silica. The first step in the process was the filling of the pores and cavities by the silica in solution, and then as the woody fiber, by gradual decomposition, disappeared the silica further took its place. Other examples are quartz after fluorite, calcite, and many other species; cassiterite after orthoclase; native copper after aragonite, etc.

2. Pseudomorphs by *incrustation* form a less important class. Such are the crusts of quartz formed over fluorite. In most cases the removal of the original mineral has gone on simultaneously with the deposition of the second, so that the resulting pseudomorph is properly one of substitution. In pseudomorphs by *infiltration* a cavity made by the removal of a crystal has been filled by another mineral.

3. The third class of pseudomorphs, by *alteration*, includes a considerable proportion of the observed cases, of which the number is very large. Conclusive evidence of the change which has gone on is often furnished by a nucleus of the original mineral in the center of the altered crystal — *e.g.*, a kernel of cuprite in a pseudomorphous octahedron of malachite; also of chrysolite in a pseudomorphous crystal of serpentine, etc.

(*a*) An example of *paramorphism* — that is, of a change in molecular constitution without change of chemical substance — is furnished by the change of aragonite to calcite (both $CaCO_3$) at a certain temperature; also the *paramorphs* of rutile after brookite (both TiO_2) from Magnet Cove, Arkansas.

(*b*) An example of the pseudomorphs in which alteration is accompanied by a loss of ingredients is furnished by crystals of native copper in the form of cuprite.

(*c*) In the change of cuprite to malachite — *e.g.*, the familiar crystals from Chessy, France — an instance is afforded of the assumption of an ingredient — viz., carbon dioxide (and water). Pseudomorphs of gypsum after anhydrite occur where there has been an assumption of water alone.

(*d*) A partial exchange of constituents — in other words, a loss of one and gain of another — takes place in the change of feldspar to kaolin, in which the potash silicate disappears and water is taken up; pseudomorphs of limonite after pyrite or siderite, of chlorite after garnet, pyromorphite after galena, are other examples.

The chemical processes involved in such changes open a wide and important field for investigation. Their study has served to throw much light on the chemical constitution of mineral species and the conditions under which they have been formed. For the literature of the subject see the Introduction, p. 4 (Blum, Bischof, Roth, etc.).

CHEMICAL EXAMINATION OF MINERALS

479. The complete investigation of the chemical composition of a mineral includes, first, the identification of the elements present by qualitative analysis, and, second, the determination of the relative amounts of each by quantitative analysis, from which last the formula can be calculated. Both processes carried out in full call for the equipment of a chemical laboratory. An approximate qualitative analysis, however, can, in many cases, be made quickly and simply with few conveniences. The methods employed involve either (*a*) the use of acids or other reagents " in the wet way," or (*b*) the use of the blowpipe, or of both methods combined. Some practical instructions will be given applying to both cases.

EXAMINATION IN THE WET WAY

480. Reagents, etc. — The most commonly employed chemical reagents are the three mineral acids, hydrochloric, nitric, and sulphuric acids. To these may be added ammonium hydroxide, also solutions of barium chloride, silver nitrate, ammonium molybdate, ammonium oxalate; finally, distilled water in a wash-bottle.

A few test-tubes are needed for the trials and sometimes a porcelain dish with a handle called a casserole; further, a glass funnel and filter-paper. The Bunsen gas-burner (p. 330) is the best source of heat, though an alcohol lamp may take its place. It is unnecessary to remark that the use of acids and the other reagents requires much care to avoid injury to person or clothing.

In testing the powdered mineral with the acids, the important points to be noted are: (1) the degree of solubility, and (2) the phenomena attending entire or partial solution; that is, whether (*a*) a solution is obtained quietly, without effervescence, and, if so, what its color is; or (*b*) a gas is evolved, producing effervescence; or (*c*) an insoluble constituent is separated out.

481. Solubility. — In testing the degree of solubility hydrochloric acid is most commonly used, though in the case of many metallic minerals, as the sulphides and compounds of lead and silver, nitric acid is required. Less often sulphuric acid and aqua regia (nitro-hydrochloric acid) are resorted to.

The trial is usually made in a test-tube, and in general the fragment of mineral to be examined should be first carefully pulverized in an agate mortar. In most cases the heat of the Bunsen burner must be employed.

(*a*) Many minerals are completely *soluble without effervescence;* among these are some of the oxides, as hematite, limonite, göthite, etc.; some sulphates, many phosphates and arsenates, etc. Gold and platinum are soluble only in aqua regia or nitro-hydrochloric acid.

A yellow solution is usually obtained if much iron is present; a blue or greenish blue solution (turning deep blue on the addition of ammonium hydroxide in excess) from compounds of copper; pink or pale rose from cobalt, etc.

(*b*) *Solubility with effervescence* takes place when the mineral loses a gaseous ingredient, or when one is generated by the mutual reaction of acid and mineral. Most conspicuous here are the *carbonates,* all of which dissolve with effervescence, giving off the odorless gas *carbon dioxide* (CO_2), though some of them only when pulverized, or, again, on the addition of heat. In applying this test dilute hydrochloric acid is employed.

Hydrogen sulphide (H_2S) is evolved by some sulphides when dissolved in hydrochloric acid: this is true of sphalerite, stibnite, etc. This gas is readily recognized by its offensive odor.

Chlorine is evolved by oxides of manganese and also chromic and vanadic acid salts when dissolved in hydrochloric acid.

Nitrogen dioxide (NO_2) is given off, in the form of red suffocating fumes, by many metallic minerals, and also some of the lower oxides (cuprite, etc.), when treated with nitric acid.

(*c*) The *separation of an insoluble ingredient* takes place: With many silicates, the *silica* separating sometimes as a fine powder, and again as a jelly; in the latter case the mineral is said to *gelatinize* (sodalite, analcite). In order to test this point the finely pulverized silicate is digested with strong hydrochloric acid, and the solution afterward slowly evaporated nearly to dryness. With a considerable number of silicates the gelatinization takes place only after the mineral has been previously fused; while some others, which ordinarily gelatinize, are rendered insoluble by ignition.

With many sulphides (as pyrite) a separation of *sulphur* takes place when they are treated with nitric acid.

Some compounds of titanium and tungsten are decomposed by hydrochloric acid with the separation of the oxides of the elements named (TiO_2, WO_3). The same is true of salts of molybdic and vanadic acids, only that here the oxides are soluble in an excess of the acid.

Compounds containing silver, lead, and mercury give with hydrochloric acid insoluble residues of the *chlorides.* These compounds are, however, soluble in nitric acid.

When compounds containing tin are treated with nitric acid, the *tin dioxide* (SnO_2) separates as a white powder. A corresponding reaction takes place under similar circumstances with minerals containing arsenic and antimony.

Insoluble Minerals. — A large number of minerals are not sensibly attacked by any of the acids. Among these may be named the following oxides: corundum, spinel, chromite, diaspore, rutile, cassiterite, quartz; also cerargyrite; many silicates, titanates, tantalates, and niobates; some of the sulphates, as barite, celestite; many phosphates, as xenotime, lazulite, childrenite, amblygonite; also the borate, boracite.

482. Examination of the Solution. — If the mineral is difficultly, or only partially, soluble, the question as to solubility or insolubility is not always settled at once. Partial solution is often shown by the color given to the liquid, or more generally by the precipitate yielded, for example, on the addition of ammonium hydroxide to the liquid filtered off from the remaining powder. The further examination of the solution yielded, whether from partial or complete solution, after the separation by filtration of any insoluble residue, requires the systematic laboratory methods of qualitative analysis.

It may be noted, however, that in the case of sulphates the presence of *sulphur* is shown by the precipitation of a heavy white powder of barium sulphate ($BaSO_4$) when barium chloride is added. The presence of *silver* in solution is shown by the separation of a white curdy .precipitate of silver chloride ($AgCl$) upon the addition of any chlorine compound; conversely, the same precipitate shows the presence of *chlorine* when silver nitrate is added to the solution.

Again, *phosphorus* may be detected if present, even in small quantity, in a nitric acid solution of a mineral by the fine yellow powder which separates, sometimes after standing, when ammonium molybdate has been added.

EXAMINATION BY MEANS OF THE BLOWPIPE *

483. The use of the blowpipe, in skilled hands, gives a quick method of obtaining a partial knowledge of the qualitative composition of a mineral. The apparatus needed includes the following articles:

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* The subject of the blowpipe and its use is treated very briefly in this place. The student who wishes to be fully informed not only in regard to the use of the various instruments, but also as to all the valuable reactions practically useful in the identification of minerals, should consult a manual on the subject. The Brush-Penfield Manual of Determinative Mineralogy, with an introduction on Blowpipe Analysis, is particularly to be recommended.

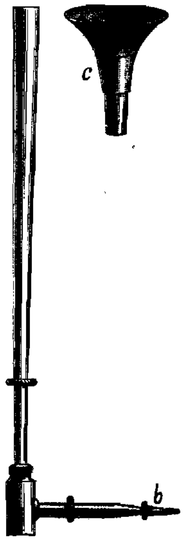
Blowpipe, lamp, forceps, preferably with platinum points, platinum wire, charcoal, glass tubes; also a small hammer with sharp edges, a steel anvil an inch or two long, a horseshoe magnet, a small agate mortar, a pair of cutting pliers, a three-cornered file.

Further, test-paper, both turmeric and blue litmus paper; a little pure tin-foil; also in small wooden boxes the fluxes: borax (sodium tetraborate), soda (anhydrous sodium carbonate), salt of phosphorus or microcosmic salt (sodium-ammonium phosphate), acid potassium sulphate ($HKSO_4$); also a solution of cobalt nitrate in a dropping bulb or bottle; further, the three acids mentioned in Art. **480**.

484. Blowpipe and Lamp. — A good form of *blowpipe* is shown in Fig. 622. The air-chamber, at *a*, is essential to stop the condensed moisture of the breath, the tip (*b*), which is removable, is usually of brass, (*c*) is a removable mouthpiece which may or may not be used as preferred.

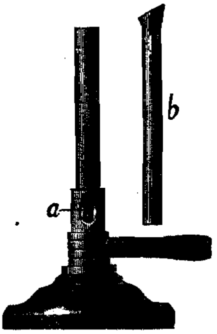
The most convenient form of *lamp* is that furnished by an ordinary Bunsen gas-burner * (Fig. 623), provided with a tube, *b*, which when inserted cuts off the air supply at *a*; the gas then burns at the top with the usual yellow flame. This flame should be one to one and a half inches high. The tip of the blow-pipe is held near (or just within the flame, see beyond), and the air blown through it causes the flame to take the shape shown in Fig. 625.

622



It is necessary to learn to blow *continuously*, that is, to keep up a blast of air from the compressed reservoir in the mouth-cavity while respiration is maintained through the nose. To accomplish this successfully and at the same time to produce a clear flame without unnecessary fatiguing effort calls for some practice.

623



When the tube, *b*, is removed, the gas burns with a colorless flame and is used for heating glass tubes, test-tubes, etc.

485. Forceps. Wire. — The *forceps* (Fig. 624) are made of steel, nickel-plated, and should have a spring strong enough to support firmly the small fragment of mineral between the platinum points at *d*. The steel points at the other end are used to pick up small pieces of minerals, but must not be inserted in the flame. Care must be taken not to injure the platinum by allowing it to come in contact with the fused mineral, especially if this contains antimony, arsenic, lead, etc. Cheaper forceps, made of steel wire, etc., while not so convenient, will also serve reasonably well.

A short length of fairly stout platinum wire to be used in the making of bead tests should be available. A similar length of finer wire for making flame tests is also desirable.

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* Instead of this, a good stearin candle will answer, or an oil flame with flat wick.

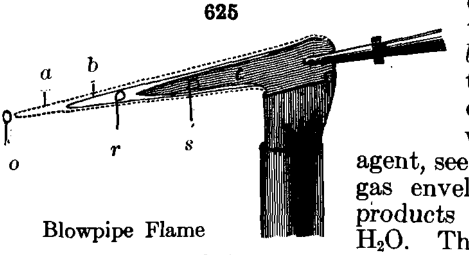
486. Charcoal. — The *charcoal* employed should not snap and should yield but little ash; the kinds made from basswood, pine or willow are best. It is most conveniently employed in rectangular pieces, say four inches long, an inch wide, and three-quarters of an inch in thickness. The surface must always be perfectly clean before each trial.

487. Glass Tubes. — The glass tubes should be preferably of two grades; a hard glass tubing with about 5 mm. interior diameter to be cut in five inch lengths and used in open tube tests and a soft glass tubing with about 3 mm. interior diameter to be in about six inch lengths, each length yielding two closed tubes.

624

488. Blowpipe Flame. — The blowpipe flame, shown in Fig. 625, consists of three cones: an inner of a blue color, *c*, a second pale violet cone, *b*, and an outer invisible cone, *a*. The cone *c* consists of unburned gas mixed with air from the blowpipe. There is no combustion in this cone and therefore no heat. The cone *b* is the one in which combustion is taking place. This cone contains carbon monoxide which is a strong reducing agent, see below. Cone *a* is merely a gas envelope composed of the final products of combustion, CO_2 and H_2O. The heat is most intense near the tip of the cone *b*, and the mineral is held at this point when its *fusibility* is to be tested.

625



Blowpipe Flame

The point *o*, Fig. 625, is called the OXIDIZING FLAME (O.F.); it is characterized by the excess of the oxygen of the air and has hence an *oxidizing* effect upon the assay. This flame is best produced when the jet of the blowpipe is inserted a very little in the gas flame; it should be entirely non-luminous.

The cone *b* is called the REDUCING FLAME (R.F.); it is characterized by the excess of the carbon or hydrocarbons of the gas, which at the high temperature present tend to combine with the oxygen of the mineral brought into it (at *r*), or, in other words, *to reduce* it. The best reducing flame is produced when the blowpipe is held a little distance from the gas flame; it should retain the yellow color of the latter on its upper edge.

489. Methods of Examination. — The blowpipe investigation of minerals includes their examination, (1) in the forceps, (2) in the closed and the open tubes, (3) on charcoal or other support, and (4) with the fluxes on the platinum wire.

1. EXAMINATION IN THE FORCEPS

490. Use of the Forceps. — Forceps are employed to hold the fragment of the mineral while a test is made as to its fusibility; also when the presence of a volatile ingredient which may give the flame a characteristic color is tested for, etc.

The following practical points must be regarded: (1) Metallic minerals, especially those containing arsenic or antimony, which when fused might injure the platinum of the forceps, should first be examined on charcoal;* (2) the fragment taken should be thin, and as small as can conveniently be held, with its edge projecting well beyond the points; (3) when decrepitation takes place, the heat must be applied slowly, or, if this does not prevent it, the mineral may be powdered and a paste made with water, thick enough to be held in the forceps or on the platinum wire; or the paste may, with the same end in view, be heated on charcoal; (4) the fragment whose fusibility is to be tested must be held in the hottest part of the flame, just beyond the extremity of the blue cone.

491. Fusibility. — All grades of fusibility exist among minerals, from those which fuse in large fragments in the flame of the candle (stibnite, see below) to those which fuse only on the thinnest edges in the hottest blowpipe flame (bronzite); and still again there are a considerable number which are entirely infusible (*e.g.*, corundum).

The exact determination of the temperature of fusion is not easily accomplished (cf Art. **431** p. 304), and for purposes of determination of species it is unnecessary. The approximate *relative* degree of fusibility is readily fixed by referring the mineral to the following scale, suggested by von Kobell:

1. Stibnite.
2. Natrolite (or Chalcopyrite).
3. Almandite Garnet.
4. Actinolite.
5. Orthoclase.
6. Bronzite.

492. In connection with the trial of fusibility, the following phenomena may be observed: (*a*) *coloration* of the flame (see Art. **493**); (*b*) *swelling up* (stilbite), or *exfoliation* of the mineral (vermiculite); or (*c*) *glowing* without fusion (calcite); and (*d*) *intumescence*, or a spirting out of the mass as it fuses (scapolite).

The color of the mineral after ignition is to be noted; and the nature of the fused mass is also to be observed, whether a clear or blebby glass is obtained, or a black slag; also whether the bead or residue is magnetic or not (due to iron, less often nickel, cobalt), etc.

The ignited fragment, if nearly or quite infusible, may be moistened with the cobalt solution and again ignited, in which case, if it turns *blue*, this indicates the presence of aluminium (as with cyanite, topaz, etc.); but note that zinc silicate (calamine) also assumes a blue color. If it becomes *pink*, this indicates a compound of magnesium (as brucite).

Also, if not too fusible, it may, after treatment in the forceps, be placed upon a strip of moistened turmeric paper, in which case an *alkaline* reaction proves the presence of an alkali, sodium, potassium; or an alkaline earth, calcium, barium, strontium.

493. Flame Coloration. — The color often imparted to the outer blowpipe flame, while the mineral held in the forceps is being heated, makes possible the identification of a number of the elements.

The colors which may be produced, and the substances to whose presence they are due, are as follows:

Color	Substance
Carmine-red	Lithium.
Purple-red	Strontium.
Orange-red	Calcium.
Yellow	Sodium.
Yellowish green	Barium.
Siskine-green	Boron.

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* Arsenic, antimony, and easily reducible metals like lead, also copper, form more or less fusible alloys with platinum.

Emerald-green	Oxide of copper.
Bluish green	Phosphoric acid (phosphates).
Greenish blue	Antimony.
Whitish blue	Arsenic.
Azure-blue	Chloride of copper; also selenium.
Violet	Potassium.

A yellowish green flame is also given by the oxide or sulphide of molybdenum; a bluish green flame (in streaks) by zinc; a pale bluish flame by tellurium; a blue flame by lead.

494. Notes. — The presence of soda, even in small quantities, produces a yellow flame, which (except in the spectroscope) more or less completely masks the coloration of the flame due to other substances, *e.g.*, potassium. A filter of blue glass held in front of the flame will shut out the monochromatic yellow of the sodium flame and allow the characteristic violet color of the potassium to be observed. Silicates are often so difficultly decomposed that no distinct color is obtained even when the substance is present; in such cases (*e.g.*, potash feldspar) the powdered mineral may be fused on the platinum wire with an equal volume of gypsum, when the flame can be seen (at least through blue glass). Again, a silicate like tourmaline fused with a mixture of fluorite and acid potassium sulphate yields the characteristic green flame of boron. Phosphates and borates give the green flame in general best when they have been pulverized and moistened with sulphuric acid. Moistening with hydrochloric acid makes the coloration in many cases (as with the carbonates of calcium, barium, strontium) more distinct.

2. HEATING IN THE CLOSED AND OPEN TUBES

495. The tubes are useful chiefly for examining minerals containing volatile ingredients, given off at the temperature of the gas flame.

In the case of the *closed tube*, the heating goes on practically uninfluenced by the air present, since this is driven out of the tube in the early stages of the process. In the *open tube*, on the other hand, a continual stream of hot air, that is, of hot oxygen, passes over the assay, tending to produce oxidation and hence often materially changing the result.

496. Closed Tube. — A small fragment is inserted, or a small amount of the powdered mineral — in this case with care not to soil the sides of the tube — and heat is applied by means of the ordinary Bunsen flame. The presence of a volatile ingredient is ordinarily shown by the deposit, or *sublimate*, upon the tube at some distance above the assay where the tube is relatively cool.

Independent of this, other phenomena may be noted, namely: *decrepitation*, as shown by fluorite, calcite, etc.; *glowing*, as exhibited by gadolinite; *phosphorescence*, of which fluorite is an example; *change of color* (limonite), and here the color of the mineral should be noted both when hot, and again after cooling; *fusion;* giving off *oxygen*, as mercuric oxide; yielding *acid* or *alkaline vapors*, which should be tested by inserting a strip of moistened litmus or turmeric paper in the tube.

Of the *sublimates* which form in the tube, the following are those with which it is most important to be familiar:

Substance	**Sublimate in the Closed Tube**
Water (H_2O)	Colorless liquid drops.
Sulphur (S)	Red to deep yellow, liquid; pale yellow, solid.
Tellurium dioxide (TeO_2)	Pale yellow to colorless, liquid; colorless or white, solid.
Arsenic sulphide (As_2S_3)	Dark red, liquid; reddish yellow, solid.
Antimony oxysulphide (Sb_2S_2O)	Black to reddish brown on cooling, solid.
Arsenic (As)	Black, brilliant metallic to gray crystalline, solid
Mercury sulphide (HgS)	Deep black, red when rubbed very fine.
Mercury (Hg)	Gray metallic globules.

In addition to the above: Tellurium gives black fusible globules; selenium the same, but in part dark red when very small; the chloride of lead and oxides of arsenic and antimony give white solid sublimates.

497. Open Tube. — The small fragment is placed in the tube about an inch from the lower end, the tube being slightly inclined (say 20°), but not enough to cause the mineral to slip out, and heat applied beneath. The current of air passing upward through the tube during the heating process has an oxidizing effect. The special phenomena to be observed are the formation of a *sublimate* and the *odor* of the escaping gases. The acid or alkaline character of the vapors is tested for in the same way as with the closed tube. The most common gas to be obtained in this way is sulphur dioxide, SO_2, when sulphides are being oxidized. This gas is to be recognized by its irritating, pungent odor and its acid reaction upon moistened blue litmus paper.

The more important sublimates are as follows:

Substance	Sublimate in the Open Tube
Arsenic trioxide (As_2O_3)......	White, crystalline, volatile.
Antimony antimonate (Sb_2O_4)	Straw-yellow, hot; white, cold. Infusible, non-volatile, amorphous, settling along bottom of tube. Obtained from compounds containing sulphur as stibnite, also the sulphantimonites (*e.g.*, bournonite) as dense white fumes. Usually accompanied by the following:
Antimony trioxide (Sb_2O_3)...	White, crystalline, slowly volatile, forming as a ring on walls of tube.
Tellurium dioxide (TeO_2)....	White to pale yellow globules.
Selenium dioxide (SeO_2)......	White, crystalline, volatile.
Molybdenum trioxide (MoO_3)	Pale yellow, hot; white, cold.
Mercury (Hg)..............	Gray metallic globules, easily united by rubbing.

It is also to be noted that if the heating process is too rapid for full oxidation, sublimates, like those of the closed tubes, may be formed, especially with sulphur (yellow), arsenic (black), arsenic sulphide (orange), mercury sulphide (black), antimony oxysulphide (black to reddish brown).

3. HEATING ON CHARCOAL

498. The fragment (or powder) to be examined is placed near one end of the piece and this so held that the flame passes along its length. If the mineral decrepitates, it may be powdered, mixed with water, and then the material employed as a paste.

The reducing flame is employed if it is desired to *reduce* a metal (*e.g.*, silver, copper) from its ores: this is the common case. If, however, the mineral is to be *roasted*, that is, heated in contact with the air so as to oxidize and volatilize, for example, the sulphur, arsenic, antimony present, the oxidizing flame is needed and the mineral should be in powder and spread out.

The points to be noted are as follows:

(*a*) The *odor* given off after short heating. In this way the presence of *sulphur*, *arsenic* (garlic or alliaceous odor), and *selenium* (odor of decayed horseradish) may be recognized.

(*b*) *Fusion.* — In the case of the salts of the alkalies the fused mass is absorbed into the charcoal; this is also true, after long heating, of the carbonates and sulphates of barium and strontium. (Art. **501**.)

(*c*) *The Sublimate.* — By this means the presence of many of the metals may be determined. The color of the sublimate, both near the assay (N) and at a distance (D), as also when hot and when cold, is to be noted.

The important sublimates are the following:

Substance	Sublimate on Charcoal
Arsenic trioxide (As_2O_3)	White, very volatile, distant from the assay; also garlic fumes.
Antimony oxides (Sb_2O_3 and Sb_2O_4)	Dense white, volatile; forms near the assay.
Zinc oxide (ZnO)	Canary-yellow, hot; white, cold; moistened with cobalt nitrate and ignited (O.F.) becomes green.
Molybdenum trioxide (MoO_3)	Pale yellow, hot; yellow, cold; touched for a moment with the R.F. becomes azure-blue. Also a copper-red sublimate (MoO_2) near the assay.
Lead oxide (PbO)	Dark yellow, hot; pale yellow, cold. Also (from sulphides) dense white (resembling antimony), a mixture of oxide, sulphite, and sulphate of lead.
Bismuth trioxide (Bi_2O_3)	Dark orange-yellow (N), paler on cooling; also bluish white (D). See further, p. 338.
Cadmium oxide (CdO)	Nearly black to reddish brown (N) and orange-yellow (D); often iridescent.

To the above are also to be added the following:

Selenium dioxide, SeO_2, sublimate steel-gray (N) to white tinged with red (D); touched with R.F. gives an azure-blue flame; also an offensive selenium odor.

Tellurium dioxide, TeO_2, sublimate dense white (N) to gray (D); in R.F. volatilizes with green flame.

Tin dioxide, SnO_2, sublimate faint yellow hot to white cold; becomes bluish green when moistened with cobalt solution and ignited.

Silver (with lead and antimony), sublimate reddish

(*d*) *The Infusible Residue.* — This may (1) glow brightly in the O.F., indicating the presence of calcium, strontium, magnesium, zirconium, zinc, or tin. (2) It may give an alkaline reaction after ignition: alkaline earths. (3) It may be magnetic, showing the presence of iron (or nickel). (4) It may yield a globule or mass of a metal (Art. **499**).

499. Reduction on Charcoal. — In many cases the reducing flame alone suffices on charcoal to separate the metal from the volatile element present, with the result of giving a globule or metallic mass. Thus silver is obtained from argentite (Ag_2S) and cerargyrite ($AgCl$); copper from chalcocite (Cu_2S) and cuprite (Cu_2O), etc. The process of reduction is always facilitated by the use of sodium carbonate or borax as a flux, and this is in many cases (sulpharsenites, etc.) essential.

The finely pulverized mineral is intimately mixed with two or three times its volume of soda, and a drop of water added to form a paste. This is placed in a cavity in the charcoal, and subjected to a strong reducing flame. More soda is added as that present sinks into the coal, and, after the process has been continued some time, a metallic globule is often visible, or a number of them, which can be removed and separately examined. If not distinct, the remainder of the flux, the assay, and the surrounding coal are cut out with a knife, and the whole ground up in a mortar, with the addition of a little water. The charcoal is carefully washed away and the metallic globules, flattened out by the process, remain behind. Some metallic oxides are very readily reduced, as lead, while others, as copper and tin, require considerable skill and care.

The metals obtained (in globules or as a metallic mass) may be: *copper*, color red; *bismuth*, lead-gray, brittle; *gold*, yellow, not soluble in nitric acid; *silver*, white, soluble in nitric acid, the solution giving a silver chloride precipitate (p. 340); *tin*, white, harder than silver, soluble in nitric acid with separation of white powder (SnO_2); *lead*, lead-gray (oxidizing), soft and fusible. The coatings (see the list of sublimates above) often serve to identify the metal present.

500. Detection of Sulphur in Sulphates. — By means of soda on charcoal the presence of sulphur in the sulphates may be shown, in the following manner. Fuse the powdered mineral with soda and charcoal dust. The latter acting as a strong reducing agent changes the sulphate to a sulphide with the formation of sodium sulphide. When the fused mass is placed with a drop of water upon a clean silver surface a black or yellow stain of silver sulphide will be formed. A similar reaction would of course be obtained from a sulphide. The latter can however be readily distinguished by roasting in the open tube or upon charcoal and noting the formation of SO_2.

4. Treatment on the Platinum Wire

501. Use of the Fluxes. — The three common fluxes are borax, salt of phosphorus, and carbonate of soda (p. 330). They are generally used with the platinum wire, less often on charcoal (see p. 335). If the wire is employed it must have a small loop at the end; this is heated to redness and dipped into the powdered flux, and the adhering particles fused to a bead; this operation is repeated until the loop is filled. Sometimes in the use of soda the wire may at first be moistened a little to cause it to adhere.

When the bead is ready, it is, while hot, brought in contact with the powdered mineral, some of which will adhere to it, and then the heating process may be continued. Very little of the mineral is in general required, and the experiment should be commenced with a minute quantity and more added if necessary. The bead must be heated successively first in the oxidizing flame (O.F.) and then in the reducing flame (R.F.), and in each case the color noted when *hot* and when *cold*. The phenomena connected with fusion, if it takes place, must also be observed.

Minerals containing sulphur or arsenic, or both, must be first *roasted* (see p. 334) till these substances have been volatilized. If too much of the mineral has been added and the bead is hence too opaque to show the color, it may, while hot, be flattened out with the hammer, or drawn out into a wire, or part of it may be removed and the remainder diluted with more of the flux.

With salt of phosphorus, the wire should be held above the flame so that the escaping gases may support the bead; this is continued till quiet fusion is attained.

It is to be noted that the colors vary much with the amount of material present; they are also modified by the presence of other metals.

502. Borax. — The following list enumerates the different colored beads obtained with borax, both in the oxidizing (O.F.) and reducing flames (R.F.), and also the metals to the presence of whose oxides the colors are due. Compare further the reactions given in the list of elements (Art. **504**).

Color in Borax Bead	**Substance**
	1. Oxidizing Flame
Colorless, or opaque white...	Silica, calcium, aluminium; also silver, zinc, etc.
	Iron, cold — (pale yellow, hot, if in small amount).
Red, red-brown to brown....	Chromium (CrO_3), hot — (yellowish green, cold).
	Manganese (Mn_2O_3), amethystine-red — (violet, hot).
	Iron (Fe_2O_3), hot — (yellow, cold) — if saturated.
	Nickel (NiO) red-brown to brown, cold — (violet, hot).
	Uranium (UO_3), hot — (yellow, cold).
Green....................	Copper (CuO), hot — (blue, cold, or bluish green if highly saturated).
	Chromium (CrO_3), yellowish green, cold — (red, hot).

Yellow	Iron (Fe_2O_3), hot — (pale yellow to colorless, cold) — but red-brown and yellow if saturated. Uranium (UO_3), hot, if in small amount; paler on cooling. Chromium (CrO_3), hot and in small amount — (yellowish green, cold).
Blue	Cobalt (CoO), hot and cold. Copper (CuO), cold if highly saturated — (green, hot).
Violet	Nickel (NiO), hot — (red-brown, cold). Manganese (Mn_2O_3), hot — (amethystine-red, cold).

2. REDUCING FLAME (R.F.)

Colorless	Manganese (MnO), or a faint rose color.
Red	Copper (Cu_2O, with Cu), opaque red.
Green	Iron (FeO), bottle-green. Chromium (Cr_2O_3), emerald-green. Uranium (U_2O_3), yellowish green if saturated.
Blue	Cobalt (CoO), hot and cold.
Gray, turbid	Nickel (Ni).

503. Salt of Phosphorus. — This flux gives for the most part reactions similar to those obtained with borax. The only cases enumerated here are those which are distinct, and hence those where the flux is a good test.

With *silicates* this flux forms a glass in which the bases of the silicate are dissolved, but the silica itself is left insoluble. It appears as a skeleton readily seen floating about in the melted bead.

The colors of the beads, and the metals to whose oxides these are due, are:

Color	Substance
Red	Chromium in O.F., hot — (fine green when cold).
Green	Chromium in O.F. and R.F., when cold — (red in O.F., hot). Molybdenum in R.F., dirty green, hot; fine green, cold — (yellow-green in O.F.). Uranium in R.F., cold; yellow-green, hot. Vanadium, chrome-green in R.F., cold — (brownish red, hot). In O.F., dark yellow, hot, paler on cooling.
Yellow	Molybdenum, yellowish green in O.F., hot, paler on cooling — (in R.F., dirty green, hot; fine green, cold). Uranium in O.F., hot; yellowish green, cold — (in R.F., yellowish green, hot; green, cold). Vanadium in O.F., dark yellow, hot, paler on cooling — (in R.F., brownish red, hot; chrome-green, cold).
Violet	Titanium (TiO_2) in R.F., yellow, hot. (Also in O.F., yellow, hot; colorless, cold.)

CHARACTERISTIC REACTIONS OF THE IMPORTANT ELEMENTS AND OF SOME OF THEIR COMPOUNDS

504. The following list contains the most characteristic reactions, chiefly before the blowpipe and in some cases also in the wet way, of the different elements and their oxides. It is desirable for every student to gain familiarity with them by trial with as many minerals as possible. Many of them have already been briefly mentioned in the preceding pages. For a thoroughly full description of these and other characteristic tests (blowpipe and otherwise) reference should be made to the volume by Brush and Penfield referred to on p. 329.

It is to be remembered that while the reaction of a single substance may be perfectly distinct if alone, the presence of other substances may more or less entirely obscure these reactions; it is consequently obvious that in the actual examination of minerals precautions have to be taken, and special methods have to be devised, to overcome the difficulty arising from this cause. These will be gathered from the "pyrognostic characters" (**Pyr.**) given in connection with the description of each species in the Fourth Part of this work.

Aluminium. — The presence of aluminium in most infusible minerals, containing a considerable amount, may be detected by the blue color which they assume when, after being heated, they are moistened with cobalt solution and again ignited (*e.g.*, cyanite, andalusite, etc.). Very hard minerals (as corundum) must be first finely pulverized. The test is not conclusive with fusible minerals since a glass colored blue by cobalt oxide may be formed. It is to be noted that the infusible calamine (zinc silicate) also assumes a blue color when treated with cobalt nitrate. From solutions aluminium will be precipitated as a flocculent white or colorless precipitate on the addition of ammonium hydroxide in excess.

Antimony. — Antimonial minerals roasted on charcoal give dense white odorless fumes; metallic antimony and its sulphur compounds give in the open tube a white sublimate of oxide of antimony (see p. 334). Antimony sulphide (stibnite), also many sulphantimonites, give in a strong heat in the closed tube a sublimate of antimony oxysulphide, black when hot, brown-red when cold. See also p. 333.

In nitric acid, compounds containing antimony deposit white insoluble metantimonic acid.

Arsenic. — Arsenides, sulpharsenites, etc., give off fumes when roasted on charcoal, usually easily recognized by their peculiar garlic odor. In the open tube they give a white, volatile, crystalline sublimate of arsenic trioxide. In the closed tube arsenic sulphide gives a sublimate dark brown-red when hot, and red or reddish yellow when cold; arsenic and some arsenides yield a black mirror of metallic arsenic in the closed tube. In arsenates the arsenic can be detected by the garlic odor yielded when a mixture of the powdered mineral with charcoal dust and sodium carbonate is heated (R.F.) on charcoal.

Barium. — A yellowish green coloration of the flame is given by all barium salts, except the silicates; an alkaline reaction is usually obtained after intense ignition.

In solution the presence of barium is proved by the heavy white precipitate ($BaSO_4$) formed upon the addition of dilute sulphuric acid.

Bismuth. — On charcoal alone, or better with soda, bismuth gives a very characteristic orange-yellow sublimate; brittle globules of the reduced metal are also obtained (with soda). Also when treated with 3 or 4 times the volume of a mixture in equal parts of potassium iodide and sulphur, and fused on charcoal, a beautiful red sublimate of bismuth iodide is obtained; near the mineral the coating is yellow.

Boron. — Many compounds containing boron (borates, also the silicates, datolite, danburite, etc.) tinge the flame intense yellowish green, especially if moistened with sulphuric acid. For some silicates (as tourmaline) the best method is to mix the powdered mineral with one part powdered fluorite and two parts potassium bisulphate. The mixture is moistened and placed on platinum wire. At the moment of fusion the green color appears, but lasts but an instant.

A dilute hydrochloric acid solution containing boron gives a reddish brown color to turmeric paper which has been moistened with it and then dried at 100°; the color changes to black when ammonia is poured on the paper.

Calcium. — Many calcium minerals (carbonates, sulphates, etc.) give an alkaline reaction on turmeric paper after being ignited. A yellowish red color is given to the flame by some compounds (*e.g.*, calcite after moistening with HCl); the strontium flame is a much deeper red.

In weakly acid or alkaline solutions calcium is precipitated as oxalate by the addition of ammonium oxalate.

Cadmium. — On charcoal with soda, compounds of cadmium give a characteristic sublimate of the reddish brown oxide.

Carbonates. — All carbonates effervesce with *dilute* hydrochloric acid, yielding the odorless gas CO_2 (*e.g.*, calcite); many require to be pulverized, and some need the addition of heat (dolomite, siderite). Carbonates of lead should be tested with nitric acid.

Chlorides. — If a small portion of a mineral containing chlorine (a chloride, also pyromorphite, etc.) is added to the bead of salt of phosphorus, saturated with copper oxide, the bead when heated is instantly surrounded with an intense purplish flame of copper chloride.

In solution chlorine gives with silver nitrate a white curdy precipitate of silver chloride which darkens in color on exposure to the light; it is insoluble in nitric acid, but entirely soluble in ammonia.

Chromium. — Chromium gives with borax a bead which (O.F.) is yellow to red (hot) and yellowish green (cold) and R.F. a fine emerald-green. With salt of phosphorus in O.F. the bead is dirty green (hot) and clear green (cold); in R.F. the same. Cf. Vanadium beyond (also pp. 336, 337).

Cobalt. — A beautiful blue bead is obtained with borax in both flames from minerals containing cobalt. Where sulphur or arsenic is present the mineral should first be thoroughly roasted on charcoal.

Copper. — On charcoal, at least with soda, metallic copper can be reduced from most of its compounds. In the case of sulphides the powdered mineral should be roasted first in order to eliminate the major part of the sulphur before fusion with soda. With borax it gives (O.F.) a green bead when hot, becoming blue when cold; also (R.F.), if saturated, an opaque red bead containing Cu_2O and often Cu is obtained. Copper chloride, obtained by moistening the mineral with hydrochloric acid (in the case of sulphides the mineral should be previously roasted) yields a vivid azure-blue flame; copper oxide gives a green flame.

Most metallic compounds are soluble in nitric acid. Ammonia in excess produces an intense blue color in the solution.

Fluorine. — Heated in the closed tube with potassium bisulphate and powdered glass produces a white sublimate of SiO_2. This sublimate and the hydrofluosilicic acid present form a volatile combination. But if the lower end of the tube is broken off and the open tube then dipped in a test tube of water so that the acid is removed, the deposit of SiO_2 which will appear when the tube is dried will be found to be no longer volatile.

Heated gently in a platinum crucible with sulphuric acid, many compounds (*e.g.*, fluorite) give off hydrofluoric acid, which corrodes the exposed parts of a glass plate placed over it which has been coated with wax and then scratched.

Iron. — Minerals which contain even a small amount of iron yield a magnetic mass when heated in the reducing flame. With borax iron gives a bead (O.F.) which is yellow to brownish red (according to quantity) while hot, but is colorless to yellow on cooling; R.F. becomes bottle-green (see pp. 336, 337).

Lead. — With soda on charcoal a malleable globule of metallic lead is obtained from lead compounds; the coating has a yellow color near the assay; the sulphide gives also a white coating ($PbSO_3$) farther off (p. 335). On being touched with the reducing flame the coating disappears, tingeing the flame azure-blue.

In solutions dilute sulphuric acid gives a white precipitate of lead sulphate; when delicacy is required an excess of the acid is added, the solution evaporated to dryness, and water added; the lead sulphate, if present, will then be left as a residue.

Lithium. — Lithium gives an intense carmine-red to the outer flame, the color somewhat resembling that of the strontium flame but is deeper; in very small quantities it is evident in the spectroscope.

Magnesium. — Moistened, after heating, with cobalt nitrate and again ignited, a pink color is obtained from some infusible compounds of magnesium (*e.g.*, brucite). In solution the addition of ammonium hydroxide in large excess and a little hydrogen sodium phosphate produces a white granular precipitate of NH_4MgPO_4. Elements precipitated by ammonium hydroxide or ammonium oxalate should be removed first.

Manganese. — With borax manganese gives a bead violet-red (O.F.), and colorless (R.F.). With soda (O.F.) it gives a bluish green bead; this reaction is very delicate and may be relied upon, even in presence of almost any other metal.

Mercury. — In the closed tube a sublimate of metallic mercury is yielded when the mineral is heated with dry sodium carbonate. In the open tube the sulphide gives a mirror of metallic mercury; in the closed tube a black lusterless sublimate of HgS, red when rubbed, is obtained.

Molybdenum. — On charcoal molybdenum sulphide gives near the assay a copper-red stain (O.F.), and beyond a white coating of the oxide; the former becomes azure-blue when for a moment touched with the R.F. The salt of phosphorus bead (O.F.) is yellowish green (hot) and nearly colorless (cold); also (R.F.) a fine green.

Nickel. — With borax, nickel oxide gives a bead which (O.F.) is violet when hot and red-brown on cooling; (R.F.) the glass becomes gray and turbid from the separation of metallic nickel.

Niobium (Columbium). — An acid solution boiled with metallic tin gives a blue color. The reactions with the fluxes are not very satisfactory.

Nitrates. — These detonate when heated on charcoal. Heated in a tube with sulphuric acid they give off red fumes of nitrogen dioxide (NO_2).

Phosphorus. — Most phosphates impart a green color to the flame, especially after having been moistened with sulphuric acid, though this test may be rendered unsatisfactory by the presence of other coloring agents. If they are used in the closed tube with a fragment of metallic magnesium or sodium, and afterward moistened with water, phosphureted hydrogen is given off, recognizable by its disagreeable odor.

A few drops of a nitric acid solution, containing phosphoric acid, produce in a solution of ammonium molybdate a pulverulent yellow precipitate of ammonium phosphomolybdate.

Potassium. — Potash imparts a violet color to the flame when alone. The flame is best observed through a blue glass filter which will eliminate the sodium flame color which will almost invariably be present. It is best detected in small quantities, or when soda or lithia is present, by the aid of the spectroscope. See also p. 333.

Selenium. — On charcoal selenium fuses easily, giving off brown fumes with a peculiar disagreeable organic odor; the sublimate on charcoal is volatile, and when heated (R.F.) gives a fine azure-blue flame.

Silicon. — A small fragment of a silicate in the salt of phosphorus bead leaves a skeleton of silica, the bases being dissolved.

If a silicate in a fine powder is fused with sodium carbonate and the mass then dissolved in hydrochloric acid and evaporated to dryness, the silica separates as a gelatinous mass and on evaporation to dryness is made insoluble. When strong hydrochloric acid is added and then water to the dry residue in the test tube, the bases are dissolved and the silica left behind.

Many silicates, especially those which are hydrous, are decomposed by strong hydrochloric acid, the silica separating as a powder or, after evaporation, as a jelly (see p. 328).

Silver. — On charcoal in O.F. silver gives a brown coating. A globule of metallic silver may generally be obtained by heating on charcoal in O.F., especially if soda is added. Under some circumstances it is desirable to have recourse to cupellation.

From a solution containing any salt of silver, the insoluble chloride is thrown down when hydrochloric acid is added. This precipitate is insoluble in acid or water, but entirely so in ammonia. It changes color on exposure to the light.

Strontium. — Compounds of strontium are usually recognized by the fine crimson-red which they give to the blowpipe flame; many yield an alkaline reaction after ignition. (Cf. barium.)

Sodium. — Compounds containing sodium in large amount give a strong yellow flame.

Sulphur, Sulphides, Sulphates. — In the closed tube some sulphides give off sulphur; in the open tube they yield sulphur dioxide, which has a characteristic odor and reddens a strip of moistened litmus paper. In small quantities, or in sulphates, sulphur is best detected by fusion on charcoal with soda and charcoal dust. The fused mass, when sodium sulphide has thus been formed, is placed on a clean silver coin and moistened; a distinct black stain on the silver is thus obtained (the precaution mentioned on p. 336 must be exercised).

A solution of a sulphate in hydrochloric acid gives with barium chloride a white insoluble precipitate of barium sulphate.

Tellurium. — Tellurides heated in the open tube give a white or grayish sublimate, fusible to colorless drops (p. 334). On charcoal they give a white coating and color the R.F. green.

Tin. — Minerals containing tin (*e.g.*, cassiterite), when heated on charcoal with soda or potassium cyanide, yield metallic tin in minute globules; these are malleable, but harder than silver. Dissolved in nitric acid, white insoluble stannic oxide separates out.

Titanium. — Titanium gives in the R.F. with salt of phosphorus a bead which is violet when cold. Fused with sodium carbonate and dissolved with hydrochloric acid, and heated with a piece of metallic tin, the liquid takes a violet color, especially after partial evaporation.

Tungsten. — Tungsten oxide gives a blue color to the salt of phosphorus bead (R.F.). Fused and treated as titanium (see above) with the addition of zinc instead of tin, gives a fine blue color.

Uranium. — Uranium compounds give to the salt of phosphorus bead (O.F.) a greenish yellow bead when cool; also (R.F.) a fine green on cooling (p. 337).

Vanadium. — With borax (O.F.) vanadates give a bead yellow (hot) changing to yellowish green and nearly colorless (cold); also (R.F.) dirty green (hot), fine green (cold). With salt of phosphorus (O.F.) a yellow to amber color (thus differing from chromium); also (R.F.) fine green (cold).

Zinc. — On charcoal in the reducing flame compounds of zinc give a coating which is yellow while hot and white on cooling, and moistened by the cobalt solution and again heated becomes a fine green. Note, however, that the zinc silicate (calamine) becomes blue when heated after moistening with cobalt solution.

Zirconium. — A dilute hydrochloric acid solution, containing zirconium, imparts an orange-yellow color to turmeric paper, moistened by the solution.

DETERMINATIVE MINERALOGY

505. Determinative Mineralogy may be properly considered under the general head of Chemical Mineralogy, since the determination of minerals depends mostly upon chemical tests. But crystallographic and all the physical characters have also to be carefully observed.

There is but one exhaustive way in which the identity of an unknown mineral may in all cases be fixed beyond question, and that is by the use of a complete set of determinative tables. By means of such tables the mineral in hand is referred successively from a general group into a more special one, until at last all other species have been eliminated, and the identity of the one given is beyond doubt.

A careful preliminary examination of the unknown mineral should, however, always be made before final recourse is had to the tables. This examination will often suffice to show what the mineral in hand is, and in any case it should not be omitted, since it is only in this way that a practical familiarity with the appearance and characters of minerals can be gained.

The student will naturally take note first of those characters which are at once obvious to the senses, that is: *crystalline form*, if distinct; *general structure, cleavage, fracture, luster, color* (and *streak*), *feel;* also, if the specimen is not too small, the apparent weight will suggest something as to the *specific gravity.* The characters named are of very unequal importance. Structure, if crystals are not present, and fracture are generally unessential except in distinguishing varieties; color and luster are essential with metallic, but generally very unimportant with nonmetallic, minerals. *Streak* is of importance only with colored minerals and those of metallic luster (p. 247). Crystalline form and cleavage are of the highest importance, but may require careful study.

The first trial should be the determination of the *hardness* (for which end the pocket-knife is often sufficient in experienced hands). The second trial should be the determination of the *specific gravity.* Treatment of the powdered mineral with acids may come next; by this means (see pp. 328, 329) a carbonate is readily identified, and also other results obtained. Then should follow blowpipe trials, to ascertain the *fusibility;* the *color* given to the flame, if any; the character of the *sublimate* given off in the tubes and on charcoal; the metal reduced on the latter; the reactions with the *fluxes,* and other points as explained in the preceding pages.

How much the observer learns in the above way, in regard to the nature of his mineral, depends upon his knowledge of the characters of minerals in general, and upon his familiarity with the chemical behavior of the various elementary substances with reagents and before the blowpipe (pp. 338 to 341). If the results of such a preliminary examination are sufficiently definite to suggest that the mineral in hand is one of a small number of species, reference may be made to their full description in Part. IV of this work for the final decision.

A number of tables, in which the minerals included are arranged according to their crystalline and physical characters, are added in the Appendix. They will in many cases aid the observer in reaching a conclusion in regard to a specimen in hand.

The first of these tables gives lists of minerals arranged primarily according to their principle basic elements and secondarily according to their acid radicals.

The second of these tables is intended to include all well-defined species, grouped according to the crystalline system to which they belong and arranged under each system in the order of their specific gravities; the hardness is also added in each case. The relative importance of the individual species is shown by the type employed. Following this are minor tables enumerating species characterized by some one of the prominent crystalline forms; that is, those crystallizing in cubes, octahedrons, rhombohedrons, etc. Other tables give the names of species prominent because of their cleavage; structure of different types; hardness; luster; the various colors, etc. The student is recommended to make frequent use of these tables, not simply for aid in the identification of specimens, but rather because they will help him in the difficult task of learning the prominent characters of the more important minerals.