

§ 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

litharge can be kept melted without eating its way through the bottom of the crucible.

Exp. 64. Take one part of raw clay by measure and an equal amount of crushed coke; well knead together with water, and mould into crucibles, as in Experiment 62. Dry, bake, and test the refractory power as before. Instead of the coke powder, good native graphite may be used.

§ 35. Exp. 65. Take 200 grains of white sand; place in crucible; lute on the lid, and expose to a high temperature for two hours. Notice that it remains unmelted.

Exp. 66. Take 348.8 grains of silica* and 651.2 grains of lime; place in clay crucible lined with charcoal paste; lute on the lid; expose to a white heat for two hours, and observe that the mass is infusible at that temperature. These proportions correspond to the formula 2CaO.SiO_2 , the ratio of the oxygen in the acid to that of the base being as 1 : 1.

Exp. 67. Take 517.2 grains of silica and 482.8 grains of lime and expose in a covered and luted crucible as before to a white heat for two hours. Notice that the mass is well melted, compact, and has a glassy fracture. This silicate corresponds to the formula CaO.SiO_2 , the ratio of oxygen in the acid to that in the base being as 2 : 1.

Exp. 68. Take 445.5 grains of silica and 554.5 grains of lime and proceed as before. The mixture melts and forms a compact slightly crystalline silicate, the formula of which is 4CaO.3SiO_2 , the oxygen ratio of acid to base being as 3 : 2.

Exp. 69. Mix together 211.3 grains of silica and 788.7

* Ca=40 . O=16 . Si=28.

grains of lime; heat strongly for two hours as before. Observe that the mixture does not melt. This corresponds to the formula 4CaO.SiO_2 , the oxygen ratio of acid to base being as 1 : 2.

Exp. 70. Mix together 616.4 grains of silica and 383.6 grains of lime in a brasqued crucible; cover; lute on lid, and expose to a white heat for two hours. Observe that the mass is unmelted, or only partly melted into a compact glassy slag. This mixture when combined would have the formula 2CaO.3SiO_2 , the oxygen ratio of acid to base being as 3 : 1.

(a) From the foregoing series of experiments it will be observed that silicates of lime having a composition corresponding to CaO.SiO_2 and 4CaO.3SiO_2 are fusible at the temperature of an assay furnace. That 2CaO.3SiO_2 is partly fusible; and that 2CaO.SiO_2 and 4CaO.SiO_2 are infusible, which shows that an excess of silica is an advantage, and an excess of lime a disadvantage in inducing fusibility in slags.

§ 36. The fusibility or otherwise of the corresponding silicates of magnesia may be determined in a similar way to those of lime.

Exp. 71. Take 428.6 grains of silica and 571.4 grains of calcined magnesia (obtained by igniting the carbonate); place in a brasqued crucible; lute on lid, and expose to a white heat for two hours. Observe that the substances only partially fuse into a white hard semi-crystalline mass. These proportions correspond to 2MgO.SiO_2 , with oxygen ratio of acid to base as 1 : 1.

Exp. 72. Take 600 grains of silica and 400 grains of magnesia. Heat as above, and notice that the mass only just melts, or partly melts. The formula is MgO.SiO_2 and oxygen ratio of acid to base as 2 : 1.

Exp. 73. Take 529.4 grains of silica and 470.6 grains of magnesia. Heat as before. The mass is well melted and has a crystalline fracture. The formula is $4\text{MgO} \cdot 3\text{SiO}_2$, and oxygen ratio of acid to base as 3 : 2.

Exp. 74. Heat as before 272.7 grains of silica and 727.3 grains of magnesia. The mass does not melt, but frits together. The formula is $4\text{MgO} \cdot \text{SiO}_2$, and oxygen ratio of acid to base is as 1 : 2.

Exp. 75. Heat as before 692.3 grains of silica and 307.7 grains of magnesia. The mass melts, forming a hard porous substance corresponding to the formula $2\text{MgO} \cdot 3\text{SiO}_2$; the oxygen ratio of acid to base is as 3 : 1.

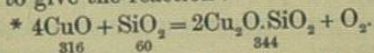
(a) With regard to the above experiments on silica and magnesia, we find that those substances having a composition corresponding to $4\text{MgO} \cdot 3\text{SiO}_2$ and $2\text{MgO} \cdot 3\text{SiO}_2$ are fusible; that $\text{MgO} \cdot \text{SiO}_2$ is just fusible; that $2\text{MgO} \cdot \text{SiO}_2$ is partly fusible; and that $4\text{MgO} \cdot \text{SiO}_2$ is infusible.

§ 37. No silicate of alumina is completely fusible at the highest temperature of an assay furnace, but the proportions requisite to form the compounds represented by the formulæ $\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$ and $2\text{Al}_2\text{O}_3 \cdot 9\text{SiO}_2$ respectively soften at a high temperature, which may be verified by heating in brasqued crucibles, with lids luted on (Fig. 34), 643 grains of silica with 357 grains of alumina, and 730 grains of silica with 270 grains of alumina. The alumina may be obtained by strongly calcining sulphate of alumina.

(a) Although, as stated above, silicates of alumina are infusible alone, the addition of a small amount of another base greatly reduces their melting points. The substance commonly added for this purpose is lime. Magnesia may also be used, but it is much less fluxing than lime. The most fusible compounds of lime and alumina are comprised between those in which the oxygen of the

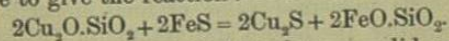
silica is double the sum of that of the lime and alumina, and those in which the oxygen of the silica is half the sum of that of the lime and alumina; and these compounds are fusible in proportion as the relation between the bases approaches that of $6\text{CaO} : \text{Al}_2\text{O}_3$. They still melt well when the relation is as $3\text{CaO} : \text{Al}_2\text{O}_3$; but they become much less fusible when the relation is as $3\text{CaO} : 2\text{Al}_2\text{O}_3$. The composition of clays, which are richest in alumina, may, with some exceptions, be expressed by the formula $2\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$. Hence it follows that by the addition of an amount of lime intermediate between 3CaO and 6CaO , or the equivalent in carbonate of lime, they ought always to melt well; and that the fusion ought to be still more easy when, in addition to lime, silica is added in a proportion ranging from SiO_2 to 4SiO_2 .

§ 38. Exp. 76. *To Prepare Silicate of Copper.*—Take 300 grains of the black oxide of copper (CuO) and sufficient white sand to give the reaction:—



Well mix the two substances together, place in a scorifier, and heat strongly in a muffle for half an hour, when the ingredients will be fritted together without melting.

Exp. 77. Take 200 grains of the above silicate of copper, crush up fine, and mix with sufficient ferrous sulphide to give the reaction:—

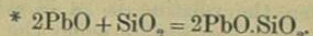


Place the mixture in a crucible; lute on a lid, and heat strongly for half an hour in a furnace. Remove from fire; allow to cool; then break the crucible, and describe the contents, which consist of a dark bluish grey fine-

* $\text{Cu} = 63 : \text{Si} = 28 : \text{Pb} = 207.$

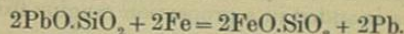
grained copper regulus at the bottom, and a black glassy slag of silicate of iron on the top.

§ 39. Exp. 78. *To Prepare Silicate of Lead.*—Take 300 grains of litharge, mix with sufficient sand to give the reaction:—



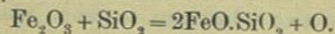
Place the mixture in a roasting dish, and heat at a low temperature in a muffle, so as to frit the ingredients together, or melt together in a crucible and pour into a mould. Describe the result.

Exp. 79. Take 200 grains of the above silicate, 10 grains of charcoal, and sufficient iron, to give the reaction:—



Place in crucible, and heat for about 15 minutes, until all action ceases, then pour into mould, and describe the result.

§ 40. Exp. 80. *To Prepare Silicate of Iron.*—Take 300 grains of powdered hematite and sufficient sand to give the reaction:—

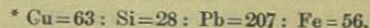


Heat together in a crucible; allow to cool; then break the pot, and describe the result.

EXAMINATION OF FUELS.

§ 41. The industrial value of fuels depends on the combustibility, the length and volume of the flame, the purity, the calorific power, and the calorific intensity.

Exp. 81. The temperature at which a given fuel inflames may be determined by passing hot air over it at



different temperatures in a glass tube. In this way dry peat has been found to inflame at 225° C.; pine charcoal at 280° C.; pine wood at 300° C.; ordinary coal at about 350° C.; coke and anthracite at a nascent red heat. The igniting points of charcoal and coke depend upon the temperature at which they have been produced; the higher this point, the less inflammable they become. The most inflammable kinds of wood and coal are those which are richest in hydrogen.

Exp. 82. Weigh a lump of wood of a convenient size to go inside an ordinary clay crucible, cover with a lid, place inside a furnace, and observe the character of the flame produced from its decomposition. It will be voluminous, white, and mixed with dense fumes of tarry matter. When all flame ceases remove the crucible, taking care to keep the lid on until quite cold; then weigh the charcoal produced, and estimate the percentage obtained from the sample of wood taken.

This should be repeated with different kinds of wood.

Exp. 83. Repeat the last experiment with a weighed portion of compressed peat.

Exp. 84. Take a lump of lignite weighing about 2000 grains; crush to a powder in an iron mortar, and weigh 500 grains. Place in a clay crucible; cover, and heat in a furnace. Notice the long smoky flame, the disagreeable odour peculiar to itself, and very different from that of ordinary coal. Remove from furnace; cool with lid on, and weigh the carbon residue. This residue retains the form and colour of the lignite from which it has been produced.

Exp. 85. Weigh 500 grains of a sample of ordinary coal which has been crushed to powder, and treat as in Exp. 84. Observe that the flame is less voluminous, and the odour quite different; that the particles have caked

together, forming a more or less dense and compact coke according to the temperature and duration of the coking operation, and that a larger percentage of fixed carbon remains. Its colour will be greyish black, with a semi-metallic lustre, especially if a high and prolonged temperature has been employed. Notice also that coke is much less combustible than charcoal.

Exp. 86. Repeat Experiment 85 with 500 grains of powdered anthracite. Observe that the flame in this case is very short, and the powdered fuel presents much the same appearance at the conclusion as before the coking operation, still retaining its pulverulent form, showing that it is non-caking; also note that the amount of the fixed residue is greater than in any of the above cases.

Exp. 87. Take different samples of coal, crush to a fine powder, and pass through a sieve with 80 meshes to the inch; weigh out 50 grains of each; place in small scorifiers, and heat in a muffle until all the carbon is removed and a white or reddish white ash remains; cool and weigh to obtain the amount of ash, and as 50 grains were taken, the result, when multiplied by 2, will give the percentage.

Exp. 88. Weigh out 50 grains each of wood and peat, and decarbonise, as with coal, to determine the amount of ash present in each case.

Exp. 89. The estimation of moisture may be made by heating 25 grains of finely powdered coal in a water oven for an hour, cooling and weighing. First carefully dry a pair of watch glasses, having ground edges so as to fit accurately, and obtain their exact weight. Then weigh in one of them the coal to be examined, and expose the whole to a temperature of 100° C. for an hour; cool and weigh. Notice the loss; repeat the experiment for

another half hour; again weigh, and repeat until the weight is constant.

Exp. 90. To determine the amount of sulphur in coal; take 50 grains of finely powdered coal, and oxidise the sulphur present into sulphate; then precipitate the sulphuric acid with barium chloride.

1st Method. Powder a quantity of pure sodium chloride and nitre; dry at 100° for some time, then incorporate the 50 grains of dry coal with 500 grains of a mixture consisting of $\frac{2}{3}$ sodium chloride and $\frac{1}{3}$ nitre. Half fill a porcelain crucible with this mixture; partly cover with lid, and heat gently over a gas flame until the mass is completely deflagrated, which may be known by its white colour; then add more of the mixture, and repeat the heating until the whole of the coal has been oxidised. Allow the crucible to cool, place it in a large evaporating basin; cover with water; heat to boiling, and continue the ebullition until all soluble matter is dissolved. Remove the crucible and carefully wash it with hot water. Now add hydrochloric acid to the solution, and heat until all gases are expelled. Then filter off any insoluble matter, allowing the filtrate to run into a beaker; again boil and add a solution of barium chloride, stirring at the same time with a glass rod, until all the sulphate is precipitated; allow to stand in a warm place for the solution to clear; filter; well wash several times with hot water; dry; then brush off the precipitate on to a sheet of glazed paper; burn the filter paper completely in a weighed porcelain crucible; then add the precipitate and again ignite for some time; allow to cool in a desiccator, and weigh the barium sulphate (BaSO_4). If the paper be of the kind now sold as ashless, no deduction need be made for its ash. Then as 233 : 32 = weight of ppt. : the S present : and as 50 grains of the fuel were taken, the result when multiplied by 2 will give the percentage of sulphur present.

2nd Method. Mix 50 grains of coal with 100 grains of pure quick lime, place in a roasting dish, and cover the mixture with another 100 grains of lime, then strongly heat in a muffle for an hour, or until the mass is quite white. Digest the roasted mass with hydrochloric acid, filter off any insoluble residue, and treat the filtrate with barium chloride and proceed as in the former method.

Exp. 91. To determine the calorific power of a given fuel, carefully dry a quantity of finely powdered potassium chlorate and nitrate in separate dishes. Also a quantity of the fuel very finely powdered should be dried, for half an hour at least, in a water oven. Weigh out 30 grains of the dried coal, 225 grains of potassium chlorate, and 75 grains of nitre; well mix and introduce into the perfectly dry copper tube of what is known as Thompson's or Wright's calorimeter; ram it moderately tight; insert a small piece of "fuse" (prepared by soaking cotton wick in a solution of nitre and drying it), and place the tube in the stand ready for the experiment. Now take the temperature of the water in the large vessel; light the fuse; cover with the bell tube, taking care that the tap is closed, and put the whole in the water before the ignited fuse has had time to burn down to the mixture. When the combustion appears complete, open the tap so that the water may absorb the residual heat in the tube, and again take the temperature of the water. The increase of temperature, plus 10 per cent. added for loss, gives the evaporative power of the fuel.

EXAMPLE:

Temp. of water before the experiment	=	55° Fahr.
" " after " "	=	66° "
" produced by the combustion,	=	11
Plus 10%.	=	1.1
Total evaporative effect,	=	<u>12.1</u>

i.e., 1 lb. of the coal on perfect combustion will generate 12.1 lbs. of steam.

(a) The apparatus (Fig. 46) consists of a tall glass cylindrical vessel *a* capable of holding 60,000 grains of water. At a given distance a mark is scratched on the outside to indicate 29,010 grains of water, which equals 967×30 (the former figure being the latent heat of steam and the latter the weight of fuel taken). A copper vessel (*b b*) fitted at the top with a stopcock, and a perforated stand *c*, into the socket of which the copper crucible *d* fits. This stand has 3 springs *e* for keeping the cylinder *b* tightly fixed to the stand.



Fig. 46. *

Note.—The quantity of water used is immaterial, provided its weight is known, and that there is sufficient to cover the lower portion of the copper vessel *b b*.

(b) The calorific value of fuel may be estimated in "calories" or in vapour units. The calorie represents the heat absorbed by the unit weight of water when its temperature is raised one thermometric degree, the centigrade scale being most generally employed. The vapour unit was proposed by Rankine, and corresponds to 537 calories if the Centigrade scale be used, and 967 calories for the Fahrenheit scale. In order to express calories in vapour units, divide by 537 or 967 respectively. To convert vapour units into calories multiply by these numbers. Take the example given above, *viz.*, 12.1 vapour units; $12.1 \times 967 = 11,700.7$ calories.

SPECIFIC GRAVITY OF SOLIDS.

§ 42. By this term is understood the ratio of the weight of a body to the weight of an equal bulk of another body taken as a standard. Pure distilled water is almost invariably employed for this purpose. As the volume of bodies is altered by heat it is necessary to conduct the experiment at a known temperature, which is generally $15^{\circ}5$ C. There are several ways by which the density may be determined, but the most useful and generally applicable is as follows:—

Exp. 92. The body is suspended by a horse hair from the pan of a specific gravity balance, and accurately weighed in air; then it is completely immersed in a beaker of distilled water placed underneath the pan and reweighed. The body will now appear lighter than before, the loss in weight being exactly equal to the weight of an equal volume of the water; so that a body immersed in water loses a portion of its weight equal to that of the displaced water. Now place weights in the short pan, *i.e.*, the one from which the body is suspended, to counterbalance this diminution and observe the amount added. Then divide the weight in air by the loss of weight in water, and the result gives the specific gravity.

Suppose the body is lighter than water. Take its weight in air as before, then the weight in water of a piece of lead sufficient to sink it, and the weight in water of the light substance with the lead attached. The density D is then obtained by the following equation.

$$D = \frac{\text{Weight of substance in air.}}{\text{Wt. of lead} - (\text{Wt. of lead} + \text{light subs.}) + \text{Wt. of substance in air.}}$$

In more accurate work it is usual to reduce the weight of the substance to its weight in vacuo, but for further information the student is recommended to refer to the "Theory and Use of a Physical Balance," by James Walker, M.A. (Clarendon Press, 1887.)

Exp. 93. Suppose the body to be soluble in water or decomposed by that liquid; then substitute for the water some liquid in which the body is insoluble, oil of turpentine or spirits of wine being generally applicable. Weigh in air as before, then note the difference when weighed in turpentine; this gives the weight of a volume of turpentine equal to that of the substance. The specific gravity of oil of turpentine compared with water is known, and as the specific gravity of turpentine is to the specific gravity of water, so is the weight of a bulk of turpentine equal to that of the body, to the weight of a bulk of water equal to that of the body. The weight in air of the body is then divided by the latter. Let A = the weight of body in air: A' = the weight of body in turpentine. Then the weight of a bulk of turpentine equal to that of the body is $A - A' = A''$. Let S = the density of the turpentine, and S' = the density of water; then $S : S' :: A'' : x$, and the specific gravity of the body = $\frac{A}{x}$.

The density of oil of turpentine varies from .86 to .88.
 " " alcohol is .794 at $15^{\circ}5$ C.

Exp. 94. The specific gravity of a metal or alloy may be determined approximately by removing the pan and suspension of an ordinary laboratory balance, carefully counterpoising by wrapping sheet lead round the end of the beam, and suspending the metal by means of a horse hair, weighing in air, then taking the weight when suspended in water, and calculating as described in Exp. 92.

Exp. 95. Determine by the above method the specific gravity of lead, iron, copper, zinc, tin, etc. Also various alloys, such as gun metal containing 900 parts by weight of copper and 100 parts of tin. Now calculate the density of the gun metal, and notice whether it agrees with the experimental determination thus:—

Let the weight of the above gun metal in air = 1000 grains. The amount of copper present in the alloy is therefore 900 grains and the tin 100 grains.

$$\begin{array}{rcl} \text{Weight of copper in air} & = & 900 \\ \text{'' '' water} & = & 900 - x \end{array}$$

The difference between these weights = x

And $\frac{900}{x} = 8.8 =$ the specific gravity of copper.

$$\begin{array}{rcl} \text{Weight of tin in air} & = & 100 \\ \text{'' '' water} & = & 100 - y \end{array}$$

The difference between these weights = y

And $\frac{100}{y} = 7.3 =$ the specific gravity of tin.

$$\text{Now } x = \frac{900}{8.8} = 102.3$$

$$\text{And } y = \frac{100}{7.3} = 13.7$$

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Therefore 116 is the weight of an amount of water equal in bulk to 1000 grains of the alloy, and $\frac{1000}{116} = 8.62 =$ the specific gravity of the bronze.

The following formula for calculating the theoretical density on the assumption that the union of two metals

has been attended neither by contraction nor expansion will show whether the alloy has actually expanded or contracted:—

$$\frac{W}{D} = \frac{w}{d} + \frac{w_1}{d_1}$$

W and D representing the weight and density of the resulting alloy, and w, w_1, d, d_1 the same with regard to the constituents.