown, dark yellow, or light yellow color".

6.3.1 Pure copper. Copper tubing shall be in accordance with MIL-T-24107.

6.3.2 Copper-nickel. Copper-nickel is as follows:

(a) Type II, 90/10 Cu-Ni shall be in accordance with MIL-C-20159.
(b) 90/10 Cu-Ni tubing shall be in accordance with MIL-T-16420.

6.3.3 Aluminum bronze. Aluminum bronze is as follows:

(a) Aluminum bronze, alloy 952 shall be in accordance with QQ-C-390.
(b) Nickel-aluminum bronze shall be in accordance with MIL-B-24480.

6.3.4 Brasses. Brasses are as follows:

(a) Leaded brass, composition 360 shall be in accordance with QQ-B-626.
(b) Naval brass, alloy 464 shall be in accordance with QQ-B-639.
(c) High-strength yellow brass (manganese bronze), alloys 864 and 865 shall be in accordance with QQ-C-390.

6.3.5 Other bronzes. Other bronzes are as follows:

(a) Silicon bronze, alloy 655 shall be in accordance with QQ-C-591.
(b) Valve bronze ("M"), alloy 922 shall be in accordance with QQ-C-390.
(c) Gun bronze ("G"), alloy 903 shall be in accordance with QQ-C-390.
(d) Phosphor bronze, composition A shall be in accordance with QQ-B-750.
(e) Manganese bronze, alloy 863 shall be in accordance with QQ-C-390.
(f) Manganese bronze, alloy 861 shall be in accordance with QQ-C-390.

7. PROCEDURE

7.1 General.

7.1.1 This handbook provides a procedure for metal alloy identification or classification of an unknown metallic material into the categories or subcategories of section 6. The further separation of metals and alloys within the groups is as specific as possible within practical limits, i.e., a reasonable number of chemicals and no complex apparatus.

7.1.2 The following are examples of use of this procedure:

7.1.2.1 If the sample is completely unknown, such as scrap in the yard, foundry, or forge shop, then the entire procedure shall be used to tentatively identify the unknown. The procedure should then be repeated with the unknown and a known standard for comparison.

7.1.2.2 If the unknown is expected or suspected to be some specific material, then a known standard of the desired material shall be acquisitioned and run through the procedure with the unknown for positive identification.

7.1.2.3 In the typical example of submarine air flask heads, which can be either UNS N04400 or stainless steel, all that is required is to compare the reaction of the unknown to 50 percent nitric acid with that of known standards of nickel-copper alloy and a stainless steel.

7.1.2.4 In many other cases, the form, shape, or usage of the unknown will eliminate many possible metals and thus narrow the search to two or three possibilities. The unknown can then be compared with standards of these materials.

7.1.3 The procedure specified herein has been developed with the most difficult field (or shipboard) condition in mind. The technique of applying reagents and observing reactions in filter paper is presented wherever feasible so that testing can be done even on vertical surfaces or overhead piping systems. In less rigorous situations in which a flat, horizontal surface is available for testing, the same reagents and reactions prevail but can be observed in droplets on the metal surface rather than in the filter paper, always in comparison with known standards.

7.1.4 This handbook is not intended for use on weld metals. Such use can provide erroneous results, for example, Ni-Cu weld metal will provide the same color reaction as nickel-copper-aluminum alloy provides.

7.1.5 Although the procedures set forth in this handbook are commonly referred to as spot tests, they should not be confused with the Acid Spot Absorption Test (ASAT), a more quantitative test which utilizes spectrographic analysis and is not covered by this handbook.

7.1.6 To facilitate visualizing the spot test procedures package presented herein, flow charts (figures 1 through 4) are necessarily abbreviated and shall be used only with the aid of the step-by-step procedures as specified in 7.2.

7.2 Step-by-step procedures.

7.2.1 Prior to testing, clean a spot of at least 6.25 cm^2 on the metal surface with a degreasing solvent, abrasive wheel, abrasive cloth, or file to remove dirt, grease, corrosion products, oxide or temper films, or metallic coatings. When testing for coating identity, do not use abrasives or otherwise remove coating prior to testing.

7.2.2 Examine the clean surface and classify on the basis of color into two groups, the first group having "white or silvery, steel-like metallic luster" and the second group having "reddish, red-brown, dark yellow, or light yellow color".

7.2.3 Figures 1, 2, and 3 comprise a schematic flow chart for identifying the unknown materials that are color identified as having "white or silvery, steel-like metallic luster". The following procedures shall be performed sequentially on this group of metals as specified by the flow charts.

7.2.3.1 Separate aluminum, magnesium, or zinc alloys by the following screening tests.

7.2.3.1.1 These metals are always nonmagnetic and extremely soft when abraded or filed; and the normal air-oxidized surface is dull gray or white. Aluminum and magnesium are low density and, therefore light, while zinc is as heavy as common steels.

7.2.3.1.2 Aluminum, exclusive of all other white or silvery metals, undergoes a vigorous white reaction when a drop of 40 percent sodium hydroxide reagent (see 5.6) is applied to a clean spot.

7.2.3.1.3 Magnesium and zinc can be screened from other white or silvery metals by placing a drop of silver nitrate reagent (see 5.3) on a clean spot. The drop will turn black and leave a black spot on the metal when reactant product is absorbed in filter paper. The two metals can be separated by applying a drop of 50 percent sulfuric acid reagent (see 5.11) to a clean spot. The spot on magnesium becomes chalky white in less than 15 seconds and the spot on zinc slowly turns gray in about 1 minute.

7.2.3.2 After eliminating the possibility of the white metals as above, apply two drops of 50 percent nitric acid reagent (see 5.1) to a strip of filter paper, press on a clean spot for at least 2 minutes, and remove.

7.2.3.2.1 No visible color on the filter paper indicates 300/400 series CRES, 17-4 PH CRES, nickel-chromium-iron alloy, chromium-tungsten-cobalt alloy, cobalt-chromium-tungsten-nickel alloy, or titanium.

7.2.3.2.2 A vigorous reaction which turns both metal and filter paper brown or black indicates plain carbon steel, low alloy steel, or cast iron.

7.2.3.2.3 An immediate reaction and a bluish-green (turquoise) coloration on the filter paper indicates copper-nickel 70/30.

7.2.3.2.4 A slower reaction and a green coloration on the filter paper indicates nickel-copper alloy, nickel-copper-aluminum alloy, or pure nickel.

7.2.3.3 If the green color of 7.2.3.2.4 indicates nickel-copper alloy, nickel-copper-aluminum alloy, or pure nickel, add one drop of 5 percent chromotropic acid solution reagent (see 5.2) to the green spot on the filter paper. An immediate red to scarlet coloration indicates nickel-copper-aluminum alloy. The red color is most visible when the filter paper is equal to or greater in absorbency than Whatman No. 3--double thickness paper.

7.2.3.4 Nickel-copper alloy and nickel can be separated by applying two drops of 50 percent nitric acid reagent (see 5.1) and one drop of concentrated hydrochloric acid reagent (see 5.4) on a clean spot and reacting for 1 minute. If, after rubbing a clean steel nail in the reactants for 1 minute, copper is plated on the nail, nickel-copper alloy is present. Absence of copper on the nail indicates pure nickel.

7.2.3.5 The carbon steels, alloy steels, and cast irons identified by the vigorous brown or black reaction of 7.2.3.2.2 encompass so many specifications and forms (bars, castings, tubing, and forgings) with overlapping chemical compositions that positive identification of an unknown is not possible. However, spot tests for nickel, molybdenum and cast iron are relatively simple and provide a limited basis for further classification. If it is necessary to identify cast iron, or to otherwise separate it from the family of steels, add one drop of silver nitrate reagent (see 5.3) to the metal surface. The drop will remain clear on steels and turn black or gray on cast iron.

7.2.3.5.1 Test for molybdenum in steels by dropping one drop of 50 percent nitric acid solution reagent (see 5.1) and 50 percent hydrochloric acid reagent (see 5.14) on a clean spot. Absorb the reactant product on filter paper and add four drops of stannous chloride reagent (see 5.15). A drop or two of ammonium thiocyanate reagent (see 5.16) on the filter paper will develop a pink or red color in the presence of approximately 0.2 percent, or greater molybdenum. The intensity of color development is proportional to the molybdenum content.

7.2.3.5.2 Test for nickel in steels by dropping one drop of 10 percent nitric acid reagent (see 5.9) on a clean spot, reacting for 1 minute, and inserting a strip of dimethylglyoxime (paper) reagent (see 5.10) into the drop. Pink to red color on the paper indicates nickel. The lower limit of detectability is 0.05 percent nickel and the pink color is intensified to red at higher nickel contents.

7.2.3.5.3 The above tests are not sufficiently sensitive to identify alloys which have nickel and molybdenum contents less than 0.15 percent, but are useful for rejection if receipt inspection or shipboard verification require steels with nickel or molybdenum and none is found. The limits of nickel and molybdenum content in typical shipboard steels are specified in table I for guidance. Two examples of proven utility are specified in 7.2.3.5.3.1 and 7.2.3.5.3.2.

TABLE I. Significant alloy content of common steels.

Alloy and specification	Alloy content percent; min/max	
	Ni	Mo
Carbon steels		
(1) AISI 1030 of MIL-S-16974	---	---
(2) Grade D plate of MIL-S-22698	---	---
(3) Tube and pipe of MIL-T-20157	---	1.25 max
Alloy steels		
C-Mo steels		
(1) Tube and pipe of MIL-T-20155	---	.44/.65
(2) Bar or forgings of MIL-S-872	.25 max	.40/.60
(3) Castings of MIL-S-870	.25 max	.40/.60
(4) Grade 4 nuts of MIL-S-1222	---	.20 min
(5) ASTM A 217, Gr. WC1 castings	---	.45/.65

TABLE I. Significant alloy content of common steels. - Continued

Alloy and specification	Alloy content percent; min/max	
	Ni	Mo
Cr-Mo steels		
(1) 1-1/2 Cr-1/2 Mo of MIL-S-18410	---	.45/.65
(2) 2-1/2 Cr-1 Mo of MIL-S-18410	---	.90/1.10
(3) AISI 4140 of MIL-S-16974	---	.15/.25
(4) B-14 bar and studs of MIL-S-1222	---	.28/.42
(5) B-16 bar and studs of MIL-S-1222	---	.42/.68
(6) ASTM A 217, Gr. WC6 castings	---	.45/.65
(7) ASTM A 217, Gr. WC9 castings	---	.90/1.20
Ni-Cr-Mo steels		
(1) AISI 4340 of MIL-S-16974	1.65/2.00	.20/.30
(2) AISI 8630 of MIL-S-16974	.40/.70	.15/.25
(3) Grade HY-80 plate of MIL-S-16216	2.00/3.25	.20/.60
(4) Grade HY-100 plate of MIL-S-16216	2.25/3.50	.20/.60

7.2.3.5.3.1 In steel pipe or tube materials, absence of molybdenum indicates carbon steel. Carbon-molybdenum steel and the chromium-molybdenum steels of MIL-S-18410 are distinguishable by comparison of the intensity of red color with known standards.

7.2.3.5.3.2 Submarine plate steels of HY-80 or HY-100 shall have positive Mo and Ni spot tests. Absence of Ni and Mo may indicate grade D of MIL-S-22698.

7.2.3.6 If the lack of reaction to 50 percent nitric acid reagent (see 5.1) indicates 300 or 400 series CRES, 17-4 PH CRES, nickel-chromium-iron alloy, chromium-tungsten-cobalt alloy, cobalt-chromium-tungsten-nickel alloy, or titanium alloys, check the magnetism of the sample. If the sample is fully ferromagnetic, in comparison with a standard, it is either 400 series CRES or 17-4 PH CRES. Samples with less than the full ferromagnetism of a 400 series CRES standard will be 300 series CRES, nickel-chromium-iron alloy, chromium-tungsten-cobalt alloy, cobalt-chromium-tungsten-nickel alloy, or titanium alloys.

7.2.3.6.1 Apply two drops of concentrated hydrochloric acid reagent (see 5.4) to a strip of filter paper, apply to a clean spot, react for 30 seconds, and remove. Then apply one drop of 10 percent potassium ferricyanide reagent (see 5.5) onto the reactants on the filter paper. A yellow ring with a green or blue-green center indicates 300 series CRES and a plain yellow ring indicates nickel-chromium-iron, chromium-tungsten-cobalt, cobalt-chromium-tungsten-nickel alloy, or titanium. The yellow spot shall then be placed back onto the same spot on the metal, held for 2 minutes, and then removed. If the residue or spot on the metal is bluish-green all over, nickel-chromium-iron is indicated. If the residual spot on the metal contains a purplish-black center with a bluish-green fringe, chromium-tungsten-cobalt alloy is indicated. If the residual spot is gray or black in the center with a bluish-green fringe, cobalt-chromium-tungsten-nickel alloy is indicated. If no spot remains on the metal, a titanium alloy is indicated.

7.2.3.6.2 If it is desired to separate the various nickel-chromium-iron alloys specified in 6.2.9, place two drops of 50 percent nitric acid reagent (see 5.1) and one drop of concentrated hydrochloric acid reagent (see 5.4) on a clean spot, react for 3 or 4 minutes, and compare with the distinctive shades of green on known standards.

7.2.3.6.3 After the identification of an unknown as 400 series or 17-4 PH, 17-4 PH can be separated by applying two drops of 50 percent nitric acid reagents (see 5.1) and one drop of concentrated hydrochloric acid reagent (see 5.4) on a clean spot and reacting for 1 minute. If, after rubbing a clean steel nail in these reactants for 1 minute, copper is plated on the nail, 17-4 PH CRES is indicated.

7.2.3.7 A magnet is a very useful tool for confirming the chemical identification scheme.

7.2.3.7.1 Carbon steels, low-alloy steels, nickel, 17-4 PH CRES, and 400 series CRES are always strongly magnetic.

7.2.3.7.2 70/30 copper-nickel, nickel-copper-aluminum, nickel-chromium-iron, chromium-tungsten-cobalt, cobalt-chromium-tungsten-nickel, and titanium alloys are always nonmagnetic.

7.2.3.7.3 The other metals in this group exhibit magnetic variances as follows:

7.2.3.7.3.1 Molybdenum-bearing 300 series CRES such as 316, 316L, 317, and 319 are usually nonmagnetic, but can sometimes be strongly magnetic in either the annealed or cold-worked condition.

7.2.3.7.3.2 The other 300 series CRES classes are nonmagnetic in the annealed condition, but can range to strongly magnetic, if not annealed after cold working.

7.2.3.7.3.3 Nickel-copper alloy is usually nonmagnetic, but can range to strongly magnetic.

7.2.3.7.4 In figures 1 through 4, these magnetic characteristics are specified as follows:

SM - always strongly magnetic
NM - always nonmagnetic
nm - usually nonmagnetic, but subject to variances as specified above

7.2.3.8 If, for any reason, the chemical reaction and magnetic response do not agree, or interpretations of either chemical reactions or magnetism are doubtful, submit the unknown to quantitative chemical analysis.

7.2.4 Figures 1 and 4 consist of a schematic flow chart for identifying the unknown materials that have "reddish, red-brown, dark yellow, or light yellow" color. The following procedures are to be performed sequentially by following the flow charts.

7.2.4.1 By comparison with a known standard of pure copper, visually separate the reddish, red-brown pure copper from the dark yellow or light yellow copper alloys.

7.2.4.2 For classifying the balance of these materials, apply one drop of silver nitrate reagent (see 5.3) to a clean spot. No reaction or the slow development of a gray coloration indicates aluminum bronze or nickel-aluminum bronze. A rapid black reaction in 2 seconds or less indicates brass. A black or gray reaction in 3 to 15 seconds indicates the copper-nickel alloys specified in 6.3.2 or the other bronzes specified in 6.3.5.

7.2.4.2.1 Nickel-aluminum bronze can be separated from aluminum bronze. Apply one drop of 50 percent silver nitrate reagent (see 5.1) to a clean spot for 30 seconds, either as a surface drop or in filter paper. A reaction of turquoise color indicates aluminum bronze. A gray-green or dirty green reaction indicates nickel-aluminum bronze. The identification of nickel-aluminum bronze can be confirmed by conducting the nickel test specified in 7.2.3.5.2.

7.2.4.2.2 Further separation within the identified groups in 7.2.4.2 can be made using the magnetic responses specified in figure 4 and as follows.

7.2.4.3 Apply magnet to confirm the classification of metals. The magnetic characteristics of this group of metals are as specified in 7.2.4.3.1 through 7.2.4.3.4.

7.2.4.3.1 Pure copper (see 6.3.1), leaded and naval brasses (see 6.3.4(a) and (b)) are always nonmagnetic.

7.2.4.3.2 90/10 copper-nickel tubing is nonmagnetic but the cast 90/10 copper-nickel (see 6.3.2(a)) can be faintly magnetic as a result of iron content of about 1.50 percent.

7.2.4.3.3 The aluminum bronze and nickel-aluminum bronze (see 6.3.3) and the manganese bronzes (see 6.3.4(c), 6.3.5(f), and 6.3.5(g)) exhibit faint to moderate magnetism due to iron content of about 2.0 percent minimum.

7.2.4.3.4 The remaining alloys specified in 6.3.5 are always nonmagnetic.

NOTE: Some of the copper-base alloys such as aluminum bronze, are subject to a de-alloying type of corrosion in seawater service. Copper-base alloys that do not normally react to the application of silver nitrate reagent (see 5.3) may react strongly if they have suffered de-alloying corrosion in service. When unknown materials are suspected of this condition, spot testing shall be used with caution. The de-alloyed condition is not readily determined visually, but can be detected by the eddy-current type of nondestructive testing.

7.2.5 After tentatively identifying the unknown by the preceding procedures, the unknown shall be cleaned again and the observations of color, chemical reactions, and magnetism shall be repeated and compared to a known standard of the tentatively identified material.

7.3 Miscellaneous auxiliary procedures.

7.3.1 Spot testing for identification of silver brazing alloys is infeasible. Where possible, suitable samples to permit analytical chemistry tests shall be secured. If etching is done to detect residual sil-braze metal in joints, this shall be accomplished by swabbing the joint with 50 percent siver nitrate reagent (see 5.1). Instant black coloration will occur only on the sil-braze deposit. No standard is required.

7.3.2 In the repair of lead-shielded stainless steel piping systems, it is essential that the lead be completely removed prior to welding of the stainless steel. To check for complete lead removal, wrap filter paper wetted by 5 percent sodium hydroxide reagent (see 5.12) around the weld prep area and hold there for 1 minute. Then remove the paper and place drops of 5 percent sodium sulfide reagent (see 5.13) on the filter paper. Lead is present if the paper turns brown. A standard for this test shall be manufactured locally by rotating a CRES bar in a lathe or drill press and smearing lead onto its surface.

7.3.3 To separate cadmium, tin, or zinc coatings on items such as fasteners, apply one drop of 50 percent hydrochloric acid reagent (see 5.14). If a rapid reaction takes place, the coating is zinc. If no obvious reaction occurs, hold a clean uncoated nail in contact with the surface beneath the drop of acid. If rapid gas evolution occurs at the interface of the nail and acid, the metal is cadmium. If no obvious gas evolution of this nature occurs, the metal is tin. Fasteners coated with cadmium, tin, and zinc shall be acquisitioned or made up locally for use as standards for this test. Caution: Positive cadmium identification evolves toxic gases. Minimize testing time and ensure adequate ventilation when cadmium coatings are suspected.

Preparing activity:
Navy - SH
(Project 95GP-N007)

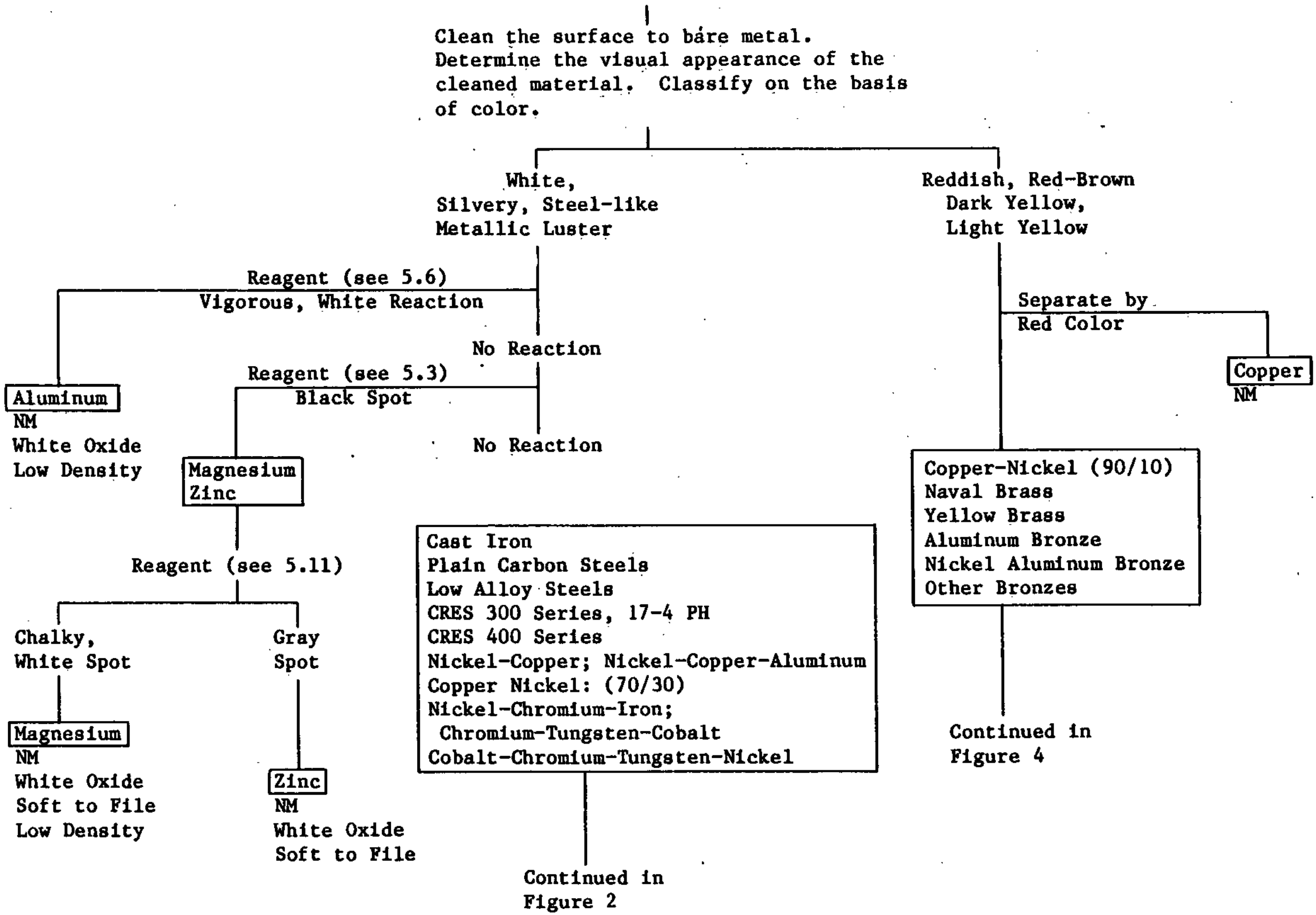

FIGURE 1. Identification scheme outline.

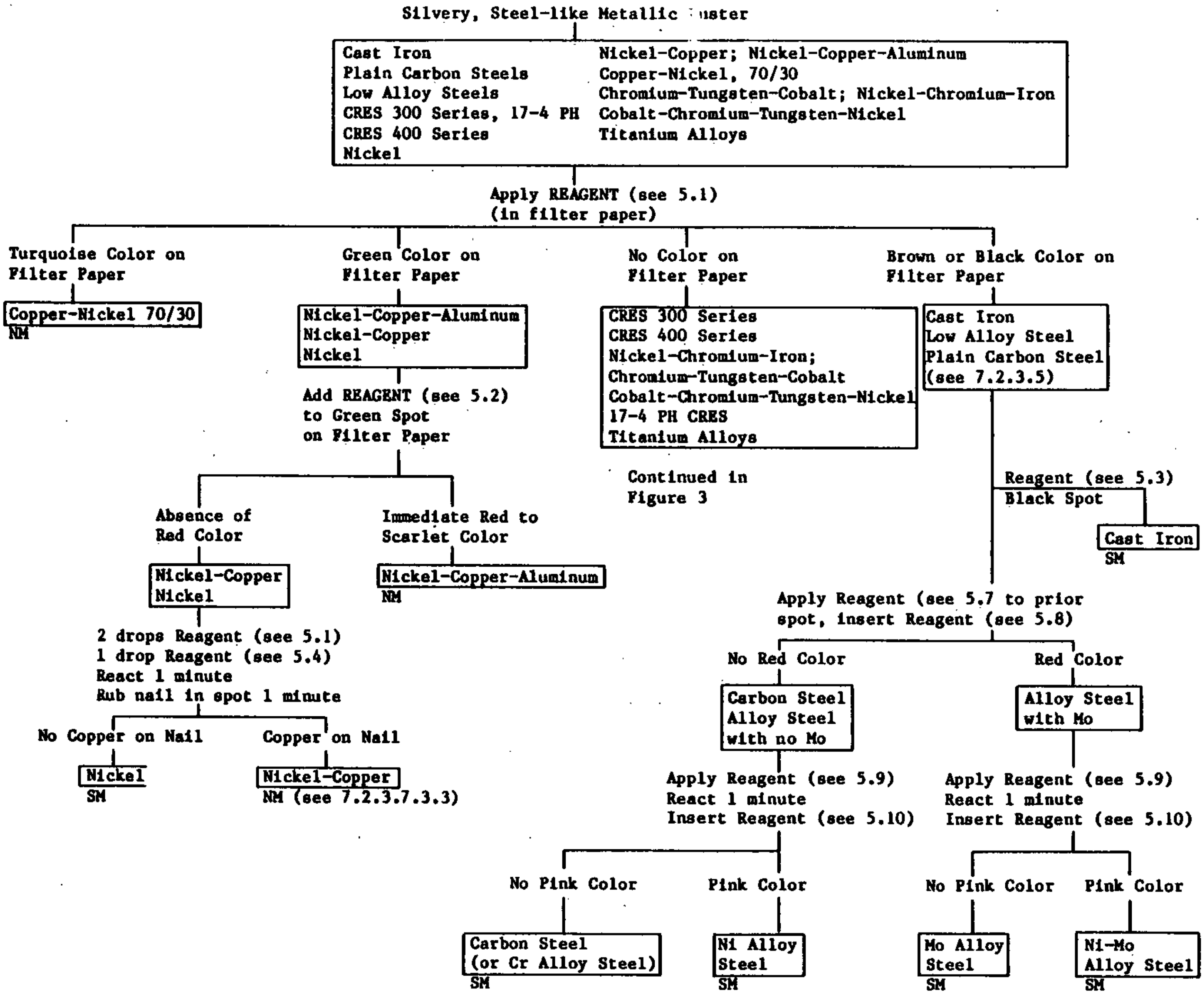

FIGURE 2. Identification scheme outline (continued from figure 1).

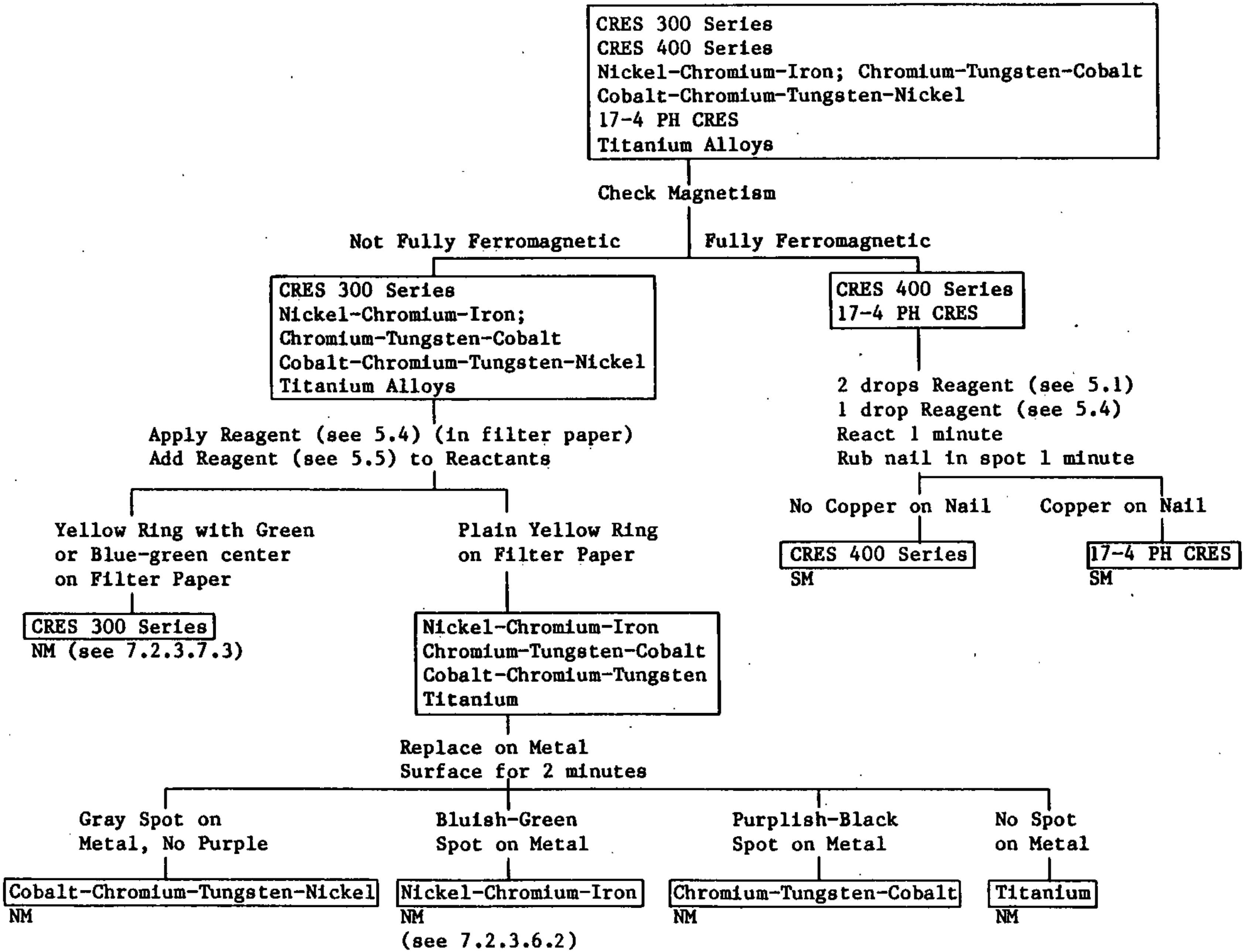

FIGURE 3. Identification scheme outline (continued from figure 2).

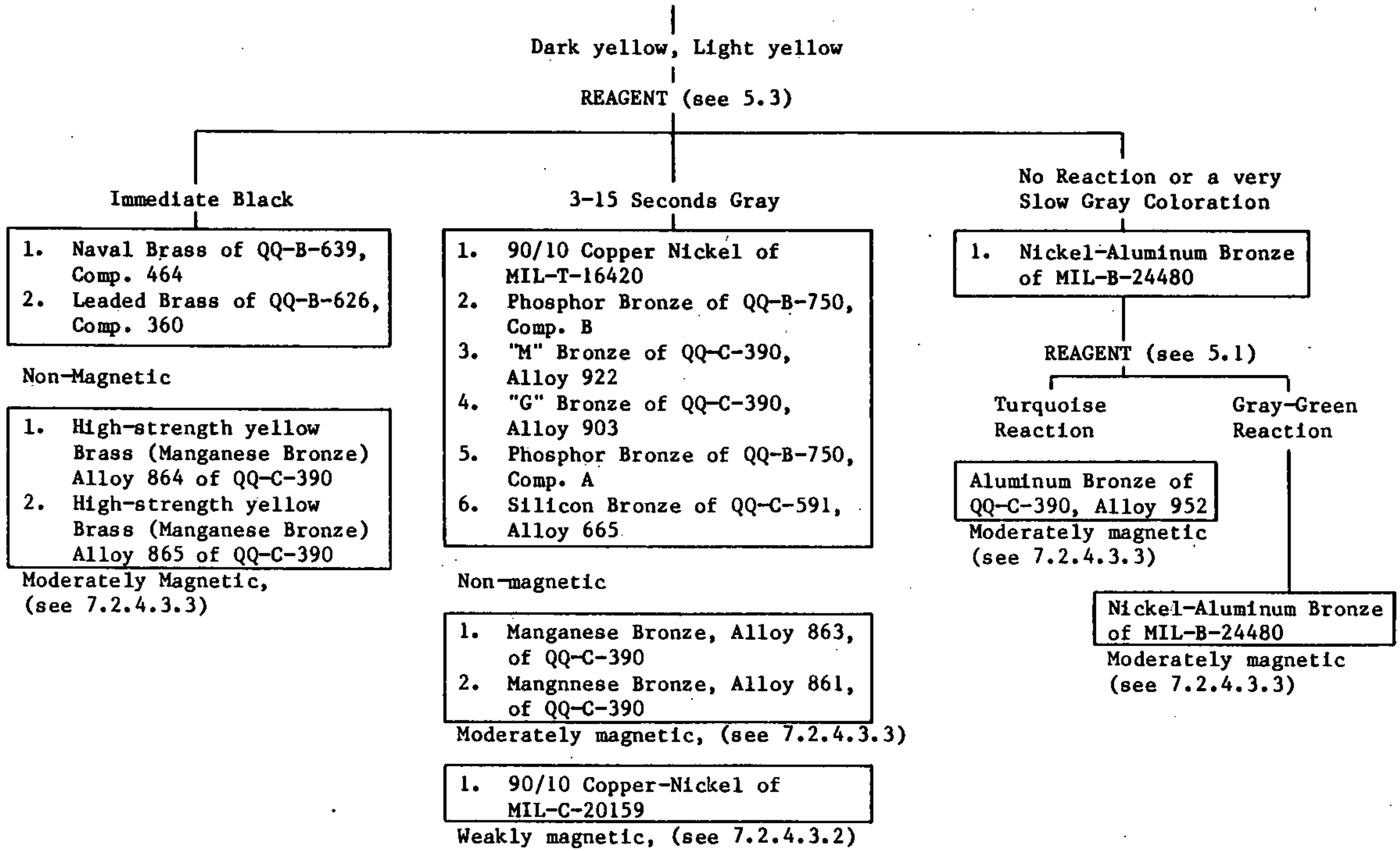

FIGURE 4. Identification scheme outline (continued from figure 1).

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions – Reverse Side)

1. DOCUMENT NUMBER	2. DOCUMENT TITLE
DOD-HDBK-249A(SH)	

3a. NAME OF SUBMITTING ORGANIZATION	4. TYPE OF ORGANIZATION *(Mark one)*
	☐ VENDOR ☐ USER ☐ MANUFACTURER ☐ OTHER *(Specify)*: ______
b. ADDRESS *(Street, City, State, ZIP Code)*	

5. PROBLEM AREAS

a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

6. REMARKS

7a. NAME OF SUBMITTER *(Last, First, MI)* – Optional	b. WORK TELEPHONE NUMBER *(Include Area Code)* – Optional
c. MAILING ADDRESS *(Street, City, State, ZIP Code)* – Optional	8. DATE OF SUBMISSION *(YYMMDD)*

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