PRACTICAL INSTRUCTIONS

IN

QUANTITATIVE ASSAYING WITH THE BLOWPIPE.

CONTAINING ALSO READILY APPLIED

QUALITATIVE BLOWPIPE TESTS.

FOR THE PROSPECTOR, MINER, AND ASSAYER,
AND FOR USE IN COLLEGES.

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BY

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PREFACE.

THE Author has long believed that a practical, simple, and at the same time cheap work on the blowpipe, that included quantitative assaying, would be appreciated by many who are interested in "mineral." This book is the result of that belief.

The methods of assaying by means of the blowpipe furnace, as advocated herein, I can recommend as more certain and practical in many cases than the old way. A larger quantity of ore is operated on, and the outcome does not depend so much on the skill, or want of skill, of the assayer.

Wet methods I have omitted: they to a great extent destroy some of the distinguishing merits of the blowpipe,—its quickness and simplicity. Moreover, when wet methods are combined with the use of the blowpipe it is very often either for purposes of Determinative Mineralogy or to detect substances that have little or no commercial value. The only use of acids in this book is in *parting* in the gold assay, and to aid in obtaining some flame-colorations.

Prof. H. B. Cornwall's most excellent and valuable work on Qualitative and Quantitative Blowpipe Analysis can be consulted with profit, especially in the department of Qualitative Tests.

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Jan. 16, 1894.

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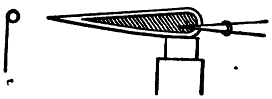
QUANTITATIVE ASSAYING WITH THE BLOWPIPE.

CHAPTER I.

USE OF BLOWPIPE.—APPARATUS.

1. By the proper use of the blowpipe two very different chemical reactions take place in the substance on which we operate.

Oxidation and Reduction (the giving and taking away of oxygen).—The Oxidizing Flame (abbreviated O. F.) is produced by holding the jet of the blowpipe just above the wick and inserting it into the flame about one third of the breadth of the wick, blowing parallel to the wick, and rather strongly, making a pointed blue flame rather long and cone-shaped. The assay (or substance we are submitting to the action of the blowpipe flame) is held beyond the point of the extreme nearly invisible yellowish flame that ter-

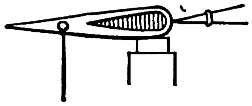


Oxidising Flame

FIG. 1.

minates the blue (Fig. 1). The blue is the "inner flame."

The Reducing Flame (abbreviated R. F.).—The blowpipe jet is lifted higher above the wick than in the O. F. Instead of being inserted *in* the flame, as when using the O. F., the jet barely touches the side of the flame. The blast must not be as strong as with the O. F., and a thick yellow flame is produced. The assay is held *in* the thick yellow reducing flame, beyond the blue, which now is shorter than in the O. F. (Fig. 2).



Reducing Flame
FIG. 2.

2. *Cautions.*—The R. F. is harder to produce well than the O. F. Do not blow too hard: it might have an oxidizing effect. Do not let the blue flame play on the assay: keep it in the yellow. Keep soot from forming on the assay: it often will, with too weak blowing.

3. A test for the R. F. mentioned by Berzelius is good. Tin oxidizes (combines with oxygen) easily. Put a small piece of tin in a cavity on a piece of charcoal. Blow on the tin with the R. F. The longer you can keep the tin bright, the better control you have over the R. F., as the tin will cover itself with a powdery-looking oxide if the R. F. is not pure.

4. In using the blowpipe the blast must be kept up uninterruptedly, blowing all the time without stopping to inhale. This seems hard, but it is easily acquired. Printed directions are

not of much use. If the beginner can have the method of blowing shown him by some one who is proficient, he can by practice easily acquire it. Suffice it to say, that a good breath is taken, the cheeks are puffed out like bellows, and the air contained between them is sufficient to continue the blast without stopping, while air in inhaled through the nose. *Don't stop to think about the inhaling.* Forget it, and think of the assay. The blowing will proceed mechanically and without trouble.

5. *Blowpipes.*—The platinum-pointed Plattner blowpipe is the one generally recommended in books, while the cheaper Black's blowpipe is only mentioned as a kind of makeshift. I have, on the contrary, found Black's blowpipe capable of all the work, including quantitative assaying, that the more expensive Plattner blowpipe can do. The Black (Fig. 3) has the merit of simplicity and cheapness, costing twenty-five cents to the Plattner's two dollars or more. The jet (the "tip" through which the air issues) is movable, and is made of brass. These jets are simple and durable. For quantitative work with the coal crucible the aperture of the jet should be *somewhat*

FIG. 3.—BLACK'S BLOWPIPE.

larger than for ordinary qualitative work, but the jet with larger orifice can be used for both.

Soot collects on the jet. With the Plattner platinum-pointed jet it can be burned off; but the soot can be easily cleaned from the brass jet with a stiff brush.

Reverse the blowpipe from time to time, and let the moisture which has condensed in it run out. The lips get tired more quickly if a mouthpiece is not used. But many of the mouthpieces for sale are too small, or badly shaped, and are worse than none. One of the best shapes is Fletcher's * (sec. 108).

The author in using Black's blowpipe (which as furnished generally has no mouthpiece) presses the tube against the teeth; this will be found to be not as tiresome as placing the tube between the teeth.

In testing for flame-coloration be sure the brass jet is clean, or it may impart a color to the flame.

Black's blowpipe may be had from Taylor & Co., San Francisco, Cal., with a mouthpiece fitted on at a small additional cost.

6. (*a*) *Lamps and Fuel.*—Plattner's lamp has the advantage of cleanliness; but it is expensive, as is the fuel used—rape-seed oil, or walnut oil. The use of oil is against its portability. Moreover, in many of those for sale the wick is so far from the side of the lamp that good work with the R. F. cannot be done. Fletcher's tin tallow lamp * (Fig. 4) I can recommend, as I have found it effective, and it is cheap, and the fuel—tallow, candles

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* These and other implements referred to as Fletcher's are made by a well-known dealer in assayers' supplies in England

broken up, almost any kind of solid fat—easily obtained. In using this lamp, first melt the tallow on top by blowing with the blowpipe a blast from the alcohol lamp, until the grease is well melted, then light the wick. Take a small piece of grease in the lamp forceps, and hold it over the wick until it melts. A good flame is thus obtained. The grease on top must be kept melted; if it shows a tendency to become solid in time, it must be kept melted by an occasional blast from the alcohol lamp.

FIG. 4.—TALLOW LAMP.

(*b*) An *Alcohol Lamp* is used in the blowpipe furnace assay. It is needed for many other purposes, as when glass tubes are used, etc.

(*c*) *Wicks.*—The wick for the tallow lamp should be kept clean and trimmed. It should be of soft material, affording an easy flow to the melted grease. It should be rather close in texture, and must not be apt to char. For a good R. F. the wick should rise $\frac{3}{4}$ in. above the top of the wick tube.

7. (*a*) *Supports.*—Charcoal is best of soft wood well burnt. It is well to try the flame on the coal, before placing on it the substance to be assayed, as I have known coal to snap with such force as to throw pieces several feet. The coal can be purchased sawed into strips ready for use.

(*b*) *Platinum Wire* is used as a support for the

" glass fluxes " (borax and " salt of phosphorus "). I have found the best and handiest holder for the wire (as stated by Ross) to be a right-line pen used in this manner (Fig. 5). It has the advantage of enabling one to make two beads without adjusting the wire. A right-line pen can be bought at any stationer's store.

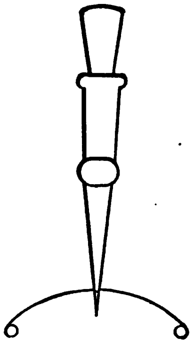

FIG. 5.—RIGHT-LINE PEN HOLDING PLATINUM WIRE.

(*c*) Bead is the name given to the globule of borax or salt of phosphorus in which the assay is dissolved in certain operations. To form the bead, bend the end of the platinum wire round till it meets itself, making a loop about twice as large as can be formed by bending the wire around an ordinary match. Heat the wire to redness in the O. F., dip in the flux, heat again in the O. F., forming a *clear* limpid bead. Then while hot take up some of the powdered substance,—*about* as much as twice the head of a pin at *first*,—increasing the amount as may be necessary.

This does for borax. For salt of phosphorus (abbreviated to SPh) the method is modified. SPh is apt to run off the wire when first heated. To avoid this, hold the wire loop *above* the flame until the SPh has done bubbling. Then heat in O. F. as above.

(*d*) Wire should be perfectly clean before using, or mistakes may happen. (To detach a bead from the wire, break it by gently tapping with the hammer on the anvil.) Clean the wire by dipping in *dilute* hydrochloric acid.

8. *Mortars.*—Agate mortars are expensive, and can be dispensed with. I have found most serviceable an iron mortar, half-pint size. Ore can be pulverized in it fine enough to go through a sieve with 100 meshes to the inch, and this is sufficient. A small Wedgwood mortar, 3 in. in diameter, is useful in pulverizing slag from soda reductions, and for crushing certain fluxes, etc.

9. A flat piece of steel, 1½ × 1½ in., about, known as an *anvil*, will be needed. It should have an iron ring (a piece of gas-pipe 1½ in. high does well) or cylinder to keep fragments of the material that is being broken on the anvil from flying. A small blowpipe hammer (a hammer the size of a tack hammer, but heavier, will do) and a larger hammer (½-lb., or so) are required.

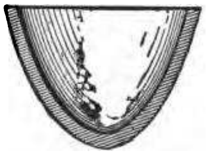
FIG. 6.

10. (*a*) *Clay Crucibles.*—These are small crucibles for the Furnace assay (Fig. 6).

(*b*) *Coal Crucibles* (for the ordinary blowpipe assay).—In using them the hole should be enlarged with the borer, and still more enlarged by cutting with a penknife so as to make a hole *about* half an inch wide at top. The *borer* is not only used for this purpose, but for boring cavities in the pieces of charcoal used as supports.

(*c*) *Clay Cylinder*, or Coal Crucible-holder.—A cylinder of clay with a depression at the end to receive the coal crucible.

(*d*) *Clay Capsules.*—Shallow clay dishes, used for roasting ore, and as scorifiers in the scorification of rich-lead in the silver and gold assay. For this latter purpose they are placed in the clay cylinder.

11. *Fletcher's Blowpipe Furnace.*—This is an admirable piece of blowpipe apparatus, and well-nigh indispensable for quantitative assays, as one is able to operate on a much larger amount of ore than by the old coal-crucible method; and this, when assaying ores containing the precious metals, is a great advantage, especially in the case of gold. A great objection to blowpipe assays has been the small amount of ore used, which in the case of poor ores made it difficult to get a satisfactory result. The coal square and holder formerly used with the clay crucibles is expensive and troublesome, and new coal squares had to be provided each time. The Fletcher furnace (Fig. 7) consists of a cylinder of refractory material and a cover. The furnace is $\frac{7}{8}$ in. high. It is cheap and durable. It costs twenty-five cents, and the author has made over three hundred assays in one of these furnaces before it had to be discarded. The furnace is made in two styles—with a hole for the flame in the side, and with the hole in the bottom for use with a Bunsen burner. *Only the one with the side hole* is suited for the method of assaying described in this

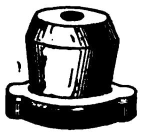

FIG. 7.*

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* The base is no part of the furnace; merely what it stands on.

work. The furnace has three small projections inside, which hold the clay crucible in place.

12. *Sieves.*—A tin box sieve of 100 holes to the inch, with a cover and cup at bottom, and an open sieve of 60 holes to the inch, are required.

13. An *Ivory Spoon.*—This is made of a certain size, and is a ready measure for some of the fluxes.

A *mixing capsule* of horn, in which the charge is mixed.

A *Test-lead Measure.*—A cylinder of glass, with graduations to measure test-lead instead of weighing, saves time, when its use is admissible.

A small cylinder of wood or *soda-paper form* is used to form the soda-paper cylinders required in the coal-crucible assay.

14. *Forceps.*—A forceps for use with the lamp (for the wick). An iron pincer 5 or 6 in. long for use with the blowpipe furnace. A small brass forceps for handling the weights.

A small steel forceps 3½ in., with fine points, will be in constant use.

A platinum-pointed forceps for testing flame-coloration and for fusibility tests.

15. *Cupels.*—In books on the blowpipe directions are generally given for making small cupels, especially for blowpipe use; but it is troublesome, and requires extra apparatus. I have found that ordinary assaying cupels of 1¼ in. or so in diameter answer every purpose. They can be used for a number of different cupellations, as the "rich-lead" button from a blowpipe assay is so small,

that a number of cupellations can be made on the same regular assay cupel by using a clean part of the cupel each time.

16. *Cupel Support.*—This can be "home-made." Take a piece of wood 5 or 6 in. long, and 2 in. at widest end, tapering to a handle for the hand to grasp. Near the edge at the widest end are driven four tacks, and against these the side of the cupel rests, the support being slightly inclined, when in use, so as to let the flame impinge squarely on the cupel.

17. A few small *porcelain crucibles*, 1 in. or so in diameter, for use "in parting" in the gold assay.

18. *Glass Tubes.*—These must be free from lead, otherwise mistakes may occur. *Open tubes*, used when the substance is to be heated in free contact with the air. Tubes 5 or 6 in. long and ¼ in. in diameter are handy. *Closed tubes*, used when the substance is to be heated out of contact with the air as much as possible. They can easily be made from the open tubes by fusing one end with the blowpipe flame. A matrass is a closed tube, with a bulb at the end, sometimes useful.

19. (*a*) A good *magnifying-glass* is constantly needed.

A small magnet, scissors for the wick, and a small file, a *camel's-hair* brush for cleaning pans of balance, one for brushing out ore from the mixing capsule, and a larger brush for brushing out the mortar, are also required.

(*b*) A "matrass-holder," or holder for the glass tubes when they are heated. Soda-paper for making the soda-paper cylinders.

20. (*a*) *Balances.*—This is the most expensive part of the blowpipe outfit. When such small objects are weighed, delicate, and consequently expensive, apparatus is required. Fortunately the gold and silver buttons from a blowpipe assay are measured by a scale (to be described).

Plattner's blowpipe balance can be obtained from $18 to $60, according to delicacy and finish. The best of them show $\frac{1}{10}$ of a milligramme ($\frac{1}{650}$ of a grain). But there is no need of weighing such small buttons, measurement in their case being better.

(*b*) Whittell's balance is the best moderate-price balance the author knows of. It is simple and does not easily get out of order, and is sensitive to $\frac{1}{10}$ milligramme. The weighing is done by substitution (see Fig. 8). Place the button in the pan, and balance it by sliding the weights. Then remove the button and place in the pan proper weights to balance. Including weights, the price is $20. It can be obtained from John Taylor & Co., San Francisco, Cal. Although excellent for weighing buttons, it is not well adapted to the weighing of ores like Plattner's or the next balance to be described.

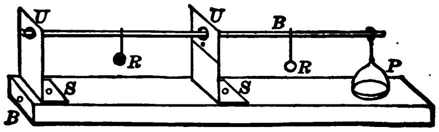

FIG. 8.—WHITTELL'S BALANCE.

(*c*) Henry Heil Chemical Co., St. Louis, Mo., furnish a "Pocket Assay Balance," which is serviceable (Fig. 9), for $15.30. It shows $\frac{1}{4}$ milligramme. With the amount of ore recommended for assay in this work—3grains—$\frac{1}{4}$ milligramme represents a little more than $\frac{12}{100000}$ of the amount assayed. Or, to be accurate, if 3 grains troy of ore were taken, the smallest button resulting from the assay that this balance would show would represent 37½ ounces of metal in a ton of ore. This is close enough for the base metals. But by making two or three assays and weighing the results together, a much smaller percentage would be shown. For the precious metals Plattner's Ivory Scale (for measuring the button) is used for anything less than this; and the assays of silver should *always* be made at least in duplicate, and when assaying gold ores by the blowpipe at least three or four different assays should be made. The above balance can also be used for weighing the charges of ores of the precious metals.

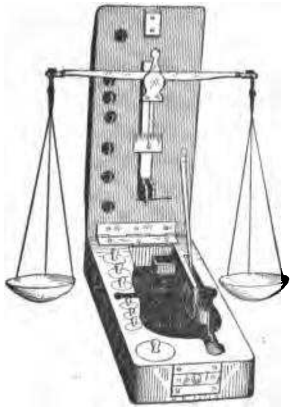
FIG. 9.

(*d*) For weighing fluxes and charges of the base metals a *good* hand balance will be found very useful. These balances can be obtained well made and reliable, and are of more accuracy than is often supposed. They are said to have a sensibility of $\frac{1}{10}$ to $\frac{1}{20}$ of a grain, but I have seen those that would turn with much less than this. They are made with horn pans and silk threads, also with pans hung by chains. These latter do not get out of order as quickly as the former, whose silk threads fray and pull out with much use, requiring new threads to be put in. When using these hand balances, hang them by the ring to a tack driven into the table edge.

In using balances, reserve one pan always for the weights, and the other one solely for the substance to be weighed.

(*e*) Troy weights or French metric weights may be used. For various reasons the author prefers the former, and they are the ones employed in this work. French metric weights are *not* used commercially in this country for mineral transactions.

Whittell's balance is furnished with weights running from 200 milligrammes to $\frac{1}{10}$ milligramme, but the corresponding troy weights are furnished when desired.

The Pocket Assay Balance has weights from 10 grammes ($154\frac{3}{10}$ grains) to 1 milligramme ($\frac{15}{1000}$ grain). The needle shows four divisions for 1 milligramme. Troy weights are furnished when desired. For the methods used in this work, weights from 20 grains to $\frac{1}{200}$ grain, with a set for coarser weighing, 10 pennyweights to $\frac{1}{2}$ pennyweight, will suffice.

21. *Plattner's Ivory Scale* (for measuring but-

tons).—This most useful instrument is of prime importance to the blowpipe assayer. It is constructed on the principle that buttons of silver or gold *obtained* by cupellation vary in size proportionally to their weight, and can hence be measured when they are too small for the most delicate balance to weigh. The scale (Fig. 10) is made of ivory, on which are drawn two fine lines converging to a point. These lines are crossed by transverse lines numbered on the left, while on the right the figures indicate the weight of a silver button in milligrammes. To measure the button, place it on the scale between the diverging lines, and carefully move it until it just fits in these lines; that is, until they are tangent to the button The figures on the right give the weight of the silver button.

The button must be carefully and slowly pushed, care being taken that it is not snapped off the scale and lost.

The magnifying-glass must be used, to be sure the button is in the right place. Guard against the scale being placed in such a light that the reflection from the bright shining silver button might cause error in properly placing it between the lines.

The scale, to be of use, must be perfectly accurate, and the lines finely and properly drawn. Unfortunately there are many on the market with lines so badly drawn, and so badly made generally, that they are entirely useless. The author can recommend those obtained from John Taylor & Co., San Francisco.

22. The silver button to be measured on this scale must be one resulting from cupellation, as the scale is calculated from buttons that have been cupelled. They have a certain form. Any silver button that has been melted to a spherical shape has not the exact form of a cupelled button, and cannot be measured on this scale.

23. It has already been said that the scale is numbered on the right to indicate the weight of the silver button which fits in the diverging lines at that place. This weight is in milligrammes, and also represents the per cent of silver *if one decigramme* (1.5432 grains) of ore was taken for assay. These figures on the scale can be used for other weights in the following manner: There are 29,166$\frac{66}{100}$ troy ounces in a ton of 2000 lbs. There are 29,166$\frac{66}{100}$ milligrammes in 450.09992 grains troy. (This latter weight is called the assay ton in the regular furnace assays.) If an assay ton of ore be taken for assay, each milligramme of metal found would represent 1 oz. of metal per ton of ore. If two assay tons are used, each milligramme of metal found represents $\frac{1}{2}$ oz. per ton. If one-half assay ton is taken for assay, each milligramme found represents 2 oz. per ton of metal. The principle involved in this last case is the one applicable to the use of the Ivory Scale for blowpipe buttons. For instance, 3 grs. of ore are taken for a Fletcher blowpipe-furnace assay. The resulting silver button after cupellation is just contained between the two diverging lines, at transverse line No. 16. What number of

ounces of silver per ton does the ore carry? Divide the number of grains in one assay ton (450.09992) by the number of grains taken for assay (3); multiply the result by the weight of the button (the number on the right of line No. 16 is .11, $\frac{11}{100}$ of a milligramme, which is the weight of the button in milligrammes): the result is the number of ounces of silver the ore carries to the ton.

6)450.09992
75.016653

[illegible] 3)450.09992
150.033
.11 (weight of button)
150.033
1500.33
16.50363 or 16.5 (16½) oz. of silver to the ton.

To save calculations, Table IV (see Silver Assay) has been computed, showing the number of ounces of silver the ore carries to the ton, corresponding to each line on the scale when 3 grains were taken for assay. Also, a similar table (Table VI, Gold Assay) for the gold button. Gold weighs more than silver; consequently the figures on the right of the scale which show the weight of the silver button will not answer for gold. Table V, Gold Assay, shows the weight of the gold button corresponding to each transverse line on the scale.

24. Any weight of ore can be taken, and a simple calculation gives the number of ounces per ton the ore carries, as shown by the scale. For instance, a piece of galena is assayed for silver (by

the blowpipe-furnace method); 8 grains are taken. The resulting silver button on the scale measures to line 20. The figures on the right are .22, showing the button weighs $\frac{22}{100}$ milligramme. 450.09992 divided by 8 = 56.2624; this multiplied by .22 gives 12.37, or $12\frac{37}{100}$, ounces of silver per ton the galena carries.

The scale is intended for buttons too small to weigh easily. When large enough use the balance.

Some old-fashioned scales were computed for weights formerly used in Germany, which have different numbers on the right from those showing the weight in milligrammes. In this case measure the button on the scale, and see by Fig. 10 what number on the right corresponds to the position of the button on the scale.

If the button measures between two transverse lines, proceed as follows to obtain its weight: A button fits in the diverging lines *half*-way between transverse lines No. 14 and No. 15. The number on the right corresponding to line 15 is

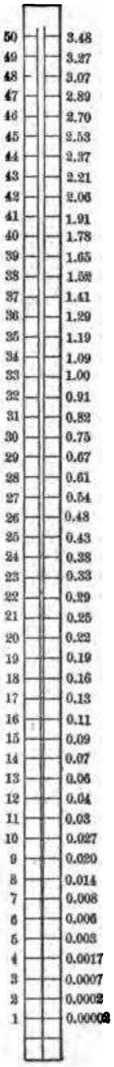

FIG. 10.—PLATTNER'S IVORY BUTTON SCALE.

.09; the number corresponding to 14 is .07., .09 − .07 = .02. Divided by .02 (as the button is half-way between the two cross-lines) = .01. Add .01 to the weight opposite line 14. .07 + .01 = .08, the weight of the button.

25. A *sampler* is necessary in getting a fair average sample of the ore (see section 107). It would be more convenient if smaller ones could be obtained. A *glass spoon* with *spatula handle* for mixing the ore and for use in sampling.

26. A "*batea*," or washing pan of peculiar form (see Gold Assay). A *tripod* and *triangle* for use with the porcelain crucible in parting.

A Fletcher patent *blowpipe*, *No.* 42, for use in blowpipe-furnace assay (see section 108).

Soda Paper.—Used for assays in the coal crucible.

CHAPTER II.

REAGENTS.

26. (*a*) *Borax* is an invaluable reagent. It is used for making the borax bead and other purposes, and in the quantitative assay as a flux.

It should be *fused*, as ordinary borax is hard to manage on account of its swelling up. *Fused* borax glass is in small grains used for making the bead. *Powdered*, it is used for assays. It can be purchased pure. The commercial article may contain slight amounts of iron as an impurity, which would interfere with properly judging what colors are produced in the borax bead by the substance dissolved in it. Test the borax for iron colors by making a bead and examining it carefully (section 75).

(*b*) It is not the metal, but the *oxide* of the metal, that dissolves in a borax bead, and which imparts to it characteristic colors, by which the presence of the metal is indicated.

(*c*) In the crucible assay borax acts as a solvent for many infusible substances we wish to slag off or get rid of, as it forms fusible compounds with them, which enter the slag. It combines with most of the oxides of the base metals, which thus pass into the slag. It is a good flux for basic substances, such as lime, iron, etc., when they are present in the gangue, it acting as an "acidic" flux.

27. *Salt of Phosphorus* (Microcosmic Salt).—SPh produces with the metallic oxides different colors often from those given by borax, and gives in certain cases more delicate reactions than borax. As in the case of borax, it is not the metal, but the metallic oxides, that dissolves in the SPh bead. The precautions to be taken with the SPh bead until its water of crystallization is gotten rid of, are mentioned in section 7, (*c*).

SPh is not used in the crucible assay.

28. (*a*) *Soda.*—This is "bi-carbonate of soda.' Used in reductions on coal as a test in certain cases for manganese and sulphur, and for other purposes which will be noted in the proper place. It is used as a flux in the crucible assays. It should be perfectly pure from sulphur when used to test for sulphur. The pure article had better be used in the copper assay. The commercial article suffices in other assays.

(*b*) Its action in the crucible assay is as follows: Soda forms very fusible compounds with otherwise infusible parts of the gangue, retaining these substances without losing its fluid condition, as charcoal, clayey, earthy matter, etc.; or it combines chemically with silicates in the gangue, —quartz, for instance, which is silica, forming a sodium silicate, which passes into the slag. It forms fusible compounds with most metallic oxides. It oxidizes iron and tin, which then pass into the slag. (It oxidizes zinc, which partly volatilizes.) It desulphurizes, or takes away sulphur, from its combinations (more or less perfectly, and in the case of many metallic sulphides soda has to be helped by the other "desulphurizers" for the sulphide to be completely reduced).

It acts as a basic flux, and consequently is used to get rid of substances of an opposite or "acidic" nature, as in the case of quartz mentioned above.

29. *Potassium Carbonate.*—It is only used for the crucible assay. It acts in the same manner as soda. A mixture of potassium carbonate and soda makes a more fusible flux than either alone, and hence it is very useful in blowpipe assays (where the amount of heat is limited) in the case of difficultly fusible gangues.

30. *Nitre* ("Saltpetre").—It is used in certain tests for manganese.

It is especially useful in the assay of silver-bearing galena. In the crucible it acts as a desulphurizer. When enough is used (varying with the metallic sulphide acted on) it takes away the sulphur, and if in excess, oxidizes the base metals, which pass into the slag. But many metallic sulphides require such a large amount of nitre to do this that it is not used by itself alone in blowpipe assays to remove the sulphur, except in the case of lead-sulphide galena. Moreover, too much nitre puffs up the assay or throws it out of the crucible.

31. *Tin*, in the form of foil, is used in many

tests in qualitative work. It helps reductions; that is, helps to take away oxygen from the substance dissolved in the bead, owing to the affinity of tin for oxygen.

32. *Boric acid, fused,* and in the form of a "glass," is used to separate copper from lead in quantitative assays.

Copper Oxide.—But a very small quantity is needed for tests for chlorine.

33. *Nitrate of Cobalt Solution.*—Made by dissolving in rain-water a few crystals, about one tenth weight of water. Invaluable in detecting zinc and some other substances by the color it imparts.

Keep in a glass-stoppered bottle. Taking a drop or two out of the bottle with a "dropper" purchased at any drug-store) is the best way to use it.

34. *Test-lead* (in powder).—Lead, it may safely be said, nearly always contains silver,—in very minute quantities, it is true, but still enough to vitiate an assay for silver unless allowed for, especially when the question of the presence or absence of silver in the ore is in doubt, as mistakes are bound to occur from the silver contained in the test-lead used being taken for silver contained by the ore assayed. The best lead, advertised as chemically pure and free from silver, the author has found to contain silver in varying amounts at the rate of from .12 oz. to the ton up. All test-lead must be tested for silver (section 112), and the amount determined and allowed for.

Test-lead is used as a collecting medium to alloy with the silver (or gold) in the coal-crucible assay, the resulting alloy being afterwards cupelled to get rid of the lead. It is also used to collect copper in the same manner in the coal-crucible assay of copper, the lead being separated by treating the alloy, besides boracic-acid glass. It is also used alongside of a borax bead saturated with metallic oxides to collect those metals which are "reducible" under these circumstances. The alloy is then treated by suitable means to show the presence of these "reducible" metals. It is used in the clay-crucible assay as a "collecting medium" in certain of the copper assays.

35. *Litharge* (Oxide of Lead).—Invaluable in the blowpipe-furnace assay. Litharge contains silver as well as test-lead does. The remarks under Test-lead in relation to silver are applicable here.

The amount of silver the litharge contains must be determined and allowed for (see section 112). The best and purest litharge I have ever used was obtained from John Taylor & Co., San Francisco, who make a specialty of pure blowpipe reagents.

The action of litharge in the crucible is as follows: It oxidizes most of the metals, except the noble ones. It acts as a desulphurizer, but larger amounts are required of litharge than can be used in the small blowpipe crucible; and if used for this purpose, and without the help of other desulphurizing agents, it is in less quantity than is required to entirely desulphurize the metallic sulphide. Moreover, an oxysulphuret is liable to be formed (composed, in part, of the unacted-on metallic sulphide), which goes into the slag; hence loss of a portion of the metal. This is one reason why ores containing sulphides should be well roasted before assaying in the furnace. An exception is in the assaying of silver-bearing galena by the nitre method.

Litharge also to a certain degree acts as a flux. In the clay-crucible assay for the precious metals the litharge is reduced (its oxygen taken away) by suitable reducing agents to metallic lead, which alloys itself with the gold and silver in the ore (or copper in the case of certain copper assays), and the lead is removed afterwards from the alloy, as described in section 34.

36. *Silica.*—Used as an "acid" flux in the crucible-furnace assay when the gangue contains "basic" impurities, such as lime, iron oxides, iron carbonates, especially when little quartz (silica) is present. It is of great use when much lime is present, or barytes; but with some metals, like tin, it forms silicates which pass into the slag. Hence, if the object be to save the metal, it is frustrated unless the silicate can be decomposed by other agents in the charge. If we wish to get rid of the metal, our object is accomplished by the formation of the silicate which goes into the slag.

Silica is also used in a qualitative test for sulphur. Some ores which are wanting in silica will take it at the expense of the crucible, thus eating away or cutting through the clay crucible if silica is not furnished.

37. *Nickel Oxalate.*—Of very limited use. Used to detect potassium. *Silver* (foil), for inquartation.

38. *Charcoal.*—Used as a reducing agent—one that takes away oxygen or reduces an oxide. Also as a desulphurizer. It should be powdered, and well mixed with the assay. Though it is the most powerful reducing agent used in the dry way, the author does not recommend its use in the blowpipe-furnace assay, as if used in excess it has the disadvantage of remaining unconsumed in the slag, retaining minute globules of reduced lead (from the litharge), alloyed with the metal sought, in the slag, and thus spoiling the assay. It has a tendency to make the slag more difficultly fusible also. This is especially true of the blowpipe assay. It is also used to mix with soda in the soda reduction on coal. Here its use is excellent.

39. *Argol* is a better reducing agent for crucible assays for blowpipe use.

Flour and *Starch* are also excellent reducing agents. They should be well dried.

The comparative power of these reducing agents is about as follows:

Charcoal..............	28
Argol.................	8½
Flour.................	12 (dry, more)
Starch................	11½ " "

That is, from the same amount of litharge they will reduce that many times their weight of metallic lead. The reducing power varies in different samples—due to impurities, their being dry or not, etc.

40. *Metallic Arsenic.*—Used in the quantitative assay of nickel and cobalt, to form arsenides of these metals, which are too infusible to obtain by direct fusion. It is also used in certain qualitative tests for the same metals. It is hardly necessary to caution the operator against its poisonous qualities.

41. *Potassium Cyanide.*—Used in the furnace assay of tin. It also is very poisonous.

42. *Iron.*—Metallic iron in the form of wire, like hairpins in thickness, is used as a desulphurizer, or taker away of sulphur from certain bodies, owing to the strong affinity iron has for sulphur. Amongst the metals whose quantitative assays are treated of in this book, iron *completely* desulphurizes silver sulphide, lead sulphide, tin sulphide, and bismuth sulphide. Copper sulphide is not completely reduced. Cast iron should not be used.

Iron is also used in certain qualitative tests, as to find antimony in the presence of galena.

43. *Test-papers.*—*Blue* litmus-paper, to indicate the presence of acids by turning red.

Turmeric paper, turned a rich brown by alkalies.

Brazil-wood paper is used in tests for fluorine.

44. ACIDS.—*Hydrochloric Acid.*—Used in helping to intensify certain flame colors.

45. *Nitric Acid.*—Used in parting gold from silver in the gold assay.

CHAPTER III.

COATS.—FLAME-COLORATION.—GLASS TUBES.

46. *Coats.*—(*a*) A number of substances when submitted to the action of the blowpipe flame, on coal, give a coat; that is, incrust the coal with a coating which has characteristic colors.

(*b*) The first thing to be done with a mineral before testing it is to pulverize it well on the steel anvil. If very hard, reduce it to powder in the iron mortar. Mix well the powder, pour it into the mixing capsule. Bore a shallow hole with the borer on a piece of charcoal (near the end). Take about as much of the powder as will cover a half-dime (or less). The most suitable quantity varies with the substance, and will soon be learned by experiment. Place the powder in the hole; press it well down so the blast from the blowpipe will not blow it away. It may be well to slightly moisten it, to make it coherent. Subject it to a good and pure R. F. Certain coats are best formed by the O. F. If no coat is obtained by the R. F. the substance should be tried with the other flame. If certain volatile bodies are present they will soon coat the coal beyond the assay, some nearer, some farther, with a coat whose colors both while hot and while cold must be noted.

TABLE I.

COATS PRODUCED ON COAL.

(In the R. F. unless otherwise stated.)

Color.	Given by—	Hot.	Cold.
YELLOW.	Pb.	Lemon-yellow.	Sulphur-yellow.
	Bi.	Orange-yellow.	Lemon-yellow.
	Cd.		Orange-yellow.
	See also under BROWN.		
	(O. F.) In.	Dark yellow.	Light yellow.
	Te.		Yellow *border*.
	See also under WHITE.		
RED.*	(O. F.) Ag.		Dark red to brownish red.
	(O. F.) Ag and Bi, Ag and Pb.		Red, at first yellowish.
	(O. F.) Ag and Sb.		Red, at first whitish.
	(O. F.) Mo.		Copper-red.
	See also under WHITE and BLUE.		
	Se.		Red *border*.
	See also under GRAY.		
	Te.		Red *border*.
	See also under WHITE.		
GRAY.	Se.		Light gray near, dark gray far from assay; steel-like.
	Hg.		Light grayish far from assay.
	As.		Steel-gray to dark gray.
	See also under WHITE.		
WHITE.†	Zn.	Yellow.	White.
	Sn.	Faint yellow.	White.
	Te.		White, with red or yellow border.
	Sb.		White.
	As.		White far from assay.
	(O. F.) Mo.	Yellow.	White. The coat touched with R. F. blue mark. Copper-red when coat is treated with O. F.
	See also under RED.		
	Tl.		White. Near assay brown.
	Pb (carbonate).		White *border* to bluish.
	See also under YELLOW.		

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* Sulphides of As and Sb too quickly heated may give reddish or reddish yellow coats, instead of the coats proper to them. Certain minerals containing Mo give a slight whitish coat, copper-red near assay, and with a blue border at the extreme edge of coat.

† Certain chlorides, sulphides, etc., give white coats. See section 46, (*d*).

TABLE I—(*Continued.*)

Color.	Given by—		Cold.
BROWN.	Cd. See also under Yellow. Tl. See also under White.		Reddish brown to dark yellow, with iridescent *border*. Brown near assay.
BLUE.	(O. F.) Mo. See under Red and White. Pb *blue border*. See under Yellow.		The coat may have a blue border. The white coat touched for an instant with R. F. is blue. The yellow coat has often a bluish white *border*.

(*c*) The side of the hole farthest from the blowpipe must be slightly cut away, sloping so that the volatile metal, whose oxidization on contact with the air produces the coat, may not be deflected from the coal.

(*d*) If no coat is produced, or even if a white one is, try the experiment again, this time mixing the substance with its bulk or more of soda; moisten slightly the mass, and submit to the R. F. The reason for this is, that some chlorides, bromides, iodides, sulphides, and sulphates yield white coats which are not characteristic, and when soda is used these compounds are decomposed, and we obtain coats of the oxides, with their distinguishing colors.

47. Under the head of each element in Chapter VI will be given the coat it produces. The preceeding table is so arranged that the metal producing a given coat may be ascertained, conforming it by referring to Chapter VI.

(For the symbols of the elements, or abbreviations of their names, see Appendix.)

48. (*a*) Some of these coats are volatile. Under the head of each element in Chapter VI will be given full descriptions of the coats.

(*b*) Some of the coats color the inner flame—

Blue.—	Faint azure-blue	Pb.
	Azure-blue	Se.
	Light blue when abundant	As.
Green.—	Pale bluish green, with a whitish yellowish tinge	Sb.
	Green	Te.
	Intense green	Tl.

(*c*) The white coats produced by the sulphides of Pb and Bi give a yellow spot when heated with O. F., which serves to distinguish them from the white coats caused by the oxides of some other elements; but it is always best to use soda when a white coat is obtained, unless the use of the nitrate-of-cobalt solution, section 49, shows Zn or Sn.

(*d*) Certain of the white coats produced by chlorides, bromides, etc., tinge the flame; but the reactions are not very practical, and are omitted.

(*e*) Copper chloride gives a brown to dark yellow coat, gray nearer the assay, and tinges the flame azure-blue.

(*f*) A white coat may also be caused by ammonium chloride.

(*g*) In obtaining a coat by means of soda, when iron is present in the mineral, the addition of some borax is useful, as will be noticed under the head of Zinc. Two parts of soda, one of borax, and one of the substance assayed is a good mixture.

(*h*) When soda is treated with minerals containing sulphur in the R. F. a liver-red-colored compound of soda is often obtained in small spots on the coal, and by long blowing a *slight* reddish to reddish brown coat may be formed. This is mentioned to warn the operator against error. Test also the coal before forming a coat to see that no change of color takes place on the coal.

(*i*) The use of the coats is most practical and important in detecting Pb, Bi, Zn, Sn, Sb, Cd; in a less degree As and Mo.

49. *Nitrate-of-cobalt Solution.*—The colors imparted by this solution, either to the coats or to the minerals themselves, after treatment in the O. F. are very characteristic in certain cases.

Generally this method is only applicable to substances which are not fusible, and which turn white or whitish after treatment with the O. F.

Powder the substance, place it in a shallow cavity on coal, press it down, treat with O. F., drop a drop of the nitrate-of-cobalt solution on the substance, and after drying at first, treat strongly with O. F. The colors appear best after the substance is cold.

In the case of coats: Form first the coat; when cold, moisten with the solution, and then treat with O. F. Form coats when testing for Zn and Sn. The following are the colors:

Green.—Beautiful grass-green or green
with a yellow tinge...........Zn.
Bluish green...................Sn.

(The above must not be confounded with a dirty green given by Sb.)

The yellowish green of Ti and the dirty green of Cb are obtained by moistening the substances themselves, as they form no coats.

Pink or Flesh Color.—Magnesia. This is not always easy to obtain, and sometimes is of a brick-red or dingy shade.

Blue.—Alumina. Not to be confounded with the blue yielded by some fusible compounds or by silica, which give a lustrous, glassy appearance.

This test is of most practical importance in the detection of Zn, and less so in the case of Sn.

50. GLASS TUBES.—(*a*) The use of the glass tubes is generally given much prominence in works on the blowpipe, but the author finds that many other reactions are more useful in detecting substances. Only the most useful of the phenomena that take place in using the tubes will be noticed. (A full account will be found in Cornwall's "Blowpipe Analysis.")

(*b*) In the open tube the substance is burned in the air. Insert the material carefully in the open tube, a short distance from the end. Hold the tube in the "matrass-holder" and heat in the flame of the alcohol lamp. The tube should be inclined so the air may flow through it.

An odor of *sulphur* is caused by many substances containing it. A small strip of moistened blue litmus-paper inserted in the upper end of the tube is reddened when sulphur fumes arise.

An odor of *garlic* indicates arsenic.

An odor of rotten horse-radish, selenium.

A white *crystalline* coating or sublimate forming inside the tube, and which can be driven about by heat, As. White fumes condensing to a coating in the tube, Sb. White fumes giving a gray sublimate which can be fused to colorless drops, Te.

Gray shining metallic, *mercury*. Sometimes in scattered drops.

(*Note.*—Too rapid heating may give colored sublimates, as sulphides of arsenic, red, yellow; sulphide of mercury, black.)

(*c*) *Closed Tube.*—The powdered mineral is put in a long narrow slip of paper, which is folded lengthwise on itself. The tube is held horizontal, the paper slipped in to the bottom, the tube turned up straight, and the paper withdrawn, thus placing the powder at the bottom without getting any on the sides. Heat end of tube in flame. *Water* collects in drops on the tube.

Sublimate dark yellow to light sulphur-yellow, cold—sulphur.

Black with red powder, *mercury* sulphide.

Shining grayish white coating, *mercury* (rub together with a feather the drops of mercury). If the sulphide is mixed with *dry* soda and heated, it is decomposed, giving the shining mercury drops.

Lustrous metallic black arsenic (metallic), the sulphide of arsenic, gives a reddish yellow (cold) sublimate.

Gray to black metallic, with brown border, cadmium.

51. *Flame-coloration.*—(*a*) Many bodies when heated give certain colors to the blowpipe flame.

The mineral in a small fragment—the smaller the better—is held in the platinum-pointed forceps and heated intensely by the tip of the inner flame. (The blowpipe is held as when producing the O. F., and the blue cone is meant by the inner flame.) Some minerals are best heated on coal.

The room should be darkened, as the colors show better. A drop of hydrochloric acid often increases the intensity of the color.

(*b*) Flame-colorations given by the following:

Blue:

Azure-blue	Pb (on coal).
Light blue	As (on coal).
Blue	Se (on coal).
Intense blue	Copper chloride produced if the mineral contains copper, and is moistened with hydrochloric acid (on coal).

Green:

Yellowish green	Barium.
A light bluish green, with (especially the coat) a whitish yellow tinge, hard to describe exactly in words.	Sb (on coal).
Emerald-green	Cu.
Pale bluish green	Some compounds of phosphorus (on wire).

Intense green..........Tl.
Intense green..........Te.
Whitish green..........Zn (on coal).
Yellowish green........Mo.
Green ....Boracic acid.

Yellow:

Sodium.

Red:

Intense carmine........Sr.
Red, not as intense as the strontium nor lasting as long, best brought out by a drop of hydrochloric acid, calcium (lime).
Reddish purple.........Lithium.
Violet.................Potassium.

(*c*) The mere absence of color does not indicate the absence of an element which gives that color. For instance, lime gives a red flame, but lime (calcium) in combination with silica in most silicates does *not* give the red color to the flame. The same holds good as to barium in silicates.

Barium interferes with the strontium flame.

The yellow of sodium powerfully masks the violet of potassium in compounds where they occur together.

(*d*) If the mineral is in the form of a powder, it may be moistened slightly and placed in the loop of a platinum wire. See that the forceps and the wire are perfectly clean. Wiping them on the hand may give a sodium flame.

(*e*) The flame-coloration is of most practical importance in the detection of Cu and Sb, and is a very delicate test for Na.

CHAPTER IV.

ROASTING.—USE OF GLASS FLUXES.

52. (*a*) MOST minerals containing sulphur or arsenic have to be oxidized or roasted before they can be properly examined by means of borax and SPh.

For it is the metallic oxides that color the glass beads with their distinguishing colors, and it is essential to drive off the sulphur or arsenic. So as to make sure, it is best generally to roast the substance.

Before making the "soda reduction" the substance should also be roasted.

(*b*) Bore a shallow cavity in the coal. The powdered mineral is spread thinly over this, moistened if necessary to keep it from blowing away, and heated with the O. F., slowly at first, to redness.

Smell the assay. The fumes of sulphur or the garlic odor of arsenic are given off by these elements. Continue the roasting until no more odors are given off. Care must be taken not to melt or fuse the assay together; some minerals have a tendency to do this. If this happens, throw away the assay and begin again; or if it can't be spared, pulverize it afresh, and commence over. In the roasting, sulphides may be oxidized to sulphates and arsenides to arsenates: these must be reduced by the R. F.; then repeat the treatment with the O. F., followed by the R. F. until the assay is "sweet." It is now turned carefully over (it generally sticks together) so as to expose the bottom, and the operation of roasting using the O. F. and R. F. alternately repeated. A well-roasted assay should be dull and easily powdered.

(*c*) If there is a tendency to fuse, as in some minerals containing lead sulphide, mix a little silica with the assay.

(*d*) It should not be necessary to caution the operator against inhaling the fumes given out freely by some minerals containing *arsenic.*

(*e*) For the quantitative assay, sulphides, etc., have to be well roasted. The method will be indicated in the proper place.

53. *Glass Fluxes* (Borax and SPh).—A bead of borax is formed (as described in section 7, (*c*)) and dipped in the powdered assay, taking up but a small quantity, and the bead treated in a strong O. F. until all is dissolved, noting the color while hot and when cold. Do not look at the flame through the bead. Do not look at it with a colored window-shade or a tree as a background. This would interfere with a proper recognition of the color. Look at the bead against a white wall or over a plate.

Small portions of the assay should be added successively rather than all at once, as certain minerals color so intensely the bead as to make it almost black. Manganese, for instance, which in right proportion gives a beautiful purple, turns the borax bead nearly black by an excess.

(Compounds of arsenic, antimony, and sulphur, and metals also, must be kept from the platinum wire, lest it be injured.)

54. (*a*) After noting the color of the bead hot and cold in the O. F., submit it to the R. F. (minding the precautions mentioned in par. 2), and note the colors hot and cold of the bead. It is best for beginners not to trust to memory, but to write down the colors, which can then be compared with the tables.

Make now a SPh bead (section 7, (*c*)), dip in the assay, and note in the same way the colors produced (hot and cold) by the O. F. and R. F.

(*b*) If too much of a mineral has been used, giving too dark a color, add more borax (or SPh), or, what is still better, while hot lay bead on anvil, break off half of it with the hammer, add more of the flux, and fuse again in the flame.

(*c*) The following table, modified from Plattner, and which has appeared since in many books on the blowpipe, is given as a reference by which the color of a bead can be traced to the producing metallic oxide.

Reference should then be made to Chapter VI for further particulars.

(*d*) Pure oxides give these colors, but in minerals we often have mixed oxides to deal with, which often give mixed colors very different from that produced by the pure oxide alone. In this case the color of the oxide is produced in various ways noted in Chapter VI, such as by obliterating the color of one of the oxides by changing the flame, or by changing the flux, or by the use of tin.

For instance, manganese (when in certain proportion) with iron in the borax bead masks the color of the iron when treated with the O. F., and the bead shows neither the violet of Mn nor the yellow of Fe. But when the same bead is submitted to the R. F. the Mn color is destroyed, and that of Fe (bottle-green in R. F.) can be detected. Or by dissolving the mineral in SPh, which is colored much less by Mn than is the borax bead, the color of Fe may be ascertained. Fe in certain proportions with Ti in the borax bead in O. F. masks the color of Ti, giving a red bead. Treat the substance in a SPh bead on coal in a cavity alongside of a piece of tinfoil tightly rolled and submit to a strong R. F., and the violet color of Ti appears, if too much iron is not present (see sec. 55).

(*e*) *Flaming.*—When a bead is well charged with *certain* bodies and alternately heated and cooled by the flame, or taken out of it and introduced again several times, it becomes opaque, and changes color. This operation is called *flaming.* Some beads when saturated with certain elements become opaque on cooling.

TABLE II.

COLORS PRODUCED BY OXIDES IN THE BORAX AND SPH BEADS.

(For explanation of the abbreviations used see Appendix.)

(A) IN THE BORAX BEAD WITH THE O. F.

Color.	When Hot.	When Cold.	
COLORLESS.	Si, Al, Sn.	The same cold.	
	Ba, Sr, Ca, Mg, Be, Y, Zr, Th, La, Ag, Ta, Te.	" "	White on cooling if saturated.
	Ti, W, V, Mo, Di, In, Zn, Cd, Pb, Bi, Sb.	" "	With slight amounts.
YELLOW.	Ti, W, Cb, Mo, Zn, Cd, Pb.	Colorless.	When saturated. Opaque by flaming.
	Bi, Sb.	Colorless.	When saturated.
	Ce, U, Fe.	Colorless.	
	Cr.	Greenish yellow.	Small quantity.
	V (a larger amount).	" "	
RED.	Fe.	Yellow.	In large quantity.
REDDISH BROWN.	Ce, U.	"	Large quantity. Enamel-like by flaming.
	Cr.	Yellowish green.	Large quantity.
	Fe and Mn.	Yellowish red.	" "
		Ni, Ni and Co, Mn (see under VIOLET).	
VIOLET. TO PURPLE.	Mn.	Violet-red.	*Nearly* black if in excess.
	Ni.	Brown, reddish brown.	
	Ni and little Co.	Violet-brown.	
	Ni and much Co.	Same.	
	Co and Mn.	Same.	
BLUE.	Co.	Same.	
		Cu (see under GREEN).	

TABLE II—(*Continued.*)

(A) In the Borax Bead with the O. F.

Color.	When Hot.	When Cold.	
Green.	Cu.	Blu to greenish blue.	
	Fe and Co, Fe and Cu, Cu and Ni.	Light green, blue, or yellow, according to proportions in which they are present and amount used. When hot.	
	Cr.	Yellowish green (see under Red).	

(B) In the Borax Bead with the R. F.

Color.	When Hot.	When Cold.	
Colorless.	Si, Al, Sn, In.	Same.	
	Mn.	Same; may acquire a slight reddish tinge.	
	Ba, Sr, Ca, Mg, Be, Y, Th, Zr, La, Ce, Ta.		Opaque by flaming.
	Cb.	Same.	In small quantity.
	Ag, Zn, Cd, Pb, Bi, Sb, Ni, Te.	Same.	Gray at first; after long blowing, colorless.
	Cu.	Opaque red if saturated.	
Yellow.	Ti.		If highly saturated, enamel - blue on flaming.
	W.	Brownish.	Saturated.
Brown.	Ti.		If highly saturated, enamel - blue on flaming.
	Mo, nearly black.		Highly saturated.
	When Hot.	When Cold.	
	V.	Emerald-green, W. (See under Yellow.)	
Blue.	Co.	Same.	
Green.	Fe, bottle-green.	Same.	Nearly black if in excess.

TABLE II—(*Continued.*)

(B) In the Borax Bead with the R. F.

Color.	When Hot.	When Cold.	
Green.	**U, yellowish green.**	**Same.**	**Saturated; black by flaming.**
	Cr, light to dark green.	**Same.**	
		V (see under Brown).	
Gray.		**Ag, Zn, Cd, Pb, Bi, Sb, Tl, Ni, Te.**	**At first, after long blowing, colorless.**
		Cb.	**Highly saturated.**
Red.		**Cu highly saturated and not well reduced; opaque red.** **Mn highly saturated and not well reduced; faint color.** **Di rose; highly saturated.**	

(C) In the SPh Bead with the O. F.

Color.	When Hot.	When Cold.	
Colorless.	**Si, but little soluble. Al, Sn, with difficulty dissolved.**	**Same.**	
	Ba, Sr, Ca, Mg, Be, Y, Th, Zr, La, Di, Te.	**Same.**	**White by flaming.**
		Ta, Cb, Ti, W, Zn, Cd, Pb, Bi, Sb (see under Yellow).	**If but slightly saturated, the same hot.**
Yellow.	**Ta, Cb, Ti, W, Zn, Cd, Pb, Bi, Sb.**	**Colorless.**	**Saturated.**
	Ag.	**Opalescent.**	
	Fe, Ce.	**Colorless.**	**Slightly saturated.**
	U.	**Yellowish green.**	
	V.	**Paler yellow.**	
		Ni (see under Red).	
		Fe, Ce, highly saturated.	

TABLE II—(*Continued.*)

(c) In the SPh Bead with the O. F.

Color.	When Hot.	When Cold.	
Red.	Fe, Ce, highly saturated.	Yellow.	
	Ni reddish.	Yellow.	
	Cr reddish.	Emerald-green.	
	Mn, brownish violet.	Reddish violet.	
Blue.	Co.	Same.	
		Cu (see under Green); if saturated, greenish blue.	
Green.	Cu.	Blue to greenish blue.	
	Mo, yellowish green.	Lighter.	
	Fe and Cu, Fe and Co, Cu and Ni.	Light green, blue, or yellow, according to proportions of oxides and amount taken when hot.	
		U yellowish green (see under Yellow).	
		Cr emerald-green (see under Red).	

(d) In the SPh Bead with the R. F.

Color.	When Hot.	When Cold.	
Colorless.	Si slightly soluble.	Same.	
	Al Sn, with difficulty dissolved.	"	
	Ba, Sr, Ca, Mg, Be, Y, Zr, Th, La.	"	White by flaming if saturated.
	Mn, Ce, Di (small amount).	Same.	
	Ta, Te, Ag, Zn, Cd, Pb, Bi, Sb.	Same.	Gray at first; after long treatment colorless.
		Fe small quantity (see under Yellow and Red).	
Yellow.	Fe.	Smoky reddish brown.	
	Ti.	Violet.	
	Cb.	Brownish (saturated).	
	Ti and Fe, W and Fe.	Brownish red to blood-red	
		Cb and Fe dark yellow (see under Red).	

TABLE II—(*Continued.*)

(D) IN THE SPH BEAD WITH THE R. F.

Color.	When Hot.	When Cold.	
RED.	Fe red (*much*).	Smoky brownish red.	
BROWN.	V brownish.	Emerald-green.	
	Cb and Fe brownish red.	Dark yellow.	
		Cu highly saturated (see under GREEN).	
BLUE.	Co.	Co.	
		W blue to bluish green (see under GREEN).	
VIOLET.		Ti (see under YELLOW).	
		Di highly saturated and after long treatment.	
GREEN.	Cu.	Opaque red.	
	W dirty green.	Blue to bluish green.	
	U dirty green.	Fine green.	
		Cr (see under RED).	
		V (see under BROWN).	
	Mo dirty green.	Green.	
GRAY TO BLACK AND CLOUDY.		Ag, Zn, Cd, In, Ni, Tl, Pb, Sb, Bi, Te.	At first, with long blowing, colorless.

(*g*) The glass beads are of most use in the detection of Co, Ni, Mn, Fe, Cu, Mo, U, V, and Cr.

55. (*a*) SPh beads reduced on coal besides tin. Dissolve the powdered mineral in a SPh bead in O. F. Bore a cavity in a piece of coal. Place the bead in the cavity. Roll the tin-foil up tight; take about one half the size of the bead. Submit to the R. F. Be careful to keep the tin button bright: it must not be covered with oxide. Do not submit too long to the flame. The tin button and bead should be fused together. Tin is greedy for oxygen, and aids the R. F. in taking away (or making less) the oxygen combined with

the element in the bead, and changes take place in the color of the bead which are often characteristic (see section 2 in relation to soot-forming).

(*b*) TABLE III.

SPh Bead treated on Coal, alongside of Tin, with the R. F.

Color.		
Red.	Cu.	Colorless hot; on cooling, red to brownish red; opaque.
Gray (to Cloudy).	Sb.	Gray, but on longer blowing clear.
	Bi.	Clear hot; blackish gray, *opaque*, cold.
	Pb.	Cloudy and gray; never quite opaque.
Green.	Fe.	Green on cooling, then colorless.
Violet.	Ti.	Violet, unless too much Fe is present.
Blue.	W.	Blue, unless too much Fe is present.

The reduction of the SPh bead with tin is of most use in the detection of Cu, Ti, W, and Bi.

(*c*) When the borax bead is reduced with tin on coal (as above) Fe produces a vitriol-green color on long blowing. If there is much Fe the bead will be nearly black.

56. (*a*) *Reduction of Metals from Borax Beads with the Aid of Lead.*—When a bead of borax saturated with metallic oxides is treated on coal, with the R. F. alongside of a button of lead, certain metallic oxides are reduced to metals, some of which alloy themselves with the lead.

Certain others are not reduced.

This is an important operation, and must be well practised until the operator is master of it.

On it depends one method of quantitative blowpipe assaying. It also is of constant use in qualitative tests.

(*b*) The following can be so reduced from borax: gold, silver, copper, nickel, lead, bismuth, tin, antimony, arsenic, tellurium, zinc, and cadmium.

Of these, *gold*, *silver*, *copper*, and *nickel* alloy with the lead button; that is, are "collected" by the lead.

Of the remainder, some will volatilize partly, and some of the more volatile ones entirely.

(*c*) The following are not so reduced:

Fe, Co, Cr, Mn, U, V, W, Mo, Ti, Cb, Ta; the oxides of the "earths," Al, Ca, Mg, Sr, etc.; and of the "alkalies," Na, K, etc.

(*d*) The roasted mineral may be first dissolved in the borax bead (which should be made larger than usual, so more mineral can be used), and placed in a coal crucible (prepared as in section 10). A small button of test-lead is formed by melting the lead with the flame (it should be one fourth or less the size of the borax bead), and placed in the coal crucible beside the borax bead, and treated with the R. F. Or, which is better, the roasted mineral is mixed with its bulk or so of borax (an ivory spoon of each), and some test-lead, in the mixing capsule; placed in a soda-paper cylinder, which is put in a coal crucible held in the clay cylinder (like the silver assay in the coal crucible, section 104, which consult), and submitted to the R. F.

(*e*) The lead must unite in one button. None must remain scattered through the bead. The button and bead must be kept in motion around the crucible, so the lead button will pass over every part of the bead and be able to collect all reducible metals, with which it can alloy. The R. F. must be strong and pure; no lead must oxidize: if it does, the borax has a tendency to lose its globular shape, and spread over the coal and stick to it, whereas it should freely move around the crucible. To keep the borax bead in its proper shape is the most difficult part of this operation. If the borax does commence to spread, a strong blast R. F. may bring it back to its globular form. If not, let it get cold, insert a pin under the borax (being careful not to break the crucible), and turn the assay over so its under side will now be up, and again submit to the R. F. By this means, and with care, it may be made to assume its proper shape and be kept from sticking to the coal. But the results are apt to be more certain if from the start the borax is not allowed to spread itself over the coal. When the lead button has freely revolved several times around the bead, and there are no scattered lead globules in the bead, the operation is finished. (When the soda-paper cylinder is used, see directions in sec. 104. Silver Assay, in relation to causing it to " turn " at proper time.)

(*f*) We have now in the borax bead the non-reducible oxides (see "*c*" of this section), and alloyed with the lead button the reducible ones that are non-volatile, and perhaps a part of some of the volatile ones.

(*g*) Of the non-reducible ones the following give colors sufficiently characteristic to the borax bead to by this means determine if they were in the mineral: Fe, Mn, and Co. The bead is broken when cold, and a piece of it submitted on the platinum wire to the O. F. and R. F., and from the colors produced by aid of Table II can be determined the metal whose oxide produces the color.

For the mixed colors of Fe, Mn, and Co, see Chapter VI.

(*h*) Of the metals in the lead button that are found by this method, gold and silver are found by cupellation (see Silver Assay); copper, by removing the excess of lead by means of fusing it, besides boracic acid (see Copper Assay, sec. 126), then placing the small button alongside of a SPh bead, in a shallow cavity on coal, and submitting the button to the O. F., when the copper oxidizes and colors the SPh bead blue cold (sec. 54). Or, in the case of nickel, the same method is used, and the SPh bead becomes yellow cold.

If the SPh bead becomes green, it shows that both nickel and copper are present. If no very decided color is obtained after treating the button with a good O. F., remove the SPh bead to another cavity on the coal, and fuse it beside tin in the R. F.

If it becomes red, it shows that copper is present. This is a more delicate test for copper than the other (sec. 55).

CHAPTER V.

SODA REDUCTION.

57. (*a*) BY the use of soda many metallic oxides are reduced (oxygen taken away), when this could not be easily accomplished, if at all, by the use of R. F. alone.

(*b*) The assay is mixed with two or three times its bulk of dry soda, moistened if needful, and placed in a deep cavity on coal, and the R. F. made to play on it.

The soda partly sinks into the coal, leaving many of the metals in the form of small globules or scales, and others as metallic dust or powder.

(*c*) The blast should be strong, and a pure R. F., and continued sufficiently to make a good reduction. More soda may be added from time to time. The minute globules of metal obtained by this method are liable to be lost, unless great care is taken in the subsequent manipulations.

(*d*) After the reduction examine the assay carefully through the lens, and buttons of metal may frequently be seen on top of the coal. Now cut with a sharp knife carefully around the assay and the surrounding coal, remove the mass to the Wedgwood mortar, and pulverize it. Fill the mortar half full of water, and wash carefully away the coal and lighter portions. Pour more water in, and continue the washing, which if carefully done will get rid of the soda and coal, and finally leave the reduced metal in the mortar. With the last drop in the mortar pour the metal in a cavity on coal and gently dry with the O. F. It can now be often determined by examining it with the magnifying-glass. But it is safer, in case of doubt, to treat the metal with the O. F. beside some borax until it is saturated, and then treat the bead beside lead in the R. F., as in the last chapter. From some of the metals reduced by this method characteristic coats will be obtained on coal.

(*e*) Gold, silver, copper, tin, lead, in buttons, whose appearance generally determines them, are obtained by the soda reduction. In case of doubt between silver and tin, test with O. F. Tin oxidizes easily, the button becoming covered with a powdery-looking white oxide.

Antimony is obtained at times if in quantity, but is volatile, and is apt to be lost. It is in the form of globules or grains, white, brittle, and which yield the Sb coat on coal (see chapter on Coats).

The volatile arsenic, zinc, tellurium, indium, and cadmium are practicably lost by volatilization. Arsenic may form an alloy with other metals.

Bismuth.—Whitish buttons, with a reddish or pink tinge; very slight tinge unless the buttons be of some size. Examine for coat, etc. (see Chapter VI).

Iron, Cobalt, and *Nickel* are obtained in gray metallic powders, or shining white or steel-colored scales (especially nickel in this last form). They are all three magnetic. Test with the magnet. *Tungsten* and *Molybdenum* as metallic infusible powders may be obtained.

58. (*a*) Charcoal dust, finely powdered and mixed with the soda and mineral, makes the reduction more easy. Use about as much coal (in bulk) as mineral.

(*b*) If we wish to make a soda reduction, and do not wish any iron oxide present in the mineral to be reduced, add borax to the soda. Iron oxide remains dissolved in the borax (see last chapter). Add one third (or less) as much borax as mineral.

(*c*) When several metals are present and alloys are formed, treat the alloy with the R. F. on coal to find by the odor arsenic, and by the coats antimony and tin. When arsenic has been driven off (if present), place the alloy beside a borax bead, and treat the button with the O. F. until the borax is well saturated with oxides; then remove the borax bead to a coal crucible and treat it in the R. F. beside lead (as in sec. 56, (*a*)), and the presence of Fe or Co being determined as in sec. 56, (*g*), and the presence of Cu and Ni as in 56, (*h*).

(*d*) Great care must be used in the washing of the substance in the mortar; but skill in the operation of "washing" must be acquired, and it is needed in the assay of gold and of tin, as will be shown under the assays of those metals.

(*e*) It is better to put the mass of coal and soda, cut from the coal after it is powdered, into a small porcelain crucible, and heat it with water over the alcohol lamp to dissolve out the soda. Let it stand a while. The washing is rendered easier by this means.

CHAPTER VI.

QUALITATIVE TESTS.

59. ALUMINUM.—Powdered, placed in a cavity on coal, moistened with a drop of nitrate-of-cobalt solution, and treated with the O. F., gives a blue color (sec. 49).

Caution.—This test is nearly restricted to substances which are nearly white after ignition, and which are *infusible*. Certain substances give a blue, glassy and fused; silica, a reddish blue. In many compounds the wet way has to be invoked in testing for aluminum.

60. ANTIMONY.—(*a*) *Coat.*—On coal in the R. F. compounds of antimony give a white coat nearer the assay than the arsenic coat. The coat is volatile, and colors the inner flame light bluish green, with a yellowish or whitish tinge, which is characteristic (see sec. 46, coats produced by too rapid heating of sulphides). The metal itself, treated on coal in O. F. and removed from the flame, continues melted for some time, the button becoming covered with white crystals. The metal, as well as the coat formed from its treatment with the flame, colors the inner flame pale light bluish green, with a yellowish tinge.

With nitrate-of-cobalt solution (sec. 49) the coat gives a dirty green, to be especially distinguished from the green of a zinc coat treated with the same solution.

(*b*) *Open Tube.*—White fumes, amorphous white sublimate. (Sulphide of antimony too rapidly heated in tube, reddish sublimate).

(*c*) *Flame-coloration.*—Light bluish green, with a yellowish whitish tinge.

(*d*) *In "Glass" Beads.*—No very good reactions (see Table II), except in SPh bead with tin on coal in R. F. (Table III). Thus treated, gray; on longer blowing, clear (to be distinguished from Bi, which gives under same circumstances a blackish gray and opaque bead; and from Pb, a gray bead, not quite opaque).

An antimony coat scraped carefully off the coal, and dissolved in a SPh bead (on wire) in O. F., and then treated in a cavity on coal alongside of tin, in the R. F. gives a bead gray to nearly black, if saturated (to be distinguished from Bi, which with the same treatment gives a bead brownish to black).

(*e*) *Soda Reduction.*—Partly volatilizes, and partly obtained in white shining, brittle metallic buttons.

(*f*) *Alloys* (*or Compounds*) *of Sb and Pb, or Sb and Bi.*—Place the alloy in a cavity on coal, beside a piece of boracic acid; fuse in R. F., keeping flame mostly on flux. The Pb is dissolved in the flux, and an Sb coat is formed which is tested for the flame-coloration as in (*a*).

Sb and Cu, or Sb aud Sn.—As Bi might be present, the quickest way is to fuse the alloy with lead and proceed as above.

(*g*) When galena or other lead sulphides are present, the lead coat might mask the Sb coat, and the object should be to alloy the Sb with the Pb, which is afterwards gotten rid of by means of boracic acid. But the lead must first be reduced to the metallic state. This is done by placing in a cavity on coal a bit of iron wire, with the powdered mineral and a mixture of soda and borax (twice to three times as much soda as mineral, and half as much borax as mineral), and treating with the R. F. Owing to the affinity of metallic iron for sulphur, the latter is "taken up" by the iron, the sulphide is decomposed, and the resulting lead button, freed from slag, is placed beside boracic acid, treated as in (*f*), and a coat of Sb obtained.

(*h*) *Antimony and Arsenic.*—As is more volatile than Sb, and the substance treated on coal with O. F. is gently heated at *first*, then strongly. The As coat may be driven off, and the Sb coat tested for flame-coloration.

61. ARSENIC.—(*a*) *Coat.* — On coal in R. F. gives a grayish to white coat, far from assay, volatile, and coloring the flame light blue.

(*b*) *The garlic-like odor* given out by arsenic compounds when tested on coal with R. F. is *characteristic.* It is best obtained in doubtful cases by mixing the powdered mineral with soda and treating with R. F. In some compounds with Ni and Co and little arsenic the odor is hardly perceptible. In this case treat beside lead in O. F. on coal, and the odor will be obtained.

Also when As is contained in metallic Zn, use the same method. The garlic-arsenic odor must be distinguished from the rotten horse-radish odor of selenium. Some compounds containing arsenic and sulphur too rapidly heated may give reddish or yellowish coats.

(*c*) *Closed Tube.* — Black metallic sublimate, arsenic odor. (*Arsenic sulphides*, brown to black *hot*, red to reddish yellow sublimate *cold*. *Arsenous acid*, white crystalline sublimate.)

(*d*) *Flame-coloration.*—Light blue.

(*e*) *Soda Reduction.*—Arsenic volatilizes and is lost, unless other metals are present with which it alloys. Treat powdered alloy in R. F. on coal for arsenic odor.

(*f*) *Special Tests.*—Place powdered mineral in a closed tube or " matrass," cover with about six times its weight of soda and cyanide of potassium. Heat carefully in alcohol-lamp flame. Wipe out any moisture that may form. Repeat this until no more moisture is given off. Then heat more strongly for a few minutes, and a black lustrous metallic sublimate (" a mirror," so called) of arsenic forms on cool part of tube. Cut off tube (with aid of file) above the " mirror," and heat the part of tube containing the sublimate in the alcohol flame, to get the arsenic odor.

(*g*) In some compounds with a very small quantity of As the wet way has to be used.

62. BARIUM. — The flame-coloration (sec. 51) is the best test (except for silicates). The yellowish green color is more strongly brought out

by wetting the substance with a drop of hydrochloric acid. In the case of silicates wet methods have to be employed.

63. BISMUTH.—(*a*) *Coat.*—Volatile; orange-yellow hot, lemon-yellow cold. (Coat gives no color to flame: distinction from Pb coat, which colors flame azure-blue.) Coat carefully scraped off coal, and treated as in sec. 60, (*d*), gives a brown to black bead.

(*b*) When other volatile metals are present, such as Pb, etc., the Bi coat may be masked. Treat the powdered mineral on coal with a *gentle* O. F. and *gentle* R. F. to get rid of As, Sb, or Cd, if present. Then mix mineral with equal parts (by weight) of iodide of potassium and sulphur, and treat with R. F. (on a fresh piece of coal). A brilliant red coat of iodide of bismuth forms at some distance from assay.

(*c*) The following method of Cornwall ("Blowpipe Analysis," edition 1888, page 121, *c*) is more delicate in presence of much Pb: Mix substance with somewhat more than its volume of a mixture of one part of iodide of potassium and five of sulphur. Heat in open tube. If Bi is present a red (dull brick-red) sublimate is deposited a short distance above the yellow iodide of lead sublimate. In case Sb is present in quantity, as sulphide, remove excess by alternate treatment with *gentle* O. F. and R. F. on coal, *before mixing* with the iodide mixture. Antimony oxide being present, it may be necessary to fuse with an equal volume of powdered sulphur in deep cavity on coal with

R. F., the resulting sulphides being treated as directed for antimony sulphide.

(*d*) *Glass Beads.*—No characteristic reactions of value, except in SPh bead treated on coal with tin in R. F., dark gray or blackish gray and *opaque* when cold. (Table III.)

(*e*) *Soda Reduction.*—White shining metallic buttons. Examine through lens. They have a faint pink color. Treated with O. F. on coal the coat gives no color to flame (distinction from Pb).

Alloys—Treat with strong R. F. on coal for Bi coat.

(Test any coat formed with SPh and tin, sec. 63, (*a*), sec. 60, (*d*).

Make special tests (*b*) or (*c*).

64. BORON.—Flame-coloration (sec. 51) is the best test. In difficult cases use Turner's flux, consisting of 4½ parts bisulphate of potassium, 1 of fluor-spar (free of course from boracic acid). Mix one part of flux with finely powdered substance, moisten with enough water to make a paste, place in loop of platinum wire, and fuse within point of blue flame. A very transient yellowish green flame is produced if boron is present.

Bromine. See Chlorine.

65. CADMIUM.—(*a*) *Coat.*—Reddish brown near assay to orange-yellow farther from it. Iridescent border often. Coat is easily volatilized. (The *metal* burns with brown smoke.)

(*b*) *Closed Tube.*—Lustrous dark gray to black, metallic sublimate, brown border; from certain compounds of cadmium.

(*c*) *Soda Reduction.*—Reduced but volatilizes, with formation of coat.

(*d*) When zinc is present, by careful heating with R. F. (the substance being mixed with soda) the cadmium forms a coat before the less volatile zinc does.

(*e*) The following from Cornwall's "Blowpipe Analysis" is more delicate: "Whether dealing with oxides, metals, or alloys, form a coat in R. F., scrape it off, mix it with a little fine charcoal dust, and heat with a *moderate* O. F. in closed tube. (Too hot a flame may drive off zinc, forming a zinc-oxide sublimate.) A reddish brown ring of cadmium oxide will form a little above the assay, sometimes only perceptible by reflected light. Much cadmium gives a metallic mirror. (*a*) This method will detect $\frac{2}{10}$ of one per cent of cadmium oxide in any mixture of lead and zinc oxide, and much less cadmium in commercial zinc. Arsenic, if present, must be removed by gently heating the substance in O. F. on coal, since it interferes with the test."

Cæsium. See sec. 100.

66. CALCIUM. — (*a*) *Flame-coloration.* — Moistened with a drop of hydrochloric acid and *strongly* heated with tip of inner flame, a red to reddish yellow flame. This is often obscured by other bodies. (Is not given by silicates containing calcium, except wollastonite.)

With the borax and SPh beads does not give reaction of value.

The carbonate (limestone) dissolves with effervescence in the bead, but so do certain other carbonates.

(*b*) *Soda Reduction.*—Infusible, the soda sinks into the coal, leaving lime behind. (Distinction from Sr and Ba, which are absorbed with the soda.)

To detect calcium in some of its combinations the wet way must be employed.

Carbon. See sec. 100.

67. CHLORINE.—(*a*) Form a SPh bead; dissolve copper oxide in it with the O. F. The bead should contain enough to be opaque. Dissolve in this bead the substance under examination, and treat with O. F. If chlorine is present an intense azure-blue flame of copper chloride surrounds the bead. (Bromine gives a similar flame, but greenish blue; greenish tinge along the edges. Iodine, an intense green flame.)

(*b*) *Goldschmidt's Test.*—Fused with powdered bismuth sulphide on coal, a compound of chlorine gives a white coat, of bromine a yellow coat, and a compound of iodine a red coat. (Metallic Bi fused with sulphur makes the bismuth sulphide.)

(*c*) The wet way must be used in many cases to detect chlorine, bromine, and iodine.

68. CHROMIUM.—(*a*) *Glass Beads.*—In *borax*, in O. F., small amount yellow hot, greenish yellow cold. Larger amount, dark yellow to dark red hot, yellowish green cold. In *borax* with R. F., green hot and cold.

In SPh in O. F., reddish hot, fine green cold. In R. F. same as O. F.

(*b*) The presence of Fe may interfere with these colors. Fe colors the SPh bead less strongly than borax. Mn also colors the SPh bead less than the borax. Examine for chromium carefully with the SPh.

(*c*) Pb or Cu with Cr may give opaque grayish to reddish beads cold.

(*d*) The wet way has to be employed if mixed colors prevent the detection of Cr.

69. COBALT.—(*a*) *Glass Beads.*—In borax in O. F. beautiful blue hot and cold. R. F., same.

In SPh, same.

Too much may give a blue nearly black.

(*b*) *Mixed Colors.*—With much Fe and little Co the borax bead is yellowish green to green hot and cold. With more cobalt, bluish green in O. F. To detect Co with much Mn use a SPh bead with the R. F.

Reduce the roasted substance dissolved in a borax bead in R. F. in a cavity on coal beside tin. Add more borax to the bead if there is much Fe. Examine carefully for traces of the blue of cobalt.

(*c*) If the substance contains Ni or Cu, dissolve the roasted mineral in borax. Treat bead beside lead in a cavity on coal with R. F. (sec. 56). If Fe interferes with the detection of the Co, treat the borax bead (after separating it from the lead button) as in (*b*). See (*g*), below.

(*d*) *Soda Reduction.*—Gray magnetic powder, or shining magnetic scales.

(*e*) *Special Tests.*—Arsenides of Co, Ni, and Fe are fused in R. F. on coal till arsenic fumes cease. Borax is then placed beside the assay, the borax melted to a bead, and the assay treated with the O. F. The bead is detached, when cold, from the assay, and a piece of the borax bead treated on platinum wire with the O. F., noting the colors. Fresh borax is placed beside the assay, and the treatment with the O. F. repeated. When the borax glass gets dark on the coal, remove it and test it on the platinum wire. Fe oxidizes and passes into the borax first, then Co, then Ni. They successively give their colors to the different borax beads placed beside the assay. Use but a portion of the bead when testing it on the wire, and be sure it is free from any pieces of the assay. The borax may have to be renewed several times, especially if the first bead is removed before all the Fe oxidizes, or if much Fe is present.

(*f*) In the case of metallic nickel, to detect cobalt, fuse with metallic arsenic in a cavity on coal with R. F. and proceed as in (*e*).

(*g*) Alloys may be treated on coal with R. F., driving off volatile substances; then, when no more odor is perceived, or no more coat is formed, place in a fresh cavity, beside borax bead, and oxidize the assay in the O. F. When the borax is saturated, treat it beside lead with the R. F. (as in sec. 56). Detach the lead button when cold, and examine the borax for Co, treating the bead if necessary as in (*b*).

70. COLUMBIUM.—The wet way must be employed. Only in the determination of certain minerals is it generally required to detect columbium. There are no good reactions before the

blowpipe. With nitrate-of-cobalt solution the mineral assumes a brownish gray color. Columbic acid, dirty green. But these reactions are not of much force.

71. COPPER.—(*a*) *Flame-coloration.*— Pulverize the mineral, roast it in a shallow cavity on coal, and while hot moisten it with a drop of hydrochloric acid; dry it gently, and then heat with the point of the R. F. Most copper compounds will give an *intense azure-blue* flame, which is a *characteristic* and delicate test. (Many copper compounds do not require the roasting, but it saves time in the long-run to proceed as above.)

(*b*) *Glass Beads.*—In *borax* in O. F., small amount, green hot, blue cold; more, dark green hot, greenish blue cold.

In borax in R. F. colorless hot, red and opaque cold.

In SPh in O. F. same as borax, not as intense.

In SPh in R. F. dark green hot, opaque red to brownish red cold.

With tin on coal in R. F. the SPh bead is red *cold* (Table III). This is a good test for even *very* small amounts of copper, the bead becoming perhaps only reddish or red in spots.

(*c*) *Soda Reduction.*—Easily reduced to buttons of metallic copper.

(*d*) *Copper and Lead.*—Place the alloy in a cavity on coal alongside of fused boracic acid. Fuse the button, and when melted keep the inner flame on the flux. The button must not become covered with the flux. After *most* of the lead has been dissolved in the boracic acid remove the button from the slag, and place it in a fresh cavity alongside of a previously made SPh bead. Treat the button with the O. F. The copper oxidizes, and the SPh bead becomes blue (cold) from copper, or green if copper and nickel are present (sec. 56). Or, what is better, determine the presence of copper by treating the SPh bead (after its treatment beside the button removed from the boracic acid) with tin in the R. F. on coal (*b*). The tin and bead must be fused just long enough to adhere well together (sec. 55).

(The red color may be darkened from the presence of certain other bodies; it may be reddish brown.) The bead, colorless while hot, suddenly becomes red on cooling.

(*e*) With minerals it is sometimes best to use a larger quantity, treating it with borax and lead, as directed in 56, (*a*), and separating the lead as in the preceding paragraph, determining the presence of copper with the SPh bead and tin as there described.

(*f*) When volatile metals are present in alloys, drive them off as much as possible by treatment with the O. F. on coal. Fuse the alloy with a little lead in the R. F. on coal, and proceed as in (*d*).

(*g*) *Non-reducible Metals Present in Alloys.*—Treat strongly the alloy with the O. F. beside a borax bead on coal. When the bead is saturated with dissolved oxides, remove it from the button; treat the bead beside lead on coal with the R. F. (as in 56). The "non-reducible" metals will remain in the borax, and the lead button is treated as in (*d*). (The borax may have to be renewed several times, and the several saturated beads fused into one, which is treated beside the lead. This especially if there is much iron present.)

(*h*) *Nickel and Copper.*—Treat the alloy on coal in the O. F. beside a SPh bead, and proceed as in (*d*).

Didymium, Erbium. See sec. 100.

72. FLUORINE.—Form a bead of SPh, and powder it when cold; mix it with the powdered mineral, and place in an open tube, with a strip of moistened Brazil-wood paper inserted in the upper end of the tube. Heat in the flame of the alcohol lamp; *the flame must pass into the tube.* The inside of the tube will be corroded and etched by the hydrofluoric acid which is formed (examine with lens), and the Brazil-wood is turned light yellow.

Gallium, Glucinum. See sec. 100.

73. GOLD.—The only sure and proper test for gold is to make a quantitative assay (see Gold Assay).

74. INDIUM.—(*a*) *Flame-coloration.*—Violet.

(*b*) *Soda Reduction.*—Reduced, but volatilizes and is lost unless very abundant, when it is obtained in white globules. The coal is covered with a coat; dark yellow hot, light yellow cold, volatile in R. F. with the violet flame.

Iodine. See Chlorine.

Iridium. See Platinum.

75. IRON.—(*a*) *Glass Beads.*—With *borax* in O. F., small amount, yellow hot, colorless cold; more,

dark yellow to red hot, *yellow* cold; saturated, dark red hot, dark yellow cold.

With *borax* in R. F., dark red hot, bottle-green cold.

With SPh in O. F., yellow hot, colorless cold; with much, dark red hot, brownish red cold.

With SPh in R. F., with small amount, unchanged; more, red hot, smoky brown cold.

(*b*) *Mixed Colors.—Fe and Mn.*—In borax O. F., yellowish red cold. Dissolve in SPh bead in R. F., when the Fe color can be detected. *Fe and Ni.*—In borax in R. F. may be gray from reduced Ni. Dissolve roasted substance in borax, treat with R. F. on coal beside lead (as in sec. 56). When cold detach button and treat a piece of the bead on platinum wire with O. F. and R. F. to obtain Fe colors. *Fe and Cu.*—Treat in the same way. *Fe and Co.*—The bead of borax in O. F., greenish hot, blue cold depending on relative amounts present. When much Co is present, and only a little Fe, it is best to make a soda reduction, and examine carefully for metallic iron. When but little Fe is present with U or Cr, test in the same way by soda reduction, but the wet way may have to be employed to detect a small quantity of Fe in the presence of U, Cr, or much Co and Mn.

(*c*) *Soda Reduction.*—Obtained as a magnetic iron-gray powder or in iron-colored magnetic scales. (Best to mix charcoal-dust with the soda; sec. 58.)

(*d*) Treat alloys with O. F. alongside of a borax bead on coal, until the bead becomes well saturated with the oxides. Place the bead beside lead, treat with R. F., and proceed as in the case of Fe and Ni, above. If the alloy is very fusible, containing Sn, Zn, etc., the R. F. is made to play on the borax bead, and when the bead has taken up the oxides, proceed as above.

Lanthanum. See sec. 100.

76. LEAD.—(*a*) *Coat.*—Lemon-yellow hot, sulphur-yellow cold. Volatile, and gives an azure-blue color when treated with the inner flame; distinction from Bi coat, which does not give a color to the flame. If Se is present its odor detects it. (See sec. 48 in relation to coats of sulphates, etc.)

(*b*) *Flame-coloration.*—Azure-blue.

(*c*) *Soda Reduction.*—Metallic lead obtained.

(*d*) *Glass Beads.*—In borax in O. F., when the bead is *highly saturated*, it becomes yellow and opaque cold. In the SPh bead, heated on coal in the R. F. beside tin, the bead becomes cloudy and gray, but not quite opaque.

(*e*) *Special Tests.*—When treated with iodide of potassium and sulphur (as directed under Bismuth, sec. 63, (*b*)) on coal, a yellow coating of iodide of lead is formed far from the assay.

In doubtful cases the reduction with soda yielding metallic lead is often the most available test.

(*f*) Treat alloys pulverized with soda in R. F. on coal for lead coat. When but very little lead is present, treat as in (*e*).

77. LITHIUM.—*Flame-coloration.*—Carmine. But the flame is masked by Ca, Na, and Sr, and special tests have to be made. (See Cornwall, page 94.)

78. MAGNESIUM.—With nitrate-of-cobalt solution (sec. 49) a flesh-red, which is characteristic. Heat strongly. But with much Fe present the color may be a brownish red. (Magnesium arsenate, borate, and phosphate give a violet color with the nitrate-of-cobalt solution.) The wet way must be used to detect Mg in many of its combinations.

79. MANGANESE.—(*a*) *Glass Beads.*—In *borax* with O. F. purple hot, purple-red cold. In borax with R. F. colorless.

In SPh with O. F., with little colorless, with much brownish violet hot, reddish violet cold. In SPh with R. F. colorless.

Mn colors borax intensely in O. F. Too much makes the glass so dark as to be nearly black. Mn colors SPh much less than borax. I have seen minerals that colored the borax bead a deep purple from Mn, give with the same amount hardly a tinge of color to a SPh bead.

(*b*) Roasted substances containing but little Mn are dissolved in SPh bead in O. F., and while the bead is still hot it is touched to a small piece of nitre. The bead swells or froths, and becomes when cold either violet, or streaked or spotted with violet, according to the amount of Mn present.

(*c*) The roasted substance is dissolved in a borax bead; powder it when cold. Mix with 3 parts of soda and 1 part of nitre. Moisten it slightly, so as to form enough of a paste to place it in the loop of a platinum wire. Fuse intensely in O. F. When Mn is present a fine bluish green color is produced. The color must not be comfounded with a yellowish green color produced by Cr. (Silica and cobalt both present would obscure this test, as they would give a blue mass.)

(*d*) *Alloys.*—Place the roasted and pulverized alloy beside borax on coal, and treat with O. F. until the bead has dissolved manganese oxide. Examine the color of bead.

80. MERCURY.—(*a*) *Coat.*—Very volatile gray coat from substances containing much mercury. (Mercury iodide yellowish to reddish coat.)

(*b*) *Closed Tube.*—Mix substance with 3 times its volume of *dry* soda. Introduce it into a glass tube carefully, so that none of the assay will remain on sides of tube (sec. 18). Heat tube with alcohol lamp, gently at first. *Wipe out carefully all moisture that may form in the tube.*

Metallic mercury condenses on tube above assay. It may have a grayish slightly metallic appearance, and should be carefully examined with the magnifying-glass.

The minute globules may be collected into larger ones by rubbing with a feather, or tapping the tube on a saucer.

81. MOLYBDENUM.—(*a*) *Coat.*—Treated with O. F., a coat yellow hot, white cold, partly volatile, and when treated with O. F. becomes copper-red. Touched for a moment with the R. F. the coat

becomes blue. Some minerals containing Mo give a copper-red coat near the assay, a very slight whitish coat beyond, which terminates in a bluish to blue border.

(*b*) *Flame-coloration.*—Yellowish green.

(*c*) *Glass Beads.*—In *borax* in O. F., little, yellow hot, colorless cold; much, dark yellow to red hot, opaline to bluish gray cold. In *borax* with R. F. saturated glass becomes brown to opaque.

In SPh in O. F. yellowish green hot, nearly colorless cold.

In SPh with R. F. dirty green hot, fine green cold.

(*d*) *Soda Reduction.*—Steel-gray powder.

82. NICKEL.—(*a*) *Glass Beads.*—In *borax* with O. F., purple hot, reddish brown cold. In *borax* with *R. F.* gray from reduced nickel, longer blowing clear. In SPh with O. F. reddish hot, yellow cold; with much, red hot, reddish yellow cold.

(*b*) When mixed colors are produced from Fe and Ni, Co and Ni, or Mn and Ni to separate the nickel, roast the mineral, dissolve in borax, and treat with R. F. beside lead, in a coal crucible, according to the method given in sec. 56. The lead button is treated beside boracic acid as described under Copper, sec. 71, (*d*), and the nickel determined by treating the small button alongside of a SPh bead on coal with the O. F., as described in sec. 71, (*d*).

When nickel is present alone, the bead will be yellow cold; if nickel is present with copper, the bead is green cold.

(*c*) *Alloys.*—Treat alongside of a borax bead on coal with the O. F. until the borax has taken up the oxides; then reduce with lead, as above (see sec. 56). If the alloy contains much lead, separate by treatment with boracic acid and test the remainder with SPh on coal (see sec. 71, (*d*)).

(*d*) *Arsenides.* See under Cobalt.

Care must be used in observing the colors successively imparted to the borax. If the cobalt has begun to oxidize and pass into the bead containing iron oxide, the mixed colors of Fe and Co, green or bluish green in O. F. and more blue in R. F., will appear, when pieces of the borax bead are examined on platinum wire. A violet-brown or violet cold shows the nickel has commenced to oxidize and pass into the bead containing cobalt, producing mixed colors. See the colors produced by Fe alone, Co alone, and nickel alone, under their respective heads. This method is only mentioned, as on it depends the theory of the nickel and cobalt quantitative assay. To determine the presence of Ni, it would be simpler to roast the arsenides and proceed as in (*b*).

83. PLATINUM. — (*a*) *Palladium, Iridium, Osmium, Rhodium, Ruthenium.* — Platinum occurs generally alloyed with these metals, or associated with some of them. It gives no reactions with the glass fluxes, etc., the small amount of Fe or Cu it may contain alone coloring the bead; and about the best way to determine the presence of platinum is by its appearance and physical properties, etc. Platinum alone cannot be cupelled, as some lead will remain with the platinum, leav-

ing an infinite mass on the cupel. (For platinum with gold, see section 123.)

(*b*) Platinum is generally found in flattened grains (much more rarely in small nuggets), which have a metallic lustre with a bright gray color (but they are frequently tarnished to a dark gray or black). The color is somewhat between that of Fe and that of Ag. The "streak" (the mineral scratched or powdered and its color then observed) is the same color as the metal itself. (Flattened scales of iron oxide, hematite, with a metallic lustre, would give a red streak or powder.) The grains are malleable, but hard. The hardness and specific gravity are: hardness, 4-4.5; specific gravity, 16-19. The metal is *infusible*. It may be slightly magnetic from the iron it contains. It is not dissolved by single acids, being, like gold, soluble only in aqua regia.

The wish is often "father to the thought" in searching for minerals, as in other things; but the substances which might be mistaken for platinum, even by the most enthusiastic imagination (coupled with lack of familiarity with platinum), can be distinguished by remembering—

1st. That platinum is malleable;

2d. That it is infusible;

3d. Noting its great (comparative) weight;

4th. The color of the metal and streak is the same;

5th. That it gives no *persistent* colors with the glass fluxes (at most, colors from the Fe or Cu contained).

The author has even known flattened scales of tarnished dark colored iron pyrites to be mistaken for platinum.

It is needless to call attention to the fact that the pyrites would give, when tested with the blowpipe, reactions for sulphur and persistent reactions for iron. The latter may be entirely oxidized with the O. F. and made to pass into the borax bead. This would not happen with platinum, which, treated beside borax on coal with the O. F., would give to the borax bead the color of the iron it contained (oxidized by the treatment), but would *not* itself oxidize and pass into the borax. Also, pyrites is brittle, is much harder than platinum and much lighter in weight.

The rare native or metallic iron may have a color very much like platinum. But the reactions for iron referred to above at once distinguish it. Native iron is also malleable, and has about the same hardness as platinum, but is much lighter in weight. Specific gravity, 7.8, at most. It is strongly magnetic (platinum at times slightly so).

(*c*) Platinum is generally alloyed in varying proportions with certain other metals, typical specimens of platinum yielding, platinum, 70 to 89 per cent (some specimens less); iron, up to 19 per cent (*generally* from 7 to 12 per cent); and smaller amounts of iridium, osmium, rhodium, palladium, and copper, the last in some specimens as high as 5 per cent, but very much less, generally. A specimen of California platinum (Dana) gave—

Platinum	85.50	per cent
Iridium	1.05	"
Osmiridium	1.10	"
Rhodium	1.00	"
Palladium	0.60	"
Iron	6.75	"
Copper	1.40	"

(*d*) *Palladium.*—In grains, malleable; light steel-gray, rather whiter than platinum; hardness about the same as platinum; lighter in weight; specific gravity, 11.8–12.2; soluble in *nitric acid* (platinum is not); infusible.

(*e*) *Osmiridium and Iridosmium.*—Compounds of iridium and osmium, with traces of rhodium and ruthenium, in flattened grains. The first whiter than platinum; the last dark, nearly lead color. Infusible; not soluble in aqua regia. Specific gravity of osmiridium, 19.5; of iridosmium, about 21. Fused in open tube with nitre give off pungent, irritating fumes of osmium.

84. PHOSPHORUS.—*Flame-coloration.*—The bluish green flame is the most available test, but the wet way has to be used to detect this element in many of its combinations.

85. POTASSIUM.—(*a*) *Flame-coloration.*—The violet-colored flame is often obscured by the yellow flame of sodium, and by the flame of lithium. (The potassium flame may often be detected, even when the other substances are present, by looking

at the flame through two thicknesses of pure cobalt-blue glass.)

(*b*) Make on wire a bead of borax and add a *little* boric acid (powdered), then add oxalate of nickel, and treat with O. F. Enough oxalate of nickel should be used to cause a brown bead when cold. Dissolve some of the substance in the bead in the O. F. If the bead is blue when cold, it shows potassium was present in the substance. In many cases the wet way must be employed.

Rhodium, Ruthenium. See sec. 83.

Rubidium. See sec. 100.

86. SELENIUM. — (*a*) *Coat.* — Steel-gray near, darker gray far from assay. Sometimes with a reddish violet border. Volatilizes, tingeing flame blue. In fusing Se yields its odor of rotten horseradish, which is characteristic.

(*b*) *Glass Tubes.—Closed Tube.*—Dark red, nearly black, sublimate, with Se odor.

Open Tube.—Steel-gray sublimate with reddish edge, often with white volatile crystals, with Se odor.

87. SILICA.—It is detected by being *virtually* undecomposed in the SPh bead. The substance, in this case preferably in very small fragments, is treated in the SPh bead with the O. F. The bases of the silicate are decomposed, but the silica is left floating around in the bead, the so-called "silica skeleton," of the same general form as the original fragment, and translucent.

88. SILVER.—The proper and sure way to de-

termine the presence of silver is by a quantitative assay (see Silver Assay).

(*a*) *Coat.*—Heated strongly in O. F. on coal a reddish to reddish coat, often slight. (For coats of silver when with certain other metals, see Table I.)

(*b*) *Glass Beads.*—In SPh with O. F. yellow, highly saturated opaline, and yellow cold.

(*c*) *Soda Reduction.*—Buttons of metallic silver.

89. SODIUM. —*Flame-coloration.* — The yellow flame is characteristic. (If much lithia is present the flame may be reddish yellow. *Much* potassium may tinge the flame violet, but the yellow of sodium will also be detected.)

90. STRONTIUM. — (*a*) *Flame-coloration.* — The red flame is the most characteristic test. A little strontia, with calcium, lime, or barium, may have to be treated by wet methods, which also have to be employed in the case of certain silicates.

(*b*) *Soda Reduction.*—The carbonate goes into the coal with the soda (as does the carbonate of barium): distinction from calcium carbonate, which does not sink into the coal.

Tantalum. See sec. 100.

91. SULPHUR.—(*a*) Many compounds of sulphur give a sulphurous odor when roasted on coal.

(*b*) Place powdered substance in open tube, insert at end a strip of moistened blue litmus-paper, and heat tube in the alcohol-lamp flame. (The tube is held in the " matrass-holder.") Sulphur fumes will redden the paper, and the odor of sulphur will be perceived.

In *closed tube* sulphur gives a yellow sublimate.

(Sulphur and arsenic, sublimate brown to black hot, red to reddish yellow cold. Sulphur and antimony, black hot, brownish red cold.)

(*c*) Fuse substance mixed with soda (which must be pure, and entirely free from sulphur) on coal with O. F. When cold, cut out the mass with adhering coal. Pulverize. Place on a *bright* silver coin, and moisten with a drop or two of pure rain-water. Sulphur will cause a black to brownish black stain to form on the silver. Se may do the same, but its peculiar odor will be apt to betray it. (Te may also give a stain.)

92. TELLURIUM.—(*a*) *Coat.*—White, with a red or yellow border; volatile, with green flame.

(*b*) *Flame-coloration.*—Green.

(*c*) *Open Tube.*—Gray sublimate, fusible to colorless drops.

Closed Tube.—Strongly heated, lustrous metallic drops.

(*d*) Mix with soda and charcoal-dust, place in a closed tube, and fuse the mass. When cold drop in a few drops of boiling water. The water becomes of a purple color when Te is present.

93. THALLIUM.—(*a*) *Coat.*—White, near assay brown; volatile, with green flame.

(*b*) *Flame-coloration.*—Green.

Thorium. See sec. 100.

94. TIN.—(*a*) *Coat.*—Pale yellow hot, white cold. (The coat is *practically* non-volatile, being only with difficulty volatile in R. F.) The coat moistened with nitrate-of-cobalt solution (as directed in par. 49) gives when cold a bluish green.

To be carefully distinguished from the fine yellowish green or grass-green of zinc.

(*b*) *Soda Reduction.*—Metallic buttons of tin (add borax to the soda and coal-dust, sec. 58).

(*c*) When Sb is present, treat with O. F. to drive off Sb before the formation of the tin coat. In the case of other volatile metals, whose coats might interfere with the tin coat, moisten the coat when cold with nitrate-of-cobalt solution, and treat carefully with O. F. The tin coat yields its color and the other coats are volatilized.

(*a*) Tin in the presence of much zinc in some alloys will hardly afford certain indications of its presence by its coat, and the wet way must be employed. In the case of minerals, treat with strong R. F. for tin coat. If much Pb or Bi are present, observe cautions in (*c*).

95. TITANIUM.—(*a*) In *borax* with O. F., much, yellow hot, colorless cold. Becomes enamel-white when saturated.

In *borax* with R. F., much, dark yellow to brown. Saturated can be "flamed" enamel-blue.

With SPh in R. F. yellow hot, fine violet cold; but if Fe is present, brownish yellow to brownish red cold.

With tin on coal in R. F. violet, if too much Fe is not present.

(*b*) When moistened with nitrate-of-cobalt solution and treated in O. F. yellowish green (not to be confounded with the green of zinc, which, moreover, yields a coat; Ti does not).

96. TUNGSTEN.—(*a*) With *borax* in O. F. behaves

much like Ti; with *borax* in R. F., much, yellow to dark yellow hot, yellowish brown cold; with SPh in R. F. dirty green hot, blue cold.

If Fe is present the SPh bead in R. F. is yellow hot, brownish red to blood-red cold. Treated with tin on coal in R. F. the SPh bead is blue cold, if too much Fe is not present.

(*b*) *Soda Reduction.*—Metallic infusible powder. If too much soda is used a yellow compound of soda and tungsten, with a metallic lustre, is left on the coal. Both with tungsten and titanium wet methods have to be employed when the colors of the beads are not characteristic.

97. URANIUM.—With borax much like Fe.

With SPh in O. F. yellow hot, yellowish green cold.

With SPh in R. F. dirty green hot, fine green cold. In the presence of Fe the wet way must often be used.

98. VANADIUM.—Much, with *borax* in O. F., yellow hot, greenish yellow cold. With *borax* in R. F. brownish hot, chrome-green cold.

In SPh with O. F. dark yellow hot, light yellow cold (much).

In SPh with R. F. as with borax.

The wet way has to be used often when "non-reducible" metals which color the beads are present.

(For determining small quantities of W, Ti, V, etc., when the ordinary tests fail, by means of the colors of their solutions, see Cornwall, page 133.)

Yttrium. See sec. 100.

99. ZINC.—(*a*) *Coat.*—Yellow hot, white cold. Treated with nitrate-of-cobalt solution (as directed in sec. 49) the coat becomes of a fine grass-green. (A more yellowish green than the coat of tin.) This is characteristic.

Roast the mineral, mix with same amount of soda and half as much borax, and treat with a good R. F. on coal. Moisten the coat with a drop of nitrate-of-cobalt solution, dry gently, and then treat well with the O. F. Before fusing the assay in R. F. it is often well to moisten the coal (before the formation of the coat with the nitrate-of-cobalt solution). Though roasting is not needed with all ores, yet it serves to help get rid of other volatile metals whose coats might interfere with the zinc coat.

(*b*) *Soda Reduction.*—Volatilizes. In *traces* may alloy with other metals present.

(*c*) If Sb is present it must be gotten rid of by treatment in the O. F. before formation of coat. In the case of other volatile metals (Pb, Bi, Cd) proceed as in 94 (*c*), moistening the coat formed with nitrate-of-cobalt solution, and treating with O. F. carefully at first, to drive off the other volatile coats.

(*d*) Treat alloys with strong R. F. for coat. Even when much tin is present the Zn coat may be detected, as it forms first. In the case of some artificial alloys wet methods must be used.

100. The following elements, mostly of no commercial importance, do not afford before the blowpipe characteristic reactions, and must be tested

for by the wet way: Cs, Ce, Di, E, Ga, Be, La, Rb, Ru, Ta, Th, Y.

CARBON.—Minerals containing carbonic acid effervesce when a few drops of dilute nitric acid is poured on the pulverized substance, heating if necessary. Carbon in the form of graphite, and the diamond, finely powdered and ignited with the O. F., burns.

In the closed tube a bituminous or burnt odor, fumes, and an oily coating are given by many hydrocarbons.

CHAPTER VII.

QUANTITATIVE ASSAYING.

101. (*a*) *Sampling.*—This is one of the most important things in an assay. Too much attention cannot be given to obtaining a fair sample of the ore for assay. That is, the substance assayed should be a fair average of the ore, carrying the metal sought for, in the same proportion it is found in the ore.

No matter how carefully and skilfully an assay is conducted, it is useless if the sampling was not properly attended to, for the result obtained will not represent the true amount of metal in the ore: it may be too much or too little. Even with the regular furnace assays the proper sampling of the ore is of prime importance. In a blowpipe assay it is still more to be insisted on; for when it is considered that gold ores are worked at a profit in many instances when they contain as little as half an oz. troy to the ton of rock, or in the proportion of about 58,000 parts of gangue to one part of metal, it will be readily seen that a blowpipe assay of perhaps 1½ grain taken from a piece of such ore weighing, say, an ounce (and in which there is less than ⅛ of a grain of metal), may show no gold at all, if taken from a barren part of the rock. Or, again, the

$\frac{1}{8}$ grain of gold might be concentrated in the part of the ounce-piece taken for assay. The result would show the ore to carry gold at the rate of 6148 ounces to the ton. Although the case of gold is an extreme one, the same remarks as to the necessity of a good sample apply to all other assays, especially silver.

No labor should be spared in endeavoring to obtain a fair sample, nor should the operation be slighted.

102. For the blowpipe assay, take a pound or more of the ore, endeavoring to select a part that faithfully represents the original mass. Break it up with the heavy hammer, using a flat hard rock as an anvil, or a heavy piece of iron, or an old blacksmith's anvil.

The ore should be well wrapped in thick paper to prevent fragments from flying. When it is broken into pieces about an inch square they can be treated on the small anvil with ring, which should be placed on a large sheet of paper to save any dust or pieces that may fall off the anvil.

(Sometimes small particles of the mineral adhere to the paper wrapped around the pieces; these should be carefully brushed off and added to the remainder of the broken rock.)

When the pieces are about the size of filberts, lay the sampler on a sheet of paper, collect all the broken rock and dust in a thick piece of paper folded like a scoop, and toss them *gently* on the sampler. After all the fragments have been thrown on the sampler, remove the pieces, etc., that have been caught by it, and spread them thinly, after well mixing on a sheet of paper in the form of a square.

(The part that has gone through the sampler bars may be rejected or laid aside for further examination.)

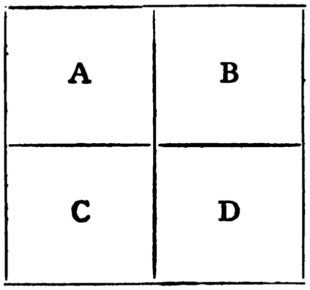


FIG. 12.

Now take the spatula and make a cross with it through the mass on the paper, thus:

Take a spoonful (with large spoon) from *A*, then one from *B*, *C*, and *D*, and repeat twice or more, taking each time spoonfuls from different parts of each quarter of the square. These spoonfuls are spread on a new sheet of paper, and this operation of quartering is again repeated. Break up the fragments thus selected as small as peas; continue quartering until the amount has been reduced to 30 or 40 grains. This is pulverized finely in the iron mortar. Do not try to pulverize all at once, but put a little at a time in the mortar, pouring out the result on a piece of paper, so arranged that the powdered ore may not be knocked off and lost.

(Examine the mortar before using, and see that it is clean; brush it out well with the large brush.)

The fine powder is now *carefully* brushed from the mortar and poured into the 100-mesh box sieve (which should have been brushed out before using). Sift it. If the pulverizing in the mortar has not been thoroughly done, some of the material may refuse to pass through the sieve. Take out the middle part of the sieve from the bottom or cup, and pour *carefully* back into the mortar the portion too coarse to go through the meshes, and repulverize, sifting this through the sieve. Carefully pour out the powdered ore on a clean piece of paper, invert over the paper the top of the sieve, and brush out carefully with the camel's-hair brush. Do this with the bottom of the sieve, and with the part containing the meshes, which should be tapped on the sides smartly with the finger-nail, and carefully brush out on both sides. The powdered ore is now well mixed and requarted, taking with the ivory spoon the proper amount for assay, which is immediately weighed on the balance. (What is left over of the powder is carefully folded in the paper, on which is written the nature of ore, locality, date, and any other useful data, and put away in a box.)

(For the method of procedure in the case of metallic scales that will not go through the sieve, see Gold Assay.)

The above is, of course, susceptible of modification, the object merely being to obtain a fair sample of the ore; but the author has found the method above described the best for this end, in spite of the time it takes. This sampling is not necessary when there is no gangue, and the metal sought is diffused evenly through a metallic mineral, as in the case of galena carrying silver, where a piece of the galena free from gangue is taken, or of iron pyrites under similar circumstances (free from gangue) assayed for gold.

103. SILVER ASSAY.—(*a*) *Coal-crucible Assay.*—Weigh out accurately 1½ grains troy of the sampled ore; remove carefully from balance-pan to the mixing capsule. Take one ivory spoonful of powdered borax-glass; place it in the mixing capsule with the ore. Pull out the piston of the "lead measure" until the top of the piston coincides with the line marked 5 on the measure. Now pour into the measure test-lead until filled. Add the lead to the ore and borax in the mixing capsule.

(*b*) In case of very infusible ores a little more borax may be used. For the minerals mentioned below more lead is required. They need as follows (the more copper present the more lead is needed):

Tin pyrites (with Cu)......	7 parts lead required
Bournonite...............	7 " " "
Copper pyrites Bismuth (with Cu) Tetrahedrite	......10 " " "
Bornite.................	..12 " " "
Chalcocite................	..15 " " "

In general, minerals with over 7 per cent copper or 10 per cent nickel require more than the 5 parts of lead. Lead can be added, however, during the cupellation (which see).

(*c*) Molybdenite requires soda, as well as borax and lead; 1½ grains ore, 1½ grains soda, 1½ grains borax, and 5 parts lead.

Oxidized lead compounds need the addition of soda and may need no lead; but they had by far better be assayed by Fletcher furnace method as had galena.

104. (*a*) The contents of the mixing capsule is now thoroughly mixed with the ivory spoon. A piece of soda-paper (cut in a rectangle about 1½ in. by ⅞ in.) is wrapped around the "soda-paper form" firmly, about one eighth of an inch of paper projecting past the end; this is neatly pressed down (first pinched together and folded down on itself) with the finger-nail. The "form" is inverted and removed from the paper cylinder, which is held between the thumb and forefinger of the left hand, resting on the second finger. The mixing capsule is taken in the right hand, and its contents carefully poured into the paper cylinder; the mixing capsule is tapped with the finger, and any particles adhering to the capsule carefully brushed into the paper cylinder. The top of the paper is pinched together and folded smoothly down on the contents; the paper cylinder is pressed into a coal crucible, whose cavity has been enlarged as directed in sec. 10, (*b*). The coal crucible rests in the socket of the "clay cylinder." The clay cylinder is held by the left hand, and the contents of the crucible treated in a good and strong R. F. At *first*, to burn the paper, it may be necessary to hold the assay nearer the outer part

of the flame than in a pure R. F., but after the paper is consumed on top only a strong pure R. F. is used.

(*b*) The clay cylinder is kept gently turning in the left hand. The borax melts; the lead buttons run together. When the upper part of the assay is fused, and the lead that can be seen is in one button, the cylinder is inclined a little more towards the flame, the blast is made a little stronger, and directed between the borax bead and the side of the crucible. This is done to cause the assay to roll over, so the flame can be directed on the under part of the bead, which is as yet unfused. The bead rolls over, either entirely or on its side, the paper on the bottom is consumed, and the assay then powerfully played on with the R. F., which should cover the borax. The lead must be made to run into one button. There must be no scattered globules of lead. The assay must be kept moving around the crucible, the borax bead partly on top of the lead button. After the lead button has been made to revolve several times around the borax bead this part of the operation is finished.

(*c*) The borax must be globular, and must ***not spread*** over the ***coal.*** This is the greatest difficulty to contend with in this part of the assay. If the R. F. is not strong and ***pure*** the lead oxidizes and the borax spreads out, spoiling the assay. A strong blast of the R. F. directed on any part of the bead that commences to stick to the crucible ***may*** bring back the bead to its proper shape. Generally, a minute button of lead, separated from the button to which it should be joined, will be seen at the point where the borax bead has commenced to stick to the coal crucible. If the borax cannot be brought back to its proper shape, let the assay get cold, and, inserting a pin under the slag, turn it over as in sec. 56, and then treat again with R. F.; but the result will not be as certain as if the assay had run properly from the beginning.

The time for this fusion of the assay varies with the skill of the operator and the material assayed, but from four to five minutes may be taken as an average.

105. REFINING.—The button and slag, when cold, is taken out of the crucible, and button freed from slag by wrapping both in paper and *gently* hammering on the steel anvil. If the lead button is dull and dark (before the hammering), instead of showing the light appearance of melted lead it should be refined. The button is placed beside twice its volume of borax (previously made to a bead) in a coal crucible. If not too much burned away, the same coal crucible that was used for the fusion will do. The button and borax are now melted with the R. F., which is changed to an O. F. and directed on the button, and the treatment continued to get rid of S or Sb until the button becomes bright and "refined." If copper is present, the copper cannot be removed by this treatment, and the lead, when cold, will still be dark or dull. The button must

be kept from running under the slag. Necessarily, in oxidizing the lead, many small globules of lead will appear around the edges of the slag (which is not globular in form, as in the fusion). No attention should be paid to these, as they do not now matter. When the lead bubbles gently it generally indicates the end of the refining. When cold, remove the lead from the slag, and carefully cleanse it from any slag, as before.

106. SCORIFICATION.—We should now have the silver originally contained in the ore taken for assay, in the lead, called the "rich-lead." It contains, however, too much lead for a blowpipe cupellation, which excess of lead we get rid of in the next operation of scorification.

Place a clay *capsule* in the end of the "clay cylinder." In this capsule place the rich-lead. Melt it with a R. F., and as soon as melted oxidize it with the O. F., *gently* at *first*. Keep the button moving around the capsule. It grows smaller and smaller as the operation continues, the lead oxide coating the capsule. The clay cylinder should be gently turned from side to side in the hand. When the lead has grown as small as half a hempseed or so, bring the button, by inclining the cylinder and by the force of the flame, towards the edge of the capsule, and *slowly* remove it from the flame. When cold, place the edge of the capsule (near which the button is fixed) on the anvil, and break it off by very gently tapping with the small end of the hammer.

In most works on blowpipe assaying the concentration of the rich-lead is directed to be done by a rough cupellation; but I have found the method described above—suggested by Atwood—superior for the following reasons, the first of which Atwood gives:

1st. There is less loss of silver in scorifying in the capsule than by cupellation.

2d. In concentration by rough cupellation small portions of the bone-ash soaked with litharge are very apt to remain firmly fixed to the lead button, are very difficult to remove, and if not entirely removed interfere very materially with the fine cupellation.

107. (*a*) *Cupellation.*—Lead oxidized on the surface of a cupel made of bone-ash has the property of sinking into the cupel, oxidizing at the same time base metals which are present, and carrying them with it into the cupel. This property is taken advantage of in separating the noble metals from an alloy of lead.

(*b*) The cupel is placed on the cupel support, carefully examined to see that it is clean. Any dust is removed by blowing. The rich-lead is placed in the cupel, melted with the R. F., and as *soon as melted* the O. F. is directed on the cupel immediately in front of the lead button. The cupel becomes red hot; the button is kept gently moving about the cupel; it grows smaller and smaller by the absorption of the oxidized lead by the cupel. Just before the operation is completed the button becomes fixed on the cupel is covered with a play of prismatic colors which rapidly revolve, and as the last of the lead leaves the button it "brightens," throwing out a flash of light called the "brightening,"—hard to describe, but when seen easily recognized. (This last phenomenon may not be noticed with very small buttons before the blowpipe.)

Now nearly touch the button with the point of the inner flame, and then *very slowly* remove from the flame. If care is not taken, and the silver button is too suddenly chilled, if it is of any size it is apt to "spit," or violently project from itself small particles of silver, which are lost, and the assay ruined. (This spitting is supposed to be due to the fact that silver [melted] absorbs oxygen, which when it is suddenly cooled it throws out violently, accompanied by particles of silver. Some claim it is merely due to sudden contraction of the cold outer crust on the hot interior; but as the spitting is confined to silver buttons, the first explanation is probably the right one.)

Too hot a blast causes loss of silver. Do not cupel too hot: silver is oxidized. On the other hand, do not cupel too cold: the button then becomes surrounded with unabsorbed litharge and is "frozen" to the cupel. In this case increase the intensity of the flame, and the litharge can be caused to be absorbed by the cupel. It is not easy to describe the exact degree of heat. The cupel should be *just* hot enough for the litharge (lead oxide formed from the oxidizing of the lead) to be rapidly-absorbed.

(*c*) Examine now the silver button through the lens. The button should be round, bright, and silver colored. If dull dark, and flattened, with a dark stain (dark green to nearly black) on cupel, copper may be present. If dull grayish white and reticulated, tellurium. In either case melt about one grain of test-lead in a cavity on coal. When in a button, remove it (after it is cold) to the anvil, and cleanse it from any charcoal. Flatten it slightly and place it on the cupel, touching the silver button; melt the two together in a gentle R. F. As soon as melted, use the O. F. and begin the cupellation again, directing the button to a fresh place on the cupel. When the operation is completed, if the button is still not of the proper silver color, repeat the cupelling with more lead. Tellurium may cause the treatment to be repeated several times, as may much copper. The silver button must be carefully removed from the cupel with the pliers, or with point of knife if too small for pliers, cleaned with the brush from any adhering bone-dust, and is now ready for weighing, or, if too small for weighing, is measured on the ivory scale, according to the directions in secs. 21, 22, which see. The mode of calculating the amount of silver the ore carries is explained in sec. 24. For instance, 1½ grains were taken for assay.

Divide 450.09992 (the "assay ton") by 1½: the result is 300.06. If the silver button is contained between the diverging lines at line numbered 18, see the number on the right, which is .16 ($\frac{16}{100}$ of a milligramme, the weight of the button). Multiply .16 by 300.06: the result, 48, shows the ore carries 48 ounces of silver to the ton.

If the button is large enough to weigh, then weight of ore assayed : to weight of button :: 29166.66 : number of ounces of metal the ore carries to the ton. If French metric weights are used, multiply weight of button in milligrammes by 300.06 (when 1½ grs. are taken).

(*d*) With some ores, during the fusion, the borax bead becomes covered in spots with a scum that revolves rapidly over the borax, from time to time passing over the melted lead button, not uniting with it, but passing from it again to the borax. When cold, the borax is covered (maybe only in spots) with a gray metallic scum. This is caused by carbon in the form of graphite.

(*e*) Certain of the base metals require a large amount of lead to carry on the cupellation properly and get rid of all but the noble metals, as (copper, tin, nickel, etc.). If enough lead was not used at the start, more can be added as required during the cupellation, as described in (*c*) in relation to copper.

Silver undergoes a slight loss by cupellation. The more lead that is used the greater the loss. The loss arises from oxidization, and most from silver being carried into the cupel with the lead oxide.

In the blowpipe assay the loss is less than with the regular furnace assay, for there the lead button is generally at once cupelled; in the blowpipe assay it is first reduced in size by scorification, and there is less loss by scorification than by cupellation.

A table was drawn up by Plattner (modified by Atwood) showing the loss sustained by the silver button with different amounts of lead. *Practically, no attention* is paid to the cupellation loss, nor is it reported in assays. The table only shows the amount of loss sustained by buttons containing from one per cent of the weight of the assay in silver up. (This is a pretty rich ore—nearly 300 oz. to the ton.) For small buttons the table is not needed. For these reasons the Author deems it of not sufficiently practical importance to insert here.

The following extracts serve as an illustration from the table:

The silver button was 1 per cent of weight of ore taken. Five times the weight of the assay had been added in lead. The cupellation loss would be .01; that is, the true amount of silver was $\frac{101}{10000}$ of the ore taken instead of $\frac{100}{10000}$, or the ore carries silver at the rate of 294.5 oz. instead of 291.602.

The silver button was of the same weight as before, same amount of ore taken for assay. Lead to the amount of 16 times the weight of the ore was added at different times during the assaying. The cupellation loss would be .05; that is, the true amount of silver was $\frac{105}{10000}$ instead of $\frac{100}{10000}$ of the ore taken, or 306.202 instead of 291.602.

108. *Fletcher Blowpipe-furnace Assay.*—(*a*) This useful piece of apparatus has been described in

sec. 11. By its use larger amounts of ore can be assayed, and the process is more accurate than in the *coal*-crucible assay, as there is not so much danger of unskilful manipulation vitiating the assay. The blowpipe used is Fletcher's New Patent No. 42. This is furnished with two jets—Fletcher's patent hot-blast jet (consisting of a coiled tube in which the air is heated by the lamp flame), and an ordinary jet. *The latter* is the *only* one *used* in *this* assay. (The patent coiled jet can be discarded. I have not found it serviceable in the operations of blowpipe assaying.) The blowpipe consists of a mouthpiece, connected by a rubber tube to the "stem" in which a tube having the jet at its extremity is screwed. A moisture-chamber collects the moisture. *The use of this blowpipe is only recommended with the blowpipe furnace, and not for other blowpipe work.*

(*b*) Place a small wooden box, some three or four inches high, on the table, and to the left of the operator. On this box put the small anvil, and on this the furnace (it is better, if one has a spare furnace cover, to place it on the anvil, and the furnace on the spare cover). The edge of the anvil should touch the right side of the top of the box, and the furnace so placed that its side-hole should be above and on a line with the right side or face of the box (Fig. 13).

A *clay* crucible is examined to see that it is clean, and is placed in the furnace. The charge (see farther on) is now carefully poured from the mixing capsule into the crucible, a little at a time, and pressed firmly down with the end of the "soda-paper form." More of the charge is then placed in the crucible, and again pressed down. When all is in, the mixing capsule is tapped with the finger-nail, and any particles carefully brushed with the camel's-hair brush into the crucible. Three or four ivory spoonfuls of dry salt are placed on top of the charge and smoothed down with the soda-paper form. The cover of the furnace is now placed on it.

The *alcohol* lamp is placed as close to the box as possible, and it should stand at such a height that the top of the wick reaches just *under* the side-hole in the furnace; that is, the top of the wick should be on a line with the bottom of the side-hole, or a little below it. (The wick must not be pulled out too much, giving a "bulky," unmanageable, flaring flame, as is the case when alcohol lamps are used for ordinary purposes. The flame must be compact.) The lamp is lighted, the blowpipe mouthpiece pressed against the lips, the small end-piece grasped with the teeth, the main stem of the blowpipe held in the right hand, the jet just outside of the flame,

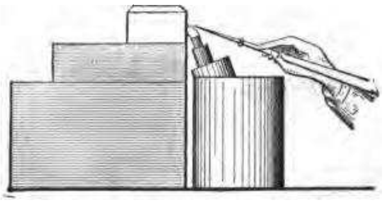


FIG. 13.

and against it. The jet is pointed upwards, and the flame blown upwards through the side-hole.

(*c*) Heat gently for a few instants until the furnace is warmed, and then powerfully. Soon the furnace gets red hot near the hole. In a short time the salt begins to decrepitate, and some of it flies out of the hole in the cover. (To guard against being hit by any of the flying salt, do not put the face near the furnace, but keep the length of the rubber tube away.) The furnace all this time is getting hotter and hotter, and soon the odor of the burning argol or flour is perceived. Now the interior of the furnace as seen through the hole in the cover grows red, then bright red, then orange-red, then bright orange-red, and flashes of light seem to come from it (through the hole in the cover). This is caused by the bubbling of the contents of the crucible. The furnace interior is now of a *clear* orange; and look through the hole in the cover (without stopping the blowing) to see if all boiling has stopped, and the contents of the crucible is in quiet fusion, also to see if no separate globules of lead are on top of the slag. If they are, continue the heat powerfully; they will unite with the main button (if a proper charge has been used). When the slag is fluid and clear, the main button may be often seen, in the middle of the crucible and at the bottom.

Blow sharply for another half-minute, and the fusion is done.

The time taken varies with the nature of the ore, the fluxes used (their quantity, etc.), and the degree of heat employed; from four minutes to eight minutes generally; five minutes is about an average; rarely as long as ten minutes.

(*d*) Let the furnace and crucible become cold. Lift off the cover with the large pincers. Lift the furnace off (with the large forceps) and place it on the table. Take out the crucible. If this should stick to the furnace, on account of some of the salt having melted between it and the furnace body, it can be detached by breaking off the offending edge of the crucible with the forceps. If the cover of the furnace should stick from some cause, put the end of a file, or a nail, etc., in the side-hole of the furnace, pressing it down to the table, and with the other hand pull off the cover by means of the forceps.

(*e*) The inside of the crucible should indicate perfect fusion; the melted charge should be well settled towards the bottom, and covered with an even, smooth coating of melted salt. Place the crucible on the anvil, within the iron ring, and break it open with the hammer. The lead should be in one button, at the bottom of the crucible, and over it the slag. Examine the slag with the lens. It should be well melted, glassy, and of uniform texture; not earthy, nor rough, indicating improper fluxes or insufficient heat. (The color of the slag varies with the kind of ore operated on, etc.) Look now carefully for any lead scattered through the slag. If found, it shows insufficient heat, too short a treatment, or that the proper kind of fluxes to make the slag fluid were not used. In this case the assay (for the precious metals) is spoiled, and has to be made over. It is true the slag may be pulverized, and washed to obtain the scattered lead buttons, which are added to the main button; but this will not do for gold or silver. Add to the charge more silica or borax or soda, according to the nature of the ore (see under Charges), and being careful to use a strong heat.

When iron has been used to remove sulphur, above the button there may be a "matte" of more or less metallic-looking sulphides. If the matte is *abundant*, it is well to detach it as completely as possible from the slag, place it in a coal crucible beside a very little test-lead and enough borax, and treat with R. F. (as in sec. 56), adding the lead button to the main button. There will not be much matte if the *roasting* has been properly *done*.

(*f*) If the assay has been successful, the button is scorified and cupelled as in sec. 106 and sec. 107 [the "refining," sec. 105, is *not* used with the furnace assays], and weighed or measured according to its size.

(*g*) *Two* assays (at least) should always be made when assaying for silver; the "rich-leads" are fused together, and cupelled as one. A better and safer and fairer result is obtained; also, the resulting silver button is larger, and more easily handled, especially in the case of very lean ores.

109. *Roasting.*—For the blowpipe-furnace crucible assay, for silver and gold, ores must be *well roasted* that contain sulphur, arsenic, zinc, or antimony. (Exception in the case of galena assayed by the nitre method.) If this roasting is not done, there is a danger of loss by the slag or by volatilization: by the slag, as in the case of sulphur, when oxysulphurets are formed which go into the slag (see under Litharge, sec. 35); by volatilization, as when zinc carries away silver. Weigh out the charge of ore; it must always be weighed before roasting. Place a clay capsule in the furnace, pour in it the ore, and proceed as in the fusion, except the cover of the furnace must be left off, and the *heat must only* be a *dull* red. The ore must not fuse together. It should be turned over and mixed from time to time with a bit of wire, so that all parts may be acted on. When the ore has a tendency to fuse together silica should be mixed with it. Charcoal-dust can be advantageously used with ores like copper pyrites and with those containing As or Sb, but the coal must be well *burned out* before placing charge in crucible. When no more fumes arise and the assay is "sweet," the roasting is done. The ore should contain no metallic particles, and should be *pulverulent.* When cold, powder it gently again if necessary. Weigh the roasted ore, for from this weight we know the proportion of fluxes to be used. From the weight of ore *before* roasting is calculated the amount of metal it carries.

110. *Charges.*—(*a*) It is impossible to give one charge for all ores. The charge necessarily varies with the nature of the kind of gangue, the combination the metal sought for is in, etc. The object is to combine with the mineral such ingredients as will make a fusible slag, to dissolve the stony and earthy gangues, and absorb the base metals and other impurities. The litharge is reduced to metallic lead by suitable reducing agents, and the lead takes up or alloys with the silver or gold in the ore. Iron is used to combine with any sulphur that may be left after roasting. Read over the action in the crucible of the different fluxes, etc., in the chapter on Reagents.

(*b*) *Owing to the different conditions of heat, etc., charges that answer for the regular furnace assay cannot be used, as a rule, for the blowpipe-furnace assay. The charges given will serve as types or guides, and have been used with success by the author on various ores, but may be varied by the operator according to the nature of the ore.*

Remember, the general rule is: acid fluxes are used for basic impurities, and basic fluxes for acid impurities. For instance, the presence of much lime (basic) calls for silica (acidic) or borax as a flux.

Iron (basic) calls for the acidic silica or borax.

Quartz (acidic) calls for soda (basic) as a flux.

Consult chapter on Reagents.

(*c*) When iron is used, insert the iron wire into the charge, and press down tightly around it. The wire had better be in two pieces.

(*d*) The amount of ore taken in these charges (3 grains) can be varied at will, limited only by the small size of the clay crucibles. With heavy ores occupying a smaller space larger amounts can be used, provided there be room for the right proportion of fluxes. The crucibles must not be filled to the top. The charge (pressed down) should leave at least ⅛ inch.

(*e*) If a charge eats through the crucible, it indicates that silica is wanting in the mixture, and the ore has taken silica from the clay of the crucible for its needs. (Litharge has a corroding action on crucibles, but hardly in the small proportions used in these assays.) It is a good plan to put at the bottom of the furnace a *small* piece of a broken crucible, right in the middle of the furnace bottom, so that if the charge at any time should eat through the crucible, the lead falls on the piece of crucible, and can be easily removed, otherwise the furnace might be ruined in trying to pry the lead away. All fluxes should be kept from touching the furnace, but with careful using they last a long time. (The Author has stated in another part of this book that he used *one* for over three hundred assays.) If heated too suddenly at first, they may crack from the side-hole up to the top,—a fine crack, which in no way interferes with the assay. But in the course of many assays it widens until the furnace may become unserviceable. They are easily replaced.

(*f*) Have a regular course of procedure in weighing. Weigh first the ore on the balance, then weigh in the hand scales the fluxes—first the soda, then the pot. carb., borax, litharge, reducing agent, silica, etc., in order named.

111. *Charges for Silver Assay.*—

(*a*) For ores containing lime, or "heavy spar:"

Ore	3	grains.
Soda	3½	"
Borax	2½	"
Litharge	5	"
Argol	½	"
Silica	½	"
Salt, cover.		

(*b*) "Dry ores," free from sulphur:

Ore	3	grains.
Soda	3	"
Pot. carb	2	"
Borax	1½	"
Litharge	6	"
Flour	1	"
Salt, cover.		

(*c*) Much iron oxide:

Ore	3	grains.
Soda	2½	"
Carb. pot	2½	"
Borax	2	"
Litharge	5	"

Argol	1 grain.
Silica	$1\frac{2}{10}$ "

Salt cover.

(*d*) Iron pyrites (not including ores with copper):

Roasted ore	3 grains.
Soda	3 "
Pot. carb.	$1\frac{1}{2}$ "
Borax	$2\frac{1}{2}$ "
Litharge	6 "
Flour	$\frac{1}{2}$ "
Silica	$\frac{1}{2}$ "
Iron wire	1 "

Salt cover.

Or:

Roasted ore	3 grains.
Soda	3 "
Borax	4 "
Litharge	$6\frac{1}{2}$ "
Flour	$\frac{1}{2}$ "
Iron wire	1 "

Salt cover.

(*e*) Galena:

	Ore	8 grains.	.600 gr
	Soda	4 " Y	.300
KNO_3	Nitre	6 "	.400

Salt cover.

(*f*) Copper, sulphides, etc.:

Roasted ore............	3 grains.
Soda....................	2½ "
Pot. carb................	1½ "
Borax....................	3½ "
Litharge..............	8 "
Flour....................	1 "
Iron wire.	
Salt cover.	

In general, ores with copper, nickel, tin, tellurium, require an increased amount of litharge: if containing no sulphur, Sb, As, or Zn, increase the amount of the reducing agent slightly also.

Roasted ores, free from sulphur, but which contain As, or Sb, can be assayed by (*d*), omitting the iron wire. See 110, (*b*).

In the case of galena, the action that takes place is different from that in the other charges. Nitre is a powerful oxidizing and desulphurizing agent. On its combustion it oxidizes the lead in the galena (lead sulphide). With the right proportion of nitre enough lead is left in the metallic state to collect all the silver in the ore. Too much nitre would oxidize all the lead, and there would be no metallic lead button.

Too little nitre and all the galena would not be desulphurized.

112. All test-lead and litharge *must* be tested for silver (see under Test-lead and Litharge in chapter on Reagents).

(*a*) For test-lead fuse in R. F. on coal 10 grains of test-lead (beside a little borax). When melted into one button, scorify and cupel. Measure the silver found, and always allow for it when using this particular lot of test-lead. If the silver button is too small to measure well, form two or three buttons of lead of 10 grains each, and scorify down, combine the "rich-leads," scorify again, and cupel.

(*b*) For Litharge, mix the following charge, and assay in clay crucible in blowpipe furnace:

Litharge	10	grains.
Soda	4	"
Argol	1½	"
Salt cover.		

Scorify, cupel, measure silver found, and allow for it, while this lot of litharge lasts, according to the amount found in the 10 grains of litharge. For instance, the 10 grains of litharge contain a certain amount of silver. Divide by 10. The result shows amount contained in one grain (call this "*a*"). If an assay is made with 6 grains of litharge employed, then 6 times "*a*" shows the amount of silver, to be deducted from the silver button obtained by the assay, before any calculation as to the amount of silver contained in the ore can be made.

119. TABLE IV.

SHOWING THE NUMBER OF OUNCES OF SILVER PER TON THE ORE CARRIES, WHEN THE SILVER BUTTON CORRESPONDS TO THE DIFFERENT NUMBERED CROSS-LINES ON THE IVORY SCALE, THREE GRAINS OF ORE BEING TAKEN FOR ASSAY.

No. of Cross-line.	Ounces of Silver per Ton.	No. of Cross-line.	Ounces of Silver per Ton.	No. of Cross-line.	Ounces of Silver per Ton.
50	522.1	33	150.	16	16.5
49	490.6	32	136.5	15	13.5
48	460.6	31	123.	14	10.5
47	433.5	30	112.5	13	9.
46	405.	29	100.5	12	6.
45	379.5	28	91.5	11	4.5
44	355.5	27	81.	10	4.
43	331.5	26	72.	9	3.
42	309.	25	64.5	8	2.1
41	286.5	24	57.	7	1.3
40	267.	23	49.5	6	.9
39	247.5	22	43.5	5	.45
38	228.	21	37.5	4	.25
37	211.5	20	33.	3	.1
36	193.5	19	28.5	2	.03
35	178.5	18	24.	1	.003
34	163.5	17	19.5		

The values of the larger buttons increase so rapidly, that most accuracy is obtained in measuring the smaller buttons. Whenever large enough, weigh the button.

114. GOLD ASSAY.—(*a*) The amount of gold generally present being so small in proportion to the amount of gangue, it is, as a rule, advisable to concentrate the ore before subjecting it to a blowpipe assay.

In most cases this is best done by means of water, by "washing." Gold is very heavy, and the lighter accompanying rock, by careful washing, can be largely removed, leaving a smaller mass to operate on. The washing skilfully done can be carried so far as to leave nearly nothing but the gold. But this should not be attempted for the purpose of assay, especially when the gold is in very fine dust and when there are many *heavy* impurities present. What is aimed at in concentration for the blowpipe assay is to reduce a mass of rock to a small proportion of its size, enabling us to operate safely and to find the amount of gold contained in a large quantity of rock, which we could not do without concentration unless a great number of assays were made.

(*b*) The best instrument for this purpose that the author knows of is the batea, a washing-pan of peculiar form, used in South America to wash gold. It is a very shallow dish of wood, with sides inclining to a point in the middle. To describe the manner of using it is difficult, but is easily learned by practice. A tub is partially filled with water. The batea is moistened and placed on the surface of the water. The ore, previously broken and powdered fine enough to pass through the No. 60 sieve, is placed in the batea and thoroughly wet. The wet mass is worked with the fingers to a mud-like consistency, care being taken that none of the fine powder, before it is wet, is floated off. The batea is inclined and a little water taken into it. The batea is now given a peculiar rotary motion, and from side to side. The water carries the lighter material around towards the edges, while the heavier bodies move towards the apex. The batea is again slightly dipped under the water, the rotary motion kept up, and the batea inclined, washing over more of the gangue. By continuing this a skilled hand can finally leave free from gangue the heavy material sought for in the apex or point of the batea.

(*c*) Of ordinary gold ores take three or four pounds; weigh amount taken, pulverize and pass through the No. 60 sieve (taking all due precautions that none of the particles or dust of the material is lost). Pour one fourth or less of the ore on the batea, and proceed as just described. When most of the gangue has been separated, and the amount of ore in batea is about one fourth of an ounce or less, hold the batea over a plate, incline it, and carefully wash off all the ore from the batea to the plate by gently pouring a stream of water from a cup on to the batea. Pour carefully the excess of water off the plate, being especially careful that no particles are carried away. Now proceed to wash another portion of the original mass in the batea; when reduced to small dimensions add it to what is on the plate, as before. Continue this until all the powdered ore has been washed and the residue is on the plate. The plate is now to be dried to expel the moisture. Place the plate on a stove and let it gently dry. When the contents are thoroughly dried, carefully brush the ore from the plate to a piece of paper; it should be weighed, then mixed and quartered as directed in sec. 102. The amount obtained by quartering is pulverized in the iron mortar, sifted through the 100 sieve, mixed, and requarted until the proper amount for assay has been taken.

(Observe the cautions and directions in par. 102 under sampling. All of the material from the plate that is not taken for assay should be carefully put away for further investigation if necessary.)

115. In case of *metallic scales* of gold which refuse to go through the sieve: these are carefully removed and weighed and set apart for assay (as they are assayed together); the pulverized material which has gone through the sieve is also weighed, and weight noted. It is then spread on paper and "sampled" to obtain the proper amount for assay. The method of calculation in this case is described in sec. 124.

116. The gold assay in the *coal* crucible is conducted in the same way as the similar silver assay.

117. *Blowpipe-furnace Crucible Assay for Gold.*—The same charges as given for silver are used for gold. (The amount of litharge may be often increased to advantage, however. Much lead causes a loss of silver in cupellation. This is not so with gold. In case the litharge is increased, increase also slightly the reducing agent used, unless the ore contains elements which themselves have a reducing effect, such as S, As, Sb, Zn.)

The method of conducting the fusion, scorification, and cupellation is the same as under the silver assay. Always make at *least* three assays of an ore for gold, concentrating the rich-leads together, and cupelling in one button.

118. *Inquartation.*—Gold contains silver in varying proportions generally. These must be separated. Silver dissolves in nitric acid, gold does not. If the silver is present in an alloy of gold and silver in greater proportion than about 2½ parts of silver to 1 of gold, all the silver is dissolved out on submitting the alloy to the action of nitric acid. If the silver is present in less proportion than 2½ times the gold, all the silver is *not* dissolved by nitric acid, but some is retained by the gold. If the silver is in too small proportion, enough has to be added to the button to cause all the silver to be acted on by the nitric acid. This adding silver is called Inquartation. By the color of the button the amount of silver necessary to add may be judged: 2 per cent of silver causes a light brass color; 60 per cent of silver destroys the gold color entirely.

The safest plan is to add from 2 to 2½ times the weight of the button in silver-foil. As the weight of an alloy of gold and silver cannot be determined by measurement on the ivory scale, it is necessary to get the weight of this button by the balance to get data from which to find the amount of silver in the button, and also to know how much silver-foil to add to the button. If the button is too small to weigh, make enough assays to obtain a weighable button. Fuse the button and the silver-foil together in a small cavity on coal with the R. F. (besides a small amount of borax). The fused alloy is removed from the coal, gently flattened on the anvil (the button is wrapped in paper to guard against loss on the anvil), and then *gently* heated in the O. F. (not more than a dull red).

119. *Parting.*—(*a*) The button is placed in one of the small porcelain crucibles, which should be wiped out, and carefully examined with the lens to see that it contains no black specks or dust, etc. The crucible is placed on the triangle on the tripod, and a few drops of water (pure rain-water or distilled water) poured in, and then five or six drops of strong nitric acid. Add acid drop by drop until the button is acted on (indicated by the bubbles, etc.). Place the alcohol lamp under the triangle, light the lamp, and warm gently the crucible for a few minutes. When the button ceases to give off bubbles and the silver is dissolved, remove the porcelain crucible and examine through the magnifying-glass. The gold will be left as *black* or dark-colored *specks.* Pour very carefully the liquid out of the crucible, taking great care that none of the black specks (the gold) is lost. Pour into a white dish, so if any of the black specks are carried over they will be seen and can be recovered. (Lean a glass tube against the crucible, and pour against it so the water may run *gently* down the side of the tube.) Now fill the crucible with more water, a fourth

full or less, and place it again on the triangle and boil gently. Pour off this water, taking all precautions against the loss of the black specks. Put more water in the crucible and boil again. (It is necessary to wash away thoroughly any nitrate of silver that may be left. It may be necessary to repeat again this boiling.) The water should be carefully poured out of the crucible, which is placed on the triangle, gently heated at first to expel moisture, then more strongly heated for a few moments, and the black-colored specks will assume their true gold color. When cold, very carefully detach them with the point of a knife. If enough gold is present it may be weighed at once; but with the blowpipe assay generally this is not the case. Brush the gold with great care into the mixing capsule; put in it a very little test-lead and borax. Make a soda-paper cylinder of half the usual size or less. Place contents of mixing capsule in the soda-paper cylinder, which is put in a prepared *coal* crucible, and treated with R. F. as in sec. 104. The lead button is then *cupelled*, and the gold button measured on the ivory scale.

(*b*) The difference between the weight of the gold button and the weight of the button *before* silver was added for inquartation gives the weight of silver in the button, and from this the amount of silver the ore carries is found (weight of silver in milligrammes multiplied by 150.033 = ounces of silver per ton ore carries, if 3 grains were taken for assay). The amount of gold in the ore is found after measuring the gold button by Table VI, if 3 grains were taken for assay. If not, Table V gives the weight in milligrammes of a gold button, corresponding to the different cross-lines on scale; and multiply this weight by the quotient obtained by dividing 450.09992 by the weight in grains of the amount assayed. The result shows number of ounces of gold the ore carries to the ton. If the button was weighed on the balance, and weight obtained in grains, then weight of amount taken for assay : weight of gold button :: 29166.66 : to number of ounces of gold the ore carries to the ton.

The foregoing calculation answers when the ore was not concentrated. When concentrated (as it should be for the gold assay) let weight of whole amount of ore taken for concentration (in Troy grains) = A, weight of residue left after washing = B, weight of gold button (in milligrammes) from assay of 3 grains = C,

$$\frac{A}{450.09992} = D. \text{ Then } \frac{\frac{B}{3} \times C}{D} = \text{ounces of gold ore carries to ton.}$$

120 ~~126.~~

TABLE V.

WEIGHT OF GOLD BUTTON IN MILLIGRAMMES CORRESPONDING TO THE CROSS-LINES ON IVORY SCALE.

No. of Cross-line.	Weight Gold Button.	No. of Cross-line.	Weight Gold Button.	No. of Cross-line.	Weight Gold Button.
25	.946	16	.248	7	.021
24	.838	15	.205	6	.013
23	.737	14	.166	5	.008
22	.645	13	.133	4	.0039
21	.561	12	.105	3	.0016
20	.485	11	.081	2	.0005
19	.416	10	.061	1	.00006
18	.353	9	.044		
17	.298	8	.031		

~~121.~~ 121

TABLE VI.

SHOWING NUMBER OF OUNCES OF GOLD PER TON THE ORE CARRIES WHEN THE GOLD BUTTON CORRESPONDS TO THE FOLLOWING CROSS-LINES ON THE IVORY SCALE, WHEN THREE GRAINS ARE TAKEN FOR ASSAY.

No. of Cross-line.	Ounces of Gold per Ton.	No. of Cross-line.	Ounces of Gold per Ton.	No. of Cross-line.	Ounces of Gold per Ton.
25	141.93	16	37.2	7	3.15
24	125.72	15	30.75	6	1.95
23	110.57	14	24.9	5	1.2
22	96.77	13	19.95	4	.58
21	84.16	12	15.75	3	.24
20	72.76	11	12.15	2	.07
19	62.41	10	9.15	1	.009
18	52.96	9	6.6		
17	44.7	8	4.65		

122. Use the greatest care in removing the gold from the porcelain crucible. It sometimes sticks to the crucible in thin sheets or flakes,—often broken up if too strong acid has been used.

Instead of alloying it with lead by treatment with R. F. on coal, as described above, it is safest, and often best, to carefully push the scales or flakes of gold from the porcelain crucible with a knife-point directly into a clay crucible, setting in the furnace. Then place in the crucible a *little* test-lead, or a little borax (just a few particles), and heat the furnace. When cold, break crucible, take out button, and cupel directly.

123. When the accompanying gangue (or impurities) is heavy and the gold in very fine dust

(as in the case of the "black sand" consisting of different iron oxides, magnetite, ilmenite, etc.), it is not safe to concentrate by washing. A ***number*** of assays must be made, and the resulting "rich-leads" fused together into one button, rescorified, and cupelled.

After parting, the gold obtained in an assay is reported as such. It ***may contain*** in some rare cases ***slight*** traces of rhodium, iridium, or other members of the platinum group. But, ***practically***, the amount of gold contained in the ore is calculated from the weight of gold obtained in an assay after the gold has been freed from silver by parting. Wet methods are employed in separating gold from metals of the platinum group. Gold containing platinum gives a dull-looking button, of a color varying (with the amount of platinum) from a whitish yellow to gray. In the latter case the button has a rough or somewhat pitted appearance.

In the case of bullion containing gold, silver, and copper, the more copper present the more lead is needed to effect cupellation. The quickest way is to treat the finely divided alloy with borax and ten to twelve times its weight in lead in a coal crucible with the R. F., as in the silver assay in coal crucible. Scorify button and cupel, if too much copper were present and not enough lead added originally, proceed as in the section on Cupelling, adding more lead, and then cupelling. The button from the cupellation is parted and amount of gold and silver determined.

124. *Metallic Scales.* — When metallic scales

refuse to go through the sieve, they are assayed by themselves in a coal crucible with borax and test-lead (see Silver Assay, coal-crucible method), and the button scorified, cupelled, and parted, and weight of gold and silver noted. The weight of the siftings, or the part of the ore (in which were the scales) which went through the sieve, is taken. These siftings are sampled, 3 grains taken for assay by the blowpipe-furnace method, the resulting button scorified, cupelled, and parted, and weights of gold and silver noted. The weight of gold found in the metallic scales, plus the weight of gold in the siftings (found by multiplying the weight of gold button, obtained by the number of times 3 grains is contained in the weight of the siftings), multiplied by 29166.66, and the product divided by the weight of the siftings plus weight of the gold scales = number of ounces of gold per ton the ore carries. Proceed in similar way to calculate the amount of silver.

125. LEAD ASSAY.—(*a*) There is always a loss of lead in the fire assay, as it is so volatile; but the loss also occurs in treating lead in the smelter. The lead button is also liable to contain certain other metals that were present in the ore. [illegible] lead

(*b*) *Charges for Lead Assay.*

Oxides, Carbonates:

Ore	3 grains.	200
Soda	3 "	
Pot. carb	2 "	
Borax	2 "	
Flour	$1\frac{3}{10}$ "	

Salt cover.

Sulphides and Ores containing Sb, As, etc.:

With Zinc.

Ore	3	grains.
Soda	4½	"
Pot. carb	2½	"
Borax	1	"
Flour	½	"
Iron wire	1	"
Salt cover.		

With Sulphur, etc.:

No Zinc.

✓ Ore	3	grains.	.200
✓ Soda	3	"	
✓ Pot. carb	2	"	
✓ Borax	1	"	.1 .05
✓ Flour	1	"	.100
Iron wire	1	"	1.59
Salt cover.			

Mix well, brush carefully into clay crucible, and follow directions in sec. 108 for the blowpipe-furnace assay.

When cold, break crucible. The lead should be in one button; the button should be soft and malleable (Sb or As present makes it hard or brittle. In the case of galena, there should be no shining particles of undecomposed galena; if there are, indicating too little soda and pot. carb., or too little iron, of course the assay is spoiled). Clean the button and weigh.

The result of an assay of the base metals is reported in per cent. Twenty pounds is one per cent of a ton (2000 lbs.).

If the lead button obtained from 3 grains of ore weighed ½ grain (.5 of 1 grain), then $\frac{.5}{3} \times 100 = 16.6$ per cent. The ore carries 16.6 per cent of lead.

126. COPPER ASSAY.—Ores containing sulphur, etc., must be well roasted. (See *Roasting*.)

(*a*) *Coal-crucible Assay*.—Mix 1½ grains of the pulverized ore (roasted, if a sulphide, or containing As or Se) with 2 grains soda, ½ grain borax, ½ grain test-lead, in the mixing capsule. Prepare a coal crucible and soda-paper cylinder, as in the silver assay. Pour mixed ore and fluxes into the soda-paper cylinder, and proceed as under the silver assay. When the "fusion" is finished, separate the button from the slag by gentle hammering on the anvil. Prepare another coal crucible, and in its cavity place the button and about its weight of fused boracic acid. Melt the button with the R. F., and when melted, change to the O. F., blowing on the *slag* with the inner flame not too strongly. The boracic acid absorbs the lead oxide. Keep button from rolling under slag by inclining clay cylinder and by the force of the flame. As soon as the button becomes of a bluish green color (melted copper) it shows that the operation is finished. When cold, examine slag carefully. It must not be red; if it is, it shows some copper has oxidized and been taken up by the slag. If it is, crush slag carefully, separate button from slag, add a little soda and test-lead to slag, and treat with R. F. on coal, to reduce the copper oxide and collect the copper. After united in one button, detach it from slag and fuse beside boracic acid to separate the lead. Now add the copper button obtained to main button, and weigh both.

(But it is better to make a fresh assay if the slag has taken up copper oxide.)

The copper button should be pure copper-colored, and should be malleable. If it is brittle and cracks, lead is still present, and it will have to be treated again beside boracic acid; but copper is very liable now to be lost in slag. Other base metals in the copper button are mostly gotten rid of by their passing into the boracic acid or volatilization.

If a crust of nickel forms on the button, detach button fuse, beside test-lead, and repeat the treatment beside boracic acid.

127. (*a*) *Blowpipe-furnace Assay.*—No dry way of assaying copper is perfectly accurate, as there is a loss by oxidization, and copper may contain other metals in the ore. But the dry assay is of great commercial importance, as the buying and selling of copper ore at the principal copper markets of the world are generally based on the results of the dry assay. Besides this, the fire assay indicates in what manner the ore will behave when treated at the smelter. [It is well to remember that *assaying* is for the purpose of

finding the amount of metal an ore carries for *practical* and *commercial* purposes. It should with great accuracy show this; but a chemical analysis which shows *all* of an element that is in a body may be misleading. It may show an amount that cannot be extracted from the ore by practical means. Hence it may be of no use.]

(*b*) *Charges.*—Ores containing sulphur, As, Sb, Zn must be roasted. (See *Roasting.*)

No. 1. Sulphides, etc.: 0

		mg
Ore (roasted)	3 grains.	.200
Soda	4 "	.100
Pot. carb.	2 "	.100
Borax	$1\frac{2}{10}$ "	.100
Flour	$1\frac{2}{10}$ "	.125
~~Litharge~~	$1\frac{1}{2}$ "	~~.125~~

A layer of flour; then
~~Salt~~ cover.

No. 2. Oxides, Carbonates, etc.:

Ore	3 grains.
Soda	4 "
Pot. carb.	2 "
Borax	$1\frac{2}{10}$ "
Test-lead	1 "
Starch	$1\frac{1}{2}$ "

Then a layer of starch.
Salt cover.

The charges are mixed, put in crucible, and then a thin layer of starch or flour placed on top, and on that the salt cover.

For ores containing native copper and gangue alone, use method No. 2, omitting the starch and varying the other ingredients to obtain a fluid flux.

The slag must not be red, which indicates that copper has been retained in the slag. In this case increase the amount of the reducing agent. (A green slag may also indicate loss of copper; but, on the other hand, the green color is often given to the slag by iron in the ore in certain amounts) The copper must now be freed from lead by treatment beside boracic acid, as in sec. 126. The button should be copper-colored and malleable. Clean button and weigh.

128. As stated in sec. 126, most base metals in the copper are eliminated by the boracic-acid treatment. Tin is not. Tin has a great affinity for silica, and may be removed from an alloy of tin and copper by taking advantage of this. Make a flux of 10 parts soda, 5 borax, 3 silica. Fuse the flux to a bead, and place this bead with the copper-tin button in a cavity on coal and melt with R. F.; then blow on the slag with O. F. The button should touch the slag on one side and the hot coal on the other. The slag "dissolves" the tin; small buttons of tin are seen around the edges of the slag. When the slag is saturated, detach the button, and fuse beside a new bead of the flux, until the button shows the bluish green of melted copper.

If the slag should be red to reddish brown, containing oxidized copper, treat slag with strong R. F. to reduce copper oxide, and add buttons of copper obtained to main button.

129. TIN ASSAY.—*Blowpipe-furnace Assay.*—Tin has such a great affinity for silica, that mixed with silica in certain proportions in an assay (when certain fluxes are used) no tin is obtained at all. It has all gone into the slag as a silicate. Quartz is silica. Metallic iron used in an assay frees the tin, as the iron itself combines with the silica, metallic tin and iron silicate being the result. But if too much iron is used, the excess alloys with the tin, and an impure button of tin and iron is obtained. If we knew the exact amount of tin and quartz in the ore, the right amount of iron could be added; but this we do not know: and of the many ways of assaying tin the author finds for blowpipe use the method with cyanide of potassium the best. This substance is very poisonous, and care must be taken in handling it.

(Sulphides, etc., should be roasted.)

Free from Silica:

Ore	3	grains.
Cyanide potassium	6	"
Soda	1	"
Starch	½	"
Salt cover.		

For ores with much silica I have obtained fair results with the following charge:

Ore	3 grains.
Cyanide potassium	9 "
Soda	3½ "
Starch	2½ "
Salt cover.	

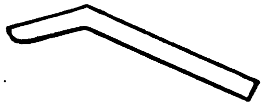
From a mixture of 40 per cent cassiterite (tin oxide) and 60 per cent silica, and using this charge, 73 per cent of the tin was obtained. This compares favorably with the regular furnace assays, that on impure tin ores get results as low as 60 per cent. Moreover, when the ore is washed properly no such amount of silica will be left.

Place a small piece of the cyanide of potassium at the bottom of the crucible, and pour in the mixed charge. Heat gently at first, break crucible when cold, and examine slag carefully for any small buttons separated from main button. If any are found, recover them by crushing the slag and washing. They are then added to the main button of tin and weighed. (With the larger quantity of cyanide of potassium the crucible will be filled; tamp with care, and no bad results will arise.) The tin button should be soft, tin-white, and malleable, but is liable to be alloyed with any iron and certain other impurities that are in the ore. In this case the perfect separation of the tin from the impurities alloyed with it has to be done by wet methods.

It is best to free the ore from silica as much as possible by using the batea. (See the Gold Assay.) Tin ore, the *oxide*, cassiterite, is very heavy, and with care most of the quartz can be washed away. Observing all the precautions as to weighing, drying, sampling, etc., as under the use of batea in gold assay, the proper amount is obtained finally for assay. Weight of whole amount washed $= A$; weight of the amount concentrated (the residue left after washing) $= B$; weight of button obtained by assaying 3 grains $= C$.

$$\frac{\frac{B}{3} \times C}{A} \times 100 = \text{per cent of tin the ore carries.}$$

130. MERCURY ASSAY.—The following, known as Domeyko's method, from its originator, is simple, and when properly executed the amount of mercury in the ore can be closely estimated. Mercury is so volatile that it has to be confined to some vessel; to obtain it a glass tube 6 inches long by about $\frac{1}{8}$ inch in diameter is used. The tube is closed at one end. The shorter arm of the tube is about $1\frac{1}{4}$ inch long. The tube is bent in the following shape:



.5 gm .8

For Sulphides.—Mix 7 grains of the ore with 10 grains litharge; introduce carefully (by means of a narrow slip of paper, folded lengthwise, and in which the mixture is placed) to the bottom of the tube. The closed end of the tube containing the charge is now heated in the alcohol-lamp flame (holding tube in matrass-holder) until the mixture is fused. The mercury forms a sublimate near the bend of the tube. When cold, cut tube with a file, brush out the mercury into mixing capsule, transfer to balance, and weigh.

For Chlorides.—Mix with the above charge soda three times the bulk of the ore, and proceed as above.

(Idrialite, a mixture of cinnabar [mercury sulphide], clay, gypsum, pyrites, and bituminous matters, gives off volatile substances which contaminate the mercury.)

131. *Nickel and Cobalt Assay* (generally found together).—When a combination of Fe, Co, Ni, and As is fused beside borax on coal in O. F., the Fe first oxidizes and passes into the borax, then the Co, leaving the Ni as an arsenide, if the operation has been carefully performed. (Read Cobalt and Nickel in Chapter VI.)

Now the elements combine with each other, not in a haphazard way, but according to fixed laws, and they are present in certain proportions expressed by their atomic weights, so that in any given chemical combination the relative proportion of each element can be ascertained. As in assaying nickel and cobalt cannot be obtained by direct fusion, they are fused with iron and with arsenic, forming arsenides of Fe, Co, and Ni. When this compound is treated as above, and the Fe and Co slagged off, a definite compound of Ni and arsenic is left, expressed by the formula Ni_2As (which means that the compound contains the two metals in the proportion of two atoms of nickel to one atom of arsenic). From the table of "Atomic Weights" the atomic weight or combining weight of nickel is 58.8, arsenic 75; but there are *two* atoms of nickel to one atom of arsenic: $58.8 \times 2 = 117.6$. 117.6 parts of nickel and 75 parts of arsenic are found in the button left. $117.6 + 75 = 192.6$. $\frac{117.6}{192.6}$ of the weight of the button is nickel, $\frac{75}{192.6}$ of it arsenic.

(*a*) Plattner's method, modified, is the one virtually given here. Take $\overset{10}{1\frac{1}{2}}$ grains ore. If sulphur is present, roast thoroughly with coal-dust. If no sulphur is present, omit the roasting. Mix the ore with $\overset{15}{1\frac{1}{2}}$ grains of metallic arsenic and $\overset{.02}{\frac{1}{4}}$ grain iron filings. Place in the clay crucible in the blowpipe furnace, and fuse at a gentle heat to a button. (The arsenical fumes are poisonous; this part of the operation had better be done out of doors if possible.) When cold, break crucible, pulverize the button of arsenides. Mix with $\overset{150}{2}$ grs. soda, $\overset{150}{2}$ grs. carb. pot., $\overset{50}{\frac{1}{2}}$ grain borax, and $\overset{50}{\frac{1}{2}}$ grain flour. Place in clay crucible, in furnace. Cover charge with salt cover, and proceed as in par. 108. (The charge may have to be varied for different gangues, etc. See sec. 110.)

When cold, break crucible, separate button from slag, and carefully clean the button.

Treat the now clean button beside borax in a cavity on coal, melting the button with the R. F. until the button assumes a rotary motion; then keep the O. F. on the borax bead. The iron arsenide oxidizes and forms on the surface of the button, and is dissolved in the borax. When the button shows a bright surface, and is no longer covered with a film, stop, and remove button. A piece of the borax bead treated on platinum wire should only show Fe colors. The cold button is covered with a black oxide. Treat the button beside fresh borax to slag off all the Fe until it is again bright, and gives off arsenical fumes. The clean button is fused with gentle R. F. on coal, and if it remains bright and gives no more arsenical fumes, the Fe is gone. Weigh the button. The button is now treated beside borax as before, to slag off the Co. The cobalt arsenide oxidizes, and is dissolved as cobalt arsenate in the borax. The borax becomes blue from cobalt. When all the cobalt is gone, and the arsenide of nickel begins to oxidize, a film of nickel arsenate forms on the button, and begins to dissolve in the borax. Stop the operation, and draw out with the forceps a thread of the hot borax glass. Examine it against the daylight. It should be rather violet than blue. If brown, the nickel has commenced to dissolve in the borax. The button should now be examined; if covered with a film of apple-green nickel arsenate, it shows the cobalt has been slagged off and the operation finished. The button containing cobalt and nickel may have to be treated beside fresh borax to slag off properly all the cobalt.

The button is cleaned and weighed; 60.7 per cent of it is nickel. The weight of this button, taken from weight of the nickel and cobalt button after iron was slagged off, gives the weight of the cobalt arsenide, 61.1 per cent of which is cobalt.

From these weights (remembering the amount of ore taken for assay) the percentage of Ni and of Co in the ore can be found. Thus if the button of nickel arsenide finally obtained weighed .4 of a grain, 60.7 per cent of this is nickel, or .24 grains nickel. $\frac{\frac{24}{100}}{1\frac{1}{2}} \times 100 =$ per cent of nickel in ore.

(*b*) When no cobalt is present proceed as above, remembering that when the Fe is slagged off a button of nickel arsenide is left.

When arsenic is contained in the ore, omit the addition of arsenic and proceed as above.

When copper is also contained in the ore, especially in any quantity, the wet way must be employed, for as far as the blowpipe assay is concerned the dry way does not then answer.

132. BISMUTH ASSAY.—*Blowpipe-furnace Assay.*—

Ore	3 grains.
Soda	3 "
Carb. pot	2 "
Borax	4 "
Flour	1 "
Salt cover.	

When ores contain sulphur, As, Sb, roast with care. (Bismuth is volatile.) If the metal is not in one button (and it often is not, in the bismuth assay), break slag, wash, and recover the buttons, which are weighed together. Bismuth is brittle; care must be taken in crushing the slag. The bismuth button will probably be alloyed with other metals in the ore. The bismuth button pure is white, with a red tinge, best seen on breaking it.

APPENDIX.

EXAMINATION OF A MINERAL FOR THE METALS OF COMMERCIAL IMPORTANCE.

SOME years ago an excellent scheme for the detection of many bodies in a complex substance, by means of the blowpipe, was published by Prof. Egleston (Cornwall, page 150).

However, for determining the presence of metals of commercial value in a mineral the following mode of procedure will be found convenient: [Refer back to Chapter VI, under the head of each, for additional information.]

1. Pulverize eight or ten grains of the substance.

2. (*a*) Treat half of it on coal (not all at once) in the O. F. and R. F.; place the roasted substance aside. Note odors produced.

(*b*) Test any coat that may form with tip of inner flame to obtain the bluish green flame of Sb.

(*c*) If yellow Pb or Bi coats are present, take a portion left from 2 (*a*), make a soda reduction (on another coal) with R. F., cut out soda, wash slag, and any button found place beside boracic acid on coal, and treat with R. F. to form a coat of Sb, which test by flame-coloration.

3. (*d*) Mix a fresh portion from 2 (*a*) with soda, and treat with R. F. on coal for coats.

(*e*) Sulphur-yellow coat (cold), coloring flame blue, Pb; lemon-yellow coat, no color to flame, Bi.

(*f*) [Test fresh portion with iodide of potassium and sulphur on coal (see Bi, Chapter VI) to determine Bi and Pb in case of doubt.]

(*g*) Test coat from (*e*) with nitrate-of-cobalt solution in O. F. for the fine grass-green of Zn coat, or bluish green of Sn coat.

(*h*) Make soda reduction on coal with R. F. (mix charcoal-dust with soda) for Sn in case of doubt.

(*i*) If no yellow coat was found by 3 (*d*), take coal from 2 (*a*), scrape off coat, dissolve in SPh bead in O. F., and treat beside tin on coal with R. F. for dark gray color imparted to bead by Sb (see table, SPh beads with tin).

4. (*j*) Mix about an ivory spoonful of roasted substance from 2 (*a*) with about two thirds as much borax, one (Troy) grain or so of test-lead; place in soda-paper cylinder in prepared coal crucible, and treat with R. F. (as in silver assay in coal crucible, which see).

(*k*) Detach lead button when cold, saving the borax bead for (*o*).

(*l*) Treat the lead button beside boracic acid on coal (as in copper assay, which see) to get rid of excess of lead.

(*m*) Place the now small button in a shallow cavity on coal beside a previously formed SPh bead, and treat button with O. F. Examine the saturated button when cold. Yellow Ni, blue Cu, green Ni and Cu.

(*n*) Place the SPh bead from (*m*) (detaching from it any button that may be left) beside a piece of tin, and treat with R. F. The bead red when cold, Cu.

(*o*) Treat small pieces of the borax-glass from (*k*) on platinum wire in O. F. and R. F., noting the colors of Fe, Mn, or Co. If the glass is too dark, add fresh borax. (See under heads for mixed colors.)

5. (*p*) Make a preliminary assay (in coal crucible) of original substance for Ag and Au. [If any traces are found, take more of the substance and make a blowpipe-furnace assay.]

6. (*q*) [Treat substance from 2 (*a*) in SPh bead on wire in O. F. and R. F. If there is *enough* of Cr, U, W, or Ti to be of *importance* in themselves, the following colors will be obtained: Green in O. F. cold and in R. F. cold, Cr; greenish yellow in O. F. cold, green in R. F. cold, U. In SPh bead with tin on coal in R. F. cold, violet Ti, blue W. [Some wolframites green. In SPh bead on wire in R. F. brownish red cold.]]

(*r*) [Treat substance from 2 (*a*) in borax bead in R. F. cold, brown to opaque, nearly black, Mo (in O. F. hot yellowish).]

7. Mix original substance with three times its bulk of *dry* soda and fuse in closed tube for Hg.

8. The presence of S, As, Se, or Te should also be determined, as the future treatment of the assay may depend on their presence or absence. The odors noted, 2 (*a*), will show Se and perhaps S and As.

Make test in open tube with blue litmus-paper for S; test in closed tube for As; test in closed tube with soda, and adding hot water, for Te. (See Chapter VI.)

9. For Pt the original substance may be examined by pulverizing, washing in batea, and testing any metallic scales for their specific gravity, fusibility, hardness (see Appendix), and malleability. Also, test beside borax on coal in O. F. (See Platinum, Chapter VI.)

TROY WEIGHT.

24 grains = 1 pennyweight (dwt.).
20 dwts. = 1 ounce.
12 ounces = 1 lb.

AVOIRDUPOIS WEIGHT.

16 drams = 1 ounce.
16 ounces = 1 pound.
25 pounds = 1 quarter.
4 quarters = 1 hundredweight (cwt.).
20 cwts. = 1 ton.

There are 29,166.66 Troy ounces in a ton of 2000 lbs.

There are 14,000,000 Troy grains in a ton of 2000 lbs.

1 Troy grain = .064798 grammes.
1 Troy grain = 64.798 milligrammes.
1 gramme = 15.43235 Troy grains.
1 milligramme = .015432 Troy grains.

In 450.09992 Troy grains there are 29,166.66 milligrammes.

The hardness of a mineral is determined by comparing it with the following scale of hardness: (*This means hardness in relation to scratching, not hardness as to breaking.*)

1. Talc.
2. Gypsum.
3. Calcite.
4. Fluorite.
5. Apatite.
6. Orthoclase.
7. Quartz.
8. Topaz.
9. Sapphire.
10. Diamond.

The following is often more available:

Scratched by the finger-nail	1
Scratched with difficulty by the nail. Does not scratch a copper coin.	2
Scratches and is scratched by a copper coin	3
Not scratched by copper coin. Does not scratch glass.	4
Scratches glass with difficulty, leaving its powder on the glass. Yields readily to the knife.	5
Yields with difficulty to the knife	6
Does not yield to the knife	7

(Not scratched by flint; harder than 7.)

The specific gravity of a substance may be obtained approximately by the following method, provided (1) that the mineral is pure; not mixed with any other body which might be heavier or lighter; (2) that great care is taken, and the operation skilfully performed:

TABLE OF ELEMENTS,

WITH THEIR SYMBOLS AND ATOMIC WEIGHTS.

Name.	Symbol.	Atomic Weight.	Name.	Symbol.	Atomic Weight
Aluminium,	Al	27.4	Mercury,	Hg	200
Antimony,	Sb	122	Molybdenum,	Mo	96
Arsenic,	As	75	Nickel,	Ni	58.8
Barium,	Ba	137.2	Nitrogen,	N	14
Bismuth,	Bi	210	Osmium,	Os	199.1
Boron,	B	11	Oxygen,	O	16
Bromine,	Br	80	Palladium,	Pd	106.6
Cadmium,	Cd	112	Phosphorus,	P	31
Cæsium,	Cs	133	Platinum,	Pt	197.4
Calcium,	Ca	40	Potassium,	K	39.1
Cerium,	Ce	92.2	Rhodium,	Rh	104.2
Carbon,	C	12	Rubidium,	Rb	85.4
Chlorine,	Cl	35.5	Ruthenium,	Ru	104.4
Chromium,	Cr	52.1	Selenium,	Se	79.5
Columbium,	Cb	94	Silicon,	Si	28.1
Cobalt,	Co	58.7	Silver,	Ag	108
Copper,	Cu	63.1	Sodium,	Na	23
Didymium,	Di	95	Strontium,	Sr	87.5
Erbium,	E	112.6	Sulphur,	S	32
Fluorine,	F	19	Tantalum,	Ta	182.3
Glucinum,	Be	9.3	Tellurium,	Te	128
Gold,	Au	196.8	Thallium,	Tl	203.5
Hydrogen,	H	1	Thorium,	Th	115.7
Iodine,	I	127	Tin,	Sn	118
Indium,	In	113.4	Titanium,	Ti	50
Iridium,	Ir	196.9	Tungsten,	W	184
Iron,	Fe	56	Uranium,	U	237.6
Lanthanium,	La	92.9	Vanadium,	V	51.3
Lead,	Pb	207	Yttrium,	Y	61.7
Lithium,	Li	7	Zinc,	Zn	65
Magnesium,	Mg	24	Zirconium,	Zr	89.6
Manganese,	Mn	55			

Weigh the mineral; note weight (weight A).

Now tie the mineral to the balance-pan by a hair, and so arrange the balance that the mineral will remain suspended in a glass of water, not touching the sides or bottom of the glass. Weigh the mineral so placed (weight B). Then $\frac{A}{A-B}$ = specific gravity.

In testing for *fusibility* use a strong O. F., holding fragment in platinum-pointed forceps. In case of metals that might injure the platinum, test on coal in a shallow cavity. Examine with lens to detect signs of fusion.

OUTFIT.

BELOW will be found the articles required to perform the various operations treated of in this book. The prices are those charged by the Henry Heil Chemical Co., St. Louis, Mo., with the exception of the articles from John Taylor & Co., San Francisco, Cal.

Black's blowpipe	$0.23
Tallow lamp	.30
Alcohol lamp (6830)	.68
1 doz. pieces charcoal	.36
3 platinum wires	.23
½-pint iron mortar	.27
Wedgwood mortar (3 in.)	.27
Anvil	.38
Blowpipe hammer	.45
½-lb. hammer	1.05
6 doz. clay crucibles	.90
2 doz. coal crucibles	.30
Clay cylinder ("holder")	.21
Coal-borer	.33
3 doz. clay capsules	.45
Fletcher blowpipe furnace with *side*-hole	.24
Tin box-sieve, No. 100, John Taylor & Co.	.40
Sieve, No. 60	.60
Mixing capsule	.12
Test-lead measure	.36
Forceps, 5415	.15
5401	.18
5416	.18
Platinum-pointed forceps	1.50
3 doz. cupels, 1¼ in.	.90
Porcelain crucibles, 4670	.15
1 doz. open tubes	.24
1 doz. matrasses	.30
Lens (double)	.75
Magnet	.08
2 camel's-hair brushes	.06
Brush, 3597	.15
Matrass-holder	.23
Pocket assay balance, No. 2584, including weights	15.30
(Or, Whittel's balance, John Taylor & Co., including weights, $20.00.)	
Best hand scale, 2½-in. pan	1.36
Dwt. weights	$0.26
Ivory button scale, John Taylor & Co	3.00
Sampler	.45
Horn spoon, spatula end	.18
Batea, 10 in., John Taylor & Co	1.00
Tripod	.18
Triangle	.06
Fletcher's patent blowpipe, No. 42	1.35
Soda-paper form	.09
Ivory spoon	.18
Borax, fused, 1 oz	1.25
SPh, ½ oz	
Soda, 1 oz	
Potassium carbonate, ½ oz	
Nitre, ½ oz	
Tin, ¼ oz	
Boracic acid, fused, ½ oz	
Nitrate of cobalt, ¼ oz	
Silica, ½ oz	
Argol, 1 oz	
Metallic arsenic, ¼ oz	
Potassium cyanide, ¼ oz	
HCl acid, ¼ oz	
Nitric acid, ½ oz	
Test-lead, 1 oz., John Taylor & Co	.20
Litharge (C. P.), 1 oz., John Taylor & Co	.10
Cu oxide	.90
Nickel oxalate	
Blue litmus-paper	
Brazil-wood paper	
Soda-paper	
Silver (foil), ¼ oz	
Potassium iodide, ¼ oz	
Sulphur, ¼ oz	
Total	$38.86

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20 div	1/.0[illegible]	12,9[illegible]
50 "	.002[illegible]	7,29[illegible]
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72[illegible]