

METALS, FLUXES, REDUCING AGENTS, RE-AGENTS, ETC., USED IN A METALLURGICAL LABORATORY.

§ 23. The following bodies should be provided for the use of students, but they need not be of a high degree of purity. Re-agents for analysis will be described in another part.

Fluxes may act as true solvents, and probably in many slags true solution occurs. More generally a flux is used for the purpose of forming a fusible compound with the earthy constituents of an ore, or to take up impurities from metals after oxidation. The gangue or earthy matter of an ore is usually a silicate of lime, alumina, magnesia, or oxide of iron; or a carbonate of lime and magnesia. Silica, and the silicates of lime, alumina, and magnesia are practically infusible when alone, but the addition of lime, or oxide of iron, or an alkali to silicate of alumina causes the formation of a fusible double silicate. Similarly the addition of oxide of iron or alumina or alkali to silicate of lime renders the latter fusible. Thus a body which is infusible in itself may be used as a flux to make another otherwise infusible body fusible. Most earthy bodies are either acid or basic in their properties and therefore require a flux of opposite properties to themselves, that is, an acid gangue requires a basic flux and *vice versa*. For operations performed in clay crucibles it is advisable not to form too basic a slag, as it rapidly corrodes them, since they are made of fire-clay and silica which unite readily with lime, soda, and most bases. Slags from which metals are to be separated should not be too acid, unless in the case where the metal is already in the metallic state mixed with dross and dirt. In the case when the metal is easily fusible and liable to oxidation or volatilisation, it should be covered with a layer of resin or fat or common salt.

(See § 23, No. 22.) In most cases the fusion of a metal, when reduced from its oxide, should be accompanied with a basic slag, as it is difficult to completely liberate the whole of a metal from acid slags such as borates and silicates. When the object is to separate one metal, say copper, without reducing an unnecessary amount of another, say iron, at the same time, a slag with much borax is an advantage, but it will contain some copper and require to be cleaned. In some cases it is advisable to treat an ore or metalliferous matter so as to form a sulphide first instead of reducing to the metallic state. Such sulphide is termed a "regulus." (See § 59.) It is usually brittle, dark in colour, and crystalline. A regulus is often reduced to metal by strongly heating it with an oxide. (Expt. 178.) Sulphide of iron and some other sulphides formed during an assay will remain diffused through the slag, instead of fusing into a button of regulus, if the slag contain sulphide of sodium and the temperature is not too high. Use is sometimes made of arsenic, in a similar way to that of sulphur, to form an arsenide, which is termed a speise. It is chiefly employed in the metallurgy of nickel, cobalt, and tin, forming brittle and easily fusible compounds.

(1) *Antimony*. Sb. Atomic weight, 120. Specific gravity, 6.75. Melts at 440° C. The commercial metal has a fern-like appearance on its surface, and may contain varying amounts of sulphur, potassium, arsenic, lead, iron, and copper. It may be purified from sulphur, iron, arsenic, and copper, by fusing with sulphide of antimony and carbonate of soda in a crucible for an hour; from sulphur and potassium by fusion with nitre. Fineness of grain is a test of purity.

(2) *Arsenic*. As. Atomic weight, 75. Specific gravity, 5.62, vaporises under ordinary conditions without melting. It exists in two forms:—1. The compact variety is obtained by condensing the metal at a temperature a

little below its melting point, so that it solidifies in an atmosphere of its own vapour. It is then white, having a strong lustre and little tendency to oxidise in air below 80°C . even when powdered. 2. When reduced from its oxide by carbon, being mixed with other gases, or when condensed on a cold surface, it is a dark grey crystalline substance less dense than the former variety, and readily oxidisable. When heated it emits an odour like garlic, and in presence of air or oxygen forms As_2O_3 .

(3) *Aluminium*. Al. Atomic weight, 27. Specific gravity, 2.6. It is a white metal with a faint tinge of blue, takes a fine polish, and may be frosted like silver by plunging into a weak solution of soda, well washing, and digesting in strong nitric acid. It is very malleable and ductile, as soft as silver, and highly sonorous. Its melting point is 625°C ., but it is not volatile when heated out of contact with air; it exhibits a crystalline structure when heated and slowly cooled; does not oxidise in air even at a red heat; does not combine with sulphur, and is not blackened with sulphuretted hydrogen; is not soluble in dilute nitric or sulphuric acid, but soluble in hydrochloric acid. It may be fused with nitre without oxidising, so that impurities may be removed by this means. Its tensile strength in the cast state is 7 tons per square inch, but this may be considerably increased by cold hammering. It unites with other metals to form valuable alloys.

(4) *Bismuth*. Bi. Atomic weight, 207.5. Specific gravity, 9.80, but this is "diminished" by pressure. Melts at 268°C . This metal is greyish white with a reddish tinge, a bright lustre, not sensibly oxidised at ordinary temperatures, but when heated in air burns with a bluish flame emitting yellow fumes. When pure, it crystallises more readily than any other metal; is

more dense in the liquid than in the solid form; is the most diamagnetic of all metals, and its best solvent is nitric acid. When mixed with lead and tin it forms fusible alloys.

(5) *Gold*. Au. Atomic weight, 196.2. Specific gravity, 19.5. Melts at about 1045°C . It possesses a characteristic yellow colour; is the most malleable and ductile of metals; has high conductive power for heat and electricity; is unaffected by air; is nearly as soft as lead; is a feeble base, and therefore readily separated from its states of combination; its best solvent is moist chlorine, generally obtained by heating the metal with two parts hydrochloric acid and one part nitric acid.

(6) *Iron*. Fe. Atomic weight, 56. Specific gravity, 7.8. Melts at above 1600°C . when pure. The pure metal is seldom required, but may be obtained by the electrolysis of a solution of its chloride, or in the dry way by melting pianoforte wire with oxide of iron and pure glass in a lime crucible. Hoop-iron, filings, turnings, and the purest kind of soft iron wire, are the forms most useful in a metallurgical laboratory. It should be soft, malleable, tenacious, practically infusible, but should soften at a white heat; should not be sensibly hardened when made red hot and plunged into cold water.

(7) *Steel* is iron containing varying proportions of from 0.15 to 1.8 per cent. of carbon and usually some manganese. It may be hardened by sudden cooling from a red heat, the hardness being in proportion to the carbon or other bodies which have a similar effect. It may be permanently magnetised. Its colour is greyish white, of a uniform granular texture, very close grained, and whiter when hardened than when soft; has very high tenacity: melts at about 1300° to 1500°C .; and is less oxidisable than soft iron at ordinary temperatures.

(8) *Cast iron* is a variety with a greater proportion of carbon and other bodies than is contained in steel. Three kinds are known in commerce, viz. :—"Grey," in which the carbon is chiefly free ; "White," in which the carbon is for the most part combined ; and "Mottled," which is a combination of the other two. A white variety is also used, combined with manganese, called ferro-manganese ; and with phosphorus, called ferro-phosphorus, etc. ; the effect being to harden, and to render it more fusible and brittle. A grey variety of pig-iron, containing about 4 per cent. of silicon, is used to produce softness in other brands of pig-iron, as the silicon induces the separation of carbon in the graphitic form. Cast-iron melts at 1100° to 1300° C.

(9) *Lead*. Atomic weight, 206.4. Symbol Pb. Specific gravity, 11.45. Melting point, 325° C. The commercial variety is sufficiently pure for laboratory purposes, except for exact assays of gold and silver. It is a bluish grey, soft, malleable metal, with feeble tenacity, tough, and may be welded by pressure. The property of viscous flow is well exhibited by lead, so that it may be forced through perforations, as in making piping and bullets. It enters into the composition of many useful alloys.

(10) *Magnesium*. Mg. Atomic weight, 24. Specific gravity 1.75. It is a malleable white metal, oxidising in moist air ; melts at 750° C., and volatilises like zinc. When ignited in air it burns with a bright light, forming magnesium oxide MgO. It is a strong base, and replaces zinc, iron, etc., from their solutions.

(11) *Mercury*. Hg. Atomic weight, 200. Specific gravity, 13.6. It is a white metal, liquid at the ordinary temperature ; freezes at -39° 44 C., forming a tin-white ductile mass ; is not affected by air unless heated ; dis-

solves in nitric acid ; boils at 350° C. ; combines with silver, gold, tin, lead, bismuth, cadmium, zinc, but not so readily with copper and iron. The commercial metal often contains zinc, bismuth, lead, antimony, and tin. When pure, a globule of the metal will roll down a slight incline as a sphere ; if impure, the particle elongates somewhat ; also, when shaken in an open flask will tarnish after a time. It is used in the extraction of gold and silver.

(12) *Nickel*. Ni. Atomic weight, 58.6. Specific gravity, 8.3. Little more fusible than iron. It is a white, malleable, ductile, tenacious metal ; welds like iron ; does not easily oxidise ; combines with sulphur to form regulus, and with arsenic to form speise. May be reduced from its oxide by carbon, carbonic oxide, and hydrogen, and takes up varying proportions of carbon like iron.

(13) *Platinum*. Pt. Atomic weight, 194.3. Specific gravity, 21.5. Melts only by aid of the electric current or by means of the oxy-hydrogen blow-pipe at a temperature of 1775° C. It is a white unoxidisable metal, malleable and ductile, very tenacious, softer than silver ; an inferior conductor of heat and electricity ; soluble in aqua-regia, but when alloyed with much silver it is dissolved by nitric acid, and may thus be separated from gold.

(14) *Silver*. Ag. Atomic weight, 107.6. Specific gravity, 10.5. Melts at 945° C. It is a pure white metal with a brilliant lustre, very malleable and ductile, soft ; has considerable tenacity ; is the best conductor of heat and electricity ; is hardened by alloying with copper ; does not oxidise, but mechanically absorbs oxygen when melted, and gives it out again on cooling, this is termed "spitting" ; it combines directly with sulphur to form sulphide ; is soluble in strong sulphuric and dilute nitric acids, and forms useful alloys with copper, gold, and other metals.

(15) *Tin*. Sn. Atomic weight, 117.4. Specific gravity, 7.3. Melts at 227° C. Commercial tin is seldom pure, the metal being generally associated with arsenic, lead, iron, copper, and occasionally with tungsten, manganese, antimony, zinc, and molybdenum. It is a white metal, with a characteristic smell when warm, and makes a peculiar crackling sound when bent, known as the "cry"; it is malleable, but has low tenacity; does not oxidise at ordinary temperatures.

(16) *Copper*. Cu. Atomic weight, 63.2. Specific gravity, 8.82. Melts at 1050° C. It is red in colour, highly malleable, ductile, tough, and tenacious, not sensibly volatile, and is one of the best conductors of heat and electricity. The common impurities are iron, arsenic, silver, antimony, and cuprous oxide; sometimes bismuth, tin, sulphur, and lead. Of these antimony and bismuth are the most injurious. It is soluble in acids, and forms with many metals useful alloys.

(17) *Zinc*. Zn. Atomic weight, 65. Specific gravity, 7.15. Melts at 415° C. It is a bluish white metal with a bright lustre; commercial zinc is brittle in the cast state with a highly crystalline structure, but becomes malleable at 100° to 150° C., above this point it again becomes brittle; the pure metal is malleable at the ordinary temperature and volatile at a bright red heat. It is soluble in acids and alkalies; is a strong base, and displaces most metals from their solutions. The chief impurities in commercial zinc are lead, iron, arsenic, and sometimes silver. Its chief alloys are formed with copper.

(18) *Carbon*. C. Atomic weight, 12. This substance in its different forms, more or less pure, is largely employed as a reducing agent, chiefly as charcoal and anthracite. In the form of coke and charcoal it is used as a fuel, and as graphite in making crucibles.

(19) *Phosphorus*. P. Atomic weight, 31. When pure it is transparent and nearly colourless; melts at 44° C.; shows a crystalline structure when broken, is very inflammable, burning with a brilliant white flame, so that it must be kept in water. Phosphorus assumes several different forms, one of which is amorphous, and red in colour. This kind may be heated to 250° without alteration; does not ignite by moderate friction, and may be handled without danger. It unites readily with most metals when heated with them, making them, as a rule, crystalline and brittle.

(20) *Sulphur*. S. Atomic weight, 32. At ordinary temperatures is yellow, brittle, solid, tasteless, and almost inodorous; melts at 120°, and boils at 440°; combines directly with most metals when heated with them, forming sulphides and evolving great heat. It is used in the laboratory in the form of powder, and as roll sulphur.

(21) *Ammonium Chloride* (AmCl) called sal-ammoniac. This substance is decomposed by several metals forming metallic chlorides and liberating ammonia, which property is taken advantage of in purifying gold. A similar reaction occurs with several metallic salts.

(22) *Sodium Chloride* (NaCl) or common salt, is employed in metallurgical operations for preserving the substance beneath from the action of the atmosphere, and to moderate the action of bodies which cause violent ebullition. It melts and volatilises at a red heat in an open crucible, but requires a white heat to vaporise it in a closed vessel. When heated to redness with silica it forms a readily fusible silicate. It forms fusible compounds with antimony and arsenic, thus removing them from other metals during the process of refining. As the crystals decrepitate when heated, common salt should be powdered before using as a flux. Under

certain circumstances it has a tendency to form volatile chlorides with a consequent loss of metal.

(23) *Borax* ($B_4O_7 \cdot Na_2$). In the crystalline form it may contain 5 or 10 molecules of water, which are given off on heating, causing an enormous increase in volume, so that the vitrified form is much more suitable for assaying. It forms fusible compounds with silica, and nearly all bases, being especially useful in uniting with metallic oxides, sulphides, and arsenides. The commercial salt is adulterated with common salt and alum.

(24) *Sodium Carbonate* (Na_2CO_3) has the property of oxidising many metals, as tin, iron, zinc, etc., by the action of its carbonic acid, and as a consequence of this action it acts as a desulphuriser. It forms fusible compounds with silica and many metallic oxides; it also melts at a low temperature, absorbing many infusible substances, such as lime, alumina, charcoal, etc. In some cases it acts as a reducing agent, as in the case of silver chloride. When mixed with carbonate of potash a double salt is formed, which fuses at a lower temperature than either taken alone, a property very useful in the fusion of silicates, etc.

(25) *Potassium Nitrate* (KNO_3), also called nitre and saltpetre, is largely used as an oxidising agent. It fuses below redness, and at a higher temperature decomposes, yielding a large volume of oxygen, whereby the sulphur of metallic sulphides is converted into sulphurous acid, and the metals into oxides. Sodium nitrate acts in the same way.

(26) *Potassium Bitartrate* (\overline{THoKo}), known also as cream of tartar or tartar. When pure this substance is white, but the variety chiefly used by assayers is coloured, and sold as red "Argol," which is cheaper, and contains other carbon-

aceous matters, which give it greater reducing power than pure cream of tartar. This reagent is very valuable in assays requiring much carbonaceous matter.

(27) *Potassium Chlorate* ($KClO_3$). This substance is sometimes used with nitre as an oxidising agent in dry assays, but much more frequently in wet assays.

(28) *Potassium Cyanide* (KCN). This flux is valuable on account of the facility with which it fuses, and the readiness with which it reduces many metallic compounds when mixed with carbonate of soda. For the purposes of dry assaying, common cyanide is preferable as a reducing agent, because it contains carbonate of potash.

(29) *Calcium Oxide* (CaO), or lime, is used in the caustic state, or combined with carbonic acid in the form of carbonate. It is a useful flux for silica and silicates, with which it combines to produce a double silicate, being in this form more fusible. It is also used to remove sulphur and phosphorus from metals, and their compounds.

(30) *Calcium Chloride* ($CaCl_2$) is useful as a desiccating agent.

(31) *Calcium Fluoride* (CaF_2) or Fluor-spar. This substance acts as a flux in two ways—1, by combining with silicates, forming fusible compounds; 2, by reacting on silicates and evolving silicon fluoride. It also forms fusible compounds with sulphates of lime and baryta, and with phosphates of lime. It should be free from pyrites, blende, and galena, with which it is likely to be contaminated.

(32) *Phosphate of Lime* ($Ca_3P_2O_8$). This substance is obtained from the calcination of bones, preferably those

of sheep and horses, until they become white; and is chiefly used in the laboratory for the manufacture of cupels. For this purpose the boneash should be of good quality and in fine powder; lime and other substances with which it is adulterated greatly interfering with its absorbent properties.

(33) *Barium Nitrate* (Ba_2NO_3). A solution of this salt is used as a test for soluble sulphates in the presence of lead or silver.

(34) *Barium Chloride* ($BaCl_2$) is used for the same purpose as the nitrate in the absence of lead and silver.

(35) *Cupric Oxide* (CuO), Black Oxide of Copper. This oxide forms the base in ordinary copper salts; melts at a white heat; is decomposed by heat, giving up oxygen and forming the lower oxide Cu_2O , thus acting as an oxidising agent; is readily reduced to metallic copper by reducing agents; when heated strongly with silica a silicate of the suboxide is formed; when heated with copper in suitable proportion it forms cuprous oxide (Cu_2O).

(36) *Cuprous Sulphide* (Cu_2S). This body is formed when copper and sulphur are heated together. It is a dark bluish grey substance, breaking with a conchoidal fracture; when heated in air a mixture of oxide and sulphate is formed; when heated with sufficient oxide of copper it is completely reduced to the metallic state.

(37) *Copper Sulphate* ($CuSO_4$) in the crystalline state contains 5 molecules of water, becoming anhydrous at $200^\circ C$. Copper is precipitated from a solution of this salt by certain metals, such as iron and zinc. When a solution of copper sulphate is submitted to electrolysis, the copper is deposited in a practically pure state.

(38) *Plumbic Oxide* (PbO) is produced when lead is heated in a current of air or oxygen. At a temperature of about $300^\circ C$. more oxygen is taken up, forming red lead; when the temperature is sufficiently high to melt the oxide, a yellow or reddish yellow crystalline mass is formed, known as litharge. Both oxides are reduced by carbon or hydrogen, producing metallic lead. Lead oxide when melted oxidises nearly all the metals, except mercury, gold, silver, platinum, etc. With other oxides it forms easily fusible compounds. With sulphur it forms sulphurous acid, metallic lead being isolated. When oxide of lead in sufficient quantity is melted with an infusible silicate a fusible double silicate is formed.

(39) *Plumbic Sulphide* (PbS). Lead and sulphur unite directly when heated together, forming a lead-grey crystalline substance known as artificial galena. It melts at a red heat, and at a higher temperature may be sublimed. Heated in air it is oxidised to sulphate or oxide, according to the temperature. It is reduced by iron. When one molecule of PbS is heated with 2 molecules of PbO the whole of the lead is isolated.

(40) *Manganese Dioxide* (MnO_2). This substance is black in colour, opaque, and a good conductor of electricity. When heated alone it is infusible, but gives off oxygen, forming Mn_2O_3 or Mn_3O_4 according to the degree of heat employed: heated with charcoal it is reduced to MnO . The facility with which it gives up oxygen makes it a valuable oxidising agent. With hydrochloric acid it is extensively used for generating chlorine. When strongly heated in a crucible lined with a paste of carbon it is reduced to the metallic state.

(41) *Silica* (SiO_2). This body occurs in crystalline and amorphous forms; it is white; infusible, except at the very highest temperatures; non-volatile; insoluble in water

and acids, except hydrofluoric; after ignition, is decomposed by carbon in the presence of iron, copper, or silver at a white heat, forming silicides of those metals. The amorphous and gelatinous varieties are slightly soluble in alkaline carbonates, but readily soluble in caustic alkalis. It combines with all the bases forming silicates, and is therefore frequently employed to effect the fusion and separation of gangues in ores; the best forms to use being pure white sand and quartz. If the silica is coloured, it should be digested with hydrochloric acid before use.

(42) *China Clay* is essentially a hydrated silicate of alumina, and when pure may be represented by the formula $(2Al_2O_3, 3SiO_2) + 3OH_2$; but the clay is generally mixed with other silicates. China clay is white, and infusible in an ordinary furnace when heated alone, but readily unites with earthy and metallic gangues to form a fusible slag, and therefore forms a useful flux for assaying ores like those of iron.

(43) *Glass* is a mixture of silicates of sodium and potassium with some insoluble silicate, such as silicate of calcium, magnesium, aluminium, iron, or lead. Being a compound silicate it fuses easily at a high temperature, and readily combines with lime and other bases containing little or no silica, so that it is often preferred to pure silica, and serves to economise borax. It is also employed as a covering in melting metals, so as to exclude the air. Plate or window glass or green bottle glass is the most useful, but flint glass, which contains much oxide of lead, would be detrimental in many cases.

(44) *Ferrous Sulphide* (FeS) is chiefly used as a source of sulphuretted hydrogen. Roasted with easily decomposable sulphides as that of silver, it converts them into sulphates. Heated with oxides of copper, nickel, etc., it

forms regulus. Heated in air it is oxidised to sulphate, and at a high temperature to oxide.

(45) *Iron Pyrites* (FeS_2). This body loses half its sulphur at a white heat, forming ferrous sulphide, and is used for similar purposes to that compound. It is chiefly employed in the metallurgy of copper, nickel, and cobalt.

(46) *Ferric Oxide* (Fe_2O_3). This oxide is very stable, non-volatile, and of a red colour. At a white heat it gives up oxygen, forming the magnetic oxide (Fe_3O_4). By heating with carbon, or carbonic oxide, it is reduced to the metallic state, but if much carbonic acid is present, ferrous oxide may be formed, which combines with any silica present, forming a fusible silicate. For this reason, it is sometimes used as a flux in copper assaying. In refining iron it acts as an oxidising agent. In presence of sulphur it oxidises that element to sulphurous acid.

(47) *Zinc Oxide* (ZnO) is a powerful base; it forms combinations with alkaline earths and several bases, and has a strong affinity for alumina. It is reduced by carbon, carbonic oxide, and hydrogen.

(48) *Cinnabar* (HgS). This sulphide is red in colour, and is decomposed by heating in a current of air. It is also reduced by many metals, and forms a better sulphurising agent than sulphur itself, being less volatile.

(49) *Black Flux*. This substance is prepared by heating a mixture of 2 parts of tartar (argol) and 1 part or less of nitre to redness in a closed crucible. The resultant mixture of potassium carbonate and carbon is often used in copper and lead assaying. It must be kept in closed bottles to prevent absorption of moisture. When great reducing power is required, black flux is prepared by

heating the tartar alone, but the excess of carbon thus produced reduces the fusibility of the slag. A mixture of sodium carbonate and charcoal is frequently used instead of black flux. The following proportions are recommended:—

Sodium carbonate,	94	88	82
Charcoal,	6	12	18

PHYSICAL PROPERTIES OF THE USEFUL METALS AND THE INFLUENCE OF SMALL QUANTITIES OF IMPURITIES IN MODIFYING THESE PROPERTIES.

Note.—Before using a mould, it should be warmed and black-leaded to prevent the metal sticking. This is assumed to be done in all the succeeding experiments in which a mould is used.

§ 24. Experiment 1. Weigh out 1000 grains of commercial lead; melt at the lowest possible temperature with a little charcoal to prevent unnecessary oxidation; pour into a rectangular mould (Fig. 17 *e*) so as to produce an ingot about 2 inches long and $\frac{1}{2}$ an inch wide. When cold, place it half-way in the jaws of a vice, and bend it backwards and forwards with a hammer until it is broken. Notice the absence of any definite structure on the fractured surface, and the toughness as exhibited by the difficulty in producing rupture. Re-melt and cast in the same mould as before; remove from mould when set, and place the ingot across two iron supports about $1\frac{1}{2}$ inches apart; heat the centre with the flame of a Bunsen burner, held in the hand, so that the flame only is under the metal, until the lead begins to melt, then strike the centre one sharp blow with a hammer, when the ingot will break short, showing its brittleness at this point.

The fractured surface will now be entirely different, showing a transverse columnar structure, the size and clearness of the columns being in proportion to the purity of the lead.

Exp. 2. Weigh out 1000 grains of the same lead, and melt with 50 grains of sulphide of lead; pour into a rectangular mould, and fracture when cold. It will be observed that this impure lead breaks much more readily than pure lead, and a crystalline or partially crystalline structure has been induced. Now re-melt, cast into mould, and place the ingot across two supports; heat with Bunsen burner as before, and break when on the point of melting. The fractured surface will still show the transverse columns, but they will be much smaller than with pure lead.

Exp. 3. Take 1000 grains of lead, and alloy with 15 grains of antimony. Fracture cold, and also when on the point of melting as described in Exp. 1. Notice that it is both hot and cold short, and that the fractured surface is coarsely granular in both cases.

Exp. 4. Melt 1000 grains of lead with 15 grains of metallic arsenic, and repeat the experiments described in Exp. 1. Notice the same results as with antimony.

Exp. 5. Test the malleability of lead by rolling the ingots obtained in Exps. 1, 2, 3 and 4 into sheets as thin as possible without cracking. It will be observed that the presence of impurities considerably impairs the malleability.

Exp. 6. Cut a strip of sheet lead about one-eighth of an inch wide, and observe how little force is required to produce rupture by applying a tensile stress. Instead of this strip, lead wire may be used.

Exp. 7. Test lead to show its feeble elasticity and the absence of sonorousness when struck, if solid. When, however, lead is cast in the shape of a mushroom, it becomes sonorous.

§ 25. Exp. 8. Melt 500 grains of grain tin; pour into the rectangular mould (Fig. 17 *e*) at as low a temperature as possible, taking precautions to prevent any dross getting into the mould; allow to cool without shaking or vibration. Notice the white, clear, and bright surface of the ingot when cold, which is a test of purity, also its full and rounded sides. The ordinary quality remains full, and rounded on the sides until the instant of becoming solid, when a frosted appearance will shoot out from the middle towards the sides, and a slight longitudinal depression will be produced on the surface. With a still commoner quality the colour becomes yellowish on cooling, and the appearance of the frosted crystalline markings takes place sooner, and completely covers the whole surface.

Bend the ingot of tin close to the ear, when a peculiar crackling sound known as the "cry" will be heard; this characteristic effect will be more marked the purer the tin.

Exp. 9. Melt and cast 1000 grains of tin; cool and fracture by bending backwards and forwards with a hammer. Notice the toughness of the metal and the absence of a definite structure on the fractured surface. In this respect it is similar to lead.

Re-melt, cast, and fracture when on the point of solidifying or melting. Notice the indefinite crystalline structure with the peculiar fern-like markings on the ruptured surface.

Exp. 10. Alloy 950 grains tin, 30 grains copper, and 20 grains lead, by melting together in a crucible with a

little charcoal. When alloyed, remove crucible from the fire and scrape the surface with a piece of stick until the surface ceases to oxidise, pour into rectangular mould, and allow to cool without shaking. Notice the longitudinal depression of the centre and the frosted appearance of the whole surface.

Exp. 11. Melt 950 grains tin with 20 grains of thin sheet iron and 30 grains lead; cast and cool as before. Notice the dull spotted appearance, and the character of the fracture, which should be finely granular, grey in colour, and the metal cold short.

Exp. 12. Melt 400 grains of tin, 10 grains of arsenic, and 600 grains of iron in a crucible with the lid luted on with clay. Break the pot, re-melt the alloy, and cast into a rectangular mould. Observe that the surface has a dull grey appearance, and that the nature of the fractured surface is of a steel-grey colour, finely granular, hard, and brittle. This is known as "hard head."

Exp. 12A. Melt 1000 grains of tin, and pour into a crucible containing 75 grains of red phosphorus which has been well rammed down. Rapidly place the crucible in a furnace, and stir with an iron rod until the mixture is uniform. Pour into an iron mould, and allow to cool. Observe that the metal is brittle, and that the fractured surface is highly crystalline. It probably contains about 5 per cent. of phosphorus.

Exp. 13. Test the malleability of pure and impure tin by flattening the various ingots with a hammer, or by rolling, observing the effect of impurities in hardening, and reducing the malleability. Test for tenacity (as in Exp. 6), which will be found to be very low as was the case with lead. Warm some tin, and notice the characteristic odour.

§ 26. Exp. 14. Weigh out 1000 grains of tough pitch copper, place in a clay crucible, and cover with charcoal. Heat in a brisk fire until melted. If the surface of the metal can then be seen, add more charcoal, and keep it in the fire a few minutes longer. The top of the pot may be covered with a piece of coke to keep the air away. Remove the crucible from the furnace, scrape the charcoal to one side, and pour the metal carefully into a rectangular mould (Fig. 17 *e*). When cool, tip the ingot out, and notice the longitudinal ridge on the upper surface which is characteristic of overpoled copper. Nick the centre with a steel chisel, place in a vice, and strike with a hammer until it breaks. Observe that it breaks somewhat short, that the fractured surface has a bright pale red colour, and is coarsely granular with some transverse fibres. This brittleness or cold-shortness is probably due to oxides of foreign metals such as iron, antimony, arsenic, etc., which have been reduced to the metallic state by carbon, and have alloyed with the copper.

Exp. 15. Melt 1000 grains of ordinary copper in a crucible without any charcoal, and heat to whiteness. Remove the crucible from the furnace; expose to the air until a thin crust of oxide forms on the surface, then shake the contents, so that the oxide may be dissolved by the copper, and pour into a warm rectangular mould. Allow to cool, and remove from mould. Observe the longitudinal furrow on the upper surface. Place it in the vice and strike vigorously with a strong hammer, when the ingot will break short, showing it to be brittle. Notice the dull deep purplish red colour of the fractured surface, the uneven finely granular structure, and the absence of fibre. It is technically known as "dry" copper, and the degree of dryness will depend on the proportion of oxygen dissolved. It is also called "underpoled" copper.

Exp. 16. Re-melt the dry copper obtained in the last experiment, with the addition of charcoal. When melted, partially remove the crucible from the fire, and stir the copper for a short time with a stick: this is termed *poling*; then pour into a rectangular mould, when, if the metal has been poled sufficiently, the ingot will present a flat surface having neither ridge nor furrow. Nick the centre when cold with a chisel, place in vice, and hammer backwards and forwards until it breaks. Observe its great toughness as compared with the overpoled and underpoled samples, its even close-grained structure when broken, its freedom from fibres or cavities, its bright metallic lustre, and fine salmon-red colour.

Exp. 17. Melt 500 grains of copper, and add a small piece of phosphorus very expeditiously to the crucible held with the tongs just inside the furnace; replace in the fire till all the uncombined phosphorus has burnt off. Repeat this addition of phosphorus two or three times, and then pour into rectangular mould. Notice the full appearance of the ingot with its longitudinal ridge resembling overpoled copper, and its lead-like colour. Fracture when cold, and observe its extreme brittleness and the facility with which it may be crushed to a fine powder; its bluish-white, coarse, semi-crystalline structure, and its hardness when tested with a file.

Exp. 18. Melt 500 grains of copper mixed with 5 grains of thin sheet iron, and add a bit of phosphorus about the size of a nut, with the precautions mentioned in Exp. 17, and pour into rectangular mould. Notice the full, convex, and compact nature of the ingot; its cold shortness when struck with hammer; its reddish grey close-grained fracture, very much resembling bronze, and its moderate hardness when tested with a file.

Exp. 19. Melt 500 grains of copper, and add 20 grains of metallic arsenic. When mixed, pour into rectangular

mould. Observe the longitudinal furrow resembling dry copper; its cold shortness, and the dull, close-grained, light reddish-grey appearance of the fractured surface.

Exp. 20. Melt 500 grains of copper, and add 10 grains of antimony. Notice that the effect is much the same as with arsenic. Some of the arsenic is lost by volatilisation in experiment 19, as is also some of the antimony in the present case.

Exp. 21. Place 500 grains of sheet copper in a crucible, well incorporate with an excess of white sand mixed with sufficient carbon to reduce the silica to silicon, lute on a lid and expose to a white heat for several hours. When cold, break open the pot, remove the metal which exists in several globules, and re-melt the whole so as to collect into one ingot. The surface will be slightly indented, and resembling gun metal in colour when filed. It can be rolled and hammered out in the cold; is much harder than copper, and fairly tough. If the temperature is too low, or not sufficiently prolonged, only a very little silicon will be reduced, and the metal will simply resemble slightly underpoled copper.

Exp. 22. Melt 500 grains copper with 10 grains of lead and cast into rectangular mould. Notice the longitudinal furrow, the cold shortness, and all the appearances of dry copper.

Exp. 23. Melt 500 grains of copper and add 25 grains of sulphide of copper; cast into rectangular mould. The surface of the ingot may be very rough, and the fractured surface very vesicular from the escape of gases. The structure may be partly crystalline and partly granular, while the colour and lustre may be bright in some parts and dull in others, resembling overpoled and underpoled copper. On re-melting and pouring, the ingot assumes the appearance and properties of moderately dry copper.

Exp. 23A. Mix 500 grains of sheet copper with 100 grains of black oxide of manganese and 25 grains of carbon, place in a crucible, cover with a layer of carbon, lute on lid, and heat strongly for two hours. When cold break open the crucible, collect any globules of metal, and re-melt the whole metal so as to collect into one ingot. The copper is whitened in proportion to the manganese alloyed with it.

§ 27. Exp. 24. Place 1000 grains of iron in a crucible and raise it to white heat, then drop in cautiously a bit of phosphorus, and repeat this at intervals until the iron melts. Remove from fire; cool; break the crucible, and detach the button of metal. Fracture and observe its brittleness, its white close-grained metallic appearance, inclining to crystalline, and its great hardness as tested by a file.

Exp. 25. Mix 1000 grains of iron with 20 grains of arsenic; place in clay crucible; lute on the lid with clay, and expose to the highest temperature of a wind furnace for an hour. Remove; cool; break the crucible, and extract the button of metal. Observe that it is brittle, hard, and that the fractured surface has a grey, granular appearance, resembling grey cast iron.

Exp. 26. Heat strongly for an hour in a covered crucible 1000 grains of iron mixed with 25 grains of ferrous sulphide. Allow to cool; break the pot, and remove the button of metal. Place the latter in a vice, and break with hammer and chisel. Notice its brittleness, hardness, and the white, granular appearance of the fractured surface, resembling white cast iron. There will probably be black patches on the surface, and also on the fractured part, which are characteristic of sulphur in iron.

Exp. 27. Place 1000 grains of hoop iron in a crucible and raise to a white heat, then add small bits of sulphur at intervals, until the whole is melted; then pour into half-round mould (Fig. 17 a); cool; fracture, and notice its brittleness, its coarse, stony appearance, its bronze-like colour, and generally its vesicular structure.

Exp. 28. Mix together in a crucible 400 grains of hoop iron and 300 grains of cast iron; lute on a lid, and expose to a white heat for an hour; allow to cool; break open the crucible, and extract the button of metal. Test the hardness with a file; observe the white granular texture, somewhat coarse, tending to crystalline, its general appearance resembling very hard coarse-grained steel.

Exp. 29. Cut up 1000 grains of hoop iron into small pieces, and thoroughly embed them in charcoal in a clay crucible; lute on the lid; place in a wind furnace, and keep the whole at a white heat for two hours; then allow the fire to burn down, remove the crucible, and when cold break open to detach the button of metal. Place the latter in a vice and break by means of a hammer and chisel. The substance will probably be a soft grey cast iron, with a medium sized granular fracture.

Exp. 30. Repeat experiment 29, with the addition of 200 grains of white sand mixed with the carbon. Keep the crucible at a white heat for three hours; allow to cool in the furnace; detach button of metal, and break in the vice as before. The substance will be harder than the former when tested with a file; of a light-grey or mottled appearance, a fine granular structure, and more compact than with carbon alone. Such a compound would probably contain about 5 per cent. of silicon.

Exp. 31. Heat strongly for an hour or two in a covered crucible, 500 grains of hoop iron cut up small, and embedded in white sand, and observe that the iron remains unaltered; showing that carbon or some other reducing agent is necessary to decompose the silica.

Exp. 32. Cut up 1000 grains of hoop iron; mix 100 grains of black oxide of manganese with carbon in excess; place the whole in a clay crucible. Lute on lid, and expose the whole to a very high temperature for two hours; allow to cool in the furnace, break crucible, detach metal, and break in vice as before. The fractured surface may present a white ground work, with almost regular concentric rings of dark stars, giving the whole the characteristic appearance of mottled cast iron. The iron, moreover, is soft when tested with a file.

§ 28. Exp. 33. Melt 10 grains of silver by placing in a red-hot cupel heated in a muffle, and keep in the molten condition for fifteen minutes. Observe that the metal does not tarnish in contact with the air. Now urge the muffle to a white heat, and maintain at that temperature for some time; the metal volatilises; cool out of contact with air very slowly; notice the clear surface; weigh and determine the loss due to volatilisation.

Re-melt and cool quickly in contact with air, when the oxygen, which has been mechanically absorbed, is disengaged on the metal solidifying, raising a blister on the surface, or covering the same with a number of small excrescences, giving it a frosted appearance. This action is called "spitting."

Exp. 33A. Re-melt the silver with 2 grains of copper, cool in air as before, and notice that the copper prevents "spitting."

Exp. 33B. Melt 10 grains of silver with sufficient

sodium chloride to cover the bead of silver, so that the metal cools under a layer of salt, and notice the even surface.

Exp. 33C. Re-melt with a quantity of nitre, instead of salt; cool as before, and observe the frosted appearance.

Exp. 33D. Re-melt the silver with $\frac{1}{3}$ rd its weight of gold, and notice that the phenomenon of "spitting" takes place.

Exp. 33E. Now alloy with more than $\frac{1}{3}$ rd of gold, cool in air, and notice that no "spitting" occurs.

Exp. 34. Melt 10 grains of silver in a cupel; add a little sulphur, and allow the bead to cool. Fracture, and notice the bluish-grey granular structure. Re-melt and heat to expel the greater part of the sulphur, cool, and observe that the metal cracks on the edges when rolled. Re-melt in a small crucible, with the addition of a bit of iron wire; pour into mould, and detach the iron regulus by striking the sides with a hammer, and prove that the silver now rolls without cracking.

Exp. 35. *To Prepare Pure Silver.*—Dissolve 100 grains of standard silver in dilute nitric acid in a beaker of about one pint capacity, then add distilled water till three parts full; heat nearly to boiling, and carefully add a hot saturated solution of common salt, until the whole of the silver is precipitated as chloride, stirring at the same time with a glass rod; allow to stand in a warm place until the liquid is clear, then decant off the greater part of the solution; add hot water; allow to settle; again decant the clear liquid, and repeat this three or four times. Now wash the precipitate on to a filter paper; wash two or three times with hot water,

and dry the whole. When dry, transfer paper and precipitate to a clay crucible; add twice the bulk of carbonate of soda, and fuse at a good red heat for about fifteen minutes; then pour into a rectangular mould, cool, and detach the slag, the last remnants of which may be removed by dissolving in hot water. Now roll out into a thin strip ready for use in other experiments.

§ 29. Exp. 36. Strongly heat 10 grains of fine gold in a muffle for some time, and observe that no oxidation takes place; remove; cool in air, and notice that there is no alteration of the surface due to liberation of oxygen, as is the case with silver under similar circumstances. Roll out the gold into a strip, and prove its great malleability. Cut with a knife to ascertain its softness.

Exp. 36A. Re-melt the gold in a small crucible with the addition of $\frac{1}{10}$ th of a grain of lead, and roll out as before. Notice that it now cracks on the edges, showing that this small amount of lead makes it brittle and alters its colour.

Exp. 37. Melt 10 grains of gold with $\frac{1}{10}$ th of a grain of iron. Cut with knife to test its hardness. Roll out thin, and notice that it cracks on the edges.

Exp. 38. Melt 10 grains of gold with $\frac{1}{10}$ th of a grain of antimony, and observe the same effect as with lead. (See Exp. 36A.)

Exp. 39. Melt 10 grains of gold in a small crucible; drop in a little sulphur; pour into mould, and observe that no change has taken place, the colour, softness, and malleability remaining unaltered.

Exp. 40. Dissolve a few grains of gold in aqua regia; evaporate to dryness to remove acid; re-dissolve in about a pint of distilled water; add a solution of sulphate of iron and heat, when the gold will be precipitated as a brown powder. Filter through a filter paper, wash residue with water, and warm till perfectly dry. Then carefully rub the brown powder with some smooth surface, when the ordinary yellow colour and lustre of gold will appear.

Exp. 41. The gold which has been rendered brittle in the previous experiments by alloying with lead, antimony, and iron, may be fused with a little nitre and double the quantity of carbonate of soda; pour into mould, cool, detach the slag, and roll out thin to test its malleability. In order to make it very tough use a little sal-ammoniac and charcoal, and fuse the metal for a few minutes. This flux should be added just as the gold is melting.

§ 30. Exp. 42. Take 500 grains of shot nickel containing about 99 per cent. of the metal; place in a clay crucible; lute on lid, and expose to a white heat for an hour. Remove crucible from fire; allow to cool; break open the pot, and examine the button of metal. Observe that it is white; malleable when hammered; hard, very tenacious, tough, and difficult to break. Also that the fractured surface is greyish white, and exhibits a mass of transverse fibres.

Exp. 43. Take 500 grains of the nickel used in the previous experiment; place in a blacklead crucible with 20 grains of carbon; lute on lid, and expose to a white heat for an hour. Remove; allow to cool; break open the pot, and extract the metal. Observe that the button is darker in colour, fairly malleable, hard, and moderately tenacious. The fractured surface is of a dark grey colour,

and granular, the fibrous structure having been destroyed by the union with carbon.

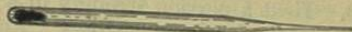
§ 31. Exp. 44. Take a portion of pure mercury; observe its silvery white colour; that it is tasteless and odourless; that it is liquid at ordinary temperatures; that it boils when heated to 350° C., and then begins to oxidise, forming HgO. Heat a portion strongly in a muffle, and observe that it completely volatilises. Rub together mercury and each of the following metals: Silver, gold, tin, lead, bismuth, zinc, iron, and copper. Notice that the iron and copper are less readily amalgamated than the others.

Exp. 45. Weigh out about 50 grains of mercury and add a little powdered zinc and antimony, but not sufficient to destroy the liquid character of the mercury, and cause the globule to roll down a slight incline. Observe that the spherical shape is not maintained as would be the case with pure mercury, but that the globule elongates, carrying a tail with it. Shake up the impure mercury in a flask, and observe a thin film of oxide on the surface impairing its lustre. Dissolve the metal in nitric acid, evaporate to dryness, and boil up with water, when a residue will be left owing to impurities.

Exp. 46. Rub together a portion of mercury and sulphur in a clear porcelain mortar for some time, when black sulphide will be formed. Place this in a porcelain crucible; cover; and heat gently for some time; cool, and observe the red crystals of vermilion. Place some of the sulphide in the centre of a hard glass tube open at both ends; rest the latter in a horizontal position on the ring of a retort stand; heat the portion containing the sulphide by means of a Bunsen burner, and notice the globules of mercury on the cooler portions of the tube,

showing the decomposition of this sulphide by the combined agency of heat and air.

Exp. 47. Take a hard glass tube about a foot long closed at one end, and introduce a little mercury, then draw out the open end with a table blowpipe to a fine point thus—



Heat the end containing the metal till it has volatilised, and observe the condensed mercury at the narrow part, proving that mercury may be readily distilled.

§ 32. Exp. 48. Melt 1000 grains of zinc, and pour into a rectangular mould; cool down to about 150°C .; place on two supports, and strike the centre several times with a hammer, when it will bend but not break. Hammer out one end of the ingot on the anvil; cut it off with a chisel; warm again if necessary, and roll into a thin sheet. It may require annealing once or twice by heating to about 200°C . during the process. The other half of the ingot will by this time have cooled down to the ordinary temperature; place it in vice, and strike with hammer, when the metal will break off short, showing its crystalline structure, and brittleness at the ordinary temperature. Heat a portion to a temperature of 200° to 300°C ., and prove the brittleness at that point as well as when cold. Hammer out another portion at 150°C .; allow to cool, and then break in the vice. Notice that the crystalline structure has been destroyed by forging, the metal having become close-grained and minutely crystalline.

Exp. 49. Heat 500 grains of zinc with 20 grains of clean iron filings in a covered crucible for an hour at a moderate temperature; remove from fire, and pour into

an iron mould; fracture, and observe that the crystals are much smaller than in pure zinc, being partly replaced by a granular groundwork, and that the lustre is less bright.

Exp. 50. Mix together in a crucible two parts of lead and one of zinc, well melt together, and pour into the upright taper mould (Fig. 17 *c*), which should have been previously heated and blacklead. When cold, knock out the ingot of metal. By close inspection of the exterior, a line will be observed where the two metals have separated; place in vice up to this mark as near as possible, and give it a few sharp blows with a hammer, when it will break off short. Notice the dull grey granular appearance of the fractured surface interspersed with a number of small crystals; also that the lower end of the ingot consists of lead which is very malleable, and the upper end of zinc which is highly crystalline and brittle.

Exp. 51. Melt 500 grains of zinc, drop in two or three bits of phosphorus, and cast into rectangular mould (Fig. 17 *e*). The phosphorus increases the brilliancy and lengthens the crystalline plates which are arranged transversely at an angle of 30 to 40 degrees in a definite manner, while the zinc before adding the phosphorus appears more irregularly crystalline.

Exp. 52. Melt 500 grains of zinc and then drop in 20 grains of arsenic—the effect is much the same as with phosphorus.

Exp. 53. Melt 50 grains of zinc in an open crucible so that air may have free access, using a hot fire; observe that the zinc is volatile, burning with a brilliant bluish white flame, forming a very light white powder, which is oxide of zinc.

§ 33. Exp. 54. Melt 300 grains of antimony in a small crucible, and when partially cool, pour out the still liquid portion; cover with lid; allow to cool; and observe the rhombohedral crystals.

Exp. 55. Melt 300 grains of antimony in a crucible; remove from fire; scrape the surface with a piece of wood until it remains bright and no film of oxide forms; pour into iron mould, and observe the nature of the surface. Before melting, crush up the metal in a mortar to prove its extreme brittleness.

Exp. 55A. Raise 100 grains of antimony to a red heat; notice that it takes fire, burning with a white flame, and forming the white trioxide. If commercial antimony be used, it will probably exhale the characteristic garlic odour of burning arsenic with which it is contaminated.

Exp. 56. Roast 50 grains of powdered commercial antimony in a muffle, and observe the odour of sulphur dioxide, showing the presence of sulphur in the metal.

Exp. 57. Melt 100 grains of common antimony with a little hoop iron; pour into round mould; cool, and observe the crust of iron sulphide on the surface of the metal.

CLAYS, SILICATES, AND SLAGS.

§ 34. Exp. 58. Measure out four medium-sized crucibles full of good fire-clay and pound in an iron mortar until sufficiently fine to pass through a sieve with 40 to 60 meshes to the inch. Then take two measures of crushed clay, which has been previously baked, pass through the same sieve, and mix the two lots together. Now weigh out three lots of 1000 grains each and reserve them for

experiments 59, 60 and 61. To the remainder add water, and knead the whole to the consistence of dough. Then shape a piece in the form of a pyramid by cutting with a knife or spatula, also cut a rectangular base for the pyramid to rest on; dry them both at about 100° C.; place in a cold gas muffle; gradually increase the heat to redness, and maintain at that temperature for fifteen minutes. Remove; place in a crucible; cover with lid, and expose to the highest temperature of a wind furnace for half an hour; remove from fire; cool, and observe the character of the sharp edges. If they show no signs of fusion, the clay is very refractory; if incipient fusion only has occurred, the clay is moderately refractory.

Exp. 59. Mix with one of the 1000 grains of clay, reserved from Exp. 58, ten per cent. of lime or oxide of iron or five per cent. of each; make a pyramid as previously described, bake, and then heat strongly. Notice that the clay partially fuses.

Exp. 60. Repeat Experiment 59, but with the addition of five per cent. of sodium carbonate in place of the lime or oxide of iron, and notice that the clay completely fuses.

Exp. 61. Repeat Experiment 59, but with the addition of three to four per cent. of iron pyrites instead of the lime or oxide of iron. (See § 18 a.)

Exp. 62. From the clay prepared in Experiment 58, make a few small crucibles and lids as described (§ 18 e); carefully dry, and bake in muffle. Line three or four of these with the charcoal brasque mentioned in § 18 h; dry, and carefully heat with lid on in a muffle until all flame ceases; remove and cool with lid on, otherwise some of the carbon will burn away.

Exp. 63. Test the power of resisting corrosion of the unlined crucibles by observing how long 100 grains of