

***NONDESTRUCTIVE
RAPID IDENTIFICATION
OF METALS AND ALLOYS
BY SPOT TEST***

STP 550

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AMERICAN SOCIETY FOR TESTING AND MATERIALS

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AND ALLOYS BY SPOT TEST

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**The Society is not responsible, as a body,
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Introduction

Quality assurance requirements at the Langley Research Center have given the impetus for the investigation and development of a reliable system of rapid identification of metals and alloys. Early in the investigation it became evident that the published existing systems were inadequate to meet the requirements at the Langley Research Center.

To assure quality fabrication of hardware and maximum safety to personnel, it is necessary to identify or verify the various metals and alloys involved. For example, if two or more metals are to be intelligently joined together by modern welding techniques, their identities are essential. Facilities for the identification of metals and alloys frequently require extensive time consuming procedures of chemical or spectrographic analyses. Facilities or techniques for nondestructive identification of the finished products likewise may be expensive or not developed.

In an effort to establish a nondestructive, relatively simple, time saving system of identification of metals and alloys that could be utilized in shops or laboratories, experimentation and applied research was conducted in the Fabrication Section, Langley Research Center in 1968-69 with the metals and alloys then in use at the Center. Chemical spot test techniques were developed which affected only the amount of material equivalent to the stroke of a smooth file or cleaning with an abrasive cloth. Many of the tests are conducted on the surface of the metals. Some tests are performed in porcelain spot plate depressions while others are accomplished on qualitative filter paper. Conclusions relative to identification are derived from unique reactions or the colors produced by the addition of reagents. Known specimens of materials, when available, should be tested simultaneously for comparison purposes and to build up the operator's confidence in the test procedures and results.

Families of metals and alloys considered in this system are aluminum and aluminum alloys, copper and copper alloys, magnesium and magnesium alloys, nickel and nickel alloys, stainless and heat resisting steels, carbon and low alloy steels, tool steels, titanium and titanium alloys, and some pure metals.

The tests are qualitative; however, an analyst can intelligently reach semiquantative conclusions for most of the tests which produce color reactions by comparison with simultaneous tests on known metals or alloys.

*M. L. Wilson*¹

Nondestructive Rapid Identification of Metals and Alloys by Spot Test

Test Techniques and Precautions

The analyst should familiarize himself with the metals and alloys he expects to encounter including normal processing methods involved in fabricating the finished products. He should become familiar with the flow diagrams and procedures for determining constituent alloys in the various metal families listed in this publication. Normal cleaning processes must be observed on metals at test areas to remove dirt, grease, oxides, or metallic coatings such as nickel, zinc, tin, aluminum, etc., prior to performing a test. Cleaning solvents, files, or abrasives can be used as applicable. After properly cleaning the test area, the analyst should follow detailed instructions for each test relative to chemicals used, number of drops, and time allowed for each reaction. Procedures for determining constituent alloys in various metal families can be used independently of the flow diagrams; however, some tests on the flow diagrams refer the analyst to tests listed in the metal family procedures. Qualitative analysis can be performed on unknown metals and alloys by following the procedures for determining alloys in a given alloy family.

Many tests will involve verifying a particular metal of which the history of processing is known. This will enable an analyst to establish a definite starting point in performing tests. He will already have some basis for suspecting the metal to be in a particular family, finding it necessary to use only a segment of the systematic procedure, and a conclusion can be derived in a few seconds. Some tests will involve longer periods of time; however, tests should not involve longer periods than 30 to 40 minutes to identify the metal. This system of identification of metals and alloys will enable an analyst to identify the metals listed in Appendix C and metals and alloys having chemical compositions of similitude.

No tests have been made to determine the effect of the chemicals on the physical properties of the materials. Therefore, chemical spot tests should be made on sample materials when possible. Should it be necessary to identify material in finished hardware, the chemical spot test should be done in a non-critical low stress area.

Chemical spot tests should not be used on materials or hardware that will

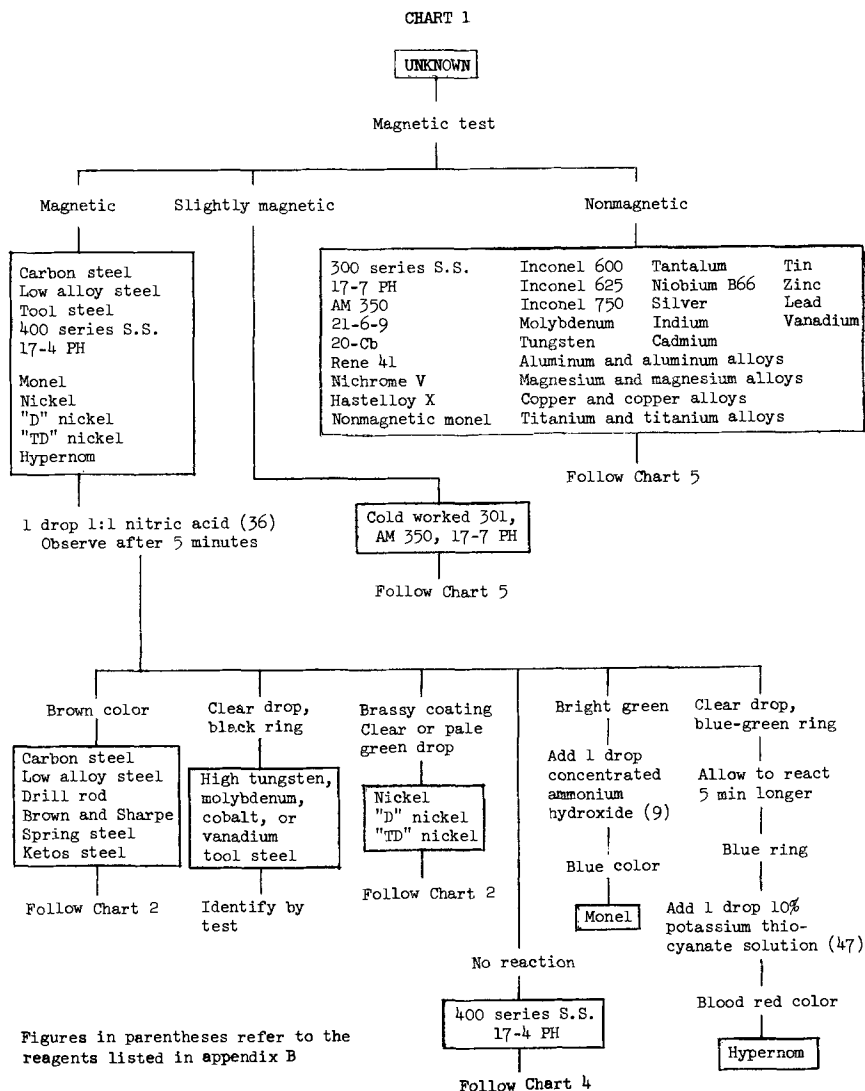
¹ Langley Research Center, Langley Station, Hampton, Va.

be subjected to elevated temperature without first determining the effect of the chemicals on the materials or hardware relative to the physical properties or fatigue life.

The apparatus necessary to employ this system of identification is listed in Appendix A. A list of the chemicals used and directions for the preparation of reagents are given in Appendix B.² The chemical compositions of all metals and alloys included in this publication are listed in Appendix C. Some precautions and notes in handling chemicals are listed in Appendix D.

²Numbers in parentheses in Charts 1 through 22 and in test procedures refer to the list of chemicals in Appendix B.

Charts, Identification of Metals and Alloys



6 NONDESTRUCTIVE RAPID IDENTIFICATION OF METALS

CHART 2

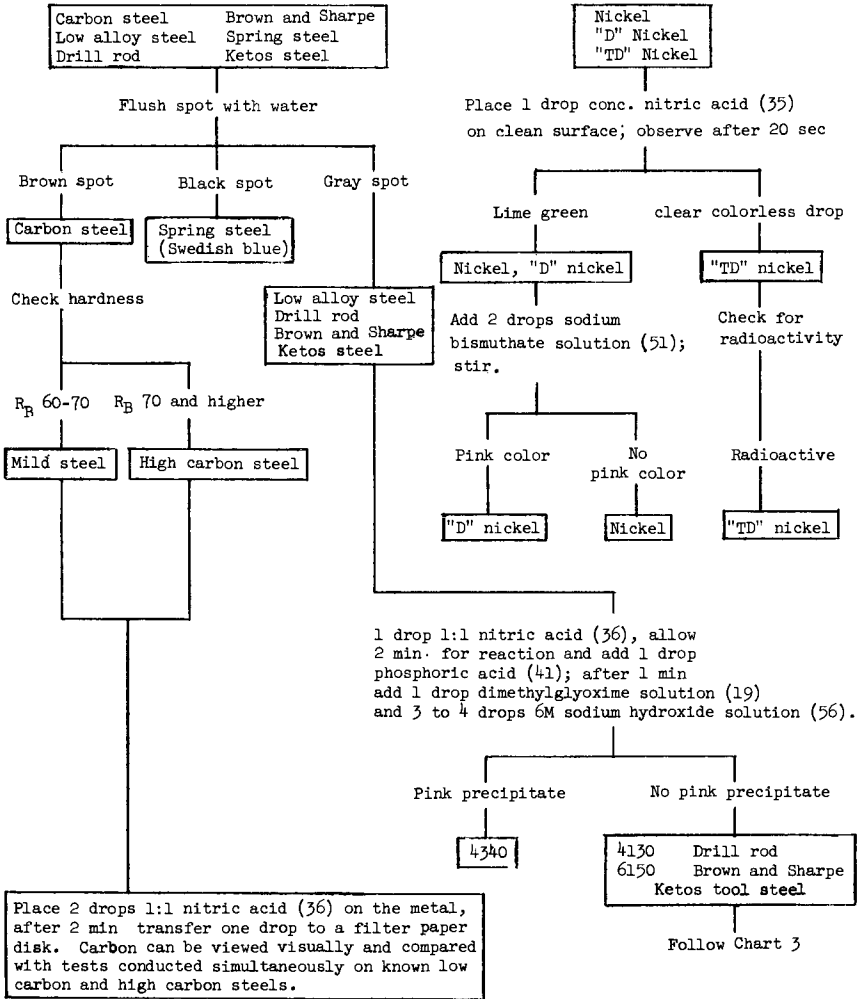
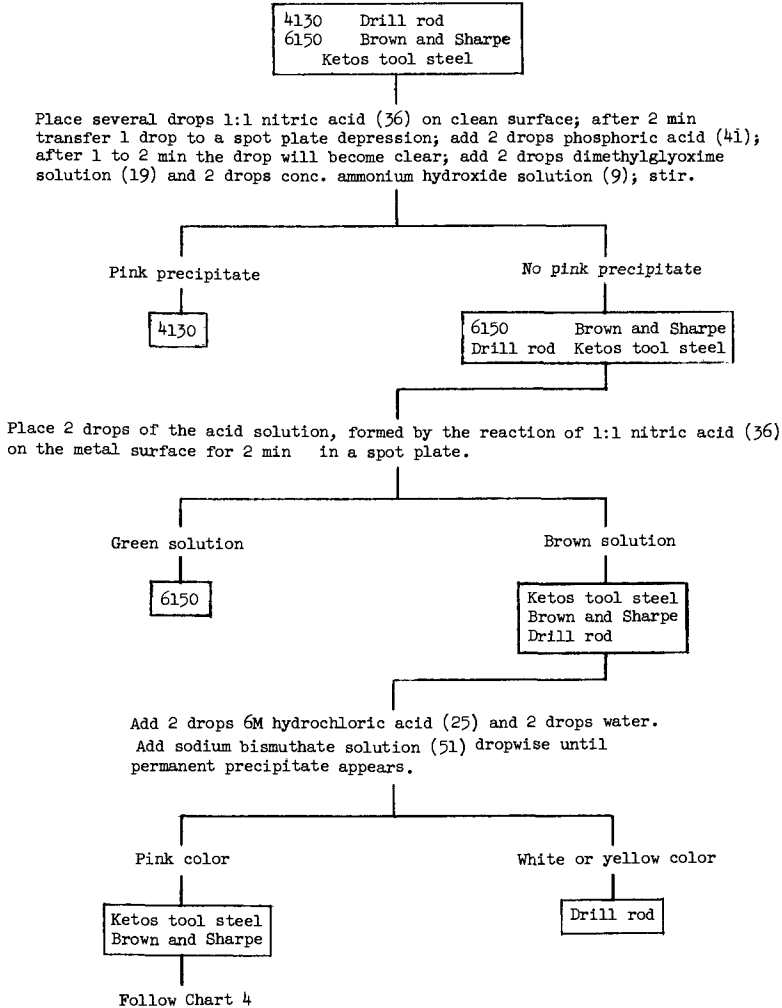


CHART 3



8 NONDESTRUCTIVE RAPID IDENTIFICATION OF METALS

CHART 4

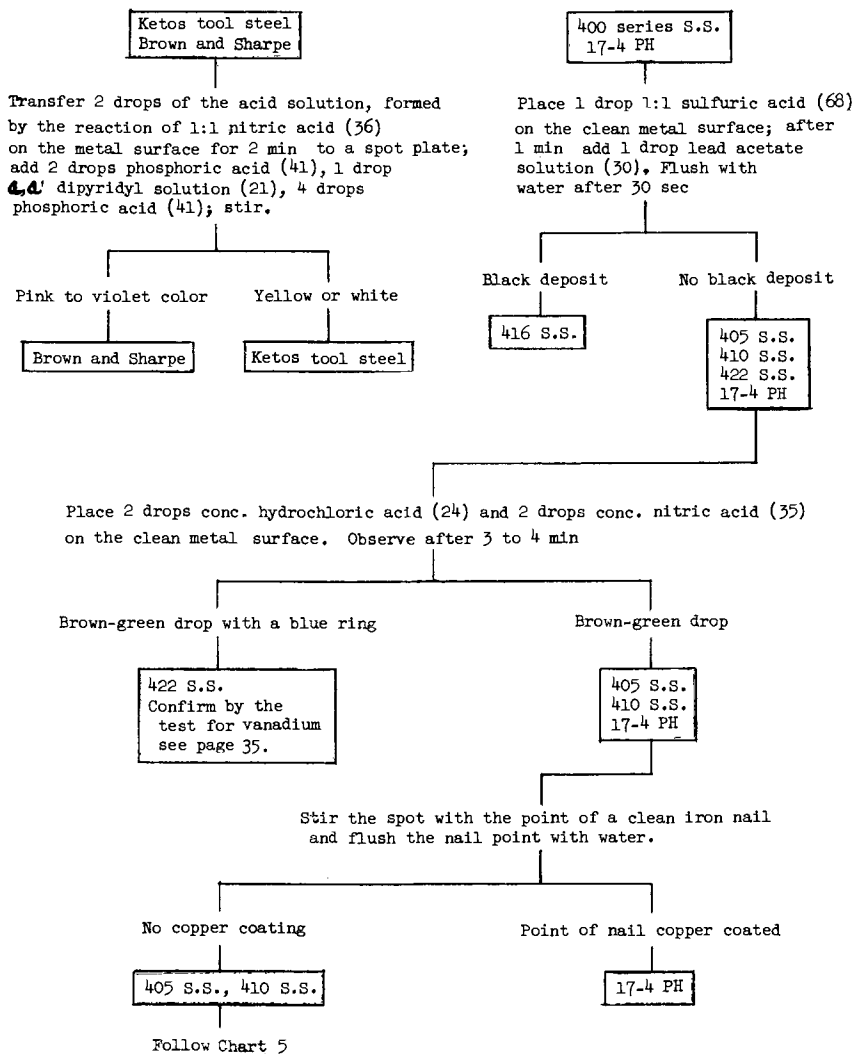


CHART 5

405, 410 stainless steel

Place 6 drops conc. hydrochloric acid (24) and 2 drops conc. nitric acid (35) on the clean metal surface. After reaction, transfer 5 drops to a spot plate. Add 1 ml sodium hydroxide (56); stir and filter using the cotton and medicine dropper method (see page 73). To 5 drops of the filtrate add 1 ml 1 M acetic acid (3), and 2 drops alizarin reagent (5); stir and add 2 drops 1M hydrochloric acid (27). Stir and observe.

Red precipitate

405 S.S.

No red precipitate

410 S.S.

300 series stainless steel	Inconel 600	Tantalum	Tin
17-7 PH	Inconel 625	Niobium B-66	Zinc
AM 350	Inconel 750	Silver	Lead
21-6-9	Molybdenum	Indium	Vanadium
20-Cb	Tungsten	Cadmium	
Rene 41	Aluminum and aluminum alloys		
Nichrome V	Magnesium and magnesium alloys		
Hastelloy X	Copper and copper alloys		
Nonmagnetic monel	Titanium and titanium alloys		

Identify copper and copper alloys by distinguishing colors reddish brown or yellow

Follow Chart 7

1 drop conc. nitric acid (35) on the metal surface

No reaction

Reaction

Tin	Molybdenum	Nonmagnetic monel
Zinc	Indium	Magnesium and magnesium alloys
Lead	Cadmium	Vanadium, silver

Follow Chart 6

Aluminum	17-7 PH
300 series S.S.	21-6-9
Hastelloy X	20-Cb
Nichrome V	Rene 41
AM 350	Titanium
Inconel 600	Tantalum
Inconel 625	Tungsten
Inconel 750	Niobium B-66

Follow Chart 12

CHART 6

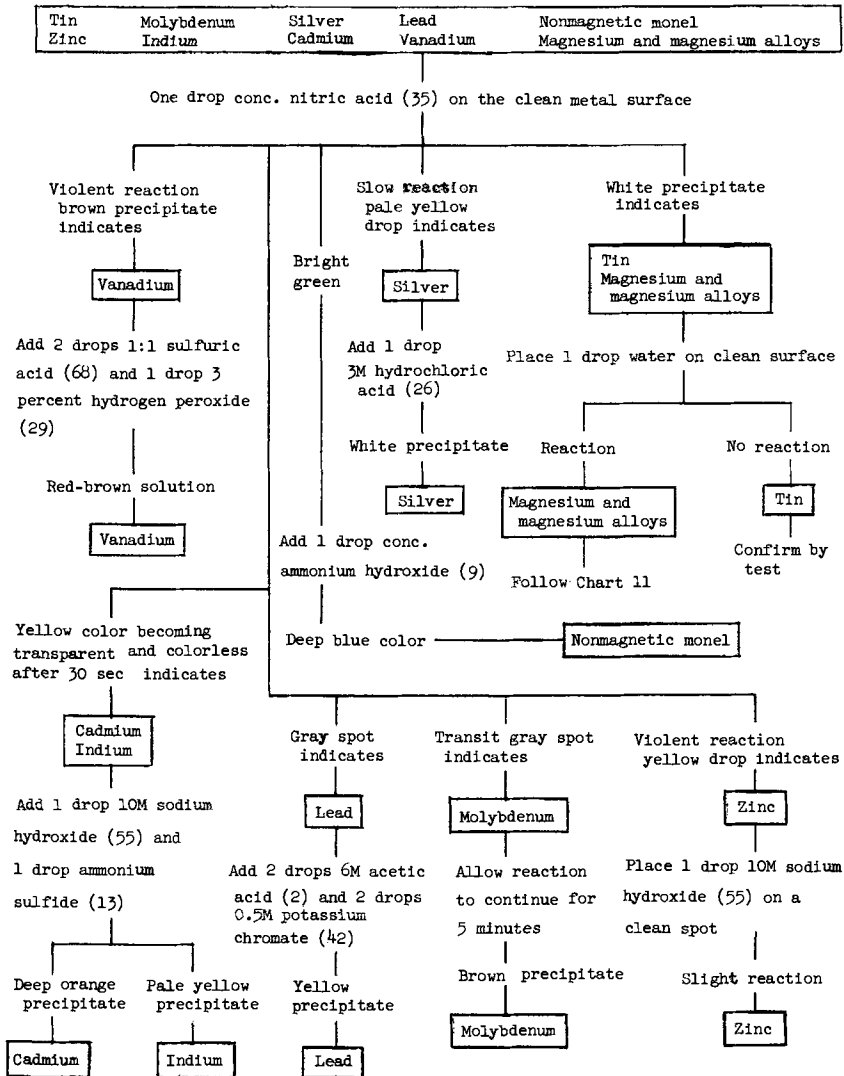


CHART 7

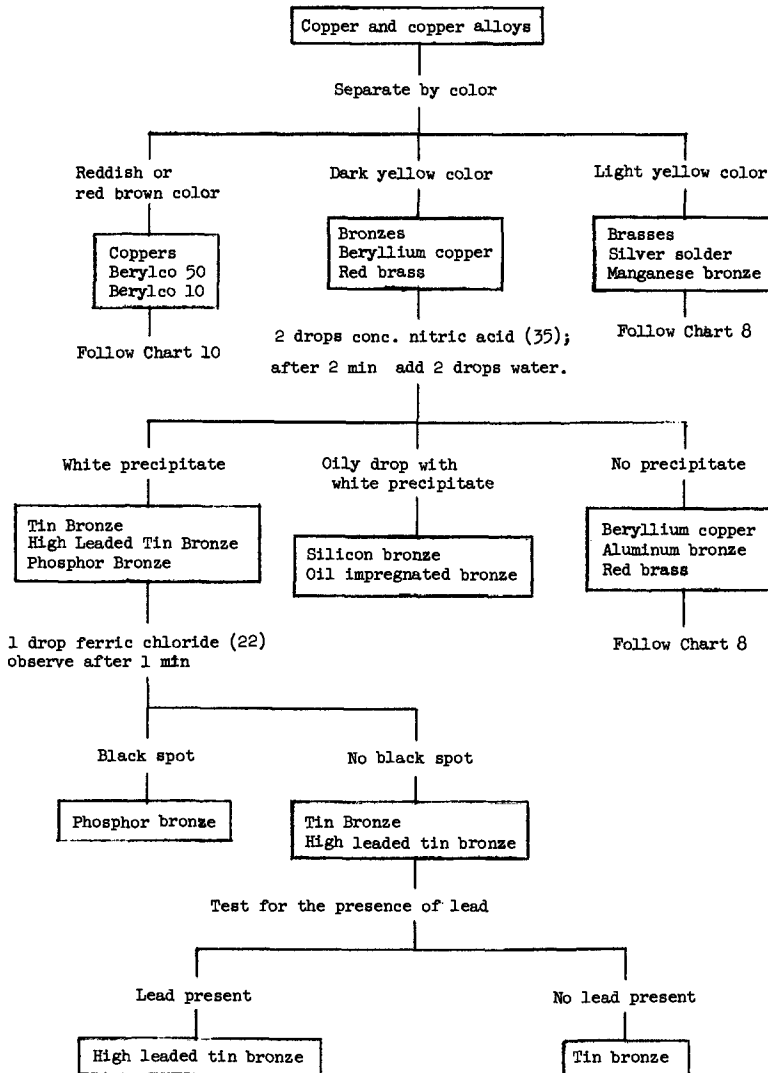


CHART 8

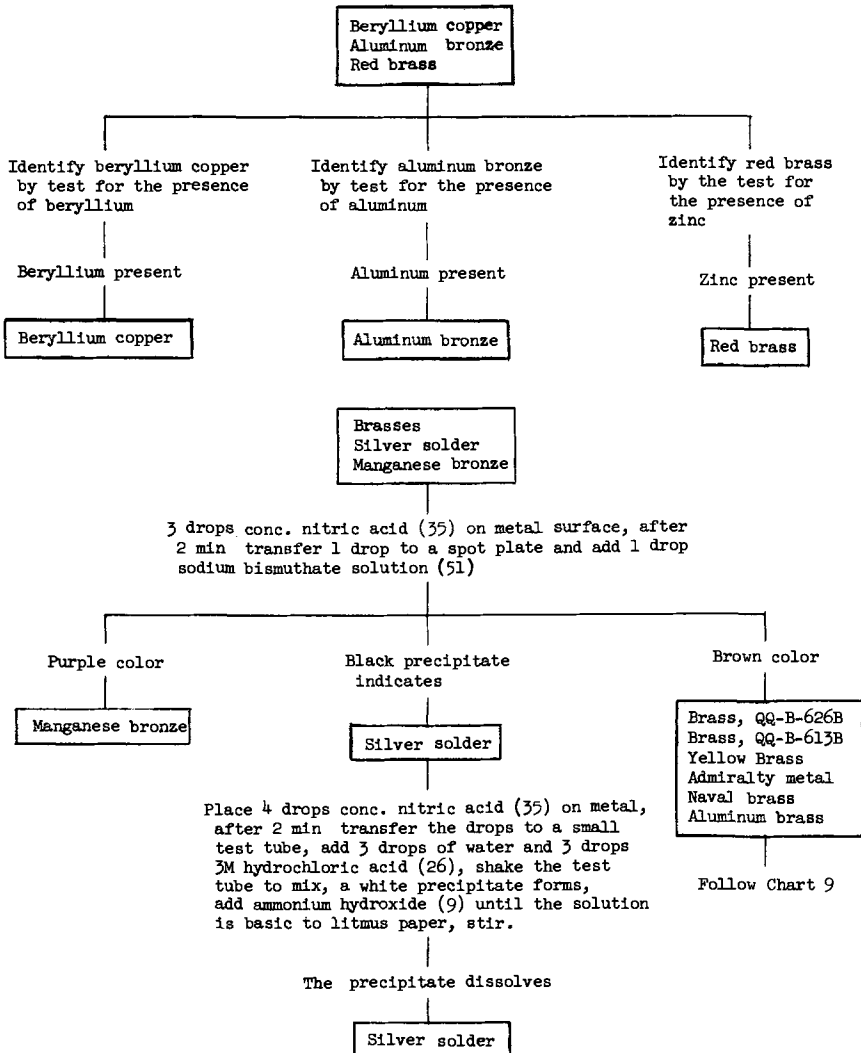


CHART 9

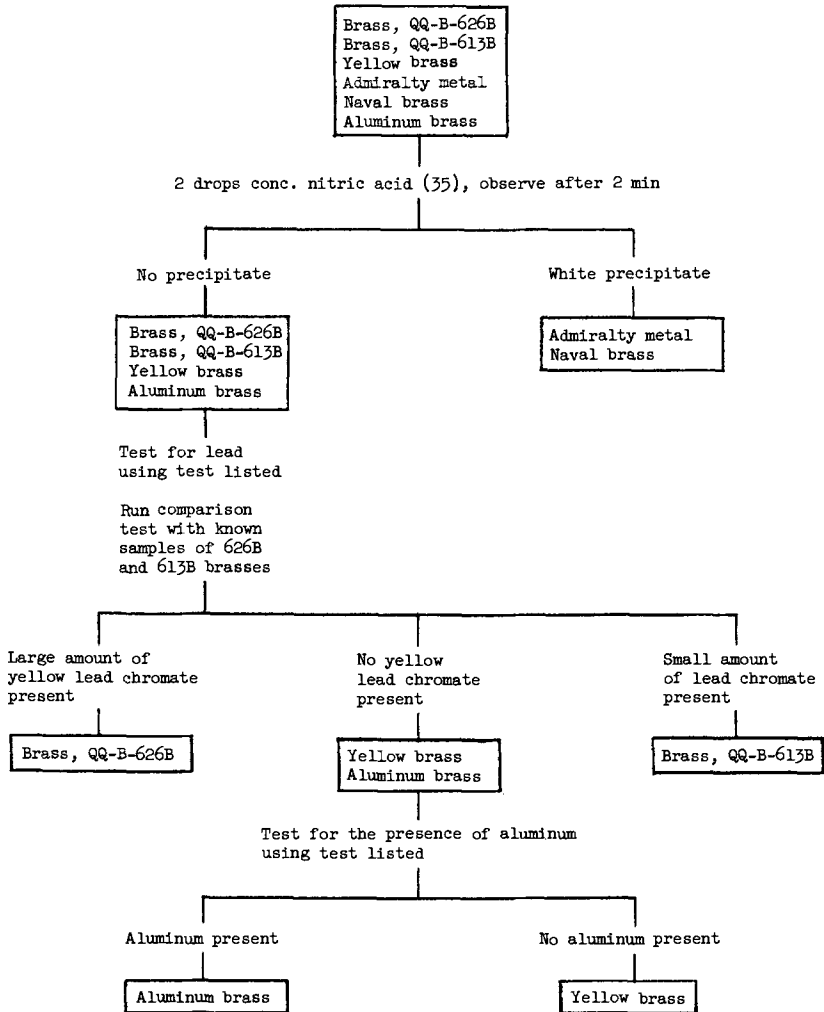


CHART 10

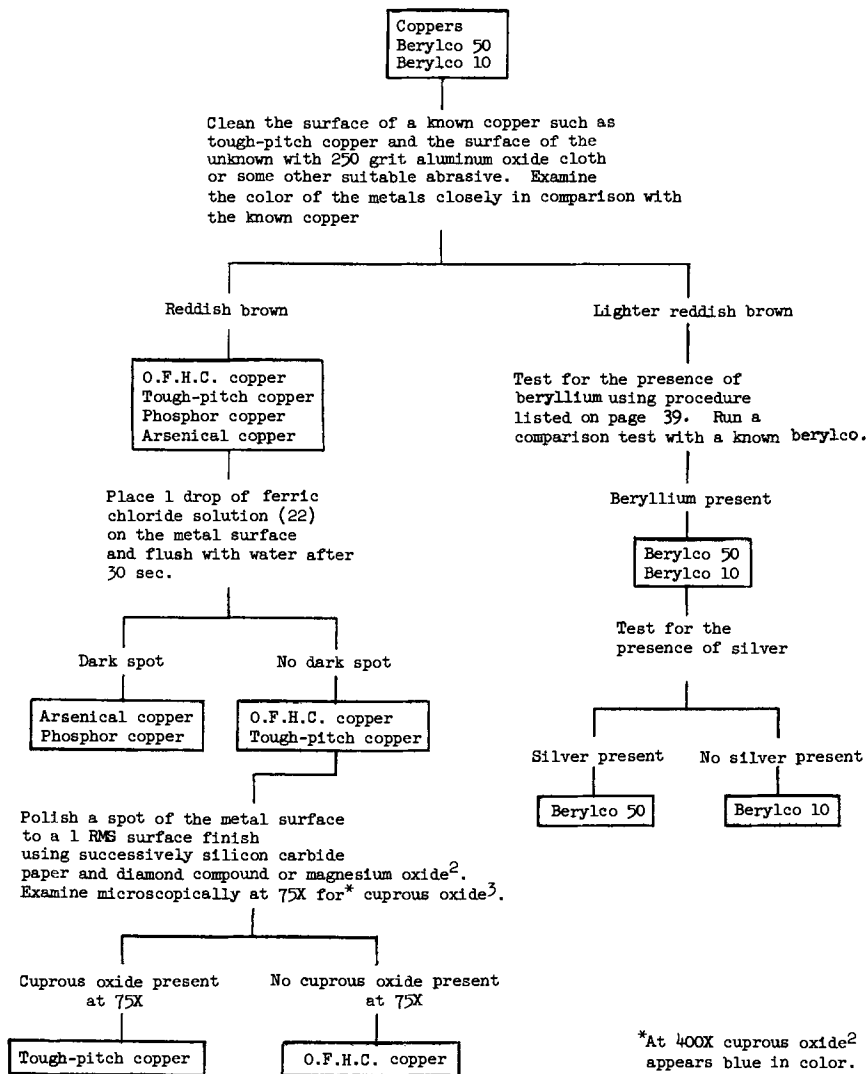
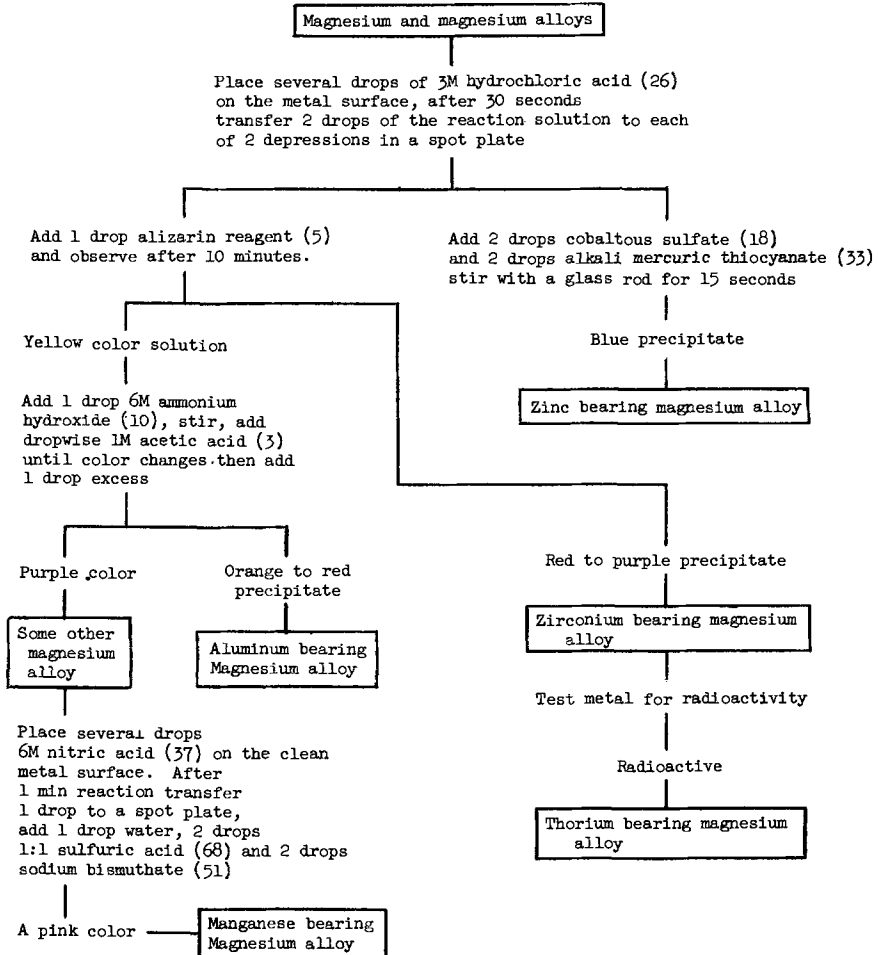


CHART 11



Note: The tests confirming Zr, Th, Al, Zn, Mn, can be applied quantitatively if simultaneous tests are made with known samples, e.g., an alloy containing 1 percent Zn is readily distinguished from one containing 3 percent Zn. This method can be employed to distinguish e.g., AZ 31 from AZ 63 or AZ 92 etc.

CHART 12

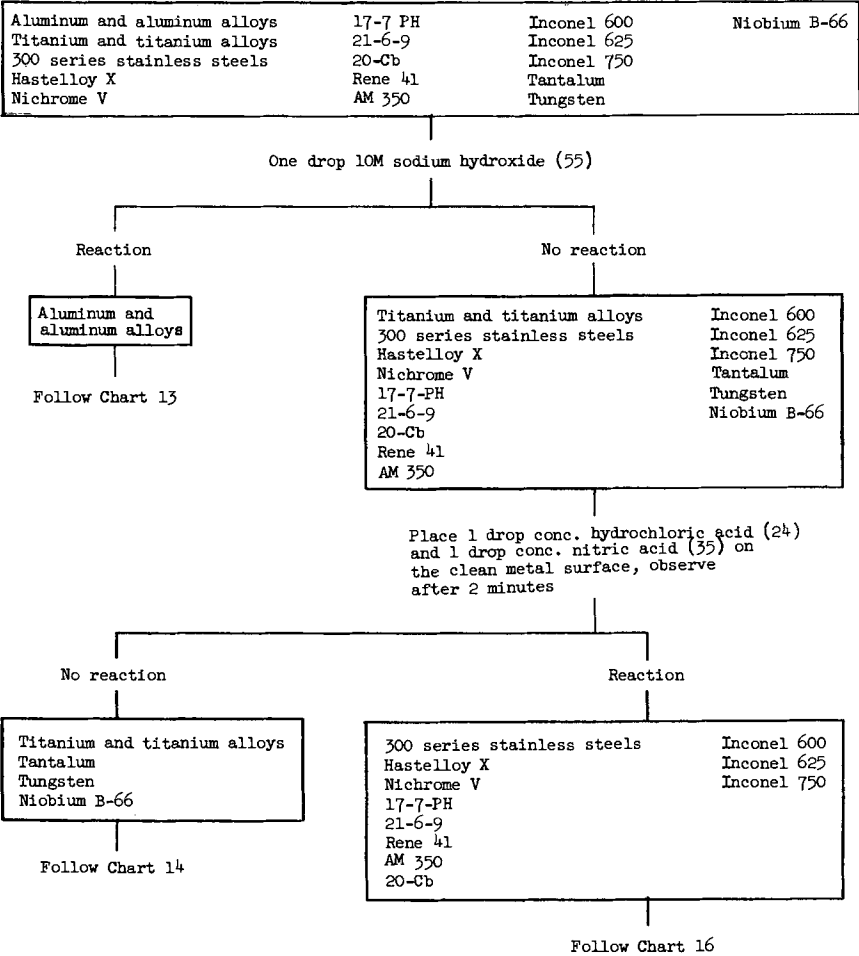


CHART 13

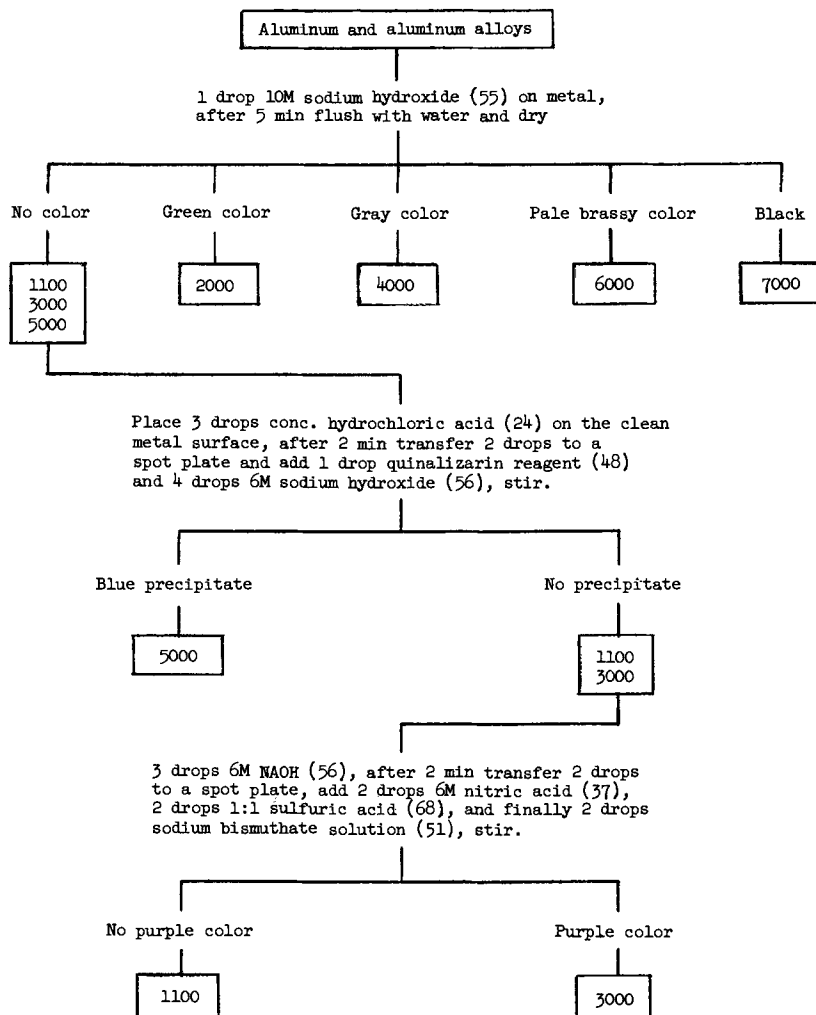


CHART 14

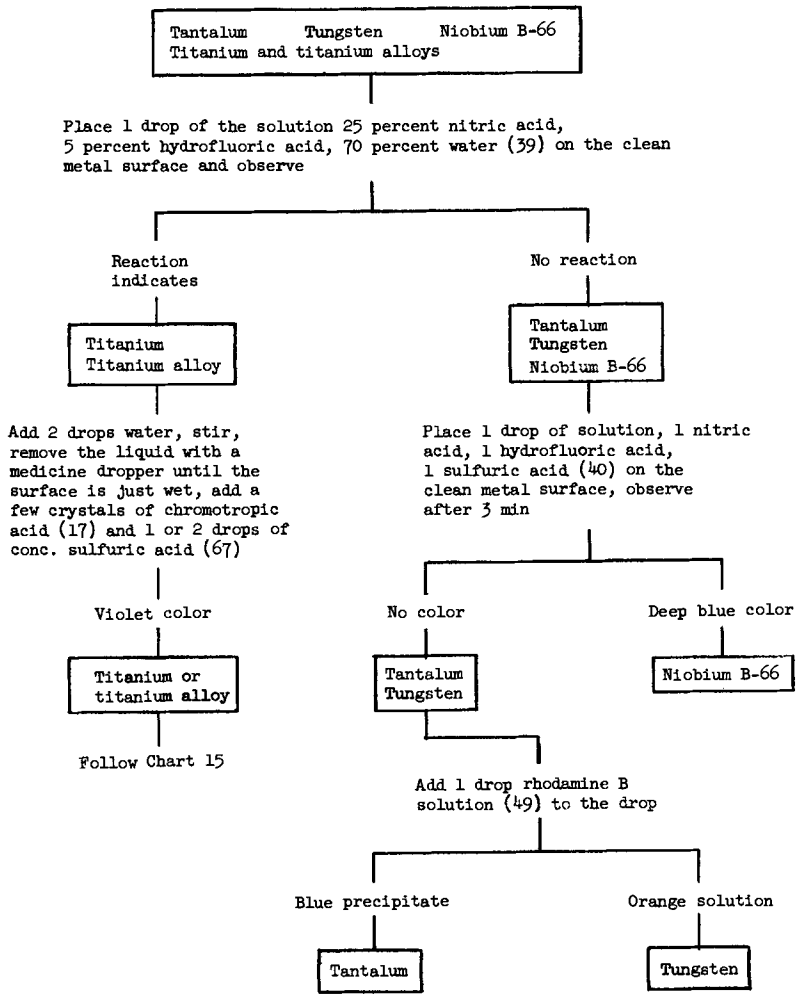


CHART 15

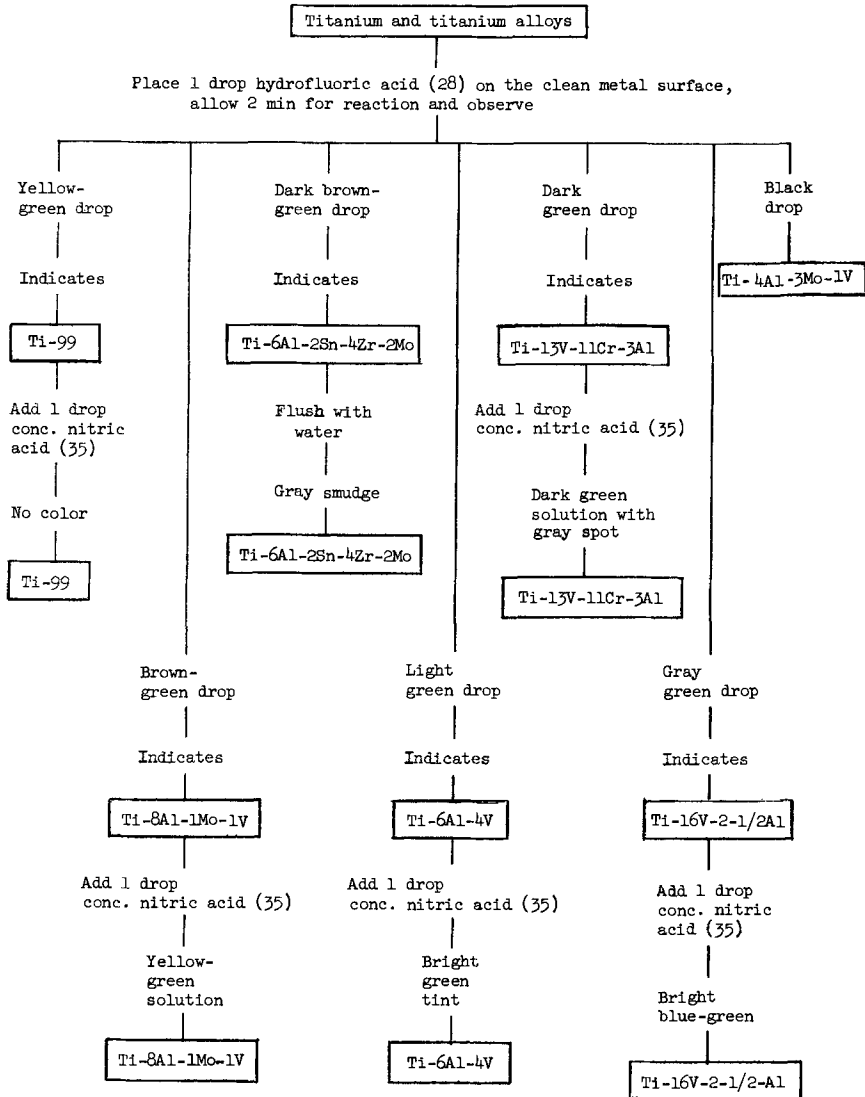


CHART 16

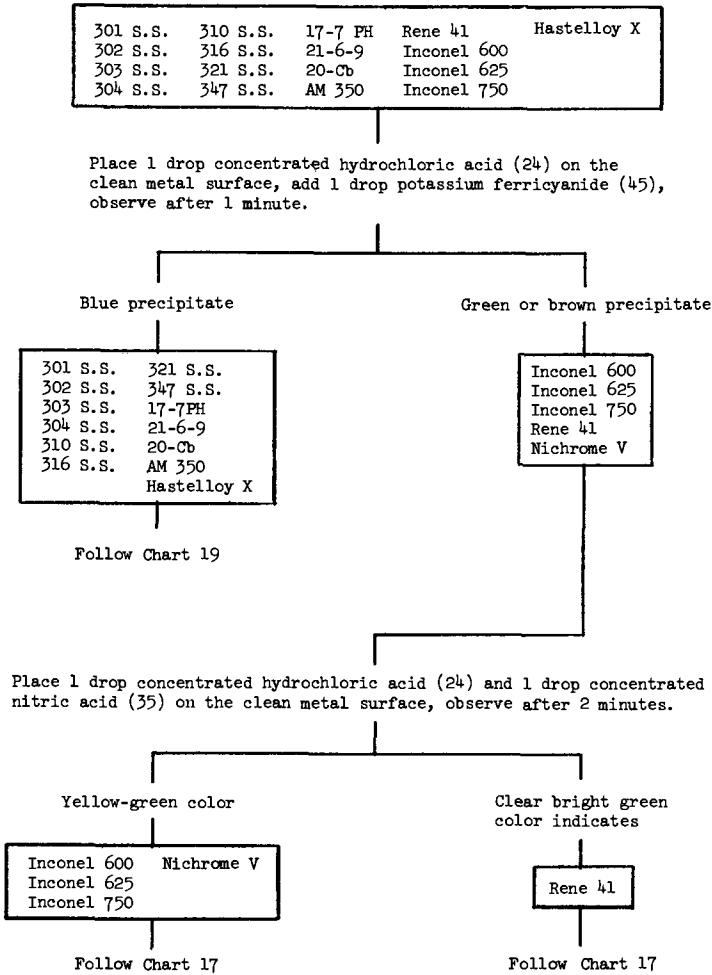


CHART 17

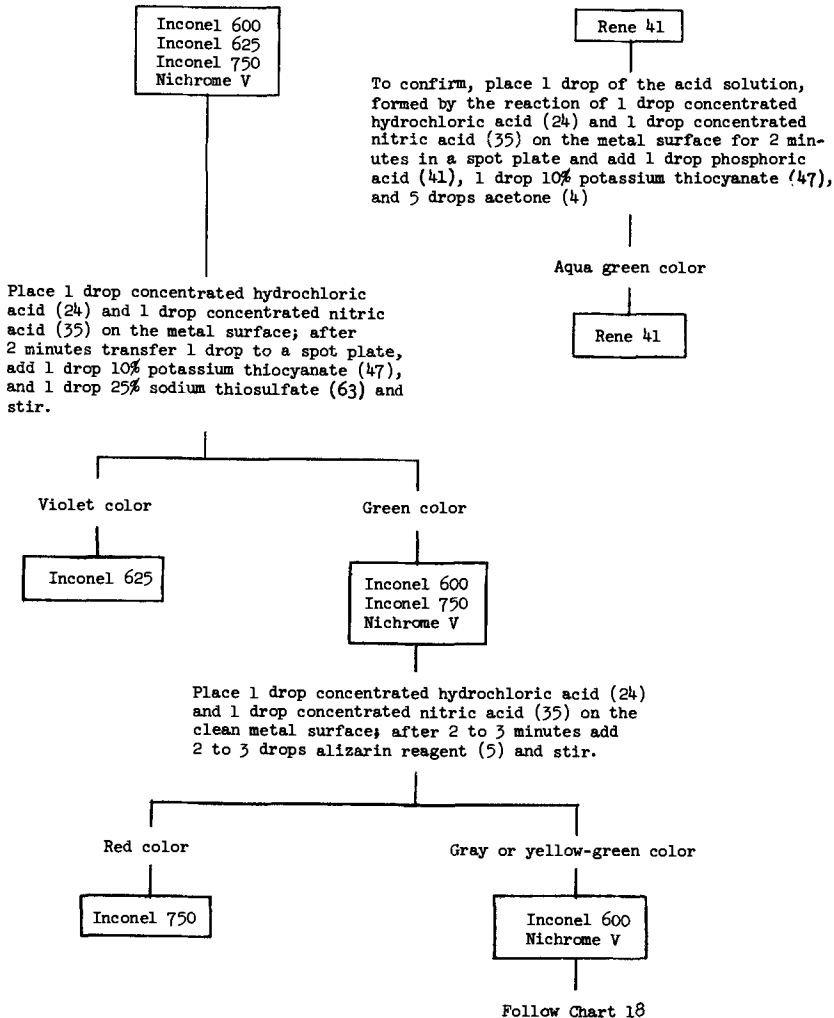


CHART 18

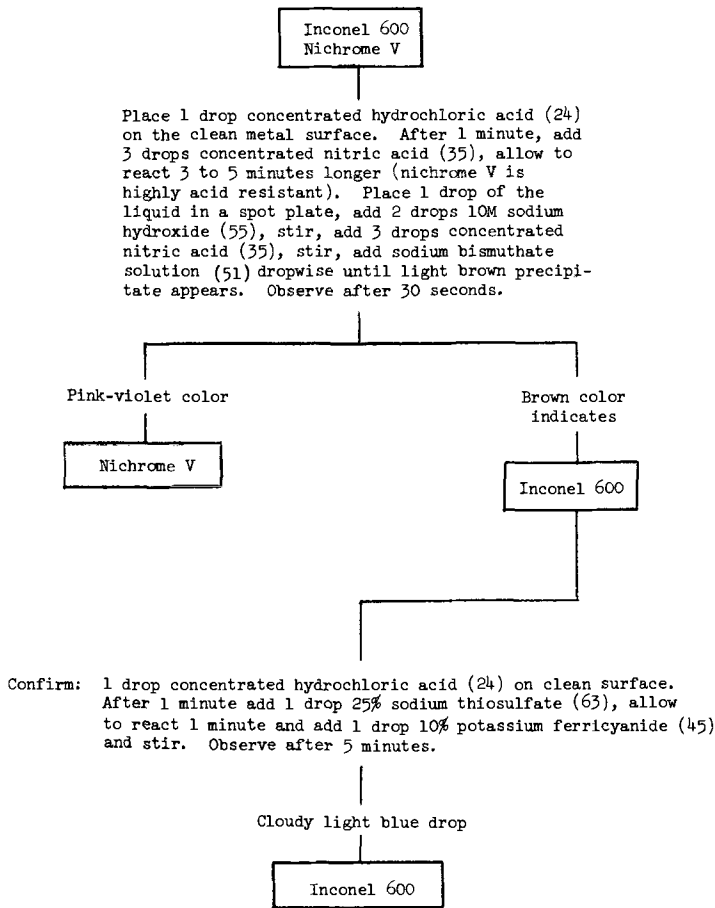


CHART 19

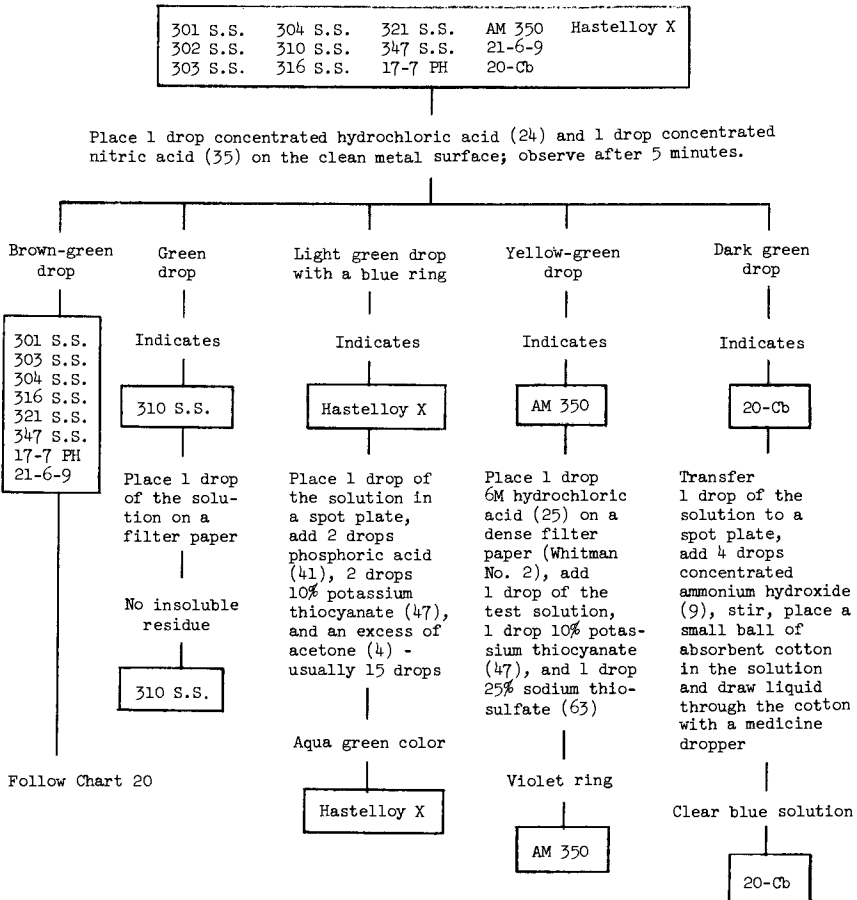


CHART 20

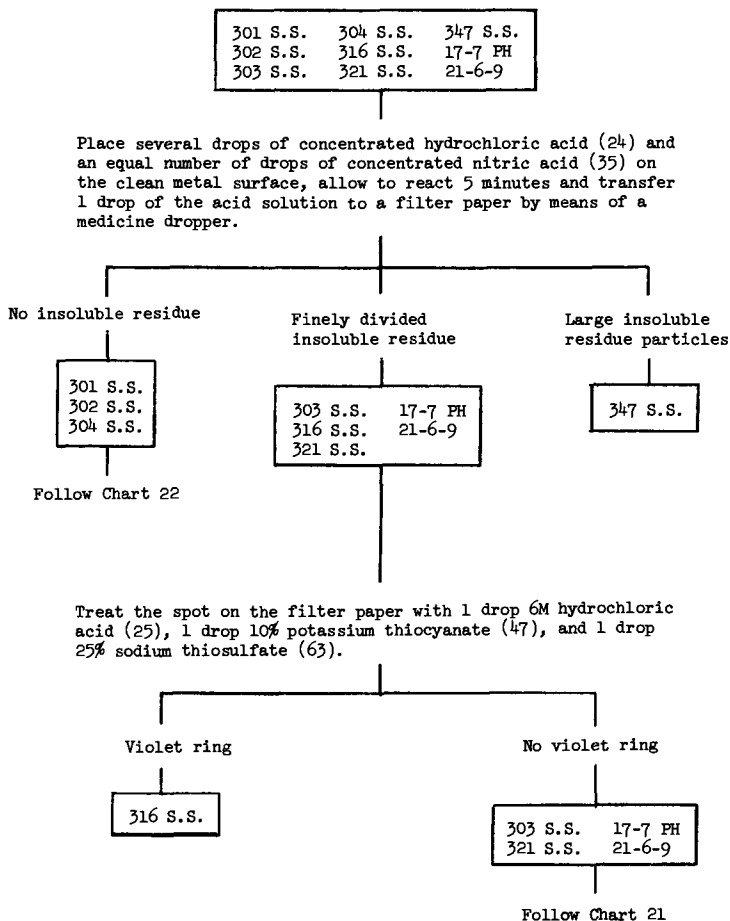


CHART 21

303 S.S.	17-7 PH
321 S.S.	21-6-9

Place 1 drop concentrated hydrochloric acid (24) and 1 drop concentrated nitric acid (35) on the clean metal surface. After 5 minutes, transfer 1 drop of the solution to a spot plate. Add 2 drops of concentrated sulfuric acid (67) to the remaining solution on the metal surface, stir with a glass rod and after 2 minutes add 2 drops of water, stir. Finally, add a small amount of chromotropic acid powder (17), stir.

Green color

Violet color

303 S.S.
17-7 PH
21-6-9

321 S.S.

Add 2 drops 10M sodium hydroxide (55) to the drop transferred to the spot plate in the above step, stir, add 4 drops concentrated nitric acid (35), stir, add 5 or 6 drops of sodium bismuthate reagent (51).

Deep red-violet color develops immediately

Slight violet color develops

21-6-9

303 S.S.	17-7 PH
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Place 1 drop concentrated hydrochloric acid (24) on the clean metal; observe after 5 minutes.

No color indicates

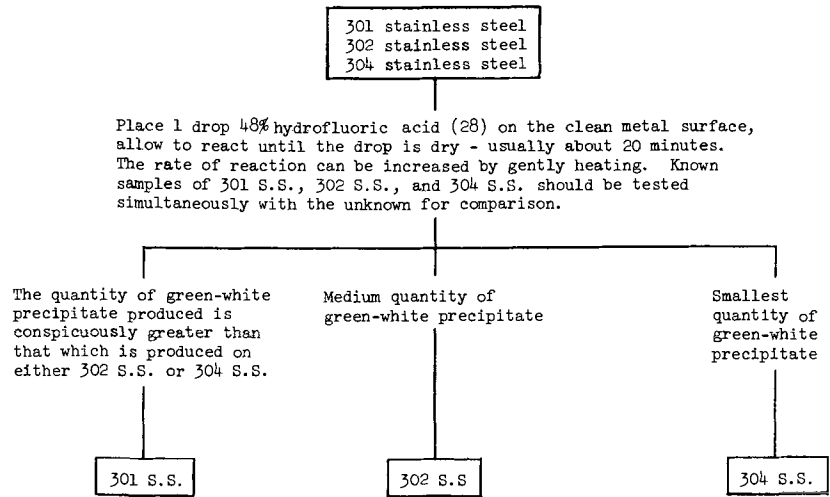
Gray spot

17-7 PH

303 S.S.

Confirm by test for the presence of aluminum

CHART 22



Procedures for Determining the Alloys in Low Alloy Steels

1. Chromium and Molybdenum

(a.) Place six drops of concentrated hydrochloric acid (24) on the clean metal surface or on a small portion of the metal filings in a small test tube, add three drops of concentrated nitric acid (35) and three drops of water. After two to three minutes transfer ten drops of the acid solution to a depression of a spot plate.

(b.) Add ten drops of 10 *M* sodium hydroxide (55) and five drops of three percent hydrogen peroxide (29). Stir with a glass rod and allow five minutes before proceeding.

(c.) Place a small ball of absorbent cotton in the solution. Using a medicine dropper, draw off the clear liquid transferring five drops (for molybdenum test) and five drops (for chromium test) to each of two depressions in a spot plate. Test for molybdenum: add a few grains of solid potassium ethyl xanthogenate (44), stir and add three or four drops of 3 *M* hydrochloric acid (26). A deep pink color identifies the presence of molybdenum (see Ref 4). Test for chromium: add two drops of diphenylcarbazide reagent (20), stir with a glass rod, after one minute add with stirring one to two drops of 3 *M* sulfuric acid (69). The bright red color should disappear leaving a faint violet color which intensifies after one to two minutes. (The solution, at this point, is acid.) For an alternate procedure on molybdenum, see Tool Steels.

2. Iron

(a.) Place one drop of 1:1 nitric acid (36) on the clean metal surface. Allow to react for one minute.

(b.) Add one drop of ten percent potassium thiocyanate (47).

A blood red color identifies the presence of iron.

3. Manganese

(a.) Place one drop of the acid test solution, from the reaction of 1:1 nitric acid (36) on the surface of the metal, in a spot plate depression.

(b.) Add sodium bismuthate reagent (51) dropwise until a brown precipitate appears. Let stand two or three minutes and add one drop of concentrated nitric acid (35).

A pink color confirms the presence of manganese.

4. Nickel

(a.) Place two drops of 1:1 nitric acid (36) on the clean metal surface. Allow two minutes for reaction and transfer one drop to a spot plate.

(b.) Add one drop of phosphoric acid (41) and stir.

(c.) Add two drops of dimethylglyoxime reagent (19) and stir.

(d.) Add one drop of concentrated ammonium hydroxide (9).

A pink to red precipitate identifies the presence of nickel.

5. *Vanadium*

(a.) Place three to four drops of 1:1 nitric acid (36) on the surface of the metal and after allowing two to three minutes to react, transfer two drops to a depression of a spot plate.

(b.) Add two drops of phosphoric acid (41) to complex the iron. Allow to react until the solution becomes colorless, however, some grey insolubles may be present. If the solution is not colorless, add another drop of phosphoric acid (41) and proceed.

(c.) Add one drop of a, a' dipyridyl reagent (21).

(d.) Add four drops of concentrated ammonium hydroxide (9) and stir.

(e.) Add two drops of phosphoric acid (41) and stir. A pink to violet color identifies the presence of vanadium (see Ref 4).

Procedures for Determining the Alloys in Tool Steels

1. *Chromium* (See Low Alloy Steels, paragraph 1.)

2. *Cobalt*

(a.) Place two drops of concentrated hydrochloric acid (24) and two drops of concentrated nitric acid (35) on the surface of the metal. Allow two to three minutes to react and transfer two drops of the solution to a spot plate.

(b.) Add two drops of a saturated solution of sodium fluoride (54), or phosphoric acid (41) may be used, to mask the interference of iron. Allow to react with stirring until the solution is clear.

(c.) Add two drops of ten percent potassium thiocyanate (47) and two to five drops of acetone (4). An aqua green to blue green color identifies the presence of cobalt. The color is not stable and will disappear; however, on the addition of a few drops of acetone (4), the color will return. A pink color following the aqua green color confirms the presence of molybdenum also.

3. *Iron* (See Low Alloy Steels, paragraph 2.)

4. *Manganese* (See Low Alloy Steels, paragraph 3.)

5. *Molybdenum*

(a.) Place one drop of 6 M hydrochloric acid (25) and one drop 1:1 nitric acid (36) on the clean metal surface, allow to react for two minutes and transfer one drop to a dense (Whatman #2) filter paper previously spotted with one drop of 6 M hydrochloric acid (25).

(b.) Add one drop of ten percent potassium thiocyanate (47) to the center of the fleck and after soaking in, add one drop of 25 percent sodium thio-sulfate (63).

A pink to violet ring around the center fleck identifies the presence of molybdenum (see Ref 4). If two to three drops of acetone (4) is added, an aqua green color identifies the presence of cobalt, if present. (See Low Alloy Steels, paragraph 1.)

6. *Vanadium* (See Low Alloy Steels, paragraph 5.)

7. *Tungsten*

Method 1—

(a.) Place four drops of concentrated hydrochloric acid (24) and four drops of concentrated nitric acid (35) on the clean metal surface. Allow to react for three to four minutes, add four drops of water and transfer about ten drops of the acid solution to a spot plate depression by means of a medicine dropper.

(b.) Add about ten drops of 20 percent sodium peroxide (60). Check for alkalinity with pink litmus paper. Solution at this point must be strongly alkaline and thoroughly stirred.

(c.) Place a ball of absorbent cotton (white surgical cotton rolled into a tight ball of about one-half inch in diameter) in the solution, draw the liquid through the cotton ball with a medicine dropper by pressing the tip firmly down in the cotton. The liquid removed in this manner should be clear; however, it may have a yellow tint. Place five drops of the liquid in a small test tube and add about five drops of water.

(d.) Add ten drops of concentrated nitric acid (35), stir by shaking the test tube in a circular motion.

(e.) Observe after four to five minutes.

A yellow precipitate identifies the presence of tungsten. Low concentrations of tungsten (one percent or less in the metal content) will be revealed by a pale yellow milky solution. In Step *b*, ten drops of 10 *M* sodium hydroxide (55) and five drops of three percent hydrogen peroxide (29) may be substituted for the 20 percent sodium peroxide (60).

Method 2—

One drop of the liquid removed with the medicine dropper in Step *c* is placed on filter paper previously spotted with two drops of 6 *M* hydrochloric acid (25), a yellow fleck will appear if tungsten is present. Add two drops of 20 percent stannous chloride (65) and the fleck will turn blue if tungsten is present. Add one drop of 10 percent potassium thiocyanate (47) and a red circle will appear outside the center fleck if molybdenum is present (see Ref 4).

Procedures for Determining the Alloys in Nickel-Chromium-Iron Metals

1. *Aluminum*

(a.) Place about eight drops of aqua regia (14) on the clean metal surface. Allow five minutes to react and transfer five drops to a spot plate.

(b.) Add 6 *M* sodium hydroxide (56) until strongly alkaline (usually about 15 drops) and stir with a glass rod.

(c.) Using a medicine dropper, filter the solution through absorbent cot-

ton (method described in Appendix A) transferring eight drops of the liquid to another depression in the spot plate.

(d.) Add 1 *M* acetic acid (3) until neutral to phydron paper or showing just pink on blue litmus paper (usually one ml of 1 *M* acetic acid (3) is required).

(e.) Add two drops of Alizarin reagent (5) and observe after one minute. An orange to red precipitate indicates aluminum is present. Add two drops of 1 *M* hydrochloric acid (27) and stir. An orange to red precipitate confirms the presence of aluminum.

2. *Chromium* (See Low Alloy Steels, paragraph 1.)

(a.) Allow a mixture of equal drops of concentrated hydrochloric acid (24) and concentrated nitric acid (35) to react on the metal surface until action ceases. Transfer ten drops of the reaction solution to a test tube, make alkaline with 6 *M* sodium hydroxide (56) plus one ml in excess.

(b.) Add ten drops of three percent hydrogen peroxide (29) and place the test tube in a boiling water bath (described in Appendix A) for about three minutes or until the bubbling caused by the hydrogen peroxide ceases. Cool, centrifuge, and pour the filtrate into a clean test tube. Discard the residue. The solution may have a faint yellow color at this point. Add one ml of 0.3 *M* barium chloride (15). If any appreciable amount of chromium is present, a yellow precipitate will appear.

(c.) Centrifuge, discard the filtrate. Add three drops of 3 *M* nitric acid (38), heat on a boiling water bath for three minutes, cool, and add three drops of three percent hydrogen peroxide (29).

A blue color confirms the presence of chromium. The color is unstable in proportion to the content of chromium in the metal being tested.

3. *Cobalt* (See Tool Steels, paragraph 2.)

4. *Copper*

(a.) Place two drops of concentrated hydrochloric acid (24) and two drops of concentrated nitric acid (35) on the clean metal surface. Allow to react for two minutes and transfer two drops of the solution to a spot plate.

(b.) Add four drops of water, eight drops of concentrated ammonium hydroxide (9), and stir.

(c.) Place a ball of absorbent cotton in the solution, using a medicine dropper draw off the liquid through the cotton. Place the liquid in another depression in the spot plate and observe.

A blue color identifies the presence of copper.

5. *Iron*

(a.) Place one drop of concentrated hydrochloric acid (24) and one drop of concentrated nitric acid (35) on the metal surface. Allow two minutes for reaction.

(b.) Add one drop of ten percent potassium thiocyanate (47). Observe after one minute.

A red color, which is proportional in intensity to the iron content of the metal, confirms the presence of iron.

6. *Manganese*

(a.) React equal drops of concentrated hydrochloric acid (24) and concentrated nitric acid (35) on the metal surface for five minutes and transfer one drop of the solution to a spot plate.

(b.) Add two drops of 10 *M* sodium hydroxide (55), stir, add four drops of concentrated nitric acid (35), stir, and add five or six drops of sodium bis-muthate reagent (51).

A pink to violet color identifies the presence of manganese. The intensity of the color is proportional to the manganese content.

7. *Molybdenum* (See Tool Steels, paragraph 5.)

(a.) Place equal drops of concentrated hydrochloric acid (24) and concentrated nitric acid (35) (aqua regia (14) may be substituted) on the metal surface. Allow three to four minutes for reaction and transfer two drops to a spot plate.

(b.) Add two drops of ten percent potassium thiocyanate (47).

(c.) Add 25 percent sodium thiosulfate (63) dropwise with stirring until the red color begins to disappear.

A violet color will appear if molybdenum is present.

8. *Nickel*

(a.) Place two drops of concentrated hydrochloric acid (24) and two drops of concentrated nitric acid (35) on the clean metal surface. After two minutes, transfer one drop to a spot plate.

(b.) Add two drops of phosphoric acid (41), stir thoroughly.

(c.) Add two drops of dimethylglyoxime reagent (19), stir, and add two drops of concentrated ammonium hydroxide (9).

A pink to red color precipitate confirms the presence of nickel.

9. *Sulfur*

(a.) Allow three drops of 1:1 sulfuric acid (68) to react on the metal for one to two minutes and add one drop of five percent lead acetate (30). After 20 seconds, flush the spot with water.

A black sulphide deposit identifies the presence of sulfur (see Ref 5).

10. *Titanium*

(a.) Place one drop of concentrated hydrochloric acid (24) and one drop of concentrated nitric acid (35) on the metal surface, and after five minutes, remove the liquid with a medicine dropper leaving the surface wet.

(b.) Add two drops of concentrated sulfuric acid (67), stir with a glass rod,

and add a small amount of chromotropic acid powder (17).

A violet color identifies the presence of titanium.

11. *Tungsten* (See Tool Steels, paragraph 7.)

12. *Vanadium*

(a.) Place two drops of concentrated hydrochloric acid (24) and two drops of concentrated nitric acid (35) on the metal. After five minutes, transfer two drops of the solution to a spot plate.

(b.) Add two drops of phosphoric acid (41). Allow to react without stirring until the solution is clear and add one drop of concentrated sulfuric acid (67). Stir and allow to react until the solution is clear.

(c.) Add two drops of a, a' dipyridyl (21) and stir.

(d.) Add four drops of concentrated ammonium hydroxide (9) and stir.

A violet color identifies the presence of vanadium.

Procedures for Determining the Alloys in Aluminum and Aluminum Alloys

1. *Copper*

(a.) Place one drop of 10 *M* sodium hydroxide (55) on the metal surface and allow to react for five minutes.

(b.) Flush the spot with tap water and dry with air. (The spot will be green or black if any appreciable amount of copper is present.)

(c.) Add one drop of 6 *M* nitric acid (37) to the green or black spot. The spot will dissolve.

(d.) Add two drops of concentrated ammonium hydroxide (9).

A blue color identifies the presence of copper. This method will determine the presence of copper in the 2000 and 7000 series aluminum alloys.

2. *Magnesium*

(a.) Place four to five drops of concentrated hydrochloric acid (24) on the metal. Allow two minutes for reaction and transfer two drops of the acid solution to a spot plate depression by means of a medicine dropper.

(b.) Add one drop of quinalizarin reagent (48) and four drops of 6 *M* sodium hydroxide (56).

A blue precipitate identifies the presence of magnesium (see Ref 4).

Alternate Procedure—

(a.) Place several drops of concentrated hydrochloric acid (24) on the metal and after two minutes, transfer five or six drops to a small test tube.

(b.) Add 6 *M* sodium hydroxide (56) until the solution is strongly alkaline to litmus paper.

(c.) Centrifuge and pour off the liquid.

(d.) Add concentrated hydrochloric acid (24) to dissolve the residue.

(e.) Add one or two drops of quinalizarin reagent (48) and two drops of 6 *M* sodium hydroxide.

A blue precipitate confirms the presence of magnesium.

3. *Manganese*

(a.) Place several drops of 6 *M* sodium hydroxide (56) on the metal surface and allow two minutes for the reaction. Transfer two drops of the solution to a spot plate.

(b.) Add two drops of 6 *M* nitric acid (37), two drops of 1:1 sulfuric acid (68), and two drops of sodium bismuthate reagent (51), and stir.

A pink color confirms the presence of manganese.

4. *Silicon*

(a.) Polish the metal surface to RMS 2 to 3 and swab with cotton dipped in a solution of two ml of 1 *M* sodium hydroxide (59) diluted to eight ml with water.

(b.) View with a microscope using a $\times 400$ to $\times 500$ lens.

Silicon does not combine with aluminum to form a compound and is present in the primary state. It generally appears in the form of dark splinters (see Ref 6). This method is suitable to determine the presence of silicon in 4000 series aluminum wrought alloys and in high silicon bearing aluminum castings.

5. *Zinc*

(a.) Place three drops of 6 *M* hydrochloric acid (25) on the surface of the metal; after two minutes, add three drops of water, stir and transfer three drops to a spot plate.

(b.) Add three drops of cobaltous sulfate solution (18), three drops of alkali mercuric thiocyanate solution (33), and stir with a glass rod for fifteen seconds rubbing the sides of the spot plate depression.

A blue precipitate confirms the presence of zinc (see Ref 4). The presence of zinc is easily determined in 7075 aluminum alloy by this method.

Procedures for Determining the Alloys in Copper and Copper Alloys

1. *Aluminum*

Method 1—

(a.) Place six drops of concentrated nitric acid (35) on the clean metal surface and allow two minutes for the reaction to complete.

(b.) Transfer four drops of the test solution to a small test tube and add four drops of 6 *M* sodium hydroxide (56) and stir. After one minute, add another four drops of 6 *M* sodium hydroxide (56), stir, place on a boiling water bath for two minutes and centrifuge.

(c.) Place one drop of the supernatant liquid in a spot plate depression, add one drop of one percent alizarin reagent (5), and stir.

(d.) Add two drops of 6 *M* acetic acid (2) and stir.

An orange color precipitate confirms the presence of aluminum.

Method 2—

(a.) Place two drops of the supernatant liquid of Step *c* (Method 1) in a spot plate depression and add two drops of 6 *M* hydrochloric acid (25) and stir.

(b.) Add one drop of aluminon reagent (7), stir, and add two drops of 6 *M* ammonium hydroxide (9) and stir.

A red precipitate confirms the presence of aluminum.

2. Beryllium

(a.) Transfer one drop of the solution formed by the reaction of two or three drops of concentrated nitric acid (35) on the surface of the metal to a spot plate depression.

(b.) Add one drop of 6 *M* sodium hydroxide (56) to neutralize or render weakly acid.

(c.) Add three drops of sodium (Di) ethylene diamine tetraacetate (52) and three drops of Morin reagent (34).

(d.) Add one drop of concentrated ammonium hydroxide (9), stir and transfer one drop to a Whatman #3 (thick, hard) filter paper previously spotted with one drop of Morin solution (34). A yellow precipitate in the central fleck indicates beryllium.

(e.) Wash the precipitate with two drops sodium (Di) ethylene diamine tetraacetate (52), two drops of water and two drops of acetone (4) successively (see Ref 4).

(f.) Add one drop of 6 *M* sodium hydroxide (56) and dry.

(g.) Place under ultra violet light.

A yellow fluorescing ring around the central fleck confirms the presence of beryllium. Beryllium 0.40 percent in copper alloys can be determined by this method.

3. Cadmium

(a.) Place several drops of the solution, taken from the reaction of concentrated nitric acid (35) on the metal, in a small test tube and make alkaline with 3 *M* ammonium hydroxide (11).

(b.) Add 4 *M* potassium cyanide solution (43) dropwise until the blue color of the ammonium-copper disappears.

(c.) Add three drops of ammonium sulfide (13).

A bright yellow precipitate confirms the presence of cadmium.

4. Chromium

(a.) Transfer three drops of the solution, from the reaction of 1:1 nitric acid (36) on the surface of the metal, to a spot plate depression.

(b.) Add three to five drops of 3 *M* sulfuric acid (69); stir and add two drops of diphenylcarbazide reagent (20).

(c.) Add one drop of three percent hydrogen peroxide (29) and two to three drops of 6 *M* sodium hydroxide (56).

A violet color confirms the presence of chromium.

5. Cobalt

Method 1—

(a.) Place several drops of concentrated nitric acid (35) on the surface of the metal. After two minutes, transfer two drops of the solution to a spot plate or small test tube.

(b.) Add one drop of concentrated ammonium hydroxide (9) and add dropwise 4 *M* potassium cyanide (43) until the blue color of the copper-ammonium is rendered colorless.

A green precipitate confirms the presence of cobalt.

Method 2—

(a.) Place three or four drops of concentrated nitric acid (35) on the metal and allow two minutes to react. Transfer one drop of the reaction solution to a spot plate or test tube and add four drops of sodium sulfate solution (62) to mask the copper. The product is a light brown precipitate.

(b.) Add four drops of ten percent potassium thiocyanate (47) (the solution turns milk white) and 15 drops of acetone (4). Let stand for two minutes and add four drops of phosphoric acid (41).

(c.) Add several drops of acetone (4).

A green to blue color identifies the presence of cobalt. The cobalt is in the acetone belt above the white precipitate. This is readily observed if the test is conducted in a small test tube.

6. Lead

Method 1—

(a.) Several drops of concentrated nitric acid (35) is placed on the surface of the metal and allowed to react for about two minutes. Transfer one drop to a Whatman #3 (thick) qualitative filter paper.

(b.) After the acid test solution has soaked into the filter paper, add one drop of 0.2 percent sodium rhodizonate (61) to the center of the fleck. A blue to violet color develops immediately.

(c.) Add one or two drops of tartaric acid-sodium bitartrate buffer solution (70).

A scarlet precipitate identifies the presence of lead (see Ref 4). This procedure will determine the presence of lead in sheet brass with as little as 0.15 percent lead content.

Method 2—

(a.) Place six drops of concentrated nitric acid (35) on the clean metal surface and allow two minutes for reaction.

(b.) Add six drops of water and transfer ten drops to a test tube.

(c.) Add ten drops of 6 *M* sodium hydroxide (56) and stir. After one minute add six more drops of 6 *M* sodium hydroxide (56) and stir. (The former addition of sodium hydroxide precipitates lead; the latter addition dissolves the lead; however, the copper remains as solid cupric hydroxide.)

(d.) Place the test tube on a boiling water bath for two or three minutes and centrifuge while hot.

(e.) To the supernatant liquid, add ten drops of 0.5 *M* potassium chromate (42), stir and add five drops of 6 *M* acetic acid (2).

A yellow precipitate confirms the presence of lead.

7. *Lead and Zinc*

(a.) Place six drops of concentrated nitric acid (35) on the surface of the metal. After two minutes add six drops of water, stir and transfer ten drops to a small test tube.

(b.) Add ten drops of 6 *M* sodium hydroxide (56) and stir. Zinc and lead will precipitate out of solution along with copper. After one minute, add another ten drops of 6 *M* sodium hydroxide (56) and stir. Zinc and lead will dissolve leaving the copper in residue.

(c.) Place the test tube in a boiling water bath for two minutes and centrifuge while hot.

(d.) Transfer three drops of the supernatant liquid to a spot plate to test for zinc. Transfer the rest of the liquid to a clean test tube and test for lead.

(1) *Zinc Test—*

(a.) Add three drops of 3 *M* hydrochloric acid (26) and stir. After one minute, add another three drops of 3 *M* hydrochloric acid (26).

(b.) Add three drops of cobaltous sulfate solution (18) and three drops of alkali mercuric thiocyanate (33).

(c.) Stir for 15 seconds with a glass rod rubbing the sides of the container.

A blue precipitate identifies the presence of zinc (see Ref 4).

(2) *Lead Test—*

(a.) Add 10 drops of 0.5 *M* potassium chromate (42), stir and add cautiously without stirring four drops of 6 *M* acetic acid (2).

A canary yellow precipitate in the acetic acid layer identifies the presence of lead. This method will determine 0.15 percent lead content in sheet brass.

8. *Manganese*

(a.) Place three drops of concentrated nitric acid (35) on the surface of the metal. Allow two minutes to react and transfer one drop to a spot plate.

(b.) Add one drop of sodium bismuthate solution (51) and observe.

A purple color confirms the presence of manganese.

9. *Nickel*

Method 1—

(a.) Place several drops of concentrated nitric acid (35) on the clean metal

surface. Allow two minutes to react and transfer two drops to a spot plate.

(b.) Add solid zinc oxide powder (72) in excess until the liquid is soaked up and a minute hill of clear zinc oxide exists.

(c.) Add two to four drops of dimethylglyoxime reagent (19).

A red color confirms the presence of nickel.

Method 2—

(a.) Place five drops of the acid solution, formed by the reaction of several drops of concentrated nitric acid (35) on the surface of the metal, in a small test tube and add five drops of concentrated hydrochloric acid (24). Add five drops of water.

(b.) Add magnesium metal (32) until all of the copper has precipitated out of solution. The solution should be clear and colorless.

(c.) Transfer two drops of the clear solution to a spot plate and add two drops of dimethylglyoxime solution (19).

(d.) Add two drops of 6 *M* ammonium hydroxide (10).

A red precipitate identifies the presence of nickel.

10. Silver

Method 1—

(a.) Place several drops of 1:1 nitric acid (36) on the clean metal surface. Allow two minutes for reaction.

(b.) Transfer two drops from the metal surface to a spot plate depression and add two drops of sodium bismuthate solution (51).

A dark grey precipitate indicates the presence of silver.

Method 2—

(a.) Place five drops of the acid solution, formed from the reaction of several drops of 1:1 nitric acid (36) on the surface of the metal, in a small test tube.

(b.) Add two drops of 3 *M* hydrochloric acid (26).

A white precipitate that dissolves upon adding several drops of concentrated ammonium hydroxide (9) confirms the presence of silver.

11. Tin

(a.) Place four drops of concentrated nitric acid (35) on the clean metal surface. Allow two minutes for reaction and transfer two drops of the acid solution to a small test tube.

(b.) Add ten drops of concentrated hydrochloric acid (24) and mix thoroughly.

(c.) Add small pieces of magnesium metal (32) to drive the copper out of solution. When all of the copper has precipitated, the solution will be clear and colorless.

(d.) Add one ml of water, stir and add four drops of mercuric chloride (31).

A white to grey precipitate confirms the presence of tin.

12. Zinc

(a.) Place two drops of concentrated nitric acid (35) on the surface of the metal. After two minutes, add two drops of water and transfer three drops of the solution to a spot plate depression.

(b.) Add four drops of 6 *M* sodium hydroxide (56) to make alkaline and stir. After one minute, add another four drops of 6 *M* sodium hydroxide (56) and stir. (The first addition of sodium hydroxide precipitates the zinc as zinc hydroxide and the latter addition dissolves the zinc hydroxide.)

(c.) Place a ball of surgical absorbent cotton in the solution and using a medicine dropper, draw off the liquid using the cotton as a filter.

(d.) Add four drops of 3 *M* hydrochloric acid (26) to four drops of the filtrate, stir thoroughly and add another four drops of 3 *M* hydrochloric acid (26) and stir.

(e.) Add three drops of cobaltous sulfate (18) and three drops of alkali mercuric thiocyanate (33). Stir for 15 seconds with a glass rod rubbing the sides of the spot plate. This starts precipitation of the zinc if present.

A blue precipitate identifies the presence of zinc.

Procedures for Determining the Alloys in Magnesium and Magnesium Alloys

1. Aluminum

Method 1—

(a.) Place several drops of 3 *M* hydrochloric acid (26) on the surface of the metal and after one minute, transfer two drops to spot plate depression.

(b.) Add one drop of alizarin reagent (5) and stir.

(c.) Add one drop of 6 *M* ammonium hydroxide (10) and stir.

(d.) Add dropwise 1 *M* acetic acid (3) until a color change is visible then add one drop in excess.

An orange to red precipitate confirms the presence of aluminum.

Method 2—

(a.) Transfer five drops of the acid solution, formed by the reaction of several drops of 6 *M* acetic acid (2) on the surface of the metal, to a small test tube.

(b.) Add five drops of 3 *M* ammonium acetate (8) and two drops of alizarin reagent (5).

(c.) Add five drops of 6 *M* ammonium hydroxide (10) and stir.

(d.) Let stand for one minute and centrifuge.

An orange to red gelintious precipitate confirms the presence of aluminum.

Method 3—

(a.) To five drops of the acid solution, formed by the reaction of several drops of 6 *M* acetic acid (2) on the metals' surface, placed in a test tube, add five drops of 3 *M* ammonium acetate (8) and two drops of aluminon reagent (7).

(b.) Add five drops of 6 *M* ammonium hydroxide (10) and stir.

(c.) Let stand for one minute and centrifuge.

A pink to red gelintious precipitate confirms the presence of aluminum.

2. Manganese

(a.) Place several drops of 6 *M* nitric acid (37) on the clean surface of the metal. Allow one minute for reaction and transfer one drop to a spot plate depression.

(b.) Add one drop of water, two drops of 1:1 sulfuric acid (68), and two drops of sodium bismuthate solution (51).

A pink to purple color identifies the presence of manganese. The intensity of the color is proportional to the content of manganese.

3. Zinc

(a.) Allow several drops of 3 *M* hydrochloric acid (26) to react for one minute on the metal and transfer two drops to a spot plate.

(b.) Add two drops of cobaltous sulfate solution (18) and two drops of alkali mercuric thiocyanate (33).

(c.) Stir 15 seconds with a glass rod rubbing the sides of the container while stirring.

A blue precipitate confirms the presence of zinc. The blue precipitate is proportional to the zinc content. Run a blank and a known to determine the quantity roughly. Magnesium alloys containing three percent and one percent zinc are easily differentiated by this method.

4. Zirconium

(a.) Place several drops of 3 *M* hydrochloric acid (26) on the surface of the metal, allow to react for one minute, and transfer two drops to a spot plate.

(b.) Add one drop of alizarin reagent (5) to the solution and let stand for ten minutes.

A red to purple precipitate identifies the presence of zirconium.

Procedures for Determining the Alloys in Titanium and Titanium Alloys**1. Aluminum**

(a.) Place one drop of concentrated hydrofluoric acid (28) on the clean metal surface, after one minute add three drops of concentrated nitric acid (35), and after one minute add six drops of water. Transfer the solution to a small test tube.

(b.) Add 0.5 ml of 6 *M* sodium hydroxide (56) and stir.

- (c.) Add another 0.5 ml of 6 *M* sodium hydroxide (56).
- (d.) Place the test tube in a boiling water bath for three minutes and centrifuge. Transfer three drops of the supernatant liquid to a spot plate depression.
- (e.) Acidify with 1 *M* acetic acid (3); usually six drops are required.
- (f.) Add two drops of alizarin reagent (5) and let stand for five to ten minutes.

A red to orange color identifies the presence of aluminum. If no aluminum is present, the solution turns yellow due to the acidified solution of alizarin having no aluminum product to absorb the alizarin.

2. *Molybdenum*

Method 1—

- (a.) Place one drop of concentrated hydrofluoric acid (28) on the metal being tested. Allow one minute for reaction.
- (b.) Add one drop of concentrated nitric acid (35) and after a minute, add a drop of water.
- (c.) Transfer two drops of the solution to a spot plate, add two drops of ten percent potassium thiocyanate solution (47), one drop of 25 percent sodium thiosulfate (63), and stir.

A pink to violet color develops after the brown color due to the addition of potassium thiocyanate disappears if molybdenum is present. The intensity of the color is proportional to the molybdenum content. Metals containing one percent and three percent molybdenum, respectively, are easily differentiated by this method.

Method 2—

- (a.) Allow two or three drops of the solution 25 percent nitric acid plus five percent hydrofluoric acid plus 70 percent water (39) to react on the surface of the metal for one to two minutes.
- (b.) Place one drop of 6 *M* hydrochloric acid (25) on a filter paper disc and add one drop of the acid test solution transferred from the metal to the center of the wetted fleck.
- (c.) Add one drop of ten percent potassium thiocyanate (47) to the center of the fleck and one drop of 25 percent sodium thiosulfate (63).

A pink to violet color develops in the center of the fleck extending outward if molybdenum is present (see Ref 4).

3. *Tin*

- (a.) Transfer ten to twelve drops of the acid solution formed by the reaction of fluoroboric acid (23) on the metal to a test tube.
- (b.) Add concentrated ammonium hydroxide (9) until just basic to litmus paper and 3 *M* hydrochloric acid (26) until just acid to litmus paper plus 0.5 ml in excess.

(c.) Add ten drops of 1 *M* thioacetamide (71) and dilute with water to five ml.

(d.) Heat in a boiling water bath for 15 minutes, centrifuge and add one ml of water to the residue, stir, centrifuge and discard the liquid.

(e.) Add two ml of 6 *M* sodium hydroxide (56) to the residue, heat in the water bath for three minutes, centrifuge and discard the residue.

(f.) Acidify the solution with concentrated hydrochloric acid (24) until faintly acid to litmus paper.

(g.) Add three drops of 1 *M* thioacetamide (71) and place in a boiling water bath for three minutes.

(h.) Cool, centrifuge, and discard the liquid.

(i.) Add one ml of 6 *M* hydrochloric acid (25) and warm in the water bath for ten minutes.

(j.) Add several pieces of magnesium metal (32) about 1/16-in. diameter and about 1/8-in. long or an equivalent in magnesium ribbon. After the magnesium metal has dissolved, centrifuge and transfer the clear liquid to a clean test tube.

(k.) Add an equal amount of water and five drops of mercuric chloride (31).

A white to grey precipitate confirms the presence of tin.

4. *Vanadium*

(a.) Place one drop of concentrated hydrofluoric acid (28) on the surface of the metal. After one minute, add three drops of 1:1 nitric acid (36). Allow about one minute to react and stir. If a brown color appears and remains, add an additional two drops of 1:1 nitric acid (36). A high vanadium content produces a bright blue-green color at this point.

(b.) Transfer two drops to a spot plate, add two drops of phosphoric acid (41) and one drop of a, a' dipyridyl reagent (21), and stir.

(c.) Add four drops of concentrated ammonium hydroxide (9), stir and add two drops of phosphoric acid (41) and stir.

A pink to red color, depending upon the vanadium content, identifies the presence of vanadium. Commercially pure titanium produces a white color.

5. *Zirconium*

(a.) Place several drops of concentrated hydrochloric acid (24) on the clean metal surface and heat gently for several minutes.

(b.) Place two drops of 2 *M* sodium hydroxide (58) on Whatman #3 (thick) qualitative filter paper and add one drop of the acid test solution taken off the surface of the metal.

(c.) Add one drop of alizarin solution (5) to the center of the fleck.

A red precipitate that is stable against 1 *M* hydrochloric acid (27) confirms the presence of zirconium.

Procedures for Determining Pure Metals**1. Cadmium**

(a.) Place one drop of concentrated nitric acid (35) on the clean metal surface and after one minute add one drop of 10 *M* sodium hydroxide (55). Allow one minute for reaction.

(b.) Add one drop of ammonium sulfide (13) and observe.
A deep orange precipitate confirms cadmium.

2. Indium

(a.) Place one drop of concentrated nitric acid (35) on the clean metal surface and after one minute add three drops of 6 *M* acetic acid (2).

(b.) Impregnate a filter paper disc with alcoholic alizarin reagent (6) and dry.

(c.) Transfer one drop of the acid test solution to the filter paper and hold the paper over a few drops of concentrated ammonium hydroxide (9) placed in a watch glass or small beaker.

(d.) Immerse the filter paper in aqueous boric acid (16).
A red to violet ring confirms indium (see Ref 4).

3. Lead

(a.) Place one drop of concentrated nitric acid (35) on the clean metal surface and after one minute add one drop of 6 *M* acetic acid (2).

(b.) Add two drops of 0.5 *M* potassium chromate (42).
A canary yellow precipitate confirms lead.

4. Molybdenum

(a.) Place one drop of 1:1 nitric acid (36) on the clean metal surface and after five minutes add one drop of concentrated hydrochloric acid (24).

(b.) Transfer one drop of the acid test solution to a spot plate and add one drop of ten percent potassium thiocyanate (47).

(c.) Add one drop of 25 percent sodium thiosulfate (63) and stir.
A violet color confirms molybdenum.

5. Silver

(a.) Place three drops of 1:1 nitric acid (36) on the metal and after one minute transfer one drop to a spot plate depression.

(b.) Add one drop of 3 *M* hydrochloric acid (26). A white precipitate indicates silver.

(c.) Add four drops of concentrated ammonium hydroxide (9) and stir.
If the white precipitate dissolves, silver is confirmed.

6. Tantalum

Place one drop of a solution of nitric acid, hydrofluoric acid, and sulfuric acid reagent (40) on the clean metal. After three minutes, add one drop of

rhodamine B solution (49), stir and observe.

A blue precipitate confirms tantalum (see Ref 7).

7. *Tin*

(a.) Place several drops of concentrated nitric acid (35) on the metal and allow to react for two or three minutes. A white precipitate indicates tin. Transfer two drops to a black spot plate.

(b.) Add five drops of concentrated hydrochloric acid (24) and a piece of magnesium metal (32) about 1/16-in. in diameter by 1/4-in. in length. Allow time for the magnesium metal to dissolve.

(c.) Add ten drops of water and three drops of mercuric chloride (31).

A white precipitate that turns grey to black upon standing confirms tin.

8. *Tungsten*

(a.) Place one drop of the solution containing nitric acid, hydrofluoric acid, and sulfuric acid (40) on the metal. Allow five minutes to react.

(b.) Add five drops of 20 percent sodium peroxide (60) to make strongly alkaline and to form tungstate if tungsten is present. Stir and after two minutes, transfer one drop to a thick filter paper disc (Whatman #3 or equivalent) previously moistened with three drops of concentrated hydrochloric acid (24).

(c.) Add one drop of stannous chloride reagent (65) and one drop of concentrated hydrochloric acid (24).

A blue color confirms tungsten (see Ref 4).

9. *Vanadium*

(a.) Place one drop of concentrated nitric acid (35) on the metal. A vigorous reaction producing a brown precipitate indicates vanadium.

(b.) Add two drops of 1:1 sulfuric acid (68) and one drop of three percent hydrogen peroxide (29).

A red-brown color confirms vanadium.

10. *Zinc*

(a.) Place three drops of 6 *M* hydrochloric acid (25) on the clean metal. After two minutes, add three drops of water and stir. Transfer three drops to a spot plate depression.

(b.) Add three drops of cobaltous sulfate (18) and three drops of alkali mercuric thiocyanate solution (33). Stir with a glass rod for 15 seconds rubbing the sides of the container with the glass rod.

A blue precipitate confirms zinc (see Ref 4).

Concluding Remarks

A system of identification of metals and alloys employing chemical spot test techniques suitable for shop and laboratory use has been developed by the author. No special training in the field of chemistry is necessary to follow

the detailed procedures. Conclusions relative to identification are derived from unique reactions or the colors produced by the addition of reagents. Tests are conducted on the metal's surface, in a spot plate depression, or on qualitative filter paper. All tests are qualitative; however, many of the tests can be employed semiquantitatively. For semiquantitative conclusions, simultaneous tests are made with specimen having known chemical compositions similar to the metal suspected. Results are compared relative to the quantity of precipitates produced or the intensities of the color reactions. The tests are nondestructive in that material affected is equivalent to the stroke of a smooth file or cleaning with an abrasive cloth.

The procedures cover aluminum and aluminum alloys, copper and copper alloys, magnesium and magnesium alloys, nickel and nickel alloys, stainless and heat resisting steels, carbon and low alloy steels, tool steels, titanium and titanium alloys, and some pure metals. The procedures are in chart form for rapidly reducing the area of possibilities. Detailed steps containing the number of drops of each chemical used and the time to allow for each reaction are listed on the charts. Stepwise, procedures for the identification of the major constituent alloys in each family of metals are also included in this system.

An example of the simplicity of this system is the separation and identification of stainless steels, Types 301, 302, and 304. One drop of 48 percent hydrofluoric acid is placed on the clean metal surface and allowed to react until dry, usually 15 to 20 minutes is required; however, gentle heat can be applied to increase the rate of reaction. The amount of green-white precipitate produced on the metal surface is distinct in the three metals. Type 301 produces the greatest quantity, Type 302 is second, and Type 304 produces the least. It is recommended that the analyst maintain a file of sample metals and alloys that he expects to encounter for simultaneous testing to compare the unknown metal with a known metal.

APPENDIX A

Apparatus

60 milliliter dropping bottles	Cotton, aseptic, absorbent
Spot plates, white and black	Crucibles, 30 ml, porcelain
Hot plate	Funnels
Test tubes, 10 ml	Forceps
Beakers, 400 ml, 100 ml, and 50 ml	Cylinders, graduated
Centrifuge	Litmus, pink and blue
Balance	pHdriion paper
Filter paper circles, 4.25 centimeter recommended, Whatman #1, #2, and #3 qualitative or the equivalent	Metals and alloys sample specimen file
Magnet	Chemical Dictionary should be available
Glass rods	Periodic table of the elements
Dropping pipets (medicine droppers)	Radiological survey meter
	Ultraviolet lamp
	Microscope
	Hardness tester

APPENDIX B

Reagents—Wet and Dry

(All reagents must be prepared with distilled water)

1. Acetic acid, concentrated	sp gr 1.05
2. Acetic acid, 6 <i>M</i>	Add 20.6 ml of concentrated acetic acid to 39.4 ml of water.
3. Acetic acid, 1 <i>M</i>	Add 3.5 ml of concentrated acetic acid to 56.5 ml of water.
4. Acetone	sp gr 0.80
5. Alizarin S, 0.1 percent	Dissolve 0.06 of Alizarin S in 60 ml water.
6. Alizarin	Prepare a saturated solution of alcoholic alizarin by adding alizarin to 60 ml of ethyl alcohol with stirring until an excess settles on the bottom.
7. Aluminon reagent, 0.1 percent	Dissolve 0.06 of aluminon in 60 ml water.
8. Ammonium acetate, 3 <i>M</i>	Dissolve 13.9 g of ammonium acetate in 60 ml water.
9. Ammonium hydroxide, concentrated	sp gr 0.90
10. Ammonium hydroxide, 6 <i>M</i>	Dilute 24 ml of concentrated ammonium hydroxide to 60 ml
11. Ammonium hydroxide, 3 <i>M</i>	Dilute 12 ml of concentrated ammonium hydroxide to 60 ml
12. Ammonium hydroxide, 1 <i>M</i>	Dilute 4 ml of concentrated ammonium hydroxide to 60 ml
13. Ammonium sulfide	22 percent aqueous solution.
14. Aqua regia	Mix 3 volumes of concentrated hydrochloric acid with 1 volume concentrated nitric acid. Prepare as needed.

Appendix B (continued).

15. Barium chloride, 0.3 *M* Dissolve 4.5 g of barium chloride in 60 ml of water.
16. Boric acid Prepare a saturated solution in 60 ml of water.
17. Chromotropic acid Dry powder
18. Cobaltous sulfate Dissolve 0.012 g of cobaltous sulfate in 2.5 ml of concentrated hydrochloric acid and dilute to 60 ml
19. Dimethylglyoxime Prepare a saturated solution of dimethylglyoxime in 60 ml of 95 percent ethyl alcohol.
20. Diphenylcarbazide Dissolve 0.6 g of diphenylcarbazide in 60 ml of 95 percent ethyl alcohol.
21. a, a' - dipyridyl Dissolve 1.2 g of a, a' dipyridyl in 60 ml 0.1 *M* hydrochloric acid.
22. Ferric chloride Dissolve 6 g of ferric chloride in 6 ml concentrated hydrochloric acid and dilute to 60 ml
23. Fluoroboric acid Dissolve 28 g of boric acid in 60 ml of 48 percent hydrofluoric acid. Store in a polyethylene bottle.
24. Hydrochloric acid, concentrated sp gr 1.19
25. Hydrochloric acid, 6 *M* Add 30 ml of concentrated hydrochloric acid to 30 ml water.
26. Hydrochloric acid, 3 *M* Add 15 ml of concentrated hydrochloric acid to 45 ml water.
27. Hydrochloric acid, 1 *M* Add 5 ml of concentrated hydrochloric acid to 55 ml water.
28. Hydrofluoric acid, concentrated 48 percent
29. Hydrogen peroxide 3 percent
30. Lead acetate, 5 percent Dissolve 3 g of lead acetate in 60 ml water.
31. Mercuric chloride Prepare a saturated solution by adding mercuric chloride with stirring to 60 ml water until an excess settles to the bottom.
32. Magnesium metal Magnesium ribbon or granules.
33. Mercuric thiocyanate, alkali solution Dissolve 5.82 g of mercuric chloride and 1.92 g of ammonium thiocyanate in 60 ml of water. Allow to stand for several days before using.
34. Morin, 0.02 percent Dissolve 0.012 g of morin in 60 ml of acetone.
35. Nitric acid, concentrated sp gr 1.42
36. Nitric acid, 1:1 Add 30 ml of concentrated nitric acid to 30 ml of water.
37. Nitric acid, 6 *M* Add 22.5 ml of concentrated nitric acid to 37.5 ml of water.
38. Nitric acid, 3 *M* Add 11.3 ml of concentrated nitric acid to 48.7 ml of water.
39. 25 percent nitric acid, 5 percent hydrofluoric acid, 70 percent water Add 15 ml of concentrated nitric acid and 3 ml of concentrated hydrofluoric acid to 42 ml of water. Store in a polyethylene bottle.

Appendix B (continued).

40. 1 nitric acid, 1 hydrofluoric acid,
1 sulfuric acid Mix 20 ml of concentrated nitric acid
with 20 ml of concentrated hydrofluoric
acid and 20 ml of concentrated sulfuric
acid.
41. Phosphoric acid, ortho, concen- 85 percent
trated
42. Potassium chromate, 0.5 *M* Dissolve 5.9 g of potassium chromate in
60 ml of water.
43. Potassium cyanide, 4 *M* Dissolve 15.6 g of potassium cyanide in
60 ml of water.
44. Potassium ethyl xanthogenate Dry crystals.
45. Potassium ferricyanide, 10 percent Dissolve 6 g of potassium ferricyanide
in 60 ml of water.
46. Potassium iodide, 1 percent Dissolve 0.6 g of potassium iodide in
60 ml of water.
47. Potassium thiocyanate, 10 percent Dissolve 6 g of potassium thiocyanate in
60 ml of water.
48. Quinalizarin Dissolve 0.012 g of quinalizarin in 60 ml
of 95 percent ethyl alcohol.
49. Rhodamine B Dissolve 0.006 g of rhodamine B in 60 ml
of water.
50. Sodium bismuthate Dry powder
51. Sodium bismuthate reagent Add 3.5 g of sodium bismuthate to 60 ml
of water, stir before using.
52. Sodium (Di) ethylenediamine Prepare a saturated solution of sodium
tetraacetate reagent (Di) ethylenediamine tetraacetate in 5 ml
of concentrated ammonium hydroxide
and 50 ml of water.
53. Sodium fluoride Dry powder
54. Sodium fluoride solution Prepare a saturated solution of sodium
fluoride in 60 ml of water.
55. Sodium hydroxide, 10 *M* Dissolve 80 g of sodium hydroxide in
200 ml of water.
56. Sodium hydroxide, 6 *M* Add 36 ml of 10 *M* sodium hydroxide to
24 ml of water.
57. Sodium hydroxide, 3 *M* Add 18 ml of 10 *M* sodium hydroxide to
42 ml of water.
58. Sodium hydroxide, 2 *M* Add 12 ml of 10 *M* sodium hydroxide to
48 ml of water.
59. Sodium hydroxide, 1 *M* Add 6 ml of 10 *M* sodium hydroxide to
54 ml of water.
60. Sodium peroxide, 20 percent Dissolve 12 g of sodium peroxide in 60
ml of water.
61. Sodium rhodizonate, 0.2 percent Dissolve 0.12 g of sodium rhodizonate
in 60 ml of water.
62. Sodium sulfate, saturated Prepare a saturated solution of sodium
sulfate in 60 ml of water.
63. Sodium thiosulfate, 25 percent Dissolve 15 g of sodium thiosulfate in
60 ml of water.
64. Stannite reagent Add two drops of Stannous reagent #66
to 3 ml of 3 *M* sodium hydroxide. This
solution should be clear and prepared
freshly as needed.

Appendix B (continued).

65. Stannous chloride, 20 percent Dissolve 12 g of Stannous chloride in 60 ml of water and add 0.5 g of metallic tin.
66. Stannous reagent Dissolve 7 g of Stannous chloride in 10 ml of concentrated hydrochloric acid, dilute to 60 ml and add 0.6 g of metallic tin.
67. Sulfuric acid, concentrated sp gr 1.84
68. Sulfuric acid, 1:1 Add 30 ml of concentrated sulfuric acid to 30 ml of water.
69. Sulfuric acid, 3 M Add 10 ml of concentrated sulfuric acid to 50 ml of water.
70. Tartaric acid and sodium bitartrate buffer, pH 2.8 Dissolve 0.9 g of tartaric acid and 1.14 g of sodium bitartrate in 60 ml of water.
71. Thioacetamide, 1 M Dissolve 4.5 g of thioacetamide in 60 ml of water.
72. Zinc oxide Dry powder

APPENDIX C

Chemical Compositions of Metals and Alloys.

Common Name of Metal or Alloy	Nominal Chemical Composition Percentage											
	Al	C	Cr	Cu	Fe	Mn	Mo	Ni	Ti	W	V	Others
Admiralty metal				70								Zn-29 Sn-1
Aluminum, 1100	99			0.20	0.5	0.05						Zn-0.1 Si-0.5
Aluminum, 2020	93.7			4.5	0.40	0.5			0.1			Zn-0.3 Si-0.4
Aluminum, 2014	92.7		0.1	4.5	1	0.9			0.15			Zn-0.3 Mg-0.5
Aluminum, 2017	93.9		0.1	3.5	1							Zn-0.3 Mg-0.4
Aluminum, 2024	92.5		0.1	4.4	0.5	0.5						Zn-0.3 Mg-1.5
Aluminum, 2117	94.7		0.10	2.5	1	0.2						Zn-0.25 Mg-0.4
Aluminum, 3003	98.2			0.2	0.7	1.2						Zn-0.1
Aluminum, 4043	94.5			0.3	0.8	0.05			0.2			Si-5.2
Aluminum, 5052	96.5		0.25	0.10	0.2	0.10						Mg-2.5
Aluminum, 5056	93.6		0.1	0.1	0.4	0.2						Mg-5.2
Aluminum, 5086	94.4		0.1	0.1	0.5	0.3			0.15			Zn-0.3 Mg-4
Aluminum, 6061	96.5		0.25	0.25	0.7	0.15			0.15			Si-0.5 Mg-1
Aluminum, 7075	88.5		0.3	1.5	0.7	0.3						Zn-5.5 Mg-2.5
Berylco 10				97.2								Be-0.6
Berylco 50				97.3								Co-2.6 Be-0.4
Brass, sheet QQ-B-613B				66	0.05							Co-1.6 Ag-1.1 Zn-34 P6-0.2

Appendix C (continued).

Common Name of Metal or Alloy	Nominal Chemical Composition Percentage											
	Al	C	Cr	Cu	Fe	Mn	Mo	Ni	Ti	W	V	Others
Brass, bar				62	0.35							Zn-35
QQ-B-626B												Pb-3
Brass, aluminum	2.25			76								Zn-22
Brass, Naval				60								Zn-39
												Sn-0.8
Brass, Red				85								Zn-15
Brass, yellow				65								Zn-35
Bronze, aluminum				92								Al-8
Bronze, leaded				88.5								Sn-10
												Pb-1.5
Bronze, Manganese				59	0.8	0.5						Zn-39
												Sn-0.75
Bronze, phosphor				95	0.10							P-0.2
												Sn-4.6
Bronze, silicon				96		1.1						Si-3.1
Bronze, Tin				89								Sn-11
Cadmium												Cd-99.9
Columbium (Niobium)												Nb-99
Copper, Arsenical				99.9								As-0.04
Copper, beryllium				97.4					Ni-0.3 or Co-0.3			Be-2.3
Copper, oxygen-free				99.9								
Copper, phosphor				99.9								P-0.02
Copper, tough-pitch				99.9								O-0.4
Hastelloy X		0.10	22		18	1	9	47		0.6		Co-1.5
Hypernom					15		4	80				
Inconel 600		0.08	14	0.2	6.5	0.25		78.5				Si-0.3
Inconel 625	0.4	0.1	21.5		5	0.5	9	61	0.4			Nb-3.7
												Co-1
Inconel 750	0.7	0.04	15	0.05	7	0.5		73	2.5			Nb-1
Indium												In-99.9
Lead												Pb-99.9
Magnesium, AZ31B	3					0.2						Zn-1
Magnesium, AZ63A	6					0.2						Mg-96
Magnesium, AZ92A	9			0.25		0.1		0.01				Zn-3
Magnesium, EZ33A												
												Zn-2
Magnesium, HK31A												R.E.-3
												Zn-2.7
												Zr-0.5
												Th-3.3
												Zr-0.2
												Th-2
Magnesium, HM21A						0.5						
Molybdenum							99.9					
Monel (400)		0.12		31.5	1.4	0.90		66				Si-0.2
Monel K (K-500)	3	0.25		27.6	2	1.5		65	0.6			Si-1

Appendix C (continued).

Common Name of Metal or Alloy	Nominal Chemical Composition Percentage											
	Al	C	Cr	Cu	Fe	Mn	Mo	Ni	Ti	W	V	Others
Nichrome V		0.25	20		1	2.5		78				Si-1.5
Nickel (200)		0.1		0.2	0.4	0.3		99				
Nickel D (211)		0.1		0.03	0.05	4.8		95				
Nickel TD								98				ThO ₂ -2
Rene 41	1.5	0.1	19		5	0.1	10	48	3.1			Co-11
Silver												AG-99.9
Silver solder				15				3				Zn-15
												CD-16
												Ag-50
Steel, low carbon		0.2			99							
Steel, high carbon		0.3 to 1.00			99.1	0.45						
Steel, 4130		0.3	0.95		97.7	0.5	0.20	0.12				
Steel, 4340		0.37	0.8		96.4	0.55	0.25	1.8				
Steel, 6150		0.48	0.95		97.8	0.70					0.15	
Steel, drill rod		1.00			98.3	0.25						Si-0.4
Steel, Brown & Sharpe		0.9	0.5		96.7	1.2				0.5	0.2	
Steel, spring, blue		1.0			98.5	0.28						Si-0.2
Steel, tool M-2		0.85	4.0		81.9		5.0			6.25	2.0	
Steel, tool, T-4		0.75	4.0		71.25					18	1.0	Co-5
Steel, tool, M-36		0.80	4.0		74		5.0			6.0	2.0	Co-8
Steel, tool, Ketos		0.90	0.50		96.9	1.25				0.50		
Steel, stain- less 301		0.15	17		72.8	2		7				
Steel, stain- less 302		0.15	18		69.8	2		9				
Steel, stainless 303		0.15	18		70	2		9				S-0.25 or Se-0.25
Steel, stain- less 304		0.08	19		68.8	2		9				
Steel, stain- less 310		0.25	25		53.2	2		20.5				Si-1.5
Steel, stain- less 316		0.08	17		65.3	2	2.5	12				
Steel, stain- less 321		0.08	18		71.5	2		10.5	0.4			
Steel, stain- less 347		0.08	18		67	2		11				Ta and Nb-0.8
21-6-9		0.08	20		63.3	9		6				N-0.2
AM 350		0.10	16.5		75.3	0.80	2.75	4.3				
17-4 PH		0.07	16.5	4.0	72.5	1.0		4.0				Ta and Nb-0.3
17-7 PH	1.0	0.09	17		72	1.0		7				
20-Cb		0.07	20	3	44.4		2	29				Ta and Nb-0.6

Appendix C (continued).

Common Name of Metal or Alloy	Nominal Chemical Composition Percentage											
	Al	C	Cr	Cu	Fe	Mn	Mo	Ni	Ti	W	V	Others
Steel, stain- less 405	0.20	0.08	13		84.3	1						
Steel, stain- less 410		0.15	12.5		86.3	1.0						
Steel, stain- less 416		0.15	13		84.4	1.25						S-0.15
Steel, stain- less 422		0.23	12		83.6	0.75	1	0.80		1	0.25	
Tantalum												Ta-99.9
Tin												Sn-99.8
Titanium									99.3			
Ti-4Al-3Mo-1V	4						3		92		1	
Ti-6Al-4V	6								90		4	
Ti-6Al-2Sn- 4Zr-2Mo	6						2		86			Zr-4 Sn-2
Ti-8Al-1Mo-1V	8						1		90		1	
Ti-13V-11Cr- 3Al	3		11						73		13	
Ti-16V-2½Al	2.5								81.5		16	
Tungsten										99.9		
Vanadium											99.9	
Zinc												Zn-99.9

APPENDIX D

Precautions and Notes

1. When mixing reagents safety rules in handling acids and bases should always be carefully followed.

(a.) Always add an acid or base to the water when mixing a solution which calls for diluting with water.

(b.) Aqua regia is unstable and should be mixed just prior to using.

(c.) Hydrofluoric acid and fluoroboric acid are *extremely* dangerous when splashed on the skin. *Always* wear rubber gloves, face mask, and apron when using these acids. If hydrofluoric acid should accidentally come in contact with skin, flush with running water for 15 minutes and report to a first aid station.

(d.) When mixing sodium peroxide reagent, allow it to stand for a few hours in a covered beaker before pouring into the dropping bottle.

2. A simple water bath can be constructed by covering a 400 ml beaker with aluminum foil and punching holes through the foil with a pencil to receive the test tubes.

3. A very handy method of separating a liquid from a solution in which an interfering constituent has been precipitated out of solution is the cotton-medicine dropper method described as follows. Roll a small amount of aseptic absorbent cotton into a ball and place it in the solution which is contained in a spot plate depression or some other suitable container. Place the tip of a medicine dropper in the cotton and draw the liquid through the cotton. With a little practice, this method will prove to be a rapid, clean, and reliable method of separation especially when only several drops of liquid are in the system to be separated from the solid.

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