

# FIELD TESTS FOR THE COMMON MINERAL ELEMENTS

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## FIELD TESTS FOR THE COMMON MINERAL ELEMENTS

### TESTING EQUIPMENT

Ultraviolet lamp (short wave black light)  
Blowpipe, 8 or 10 inches long  
Heating apparatus (candle, alcohol or lard-oil lamp, or portable propane torch)  
Charcoal sticks, 4 inches by 1 inch by  $\frac{3}{4}$  inch  
Hand lens  
Streak plate  
2 or 3-inch magnet or magnetized knife-blade  
Jackknife or micro-spatula  
Forceps (platinum tipped are the best)  
3 pieces of platinum wire (No. 26 wire gauge) and holder for the wire  
Nichrome wire, a few pieces 4 to 6 inches long. (Nos. 22 to 26 wire gauge.) Iron wire may be used in some tests.  
Pyrex test tubes (4 inches by  $\frac{1}{2}$  inch or 6 inches by  $\frac{3}{4}$  inch)  
Porcelain (china) cup  
Porcelain crucibles  
Soft glass tubing, 7 millimeters (for closed and open tubes)  
Book of litmus paper  
Window glass (a few pieces for fluorine test)  
2 glass cylinders, 10 milliliters  
1 graduated cylinder, 50 milliliters glass or polyethylene  
2 polyethylene washing bottles, 4 ounces or 125 milliliter capacity  
3 polyethylene bottles, 4 ounces or 125 milliliter capacity  
3 dropping bottles, polyethylene or glass, 1 ounce capacity  
Small beakers, 100 milliliter capacity  
Two 2-inch glass funnels  
1 package 4-inch filter papers  
Flame-color screen (Merwin's)  
3 candles (for fluorine test)  
4 plastic vials, 1 or 2 ounce, wide mouth  
1 white porcelain spot plate (for beryllium test)

## DRY REAGENTS

- ¼ ounce powdered borax (borax glass is better than ordinary borax)
- 1 ounce powdered sodium carbonate (baking soda may be substituted, except in tests for beryllium and for mercury)
- ¼ ounce powdered salt of phosphorus (sodium metaphosphate)
- 4 ounces zinc metal (mossy or granulated, 20 mesh or finer)
- 1 ounce zinc dust
- 2 ounces tin (shavings or filings)
- 1 ounce powdered manganese dioxide
- ¼ ounce sodium acid phosphate (for magnesium test)
- ¼ ounce diphenylamine crystals (for nitrate test)
- 1 ounce sodium bismuthate (for manganese test)
- 2 ounces sodium or potassium ethyl xanthate (for molybdenum test)
- 2 ounces potassium or sodium hydroxide (pellets)
- 1 ounce potassium bisulfate (acid sulfate of potassium)
- ¼ ounce ammonium oxalate (for calcium test)
- ¼ ounce ammonium molybdate (for phosphate test)
- ¼ ounce ferrous sulfate (for gold test)
- ¼ ounce stannous chloride (for gold test)
- 250 grams quinizarin (1-4 dihydroxyanthraquinone, technical grade) (used for beryllium test only)

## WET REAGENTS

- 4 ounces concentrated hydrochloric (muriatic) acid
  - 4 ounces concentrated nitric acid
  - 2 ounces concentrated sulfuric acid (oil of vitriol)
  - 8 ounces concentrated ammonia
  - 3 ounces denatured alcohol
  - ¼ ounce 10 percent cobalt nitrate solution
  - 1 ounce dimethylglyoxime solution (for nickel test)
  - 4 ounces mercury (quicksilver)
  - 1 ounce hydrogen peroxide
  - 1 ounce ether or chloroform (for the petroleum test)
- Note: Many common reagents cannot be purchased in less than 4 ounce quantities.

## OTHER USEFUL TOOLS AND SUPPLIES

- 1½-inch agate mortar and pestle (a black iron pipe cap can be used as a mortar)
- Gold pan, horn spoon, or frying pan for panning
- File (4-inch triangular)
- Hammer
- Anvil (block of steel 1½ inches by 1½ inches by ½ inch is convenient)

## BLOWPIPE OPERATIONS

G. M. Butler's *Handbook of Blowpipe Analysis* (2)\* gives the following explanations of the flames made with the aid of the blowpipe and the directions for producing them:

The blowpipe is used for the purpose of concentrating the flame into a long slender cone which can be readily directed against the substance to be heated. It is important that the blast be continuous and uniform, although this operation may seem very difficult at first. The blast is not produced by the lungs, but results from a bellows-like action of the distended cheeks. During the operation, air is inhaled only through the nose, and is exhaled largely through the mouth and blowpipe. Before trying to use that instrument, distend the cheeks, and keeping the mouth closed, breathe through the nose for a moment; then open the lips just enough to allow a little air to escape slowly, and admit air from the lungs by a kind of gulping action just fast enough to keep the cheeks fully distended. This may take some practice, but, when it is possible to allow the air to escape continuously from the mouth in this way, no matter whether it is being exhaled or inhaled through the nostrils, it is time to begin to use the blowpipe.

*Producing the Oxidizing Flame.* Place the oil lamp so that the longer dimension of the wick is from right to left, and set its right-hand edge upon a pencil or some other low support so that it will tip somewhat to the left. Insert the tip of the blowpipe about one-eighth of an inch within and just above the right-hand side of the wick. Blow steadily parallel to the wick, directing the flame to the left, producing a clear, blue flame about an inch long. If all of the flame cannot be thus diverted to the left, or if there are yellow streaks in the flame, trim or lower the wick. If the whole flame is inclined to be yellow, move the tip of the blowpipe a trifle to the left. If it is impossible to produce a flame approaching the length mentioned above, the opening in the end of the blowpipe is too small; when a very long, hissing flame is produced, this opening is too large. To succeed in blowing a steady flame, the hand must rest upon some support, or the third and fourth fingers may be placed against the lamp.

In analytical operations, it is sometimes desirable to oxidize substances to be tested, and at other times the aim is to reduce them to the metallic condition; either result can be more or less readily obtained with the blowpipe.

A flame produced in the manner above described is called an *oxidizing flame*, but the action of all portions of such a flame is not oxidizing. The blue cone contains considerable carbon monoxide and is feebly reducing in its action, but just outside of the blue cone at the tip of the flame is an extremely hot, but nearly colorless, zone which is strongly oxidizing because of the free oxygen there present, and anything held in this zone about an eighth of an inch from the tip of the blue flame will be in most favorable position for oxidation.

The oxidizing flame is hotter than the reducing, and the hottest part of this flame is just outside of the blue cone. In the absence of other instructions, substances should always be heated there.

*Producing the Reducing Flame.* Hold the tip of the blowpipe about one-sixteenth of an inch above and to the right of the wick, and a long, yellow flame containing much unconsumed carbon will be produced. This is sometimes called the smoky, reducing flame. Where greater heat is required, the inner cone of the oxidizing flame should be used. The strongest reducing action will take place at the tip of, and within the yellow cone of the reducing flame.

Note — *One of the best kinds of lamps for this work is one burning a mixture of one third kerosene and two thirds lard oil, but the flame from a candle, an alcohol or any other kind of lamp, or from a Bunsen burner may be used.*

\* Bold face numbers in parentheses refer to literature cited in Bibliography, page 43.

### SAFETY PRECAUTIONS

1. Keep all chemicals in a safe place and out of reach of children.
2. Sodium or potassium hydroxide, solid or solution, coming into contact with the eye may cause permanent injury. Also, other hydroxides can produce severe burns to the flesh. In the event of contact with the eye or flesh, immediately flood infected area with water.
3. Wear safety glasses or plastic shield when using caustic or acids.
4. Acids, especially sulfuric and nitric acids, can produce burns on contact with flesh. Strong acids will eat holes in the clothing. Flood area with water.
5. Sulfuric acid when mixed with water produces heat. To make dilute sulfuric acid, always add the acid to the water, a drop or so at a time. Never add the water to the acid, since the heat generated may cause an explosion.
6. Strong ammonium hydroxide should not be used to neutralize strong acid. The acid solutions should be diluted with water before strong ammonium hydroxide is added.

## TESTS FOR ELEMENTS AND MINERALS

### ANTIMONY

The most important antimony mineral of commerce is stibnite (antimonite, antimony glance, gray antimony, or antimony sulfide).

*Beginners practicing the following tests should use antimony sulfide.*

1. Antimony can usually be detected by the dense, white fumes given off and the heavy, white coating formed near the mineral when it is heated on charcoal with the blowpipe.

**To make this test:** Transfer to a flat stick of charcoal a little of the mineral to be tested for antimony. Use about the amount of powdered mineral that can be held on the tip of a knife blade or a piece of the mineral about as large as a grain of wheat. Heat the mineral in the oxidizing (bluish) flame of the blowpipe until the material is thoroughly fused. By this treatment, most antimony minerals give off dense, white fumes which often continue to rise even after the heating has ceased. These fumes deposit as a dense, white sublimate (coating) on the charcoal near the mineral; the outer edges, where the coating is thin, appear bluish-white. The bluish-white coating of antimony must not be confused with those of lead or zinc which are very similar on the outer edges.

Lead and zinc coatings can easily be identified; the coating deposited on the charcoal from lead is yellow near the assay when hot or cold. The coating from zinc deposited on the charcoal near the assay is yellow when hot, and white when cold.

2. Sulfides of antimony when heated in a closed tube yield a sublimate which is black when hot and reddish brown when cold.

**To make this test:** Fill a closed tube to about  $\frac{1}{2}$  inch from the bottom end with the powdered mineral. Heat the lower portion of the closed tube to a red heat and maintain for several minutes. Antimony sulfides will coat the walls of the tube with a sublimate. This coating is black when hot, but on cooling changes to reddish brown.

3. Stibnite (antimony sulfide) may be determined by the orange cloudy solution produced with caustic and dilute hydrochloric acid.

**To make this test:** Place a drop of 5 percent sodium or potassium hydroxide on the mineral, let stand one minute, then add a drop or two of dilute hydrochloric acid (1 part hydrochloric acid to 7 parts of water). If the mineral is stibnite, the spot will immediately turn orange. If a yellow spot appears, see test 5 for arsenic.

## ARSENIC

Nearly all of the arsenic sold in the United States is obtained as a by-product from the fumes given off when smelting other ores.

1. When struck a glancing blow with a hammer, many arsenic minerals give off sparks and a garlic-like odor.

*Beginners practicing this and the following tests should use arsenopyrite.*

2. Arsenides, sulfides of arsenic, and native arsenic give off a garlic-like odor when heated on charcoal with the blowpipe. A white coating forms on the charcoal at a distance from the mineral.

**To make this test:** Transfer to a flat stick of charcoal a little of the mineral to be tested for arsenic. Use about the amount of the powdered mineral that can be held on the tip of a knife blade or a piece of the mineral about as large as a grain of wheat. Heat the mineral with the blowpipe in the reducing (yellow) flame.

3. Arsenic and some arsenides when heated in a closed tube with sodium carbonate yield a black sublimate.

*Beginners practicing this test should use only arsenic.*

**To make this test:** Mix thoroughly one part of the finely powdered mineral with three parts of powdered sodium carbonate (or baking soda). Place in a closed tube about ½ inch of this mixture. Heat the lower end of the tube to a red heat for several minutes. Arsenic and some arsenides when given this treatment yield a black, mirror-like sublimate (coating) on the walls of the tube.

4. Some orpiments (arsenic trisulfide) fluoresce (glow) green when exposed to strong ultraviolet rays (black light). Always check fluorescent substances by chemical and blowpipe tests.

5. Orpiment or realgar (arsenic sulfides) may be determined by a yellow cloudy spot produced with caustic and dilute hydrochloric acid.

**To make this test:** Place a drop of 5 percent sodium or potassium hydroxide on the mineral, let stand one minute, then add a drop or two of dilute hydrochloric acid (1 part hydrochloric acid to 7 parts of water). If a yellow spot appears on the mineral, it is orpiment or realgar. If an orange spot appears, the mineral is stibnite; see test 3 for antimony.

## ASBESTOS

Asbestos is a term applied to several minerals that have flexible, fibrous, and sometimes matted structures, are fireproof, and are more or less resistant to acid attack. Among such minerals are fibrous and sometimes matted serpentine (chrysotile) and various fibrous amphibole minerals (tremolite, actinolite, anthophyllite, crocidolite, and amosite). Chrysotile is the most important asbestos mineral produced in the United States.

Asbestos can usually be recognized by its incombustibility, flexible structure, and slow conductivity of heat.

*Beginners practicing this test should compare the combustibility (taking fire and burning) of cotton, wool, and asbestos.*

**To make this test:** Twist a few fibers of the materials into a string or yarn. Hold one end of the string or yarn in a flame. If the material is asbestos, it will not burn.

## BARIUM

Barite is the most common barium mineral. It is practically insoluble in acid but can be made soluble by fusion with sodium carbonate. With-erite, a rather rare mineral containing barium, however, is soluble in hydrochloric acid.

**To make this test:** Make a loop of platinum or nichrome wire about ⅛ inch in diameter. Heat the loop to redness in a flame and insert the hot loop in sodium carbonate and return the loop to the flame. Repeat several times until a good bead is formed in the loop. Detailed instructions for making bead tests can also be found under CHROMIUM, test 1.

Touch the hot bead to the pulverized sample to be tested and again heat until the sample is thoroughly melted to glass. Repeat several times. Put the wire with the bead into a test tube and add 5 milliliters (1 teaspoon) distilled water and boil for a few minutes. Pour off the solution but keep the bead which may appear to be partly dissolved in the test tube. Add 5 milliliters (1 teaspoon) of hydrochloric acid and boil gently until the bead is dissolved. Add a few drops of diluted sulphuric acid; if barium is present, a heavy white cloud will appear in the solution. Since strontium will give the same cloudy appearance, it is advisable to filter the solution through a small filter paper. With a wire loop, scrape off a little of the precipitate which has been moistened with a drop of hydrochloric acid and ignite in a hot flame. The flame will be yellowish green if barium is present. Strontium will give a crimson flame.

## BERYLLIUM

A simple field test for beryllium in rocks has been developed by the U.S. Bureau of Mines (4). A portion of pulverized rock is fused with sodium carbonate and sodium hydroxide flux in a nichrome or platinum wire loop. The fused slag is dissolved in water, a few drops of quinizarin solution are added, and the solution is observed under ultraviolet light.

**Preparations for this test:** 1. *Quinizarin solution:* A quarter-teaspoon of quinizarin powdered reagent is mixed in 25 milliliters of alcohol (ethyl or methyl) and allowed to stand for several hours and the solution is then filtered to remove the residue. The filtrate or clear liquid is diluted with 4 times the volume of alcohol. The solution should be kept in a dropping bottle and out of the sunlight or bright electric light.

2. *Flux:* Two parts of sodium hydroxide pellets and three parts of sodium carbonate are ground together. This should be done rapidly as the mixture picks up moisture. Keep the flux in a stoppered glass or polyethylene bottle.

3. *Heat source:* A propane burner will give better heat than an alcohol lamp or blowpipe for melting the bead.

*Preparation of the sample:* Proper selection of the sample is important because such a small amount of material is used for the test. The sample should be ground to the fineness of flour in a mortar or on an anvil with a hammer.

*Beginners practicing this test should use the mineral beryl.*

*To make this test:* Make a loop in the platinum or nichrome wire (22 to 26 gauge)  $\frac{1}{4}$  inch in diameter. Heat the loop to redness in a flame, then insert the hot loop into flux and return to the flame. Repeat the process until a bead is built up in the loop. Now touch the hot molten bead to the pulverized sample and fuse the adhering powder to the bead until the sample is dissolved or completely melted.

Insert the wire loop containing the bead into a test tube and add 5 to 10 milliliters (1 to 2 teaspoons) water. Warm the test tube to dissolve the bead. After the bead is dissolved, remove the wire and cool. Add about 10 drops of the dilute quinizarin solution. Shake the test tube; a lilac-colored solution should result.

Pour a small amount of the lilac-colored solution into one of the cavities of a white porcelain spot plate. Examine the solution in a darkened room under either a short or long wave-length ultraviolet light. A pink-to-orange fluorescence indicates the presence of beryllium. If a sample contains much iron the fluorescence will be diminished.

## BISMUTH

The most common minerals of bismuth are native bismuth, bismuthinite, bismite, and bismutite.

Bismuth can be detected by an iron nail test.

*Beginners practicing this test should use bismuthinite.*

*To make this test:* Place a little of the powdered mineral to be tested in a porcelain crucible or small beaker. Use about the equivalent to two grains of wheat. Add 10 drops of nitric acid and heat gently at first, and finally bake until dry. Cool and add 10 drops of water to the residue. After a half-minute immerse the head of a nail in the solution for a half-minute and observe the head. If it turns dark, bismuth is present. Bismite or bismutite, which are oxides and carbonates of bismuth, may be tested by moistening the mineral with dilute hydrochloric acid (1 to 7). After a half-minute the head of a nail immersed in the acid solution will darken if bismuth is present. Sometimes antimony will darken the nail.

## CALCIUM

The most important commercial calcium minerals are calcite (limestone, marble, chalk, and calcium carbonate), dolomite (brown spar or calcium-magnesium carbonate), gypsum (selenite, satin spar, rock gypsum, land plaster, and hydrous calcium sulfate), and fluorite (fluorspar and calcium fluoride).

1. Calcium can usually be detected by its precipitation as an oxalate. *Beginners should use limestone, marble, chalk, or some other easily soluble calcium mineral.*

*To make this test:* Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade or the equivalent to two grains of wheat. Pour into the receptacle 5 milliliters (1 teaspoon) of concentrated (strong) hydrochloric (muriatic) acid or about twice this amount of dilute (about equal parts of acid and water) hydrochloric acid. If barium or strontium is present in the mineral which is being tested, add to the hydrochloric acid solution potassium sulfate or some other alkali sulfate and boil for a few minutes. Filter off the residue and any precipitate that has been formed and proceed with the test.

This mixture either cold or heated (if the mineral contains calcium carbonate) will effervesce (boil; bubble) and give off a colorless, odorless gas (carbon dioxide) which will not support combustion. A lighted match or other flame if held in this gas will go out. After the effervescence has ceased, add to the liquid about 25 milliliters (5 teaspoons) of cold water. To this solution add ammonia until a white precipitate begins to form or until the solution smells of ammonia. **Caution!** Be sure to add the cold water before adding the ammonia; if ammonia is added before the cold water, you may have an explosion.

The next step is to add a few crystals of ammonium oxalate or 3 milliliters concentrated ammonium oxalate solution (ammonium oxalate crystals dissolved in water). When the ammonium oxalate is added, a white precipitate will be thrown down, if the mineral used contains an appreciable amount of calcium.

2. Sulfuric acid precipitates calcium as a sulfate in moderately concentrated solutions.

*Beginners should use limestone, marble, chalk, or some other easily soluble calcium mineral.*

*To make this test:* Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested: Use about the amount of powdered mineral that can be held on the tip of a knife blade or the equivalent of two grains of wheat. Pour into the receptacle about 5 milliliters (1 teaspoon) of concentrated hydrochloric acid. After all effervescence has ceased, add to this solution a few drops of dilute sulfuric acid

(oil of vitriol) (one volume of acid added to about four volumes of water). This precipitates the calcium as colorless, white crystals of calcium sulfate (gypsum), which is distinguished from the sulfates of barium and strontium in that it will dissolve in a solution of ammonium sulfate. Calcium sulfate is also soluble in hot water. If the solution is diluted with water to about ten times its original volume and warmed, the calcium sulfate will dissolve.

3. Volatile compounds of calcium treated with hydrochloric (muriatic) acid color a nonluminous flame yellowish red if heated therein. *Beginners should use limestone, marble, chalk, or some other volatile compound of calcium.*

To make this test: Wet one end of a piece of iron wire about 4 inches long in hydrochloric (muriatic) acid. Draw the wet end of the wire through some of the powdered mineral. Heat the mineral coated end of the wire in a flame. An alcohol lamp flame is very satisfactory for this purpose. As soon as the wire and mineral are red hot, the flame will be colored yellowish red, if the mineral contains an appreciable amount of volatile compounds of calcium and if no other element masks the calcium flame.

4. With a Merwin's flame-color screen: Follow the directions outlined in test 3 above, but look at the flame through the different sections of a Merwin's flame-color screen. Through section 1, calcium gives a flashy, greenish yellow; through section 2, a green; and through section 3, a faint crimson flame.

5. Some calcium minerals (some calcite, limestone, aragonite, gypsum, and others) fluoresce (glow) when exposed to strong ultraviolet light (black light). Red, yellow, orange, and blue are among the colors emitted by these minerals when activated by this light — best seen in the dark.

Always check fluorescent substances by chemical and blowpipe tests.

Calcium Carbonate (Limestone). Many mining men and prospectors think that a conclusive test for the determination of calcium carbonate (limestone) is that it effervesces (boils; bubbles) when moistened with hydrochloric (muriatic) acid. This effervescing merely indicates that the mineral is a carbonate, if it gives off a colorless, odorless gas which does not support combustion (a lighted match if held in this gas will go out). Dolomite (calcium-magnesium carbonate) will not effervesce if a large piece is used, but a fine powder will effervesce slowly.

Various metals occur as carbonates, a few of the commonest being lead, zinc, copper, and iron. From this statement, it is evident that this effervescence is not a conclusive test for the determination of calcium carbonate, since the sample may contain a carbonate of some other metal.

## CHLORINE

1. Chlorine can usually be detected by its precipitation as silver chloride.

*Beginners practicing these tests should use halite (sodium chloride, or common table salt) or sylvite (potassium chloride).*

To make this test: Place in a test tube or some other glass receptacle a little of the material to be tested. If the material is a mineral, use about as much of the powdered mineral as can be held on the tip of a knife blade, or the equivalent of two grains of wheat. If the material to be tested is a liquid, use about 5 milliliters (1 teaspoon). Pour into the receptacle 5 milliliters (1 teaspoon) of dilute nitric acid. Use distilled water to make dilute nitric acid: 1 part of nitric acid to 7 parts of distilled water. Heat this mixture to boiling and then cool it. When a small amount of silver nitrate is added to this cool solution a white precipitate will result if the material used in the test contains an appreciable amount of chlorine. If only a small amount of chlorine is in the material tested, the precipitate will give the solution a milky appearance; if considerable chlorine is present, the precipitate will look curdy. If exposed to the sunlight for a time, this white precipitate turns dark (from a violet to brown color). This precipitate (silver chloride) is also soluble in ammonia. To make this part of the test, pour into the receptacle an excess of ammonia (until the solution smells strong of ammonia). The ammonia will dissolve the silver chloride.

2. Chlorine can usually be detected by the fumes that are given off when a chloride is heated with potassium bisulfate and manganese dioxide. These fumes have a bleaching action.

To make this test: Mix thoroughly a little of the finely powdered mineral with an equal volume of manganese dioxide (pyrolusite, psilomelane, or wad) and about four volumes of powdered potassium bisulfate (acid sulfate of potassium). Put about ½ inch of this mixture into a small test tube. Heat the lower end of the tube at a red heat for several minutes. This mixture will then give off rusty-green fumes that have a strong pungent odor if the material used in the test contains an appreciable amount of chlorine. These rusty-green fumes, if chlorine gas, have a bleaching action. This can be tested by holding a strip of moistened litmus paper inside the tube in the fumes given off.

## CHROMIUM

The most important chromium mineral is chromite (chromic iron ore or ferrous metachromite).

*Beginners should use chromite for practice testing.*

1. Chromium can usually be detected by the colors it imparts to the fluxes.

To make this test: First, prepare a glassy bead. Use a piece of No. 26 platinum wire about 2 inches long. Fasten one end in a holder so that when the wire is heated it will not burn the fingers. Special holders can be purchased, but a cork or a piece of soft wood into which one end of the wire is inserted may be used for this purpose. Make a loop about  $\frac{1}{4}$  inch in diameter at the unattached end of the wire. This loop is easily made by winding the end of the wire around the point of a lead pencil. Heat this looped end in a flame until it is red hot. An alcohol lamp flame is very satisfactory for this work. Dip the red-hot loop into some powdered borax or salt of phosphorus, a little of which will adhere to the wire loop. Fuse the borax or salt of phosphorus adhering to the wire by holding it in the flame. Continue these operations until a clear, glassy bead that fills the loop in the wire is secured.

Touch the bead while it is red hot to a little of the very finely powdered mineral. If the bead made from the borax and a very little of a mineral containing chromium is heated in the oxidizing (bluish) flame of the blowpipe, the bead will be decidedly yellow while it is warm, changing to a yellowish-green color when cold. When more of the mineral is added, the colors are deeper, changing through reddish or yellow when warm to a fine green when cold. If this bead is heated in the reducing (yellow) flame of the blowpipe, the bead assumes a fine green color when cold, but shows none of the yellow or reddish tint which is so prominent in the warm bead after heating in the oxidizing flame.

If salt of phosphorus is used instead of borax for making the bead, and the operations outlined above for the borax bead test are followed, the color of the bead formed when the oxidizing flame of the blowpipe is employed is a dirty green when the bead is warm, which changes to a fine green when the bead is cold. If the reducing blowpipe flame is used, the colors are about the same as with the oxidizing flame.

The chromium bead tests must not be confused with those for vanadium, which in the reducing flame give almost identical reactions with the fluxes. Vanadium, however, in the oxidizing (bluish) flame differs in the salt of phosphorus bead test in that it yields a yellow bead, while this flux never yields other than a green bead with chromium.

2. Chromium can usually be detected by its yellow-colored sulfuric acid solution. This turns green on the addition of alcohol.

To make this test: Mix one part of powdered ore with ten parts of powdered manganese dioxide. Place a little of this mixture in a test tube or some other glass or porcelain receptacle. Use about three times the amount of this mixture that can be held on the tip of a knife blade or the equivalent of two grains of wheat. Pour into the receptacle 5 milliliters (1 teaspoon) of water and the same amount of concentrated (strong) sulfuric acid (oil of vitriol). Heat this solution to boiling and boil strongly

until dense white fumes are freely given off. Then cool the solution and, after it is cold, dilute it with 15 milliliters of cold water. Filter the solution and catch the filtrate (the clear liquid that passes through the filter paper) in a glass receptacle. This filtrate will be yellow if the mineral used in the test contains an appreciable amount of chromium.

Add 5 milliliters (1 teaspoon) of alcohol to this yellow colored solution and boil. The solution will slowly turn green if the mineral contains an appreciable amount of chromium.

#### COBALT

The most important cobalt minerals are smaltite (tin-white cobalt or cobalt-nickel arsenide) and cobaltite (cobalt arsenide-sulfide).

1. Cobalt can usually be detected by the blue color it imparts to the fluxes.

*Beginners practicing this and the following cobalt test should use roasted smaltite or cobaltite.*

To make this test: The sulfide or arsenide ore must be thoroughly roasted (pulverized and heated on charcoal at a red heat until sulfur or arsenic fumes are no longer noticeable) before using the cobalt mineral in the bead tests. Detailed instructions for making bead tests are given under CHROMIUM, test 1. If the beads made from either borax or salt of phosphorus and cobalt mineral are heated before the blowpipe in either the reducing (yellow) flame or the oxidizing (bluish) flame, the beads formed will be deep blue in color.

2. Cobalt compounds become magnetic when heated on charcoal before the blowpipe in the reducing flame.

To make this test: Mix thoroughly a little of the finely powdered mineral with about twice its volume of powdered sodium carbonate (or baking soda). Transfer to a stick of charcoal as much of this mixture as can be held on the tip of a knife blade. Heat strongly before the blowpipe in the reducing flame until the mixture is thoroughly fused. The resulting fused mass will contain a dark colored, more or less metallic button enclosed in the slag, and this button will be magnetic when cold if the mineral used contains cobalt.

*Metallic iron and nickel are also magnetic; therefore a magnetic button obtained from any mineral, the metallic contents of which are unknown, should be tested further by applying test 1 for cobalt.*

#### COPPER

Important copper minerals are native copper, chalcocite (copper glance, vitreous copper, or cuprous sulfide), chalcopyrite (copper pyrites, yellow copper ore, or sulfide of copper and iron), bornite (purple copper ore, variegated copper ore, horse-flesh ore, peacock copper, or sulfide of



copper and iron), malachite (green copper carbonate or basic carbonate of copper), azurite (blue copper carbonate or basic carbonate of copper), cuprite (ruby copper, red copper ore, or cuprous oxide), and copper-bearing pyrites.

1. Ammonia added to an acid solution of copper produces a blue coloration.

*Beginners practicing this test should use a high grade copper mineral.*

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade or the equivalent of two grains of wheat. Pour into the receptacle about 5 milliliters (1 teaspoon) of acid. Use concentrated (strong) nitric acid or a mixture of nitric and hydrochloric acid. Heat until copper has been dissolved and then add about 15 milliliters (3 teaspoons) of cold water. To this add an excess of ammonia. Never add ammonia to a hot acid solution because the heat may cause an explosion, and always point the mouth of the receptacle in a direction where no harm can result. When the ammonia is added to the acid solution, the color of the solution will turn blue if the mineral used in the test contains an appreciable amount of copper.

2. A clean iron nail immersed in a dilute acid solution of copper will become coated (plated) with a film of copper. This test is used to detect oxidized copper minerals.

*Beginners should use malachite in practicing this test.*

To make this test: The sample to be tested need not be ground. Place a few drops of 1 : 7 hydrochloric acid (1 part hydrochloric acid and 7 parts water) in one spot on the sample. Hold the head of an iron nail in the moistened area for 10 seconds or more. The head of the nail will be plated with copper if an oxidized copper mineral is present. It is sometimes necessary to use a magnifying glass to observe the nail if the amount of copper is very small.

3. Volatile copper compounds color a nonluminous flame.

*For this test beginners should use malachite (copper carbonate) or some other volatile copper mineral.*

To make this test: Use a piece of iron wire about 4 inches long. Do not use platinum wire since volatile copper compounds alloy with the platinum in the flame, and the platinum wire would then be useless for further copper flame tests. Do not use a piece of iron wire for more than one test.

Wet one end of the wire in water, and draw the wet end of the wire through the powdered mineral. Heat the end of the wire with the mineral on it in a flame. An alcohol lamp flame is satisfactory for this purpose. As soon as the wire and mineral are red hot, the flame will be greenish

if the mineral contains an appreciable amount of a volatile copper compound.

Pour a few drops of hydrochloric acid into a glass or cup, and dip the end of the wire into it. Then take the mineral on the wire, and again hold it in the flame. If the flame is azure-blue, the mineral contains an appreciable amount of a volatile copper compound. (Never dip a wire which has been used for this or other tests in the acid bottle, since any soluble mineral present will dissolve and the acid may be made valueless for further flame tests.)

4. With a Merwin's flame-color screen: Follow the directions outlined in "Copper" (3) but look at the flame through the different sections of a Merwin's flame-color-screen. Through section 1, copper gives a bright green; through section 3, a bright blue flame fringed with green; and through section 2, the same tints, but paler.

5. Copper compounds fused with soda in the reducing flame yield a mass of metallic copper.

*For this test beginners should use chalcopyrite, cuprite, or some other easily fusible copper mineral.*

To make this test: Mix thoroughly a little of the finely powdered mineral with about twice its volume of powdered flux, sodium carbonate (or baking soda) — alone or mixed with a little borax. Transfer to a stick of charcoal as much of this mixture as can be held on the tip of a knife blade. Heat this mixture strongly before the blowpipe in the reducing (yellow) flame until it is thoroughly fused. The resulting fused mass will contain an irregular, spongy mass of metallic copper if the mineral used in the test contains an appreciable amount of copper.

6. Copper can usually be detected by the colors it imparts to the fluxes. Borax and salt of phosphorus beads are green when warm and blue when cold after having been heated in the oxidizing (bluish) flame. Saturated beads are opaque red in the reducing flame of the blowpipe. In the presence of much iron, the oxidizing flame bead is green or bluish green. Bead test instructions can be found under CHROMIUM, test 1.

## FLUORINE

The most important fluorine minerals are fluorite (fluorspar or calcium fluoride) and fluorapatite.

*Beginners practicing this test should use fluorite.*

1a. Some compounds of fluorine with sulfuric acid etch glass.

To make this test: Take a flat piece of window glass and coat a section of one side of the glass with a thin, even layer of paraffin wax. Wax melted and dropped from a burning paraffin candle can be used for this purpose. When the wax is cold write or mark through the paraffin to the glass. Use a pencil point, a sharpened piece of wood, or some other

tool that will cut through the paraffin, but will not scratch the glass. Pour into the marks made in the paraffin some of the finely powdered mineral that is being tested for fluorine. Onto this powdered mineral pour a few drops of concentrated (strong) sulfuric acid (oil of vitriol) and mix the mineral and acid together to the consistency of a thick paste. Use a pointed stick or pencil point for this purpose. In about 10 minutes wash off the mixture of acid and mineral, remove the paraffin by heating the glass, and clean the glass. If the glass was etched fluorine was present in the mineral used.

1b. Some compounds of fluorine with potassium bisulfate etch glass.

To make this test: Mix thoroughly one part of the finely powdered mineral with about three parts of potassium bisulfate (acid sulfate of potassium). Fill a closed tube or a small test tube to about  $\frac{1}{2}$  inch from the bottom end with this mixture. Heat the lower end of the tube at a red heat for several minutes. Etching (roughening or clouding) of the sides of the tube, just above the charge, indicates that fluorine is present in the mineral tested. The etching may be detected by breaking the tube, washing a fragment thoroughly, and rubbing the surface with the sharp point of a knife blade. The glass will feel slightly rough if it has been etched.

2. Some varieties of fluorite become phosphorescent when heated.

*Use violet-colored fluorite for practice testing.*

To make this test: Place in a test tube a few fragments of the mineral to be tested. The fragments should be about  $\frac{1}{4}$  inch in size. Heat the lower portion of the tube at a red heat, for a very short time. If heated too long the glowing will disappear. Some varieties of fluorite, when thus treated become phosphorescent (glow and emit light of various tints). This must be observed in the dark.

The above test can also be applied to a splinter of the material held in a pair of forceps.

3. Some fluorites fluoresce (glow) a blue or green color when exposed to strong ultraviolet light (black light).

Always check fluorescent substances by chemical and blowpipe tests.

#### GOLD

The principal gold minerals are native gold, sylvanite (gold and silver telluride), and calaverite (gold telluride).

*Beginners practicing these tests should use a piece of metallic gold or concentrates from a gold ore.*

1. The following physical properties are characteristics of metallic gold: Yellow-color; high specific gravity (nearly twice as heavy as silver); high melting point (1945° Fahrenheit); malleability, ductility, sectility, and softness (gold can be flattened if hammered on an anvil and a knife

blade, needle, or similar tool cuts and indents gold easily without crushing, cracking, or breaking it, as with metallic lead).

2. When particles of mercury-coated gold come in contact with each other, they become loosely cemented or soldered together. The resulting mass or paste is gold amalgam. Amalgamation, as used in this test, is based upon the fact that when clean, bright gold is brought into contact with clean, bright mercury, especially by a rubbing or grinding action, the mercury sticks to, coats, and catches the gold.

If the mercury is dark or tarnished, the gold, no matter how bright and clean will not be caught by or unite with the mercury. Neither will the union take place if the gold is rusty or dirty, even though the mercury is bright and clean. Both the gold and the mercury must be bright and clean to unite. Grinding the mixture in a dilute nitric acid solution will sometimes help brighten the mercury and the gold.

If gold amalgam is heated before the blowpipe on charcoal, the mercury will be distilled and leave the gold as a residue. If this residue, mixed with a little powdered borax, is then heated before the blowpipe on charcoal, there will be obtained a malleable, brass-yellow button which can be tested as explained in test 1 above.

3. Gold can usually be detected in free-milling ores, sand, and gravel, by panning.

With many complex, refractory (rebellious) ores, panning does not give satisfactory results. For such, a preliminary roasting of the ore often overcomes the difficulty.

4. In nitrohydrochloric acid solutions of gold, either metallic tin or stannous chloride give a purple precipitate and ferrous sulfate gives a brown precipitate. These salts are rather unstable; if stannous chloride changes to stannic chloride or ferrous sulfate changes to ferric sulfate, they are worthless for this test.

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the powdered mineral to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade or the equivalent of two grains of wheat. Pour into the receptacle about 5 milliliters (1 teaspoon) of concentrated (strong) nitric acid and 20 milliliters (4 teaspoons) of concentrated hydrochloric (muriatic) acid. Heat this solution to boiling and boil until any gold present has been dissolved. To half of this solution add a pinch of stannous chloride or a pinch of metallic tin filings. An excess of either will turn the solution a deep purple color if the mineral used in the test contains an appreciable amount of gold. On exposure to air the purple solution turns yellow. This test is known as the "Purple of Cassius Test for Gold."

To the remaining nitrohydrochloric acid solution add a pinch of ferrous sulfate. A brown precipitate indicates the presence of gold.

When the above tests do not indicate the presence of gold but the operator still believes that the mineral contains gold, the policy recommended for testing such a mineral is to have it assayed for gold. If an assay does not reveal the presence of gold in a mineral, it can be safely assumed that gold is not present in that mineral in commercial quantity.

## IRON

Important iron minerals of commerce are hematite (red ocher, red oxide of iron, specular iron, iron glance, ferric oxide, or iron sesquioxide), limonite (brown oxide of iron, brown ocher, brown hematite, or bog iron ore), and magnetite (black iron oxide, magnetic iron ore, or lodestone). *Beginners practicing these tests for iron should use hematite, pyrites, or some other iron mineral.*

1. Iron minerals roasted on charcoal yield a magnetic residue.

**To make this test:** Place on a stick of charcoal a little of the finely powdered mineral to be tested. Use about the amount that can be held on the tip of a knife blade. Heat before the blowpipe in the reducing (yellow) flame. If the mineral thus heated *without a flux* does not fuse (melt), but becomes magnetic, it contains iron. If it fuses and becomes magnetic it may contain iron, cobalt, or nickel.

2. Iron minerals fused with soda in the reducing flame yield a magnetic button.

**To make this test:** Mix thoroughly a little of the finely powdered mineral with about twice its volume of sodium carbonate (or baking soda). Transfer to a stick of charcoal as much of this mixture as can be held on the tip of a knife blade. Heat strongly before the blowpipe in the reducing flame until it is thoroughly fused. The resulting fused mass will contain a dark-colored more or less metallic button which is magnetic when cold, providing the mineral used in the test contains an appreciable amount of iron. Cobalt and nickel buttons produced in this manner are also magnetic. For this reason the magnetic button should be further tested for iron, cobalt, and nickel by the tests given for these metals.

3. Ammonia added to an acid solution of iron throws down a brownish-red precipitate.

**To make this test:** Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade or the equivalent of two grains of wheat. Pour into the receptacle about 5 milliliters concentrated (strong) hydrochloric (muriatic) acid and a few drops of nitric acid. Heat this solution until the iron has been dissolved, and then add about 10 milliliters (2 teaspoons) of cold water. To this solution add an excess of ammonia or until the solution smells strongly of ammonia. Be sure the solution is cold before adding the ammonia. Upon the

addition of the ammonia to this acid solution there will be thrown down a brownish-red precipitate if the mineral used in the test contains an appreciable amount of iron.

Many iron minerals are practically insoluble in the acids just mentioned. These minerals, if roasted or treated as described in IRON, test 2, become easily soluble.

4. Iron can usually be detected by the colors it imparts to the fluxes. In the oxidizing (bluish) flame of the blowpipe, the borax bead of iron is amber-colored when warm and yellow to colorless when cold. In the reducing flame the borax bead is bottle-green, providing the bead is saturated. Bead test instructions are given under CHROMIUM.

5. Some limonites and some siderites (iron carbonate) fluoresce (glow) green when exposed to strong ultraviolet rays (black light).

Always check fluorescent substances by chemical and blowpipe tests.

## LEAD

The most important lead minerals of commerce are galena (galenite, steel galena, potter's ore, or lead sulfide), cerussite (lead carbonate or white lead ore), and anglesite (lead sulfate).

*Beginners practicing this test should use galena, cerussite, or some other high-grade lead mineral or some small cuttings of metallic lead.*

1. Lead can usually be detected by the yellow sublimate and the metallic lead button formed on charcoal.

**To make this test:** Mix thoroughly a little of the finely powdered mineral with an equal volume of powdered charcoal and three volumes of powdered sodium carbonate (or baking soda). Moisten this mixture with water and transfer about the amount that can be held on the tip of a knife blade to a flat piece of charcoal or into a shallow cavity that has been made in the charcoal. Heat this before the blowpipe in a moderately strong, reducing (yellow) flame. This treatment, if the mineral used in the test contains an appreciable amount of lead, will produce small globules or buttons of metallic lead, which are soft and malleable (can be flattened out if hammered on an anvil), and also a yellowish sublimate (coating) on the charcoal, close to the mineral. This sublimate is whitish on the outer edges; the white portion should not be confused with the antimony or zinc sublimate.

2. Hydrochloric or sulfuric acid throws down a heavy, white precipitate in cold, nitric acid solutions of lead.

*Cerussite, or some other easily soluble, high-grade lead mineral, the lead buttons produced in LEAD, test 1, or some cuttings of metallic lead should be used by beginners.*

**To make this test:** Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount

of the powdered mineral that can be held on the tip of a knife blade. Pour into the receptacle about 5 milliliters (1 teaspoon) of concentrated (strong) nitric acid and about 10 milliliters (2 teaspoons) of water. Boil this solution until the lead has been dissolved, and then cool to room temperature. When cold, add a few drops of dilute sulfuric acid (oil of vitriol) or a few drops of hydrochloric (muriatic) acid. If the mineral used in the test contains an appreciable amount of lead, a white, heavy precipitate of lead will appear. This white precipitate should be further tested for lead by using test 1, above. When hydrochloric acid is used in the above test, silver and mercury, if present, will also be thrown down as a white precipitate, but silver in this form (silver chloride) turns dark if exposed to sunlight and is also very soluble in ammonia. The white mercury precipitate thus obtained usually turns dark grayish on the addition of an excess of ammonia.

The white precipitate (lead chloride) formed when hydrochloric acid is added to a nitric acid solution of lead can be tested further as follows:

3. Lead chloride is quite soluble in hot water.

To make this test: Add from ten to fifteen volumes of water to the mixture obtained when hydrochloric acid is used in making test 2 above. Heat to boiling and boil for a minute or two. If the white precipitate from lead test 2 is lead chloride, it will dissolve.

4. Anglesite gives cracking sounds and decrepitates (flies to pieces) in a hot flame. Use an alcohol torch flame or a carbide lamp flame for this work.

Lead carbonate (cerussite) is commonly associated with lead sulfate. Lead carbonate, like lead sulfate, fuses easily. Lead carbonate decrepitates in a closed tube.

5. Some cerussites fluoresce (glow) a pale blue color when exposed to strong ultraviolet light (black light).

Always check fluorescent substances by chemical and blowpipe tests.

## LITHIUM

The most important minerals are amblygonite, lepidolite (lithia mica), and petalite.

*Beginners practicing this test should use amblygonite or lepidolite.*

Lithium can usually be detected in a flame test by the brilliant crimson color. Other substances, especially sodium, obscure the crimson color. A small amount of sodium may often be eliminated by first heating the mineral in the cooler part of the flame until the sodium has burned off, then the lithium crimson color can be seen in the hotter part of the flame.

To make this test: Use an iron or nichrome wire. Wet the end of the wire with a dilute hydrochloric acid solution (1 part hydrochloric acid to 7 parts of water). Draw the wet end of the wire through the finely powdered sample to be tested. Heat the mineral coated end of the wire in a flame. An alcohol lamp is satisfactory for this purpose. As soon as the wire and the mineral are red hot, the flame will be colored crimson if lithium is present. Strontium also gives the same crimson color. Strontium minerals, however, will give an alkaline test on moistened litmus paper, after ignition on charcoal, whereas lithium minerals will not.

## MAGNESIUM

The most important magnesium mineral is magnesite (magnesium carbonate).

1. Magnesium can usually be detected by its precipitation as ammonium-magnesium phosphate.

*For this and the following test beginners should use magnesite, dolomite, or some other easily soluble magnesium mineral.*

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade or the equivalent of two grains of wheat. Pour into the receptacle about 15 milliliters (3 teaspoons) of hydrochloric (muriatic) acid, a drop of nitric acid, and about 20 milliliters (4 teaspoons) of water. Heat to boiling, and then cool to room temperature. To the cold solution add an excess of ammonia (until the solution smells strongly of ammonia). If a precipitate results, filter it off and catch the clear, filtered solution in another glass receptacle. To this clear liquid add a little ammonium carbonate or a little ammonium oxalate solution. Again filter off any precipitate which may form, and catch the clear, filtered solution in another receptacle. To this last, clear solution add a few drops of sodium phosphate solution. This will cause a white, crystalline precipitate of ammonium-magnesium phosphate to form if the mineral used in this test contains an appreciable amount of magnesium. To allow sufficient time for the ammonium-magnesium phosphate precipitate to form, it is sometimes necessary to let the solution stand up to 12 hours.

2. Some dolomites (carbonate of calcium and magnesium) fluoresce (glow) a white or gray color when exposed to strong ultraviolet light (black light).

Always check fluorescent substances by chemical and blowpipe tests.

## MANGANESE

Important manganese minerals are pyrolusite (black oxide of manganese, or manganese dioxide), psilomelane (impure hydrous manganese

dioxide), rhodochrosite (manganese carbonate), rhodonite (manganese metasilicate), and bementite (hydrous manganese silicate).

*Beginners practicing the following tests for manganese should use one of the manganese dioxide minerals.*

1. Manganese can usually be detected by the colors it imparts to the fluxes.

To make this test: Prepare a glassy bead as instructed under CHROMIUM, test 1.

a) If the bead made from borax and a *very little* manganese mineral is heated in the oxidizing (bluish) flame of the blowpipe, a bead is produced which is opaque while hot, but on cooling it becomes transparent and has a fine, reddish-violet or amethystine color. If this manganese-borax bead is heated for a time in the reducing (yellow) flame of the blowpipe, it becomes colorless when cold. This bead test is a very delicate one for manganese.

b) If sodium carbonate (or baking soda) is used instead of borax for making the bead test, the color of the bead which is formed when the oxidizing flame of the blowpipe is employed is green when the bead is warm and greenish blue when it cools. The sodium carbonate bead made in the reducing flame is colorless. Sodium carbonate beads are opaque.

If the greenish-blue sodium carbonate bead of manganese is dissolved in a drop of water on a piece of glass, and a drop of nitric acid is added to this solution, the solution will become pink.

2. Nitric acid solutions of manganese with sodium bismuthate give a wine-purple (permanganate) colored solution.

To make this test: Place a little of the mineral to be tested in a test tube or some other glass or porcelain receptacle. Use about the amount of the powdered mineral that can be held on the tip of a knife blade. Pour into the receptacle about 10 milliliters (2 teaspoons) concentrated (strong) nitric acid and about 5 milliliters (1 teaspoon) of water. Boil the mixture for 1 or 2 minutes and then let it stand for a time so that all residue settles.

In a second test tube place a pinch of sodium bismuthate powder. Pour onto the sodium bismuthate about 1 inch of the clear nitric acid solution from the first receptacle. Then add some cold water and let the mixture stand until all residue has settled. Manganese, if present in the mineral used in the test, gives a wine-purple (permanganate) colored solution with sodium bismuthate if there is no excess of chlorides or other reducing substances.

3. Red oxide of lead when added to nitric acid solutions of manganese usually gives a pink (permanganate) colored solution.

To make this test: Place a little of the mineral to be tested in a test tube or some other glass or porcelain receptacle. Use about the amount

of powdered mineral that can be held on the tip of a knife blade. Pour into the receptacle about 5 milliliters (1 teaspoon) of concentrated nitric acid and about the same amount of water. Heat the mixture to boiling, boil for a minute or two, and then let it stand until all residue settles.

In a second test tube place a pinch of powdered red oxide of lead (red-colored litharge can be used). Pour onto the red oxide of lead about 1 inch of the clear nitric acid solution from the first receptacle. Then add a little cold water and let the mixture stand until all residue has settled. Manganese, if present in the mineral used in the test, gives a pink (permanganate) colored solution with red oxide of lead, providing an excess of chlorides or other reducing substances is not present.

4. Many manganese minerals dissolve in hydrochloric acid with the evolution of chlorine gas.

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount of the powdered mineral that can be held on the tip of a knife blade or the equivalent of two grains of wheat. Pour into the receptacle about 5 milliliters of concentrated hydrochloric (muriatic) acid. Heat gently at first, then increase the temperature as much as possible. Chlorine gas will be given off if the mineral used in the test contains an appreciable amount of manganese oxides. This gas can be recognized by its yellowish-green color and its pungent odor.

5. Some manganese minerals, when heated, yield oxygen.

To make this test: Pour into a closed tube a few fragments of the mineral to be tested. Place a sliver of charcoal in the tube a little above the mineral. Heat the tube so that the charcoal alone is heated, and it will be noticed that although the charcoal gets red hot it does not burn, owing to the limited supply of oxygen in the tube. Keeping the charcoal red hot, apply the heat to the tube so that both the mineral and the charcoal are heated. If the mineral is an oxide of manganese, oxygen will be given off, and the charcoal will burn brightly.

6. Some rhodochrosites fluoresce (glow) a gray color when exposed to strong ultraviolet rays (black light).

Always check fluorescent substances by chemical and blowpipe tests.

#### MERCURY (QUICKSILVER)

The most important mercury mineral is cinnabar (native vermillion or mercuric sulfide).

*Beginners practicing the test for mercury should use cinnabar or metallic mercury.*

1. Mercury when heated with soda in a closed tube condenses as metallic globules of mercury on the sides of the tube.

To make this test: Mix thoroughly a little of the finely powdered mineral with about three volumes of *dry* sodium carbonate. Place in a test tube about ½ inch of this mixture and cover this layer with an additional layer of soda to a depth of about ½ inch, then close the tube. Heat carefully and mercury will distill and condense as globules on the walls of the tube if the mineral used in the test contains an appreciable amount of mercury (quicksilver). If only a little mercury is formed, it will appear as a gray sublimate (coating) composed of minute globules which may be made to unite by rubbing with a splinter of wood.

2a. Most compounds of mercury, if moistened with hydrochloric (muriatic) acid and rubbed on a piece of bright copper, will coat the copper. The copper will then appear as if it had been silver plated.

In this test, quicker results are usually obtained if the mineral is powdered instead of in a chunk. The addition of a little powdered manganese dioxide speeds up the reaction.

2b. This test can also be performed by boiling the mineral with hydrochloric acid in a test tube or some other glass or porcelain receptacle. Addition of a little powdered manganese dioxide expedites the reaction. A piece of bright copper immersed in this solution becomes covered by a thin coating of metallic mercury, if the mineral used in the test contains an appreciable amount of mercury.

3. The presence of mercury may be determined by viewing the mercury vapor in an ultraviolet light (3).

To make this test: Use a dark room. Place in a porcelain crucible a little of the powdered mineral to be tested. Set up an ultraviolet light 6 inches to one foot from the flame of a Bunsen burner or other heat source and a willemite-coated screen 6 inches to one foot on the opposite side of the heat source. The sample in the crucible is then heated, and the volatilized mercury will cause a shadow to appear on the willemite-coated screen. If there is no mercury the willemite-coated screen will fluoresce a uniform strong green over the entire surface. Ordinary smoke has little effect upon the willemite-coated screen, but mercury vapor appears as a dense, black cloud of smoke against the screen.

#### MOLYBDENUM

The most important molybdenum minerals of commerce are molybdenite (molybdenum sulfide or molybdenum disulfide) and wulfenite (lead molybdate).

*Beginners practicing the following tests should use molybdenite, or wulfenite.*

1. In acid solutions of molybdenum, sodium or potassium ethyl xanthate usually give a pink to purple precipitate.

To make this test: Place in a 100 or 150 milliliter beaker about ¼ teaspoon of the powdered mineral to be tested. Pour onto the mineral about 5 milliliters (1 teaspoon) of hydrochloric (muriatic) acid, about 5 milliliters of nitric acid, and about 5 milliliters of sulfuric (oil of vitriol) acid. Heat this mixture to boiling and boil it until dense white fumes are given off freely. Add about 10 milliliters (2 teaspoons) of water and boil the mixture again for a minute or so. After the mixture has cooled add strong potassium or sodium hydroxide solution until the test solution is slightly alkaline (alkalies turn red litmus blue), after which, boil it for a minute or two.

Let the mixture cool and settle; then filter out the precipitate (this contains the interfering substances), catching the filtrate (the clear solution that passes through the filter) in another test tube or glass receptacle. Pour about 1 inch of this filtrate into a 6 inch test tube and acidify it by adding an excess of hydrochloric or sulfuric acid (acids turn blue litmus red). Pour into another 6 inch test tube about 1 inch of strong sodium or potassium ethyl xanthate solution (the salt dissolved in water). Pour some of the acidified filtrate into the xanthate solution.

If molybdenum is present, a pink to purple precipitate, which sometimes rapidly fades in color, will form.

2. In nitric acid solutions of molybdenum, potassium ferrocyanide throws down a reddish-brown precipitate.

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade. Pour into the receptacle about 15 milliliters (3 teaspoons) of concentrated (strong) nitric acid. Boil for a few minutes and then add 20 to 25 milliliters (4 to 5 teaspoons) of cold water. When ferrocyanide is added to the acid solution, a reddish-brown precipitate will be thrown down if the mineral used in the test contains an appreciable amount of molybdenum.

3. Molybdenum can usually be detected by the colors it imparts to the fluxes.

Sulfide ore must be thoroughly roasted on charcoal at red heat until sulfur fumes are no longer noticeable before using the mineral for bead tests. Instructions for making bead tests are given under CHROMIUM, test 1.

In the oxidizing (bluish) flame of the blowpipe, borax beads of molybdenum are yellow when warm and colorless when cold. In the reducing (yellow) flame the warm and cold borax beads are colorless. Beads made in the reducing flame, when saturated with molybdenum, are brown when warm or cold.

All salt of phosphorus beads of molybdenum are green except the

unsaturated, cold bead formed in the oxidizing (bluish) flame, which is colorless.

If several of the green salt of phosphorus beads are dissolved in dilute hydrochloric acid and tin is added, the boiling solution turns brown.

4. Some wulfenites (lead molybdate) fluoresce (glow) a green color when exposed to strong ultraviolet rays (black light).

Always check fluorescent substances by chemical and blowpipe tests.

Several tests for molybdenum depend on whether the mineral occurs as an oxide or as a sulfide.

#### *Tests for molybdenum sulfides:*

1. Powdered molybdenum sulfide (molybdenite), if heated strongly in an open tube, gives off sulfurous fumes and deposits a pale yellow sublimate (coating) on the sides of the tube, and delicate, hairlike, white or yellow crystals on the mineral itself.

2. Molybdenite, if heated on charcoal for a long time in the oxidizing (bluish) flame of the blowpipe, deposits a sublimate on the charcoal a short distance from the assay. This sublimate is pale yellow when hot and almost white when cold and often consists of delicate crystals. If this sublimate is touched for an instant with the moderately hot reducing (yellow) flame of the blowpipe, it assumes a beautiful, deep-blue color.

3. Molybdenite is soluble (dissolves) in nitric acid. If the resulting solution is dried and then heated strongly in a porcelain receptacle, a beautiful blue coating forms on the bottom and sides. If the sample is molybdenite with no copper present, the blue coating can be moistened with water and a few drops of strong xanthate solution added. A pink or purple precipitate will be formed as in MOLYBDENUM, test 1, above.

#### *Tests for molybdenum oxides:*

1. Finely powdered molybdenum oxides are partially soluble in hydrochloric acid. Tin added to such a solution produces a green solution which changes to blue and finally to brown.

*Beginners practicing this and the following two tests should use wulfenite or some other oxidized molybdenum mineral.*

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade. Pour into the receptacle about 10 milliliters (2 teaspoons) of concentrated (strong) hydrochloric (muriatic) acid, heat to boiling and boil for a few minutes. Dilute this solution by adding about 20 milliliters (4 teaspoons) of cold water. If some metallic tin is then added to this solution, it will turn green, then, blue, and finally brown if the mineral used in the test contains an appreciable amount of molybdenum oxide.

2. Molybdenum oxides can usually be detected by their reduction to the blue oxide of molybdenum.

To make this test: Place in a small porcelain crucible or some other porcelain receptacle a little of the powdered mineral to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade. Pour onto this mineral a few drops of concentrated sulfuric acid (oil of vitriol), and heat strongly until dense, white fumes of sulfur trioxide are given off for a minute or two. Cool the receptacle by blowing strongly onto the mineral. When it is cool, a beautiful blue color develops around the sides and in the bottom of the receptacle providing the mineral used in the test contains an appreciable amount of molybdenum oxide.

This test can also be made in the following manner: Place in a test tube a little of the powdered mineral to be tested. Use about the amount of the powdered mineral that can be held on the tip of a knife blade. Drop onto the mineral a small piece of paper. Add from three to five drops of concentrated sulfuric acid and about an equal amount of water. Heat strongly until dense, white fumes of sulfur trioxide are given off freely, and then cool. The liquid when cool turns a beautiful deep blue color that disappears if the liquid is again heated to boiling, and reappears on cooling, if the mineral used in the test contains an appreciable amount of molybdenum oxide.

## NICKEL

Important nickel minerals are millerite (nickel pyrites, sulfuret of nickel, capillary pyrites or nickel sulfide) and niccolite (copper nickel, arsenical nickel or nickel arsenide). Nearly all of the nickel of commerce is obtained from nickel-bearing pyrrhotite and garnierite.

*Beginners practicing the following tests should use millerite, niccolite, or a piece of metallic nickel.*

1. Dimethylglyoxime solution throws nickel down as a red precipitate in an alkaline solution. Prepare the dimethylglyoxime solution by dissolving one part of the powdered dimethylglyoxime in about ten parts of alcohol. Several hours are needed to prepare the solution, since the powder dissolves very slowly.

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade or the equivalent of two grains of wheat. Pour into the receptacle about 5 milliliters (1 teaspoon) of concentrated (strong) nitric acid. Heat until the nickel has been dissolved and then add about 10 milliliters (2 teaspoons) of cold water to the solution. Add to this dilute solution an excess of ammonia (until the solution smells of ammonia). Filter the solution through a good filter and catch the filtrate in a glass receptacle. The filtrate

must be crystal clear; if the filtrate is not crystal clear, refilter through a fresh filter. To this clear filtrate add a few drops of dimethylglyoxime solution. When the dimethylglyoxime solution is added to this filtrate there will be thrown down a light red precipitate, if the mineral used in the test contains an appreciable amount of nickel.

2. Ammonia added to an acid solution of nickel produces a pale blue coloration.

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the powdered mineral that is to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade. Pour into the receptacle about 5 milliliters (1 teaspoon) of concentrated nitric acid or a mixture of about 5 milliliters of nitric, 5 milliliters of hydrochloric (muriatic) acid, and 5 milliliters of water. Heat this mixture to boiling until the mineral has been dissolved. If the mineral used in the test contains an appreciable amount of nickel, the solution will turn a greenish color; and if an excess of ammonia is added, the solution will turn to a pale blue color that is considerably lighter than that produced by copper.

3. Nickel minerals fused with soda in the reducing flame yield a magnetic button.

To make this test: Mix thoroughly a little of the finely powdered mineral with about twice its volume of sodium carbonate (or baking soda). Transfer to a stick of charcoal as much of this mixture as can be held on the tip of a knife blade. Heat strongly before the blowpipe in the reducing (yellow) flame until it is thoroughly fused (melted). If the mineral used in this test contains an appreciable amount of nickel, the resulting fused mass will contain a dark-colored, more or less metallic button which is magnetic when cold. Since cobalt and iron buttons produced in this manner are also magnetic, the magnetic button should be further tested for nickel by tests 1, 2, and 4 and for cobalt and iron by the tests given for those metals.

4. Nickel can usually be detected by the colors it imparts to the fluxes. The sample first should be roasted at red heat on charcoal to eliminate any arsenic and sulfur. To make a bead see CHROMIUM, test 1.

In the oxidizing (bluish) flame of the blowpipe, borax beads of nickel are violet when warm and brown when cold. In the reducing flame the borax beads are colorless unless saturated with nickel, then they are gray and opaque.

The salt of phosphorus beads made in the oxidizing flame are yellow when cold and reddish when warm.

## NITRATES

Important natural nitrate minerals are sodium nitrate (soda niter or Chile saltpeter), potassium nitrate (niter or saltpeter), and nitrocalcite (calcium nitrate).

*Beginners practicing the following tests should use sodium nitrate, potassium nitrate, or nitric acid.*

1. Nitrates can usually be detected by the dark blue precipitate formed in diphenylamine solution.

To make this test: Dissolve  $\frac{1}{2}$  gram of diphenylamine in 100 cubic centimeters of concentrated sulfuric acid (oil of vitriol) and cautiously add this to 25 milliliters (5 teaspoons) of water. Set aside until needed later in the test.

Place in a test tube about 1 teaspoon of the material to be tested for nitrates. To dissolve the nitrates pour onto this material about 15 milliliters of water. Heat to boiling, then cool and allow the residue to settle.

Pour into a second test tube about 1 inch of diphenylamine solution. Inclining this test tube slightly, carefully pour about 5 milliliters (1 teaspoon) of the liquid from the first receptacle down the inside of the test tube containing the diphenylamine solution. If nitrates are present in the material used in the test, the solution will usually give a dark blue precipitate.

2. Nitrates can usually be detected by the brown ring formed when a concentrated solution of ferrous sulfate is added to a solution of a nitrate in concentrated sulfuric acid.

Prepare a concentrated ferrous sulfate solution. This is made by dissolving ferrous sulfate in water; enough is used so that some of it remains undissolved.

To make this test: Place in a test tube or some other glass receptacle a little of the mineral to be tested. Use about  $\frac{1}{2}$  teaspoon of the powdered mineral. Pour into the receptacle about 5 milliliters (1 teaspoon) of sulfuric acid (oil of vitriol). Heat this solution to boiling and then cool. If freshly prepared, concentrated ferrous sulfate solution is slowly added to this acid solution, a brown ring will form where the ferrous sulfate solution contacts the acid solution, when the material used in the test contains an appreciable amount of nitrates. Pungent, brownish-red nitrous oxide fumes are usually given off.

3. Nitrates, when fused with potassium bisulfate, yield brownish-red nitrous oxide fumes.

To make this test: Mix thoroughly a little of the finely powdered mineral with an equal volume of powdered potassium bisulfate (acid sulfate of potassium). Place in a test tube about  $\frac{1}{4}$  inch of this mixture. Heat the lower end of the tube at a red heat for some time. Nitrates when given



this treatment give off nitrous oxide fumes which are recognized by their reddish-brown color and pungent odor.

4. Sodium nitrate, potassium nitrate, and calcium nitrate are all soluble in water and have a salty taste. Sodium nitrate and potassium nitrate give a cooling sensation to the tongue. Nitrocalcite has a sharp and bitter taste.

#### OIL SHALES

Shales are fine, dense, more or less consolidated sediments composed principally of fine silt. Their color is usually light gray to black, but yellow, brown, and reddish shales are found in some places. Shales have a noticeably clay-like odor when they are moistened. They can be readily scratched with a knife. Powdered shales are much lighter in color than uncrushed shale, and often are nearly white. Shales commonly have a noticeably laminated structure, that is, they appear to be made up of thin sheets or plates. Surface exposures often look like piles of cardboard or paper, and such masses can be readily separated into flakes or sheets. Shales are usually associated with sandstones and limestones.

Oil shales are usually dark brownish-gray to black on freshly broken surfaces. Weathered surfaces usually are lighter in color. Oil shales contain little or no oil as such. They do, however, contain variable amounts of the solid gum, kerogen. Oil shales rarely smell like petroleum although occasionally freshly broken pieces yield a faint odor. Oil shales may be of the decidedly laminated "paper shale" variety, or the material may be a massive, more or less hardened, and very tough clay.

Kerogen, the carbonaceous matter in oil shales from which petroleum is obtained by destructive distillation processes, can usually be detected if a splinter of the shale is held over the flame of a match, or of a candle. If the proportion of kerogen in the shale is fairly high, the splinter will burn for several seconds after it is removed from the flame. As soon as the flame from the kerogen goes out, white fumes having the odor of burning petroleum are given off for a short time.

#### ORGANIC MATERIAL

Laboratory studies have brought out important facts regarding sand and other materials that are used for concrete. One of these discoveries is the great importance of absolutely clean material. Very often, sand which appears to be clean contains enough humus or vegetable matter to greatly reduce the strength of the concrete made from it. Tests carried on at Lewis Institute (1) gave the following results:

Concrete made from a clean sand gave a compressive strength at twenty-eight days of 1,900 pounds. Concrete made from this same sand, but with one-tenth of one per cent of tannic acid added, gave a strength of only 1,400 pounds; in other words, one-thousandth part of organic impurities, in terms

of the weight of the sand, reduced the strength of the resulting concrete 25 per cent.

We can detect these organic impurities (humus or vegetable matter), even if we cannot see them by using the colorimetric test for organic impurities, devised at the laboratory of the Lewis Institute, Chicago.

To make this test: Take a 12 ounce, graduated prescription bottle and fill to the 4½ ounce mark with the sand to be tested. Pour onto this sand a 3 percent solution of sodium hydroxide (made by dissolving 1 ounce of sodium hydroxide (caustic soda) in enough water to make 32 ounces (1 quart), until the volume of the sand and solution, after shaking, amounts to 7 ounces). Shake thoroughly and let it stand for 24 hours. Observe the color of the clear liquid above the sand. If the solution resulting from this treatment is colorless or has a light-yellowish color, the sand may be considered satisfactory as far as organic impurities are concerned. If the liquid is a brown color, especially dark brown, reject the sand or wash it thoroughly before using it for concrete.

#### PETROLEUM

1. Petroleum (crude oil) in rocks can usually be detected by the odor of petroleum given off when the rocks are vigorously scratched or struck.

2. Petroleum when heated at a high temperature gives off vapors with the characteristic petroleum odor.

*Beginners practicing this and the following test should add a few drops of petroleum to the sample.*

To make this test: In a test tube or some other small-necked receptacle place about 1 inch of the material to be tested for petroleum. Rocks should be crushed, until the pieces used in making the test pass through a ¼ inch screen. Heat strongly over a flame until gases and vapors are given off freely. If the material used in the test contains an appreciable amount of petroleum, the gases will have the characteristic odor of petroleum, and petroleum will deposit on the cool portions of the receptacle.

3. Petroleum can usually be detected with ether or chloroform.

Caution: Ether or chloroform vapors *explode* when ignited. It is very dangerous to make this test near open flames. These vapors will anaesthetize (render insensible, put to sleep). The evaporation of these liquids should, therefore, always be done in the open, or where the fumes will escape without doing harm.

To make this test: In a test tube or glass bottle place about 1 inch of the material to be tested. Rocks should be crushed, and the pieces used in making the test should pass through a ¼ inch screen. Pour onto this material about 1 inch of ether or chloroform. Cork the receptacle tightly.

Shake this mixture at intervals until any oil has been dissolved; this may take from 1 to 10 hours. After the petroleum in the material has been dissolved by the ether or chloroform, let the mixture stand until the liquid becomes clear. Then pour the clear liquid into a shallow, clean, white china or porcelain dish. The ether or chloroform will evaporate quickly leaving a greenish-yellow or brownish ring around the edge of the dish if the material used in the test contains an appreciable amount of petroleum. The ring, if petroleum, will have an oily feel and the characteristic odor of crude oil.

#### PHOSPHORUS

1. Phosphorus can usually be detected by the yellow-colored precipitate formed when a nitric acid solution of phosphorus is added to ammonium molybdate solution.

*Beginners practicing this test should use apatite (phosphate rock or asparagus stone), guano, phosphorite, or some other easily soluble phosphate.*

Prepare molybdic acid solution in the following manner: Mix about  $\frac{1}{2}$  ounce of molybdic acid to a paste with about 40 milliliters (8 teaspoons) of water in a glass or porcelain receptacle. Add about 20 milliliters (4 teaspoons) of strong ammonia, and stir until dissolved. To this solution add about  $7\frac{1}{2}$  milliliters ( $1\frac{1}{2}$  teaspoons) of strong nitric acid, stir well, and set aside. In another receptacle make a mixture of about 50 milliliters (10 teaspoons) nitric acid and about 150 milliliters ( $\frac{3}{4}$  cup) of water. Finally, pour the first solution into the second stirring constantly with a glass rod. Dissolve several grains of phosphate salt (such as sodium phosphate) about the size of grains of table salt in 10 drops of water and add to the mixture. Let stand 24 hours before using. There will be a precipitate in the bottom of the receptacle; use only the clear solution for the test.

**To make this test:** Place a little of the mineral to be tested in a test tube or some other glass or porcelain receptacle. Use about the amount of the powdered mineral that can be held on the tip of a knife blade. Pour into the receptacle about 5 milliliters (1 teaspoon) of concentrated (strong) nitric acid and about the same volume of water. Warm this mixture for about 10 minutes and then cool. Into another test tube pour about 50 milliliters (10 teaspoons) of ammonium molybdate solution, and add a few drops of the cool solution made from the mineral. Let this stand for a few minutes. A yellow precipitate will be thrown down if the material used in the test contains an appreciable amount of phosphorus.

#### POTASH

Potash, strictly speaking, is potassium oxide ( $K_2O$ ) but potassium carbonate ( $K_2CO_3$ ) is also sometimes, although incorrectly, called potash. The "potash" salts of commerce do not necessarily contain potas-

sium oxide — for example, potassium chloride ( $KCl$ ) — and they should more properly be called potassium salts. The term "potash salts," however, is now generally accepted, and all the compounds of potassium are known commercially as potash salts.

From this description it is evident that the characteristic element of all potash salts is potassium and the test for that element should be used.

#### POTASSIUM

1. Volatile compounds of potassium color a nonluminous flame violet.

*Beginners practicing this test should use carnallite or sylvite.*

**To make this test:** Wet one end of a piece of platinum or iron wire about 4 inches long with hydrochloric (muriatic) acid. Draw the wet end of the wire through the finely powdered mineral. Heat the mineral coated end of the wire in a flame. An alcohol flame is satisfactory for this purpose. As soon as the wire and mineral are red hot, the flame will be colored violet if the mineral used in the test contains an appreciable amount of volatile compounds of potassium and provided, further, that the potassium flame is not masked or obscured by the flame of some other element. The yellow flame of sodium and the red flame of lithium obscure the violet flame of potassium. To filter out the flame colors of sodium and lithium, look at the flame through a thick, blue glass.

2. With a Merwin's flame-color screen: Follow the directions outlined above observing the flame through the different sections of a Merwin's flame-color screen. Through section 1, potassium gives a blue-violet flame, it appears violet grading into reddish through section 3, and the same tints, but fainter, are seen through section 2.

#### SILVER

Important silver minerals are native silver, argentite (silver glance, sulfuret of silver, vitreous silver, or silver sulfide), pyrargyrite (dark ruby silver, dark red silver, or silver sulfantimonite), proustite (light ruby silver, light red silver, or silver sulfarsenite), and cerargyrite (horn silver or silver chloride).

*Beginners practicing this test should use argentite, pyrargyrite, some other high-grade silver mineral, or metallic silver.*

1. Silver can usually be detected by its reduction to metallic silver.

**To make this test:** Mix thoroughly a little of the finely powdered mineral with about three times its volume of sodium carbonate (or baking soda). Transfer to a stick of charcoal as much of this mixture as can be held on the tip of a knife blade. Heat strongly before the blowpipe in the reducing (yellow) flame until it is thoroughly fused (melted). A metallic, silver globule or button results if the material contains an appreciable

amount of silver. This button is bright when hot or cold and is malleable (it can be flattened out if hammered on an anvil), but it is both harder and less easily cut than a lead button. It should be further tested for silver as explained in the next test.

2. Hydrochloric acid and soluble chlorides, when added to a nitric acid solution of silver, give a white precipitate, silver chloride. Silver chloride turns dark on exposure to light and is soluble in ammonia.

To make this test: Place in a test tube or some other glass or porcelain receptacle the button of test 1, or a little of the mineral to be tested for silver. Use about the amount of the powdered mineral that can be held on the tip of a knife blade or the equivalent of two grains of wheat. Pour into the receptacle about 10 milliliters (2 teaspoons) of dilute nitric acid (5 milliliters or 1 teaspoon of concentrated nitric acid and about 5 milliliters or 1 teaspoon of distilled or rain water). Sometimes it is better to use concentrated nitric acid to dissolve the silver. Silver minerals, such as cerargyrite are insoluble in nitric acid. Treat these minerals as described in test 1. The silver button is easily soluble in nitric acid.

Cool the solution to room temperature. Add to this cold dilute nitric acid solution a few drops of hydrochloric (muriatic) acid, or a few drops of concentrated (strong) salt water. Upon the addition of the hydrochloric acid, or the salt water to the cold dilute nitric acid solution, a white precipitate will be thrown down providing the material used in the test contains an appreciable amount of silver. If much silver is present, this white precipitate appears as a white curdy mass; if only a small amount of silver is present the precipitate gives the solution a milky appearance. Expose some of the solution containing the white precipitate to a bright light for several hours. If the white precipitate is silver chloride it will turn dark violet to brown.

Lead and mercury may also be thrown down as a white precipitate. The silver can be separated from the lead and mercury by adding ammonia until the solution smells strongly of ammonia. If the white precipitate contains silver chloride, the ammonia will dissolve it. Filter the solution. To the filtrate or the solution that passes through the filter, add nitric acid until no more ammonia fumes are evident from the smell; the silver will be reprecipitated.

#### STRONTIUM

The most important strontium minerals are celestite (strontium sulfate) and strontianite (strontium carbonate).

1. Volatile compounds of strontium color a nonluminous flame crimson.

To make this test: Use a piece of iron wire about 4 inches long. Wet one end of the wire in dilute (one part acid and four parts of water) hydro-

chloric (muriatic) acid. Draw the wet end of the wire through the finely powdered mineral. Heat the mineral coated end of the wire in a flame. An alcohol flame is satisfactory for this purpose. As soon as the wire and mineral are red hot, the flame will be colored crimson if the test mineral contains an appreciable amount of a volatile compound of strontium. The crimson flame of strontium must not be mistaken for the red flame of lithium. The flame may be of such short duration that it appears as a crimson flash.

In a second method of performing this test, moisten one end of a fragment or chunk of the mineral in hydrochloric acid. If the fragment of mineral used in the test is small, use a clean pair of iron tweezers, pliers, or pinchers for holding it in the flame. Heat the moistened end to red heat at the base of an alcohol flame. As soon as the end of the mineral is ignited (red hot) the flame will be colored crimson if the mineral used in the test contains an appreciable amount of a volatile compound of strontium.

2. With a Merwin's flame-color screen: Follow the directions outlined above, observing the flame through the different sections of a Merwin's flame-color screen. Through section 3, strontium gives a crimson-colored flame which is absorbed by sections 1 and 2.

3. Some strontianites fluoresce (glow) a white or green color when exposed to strong ultraviolet rays (black light) — best seen in the dark.

Always check fluorescent substances by chemical and blowpipe tests.

#### TIN

The only important tin mineral is cassiterite (tinstone or tin dioxide; when recovered from placers it is called stream tin).

Cassiterite when boiled with metallic zinc in hydrochloric or sulfuric acid usually becomes coated with a gray metallic deposit.

To make this test: Place in a test tube a ragged fragment of the mineral, somewhat larger than a bean. Around this fragment pour metallic zinc, allowing about one half of the fragment to project above the zinc. Granulated zinc, about 20 mesh, works satisfactorily in this test. Onto this pour about 15 milliliters (3 teaspoons) of either concentrated (strong) hydrochloric (muriatic) acid or concentrated sulfuric acid (oil of vitriol) and about 10 milliliters (2 teaspoons) of water. Heat this mixture to boiling and allow it to boil for two minutes.

Cassiterite when given this treatment usually becomes coated with a dull gray metallic deposit. If the fragment is washed in water and the coating is then rubbed dry the metallic deposit becomes bright.

2. Tin can usually be detected by the sublimate formed on charcoal and its reduction to small, metallic globules. These globules, if treated with nitric acid, yield a white powder.

**To make this test:** Mix thoroughly a little of the finely powdered mineral with an equal volume of powdered charcoal and two volumes of sodium carbonate (or baking soda). Transfer to a shallow cavity in a stick of charcoal about the amount of this mixture that can be held on the tip of a knife blade, and form a paste of the mixture with water. Heat before the blowpipe in a strong, reducing (yellow) flame. This treatment gives a scanty sublimate (coating) on the charcoal which is yellowish when hot and white when cold.

If this sublimate is moistened with a drop or two of cobalt nitrate solution, and if the assay is then heated strongly before the blowpipe in the reducing flame, the sublimate will assume a dull, bluish-green color when cold.

This treatment also yields small metallic globules, which can only with difficulty be forced to run together into one single, larger globule. These globules on cooling become coated with a white film, but if cut open show a white, metallic color. If these globules are treated with nitric acid, a white powder is produced which is insoluble in that acid.

#### TUNGSTEN

Important tungsten minerals are wolframite (wolfram, hubnerite, or tungstate of iron and manganese), ferberite (iron tungstate), tungstite (tungsten sulfide), scheelite (calcium tungstate), and cuproscheelite (calcium copper tungstate).

*Beginners practicing the following test should use scheelite or some other easily soluble tungsten mineral.*

Tests 1 and 2 for tungsten should be used only on very soluble tungsten minerals such as scheelite. The rather insoluble minerals should be given a preliminary fusion with soda ash as described in IRON, test 2, and the fused mass then tested as follows:

1. In hydrochloric acid, tungsten gives a lemon-yellow residue that is soluble in ammonia.

**To make this test:** Place in a test tube or some other glass or porcelain receptacle a little of the finely powdered mineral to be tested. Use about the amount of the powdered mineral that can be held on the tip of a knife blade. Pour into the receptacle about 10 milliliters (2 teaspoons) of concentrated (strong) hydrochloric (muriatic) acid. Heat this mixture to boiling and boil for a time, replenishing the acid if necessary. This treatment will give a lemon-yellow residue (tungstic acid) in the bottom of the receptacle if the mineral used in the test contains an appreciable amount of tungsten. If this lemon-yellow residue is soluble in ammonia, it is tungstic acid. Pour into the receptacle an excess of ammonia, until the solution smells strong of ammonia. Warm slightly if necessary. The lemon-yellow residue will dissolve in the ammonia if it is tungstic acid.

2. Solutions of tungsten reduced by tin, or zinc give a blue color in hydrochloric acid.

**To make this test:** Pour into a test tube or some other glass or porcelain receptacle a little of the finely powdered mineral to be tested. Use about the amount of the mineral that can be held on the tip of a knife blade. Pour into the receptacle about 10 milliliters (2 teaspoons) of concentrated hydrochloric acid. Heat this mixture to boiling and boil strongly, replenishing the acid if necessary, until a lemon-yellow colored residue begins to form in the bottom of the receptacle; then add a little metallic tin or metallic zinc to the solution. If no other tin or zinc is available, galvanized iron, a piece of a tin can, soft solder, or hard solder may be used for their tin or zinc content. When tin or zinc is added, the solution will turn deep blue, and later it will turn brown if the material contains an appreciable amount of tungsten.

3. Tungsten can usually be detected by the colors it imparts to the fluxes. Instructions for making bead tests are given under CHROMIUM, test 1.

All of the borax beads of tungsten are colorless except the warm, saturated beads which are yellow.

All of the salt of phosphorus beads of tungsten are colorless except the warm, saturated bead which is yellow and the cold, saturated bead, made in the reducing flame, which is greenish-blue.

4. Some scheelites fluoresce (glow) a blue or bluish-white color when exposed to strong ultraviolet rays (black light).

Always check fluorescent substances by chemical and blowpipe tests.

#### URANIUM

Uranium and its chain of decay products give off radio-active energy, which can be detected by a Geiger counter. Other elements also give off radio-active energy, therefore, the sample should be checked by chemical test. A simple bead test developed by the United States Atomic Energy Commission (5) uses ultraviolet light to determine uranium.

**To make this test:** Use a piece of No. 26 platinum, nichrome, or iron wire about 3 inches long. Fasten one end in a holder (cork or piece of soft wood) so that when the wire is heated it will not burn the fingers. Make a loop about  $\frac{1}{8}$  inch in diameter at the unattached end of the wire by winding the end of the wire around the lead of a pencil. Heat the looped end in a flame until it is red hot. Dip the hot loop into some powdered sodium fluoride or lithium fluoride, a little of which will adhere to the wire loop. Fuse the fluoride to the wire by holding it in the flame. Repeat this operation until a bead fills the loop in the wire. Touch the bead while it is red hot to a little of the powdered mineral and return to the flame. Too much mineral fused into the bead will cause the bead to appreciably

reduce the sensitivity of the test. Allow the bead to cool and place the bead under an ultraviolet light (either long or short wave). A bead made with sodium fluoride will fluoresce yellow; if made with lithium fluoride, it will fluoresce bright green if the sample contains sufficient uranium.

## VANADIUM

Important vanadium minerals are patronite (vanadium sulfide), vanadiferous asphaltite, vanadinite (chlorovanadate of lead), descloizite (hydrated basic vanadate of lead and zinc), rescoelite (vanadium mica), and carnotite (a mixture of vanadium and uranium compounds).

*Vanadinite, descloizite, or some other vanadate should be used by beginners practicing the following test.*

1. Vanadium colors concentrated hydrochloric acid a deep cherry-red. This reaction takes place with the evolution of chlorine gas. A little water added to this solution changes it to a light green color.

To make this test: Place in a bone-dry test tube or some other glass or porcelain receptacle a little of the mineral to be tested. Use about the amount of the powdered mineral that can be held on the tip of a knife blade. Pour onto the mineral about 2 milliliters ( $\frac{1}{2}$  teaspoon) of concentrated (strong) hydrochloric (muriatic) acid. Almost as soon as the hydrochloric acid comes into contact with the mineral chlorine gas will be given off, and the solution will turn to a deep, cherry-red color if the mineral used in the test contains an appreciable amount of vanadium. This chlorine gas is easily recognized by its rusty-green color and its pungent odor. If a few drops of water are added to this cherry-red solution, it changes to a light greenish tint. If too much water is added the solution becomes almost colorless.

2. Vanadium minerals impart a greenish color to sulfuric acid solutions.

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the mineral that is to be tested. Use about the amount of powdered mineral that can be held on the tip of a knife blade. Pour onto the mineral about 5 milliliters (1 teaspoon) of concentrated sulfuric acid (oil of vitriol). Heat the solution to boiling and boil until dense, white fumes of sulfur trioxide are given off. Cool to room temperature, and when cold cautiously add from 5 to 10 milliliters (1 to 2 teaspoons) of cold water. When the water is added, the color of the solution will rapidly change to a light green if the mineral used in the test contains appreciable soluble vanadium.

3. Vanadates with potassium bisulfate in a closed tube give a yellow mass.

To make this test: Mix thoroughly a little of the finely powdered mineral with an equal volume of potassium bisulfate (acid sulfate of

potassium). Place about  $\frac{1}{2}$  inch of this mixture in a closed tube. Heat the lower end of the tube to a red heat for some time. This will produce a yellow mass if the mineral used in the test contains vanadates.

4. Hydrogen peroxide gives a brownish-red color to an acid solution of vanadium.

To make this test: Place a little of the mineral to be tested in a porcelain receptacle. Use about the amount of the powdered mineral that can be held on the tip of a knife blade. Pour into the receptacle about 5 milliliters (1 teaspoon) of concentrated hydrochloric acid and boil the solution for a couple of minutes. Cool the receptacle and pour into it about 5 milliliters (1 teaspoon) of concentrated nitric acid. Boil for a couple of minutes and cool. After it is cold add about 1 teaspoon of concentrated sulfuric acid and boil this mixture over an open flame until dense white fumes are given off. Cool to room temperature and very carefully add about 15 milliliters (3 teaspoons) of cold water. Boil for a couple of minutes and then filter. Catch the filtrate (the clear solution that passes through the filter paper) in a glass or porcelain receptacle. Pour about 1 teaspoon of this filtrate into a test tube. To this add a drop or two of fresh hydrogen peroxide. When the hydrogen peroxide is added the solution will become brownish red if the mineral used in the test contains an appreciable amount of soluble vanadium.

5. Vanadium can usually be detected by the colors it imparts to the fluxes. Sulfide or arsenide ore must be thoroughly roasted (pulverized and heated on charcoal at a red heat until sulfur or arsenic fumes are no longer noticeable) before using the mineral in the bead test. To make a bead see CHROMIUM, test 1.

In the oxidizing (bluish) flame of the blowpipe, borax beads of vanadium are all yellow except the unsaturated, cold bead, which is colorless. In the reducing (yellow) flame all borax beads of vanadium are green.

All salt of phosphorus beads of vanadium made in the oxidizing flame are yellow, and all such beads made in the reducing flame are green.

6. Some vanadinites (lead vanadate) fluoresce (glow) a green color when exposed to strong ultraviolet rays (black light).

Always check fluorescent substances by chemical and blowpipe tests.

## ZINC

Important zinc minerals are sphalerite (blende, zinc blende, black-jack, false lead, false galena, or zinc sulfide), smithsonite (dry-bone ore or zinc carbonate), and calamine (electric calamine, hydrous zinc silicate, basic zinc metasilicate or silicate of zinc).

*Sphalerite, smithsonite, or a small piece of metallic zinc should be used by beginners practicing this and the following test.*

1. Zinc can usually be detected by the sublimate formed on charcoal when it is heated with soda before the blowpipe. This sublimate is yellow when hot and white when cold. If the sublimate is moistened with cobalt nitrate solution and heated, it assumes a green color.

To make this test: Mix thoroughly a little of the finely powdered mineral with an equal volume of sodium carbonate (or baking soda). Transfer to a shallow cavity in a stick of charcoal about the amount of this mixture that can be held on the tip of a knife blade, and form a paste of the mixture by moistening with water. Heat before the blowpipe in a strong, reducing (yellow) flame. This treatment gives a scanty sublimate (coating) on the charcoal which is canary-yellow in color when hot and white when cold, if the mineral used in the test contains an appreciable amount of zinc.

If the sublimate made in the test just described is moistened with a drop or two of cobalt nitrate solution and if the assay is then heated strongly before the blowpipe in the reducing flame, the sublimate will assume a bright green color that is best seen when it is cold.

2. Zinc is thrown down as a white precipitate by ammonium sulfide from an alkaline solution, thus being the only white sulfide that is insoluble in such a solution.

To make this test: Place in a test tube or some other glass or porcelain receptacle a little of the mineral that is to be tested. Use about the amount of the powdered mineral that can be held on the tip of a knife blade. Pour onto the mineral in the receptacle about 5 milliliters (1 teaspoon) of concentrated (strong) hydrochloric (muriatic) acid and a drop of nitric acid, and heat to boiling. Add about 10 milliliters (2 teaspoons) of cold water and cool the solution to room temperature. To the cold solution add ammonia until the solution smells strongly of ammonia. Any iron present will be thrown down as a brownish-red precipitate as mentioned in test 3 for iron. Filter and catch the clear, filtered solution in another glass or porcelain receptacle. To the clear, filtered solution add a few drops of ammonium sulfide solution. This will throw down zinc as a white precipitate (zinc sulfide).

3. Some zinc minerals (some willemites, smithsonites, sphalerites, and others) fluoresce (glow) when exposed to strong ultraviolet rays (black light). Green, yellow, and blue are among the colors emitted by these minerals when activated by this light — best seen in the dark.

Always check fluorescent substances by chemical and blowpipe tests.

## AVERAGE WEIGHTS OF VARIOUS SUBSTANCES

Name	Pounds per cubic foot	Cubic feet per short ton (2,000 lbs.)
Aluminum, cast .....	160	12.5
Andesite .....	181	11.0
Antimony, cast .....	418	4.8
Antimony sulfide (stibnite) .....	287	7.0
Arsenic sulfide (realgar) .....	218	9.2
Asbestos .....	175	11.4
Asphaltum .....	69 to 75	26.5 to 29.0
Barium sulfate (barite) .....	280	7.1
Basalt (traprock) .....	181	11.0
Borax .....	109	18.4
Brass (copper and zinc), cast .....	527	3.7
Brick, common .....	100 to 130	15.4 to 20.0
Calcite .....	168	11.9
Cement (Portland) .....	85 to 195	10.2 to 23.6
Chalk .....	146	13.7
Clay, loose, dry .....	65	30.8
Coal, bituminous, broken loose .....	47 to 60	36.4 to 42.5
Cobalt, nickel arsenide (smaltite) .....	405	4.9
Concrete, stone .....	130 to 150	13.3 to 15.4
Copper, cast .....	550	3.6
Copper carbonate (malachite) .....	243	8.2
Copper pyrites (chalcopyrite) .....	262	7.6
Diabase or diorite .....	187	10.7
Diatomaceous earth, pure dry blocks .....	28	71.5
Diatomaceous earth, pure dry powder, loose .....	10	200.0
Earth, loam, loose, dry .....	72 to 80	25.0 to 27.8
Feldspar .....	160	12.5
Fluorspar (fluorite) .....	199	10.1
Gold, native .....	1,184 (1,215.9 pure)	1.7
Granite .....	170	11.8
Graphite .....	140	14.3
Gravel, dry .....	112	17.9
Gypsum .....	143	14.0
Iron, cast .....	450	4.4
Iron, wrought .....	480	4.2
Iron pyrites .....	318	6.3
Iron sesquioxide (hematite) .....	312	6.4
Lead .....	711	2.8
Lead carbonate (cerussite) .....	405	4.9
Lead sulfide (galena) .....	467	4.3
Limestone, average .....	162	12.4
Magnesium carbonate (magnesite) .....	187	10.7

# AVERAGE WEIGHTS — *Continued*

Name	Pounds per cubic foot	Cubic feet per short ton (2,000 lbs.)
Manganese .....	499	4.0
Manganese carbonate (rhodochrosite) .....	224	8.9
Manganese dioxide (pyrolusite) .....	299	6.7
Marble, domestic, average .....	165	12.1
Mercury (quicksilver), native .....	849	2.36
Mercury sulfide (cinnabar) .....	505	3.9
Mica .....	183	10.9
Nickel sulfide (millerite) .....	349	5.7
Nickel arsenide (niccolite) .....	467	4.3
Platinum, native, average .....	1,091	1.83
Potash .....	141	14.2
Pumice stone .....	57	35.1
Quartz .....	162	12.4
Rhyolite .....	149	13.4
Salt, loose .....	45	44.5
Sand .....	90 to 130	15.4 to 22.2
Schist .....	168	11.9
Shale .....	162	12.4
Silver, native, average .....	655	3.06
Slate .....	175	11.4
Sulfur, native, average .....	131	15.2
Steel, cast .....	492	4.0
Steel, wrought .....	490	4.1
Talc .....	175	11.4
Tin oxide (cassiterite) .....	424	4.71
Tungsten ore (scheelite) .....	374	5.35
Tungsten ore (wolframite) .....	455	4.4
Water .....	62.4	32.0
Zinc .....	444	4.5
Zinc sulfide (sphalerite, blende) .....	249	8.0

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