LABORATORY NOTES
ON
PRACTICAL METALLURGY

WALTER MACFARLANE, F.I.C.

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PRACTICAL METALLURGY

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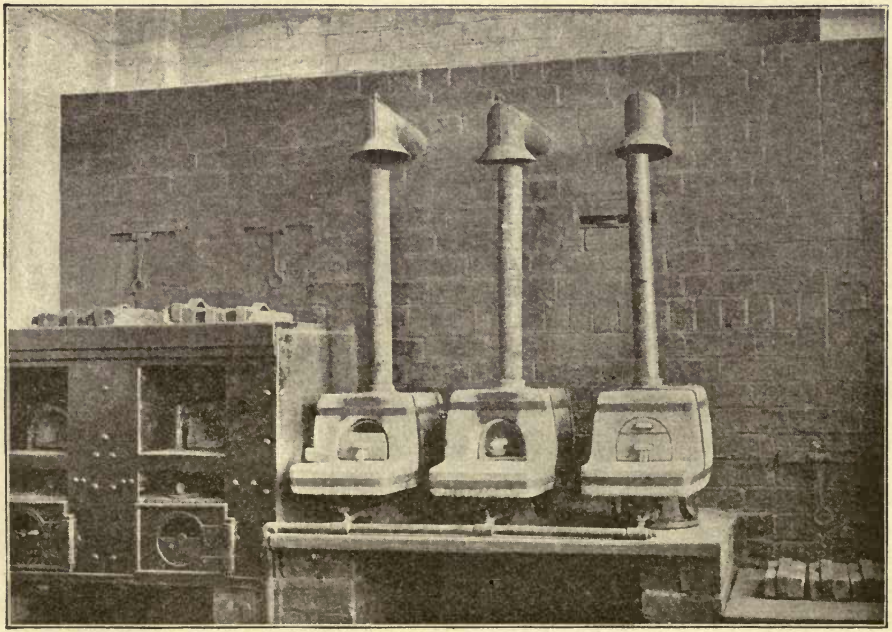

COKE AND GAS MUFFLE FURNACES.

LABORATORY NOTES

ON

PRACTICAL METALLURGY

BEING A GRADUATED SERIES OF EXERCISES

ARRANGED BY

WALTER MACFARLANE, F.I.C.

PRINCIPAL OF THE METALLURGICAL DEPARTMENT, STAFFORDSHIRE EDUCATION COMMITTEE

FORMERLY BURSAR IN TECHNICAL CHEMISTRY, ANDERSON'S COLLEGE, AND ASSESSOR IN METALLURGY, GLASGOW AND WEST OF SCOTLAND TECHNICAL COLLEGE

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PREFACE

THIS book is published in compliance with requests from several teachers and others interested in the subject.

The exercises have been helpful in clearly demonstrating metallurgical principles and firmly impressing them on the minds of students. The lessons are graduated and arranged with the view of inculcating the habit of observing results and recording the work in progress.

Thanks are due and are tendered to Professor A. Humboldt Sexton for valuable suggestions, and to Professor William Gowland for kind permission to include the Table on pages 129 and 130.

THE MUNICIPAL SCIENCE SCHOOL,
WEDNESBURY,
November, 1904.

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APPENDIX

The apparatus and materials required for the Exercises in this book can be obtained of Messrs. PHILIP HARRIS AND CO., LTD., 144 and 146, Edmund Street, Birmingham ; and 179, Great Brunswick Street, Dublin.

LABORATORY NOTES

ON

PRACTICAL METALLURGY

EXERCISE NO. 1.

Preliminary Exercise in weighing.

Weigh, one by one, the "A" set of metal triangles (which are stamped 1 to 20), noting the weight of each one as ascertained.

Report the results before proceeding with the following exercise.

All work done and results obtained should be carefully entered by the student in his laboratory note-book. The entry should be shown to the teacher before proceeding with the next exercise.

EXERCISE NO. 2.

Weighing continued.

Weigh, one by one, the "B" set of metal squares (which are stamped 21 to 40), noting the weight of each one as ascertained.

Report.

EXERCISES IN A MUFFLE FURNACE.

A muffle furnace may be heated with coal, coke, or gas.

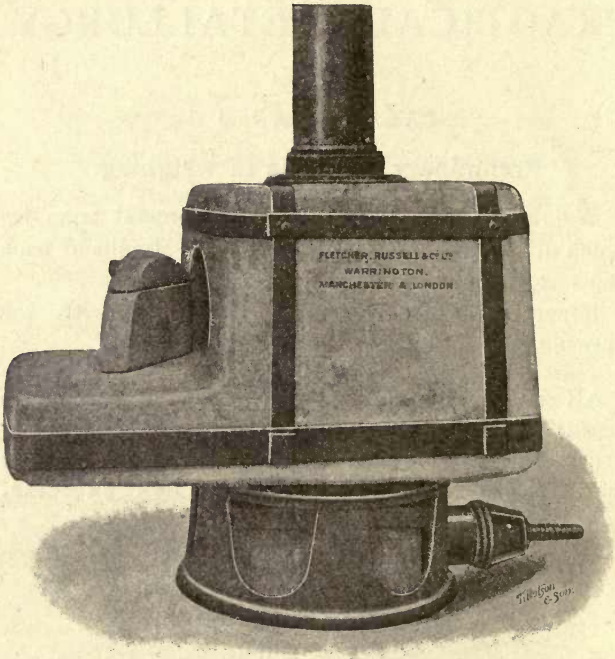

FIG. 1.—External view of gas-fired muffle furnace.

The author prefers to have the gas and air supply

arranged at the *front* of the muffle furnace: not at the back, as shown in Fig. 2. See Frontispiece.

A gas-fired muffle furnace can be easily maintained

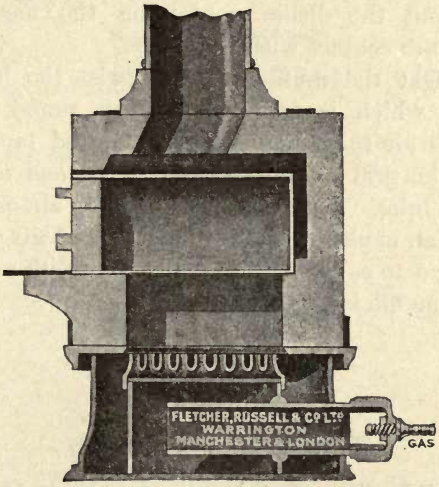

FIG. 2.—Gas-fired muffle furnace, in section, showing internal arrangement.

at a constant temperature high enough for many metallurgical operations. When the gas, mixed with

FIG. 3.—Muffle.

a regulated quantity of air, is ignited, a well-diffused flame is effectively spread, and the thick fire-clay casing around the muffle prevents outward radiation of the

heat, while reflecting it where required. A sheet-iron chimney carries off the products of combustion. In the muffle (which is of fire-clay, and may or may not have numerous slots) the various operations are carried on without the dishes containing the metals, etc., coming into contact with the flame.

To light the muffle furnace, twist the loose iron cylinder, which is near the gas-tap, round so as to cover the air inlet, apply a long, lighted taper at the edge of the grid underneath the casing, and then open the gas inlet. The gas should ignite almost immediately; an explosive mixture of gas and air must not be allowed to collect before ignition. With reasonable care the muffle is quite safe.

EXERCISE NO. 3.

To melt and pour Tin.

The intention of this exercise is to teach the beginner to weigh accurately, to melt a metal with the minimum of oxidation, and to pour it into a mould with as little loss as possible. Simple as the exercise

FIG. 4.—Scorifier.

is, the student who performs the experiment satisfactorily will have advanced a step towards success in Practical Metallurgy.

Weigh 10 grammes of granulated tin.

Place the weighed sample on a small scorifier (Fig. 4).

Set the scorifier and weighed tin on a clean scorifier,[1] and by means of scorifier tongs (Fig. 5) set in a

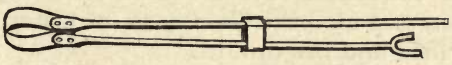

FIG. 5.—Scorifier tongs.

hot muffle, and allow to remain until the metal has melted.

Pour the melted tin into a tapered iron mould.

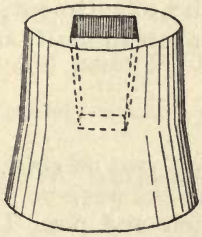

FIG. 6.—Mould for tapered ingot.

Before using a mould, it should be finely coated inside with powdered graphite. This is applied, in a slightly moist condition, with a brush. The coated mould should then be gently dried, and used while it is just warm enough to be comfortably carried by hand.

When cold, turn the little ingot of tin out of the mould.

With a hard brush (such as a tooth-brush) clean off any loose oxide from the ingot.

Weigh the ingot and note the weight.

[1] It is good practice to set each containing dish, cupel, etc., placed in the muffle on a scorifier, so as to keep the dish, etc., clear of any fusible material which may have been spilt in the muffle.

Apart from the weight obtained at the end of an experiment, both teacher and student should note the condition of the metal with regard to oxidation, and also the cleanness (or otherwise) of the scorifier and the mould.

By means of a hammer and chisel, impress a mark (T) on the ingot, and keep it intact for further use.

Report.

A report should clearly state the weight of metal taken for the experiment, the condition of the muffle, time taken to melt, and weight of the ingot obtained.

EXERCISE NO. 4.

To melt and pour Lead.

Weigh 10 grammes of granulated lead.

Place the weighed sample on a small, warm, scorifier.

Put the scorifier and weighed lead into a hot muffle and allow to remain until the metal has melted.

Pour the melted lead into a tapered iron mould which has been prepared as previously directed.

A mould should always be so prepared before pouring metal into it.

When cold, turn the little ingot of lead out of the mould.

Cooling may be hastened by leaving the mould, containing the metal, in the outlet from a cold

pot furnace to the flue, and drawing out the damper so as to allow of a "draught" of cold air. Close the outlet when done.

Brush off loose oxide.

Weigh the ingot and note the weight.

With the tang[1] of a file draw a mark (L) on the ingot and keep it intact for further use.

Report.

EXERCISE NO. 5.

To melt and pour Tin-lead Alloy.

Weigh 5 grammes of tin and 5 grammes of lead.

Granulated metals, or thin metal strips, should, as a rule, be used for experiments.

Mix the weighed samples and place on a small, warm, scorifier.

Place the scorifier and contents in a hot muffle and allow to remain until the metals are melted.

Stir with a wood slip[2] so as to help the metals to alloy thoroughly.

Alloys should always be well stirred before pouring.

Pour into a tapered mould.

When cool, clean and weigh the cast alloy.

[1] The tang is the pointed part which is intended to be fixed in a wooden handle. Try the tang on the tin ingot.

[2] Almost any odd slip of wood, say half an inch square and of convenient length, will do. It soon becomes charred at the heated end when used for stirring hot metals.

Note the weight.

Mark the ingot "S," and keep it intact for further use.

Report.

EXERCISE NO. 6.[1]

To compare Melting-points.

Arrange on a roasting dish, at intervals from each other, the three little ingots obtained in the previous experiments.

Note the position of each.

Place the roasting dish with the ingots in the centre or back part of a hot muffle.

Note the order in which the ingots melt.

Stir the alloy with a slip of wood and pour it into a half-round mould (Fig. 7).

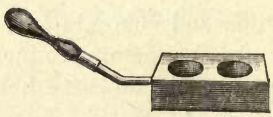

FIG. 7.—Mould for hemispherical ingots.

When cool, brush, weigh and report.

Put the ingot into an envelope marked "tin solder," with date and signature.

Hand the packet to the teacher along with the report.

[1] Where a number of students are working together, some of them might with advantage perform Exercise No. 6A as an alternative to this one.

EXERCISE NO. 6A.

To compare Melting-points.

Arrange the three small ingots and perform the experiment as detailed in No. 6, except that it is to be conducted in a newly-lighted muffle.

Note the order in which the ingots melt.

Stir the alloy with a slip of wood and pour it into a half-round mould.

When cool, weigh.

Report, and deal with the ingot as before directed.

EXERCISE NO. 7.

To ascertain the Effects of heating Zinc in a Muffle.

Weigh off 2 grammes of granulated zinc.

Place the weighed zinc on a small, warm, scorifier.

Put the scorifier and zinc into a hot muffle. Observe carefully any changes which may take place. After about 10 minutes, withdraw the scorifier.

Pour the contents of the scorifier into a broad hemispherical mould, and, when cool, examine.

Report.

EXERCISE NO. 8.

To ascertain the Effects of heating Copper in a Muffle.

Weigh off 2 grammes of granulated copper.

Place the weighed copper on a small, warm, scorifier.

Put the scorifier and copper into a hot muffle Note any change which may occur. After about 10 minutes, withdraw the scorifier, examine the contents, and report.

A perfected form of muffle furnace is shown in Fig. 7A. Muffle furnaces are also arranged for higher

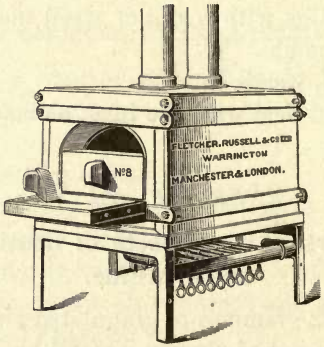

FIG. 7A.—Muffle furnace, new form.

temperatures by employing forced blast, and similar furnaces can be had for which oil may be used as fuel.

EXERCISES IN A CRUCIBLE OR "POT" FURNACE.

A crucible furnace is generally built of good fire-bricks, arranged to enclose four sides of a rectangular space.

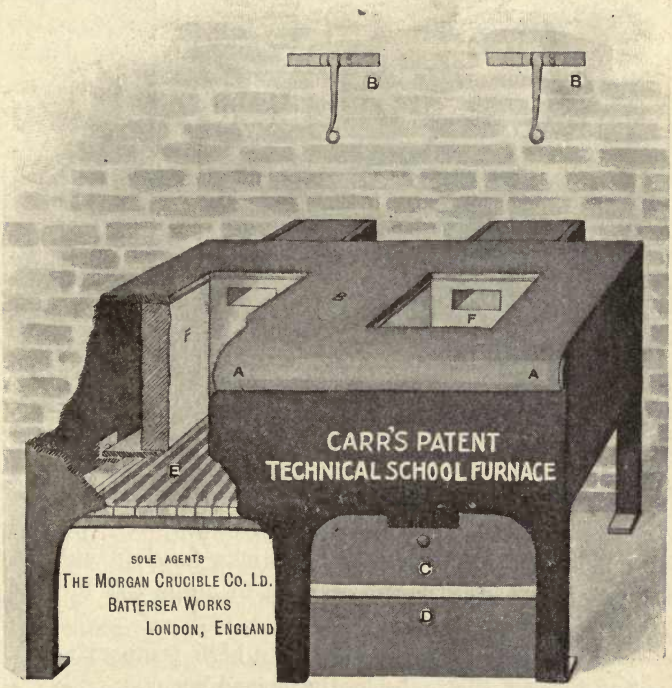

FIG. 8.—Crucible furnace.

A, Cast-iron top and flange. B, B, Dampers. C and D, Movable plates in front of ashpit. E, Fire-bars. F, Fire-brick lining.

At the back and near the top there is an opening to the flue which leads to the chimney.

At the bottom of the grate, movable cast-iron bars —the fire-bars—are placed a little apart from each other. The fire-bars are carried on iron[1] supports or

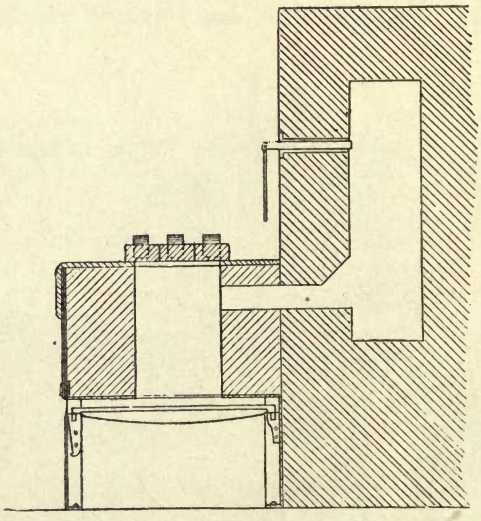

FIG. 9.

bearers extending along the front and the back of the furnace. Underneath there is an ashpit.

A cover, consisting of fire-bricks fixed in an iron frame or in iron clamps, serves to close the furnace when required.

As a smokeless fire yielding a high temperature is often needed, the fuel usually burned is coke.

To kindle a coke fire it is customary to first light a

[1] Mild steel is now much used where wrought-iron was formerly.

small coal fire in the furnace with wood, and, when the coal has kindled, to place a little coke on the top. When the coke becomes incandescent,[1] more coke is added as required. Gas coke is often good enough for a pot furnace. When a very high temperature is required, best coke, either by itself or along with some anthracite, should be used.

The temperature attainable depends on the construction of the furnace and the height and area of the chimney, on the character of the fuel, and the rate at which air is admitted to the fire.

The "draught" of air is regulated by a plate or doors, designed so as to more or less completely close the opening in front of the ashpit, and more especially by the damper.

The damper is a sliding iron plate set in a suitable frame, by which the uprush of hot gases from the furnace to the chimney may be checked if the damper is pushed in so as to partly close the flue or chimney.

When a higher temperature is needed, the cover is put on, and the damper is drawn out so as to leave a free passage for the hot gases from the furnace. As these hot gases rush off, air is freely drawn in, the oxygen of which urges the fire.

To reduce the temperature, the damper is pushed inwards, and more coke is thrown into the furnace.

By drawing the cover partly off from over the back part of the furnace, air is drawn almost directly into the chimney, and cools the upgoing gases from the furnace, thereby lessening the draught.

In making up a fire for a crucible (Fig. 10), place an old "pot" in position in the furnace and pack coke

[1] "Incandescent;" from the Latin *candeo*, to glow.

round about it. When the charge has been weighed out and put into the crucible in which it is intended to be melted, the old pot should be taken out and the

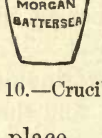

FIG. 10.—Crucible.

charged one put in its place. There should be a few inches of live coke under the crucible,[1] and the whole depth of the crucible should be embedded in glowing coke pressed against it, although not too tightly packed.

It is well, in many instances, to start working a pot furnace at a comparatively low heat, and arrange for a gradual rise of temperature.

When the contents of a pot have become fluid, they should be stirred with a slip of wood,[2] and, when in proper condition for casting, quickly poured into a prepared mould. The contents should come clean out of the pot.

EXERCISE NO. 9.

To ascertain the Effect of melting Zinc in a Covered Crucible.

Weigh off 10 grammes of zinc.

Place in an "A" Battersea round crucible, press

[1] Large crucibles are set on fire-clay stands (a half brick is often used as a stand) so as to be kept steady, and a few inches above the fire-bars.

[2] The gases evolved from the heated wood may have a beneficial effect.

it gently down, and sprinkle a little carbon powder[1] over it.

Put the crucible and contents in a live pot furnace, which for this experiment should *not* be at a high temperature.

Cover the crucible with a lid.

Occasionally uncover the crucible to see the condition of the metal.

When the metal appears to have become molten, stir and pour.

When cool, brush off the zinc oxide and weigh the metal.

Note the weight.

Examine the properties of the metal.

Report.

EXERCISE NO. 10.

To granulate Zinc.

Remelt the zinc from Exercise No. 9 and granulate it by pouring it in a thin stream into a bucket containing a good depth of cold water. While pouring, keep the crucible moving so as to spread the zinc over as much of the bottom as possible, and thus cause the granulated zinc to be in many small pieces.

Gather the granulated zinc, dry, and keep it for further use in a small envelope marked "Zinc."

[1] Either charcoal, anthracite, or bituminous coal in fine powder. Enough should be added to cover the metal when it has melted.

EXERCISE NO. 11.

To ascertain the Effect of melting Copper in a Closed Crucible.

Weigh off 10 grammes of copper, place in an A crucible, and sprinkle a little carbon powder uniformly over it.[1]

Put the crucible and contents in a live pot furnace which for this experiment may be at a fairly high temperature—say bright red.

Cover the crucible with a lid.

Increase the heat.

Examine from time to time—to ascertain the condition of the metal. When it is melted, stir and pour.

When cool, weigh.

Note the weight.

Examine the properties of the metal.

Report.

EXERCISE NO. 11A.

To granulate Copper.

Remelt the copper from Exercise No. 11 and granulate it.

Gather the granulated copper, dry, and keep it for use in the following experiment.

[1] **The precaution of melting metals *under carbon powder* should never be neglected.**

EXERCISE NO. 12.

To granulate Lead.

Melt about 20 grammes of lead, and allow to cool down till about to solidify.

Pour it quickly into a stout wooden box which has been internally blackleaded or charred.

Shake the box vigorously, so as to cause the lead to break up into fine particles.

Put the product in an envelope marked "Lead."

Report.

EXERCISE NO. 13.

To granulate Aluminium.

Melt about 15 grammes of aluminium in a covered pot.

Allow it to cool down in the pot, and, when nearly solid, stir it vigorously with a charred wooden stick until the mass becomes finely divided.

Put the product in an envelope marked "Aluminium."

Report.

EXERCISE NO. 14.

To test the Malleability of a Metal at different Temperatures.

Place five cast-zinc discs, about $\frac{1}{4}$ inch thick and $\frac{1}{2}$ inch diameter, apart from each other on a scorifier in a hot muffle.

While they are being heated, test the malleability or the brittleness of the cold metal by hammering a similar disc on an anvil.

By means of tongs (Fig. 11) withdraw one of the discs from the muffle, and test its behaviour under the hammer.

Fig. 11.—Tongs.

At suitable intervals withdraw and test each of the others.

Put all the used metals in an envelope marked "Zinc."

Report.

PREPARATION OF ALLOYS AND EXAMINATION OF THEIR PROPERTIES.

EXERCISE NO. 15.

Make a small ingot of gun metal by melting 25 grammes of copper and then adding 2·5 grammes of tin.

FIG. 12.—Long mould.

When melted, stir well, and pour into a long mould (Fig. 12).

When cool, weigh, and note the weight.

Test the alloy as directed below, and, when finished, report.

NOTE.—Melting would take place at a lower temperature if the copper and tin were charged together. But the trade practice is to melt the copper first and then add the tin. Tin may be easily oxidized at a high temperature.

GENERAL SCHEME FOR TESTING AN ALLOY.

"Nick" the ingot (that is, make a deep indentation with a chisel) *across* the top.

Fix one end in a vice, and hammer the free portion

so as to bend it alternately backwards and forwards until it breaks.

File a piece of one of the edges (*but not the fracture*), finishing with a smooth file, so as to show the appearance of the alloy when smoothed and polished.

Carefully preserve the piece.

Hammer one end of the other piece so as to test the *malleability* of the alloy.

Push the flattened end between the rollers of a mill,

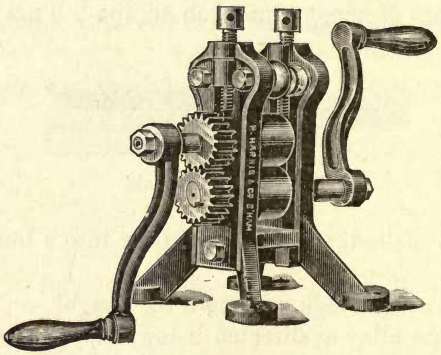

FIG. 13.—Rolling mill.

and proceed to roll out the alloy in order to test its ductility. If, during this test, it shows signs of stress by breaking at the edges, gently heat it for a little in a muffle; allow it to cool, and then continue the rolling test.

Carefully note all particulars.

Put the rolled piece, along with the other piece, in an envelope, properly marked with the date, experiment number, name of the alloy, weight of alloy enclosed, and signature.

Report.

EXAMPLE OF REPORT.

Date, .

Exercise No. 15.

Gun Metal.

Melted 25 grammes of copper in a crucible in a furnace at a full red heat, added and melted 2·5 grammes of tin, stirred with wood, and poured into a long mould which had been prepared and slightly warmed. Weighed the ingot when it had cooled.

Weight of metals used = 27·5 grammes.
Weight of ingot = 27·2 ,,

Loss = 0·3 gramme.

Nicked and broke the ingot. It was tough, and, when broken, showed a close-grained fracture. It was, when filed, moderately hard, and was rather more reddish in colour than brass. On being hammered, it flattened well before cracking. A sound portion passed repeatedly between the rolls, and was thus flattened into a thin strip before tearing.

Samples, weighing 27 grammes, enclosed.

The envelope may be marked as in the following example:—

Date, .
Gun metal, 27 grammes.
(Signature) .

Unless when directed to retain the ingot it should be handed in with the report.

EXERCISE NO. 16.[1]

To test the Effect of Cooling an Ingot slowly.

Melt 10 grammes of lead, and add thereto 11 grammes of zinc.

When the zinc has melted, stir it well.

Pour the alloy into a long mould which has been warmed to about 100° C.

Cover the mould with a slab of hot fire-brick or a hot crucible lid.

When the ingot has cooled, weigh it and note the weight.

Nick the ingot first on the top and then on the bottom.

Break the ingot, and examine the fracture.

Report.

EXERCISE NO. 17.

To test the Effect of Cooling an Ingot quickly.

Remelt the product of the previous experiment, stir it well, and pour the alloy into a long, cold mould.

When the ingot has cooled, weigh it and note the weight.

Nick the ingot on top and bottom, break the ingot, and examine the fracture.

Report.

[1] Where a number of students are working together, some of them might weigh out the materials, and try the effect of quick cooling (as instructed in No. 17), remelt, and try the effect of slow cooling.

EXERCISE NO. 18.

Make a small ingot of type metal by weighing off 27 grammes of lead, including a piece of clean, thin sheet lead of about half the weight, and 2·5 grammes of tin.

Weigh off also 5·5 grammes of roughly powdered antimony and wrap it in the piece of sheet lead.

Charge the weighed tin and the small pieces of the lead sample into a crucible and place in a pot furnace which is at a red heat.

When the metals have melted, push the antimony and the wrapping of lead down under the melted metal, and quickly cover the crucible. When all the alloy has melted,[1] stir and pour.

When cool, weigh and note the weight.

Test, and enclose the pieces in an envelope.

Report.

EXERCISE NO. 19.

Make a small ingot of brass[2] by melting together 18 grammes of copper and 11 grammes of zinc.

When melted, stir and pour.

[1] Remember that antimony is apt to volatilize considerably if kept long in a hot furnace. The alloy must, however, be thoroughly melted and mixed before pouring.

[2] One difficulty in making brass is due to the fact that zinc can assume the gaseous state while below the melting-point of copper.

When cool, weigh and note the weight.

Examine and note the properties.

Report.

EXERCISE NO. 20.

Make a small ingot of brass by *melting* 18 grammes of copper and *immediately* adding thereto 11 grammes of stick or block zinc. The zinc should be, by means of tongs, held under the melted copper for a little.

When the zinc has melted, stir and pour.

When cool, weigh and note the weight.

Examine and note the properties.

Compare the loss of zinc with that lost during Exercise No. 19.

Calculate the percentage loss (see example on p. 126).

Assuming that all the loss is due to volatilization of zinc, calculate the percentage composition (see example on p. 126).

Report.

EXERCISE NO. 21.

Make a small ingot of Muntz metal from 17 grammes of copper and 12 grammes of zinc.

Calculate the percentage loss and the percentage composition.

Examine and note the properties.
Report.

EXERCISE NO. 22.

Make a small ingot of brazing solder from 15 grammes of copper and 17 grammes of zinc. Pour into a half-round mould.[1]

Calculate the percentage loss and the percentage composition.

Examine and note the properties.

Report.

EXERCISE NO. 23.

Prepare a table of the quantities you would use to make a 30-gramme[2] ingot of each of the following alloys:—

Pewter.
Fine solder.
Dutch metal.
Bell metal.
Speculum metal.

For table of alloys, see pp. 123 and 124; and for example of calculation, see p. 128.

[1] If a number of students work this exercise together, they should arrange to cool their ingots at different rates and carefully note the effects on the appearance of the fracture.

[2] The same method will, of course, apply to the much larger quantities required for castings and for ingots in works practice.

EXERCISE NO. 24.

Prepare a table of the quantities you would use to make a 30-gramme ingot of each of the following alloys:—

Common solder.
Britannia metal.
Bronze.
Fusible alloy.

EXERCISE NO. 25.

Make a small ingot of German silver by first preparing a brass ingot from 10 grammes of copper and 11 grammes of zinc. Then melt together 8 grammes of copper and 7 grammes of nickel.

Heat the brass to redness, and add it to the melted copper-nickel alloy in the crucible.

Stir well when melted, make sure that the alloy is hot, and pour quickly into a long mould.

When the alloy is cool, weigh it and note the weight.

Test the alloy, place the pieces in an envelope, and report.

EXERCISE NO. 26.

Make a small ingot of aluminium bronze by melting 25 grammes of copper and adding 2·5 grammes of aluminium sheet clippings or aluminium powder.

Stir the alloy well when it is melted.

Allow the alloy to remain melted in the crucible for a few minutes, and then pour.

When the alloy is cool enough, weigh it and note the weight.

Test the alloy, place the pieces in an envelope, and report.

EXERCISE NO. 27.

Make a small ingot of "Regulus of Venus" by melting together 12 grammes of granulated copper mixed with 13 grammes of roughly powdered antimony.

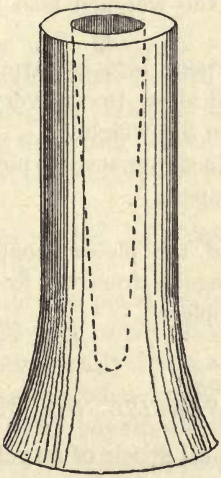

FIG. 14.—Mould for tapered circular ingots.

Be careful to keep a lid on the crucible during the melting.

When melted, stir and pour the alloy into a warm, narrow, tapered, circular mould (Fig. 14).

When cool, weigh and note the weight.

Examine and note the properties of the alloy.

Retain the alloy for the next exercise.

EXERCISE NO. 28.

To see the Separation of Lead from "Regulus of Venus."—Experiment illustrating "Segregation."

Weigh off a quantity of thin lead clippings or granulated lead equal to the weight of regulus of Venus obtained in the previous experiment.

Melt together the weighed lead and the sample of regulus.

Stir well, and pour into a warm, narrow, tapered, circular mould, and allow to cool very slowly.

Weigh and note the weight.

Break the product into several pieces.

Examine and report.

NOTE.—Some of the pieces should show a decided separation of the regulus from the lead. The separation is, however, not complete.

EXERCISE NO. 29.

To make a small stock of cupels (Fig. 15) for use in future experiments.

Weigh off about 4 ounces of bone ash from a stock which has been passed through a 40-mesh sieve.

On a board with sides, place about 3 ounces of

the bone ash in a conical heap. Make a cavity in the cone, and in the cavity put some water.

If a *small pinch* of sodic carbonate has been previously dissolved in the water firmer cupels may be made. The addition of too much sodic carbonate would, however, spoil the cupels.

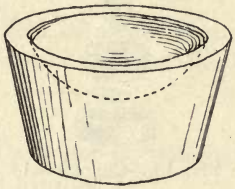

FIG. 15.—Cupel.

Mix the water with the bone ash, and knead the mass well. Enough water should be added to make the kneaded mass just clot and hold together when pressed by closing the hand. The reserved bone ash is intended to correct the addition of too much water if it has been added.[1]

The cupels are shaped in a mould by means of a plug or die (Fig. 16).

Clean the mould and place the disc in position.

Fill the mould with some of the moistened bone ash. Press the plug firmly down and strike it several smart blows with a wooden mallet, turning the plug after each blow. The cupels, although firm, ought to be porous.

Push the cupel out of the mould by pressing the

[1] If too little water is used the cupels will be apt to crumble; if too much water is used the porosity of the cupels—an important quality—is lessened.

disc upwards. Trim the edges, where necessary, with a sharp knife.

Proceed to make about a dozen cupels of different degrees of thickness.

FIG. 16.—Cupel mould.

Mark each cupel with initials and date, and put them in a warm, dry, place, to be kept there for a month or more before using.

EXERCISE NO. 30.

To put a Lining of Magnesia[1] in a Crucible.

In a mortar or other suitable vessel make a thick paste of magnesia and water.

Put a lining, about half an inch thick, of the magnesia paste in a clean A crucible.

Set to dry for two or three days, after which heat gently for about an hour.

Place the magnesia-lined crucible in a hot pot furnace, gradually raise the temperature, and allow to remain for an hour or more.

[1] Magnesia withstands the powerful reducing action of aluminium. Magnesia crucibles and plumbago crucibles lined with magnesia are now produced by manufacturers.

Withdraw the crucible and allow it to cool down very slowly.

Keep the crucible in good condition for experiments to follow.

Report.

EXERCISE NO. 31.

To test the effect of a moderate temperature on red lead, and to test the effect of the product on a fire-clay dish.

Select a broad scorifier or roasting dish, and place 10 grammes of red lead on it.

Set in a muffle which is a low red heat, and allow to remain there for about forty minutes.

With care all the red lead (Pb_3O_4) may be converted into litharge (PbO), which, while it remains unmelted, does not act on the dish. The lead oxide, when melted, acts on the dish with formation of lead silicate.

Brush off as much lead oxide as possible and keep it for further use.

Report.

EXERCISE NO. 32.

To test the effect of melted lead oxide on a fire-clay dish in a hot muffle.

Place 5 grammes of lead in a clean scorifier and set in a hot muffle.

Allow to remain there until all the lead has become oxidized, maintaining the highest possible temperature meanwhile.

Pour off as much as possible from the scorifier into a warm mould, and, when the lead oxide (PbO) is cool, weigh and keep in an envelope for future use.

Note if under these conditions the scorifier has been glazed.

Report.

EXERCISE NO. 33.

To test the effect of melted litharge (PbO) on a fire-clay crucible at a high temperature.

Put into a clean A crucible 4 grammes of litharge (PbO) mixed with about a gramme of borax.

Set the crucible and contents in a pot furnace, which is at a dull red heat.

Allow to remain for half an hour or more, the temperature being steadily raised and kept at full red.

Withdraw the crucible, and pour as much as possible of the contents into a warm mould.

Keep the pourings (if any) and the crucible for further use.

Examine the state of the crucible.

Report.

EXERCISE NO. 34.

To test the effect of melted copper oxide (CuO) on a fire-clay crucible at a high temperature.

Put into a clean A crucible 3 grammes of copper oxide mixed with about a gramme of borax.

Set the crucible and contents in a hot pot furnace.

Raise the temperature to a bright red heat.

After the contents have melted, allow to remain in the furnace for twenty minutes at least.

Take out the crucible, and pour out as much as possible of the contents.

When the crucible is cool, examine it and report.

EXERCISE NO. 35.

To test the efficiency of a magnesia-lined crucible in resisting action by hot basic oxide.

Put into the crucible, prepared as directed in Exercise No. 30, the charge prescribed for Exercise No. 33, and carry on the test as before.

When ready, withdraw the crucible, and pour out as much as possible of the contents.

Examine the state of the crucible.

Keep the crucible and the pourings for further use.

Report.

EXERCISE NO. 36.

To test the Efficiency of a "Pure Carbon" Crucible.

Select a small "pure carbon" crucible, and pack it in a larger-sized fire-clay crucible, filling the interspace with white sand, as shown in Fig. 17.

Charge into the pure carbon crucible a mixture of 3 grammes of copper oxide and 1 gramme of borax.

Proceed as directed for Exercise No. 34.

When ready, withdraw the crucible, and pour out as much as possible of the contents.

Examine the state of the crucible.
Keep the crucible and pourings for further use.
Report.

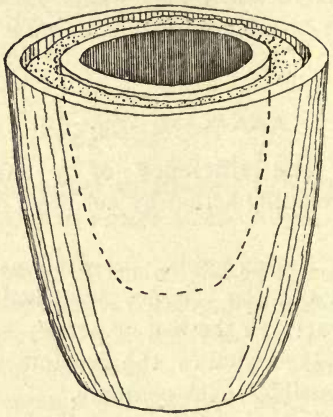

FIG. 17.—Carbon crucible in sand.

NOTE.—Pure carbon crucibles are convenient, and for certain purposes are economical. They withstand basic oxides well, and, if kept in a thick bed of glowing coke, they do not burn away readily.

A brasqued crucible may be prepared by filling a fire-clay crucible with a stiff mixture of treacle and crushed carbon, pressing the mixture, shaping a cavity, gently drying, and then firing at a red heat while so covered as to exclude air from the brasque.

The treacle should be well mixed with an equal bulk of water, and, when required, should be added to charcoal powder in quantity sufficient to make a mixture which will clot when pressed.

FORMATION OF OXIDES.

EXERCISE NO. 37.

Oxidation of Copper.

Tare a disc (Fig. 18) by heating it in a muffle at a red heat for about ten minutes, withdrawing it, and, when cool enough to be lifted by hand, placing it in a desiccator (Fig. 19) to cool, and then weighing the disc.

FIG. 18.—Disc.

FIG. 19.—Desiccator.

Note the weight of the tared disc.

Weigh off 5 grammes of clean copper filings and spread the weighed sample on the tared disc.

Place on a scorifier in a hot muffle and allow to remain at a high temperature.

Stir the filings so as to expose the portions which have not been acted on.

When oxidation is judged to be complete withdraw the disc, and, when cool,[1] reweigh.

[1] May be allowed to cool down to such an extent that the disc can be carried by hand; cooling should be finished in a desiccator.

Note the result, calculate the percentage increase and compare with the maximum theoretical increase.

Keep the cupric oxide (CuO) for use in a future experiment.

Report.

Example of record—

	grammes.
Weight of disc	= 16·35
Weight of sample	= 5·00
Weight of disc and sample before roasting	= 21·35
Weight of disc and sample after roasting	= 22·48
Increase in weight	= 1·13

For examples of calculation, see p. 126.

EXERCISE NO. 38.

Oxidation of Tin.

Tare a fire-clay disc.

Cut out several pieces of tinfoil nearly to the diameter of the disc, but not too large.

Weigh off 3 grammes of the cut foil and spread one of the pieces on the tared disc.

Place on a scorifier in a hot muffle and allow to remain at a high temperature until oxidation is judged to be complete. Then place successively the other cut pieces, allowing each one to be completely oxidized before adding the next.

When all have been oxidized withdraw the disc, and, when cool, reweigh.

Note the result, calculate the percentage increase, and compare with the maximum theoretical increase.

Keep the stannic oxide (SnO) for use in a future experiment.

Report.

EXERCISE NO. 39.

Oxidation of Iron.

Experiment on 5 grammes of clean iron filings, as in previous experiments, and report.

EXERCISE NO. 40.

Oxidation of Antimony.

Experiment on 5 grammes of powdered antimony, as in previous experiments, and report.

EXERCISE NO. 41.

Conversion of Cupric Oxide (CuO) into Cuprous Oxide (Cu_2O).

Weigh off 13 grammes of fine-pounded black copper oxide and 10 grammes of copper filings.

Mix well and place in a D crucible.

Set the crucible and contents in a pot furnace which is at a dull red heat.

Gradually raise the temperature, and, when the mass in the crucible has thoroughly melted, stir well and pour into a warm mould.

When cool, weigh.

Note the results and report.

CUPELLATION.

Cupellation is an operation by which lead and other common metals are oxidized and thus separated from the precious metals, which resist oxidation under the proper conditions for cupellation.

In the laboratory the process is conducted on a cupel in a hot muffle. If the temperature of the muffle is high enough, the oxide or oxides will be absorbed *in* the cupel, *on* which the unoxidized precious metals will remain.

The litharge (PbO) formed by the oxidation of the lead is an active oxygen carrier, and aids the oxidation of other metals; it has also great power for dissolving other oxides and carrying them into the cupel.

The cupel should be firm yet porous, and of such a nature as not to be chemically acted on by the metallic oxides. Cupels are generally made of bone ash, which is essentially calcic phosphate ($3CaO \cdot P_2O_5 + H_2O$).

Morganite cupels are not so tender as bone ash ones, and in some other respects Morganite cupels are superior.

In works, cupels are of bone ash, or marl and selected clays, or may be of cement, and a reverberatory furnace is employed.

Hints on Cupellation.

Cupels should be made several weeks before being used. They should be stored in a warm place.

A good cupel can absorb rather more than its own weight of lead oxide (litharge).

Have the muffle hot and the cupel hot before placing the alloy on it.

Start and finish cupellation with the muffle hot.

When oxidation has commenced, work at a more moderate temperature.

Do not move the charged cupel until the operation is finished.

The alloy to be cupelled should be in the form of a cube, so that when it melts a rounded top, which facilitates the removal of litharge, is formed.

An indication of the temperature of the furnace is furnished by the fumes. If they just rise over the edge of the cupel and then drop, the muffle is too cold; if they rise to the top of the muffle, the temperature is too high. When the muffle is too hot there is an undue loss of the precious metals.

The temperature of a muffle increases from the front to the back.

Sometimes cupellation ceases even in a hot muffle. This stoppage—known as "freezing"—may be caused by the cupel not being hot enough or not sufficiently porous to absorb the oxides quickly. Freezing may be overcome by raising the temperature, placing a splinter of wood, such as a doubled-up match, on the frozen mass, or by sprinkling sawdust or charcoal, or placing a small cube of clean lead on the charge on the cupel. A lump of glowing charcoal in front of the cupel may prevent or cure freezing.

EXERCISE NO. 42.

To see the Effect of Heat and Air on Various Metals during Cupellation.

Select five small cupels and mark boldly with pen or pencil a distinguishing letter, A, B, C, D, or E, on each cupel.

Set the marked cupels on scorifiers in a hot muffle.

When they are hot place by means of cupel tongs (Fig. 20) a clean lead disc,[1] weighing about 4 grammes,

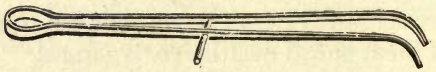

FIG. 20.—Cupel tongs.

on A; a disc of thick tin foil on B; a disc of copper foil on C; a weighed sixpenny coin, or a piece of silver of about equal weight, on D; and by means of a spoon sprinkle some powdered antimony (say about half a gramme) on E.

Keep all at a high temperature until all the lead has disappeared in the cupel.

Withdraw the scorifiers and cupels.

Examine and note the result in each case.

Report.

[1] The discs for this exercise should be about two-thirds the diameter of the cupels.

EXERCISE NO. 43.

To see the Effect of Addition of Lead, and the Action of Heat on the Metals.

By means of the scorifying tongs, set the cupels, with their charges, on scorifiers in a hot muffle, and, when all are at a red heat, place a *cube* of lead, weighing about 5 grammes, on the metal or oxide on cupels B, C, D, and E.

Close the front of the muffle, all except the space required through which to watch the operation.

When the lead has melted and oxidation and clearing have begun, lower the temperature a little so as to avoid excess loss by volatilization. Towards the end of the operation raise the temperature so as to finish hot.

When cupellation is finished withdraw the cupels, examine and note the result in each case, including the weight of the silver bead.

Report.

EXERCISE NO. 44.

Separation of Silver from Lead by Cupellation.

Melt the weighed silver bead from the previous experiment in about six times its weight of lead. Pour into a tapered mould, and, when the ingot has set, transfer it to a hot cupel in a hot muffle, and allow cupellation to proceed.

When finished, withdraw the cupel.

When cold, remove the silver bead; clean and weigh it.

Report.

EXERCISE NO. 45.

Separation of Silver from Hard (Impure) Lead by Cupellation.

Place a 25-gramme cupel on a scorifier in a hot muffle.

Weigh off about 15 grammes of lead, and about 0·5 gramme of copper and 0·5 gramme of antimony.

Melt these in a crucible along with the weighed silver bead from the previous experiment.

When well melted, stir and pour into a tapered mould, and, when the ingot has set, place it on the hot cupel. Watch the process of cupellation. If, after a time, cupellation stops, raise the temperature and place a 5-gramme cube of clean lead on the mass in the cupel. Further additions of lead may be necessary.

When the operation is finished, withdraw the cupel, clean and weigh the bead of silver.

Report.

NOTE.—The Morgan Crucible Co. has patented a muffle which allows admission of hot air at several points. This tends to prevent freezing, hastens cupellation, and lessens variations in assay results.

EXERCISES IN SCORIFICATION.

We may in the mean time consider scorification as an operation by which precious metals are concentrated in clean lead. For wider meaning of the term consult a book on theoretical metallurgy.

The previous exercises should have taught the student that lead can be melted and oxidized in a gas muffle, and that its oxide (litharge) can be absorbed in a porous bottom;[1] that certain other metals become oxidized, but their oxides are not absorbed (unless sufficient litharge is present), and that those latter metals interfere with cupellation.

EXERCISE NO. 46.

To note the Effect of attempting to cupel Impure Lead, and the result of Scorifying before Cupelling.

Make an ingot of 10 grammes of lead, 1½ grammes of bronze or brass filings, and about half a gramme of antimony.

Cast in a tapered mould, and, when the alloy is cold, cut about one-third off.

Set the small portion on a hot cupel in a hot muffle and proceed with cupellation.

[1] Bismuth behaves like lead during cupellation.

Set the large portion on a hot scorifier in the same muffle, and, when the piece has melted, add about 1 weighed gramme of borax.[1]

Allow oxidation to proceed till about half of the lead has become oxidized.

Add a small cube (about 4 grammes) of lead, and allow oxidation to continue till about 4 grammes of lead are left in the metallic state.

Pour the metal and scoriæ (or slag) into a tapered mould, and, when the mass is quite cold, separate the metal by setting the tapered ingot on one of its sides, and breaking away the scoriæ in such a way as not to drive it into the soft lead.

Clean the lead with a brush, and cut off a small portion without spoiling the cube shape of the remainder.

Test the small piece by flattening it out into a thin sheet—this, if pure enough, it will allow without cracking.

If not pure enough, scorification must be repeated.

If the test is satisfactory, place the cube on a hot cupel in the hot muffle and allow cupellation to go on till finished.

EXERCISE NO. 47.

Cupellation preceded by Scorification.

In an A crucible melt 10 grammes of lead, 1 gramme of antimony, and about half a gramme of copper.

When the alloy has melted, add the silver bead

[1] Borax absorbs and retains such portions of the metals as may become oxidized.

from the previous experiments, stir well, and cast the alloy in a tapered mould.

When the ingot has set, place it on a scorifying dish along with a gramme or two of borax.

Place in a hot muffle and allow to remain until about half of the metals have been removed by oxidation.

Pour into a tapered mould.

When the "pourings" have become cold, break away the metallic portion from the slag.

Place the clean metallic portion on a hot cupel in a hot muffle, and allow action to go on till finished.

Remove the cupel and detach the silver bead.

Clean and weigh the bead and record the weight.

Report.

NOTE.—If during scorification the metal becomes covered with slag, "clean" by adding a small quantity of powdered anthracite so as to reduce a little of the litharge. The silver will thus be concentrated in the reduced lead.

LIQUATION.

This is an operation by which a comparatively low melting-point alloy may be separated from a metal or alloy of higher melting-point. It cannot be conveniently conducted in an oxidizing atmosphere.

Liquation plays an important part in the metallurgy of tin. It is also useful for concentrating precious metals in lead and carrying them away from other metals, the lead containing the precious metals being afterwards cupelled.

EXERCISE NO. 48.

To test the Effects of Liquation.

Melt together 4 grammes of copper and the weighed silver bead from the previous experiment. Add 16 grammes of lead, melt all together, stir, and pour into a half-round mould.

When the alloy has solidified, set it on a sloping lid inside a broad-based crucible. Cover with another lid. Heat gently so as to cause some of the lead, alloyed with silver, to separate from the original alloy and flow to the bottom of the crucible.

An alternative, and perhaps better, plan is to place the alloy which is to be liquated in a perforated crucible set in another crucible. The liquated alloy should flow into the lower crucible.

Or liquation may be conducted on a sloping slab, or a hollowed-out piece of hard charcoal, in a hot muffle in which there is plenty of glowing anthracite to "correct" the atmosphere in the muffle.

Cast the liquated alloy into a cube.

Cupel the alloy.

Weigh the silver obtained and report.

EXERCISE NO. 49.

To recover more Silver by repeated Liquation and Cupellation.

Melt the unliquated mass from the previous experiment along with about three times its weight of lead.

Stir well, and pour into a half-round mould. Proceed with liquation and cupellation as before.

Weigh the silver obtained and report.

Keep the unliquated residue for further use.

EXERCISE NO. 50.

To soften Hard Lead by Oxidation.

Weigh off about 12 grammes of scrap lead. If it has some solder on it so much the better.

Melt it in a suitable crucible.

When melted, add about 1·5 gramme of powdered antimony and a pinch of copper filings.

Stir with a charred stick so as to mix well.

Pour the "metal" into a half-round mould.

When cold, hammer the casting and note its condition.

Hammer the casting into a cube-shaped mass.

Place the cube on a large scorifier and put it in a hot muffle.

Scrape aside the oxides which form on the surface of the melted mass.

Allow the action to continue for about half an hour, cleaning the surface from time to time.

With a small iron spoon, formed from a piece of hoop-iron, take out a small quantity of the melted metal, and cool it in an iron mould.

Hammer so as to test the malleability of the lead.

Continue the oxidation, and test from time to time.

When the lead is soft enough, *i.e.*, is quite soft and malleable, pour it into a mould.

When cold, break away any slag which may be clinging to the lead, taking care not to hammer any slag into the lead.

Weigh the softened lead and note the weight.

Report.

NOTE.—Lead oxide (PbO, or litharge), which is formed during the operation, has power to dissolve other (infusible) oxides; it is also a carrier of oxygen.

The student should now write up the particulars of a proposed experiment for testing the effects of foreign substances on the mechanical properties of a metal, and, when the scheme is approved, should perform the experiment and report the results. Overpoled and underpoled copper, the effects of arsenic on tin, iron on zinc, sulphur on iron, and many other exercises may be so arranged.

REDUCTION OF METALLIC OXIDES.

To reduce metals from their oxides, it is necessary that the proper temperature be attained, and that the oxide is kept in contact with a reducing agent. A reducing agent is a substance which at that temperature has a greater chemical affinity for oxygen than the metal has. By preference the oxygen (of the metallic oxide) unites with the reducing agent, and the metal is thus liberated or reduced.

The chief reducing agent for oxides is carbon.

EXERCISE NO. 51.

Reduction of Lead Oxide.

Weigh off 20 grammes of litharge and 0·5 gramme[1] of charcoal powder.

Mix thoroughly and place in an A crucible.

Set the crucible and contents in a pot furnace which is at a dull red heat and slowly raise the temperature.

When the contents of the crucible have become thoroughly melted, raise the temperature still higher, and keep the crucible in the furnace for a few minutes longer.

Pour into a warm long mould.

[1] The charcoal powder should be accurately weighed on a good chemical balance.

When cool, weigh and note the weight.

Examine the product.

Report.

EXERCISE NO. 52.

Reduction of Lead Oxide.

Reduce the lead from 20 grammes of litharge by ultimately mixing with 0·8 gramme of charcoal powder, and proceeding as directed for the previous experiment.

Cast the reduced lead, as before, and, when cool, weigh.

Examine the product.

Calculate the theoretical quantity obtainable on the basis of the equation—

$$2PbO + C = 2Pb + CO_2$$

Calculate the actual percentage yield and compare it with the theoretical yield.

Compare the yield with the result of the previous experiment.

EXERCISE NO. 53.

Reduction of Lead Oxide.

Weigh off 20 grammes of litharge and 1·2 gramme of charcoal powder, mix thoroughly, and proceed to reduce as in the previous exercises.

Pour the reduced lead, as before, and, when cool, weigh.

Examine the dross for lead shots; if any, add to the weight of the ingot.

Report, in tabular form, the weight of lead obtained in this and the two previous experiments.

EXERCISE NO. 54.

Reduction of Red Lead.

Calculate the amount of carbon required to reduce 20 grammes of red lead on the basis of the equation—

$$Pb_3O_4 + 2C = 3Pb + 2CO_2$$

To allow for moisture, etc., and for waste of charcoal by some of it burning to CO, add one-third to the theoretical quantity.

Report.

The reduction may then be proceeded with as in previous exercises.

EXERCISE NO. 55.

Reduction of Lead Carbonate.

Calculate the amount of carbon required to reduce 20 grammes of lead carbonate on the basis of the equation—

$$2PbCO_3 + C = 2Pb + 3CO_2$$

Allow for deficiencies in the charcoal as in the previous exercise, and report.

The reduction may then be proceeded with as in previous exercises.

EXERCISE NO. 56.

Reduction of Copper Oxide.

Calculate the amount of carbon required to reduce 20 grammes of black copper oxide on the basis of the equation—

$$CuO + C = 2Cu + CO$$

Report the amount of charcoal required.

Proceed with the reduction. The temperature of the furnace should be gradually raised, and the reduction and melting finished at a high temperature.

Slags and Fluxes.

Hitherto the substances selected for reduction have been such as do not yield **slags**—at least, not to an inconvenient extent.

For the present we may consider a slag as a fused substance which has not been reduced or volatilized.

In order that a metal may readily separate from a slag, the latter should be easily fused at the temperature of the operation, and it should be of lower density than the metal.

A **flux** may with advantage be added so that the slag will become fused more readily and be more fluid when melted in the crucible.

The chief fluxes in general use in laboratories are—

Borax ($Na_2B_4O_7 + Aq$), which is a suitable flux for silica and for lime and other oxides.

Sodic carbonate (Na_2CO_3), or, better, a mixture of ten

parts of sodic carbonate with thirteen parts of potassic carbonate (K_2CO_3), is useful for fluxing silica, etc.

The above fluxes are very fluid when melted, and are then capable of holding much solid matter in suspension.

Fluor spar or *calcic fluoride* (CaF_3) is a good flux for those ores which contain silica or barium sulphate (barytes) or calcic sulphate (gypsum). Fluor spar facilitates fusion and tends to form a very fluid slag.

Glass—which is a mixture of highly fusible silicates—helps to increase the fluidity of a slag.

Silica (SiO_2) unites with metallic oxides to form silicates which are fusible at the temperature attainable in laboratory furnaces.

EXERCISE NO. 57.

To reduce Ferric Oxide.

Weigh off 12 grammes of finely powdered hematite and mix with 13 grammes of powdered glass, 1 gramme of lime, and 2 grammes of charcoal.

FIG. 21.—Iron assay crucible.

Place the mixture in a tall crucible (Fig. 21), and lute the lid with moist fire-clay.

Dry carefully.

Place the crucible and contents in furnace.

Gradually raise the temperature, and maintain at highest attainable heat for about an hour. Anthracite and coke should be used for high temperatures.

Allow the crucible and contents to cool, tap gently so as to facilitate the separation of the metal and slag.

Allow to cool completely.

Break the crucible and examine the contents.

Weigh the metal.

Test the properties of the metal.

Report.

EXERCISE NO. 58.

Reduction of Ferric Oxide with Excess of Carbon.

Weigh off a charge exactly as for the previous experiment, but with an additional 2 grammes of charcoal.

Proceed as in the previous experiment.

Weigh the metallic bead.

Examine its properties.

Report.

Reducing Agents.

In the previous examples charcoal was recommended as the reducing agent on account of its comparative purity and large content of carbon.

Other reducing agents for oxides are—

For laboratory work—starch, flour, argol, potassic cyanide, and hydrogen.

For manufacturing operations — carbon, carbon monoxide, hydrogen, and hydrocarbons (all from coke, anthracite, or bituminous coal) are freely used.

EXERCISE NO. 59.

To test the Reducing Power of Substances.

Weigh off accurately 0·5 gramme of charcoal. Mix it intimately with 35 grammes of litharge. Treat the mixture so as to obtain as much lead as possible from it.

Treat in the same way 1·5 gramme of argol and 35 grammes of litharge.

Treat also in the same way 1·0 gramme of flour and 35 grammes of litharge.

Weigh the products in each case, and tabulate the percentage reducing power of each.

EXERCISE NO. 60.

Reduction of Tin Oxide by means of Potassic Cyanide.

NOTE.—*Potassic cyanide is a deadly poison: the student must be very careful with it.*

Cyanide has an affinity for oxygen, which it readily takes up and thereby becomes converted into cyanate. A large excess of cyanide is used in the experiment.

Weigh off 15 grammes of tin oxide and about 25 grammes of commercial 80 per cent. potassic cyanide.

Mix the weighed quantities intimately.

Place a layer of cyanide in the bottom of a D crucible.

Transfer the mixture of oxide and cyanide to the crucible.

Cover with a thin layer of cyanide.

Put the crucible and contents in a pot furnace which is at a dull red heat.

Raise the temperature until the mixture in the crucible has fused, and maintain it in that condition for about ten minutes afterwards. Avoid too high a temperature.

Pour the metal and slag into a warm half-round mould.

When cool, dissolve the slag in a basin (or mortar) of water, allowing a gentle stream of water to run in and the overflow water to carry off the dissolved potassic cyanate and cyanide.

Recover the metal button and any small beads of metal.

Dry, weigh, and note the weight obtained.

Carefully wash hands after using cyanide.

Calculate the percentage of tin obtained and compare with that obtainable from pure stannic oxide (SnO_2).

The reaction by which reduction is effected may be represented by the following equation:—

$$SnO + 2KCN = 2KCNO + Sn$$

EXPERIMENTS WITH SULPHIDES.

EXERCISE NO. 61.

To prepare Lead Sulphide (PbS).

Calculate the quantity of sulphur required to be added to 20 grammes of lead to form PbS.

Weigh off 20 grammes of finely granulated lead and the calculated amount of sulphur.[1]

Mix the weighed quantities intimately.

Place in a suitable crucible a layer of sulphur.

Put the weighed mixture in the crucible.

Cover with a layer of sulphur.

Cover the crucible with a lid which fits fairly close.

Set in a pot furnace which is at a good red heat.

When fusion is complete drop in a piece of roll sulphur about the size of a nut.

Allow to remain in the furnace for a few minutes more.

Quickly pour the product into a warm half-round mould.

When cold, weigh and note the weight.

Break the lead sulphide formed, examine, and note the appearance.

Report.

Pound the lead sulphide, and pass the whole of it

[1] For this and following experiment use flowers of sulphur.

through a 40-mesh sieve, for use in the following exercise.

EXERCISE NO. 62.

To reduce the prepared Lead Sulphide in an Iron Crucible.

Reaction: $PbS + Fe = Pb + FeS$
or $3PbS + 3Fe = 2Pb + (PbS + Fe_2S + FeS)$

Weigh the pounded sulphide from the previous exercise and note the weight.

Put the weighed sample in a warm cast-iron crucible and place in a pot furnace which is at a red heat.

Cover with about 3 grammes of soda.

Place a fire-clay lid on the crucible.

Gradually raise the temperature until the sulphide is fused, and, when the reaction appears to be complete, place a cover on the crucible, and raise the temperature of the furnace.

When all action appears to have ceased, scrape down the inside of the crucible with the flattened end of an iron rod, the flattened end of which has been heated.

Replace the lid of the crucible.

Maintain the furnace at a good heat.

After about five minutes withdraw the crucible and quickly pour the contents into a deep mould.

When cool, set the ingot on one of its sides and hammer off the slag. Be careful not to hammer the slag into the soft lead.

Weigh the cleaned lead and note the weight.

Note the percentage loss.

Test the malleability of the lead. If it is not soft and quite malleable, melt it again along with about 6 grammes of soda, pour, and, when cool, reweigh and note the weight.

Note the percentage loss.

Report.

NOTE.—The prepared lead sulphide may be taken as representing an artificial galena. In the next exercise natural galena—the essential constituent of which is PbS—is to be used.

EXERCISE NO. 63.

To reduce Pure Galena with Iron.

Pick out some clean crystals of pure galena and powder them till fine enough to pass through a 40-mesh sieve.

Weigh off 25 grammes of the crushed galena and place in a suitable fire-clay crucible.

Set three or four stout iron nails (head upwards) in the powdered galena.

Place in a pot furnace which is at a dull red heat.

Slowly raise the temperature until the sulphide is fused.

When the reaction appears to be complete, withdraw the nails one by one from the crucible, taking care to shake off any newly reduced lead into the crucible.

When all the nails have been withdrawn, place a cover on the crucible, and raise the temperature of the furnace.

After about five minutes, withdraw the crucible and pour the contents into a deep mould.

When cool, separate the slag as directed in the previous exercise.

Weigh the lead and note the weight.

Test the malleability of the lead and refine if necessary.

Compare the percentage of lead obtained with the percentage obtainable.

Report.

EXERCISE NO. 64.

To reduce Impure Galena with Iron—using a Flux for Siliceous Gangue.

Pick out a sample of galena with gangue and crush about 80 grammes till fine enough to pass through a 40-mesh sieve. Mix the poundings thoroughly.

Weigh off 25 grammes of the crushed and mixed sample.

Mix intimately with 20 grammes of soda[1] and 2 grammes of argol.

Place in a suitable crucible and cover with about 5 grammes of soda.

Stick in iron nails, and proceed as in Exercise No. 63.

Weigh the lead obtained.

Test the malleability of the lead and refine if necessary; or repeat the experiment, taking care to produce the maximum amount of pure lead.

Calculate the percentage of lead obtained.

Report.

[1] Sodium carbonate.

EXERCISE NO. 65.

To reduce Impure Galena with Iron—using a Flux for Calcite, etc.

Weigh off 25 grammes of the crushed and mixed sulphide and gangue.

Mix intimately with 15 grammes of soda, 2 grammes of argol, and 4 grammes of borax.

Place in a suitable crucible and cover with about 3 grammes of borax.

Stick in iron nails and proceed as in the previous exercises.

Weigh the lead obtained.

Test for malleability.

Refine or repeat if required.

Calculate the percentage of lead obtained and compare with the result of Exercise No. 64.

Report.

EXERCISE NO. 66.

To reduce Antimony Sulphide with Iron—First Experiment.

Reaction: $Sb_2S_3 + 3Fe = 2Sb + 3FeS$.

Select a sample of antimony sulphide and powder about 100 grammes so that all will pass through a 40-mesh sieve.

Mix the poundings thoroughly.

Weigh off 25 grammes of the pounded and mixed sample.

Mix with 20 grammes of soda.

Put the mixture in a suitable fire-clay crucible.

Cover with about 5 grammes of soda.[1]

Set two or three iron nails in the mixture.

Place in a pot furnace which is at a dull red heat.

Raise the temperature gently, and be careful not to overheat and cause volatilization.

After the contents of the crucible have become fused, allow to remain at a steady temperature for a few minutes so as to allow time for the separation of the reduced metal from the slag, then sharply raise the temperature, and allow to remain for a few minutes more.

Quickly pour into a warm mould.

When cold, break off the slag. This must be carefully done as antimony is a brittle metal.

Weigh the metal obtained.

Calculate the quantity of antimony obtainable.

Compare with the weight obtained.

Report.

EXERCISE NO. 67.

To reduce Antimony Sulphide with Iron—Second Experiment.

Calculate the weight of iron required to reduce 25 grammes of antimony sulphide on the basis of the equation—

$$Sb_2S_3 + 3Fe = 2Sb + 3FeS$$

Weigh off that quantity of mild steel filings or of clean sheet-iron clippings.

Weigh off 25 grammes of powdered antimony sulphide.

Mix the weighed quantities thoroughly along with 20 grammes of soda.

[1] Common salt is often useful as a covering material.

Put the mixture in a suitable fire-clay crucible.

Cover with 5 grammes of soda.

Do not set in any iron nails, but otherwise proceed as in the previous exercise.

Calculate, compare, and report.

EXERCISE NO. 68.

To reduce Antimony Sulphide with Pig Iron.

Weigh off 25 grammes of the finely-pounded antimony sulphide, 12 grammes of fine-pounded white pig iron, and 20 grammes of soda.

Mix thoroughly and put the mixture in a suitable fire-clay crucible.

Proceed as in the previous experiment.

Calculate, compare, and report.

EXERCISE NO. 69.

To reduce Antimony and Lead Sulphides—First Experiment.

Calculate the weight of iron required to reduce 15 grammes of antimony sulphide and 15 grammes of lead sulphide.

Weigh off that weight of white pig iron, 20 grammes of soda, 15 grammes of antimony sulphide, and 15 grammes of lead sulphide—all in fine powder.

Mix thoroughly and put the mixture in a suitable fire-clay crucible.

Proceed as in previous experiments until the mixture is well fused and all reaction is over.

Allow the crucible and contents to remain in the furnace till separation is judged to be complete.

Withdraw the crucible from the furnace and allow the contents to cool.

When the contents have become cold, break the crucible, carefully separate the lead, antimony, and slag. Preserve the slag.

Weigh the lead and the antimony and note the weight of each.

Calculate, compare, and report.

EXERCISE NO. 70.

To reduce Antimony and Lead Sulphides—Second Experiment.

Put into a suitable fire-clay crucible 5 grammes of finely pounded galena, the lead and the antimony from the previous experiment, and also the slag (finely pounded) from the previous exercise.

Proceed as in the previous experiment.

Compare the weight of each of the metals obtained in this with that obtained in the previous exercise.

Report.

EXERCISE NO. 71.

To test the reaction between lead sulphide (PbS) and lead oxide (PbO), represented by the equation—

$$PbS + 2PbO = 3Pb + SO_2$$

Calculate the amount of litharge (PbO) required for 11 grammes of lead sulphide (PbS).

Weigh off the required quantity of each. (Both should be in a fine state of division.)

Intimately mix the weighed quantity of each.

Place in a "pure carbon" crucible of suitable size.

Put in a pot furnace which is at a dull red heat.[1]

Gradually raise the temperature.

When action appears to have ceased, stir the charge with a charred stick.

Place a lid on the crucible.

Maintain at a higher temperature for five minutes or so.

Pour the metal into a warm mould.

When cold, brush off any slag which may be on the metal.

Weigh and note the weight.

Test the quality of the lead obtained.

Calculate the amount of lead obtainable from the substances used, and compare with the weight obtained.

Report.

EXERCISE NO. 72.

To test the reaction between lead sulphide and lead sulphate, represented by the equation—

$$2PbS + PbSO_4 = 3Pb + 2SO_2$$

Calculate the amount of lead sulphate ($PbSO_4$) required for 12 grammes of lead sulphide (PbS).

[1] If a "pure carbon" crucible is kept embedded in plenty of incandescent coke, and packed so that air does not reach it while in the hot furnace, the crucible will withstand repeated reheatings.

Weigh off the required quantity of each. (Both should be in a fine state of division.)

Intimately mix the weighed quantity of each.

Charge into a pure carbon crucible, and proceed as in the previous exercise.

Test the quality of the lead obtained.

Calculate and report.

EXERCISE NO. 73.

To partly roast Galena so as to obtain a Mixture of PbS with $PbSO_4$ and some PbO, and by Fusion to produce Lead from the Mixture.

Weigh off 15 grammes of fine-pounded pure galena.

Place the weighed sample on a large scorifier.

Heat in a muffle furnace until it is evident that a reaction has begun.

Quickly lower the temperature of the muffle.

Stir the charge with a stout iron wire and be careful to prevent clotting.

When it is judged that about half of the charge has become changed, withdraw the scorifier.

As soon as the charge can be handled, transfer it to a suitable pure carbon crucible.

Proceed as in the previous experiments.

Weigh the lead and note the weight.

Compare the weight obtained with the theoretical quantity obtainable.

Test the quality of the lead.

Report.

EXERCISE NO. 74.

To make copper sulphide according to the equation—

$$Cu_2 + S = Cu_2S$$

Calculate the amount of sulphur required to combine with 20 grammes of copper.

Weigh off that quantity of flowers of sulphur.

Weigh off 20 grammes of copper filings.[1]

Mix the weighed quantities.

Put a layer of flowers of sulphur in the bottom of a suitable sized clay crucible.

Put the weighed mixture in the crucible.

Cover with a thin layer of sulphur.

Place the charged crucible in a pot furnace which is at a dull red heat, cover with a lid, and gradually raise the temperature.

When the reaction appears to be complete and the charge is well fused, drop two pieces of roll sulphur (each larger than a nut), and stir well through the mass.

Allow to remain about two minutes with the lid on the crucible.

Withdraw the pot and pour quickly into a warm half-round mould.

Cover with a hot crucible lid.

[1] The copper filings may, if necessary, be cleaned from iron by means of a magnet. Small clippings of thin sheet copper or the finest portions of granulated copper may be used instead of filings.

When the sulphide is cold, weigh it, note the weight; break it, and examine the fracture.

Report.

Pound the prepared sulphide so that the whole of it will pass through a 40-mesh sieve. Carefully keep the powdered sample for further use.

EXERCISES IN FORMING, REDUCING, AND FUSING SILICATES.

EXERCISE NO. 75.

To make Lead Mono-silicate ($2PbO.SiO_2$).

Place a mixture of 37 grammes of finely powdered litharge and 5 grammes of white sand in a suitable crucible.

Place the crucible and charge in a pot furnace which is at a dull red heat. Gently raise the temperature until the charge has become thoroughly melted.

Pour the silicate into a warm half-round mould and quickly cover it with a hot crucible lid.

When the silicate has cooled, weigh it and note the weight.

Report.

Powder the product, pass it all through a 20-mesh sieve, and preserve it for further use.

Prepare an additional sample of lead mono-silicate, using the quantities specified above.

Compare the weight of product obtained in each case.

Report.

Powder the product, pass it all through a 20-mesh sieve, and preserve it for further use.

Mix thoroughly the powdered silicates from the previous experiment for use in the following exercises.

EXERCISE NO. 76.

Reduction of Lead Silicate by Carbon.

Weigh off 20 grammes of powdered mixed silicates, 1·2 grammes of charcoal, and 10 grammes of dry sodic carbonate.

Mix thoroughly and put in a suitable crucible.

Place in a furnace which is at a dull red heat.

Gradually raise the temperature till the mixture becomes thoroughly fused.

Keep fused for about seven minutes.

Stir and pour quickly into a warm mould.

Cover with a hot lid.

When cold, recover the lead button and pellets, searching carefully for the latter.

Weigh the lead produced and note the weight. Keep the lead in an envelope.

Weigh the slag produced and note the weight. Preserve the slag for use in the next exercise.

Report.

EXERCISE NO. 77.

Reduction of Lead from partly reduced Silicate by means of Iron.

Powder the slag from the previous exercise and pass it through a 20-mesh sieve.

Weigh the pounded slag and note the weight.

Mix with 4 grammes of grey pig-iron drillings.

Proceed to reduce as directed for the previous experiment.

Weigh the lead obtained and note the result.

Report.

EXERCISE NO. 78.

Reduction of Lead Silicate by Iron.

Weigh off 20 grammes of the powdered mixed silicates, 4·3 grammes of wrought-iron or mild steel filings, or fine turnings, and 20 grammes of dry sodic carbonate.

Mix thoroughly and proceed with the reduction.

Weigh, and note the weight of the lead obtained.

Report.

EXERCISE NO. 79.

Reduction of Lead Silicate by Carbon and Iron.

Weigh off 20 grammes of the powdered mixed silicates, 4 grammes of grey pig-iron filings or drillings, and 10 grammes of dry sodic carbonate.

Mix thoroughly and proceed with the reduction.

Weigh, and note the weight of the lead obtained.

Report.

Having gained experience in the production of lead silicates and estimation of the metal, the student should now proceed to prepare new samples and ascertain the relative fusing points of silicates.

EXERCISE NO. 80.

To prepare Lead Mono-silicate ($2PbO . SiO_2$).

Calculate the amount of white sand required to be added to 26 grammes of litharge so as to form $2PbO . SiO_2$.

See example of calculation on p. 125.

Report.

Weigh off 26 grammes of finely powdered litharge and the calculated amount of white sand.

Prepare a sample of the silicate.

Weigh the product and note the weight.

Report.

Pound the silicate, passing the whole of it through a 20-mesh sieve.

Carefully preserve the product.

EXERCISE NO. 81.

To prepare Lead Bi-silicate (PbO, SiO_2).

Calculate the amount of white sand required to be added to 26 grammes of litharge so as to form PbO, SiO_2.

Report.

Weigh off 26 grammes of finely powdered litharge and the calculated amount of white sand.

Prepare a sample of the silicate.

Weigh the product and note the weight.

Report.

Pound the silicate, passing the whole of it through a 20-mesh sieve, and carefully preserve the product.

EXERCISE NO. 82.

To test the relative Fusibilities of Lead Mono-silicate and Lead Bi-silicate — First Estimation.

Weigh off 5 grammes of the finely pounded lead mono-silicate.

Put the weighed sample on a small scorifier with a distinctive mark.

Weigh off 5 grammes of the finely pounded lead bi-silicate.

Put the weighed sample on a small scorifier with a distinctive mark.

Set the two charged scorifiers in the centre of a newly-lit muffle and note which of the two samples melts first.

When well melted pour off each into a mould.

Report.

EXERCISE NO. 83.

To test the Relative Fusibilities of Lead Mono-silicate and Lead Bi-silicate—Second Estimation.

Weigh off 5 grammes of the finely pounded lead bi-silicate.

Put the weighed sample on a small scorifier with a destinctive mark.

Weigh off 5 grammes of the finely pounded lead mono-silicate.

Put the weighed sample on a small scorifier with a distinctive mark.

Set the two charged scorifiers in the centre of a red-hot muffle and note which of the two samples melts first.

When well melted pour off each into a mould.

Report.

EXERCISE NO. 84.

Reduction of Lead from Mono-silicate.

Weigh off the remainder of the pounded lead mono-silicate, and note the weight.

Extract as much as possible of the lead from the weighed sample.

Pour, and, when cold, weigh the lead.

Note the weight of lead obtained, calculate the percentage yield, and compare with the theoretical quantity obtainable.

Test the qualities of the lead.

Report.

EXERCISE NO. 85.

Reduction of Lead from Bi-silicate.

Weigh off the remainder of the pounded lead bi-silicate, and note the weight.

Extract as much as possible of the lead from the weighed sample.

Pour, and, when cold, weigh the lead.

Note the weight of lead obtained, calculate the percentage yield, and compare with the theoretical quantity obtainable.

Test the qualities of the lead.

Report.

EXERCISE NO. 86.

To make Copper Silicate.

Tare a fire-clay disc (which has previously been heated in a muffle and allowed to cool in a desiccator) and note the weight. Arrange the record as shown in Exercise No. 37.

Weigh off 24 grammes of black copper oxide and 4·6 grammes of fine white sand.[1]

Mix the weighed quantities intimately and place on the tared disc.

Put the disc and contents in a hot muffle, and allow to remain there at the highest attainable temperature for more than half an hour. The charge should frit but not melt.

Withdraw the disc and silicate, and, when cool enough to be handled, place in a desiccator.

When cold, weigh and note the weight.

Calculate the loss of weight.

Calculate how much weight should have been lost on the basis of the equation—

$$4CuO + SiO_2 = 2Cu_2O \,.\, SiO_2 + O_2$$

Report.

[1] The quantity of sand recommended has been calculated so that $4CuO = SiO_2$. The student should verify the calculation before continuing the exercise.

EXERCISE NO. 87.

To fuse Copper Silicate.

Transfer the fritted product from the previous exercise to a suitable crucible.

Set the crucible and contents in a hot pot furnace.

Gradually raise the temperature.

When the silicate has become well fused, pour it into a warm mould and cover with a warm lid.

When cool, weigh and note the weight.

State the percentage loss on fusing and pouring.

Report.

EXERCISE NO. 88.

To make Copper Silicate Direct.

Weigh off 24 grammes of black copper oxide (CuO) and 4·6 grammes of fine white sand.

Mix the weighed quantities thoroughly and transfer to a suitable crucible.

Place the crucible and contents in a hot pot furnace which is at a dull red heat.

Gradually raise the temperature.

When the silicate has become well fused, pour it into a warm mould and cover with a warm lid.

When cool, weigh and note the weight.

State the percentage loss and compare with previous experiment.

Report.

Pound the products from two previous experiments, passing the whole through a 20-mesh sieve. Mix

thoroughly, and carefully preserve the silicate for further use.

EXERCISE NO. 89.

To reduce Copper Silicate with Iron and Carbon.

Weigh off 21 grammes of the finely pounded copper silicate and 7 grammes of pig-iron drillings.

Mix intimately and transfer to a suitable crucible.

Place the charged crucible in a pot furnace which is at a dull red heat.

Gradually raise the temperature.

When the charge has become well fused, allow to remain in the furnace for about five minutes, maintaining a high temperature meanwhile.

Withdraw the crucible and tap it so as to help the heavier portion to settle towards the bottom of the crucible.

Allow to cool.

When cool, break the crucible, extract the metal (button with perhaps beads as well) from the slag, and weigh the metal.

Calculate the theoretical quantity obtainable and compare with the weight of copper reduced and recovered.

Preserve the ferrous silicate for further use.

Report.

EXERCISES WITH SILICATES AND SULPHIDES.

EXERCISE NO. 90.

To convert Cuprous Silicate into Cuprous Sulphide.

The reaction is generally represented by the equation—

$$2Cu_2O \,.\, SiO_2 + 2FeS = 2Cu_2S + 2FeO \,.\, SiO_2$$

Pound a sample of ferrous sulphide (FeS) and pass the poundings through a 20-mesh sieve.

Weigh off 13 grammes of the finely pounded ferrous sulphide.

Weigh off 25 grammes of the finely pounded cuprous silicate from Exercises 87 and 88.

Mix the two weighed samples intimately.

Place the mixture in a suitable crucible and set in a pot furnace which is at a red heat.

Gradually raise the temperature.

Maintain at a high temperature for about ten minutes after the mixture has become well fused.

Withdraw the crucible, tap it so as to hasten the separation and settlement of the contents, and allow to cool.

When cool, break the crucible.

Weigh the product and note the weight.

Separate the layers; weigh and note the weight of each.

Examine the products.

Preserve the regulus for further use.

Compare the weight of regulus obtained with the theoretical weight obtainable.

Calculate the percentage loss.

Report.

If the experiment is not satisfactory, place the products of the experiment in a crucible and heat to bright redness. Add 12 grammes of dry sodic carbonate. Melt thoroughly, tap so as to hasten separation, and set aside to cool.

When the contents have become cold, break the crucible, separate the regulus, weigh it and note the weight.

Compare the weight of regulus obtained with the calculated percentage loss.

Report.

EXERCISE NO. 91.

To test the Effect of Iron on melted Copper Sulphide.

Weigh off 7 grammes of the powdered copper sulphide prepared in Exercises 87 and 88.

Mix it with 4 grammes[1] of finely pounded white pig iron.

[1] This is less than the quantity required to satisfy the copper.

Melt the mixture in a clay crucible.
When thoroughly melted pour.
Weigh and examine the product.
Report.

EXERCISE NO. 92.

To form and fuse Ferrous Silicate— First Experiment.

Weigh off 10 grammes of pig-iron drillings, or, still better, clean wrought-iron filings.

Roast the weighed sample in a hot muffle till the iron has become well oxidized.

Withdraw the roasted sample and sprinkle over it 6 grammes of sand.

Replace in muffle and allow to remain for fifteen minutes.

Note the effect.

Transfer the oxide and sand to a suitable crucible and proceed to melt in a pot furnace.

When fused, pour into a warm half-round mould and cover with a hot lid.

Weigh the product.

Report.

Pound and pass the poundings through a 40-mesh sieve.

Keep the poundings for further use.

EXERCISE NO. 93.

To form and fuse Ferrous Silicate— Second Experiment.

Weigh off 3 grammes of ferric oxide (powdered red hematite), 3 grammes of charcoal, and 5 grammes of fine white sand.

Mix intimately and place in a suitable crucible.

Place in a pot furnace which is at a dull red heat.

Slowly raise the temperature and note the condition of the furnace when fusion begins.

Allow to remain fused for about ten minutes.

Raise the furnace temperature to whiteness.

Take out crucible and pour the contents into a warm mould.

Cover with a warm lid and allow to cool slowly.

When cold, examine and weigh.

Report.

EXERCISE NO. 94.

Experiment on Fusibility of Slag.

Weigh off 15 grammes of kaolin and transfer to a crucible.

Weigh off 15 grammes of dry lime and transfer to a crucible.

Place the crucibles in a hot pot furnace and allow to remain at the highest temperature of the furnace for thirty minutes.

Withdraw the crucibles, and, when they are cold, examine and note the results.

Intimately mix the contents of the two crucibles and transfer the mixture to one of them.

Place the charged crucible in a hot pot furnace, and allow to remain at the highest temperature until the mixture has melted.

Pour into a warm mould and cover with a hot lid.

Allow to cool.

Weigh the product.

Report.

Testing Fire-clay.

EXERCISE NO. 95.

Preparation of Small Fire-clay Crucibles.

Pound a sample[1] (say 4 or 5 ozs.) of fire-clay so that it can pass through a 60-mesh sieve.

Put about one-third of the sample in a crucible and heat strongly in a hot furnace for about twenty minutes.

Allow to cool.

Mix together about 1 oz. of the raw fire-clay with one-third its weight of the burnt fire-clay in a large mortar and cautiously add water.

Knead thoroughly, adding more and more water until the mass is quite plastic.

If convenient, allow the kneaded mass to remain (in a place which is not too dry) in the moist condition for a week or so.

The portion which is to be made into crucibles

[1] The sample should be so selected as to fairly represent the stock of fire-clay.

should not be too wet when moulded, but be just able to take shape and to hold well together when pressed.

Clean the small crucible mould and plugs (Fig. 22), and smear them well with oil.

Fill the mould with some of the moist mixture.

Work the wired plug with a firm, rotary, pressing motion down into the clay. The excess clay will be squeezed out through the oblique slot.

Withdraw the plug.

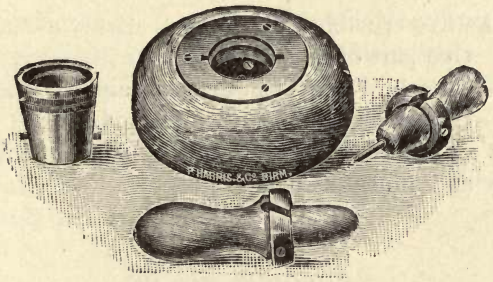

FIG. 22.

Put a pellet of the moist mixture at the hole in the bottom of the "pot," and press it well down with the finishing plug.

Dress the edge of the crucible with a sharp knife.

Carefully push the newly formed crucible out of the mould.

Make six or seven more crucibles as above.

Put a distinctive mark on crucibles of this lot.

Set them where they will dry gently.

When dry, place them in a cold muffle (the gas under which has just been lit), and allow them to remain there for an hour after the muffle has attained a full red heat.

Turn off the gas and allow the crucibles to remain in the muffle until cooled.

Make five crucibles, using—
One part of raw clay,
One-third part of burnt clay, and
5 per cent. of clean white sand.
Put a distinctive mark on each crucible of this lot.
Dry and anneal as before.

Make five crucibles, using—
One part of raw clay,
One-third part of burnt clay, and
10 per cent. of clean white sand.
Put a distinctive mark on each crucible of this lot.
Dry and anneal as before.

EXERCISE NO. 96.

To test the Corrosive Action of Oxides on the Fire-clay.

Place in one crucible of each lot 10 grammes of litharge.

Place each on a roasting dish in a *hot* muffle, and allow to remain for half an hour.

Take out and allow to cool.

Examine and report.

Remelt and pour.

Place in one crucible of each lot 10 grammes of granulated copper and 3·3 grammes of borax.

Place the crucible inside of another, put on a cover,

and heat strongly in a pot furnace for about half an hour.

Make sure that the metal is all melted.

Take out and examine.

Note results and report.

EXERCISE NO. 97.

To test Fire-clay under Oxidizing Conditions.

Make a plastic mass of the fire-clay and water.

Knead well, and allow to stand for a week.

Make from the moist mass four pyramids, each about 1½ inch high × 1 inch at base.

Form sharp edges on these pyramids.

Make four stands for these pyramids, and set the pyramids on the stands (see Fig. 23).

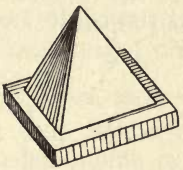

FIG. 23.—Fire-clay pyramid.

Allow to dry gently.

Place them in a hot muffle to which air has free access.

Allow to remain for an hour at the highest temperature of the muffle.

With the aid of a lens examine the edges for any sign of fusion.

Note the results and report.

EXERCISE NO. 98.

To test Fire-clay under Reducing Conditions.

Make a plastic mass of the fire-clay and water.

Knead well, and allow to stand for a week.

Make from the moist mass four pyramids, each about $1\frac{1}{2}$ inch high × 1 inch at base.

Form sharp edges on these pyramids.

Gently dry these pyramids.

Pack them in charcoal in a crucible, place in a pot furnace, and allow to remain for an hour at a high temperature.

Withdraw crucible and contents, and, when cool, examine with the aid of a lens.

Note the results and report.

Repack, replace, and reheat for an hour at the highest temperature of the furnace.

Examine again and report.

EXERCISE NO. 99.

To test the Effect of Impurities on Fire-clay: Preparation of Samples.

Mix with portions of the moistened and tempered clay—

These to be in fine powder.

- (*a*) 0·5 per cent. of sodic carbonate.
- (*b*) 3·0 per cent. of ferric oxide.
- (*c*) 5·0 per cent. of sand.
- (*d*) 2·0 per cent of lime.

Make four pyramids from each mixture.

Mark each batch distinctly.
Dry gently.

EXERCISE NO. 100.

To test the Effect of Impurities on Fire-clay, under Reducing Conditions.

Pack the prepared pyramids in charcoal in crucibles (two in each crucible) and place in a hot pot furnace.

At the end of an hour take out the crucibles and allow to cool.

Examine as before.
Note results in tabular form.
Report.

EXERCISE NO. 101.

To test the Effect of Impurities on Fire-clay, under Oxidizing Conditions.

Set two pyramids from each batch on a suitable fire-clay slab.

Place them in a hot muffle to which air has free access.

Allow them to remain for an hour at the highest temperature of the muffle.

When cool, examine.
Note the results in tabular form.
Report.

Experiments with Pyrites.

EXERCISE NO. 102.

To prepare an Artificial Ore (Copper and Iron Sulphides with Silica) for Experimental Purposes.

Pound a sample of iron sulphide (Fe_xS_y) so that the whole of it will pass through a 40-mesh sieve.

Weigh off 17 grammes of the iron sulphide and 13 grammes of the previously prepared copper sulphide.

Melt the mixture in a clay crucible.

When melted, add 5 grammes of white sand, stir the mixture well, keep in a hot furnace for fifteen minutes, and pour into a warm mould.

When cool, weigh and note the weight.

Report.

The "ore" prepared in this experiment will be much richer than the ordinary pyrites, but should prove more convenient for the purpose in view. Pound the ore so that it will all pass through a 60-mesh sieve. Keep the pounded sample for further use.

EXERCISE NO. 103.

To ascertain the Effect of roasting Copper Pyrites in Contact with Salt.

Weigh off 8 grammes of finely-pounded pyrites containing about 4 per cent of copper.

Place on a disc, or on a scorifier, and set in a hot muffle.

When the charge has become hot, lower the temperature of the muffle and continue to roast at a low temperature for an hour or more.

Withdraw the disc, or scorifier, and allow the charge to cool.

Transfer the roasted product to a small beaker (Fig. 24).

Add about 100 c.c. of water.

Boil.

FIG. 24.—Beaker.

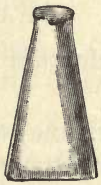

FIG. 25.—Conical flask.

Filter into a small conical flask (Fig. 25) and wash the solid residue several times with water.

Preserve the washed solid for further use, if required.

To separate metallic copper from the filtrate in the conical flask, acidify the solution by adding about 10 c.c. of hydrochloric acid, then add cautiously about $2\frac{1}{2}$ grammes of zinc dust.

Boil.

Filter the precipitated copper.

Wash several times with dilute acidulated water and, afterwards, about four times with water.

With a wash-bottle having a fine jet wash the copper into a tared porcelain basin (Fig. 26), and evaporate the water.[1]

[1] As an alternative, allow the copper to settle in the basin and pour

When dry, allow to cool in a desiccator.
Weigh the basin and the copper.
Calculate the percentage of copper obtained.
Report.

FIG. 26.—Basin.

EXERCISE NO. 104.

Examination of the Residue for Presence of Copper.

Transfer the solid residue to a conical flask and digest it in strong hydrochloric acid to which a few crystals of potassic chlorate have been added. Dilute with 100 c.c. of water. Filter into a conical flask and wash the solids well with water. To the filtrate cautiously add zinc dust, and, if copper is precipitated, collect it and ascertain the amount.

Report.

EXERCISE NO. 105.

To ascertain if Copper can be Extracted by Water from the Unroasted Pyrites.

Weigh off 5 grammes of the finely-pounded sample.
Transfer to a conical flask.

off as much as possible of the water without losing any of the copper. Flood the copper with alcohol, agitate the contents of the basin, allow the copper to subside, and pour off as before. Add more alcohol and repeat the decantation. Evaporate the remaining alcohol, allow to cool in a desiccator, ascertain the weight of the copper, calculate and report.

Boil.
Filter and wash.
Acidify the filtrate by adding hydrochloric acid.
Cautiously add zinc dust.
Collect the copper, if any, and estimate the amount.
Compare with the result of the previous experiment.
Report.

EXERCISE NO. 106.

To ascertain the Effect of roasting Copper Pyrites in Contact with Ferric Chloride.

Weigh off 8 grammes of finely-pounded pyrites.
Weigh off about 4 grammes of ferric chloride.
Mix intimately the pyrites and the ferric chloride.
Roast on a disc as in Exercise No. 103.
Leach the roasted pyrites and proceed as in No. 103, but use fine mild steel drillings to precipitate the copper.
Calculate the percentage of copper obtained.
Report.

EXERCISE NO. 107.

To extract and refine the Copper contained in Pyrites.

First Stage: Fusion of the Ore to obtain a Regulus.

Weigh off 16 grammes of the finely pounded artificial pyrites, also 16 grammes of fluor spar, 15 grammes of lime, 15 grammes of powdered glass, 12 grammes of borax, and 4 grammes of nitre.

Mix all these thoroughly and transfer to a large copper assay crucible (Fig. 27).

Place in a hot pot furnace and gradually raise the temperature.

When action has subsided and the mixture has melted, rotate the pot so as to gather the contents together, heat for two or three minutes more, and pour into a warm mould.

Allow the slag to become solid.

Cautiously plunge the mass into water in a mortar.

Withdraw the mass while it still retains heat enough to evaporate the water from its surface.

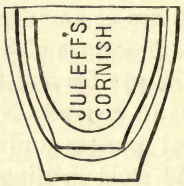

FIG. 27.—Copper assay crucible, in section.

Crumble the slag and separate the regulus from it. Search in the slag for beads of regulus. Add these, if any, to the regulus.

Examine the regulus to see if the charge has been properly proportioned.

If the regulus is round and hard, and the fracture is of a dark bluish-grey colour, it is "too fine," and a fresh charge containing less nitre should be added. If the regulus is flat and the fracture resembles iron sulphide, it is "too coarse," and a fresh charge with more nitre should be used. But if the regulus is reddish-brown and easily broken, it should be powdered and the experiment proceeded with.

Second Stage: Roasting the Regulus.

Place the powdered regulus in a scorifier in a muffle which is at a dull red heat and gradually raise the temperature.

Stir the charge constantly until danger of clotting is over.

When the sulphur has nearly all been burned off withdraw the scorifier, and, when cold enough, tip the charge into a mortar containing about 1 gramme of powdered anthracite, and mix well with the pestle.

Replace the charge on the scorifier, and continue the roasting for about fifteen minutes more,

Third Stage: Production of Coarse Copper.

Mix the roasted regulus with 17 grammes of soda, 10 grammes of argol, and 3 grammes of borax.

Put the mixture in a small copper assay crucible and place in a hot pot furnace.

Keep fused for ten minutes and pour.

When the mass has solidified, plunge it in water, and, when it is quite cold, separate the copper from the slag.

Preserve the slag for further treatment.

Fourth Stage: Production of Fine Copper.

Put the crucible which was used in the previous stage in a hot pot furnace which is well filled with coke, and when it is at a bright red heat place the coarse metal in it. Place the covering bricks on the furnace, leaving a little space over the pot through which to

watch the clearing of the button and the appearance of the bright centre spot known as the eye.

Shoot from a scoop (Fig. 28) 10 grammes of refining flux (see p. 95).

Fig. 28.—Scoop.

Cover again and allow to remain at a full red heat for about a minute after all the charge has become fluid.

Pour, and, when cool, quench as before, and separate the metal from the slag.

Preserve the slag from further treatment.

If the assay has been successful the mass will be orange coloured on the surface and the metal will possess the malleability and toughness of good copper. If it has a coarse, dull, appearance, the fourth stage must be gone over again. If the film on the button is of a red or purple hue, it is an indication that the assay is overdone and in practice it would be considered worthless.

Fifth Stage: Recovery of Copper from the Slags.

Pound the slags together, and mix them thoroughly with 5 grammes of argol, 2 grammes of soda, and half a gramme of charcoal.

Place the mixture in a red-hot crucible in a hot furnace, and, when well fused, pour.

Separate the metal from the slag.

Add the metal to that which was previously obtained.

Calculate the percentage of copper obtained and report.

In conducting an assay on the lines of this exercise, the supposed richness of the ore must be taken into account and a suitable quantity of ore weighed out. The amount of ore taken is generally between 13 grammes and 26 grammes, and the quantity of flux, etc., is varied to suit.

The refining flux used in stage four is made by measuring three parts of nitre, two and a half of argol, and one of salt, mixing intimately, putting in a capacious crucible, and, in a draught chamber, stirring with a red-hot iron rod until reaction ceases.

EXERCISE NO. 108.

To roast Copper and Silver Sulphides and Extract the Metals.

Add to 9 grammes of finely pounded copper pyrites, 1 gramme of silver sulphide, and about 10 grammes of salt.

Make an intimate mixture of the three.

Roast on a scorifier in a hot muffle for an hour or more.

When the roasted mixture has cooled brush it into a beaker.

Add about 150 c.c. of water and two or three drops of acid.

Boil.

Filter and wash well, collecting the filtrate in a

conical flask. Cautiously add to the filtrate a 2 per cent. solution of potassic iodide, and stir after each addition. Continue the additions till the solution shows a colour due to presence of free iodine.

Filter, and keep the filtrate and washings.

Wash the precipitate and dry it.

Wrap the precipitate in about 6 grammes of lead and proceed to cupel.

Weigh the silver bead and compare with the quantity taken for the experiment.

Report.

To the filtrate add a little acid and about 5 grammes of mild steel drillings or filings.

Set aside, and allow time for the precipitation of the copper.

Collect the copper, wash and dry it, and preserve it.

EXERCISE NO. 109.

To roast mixed Sulphides, extract and collect the Metals.

Weigh off 9 grammes of finely pounded copper pyrites, 1 gramme of silver sulphide, and 7 grammes of salt.

Mix intimately, and roast the charge as before.

When cool, brush the roasted mixture into a beaker.

Add about 80 c.c. of a 2½ per cent. solution of sodic hyposulphite (thio-sulphate) to dissolve the silver. (It may be advisable to repeat the leaching in a fresh solution of hyposulphite.)

Boil.

Filter the dissolved silver into a conical flask and precipitate it by the addition of ammonic sulphide.

Collect, wash, and dry the precipitate.

Heat the sulphide on a clean scorifier so as to burn off the excess sulphur.

Add 5 grammes of lead and roast further.

Separate the slag from the metallic portion and cupel the latter.

Weigh the silver bead and compare with the result of the previous experiment.

Report.

Recover and preserve the copper.

EXPERIMENTS WITH PRECIOUS METALS.

EXERCISE NO. 110.

To prepare a Solution of Silver and precipitate it by means of another Metal.

Weigh off about 0·5 gramme of the pure silver from the previous operations and note the weight.

Dissolve it in a basin with the minimum amount of pure nitric acid and evaporate the excess acid.

Dissolve the silver nitrate in water.

Place a small strip of clean zinc in the solution.

Collect the precipitated silver, wash it in a basin, dry and weigh it.

Note the weight of silver obtained.

Report.

EXERCISE NO. 111.

To recover Copper and Silver from Solutions.

Prepare an alloy of 7 grammes of copper and about 0·5 gramme of silver.

Note the weight of silver used.

Cast into a thin cake.

In a beaker or basin dissolve the alloy in strong sulphuric acid, and boil off much of the excess.

Place a thin strip of copper weighing less than a gramme in the solution, and allow to stand for a time.

Filter off the insoluble portion (the precipitated silver), and wash it several times with acidulated water and many times with pure water.

Dry in a tared porcelain crucible.

Note the weight of silver obtained and preserve it for further use.

Add to the filtrate about 10 grammes of white pig-iron borings.

Dissolve the excess of iron.

Collect the residue of copper, silica, and graphite, and preserve it.

Report.

EXERCISE NO. 112.

To precipitate Gold from Solution.

Weigh about 0·3 gramme of gold, and dissolve it in 2 c.c. of hydrochloric acid and 0·5 c.c. of nitric acid in a porcelain basin.

Heat till the excess of acid has evaporated and a thick syrup has formed.

Dissolve in about 150 c.c. of water.

Warm the solution, and add a solution of ferrous sulphate to precipitate the gold.

Wash by repeated decantation.

Transfer to a suitable crucible; dry, and heat to redness.

Weigh the gold and report.

EXERCISE NO. 113.

Extraction of Precious Metals from Sand, in Imitation of Extraction from Crushed Quartz by Amalgamation.

Weigh off small quantities (say, about 0·3 gramme) of finely divided gold and of finely divided silver, and note the weight of each.

Mix them intimately with about 10 grammes of fine sand in a mortar.

Add about 8 grammes of mercury and rub the whole well with the pestle.

Place the mortar and contents under a water-tap, and regulate the flow of water so that the sand will be carried away.

Pour off the water from the mercury, etc.

Place a piece of chamois leather over a basin or a mortar, and pour the mercury, etc., on to it.

Squeeze as much as possible of the mercury through the pores of the leather.

Place the amalgam in a suitable retort or crucible and gently volatilize the mercury.

Weigh the metal.

Recover the precious metals from the other portion of the amalgam.

Report.

EXERCISE NO. 114.

"Parting" of the Gold from the Silver obtained in the Previous Experiment.

Into a parting flask or a 5-inch × $\frac{3}{4}$-inch test-tube put about 4 c.c. of pure water and 6 c.c. of strong pure nitric acid.

Drop the metallic portions into it.

Boil, and keep boiling gently for about ten minutes.

Decant the liquor from the metal into a beaker.

Run into the test-tube 5 c.c. of pure water and an equal quantity of strong pure nitric acid.

Boil for about seven minutes and decant again.

Half-fill the test-tube with distilled water.

Boil and decant.

Fill the test-tube with distilled water.

Place a "porous cup" over it, and turn the cup and tube over so that the test-tube will be uppermost.

Gradually raise the test-tube and allow the gold to settle in the cup.

Withdraw the test-tube and pour the water out of the cup.

Heat in a muffle till the cup is at a dull red heat.

Withdraw it, and, when cool, tilt the gold into a tared watch-glass.

Weigh it and note the weight.

Report.

NOTE.—The "parting" of gold and silver is best effected when the weight of silver in the bead is equal to about $2\frac{1}{2}$ times that of the gold.

EXERCISE NO. 115.

Extraction of Gold from Sand, in Imitation of the Chlorination Method.

Dissolve a weighed quantity of gold in *aqua regia* as before, precipitate with ferrous sulphate, dry the gold obtained, and mix it well with about 7 grammes of fine sand.

Brush the mixture into a small conical flask and more than cover it with water.

Pass in chlorine gas until the water is saturated.

Cork the flask[1] and allow to remain for a time.

Repeat the treatment with chlorine gas, cork the flask, and again allow to stand.

If the liquor is not green coloured pass in more chlorine.

[1] An indiarubber stopper must not be used.

Filter, and collect the filtrate in a small beaker.

Wash well.

Boil off the free chlorine.

To the filtrate add solution of ferrous sulphate.

Allow the finely divided gold precipitate to settle.

Wash a few times by decantation.

With a fine-jet wash-bottle wash the gold into a small crucible.

Dry and weigh.

Report.

EXERCISE NO. 116.

Extraction of Gold from Sand, in Imitation of the Cyanide Method.

Weigh off a small quantity of finely divided gold and note the weight.

Mix it in a mortar with about 10 grammes of fine sand.

More than cover the mixture with a 0·75 per cent. solution of potassic cyanide. *Remember that the cyanide is a deadly poison.*

Allow to stand for three days or so.

Filter.

Collect the solution and washings in a small conical flask.

Add excess of zinc dust to precipitate the gold from the solution, and allow the precipitate to settle.

Wash well by repeated decantation.

Collect the residue in a small tared crucible.

Dry and weigh the residue and note the weight.

Fold the residue in a piece of sheet lead.

Scorify and cupel.

Weigh the bead of refined gold.

Report.

MISCELLANEOUS EXERCISES.

ASSAYING OF ORES, ETC., CONTAINING PRECIOUS METALS.

The experimental errors in assaying ores, etc., containing precious metals arise from—

(*a*) Additions due to silver in the assay lead and in the lead oxide used.

(*b*) Losses due to absorption in the cupel, and, in extreme cases, to silver carried off in the fumes.

Blank assays are made to correct errors due to (*a*) and check cupellations are conducted to correct (*b*).

EXERCISE NO. 117.

Assay of Silver in a Stock of Red Lead.

Withdraw, with the usual sampling precautions, from the stock of red lead and weigh off 100 grammes.

Intimately mix it with the proper quantity of suitable flux and 4½ grammes of flour.

Transfer to a fire-clay or a pure carbon crucible.

Set in a pot furnace.

Carefully raise the temperature till the mixture has fused.

When well melted and action has ceased, pour.

Break away the slag.
Cupel the lead, clean and weigh the bead.
Report.

EXERCISE NO. 118.

Estimation of Silver in Assay Lead.

Weigh off 50 grammes of clippings from several of the pieces of the lead in stock.

Place the weighed clippings on a warm scorifier and add enough of a suitable flux.

When more than half the lead has become oxidized, withdraw the scorifier and pour.

Clean and cupel the lead.
Weigh and note the weight of the bead.
Report.

EXERCISE NO. 119.

Estimation of Silver in Assay Lead: Alternative Method for Duplicate Assay.

Weigh off 50 grammes of clippings from several of the pieces of the lead in stock.

Melt the weighed clippings in a clean crucible.
Pour, and, when cold, hammer into a cube.
Cupel.
Weigh and note the weight of the bead.
Report.

ASSAY OF SILVER ORES.

In ores the precious metals may occur in the "native" state finely disseminated through the masses, or the ore may contain small proportions of the rare metals in combination with haloids, or with sulphur; or the ores may be of considerable complexity, and the gangue may be either acid or basic.

Given a carefully selected sample fairly representing the average composition of the ore, and which has been finely crushed and mixed thoroughly, 55 grammes should be withdrawn and pulverized until as much as possible has passed through a 60-mesh sieve. The pounding sometimes has the effect of flattening the metallic particles so that they cannot be sifted. If so, that portion must be collected carefully, wrapped in a weighed quantity of assay lead, cupelled, and the percentage of metals ascertained.

When sulphides are present the ore must be scorified.

Roasting will be resorted to with advantage when much copper or much antimony is present, or when the ore contains even a small quantity of copper or zinc. But if mercury or tellurium be present the ore must not be roasted.

The principle underlying the ordinary method of assaying ores containing precious metals by treating in a crucible and then cupelling is easily understood.

The metals are either in the native state, or are reduced, and they are caused to combine with lead, which searches out and concentrates within itself the precious metals. But, as it would be difficult to get the lead into a sufficiently fine state of division, oxide of lead is used, and a sufficiency of carbon is added (where necessary, for occasionally there is enough, and more than enough, reducing material in the ore) to reduce enough lead to collect the precious metals. The rich lead is then cupelled.

The red lead which is used supplies (*a*) lead for collecting; (*b*) oxygen for acting on certain elements; (*c*) base for combining with siliceous gangue and certain metallic compounds.

Silver ores vary so widely in composition that no definite charge can be given for an ore of any class. The button of lead obtained should weigh about 28 grammes and should be of good quality. If on trial the button is found to be not nearly of the right weight a fresh assay should be started, and if the lead is hard the amount of reducing agent must be diminished and the quantity of red lead increased. In the event of antimony being present nitre must be added to the charge.

EXERCISE NO. 120.

Assay of Silver Ore with Siliceous Gangue.

Weigh out a charge consisting of

Ore	25 grammes
Red lead . . .	32 „
Sodic carbonate . .	30 „

	Charcoal . . .	1·5 grammes
or	Flour	3·0 „

Mix the materials thoroughly and charge them into a large fire-clay crucible.

Place in a furnace which is at a dull red heat, and *slowly* raise the temperature so that the charge decidedly frits before fusing.

Raise the temperature so as to bring the charge into suitable condition for pouring.

Pour into a warm half-round mould.

When cold, break off the slag.

Crush the slag and keep any lead beads which may be present along with the lead button.

To "Clean" the Slag.

Mix the pounded slag with

	Red lead . . .	20 grammes
	Sodic carbonate . .	3 „
	Charcoal . . .	1·3 „
or	Flour	2·5 „

Mix the materials intimately and charge them into the crucible which had been used.

Melt and pour.

Detach the button.

Weigh all the lead obtained.

Hammer the buttons into cubes.

Cupel.[1]

[1] The lead produced in the first fusion may be advantageously cupelled apart from the second. It is not always necessary to clean the slag.

Weigh the bead obtained and deduct the blank.

"Part" the metals as described in Exercise No. 114.

Calculate the yield.

Report.

EXERCISE NO. 121.

Assay of Silver Ore with Basic Gangue.

Weigh out a charge consisting of

Ore	25	grammes
Red lead	42	„
Sodic carbonate	22	„
Borax	8	„
Charcoal	2·5	„
or Flour	4·5	„

Mix the materials and treat them as in the previous exercise.

Report.

EXERCISE NO. 122.

Assay of Pyritic Silver Ore.

Weigh off a charge of 25 grammes of the ore.

Roast it on a scorifier—if suitable and if judged to be necessary—in a muffle furnace at a dull red heat.

When roasting has been carried far enough and the ore has cooled, mix it with

Red lead . . .	50 grammes
Sodic carbonate . .	22 „
Borax	15 „

and charge into a large fire-clay crucible.[1]

Place the crucible and contents in a pot furnace which is at a dull red heat and cautiously raise the temperature.

Fuse the charge.

Melt and pour.

Remove the slag and the regulus, crush them and clean them as in the previous exercise.

Cupel the silver lead obtained.

Calculate the yield and report.

[1] In some instances the introduction of a strip of hoop iron is recommended.

ASSAY OF GOLD ORES.

The remarks regarding silver ores apply generally to gold ores.

EXERCISE NO. 123.

Assay of Gold Ore with Acid Gangue.

Weigh off a charge of

Ore	50 grammes
Red lead . . .	45 „
Sodic carbonate . .	60 „
Flour	3 „
or Charcoal . . .	1·3 „

Treat as directed in Exercise No. 120.

The button of lead (which should be soft) ought to weigh about 25 grammes.

Keep the slag for treatment if found necessary.

Weigh the beads obtained and note the weight.

Fuse on a clean cupel with twice their weight of silver and about half a gramme of lead.

Proceed with the parting as before and dry the remaining gold in an annealing cup.

Calculate the percentage of each of the metals.

Report.

NOTES.—Some ores contain sufficient silver to permit of "parting" without addition of silver to the bead obtained.

For proper parting the bead must contain at least two-thirds its weight of silver.

EXERCISE NO. 124.

Assay of Gold Ore with Basic Gangue.

Weigh off a charge of

Ore	50 grammes
Red lead . . .	45 „
Sodic carbonate . .	22 „
Sand	15 „
Borax	10 „
Charcoal . . .	3 „

If copper oxide, or other easily reducible matter, is present, the weight of red lead must be considerably increased.

Treat as previously directed.

Report.

EXERCISE NO. 125.

Assay of Pyritic Gold Ore, without Roasting.

Make up a charge of

Ore . . .	50 grammes
Red lead . . .	110–140 „
Sodic carbonate . .	22 „
Borax	8 „

Charcoal may be required; if so, add 1 gramme.

It may be necessary to add sand to the charge.

The presence of metallic iron is necessary.

Put the charge in a large fire-clay crucible.

Frit and fuse the charge and proceed as in previous exercises.

Report.

EXERCISE NO. 126.

Assay of Pyritic Gold Ore, after Roasting.

Weigh off 50 grammes of the ore and gently roast it at a low temperature until "sweet."

Mix the roasted residue with fluxes, etc., as for a gold ore with basic gangue.

Put the charge in a large fire-clay crucible.

Frit and fuse the charge and proceed as in previous exercises.

Report.

EXERCISE NO. 127.

Assay of Precious Metals in Old Cupels.

Weigh off a charge of

Old cupels (in fine powder)	.	20 grammes
Sodic carbonate . . .	.	25 „
Borax glass	.	25 „
Fluor spar	.	22 „
Flour	.	2 „
or Charcoal	.	1 „

Mix the materials thoroughly and charge them into a large fire-clay crucible.

Place in a pot furnace which is at a dull red heat.

Gradually raise the temperature.

When the charge is well-fused pour.

Break away the slag, clean the lead button, and cupel it.

Weigh the bead and note the weight.

Part the silver from the gold.

Calculate the percentage of each metal.

Report.

PROXIMATE ANALYSIS OF SOLID FUELS—COAL, COKE, ETC.

Particular attention should be paid to the mechanical condition of the fuel. For the estimation of ash, and of moisture, the sample operated on should be very finely pounded. If the sample supplied is not finely pounded, *a portion* of the sample should be carefully selected and the whole of the selected portion crushed fine enough. From the finely pounded sample the necessary quantities should be accurately weighed off.

The estimations required are—

(*a*) Estimation of moisture.
(*b*) Estimation of ash.
(*c*) Estimation of coke.
(*d*) Calculation of the percentage of fixed carbon; and also—
(*e*) An abstract of results.

(*a*) Estimation of Moisture.

Select a flat porcelain basin of about 3 or 4 inches diameter, wipe it dry, weigh and note the weight. The basin may be dried by heating it over the flame of a Bunsen burner (care being taken not to crack the basin) for five or ten minutes and allowing it to cool in a desiccator.

Weigh into the basin 2 grammes of the finely pounded sample.

Set the basin and contents in a steam oven at about 100° C., and allow to remain there for 1½ hours.[1]

Take the basin and contents from the steam bath, allow to cool in a charged desiccator. (Continue to keep the steam bath up to 100° C.)

When cool, weigh and note the weight.

Set the basin and contents back in the hot steam oven; allow to remain for about fifteen minutes or more, withdraw and replace in the desiccator; and, when cool, reweigh and note the weight found.

If necessary, repeat the heating, cooling, and weighing.

Example.

Before drying—

Weight of basin + sample	= 19·4375
Weight of basin only	= 17·4375
Weight of sample	= 2·0000

Weight of basin + sample, before drying	= 19·4375
Weight of basin + sample, after drying	= 19·3533
	0·0842

[1] Some, if not all, samples of coal when heated too long *gain* in weight, owing, it is supposed, to the oxidation of sulphur in the sample.

The writer found that the maximum loss of weight, from a large number of coals, was obtained on heating for 1¾ hours.

$$\frac{0{\cdot}0842 \times 100}{2} = 0{\cdot}0842 \times 50 = 4{\cdot}21$$

and 4·21 = percentage of moisture in the sample of coal.

NOTE.—Time permitting, the dried sample may be used for the estimation of ash, by cautiously burning off all combustible matter (in a muffle), allowing to cool in a desiccator, weighing and calculating.

(*b*) ESTIMATION OF ASH IN COAL.

Either use the dried sample—from the estimation of moisture—or

Select a porcelain basin about 3 inches diameter, wipe it dry, weigh and note the weight.

Weigh into the basin 2 grammes of the finely pounded sample.

Place the basin and sample in front of a hot muffle, and, after a little time, push the basin and contents further in, and, after a little more time, place them in the hottest part of the muffle.

Allow to remain until all the combustible components have burned off.

Withdraw the basin to the sole in front of the muffle.

When partly cooled, place in a charged desiccator, and, when fully cooled, weigh.

Example.

Weight of basin + ash	= 17·5622
Weight of basin only	= 17·4375
	0·1247

$$\frac{0{\cdot}1247 \times 100}{2} = 0{\cdot}1247 \times 50 = 6{\cdot}235$$

and 6·24 = percentage of ash in the sample of coal.

(*c*) Estimation of Coke.

Dry a No. 2 porcelain crucible and lid.

Weigh the crucible and lid and note the weight.

Weigh into the crucible 10 grammes of the powdered sample, taking care to see that the lid is kept on the balance-pan while weighing off the sample.

Place the lid on the crucible.

Place the whole in a warm part of a hot muffle. In a short time a flame should appear between the crucible and the rim of the lid.

Keep the crucible, with the lid on, in the muffle as long as the flame can be seen.

Withdraw the crucible, etc., to the sole in front of the muffle.

Allow to remain there until partly cooled.

Place in a charged desiccator, and, when fully cooled, weigh.

Example.

Before coking—

Weight of crucible and lid + sample	= 40·5362
Weight of crucible and lid	= 30·5362
Weight of sample taken	= 10·0000

After coking—

Weight of crucible + lid + coke = 37·2713
Weight of crucible and lid only = 30·5362

6·7351

$$\frac{6{\cdot}7351 \times 100}{10} = 6{\cdot}7351 \times 10 = 67{\cdot}35$$

and 67·35 = percentage of coke.

(*d*) Calculation of Percentage of Fixed Carbon.

Percentage of coke, as found by experiment = 67·351
Percentage of ash, as found by experiment = 6·235

61·116

and 61·116 = percentage of fixed carbon.

(*e*) Abstract of Results.

Fixed carbon	=	61·12
Volatile carbon [1] (hydrocarbons, etc.)	=	28·43
Ash	=	6·24
Moisture	=	4·21
Total	=	100·00

[1] By difference.

ESTIMATION OF SULPHUR IN COAL OR COKE.

Weigh off 9 grammes of partly slaked lime which is as free as possible from sulphur and silica. (The amount of sulphur in the lime must be estimated and deducted.)

Place about half of it in a No. 00 porcelain basin (about $2\frac{3}{4}$ inches diameter).

Weigh off (accurately) 3 grammes of the carefully selected and finely-pounded sample of coal or coke.

Transfer the weighed sample to the basin.

Stir so as to mix the lime and the weighed sample thoroughly.

Spread the remainder of the weighed lime over the mixture so as to form a covering.

Place the basin and contents in a hot muffle.

Allow to remain for at least forty-five minutes.

Draw the basin to the front of the muffle and stir up the contents so as to expose any unburnt portions.

Replace in hot part of the muffle.

Allow to remain for ten minutes or more.

Stir up contents again and search for unburnt coal.

If the coke has all been burned, place the basin in a safe place to cool a little.

Transfer the contents to a $5\frac{1}{2}$-inch $\times$ $3\frac{1}{4}$-inch beaker.

Add about 50 c.c. of water.

Add about 5 c.c. of bromine water and stir well.

Add enough strong hydrochloric acid to dissolve the lime, etc. (about 33 c.c. should be sufficient).

Boil briskly for at least five minutes.

Filter, collecting the filtrate in a clean 6-inch × 3¾-inch beaker.

Wash well with hot water.

Heat the filtrate and washings just to boiling-point.

Add 40 c.c. of warm 2½ per cent. barium chloride solution.[1]

Place a clock glass on the beaker.

Boil briskly for about seven minutes.

Allow the precipitate to settle.

Wash three times by decantation through a 9-cm. Swedish filter.

Wash the precipitate on to the filter.

Wash about ten times with hot water.

Test the washings for presence of chlorine.

If necessary, continue the washing.

Dry the filter paper and the precipitate.

Ignite in a tared No. 0 porcelain crucible for about thirty minutes in a hot muffle.

Allow to cool in a desiccator.

Weigh and calculate the result.

Example.

Weight of crucible + precipitate + ash	= 7·1343
Weight of crucible	= 7·1263
Weight of precipitate + ash	= 0·1911
Weight of ash + sulphate from lime used	= 0·0041
Weight of precipitate	= 0·1870

[1] The barium chloride prescribed is capable of precipitating 0·131 gramme of sulphur = 0·952 gramme of $BaSO_4$ = about 4·57 per cent. of sulphur from the coal: a plentiful excess.

$$\frac{0{\cdot}187 \times 32 \times 100}{233 \times 3} = 0{\cdot}187 \times 4{\cdot}587 = 0{\cdot}856$$

and 0·856 = percentage of sulphur in the sample of coal.

APPENDIX.

ELEMENTS.

THERE are about eighty elements. The following are of importance to metallurgical students :—

Name.	Chemical symbol.	Atomic weights.
METALS—		
Aluminium	Al	27·1
Chromium	Cr	52·1
Manganese	Mn	55·0
Iron (*Ferrum*)	Fe	55·9
Nickel	Ni	58·7
Copper (*Cuprum*)	Cu	63·6
Zinc	Zn	65·4
Arsenic	As	75·0
Silver (*Argentum*)	Ag	107·9
Tin (*Stannum*)	Sn	119·0
Antimony (*Stibium*)	Sb	120·2
Platinum	Pt	194·8
Gold (*Aurum*)	Au	197·2
Mercury (*Hydrargyrum*)	Hg	200·0
Lead (*Plumbum*)	Pb	206·9
Bismuth	Bi	208·5
NON-METALS—		
Hydrogen	H	1
Carbon	C	12
Nitrogen	N	14
Oxygen	O	16
Phosphorus	P	31
Silicon	Si	28·4
Sulphur	S	32

Table of Melting-points in Centigrade Degrees.

Mercury	melts at − 39	Gold	melts at + 1060
Tin	„ + 232	Cobalt	„ 1500
Bismuth	„ 268	Iron	„ 1600
Cadmium	„ 320	Nickel	„ 1600
Lead	„ 326	Platinum	„ 1775
Zinc	„ 420	Manganese	„ 1900
Aluminium	„ 625	Chromium. The melting-point is probably above that of platinum	
Antimony	„ 632		
Silver	„ 960		
Copper	„ 1050		

Table of Specific Gravity of Metals.

Aluminium	2·56	Nickel	8·80
Arsenic	5·66	Copper	8·82
Antimony	6·70	Silver	10·53
Chromium	6·80	Lead	11·37
Zinc	7·15	Mercury	13·60
Tin	7·30	Gold	19·30
Iron	7·86	Platinum	21·50
Manganese	8·00		

Table of Percentage Composition of some Important Alloys.

	Copper.	Zinc.	Tin.	Lead.	Antimony.	Iron.	Manganese.	Aluminium.	Nickel.	Phosphorus.
Brass	63–72	36–40	—	—	—	—	—	—	—	—
Muntz metal	60–64	36–40	—	—	—	—	—	—	—	—
Delta metal	55	43·5	—	0·4	—	1·0	—	—	—	0·1
Sterro metal	55	42	1·5	—	—	1·5	—	—	—	—
Dutch metal	85	15	—	—	—	—	—	—	—	—
Brazing solder	50	50	—	—	—	—	—	—	—	—
Gun metal	91	—	9	—	—	—	—	—	—	—
Bell metal	77	—	23	—	—	—	—	—	—	—
Speculum metal	67	—	33	—	—	—	—	—	—	—
Bronze	84	2	14	—	—	—	—	—	—	—
Aluminium bronze	90–95	—	—	—	—	—	—	5–10	—	—
Manganese bronze	81	—	—	—	—	2	17	—	—	—
Phosphor bronze	82·7	—	13	4	—	—	—	—	—	0·3

TABLE OF PERCENTAGE COMPOSITION—*continued.*

	Copper.	Zinc.	Tin.	Lead.	Antimony.	Iron.	Manganese.	Aluminium.	Nickel.	Phosphorus.
Britannia metal .	1	—	91	—	8	—	—	—	—	—
Type metal . .	—	—	5	80	15	—	—	—	—	—
Bearing metal .	4	86	—	—	10	—	—	—	—	—
German silver .	60	20	—	—	—	—	—	—	20	—
Pewter, French .	—	—	82	18	—	—	—	—	—	—
Solder, plumbers'.	—	—	33·3	66·7	—	—	—	—	—	—
Do. tin . .	—	—	50	50	—	—	—	—	—	—
Do. fine . .	—	—	66·7	33·3	—	—	—	—	—	—

	Bismuth.	Cadmium.	Tin.	Lead.
Fusible alloy (Darcets) . .	50	—	25	25
Do. do.	50	10	13	27

British coins.	Gold.	Silver.	Copper.	Zinc.	Tin.
Gold	91·66$\frac{6}{}$	8·33$\frac{3}{}$	—	—	—
Silver	—	92·5	7·5	—	—
Bronze	—	—	95	4	1

EXAMPLES OF CALCULATIONS.

(*a*) To find the Weight of an Element (or Compound) required for a Reaction.

Rule.—From the equation find the atomic (or the molecular) weights of the reacting substances. Divide the one by the other, and then multiply by the actual weight of the substance which is to be acted on.

Example.—How much carbon is required to reduce the copper in 30 grammes of black copper oxide (CuO)?

Equation.—$\underbrace{CuO}_{\|} + C = \underset{\|}{Cu} + \underset{\|}{CO}$

$\quad 79{\cdot}6 \quad\quad 12$

which shows that 12 units of carbon can reduce 79·6 grammes of CuO, and as a *less* weight of carbon than of CuO is required, divide by the greater number. Then multiply by the actual weight of the substance which is to be acted on.

$$\frac{12 \times 30}{79{\cdot}6} = 4{\cdot}52 = \text{grammes of C required.}$$

Note.—Allowance must be made for the moisture, etc., in the charcoal.

(*b*) To find the Weight of Metal obtainable from a Reaction.

Example.—How much lead can be obtained from 33 grammes of pure litharge (PbO)?

$$\text{Equation.}—2PbO + C = 2Pb + CO_2$$

$$\underset{445 \cdot 8}{\parallel} \qquad \underset{413 \cdot 8}{\parallel}$$

The weight of lead obtained will, of course, be less than the oxide from which it is reduced.

$$\frac{413 \cdot 8 \times 33}{445 \cdot 8} = 30 \cdot 63 = \text{weight of lead obtainable.}$$

(*c*) To find the Percentage Loss or Gain.

Example.—23 grammes of one metal and 16 grammes of another metal are charged into a crucible. The resulting ingot weighed 36·4 grammes. What is the percentage loss?

Metals charged 23 + 16 =	39·0
Ingot obtained =	36·4
Loss =	2·6

$$\frac{2 \cdot 6 \times 100}{39} = 6 \cdot 67$$

(*d*) To find the Composition of an Alloy from the Weights of its Constituents.

Example.—An alloy is made from 27 grammes of copper and 8 grammes of tin—total, 35 grammes. What should be its percentage composition?

$$\text{Cu } \frac{27 \times 100}{35} = 77 \cdot 14$$

$$\text{Sn } \frac{8 \times 100}{35} = 22 \cdot 86$$

$$100 \cdot 00$$

(*e*) To find the Percentage Composition of an Alloy from the "Parts" stated.

Example.—Dutch metal consists of 11 parts of copper and 2 parts of zinc. What is its percentage composition?

$$\text{Cu } 11 + \text{Zn } 2 = 13$$

$$\text{Cu } \frac{11 \times 100}{13} = 84{\cdot}62$$

$$\text{Zn } \frac{2 \times 100}{13} = 15{\cdot}38$$

$$100{\cdot}00$$

(*f*) To find the Percentage Composition of a Compound from its Chemical Formula.

Rule.—Divide each constituent by the molecular weight, and, in order to find percentage, multiply each result by 100.

Example.—What is the percentage composition of galena (PbS)?

$$\text{Galena contains Pb} = 206{\cdot}9$$
$$\text{and S} = 32{\cdot}0$$
$$238{\cdot}9$$

$$\text{Pb } \frac{206{\cdot}9 \times 100}{238{\cdot}9} = 86{\cdot}61$$

$$\text{S } \frac{32 \times 100}{238{\cdot}5} = 13{\cdot}39$$

$$100{\cdot}00$$

(*g*) To find the Weight of the Respective Metals required to produce a given Weight of Alloy.

Rule.—Multiply the percentage of each constituent by the weight of the intended mass of alloy and divide each result by 100.

Example.—What is the weight of each metal required to produce a 30-gramme ingot of brass containing copper 66 per cent., zinc 33 per cent., and tin 1 per cent.?

$$\text{Cu } \frac{66 \times 30}{100} = 19{\cdot}8$$

$$\text{Zn } \frac{33 \times 30}{100} = 9{\cdot}9$$

$$\text{Sn } \frac{1 \times 30}{100} = 0{\cdot}3$$

$$30{\cdot}0$$

Note.—Allowance must be made for unavoidable volatilization of zinc.

Royal College of Science, London.

TABLE FOR COMPUTING FROM THE PERCENTAGE OF SILVER THE TROY WEIGHT OF SILVER PER STATUTE TON.

Per cent.	Per ton of 2240 lbs.			
	Grains.	ozs.	dwts.	grs.
0·0001	15·68	0	0	15·68
0·0002	31·36	0	1	7·36
0·0003	47·04	0	1	23·04
0·0004	62·72	0	2	14·72
0·0005	78·40	0	3	6·40
0·0006	94·08	0	3	22·08
0·0007	109·76	0	4	13·76
0·0008	125·44	0	5	5·44
0·0009	141·12	0	5	21·12
0·001	156·8	0	6	12·8
0·002	313·6	0	13	1·6
0·003	470·4	0	19	14·4
0·004	627·2	1	6	3·2
0·005	784·0	1	12	16·0
0·006	940·8	1	19	4·8
0·007	1,097·6	2	5	17·6
0·008	1,254·4	2	12	6·4
0·009	1,411·2	2	18	19·2
0·01	1,568·0	3	5	8·0
0·02	3,136·0	6	10	16·0
0·03	4,704·0	9	16	0·0
0·04	6,272·0	13	1	8·0
0·05	7,840·0	16	6	16·0
0·06	9,408·0	19	12	0·0
0·07	10,976·0	22	17	8·0
0·08	12,544·0	26	2	16·0
0·09	14,112·0	29	8	0·0
0·1	15,680·0	32	13	8·0
0·2	31,360·0	65	6	16·0
0·3	47,040·0	98	0	0·0
0·4	62,720·0	130	13	8·0
0·5	78,400·0	163	6	16·0
0·6	94,080·0	196	0	0·0
0·7	109,760·0	228	13	8·0
0·8	125,440·0	261	6	16·0
0·9	141,120·0	294	0	0·0

TABLE FOR COMPUTING FROM THE PERCENTAGE OF SILVER THE TROY WEIGHT OF SILVER PER STATUTE TON—*continued.*

Per cent.	Per ton of 2240 lbs.			
	Grains.	ozs.	dwts.	grs.
1·0	156,800·0	326	13	8·0
2·0	313,600·0	653	6	16·0
3·0	470,400·0	980	0	0·0
4·0	627,200·0	1306	13	8·0
5·0	784,000·0	1633	6	16·0
6·0	940,800·0	1960	0	0·0
7·0	1,097,600·0	2286	13	8·0
8·0	1,254,400·0	2613	6	16·0
9·0	1,411,200·0	2940	0	0·0
10·0	1,568,000·0	3266	13	8·0

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THE END.

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