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CONTRIBUTIONS

TO

BLOWPIPE-ANALYSIS.

By E. J. CHAPMAN, Ph. D.,

PROFESSOR OF MINERALOGY AND GEOLOGY IN UNIVERSITY COLLEGE, TORONTO,
AND CONSULTING MINING ENGINEER.

TORONTO, CANADA WEST:
PRINTED BY LOVELL & GIBSON, 67 YONGE STREET.
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CONTENTS.

CONTRIBUTIONS

TO

BLOWPIPE-ANALYSIS.

1. DETECTION OF LITHIA IN THE PRESENCE OF SODA.

[*First published in the Chemical Gazette: November* 15, 1850.]

This test may be applied to mixtures of these alkalies in the simple state, or to their carbonates, sulphates, nitrates, or other compounds capable of being decomposed by fusion with chloride of barium. The test-substance, in powder, is to be mixed with about twice its volume of chloride of barium, and a small portion of the mixture is to be exposed on a loop of platinum wire to the point of a well-sustained oxidating flame. A deep-yellow coloration of the flame-border, produced by the volatilization of chloride of sodium, at first ensues. This gradually diminishes in intensity, and after a short time, a thin green streak, occasioned by chloride of barium, is seen to stream from the point of the wire, as the test-matter shrinks further down into the loop. On the fused mass being then brought somewhat deeper into the flame, the point and edge of the latter will at once assume the rich crimson tinge characteristic of the presence of lithium compounds; and the colour will endure sufficiently long to prevent the slightest chance of misconception or uncertainty. The presence of strontium compounds does not affect this reaction, as these compounds, when fused with chloride of barium, cease to impart a red color to the flame (see No. 2). In order to ensure success, or rather to avoid the least risk of failure, in the application of this test, it is only necessary to keep up a clear and sharply-defined flame for about a couple of minutes. If the red coloration do not appear by that time, the absence of lithia

—unless the latter substance be present in traces only—may be safely concluded.*

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2. METHOD OF DISTINGUISHING THE RED FLAME OF LITHIA FROM THAT OF STRONTIA.

[*First published in the Chemical Gazette: May* 1, 1848.]

It has been long known that the crimson coloration imparted to the blowpipe flame by strontia, is destroyed by the presence of baryta. This reaction, confirmed by PLATTNER—see, more especially, the *third* edition of his *Probirkunst*, page 107—was observed as early as 1829 by BUTZENGEIGER (*Annales des Mines*, *t.* v., *p.* 36). The latter substance, however, as first indicated by the writer, does not affect the crimson flame-coloration produced by lithia. Hence, to distinguish the two flames, the test-substance may be fused with 2 or 3 volumes of chloride of barium in a loop of platinum wire, the fused mass being kept just within the point or edge of the blue cone. If the original flame-coloration proceeded from strontia (or lime), an impure brownish-yellow tinge will be imparted to the flame-border; but if the original red colour were caused by lithia, it will not only remain undestroyed, but its intensity will be much increased.

This test may be applied, amongst other bodies, to the natural silicates, *Lepidolite*, *Spodumene*, &c. It is equally available, also, in the examination of phosphates. The mineral *Triphylline*, for example, when treated *per se*, imparts a green tint to the point of the flame, owing to the presence of phosphoric acid; but if this mineral be fused (in powder) with chloride of barium, a beautiful crimson coloration in the surrounding flame-border is at once produced.

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* In testing this method, a mixture was prepared of 2 parts of ignited carbonate of soda with 1 part of carbonate of lithia, and portions of this were placed in six little porcelain capsules, distinguishing upon their under sides by a spot of ink; whilst into six similar but unmarked capsules, some carbonate of soda, only, was placed. The capsules being then arranged indiscriminately upon a tray, each was separately examined, and it was found that those which contained lithia could be separated from the rest without the slightest difficulty (November, 1850). This plan was repeated with equal success, on mixtures of 3 (NaO, CO^2) + 1 (LiO, CO^2), and 6 (NaO, CO^2) + 1 (LiO, CO^2), in May, 1865. When the lithia is in very small quantity, the blowpipe flame must not be too large.

3. DETECTION OF ALKALIES IN THE PRESENCE OF MAGNESIA.

[*First published in the Chemical Gazette: September*, 1847.]

In the analysis of inorganic bodies, magnesia and the alkalies (if present) become separated from other constituents towards the close of the operation. In continuation of the analysis, it then becomes desirable to ascertain, at once, whether magnesia is alone present, or whether the saline mass, produced by the evaporation of a portion of the solution, consists of magnesia and one or more of the alkalies, or of the latter only. By fusing a small quantity of the test-matter with carbonate of soda, the presence of magnesia is readily detected, as this substance remains undissolved; but the presence or absence of alkalies is not so easily determined, the coloration of the flame being frequently of too indefinite a character to afford any certain evidence on this point. The question may be solved, however, by the following simple process. Some boracic acid is to be mixed with the test-matter and with a few particles of oxide of copper, and the mixture is to be exposed for a few seconds, on a loop of platinum wire, to the action of an oxidating flame. In the absence of alkalies, the oxide of copper will remain undissolved; but if alkalies be present, an alkaline borate is produced, forming a readily fusible glass, in which the copper oxide is at once dissolved, the glass becoming green whilst hot, and blue when cold. If magnesia also be present, white specks remain for a time undissolved in the centre or on the surface of the bead. Any metallic oxide which imparts by fusion a colour to alkaline borates, may, of course, be employed in place of oxide of copper; but the latter has long been used in other operations, and is therefore always carried amongst the reagents of the blowpipe-case.

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4. REACTION OF MANGANESE SALTS ON BARYTA.

[*First published in Chemical Gazette: August* 1, 1846.]

When moistened with a solution of any manganese salt, and ignited in an oxidating flame, Baryta and baryta compounds, generally, assume on cooling a blue or greenish-blue colour. This arises from the formation of a manganate of baryta. Strontia and other bodies (apart from the alkalies) when treated in this manner, become brown or dark-grey. A mixture of baryta and strontia also assumes an

indefinite greyish-brown colour. If some oxide of manganese be fused with carbonate of soda so as to produce a greenish-blue bead or "turquoise enamel," and some baryta or a baryta salt be melted into this, the colour of the bead will remain unchanged; but if strontia be used in place of baryta, a brown or greyish-brown enamel is produced.

NOTE:—Some examples of *Witherite, Barytine, and Baryto-calcite,* contain traces of oxide of manganese. These, after strong ignition, often assume *per se* a pale greenish-blue colour.

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5. DETECTION OF BARYTA IN THE PRESENCE OF STRONTIA.

[*First published in the Chemical Gazette: August* 1, 1846.]

This test is chiefly applicable to the detection of baryta in the natural sulphate of strontia; but it answers equally for the examination of chemical precipitates, &c., in which baryta and strontia may be present together. The test-matter, in fine powder, is to be melted in a platinum spoon with 3 or 4 volumes of chloride of calcium, and the fused mass treated with boiling water. For this purpose, the spoon may be dropped into a test-tube, or placed (bottom upwards) in a small porcelain capsule. The clear solution, decanted from any residue that may remain, is then to be diluted with 8 or 10 times its volume of water, and tested with a few drops of chromate (or bichromate) of potash. A precipitate, or turbidity, indicates the presence of baryta.

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6. ACTION OF BARYTA ON TITANIC ACID.

[*First published in Chemical Gazette:* 1852.

Fused with borax in a reducing flame, Titanic acid forms a dark amethystine-blue glass, which becomes light-blue and opaque when subjected to the flaming process. The amethystine colour arises from the presence of Ti^2O^3: the light-blue enamelled surface, from the precipitation of a certain portion of TiO^2. The presence of baryta, even in comparatively small quantity, quite destroys the latter reaction. When exposed to an intermittent flame, the glass (on the addition of

baryta) remains dark-blue, no precipitation of titanic acid taking place. Strontia acts in the same manner, but a much larger quantity is required to produce the reaction.

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7. DETECTION OF OXIDE OF MANGANESE WHEN PRESENT IN MINUTE QUANTITY IN MINERAL BODIES.

[*First published in the Philosophical Magazine: February*, 1852.]

It is usually stated in works on the Blowpipe, that the smallest traces of maganese may be readily detected by fusion with carbonate of soda, or with a mixture of carbonate of soda and nitrate of potash: but this statement is to some extent erroneous In the presence of much lime, magnesia, alumina, scsquioxide of iron, or other bodies, insoluble, or of difficult solubility, in carbonate of soda, traces of oxide of manganese may easily escape detection. By adding, however, a small portion of borax or phosphor-salt to the carbonate of soda, these bodies become dissolved, and the formation of a "turquoise enamel" (manganate of soda) is readily effected. The process may be varied by dissolving the test-substance first in borax or phosphor-salt, and then treating the fused bead with carbonate of soda: the latter being, of course, added in excess. By this treatment, without the addition of nitrate of potash, the faintest traces of oxide of manganese in limestone and other rocks, are at once made known.

NOTE:—This method of examining bodies for the presence of manganese, was recommended by *Dr. Leop. H. Fischer* in 1861 (*Leonh. Jahrbuch:* [1861] 653), but the writer had forestalled him by nine years, having already described it in 1852—a fact apparently unknown to the editor of the 4th edition of Plattner's *Probirkunst.**

* This new edition of Plattner's treatise, although containing some valuable additions from the pen of its editor, *Dr. Theodor Richter*, is not altogether free from errors of omission. One of these, the writer may perhaps be allowed to point out on personal grounds. In the third edition, p. 273, *Plattner* states under the head of cryptolite,—"Das Verhalten dieses seltenen Minerals vor dem Löthrohre ist noch nicht ermittelt." In the new edition, *Dr. Richter* expands this statement as follows:—"Kryptolit (Phosphocerit)—Das Löthrohrver halten dieses seltenen Minerals, welches beim Auflösen des grünen and röthlichen Apatits von Arendal, sowie des gerösteten Kobaltglanzes von Johannisberg in Schweden, in Säuren zurückbleibt, ist noch nicht ermittelt." Now, the blow-

8. METHOD OF DISTINGUISHING THE MONOXIDE OF IRON (FeO) FROM THE SESQUIOXIDE (Fe^2O^3) IN SILICATES AND OTHER COMPOUNDS.

[*First published in the Chemical Gazette: March* 1, 1848.]

This test serves to indicate, with great certainty, the presence or absence of FeO in bodies generally. It is performed as follows:—A small quantity of black oxide of copper (CuO) is dissolved in a bead of borax on platinum wire, so as to form a glass which exhibits, on cooling, a decided blue colour, but which remains transparent. To this, the test-substance in the form of powder is added, and the whole is exposed for a few seconds, or until the test-matter begins to dissolve, to the point of the blue flame. If the substance contain Fe^2O^3 only, the glass on cooling will remain transparent, and will exhibit a blueish-green colour. On the other hand, if the test-substance contain FeO, this will become at once converted into Fe^2O^3 at the expense of some of the oxygen of the copper compound; and opaque red streaks and spots of Cu^2O will appear in the glass, as the latter cools.*

NOTE:—Although this test is quoted by Plattner—perhaps the best criterion of its accuracy—it is passed over, without mention, in many works on chemical analysis. The writer may therefore be allowed to call to mind, in proof of its efficacy, that by its use in 1848 he pointed out the presence of FeO in the mineral *Staurolite* (*Chem. Gaz.*, *July* 15, 1848; see also *Erdmann's Journal für pract. Chem.*, XLVI., 119), nearly thirteen years before this fact—now universally admitted—was discovered and announced by *Rammelsberg* (*Berichte d. Kongl. preuss. Akad. d. Wiss. zu Berlin: Marz*, 1861.)

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pipe characters of Phosphocerite were given in a paper on that mineral, published in the journal of the Chemical Society of London in 1848; and these characters are referred to, from the paper in question, in the third volume of *Henry Watt's* English translation of *Gmelin's Handbuch*, published by the Cavendish Society in 1849, as well as in both the third and fourth editions of *Dana's* System of Mineralogy.

* Provided too much copper oxide be not dissolved in the glass—so as to become reduced *per se*—this test may be performed with either a reducing or an oxidating flame. If the method be tried with a few bodies of known composition (in some of which FeO is present, and in others absent) the operator will see, at once, that it offers no risk of failure—always assuming, of course, the absence of other reducing bodies, a point easily ascertained by the blowpipe.

9. DETECTION OF MINUTE TRACES OF COPPER IN IRON PYRITES AND OTHER BODIES.*

Although an exceedingly small per-centage of copper may be detected in blowpipe experiments, by the reducing process, as well as by the azure-blue coloration of the flame when the test-matter is moistened with chlorhydric acid, these methods fail in certain extreme cases to give satisfactory results. It often happens that veins of Iron Pyrites lead at greater depths to Copper Pyrites. In this case, according to the experience of the writer, the Iron Pyrites will almost invariably hold minute traces of copper. Hence the desirability, on exploring expeditions, more especially, of some ready test, by which, without the necessity of employing acids or other bulky and difficultly portable reagents, these traces of copper may be detected.† The following simple method will be found to answer the purpose:—The test-substance, in powder, must first be roasted on charcoal, or, better, on a fragment of porcelain,‡ in order to drive off the sulphur. A small portion of the roasted ore is then to be fused on platinum wire with phosphor-salt; and some bisulphate of potash is to be added to the glass (without this being removed from the wire) in two or three successive portions, or until the glass becomes more or less saturated. This effected, the bead is to be shaken off the platinum loop into a small capsule, and treated with boiling water, by which either the whole or the greater part will be dissolved; and the solution is finally

* This method has not been hitherto published; but it is inserted here, as it has been shown to various persons interested in blowpipe experiments. The same remark applies to No. 11.

† In Blowpipe Practice—as far, at least, as this is possible—the operator should make it an essential aim to render himself independent of the use of mineral acids and other liquid and inconvenient reagents of a similar character. If these reagents cannot be dispensed with altogether, their use, by improved processes, may be greatly limited.

‡ In the roasting of metallic sulphides, &c., the writer has employed, for some years, small fragments of Berlin or Meissen porcelain, such as result from the breakage of crucibles and other vessels of that material. The test-substance is crushed to powder, moistened slightly, and spread over the surface of the porcelain; and when the operation is finished, the powder is easily scraped off by the point of a knife-blade or small steel-spatula. In roasting operations, rarely more than a dull red heat is required; but these porcelain fragments may be rendered white-hot, if such be necessary, without risk of fracture. *Canadian Journal*, September, 1860.

to be tested with a small fragment of ferrocyanide of potassium ("yellow prussiate.") If copper be present in more than traces, this reagent, it is well known, will produce a deep red precipitate. If the copper be present in smaller quantity, that is, in exceedingly minute traces, the precipitate will be brown or brownish-black; and if copper be entirely absent, the precipitate will be blue or green—assuming, of course, that Iron Pyrites or some other ferruginous substance is operated upon. In this experiment, the preliminary fusion with phosphor-salt greatly facilitates the after solution of the substance in bisulphate of potash. In some instances, indeed, no solution takes place if this preliminary treatment with phosphor-salt be omitted.

10. DETECTION OF LEAD IN THE PRESENCE OF BISMUTH.

[*First published in the Chemical Gazette: Sept.* 15, 1848.]

When lead and Bismuth are present together, the latter metal may be readily detected by its known reaction with phosphor-salt in a reducing flame—antimony, if present, being first eliminated; but the presence of lead is less easily ascertained. If the latter metal be present in large quantity, it is true, the metallic globule will be more or less malleable, and the flame-border will assume a clear blue color when made to play upon its surface, or on the sublimate of lead-oxide as produced on charcoal; but in other cases, this reaction becomes exceedingly indefinite. The presence of lead may be detected, however, by the following plan, based on the known reduction and precipitation of salts of bismuth by metallic lead: a method which succeeds perfectly with brittle alloys containing 85-90 per cent. of bismuth. A small crystal or fragment of nitrate of bismuth is placed in a porcelain capsule, and moistened with a few drops of water, the greater part of which is afterwards poured off; and the metallic globule of the mixed metals, as obtained by the blowpipe, having been slightly flattened on the anvil until it begins to crack at the sides, is then placed in the midst of the sub-salt of bismuth formed by the action of the water. In the course of a minute or even less, according to the amount of lead that may be present, an arborescent crystallization of metallic bismuth will be formed around the globule.

This reaction is not affected by copper; but a precipitation of bismuth would ensue, in the absence of lead, if either zinc or iron were present. These metals, however, may be eliminated from the test-globule by exposing this on charcoal for some minutes, with a mixture of carb-soda and borax, to a reducing flame. The zinc becomes volatilized, and the iron is gradually taken up by the borax. If a single operation do not effect this, the globule must be removed from the saturated dark green glass, and treated with further portions of the mixture, until the resulting glass be no longer colored.

11. DETECTION OF ANTIMONY IN TUBE-SUBLIMATES.

In the examination of mineral bodies for antimony, the test-substance is often roasted in an open tube for the production of a white sublimate. The presence of antimony in this sublimate may be detected by the following process—a method more especially available when the operator has only a portable blowpipe-case at his command. The portion of the tube to which the chief portion of the sublimate is attached, is to be cut off by a triangular file, and dropped into a test-tube containing some tartaric acid dissolved in water. This being warmed or gently boiled, a part at least of the sublimate will be dissolved. Some bisulphate of potash—either alone, or mixed with some carb-soda and a little borax, the latter to prevent absorption—is then to be fused on charcoal in a reducing flame; and the alkaline sulphide thus produced, is to be removed by the point of the knife-blade, and placed in a small porcelain capsule. The hepatic mass is most easily separated from the charcoal by removing it before it has time to solidify. Some of the tartaric acid solution is then to be dropped upon it, when the well known orange-colored precipitate of $Sb\,S^3$ will at once result.

In performing this test, it is as well to employ a somewhat large fragment of the test-substance, so as to obtain a thick deposit in the tube. It is advisable also to hold the tube in not too inclined a position, in order to let but a moderate current of air pass through it; and care must be taken not to expose the sublimate to the action of the flame—otherwise it might be converted almost wholly into a compound of $Sb\,O^3$ and $Sb\,O^5$, the greater part of which would remain undissolved in the tartaric acid solution. A sublimate of arsenious acid, treated in this manner, would, of course, yield a yellow precipitate,

easily distinguished by its color, however, from the deep orange antimonial sulphide. The crystalline character etc., of this sublimate, would also effectually prevent any chance of misconception.

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12. THE COAL ASSAY.

[*First published in the Canadian Journal: May, 1858; and in the Philosophical Magazine for July of that year.*]

In the practical examination of Coals, the following operations are essentially necessary:*—(1) The estimation of the water or hygro-

* To these might be added, the determination of the heating powers or "absolute warmth" of the coal, but this may always be estimated with sufficient exactness for practical purposes by the amount of coke, ash, and moisture, as compared with other coals. Properly considered, the litharge test, resorted to for the determination of the calorific power of coals, is of very little actual value. The respective results furnished by good wood charcoal and ordinary coke, for example, are closely alike, if not in favour of the charcoal; and yet experience abundantly proves the stronger heating powers of the coke. In practice, moreover, the actual value of a coal does not always depend upon the "absolute warmth" of the latter, as certain coals, such as brown coals rich in bitumen, may possess heating powers of considerable amount (as estimated by the reduction of litharge) though only of brief duration. Thus, the lignites of the department of the Basses Alpes in south-eastern France, and those of Cuba, yield with litharge from 25 to 26 parts of reduced lead; whilst many caking coals, practically of much higher heating power, yield scarcely a larger amount. When pyrites also is present in the coal—a condition of very common occurrence—the litharge test becomes again unsatisfactory, the pyrites exerting a reducing action on the lead compound.

As described, however, by Bruno Kerl, in quoting the writer's Coal Assay (*Löthrohr-Untersuchungen: Zweite Aufl.* 1862, p. 146) the so-called absolute warmth or heating power of a coal sample may be determined, if desired, in blowpipe practice, by the following modification of Berthier's method:—20 milligrammes of the coal, in fine powder, are to be mixed intimately with 500 milligrammes of oxy-chloride of lead (consisting of 3 parts of litharge + 1 part of chloride of lead, fused together and finely pulverized). The mixture is to be placed in a blowpipe-crucible, and covered with about an equal amount of the lead compound, a second cover of 8 blowpipe-spoonfuls of powdered glass + 1 spoonful of borax, being spread over this. The crucible, covered with a clay capsule, is then to be fitted into a charcoal block in the ordinary blowpipe furnace, over which a charcoal lid is placed, and the flame directed against its under side, so as to keep it at a red heat for from 5 to 8 minutes. The weight of the reduced lead divided by 20 gives the amount of the lead mixture reduced

metric moisture present in the coal; (2) the determination of the weight and character of the coke; (3) the estimation and examination of the ash or inorganic matters; and (4) the estimation of the sulphur, chiefly present in the coal as FeS^2.

Estimation of Moisture:—This operation is one of extreme simplicity. Some slight care, however, is required to prevent other volatile matters from being driven off during the expulsion of the moisture. Seven or eight small particles, averaging together from 100 to 150 milligrammes, are to be detached from the assay specimen by means of the cutting pliers, and carefully weighed. They are then to be transferred to a porcelain capsule with thick bottom, and strongly heated for four or five minutes on the support attached to the blowpipe lamp, the unaided flame of the lamp being alone employed for this purpose. It is advisable to place in the capsule, at the same time, a small strip of filtering or white blotting-paper, the charring of which will give indications of the temperature becoming too high. The coal, whilst still warm, is then to be transferred to the little brass capsule in which the weighings are performed, and its weight ascertained. In transferring the coal from one vessel to the other, the larger pieces should be removed by a pair of fine brass forceps, and the little particles or dust afterwards swept into the weighing capsule by means of the camel's-hair pencil or small colour-brush belonging to the balance-case. The weighing capsule should also be placed in the centre of a half-sheet of glazed writing-paper, to prevent the risk of any accidental loss during the transference. After the weighing, the operation must always be repeated, to ensure that no further loss of weight occur. In place of the blowpipe-lamp, the spirit-lamp may be employed for this operation; but, with the former, there is less danger of the heat becoming too high. By holding a slip of glass for an instant every now and then, over the capsule, it will soon be seen when the moisture ceases to be given off. It should be remarked, that some anthracites decrepitate slightly when thus treated, in which case the porcelain capsule must be covered at first with a small watch-glass.

In good samples of coal, the moisture ought not to exceed 3 or 4 per cent., but in coals that have been long exposed to damp it is often

by 1 part of the coal. One part of pure carbon reduces 34 parts of this mixture; one part of charcoal, 30–33 parts; one part of bituminous coal, 19–33; one part of brown coal, 14–26; one part of peat, 8–27; and one part of wood, 12–15 parts.

as high as 6 or 7, and even reaches 15 or 20 per cent. in certain lignites. Where large quantities of coal are consumed, therefore, a serious loss is entailed on the purchaser unless the moisture be properly determined and allowed for.

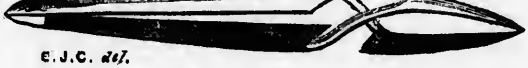
Estimation, &c., of Coke:—In this operation, a small crucible of platinum is most conveniently employed. This may consist of a couple of rather deep spoons—the larger one without a handle, so as to admit of being placed over the smaller spoon, as in the annexed figure: thus serving as a lid. The long handle of the crucible-spoon must be bent as shown in the drawing, in order that the spoon may retain an upright position when placed on the pan of the balance.

The spoon-crucible of this kind employed by the writer, weighs (with its lid) only 2.33 grammes, or rather less than 36 grs. About 150 milligrammes of coal are detached as before, in several small fragments, from the assay-specimen. These may be weighed directly in the crucible, the latter being placed in the little weighing capsule of horn or brass, with its handle-support projecting over the side of this. The crucible, with its cover on, is then taken up by a convenient forceps (see the note on page 19) and brought gradually before the blowpipe to a red heat. The escaping gases will take fire and burn for a few seconds around the vessel, and a small amount of carbonaceous matter may be deposited upon the cover. This rapidly burns off, however, on the heat being continued. As soon as it disappears, the crucible is to be withdrawn from the flame, and placed on the blowpipe-anvil to cool quickly. Its weight is then ascertained: always without removing the cover. The loss, minus the weight of moisture as found by the first process, gives the amount of volatile or gaseous matter. The residue is the coke and its contained ash. The coke in some anthracites exceeds 89 or 90 per cent. In anthracitic or dry coals it usually varies from 70 to 80 per cent., and the fragments are sometimes slightly agglutinated. In ordinary bituminous or caking coals, it amounts in general to about 65 or 70 per cent., and presents a fused and mamillated surface. In cannel or gas coals, the per centage of coke may be assumed to equal 50 or 60, but it is sometimes as low as 30. The coke fragments are often partially agglutinated, but they never present a fused, globular aspect. Finally, in lignites or brown coals, the coke may vary from 25 to 50 per cent. It forms sharp-edged fragments of a dull charcoal-like appearance, without any signs of fusion.

Estimation of Ash or Inorganic Matters :—A platinum capsule is employed for this operation. One of about half an inch in diameter, with a short ear or handle, is sufficiently large. A somewhat smaller capsule, with its handle cut off, may be fitted into this (in reversed position) to serve as a lid. The weight of the two together need not exceed $2\frac{1}{2}$ or $2\frac{3}{4}$ grammes.* The coal must be reduced to a coarse powder, and about 150 milligrammes weighed out for the experiment. The platinum capsule is then to be fixed in a slightly-inclined position above the spirit-lamp, and heated as strongly as possible. If the wick of the spirit-lamp be raised sufficiently, and the capsule be light and thin, the temperature will be sufficient to burn off the carbon: at least, in the majority of cases. The lid of the capsule must be placed above the coal powder until combustion cease, and all the more gaseous products are driven off, as otherwise, a portion of the powder might very easily be lost. During the after combustion, the powder must be gently stirred, and if agglutination take place, the particles must be carefully broken up, by a light steel-spatula or by a piece of stout platinum-wire flattened at one end. If the carbonaceous matter be not burnt off by this treatment, the blowpipe may be used to acce-

* It is convenient to have counterpoises for the platinum vessels described above, as the weights which accompany the blowpipe-balance only range, in general, from a gramme downwards. A small platinum capsule forms an excellent counterpoise. It can be trimmed down by a pair of fine scissors until brought by repeated trials to the proper weight. The writer has cut out receptacles for two platinum vessels and counterpoises of this kind, in the little box into which his travelling balance packs; and he recommends other operators to do the same, as these vessels are of very convenient use, not only in coal assaying, but in ascertaining the amount of water in minerals, as well as for other purposes.

These platinum vessels are held most conveniently, during ignition, by a pair of steel forceps, of the annexed pattern, so constructed as always to remain closed at the points except when subjected to pressure. With forceps of this kind, the vessels in question may be taken up and disengaged in an instant, without the intervention of the right hand. The forceps may be laid down also, whilst the vessels are red hot, without risk of the latter coming in contact with the table.



C. J. C. *del.*

lerate the process; but the operator must blow cautiously, and direct the flame only against the under side of the capsule, in order to avoid the risk of loss. Finally, on the ash ceasing to exhibit in any of its particles a black color, the lid of the capsule is to be carefully replaced, and the whole cooled and weighed.*

In good coals, the amount of ash is often under 2 per cent., and it rarely exceeds 4 or 5 per cent. In coals of inferior quality, however, it may vary from 8 or 10 to even 20 per cent. As regards its composition, the ash may be:—(1) argillaceous, consisting essentially of a silicate of alumina; (2) argillo-ferruginous; (3) calcareous; and (4) calcareo-ferruginous. If free from iron, it will be white or pale grey; but if more or less ferruginous, it will present a red, brown, or yellowish color. Phosphor-salt, so useful in general cases for the detection of siliceous compounds, cannot be safely used to distinguish the nature of the ash obtained in blowpipe assays. Owing to their fine state of division and to the small quantity at command, argillaceous ashes dissolve in this reagent with as much facility as those of a calcareous nature, and without producing a characteristic silica skeleton, or causing the opalization of the glass. With calcareous ashes also, the amount obtained is rarely sufficient to saturate even an exceedingly minute bead of phosphor-salt or borax, and hence no opacity is produced by the flaming process. The one kind of ash, may be distinguished, nevertheless, from the other, by moistening it, and placing the moistened mass on reddened litmus-paper. Calcareous ashes always contain a certain amount of caustic lime, and thus restore the blue color of the paper. These calcareous ashes, also, though principally composed of carbonate of lime, sometimes contain small portions of phosphate and sulphate of lime. The presence of the latter may be readily detected by the well known production of an alkaline sulphide by fusion with carbonate of soda in a reducing flame—the fused mass exhibiting a reddish color, and imparting when moistened a dark stain to a plate of silver or piece of lead test-paper. The latter may

* If the ash be very ferruginous—in which case it will present a red or tawny color—the results, as thus obtained, will require correction: the original iron pyrites of the coal being weighed as sesquioxide of iron. In ordinary assays, however—as distinguished from analyses—this may be fairly neglected. When also the ash happens to be calcareous and to occur in large quantity, it should be moistened with a drop or two of a solution of carbonate of ammonia, and gently heated, previous to being weighed.

be replaced by a glazed visiting-card. In examining earthy sulphates by this method, a little borax ought always to be added to the carbonate of soda, in order to promote the solution of the test-matter. If oxide of manganese be present in the ash, the well-known manganate of soda, or "turquoise enamel," will also be obtained by this treatment.

Estimation of Sulphur:—The following plan is perhaps the most simple that can be employed for the determination of sulphur in coal samples. It is merely an adaptation to blowpipe practice of the process very generally employed for that purpose:—

As large an amount of coal as practicable, several pounds at least, taken from different parts of the same heap or bed, must be broken into powder and well stirred together. About 150 milligrammes are to be weighed out for the assay. This amount is to be intimately mixed with about 450 milligrammes of nitrate of potash and an equal quantity of carbonate of potash, and the mixture, with a good covering of salt, is to be fused in a small platinum crucible of about a quarter of an ounce capacity. The crucible may be fixed in an ordinary blowpipe-furnace, in the centre of an already used charcoal-block, as the cavity of the latter will require to be larger than usual. The heat at first must be very moderate, as the mixture swells up greatly; but after a couple of minutes, or thereabouts, a tolerably strong blast may be kept up for from two to three minutes in addition, when the operation will be finished. The alkaline sulphate, thus produced, is dissolved out by boiling water, and the solution, acidified by a few drops of chlorhydric acid, is then treated with chloride of barium. The weight of the precipitate divided by 7.28 gives the amount of sulphur. An ordinary blowpipe crucible of clay may be employed for this operation; but it is always strongly attacked by the mixture during fusion, and is otherwise less convenient for the purpose than one of platinum.

When the iron pyrites in the coal is not in a state of semi-decomposition, its amount, and consequently the amount of sulphur, may be arrived at far more nearly than might at first thought be supposed, by the simple process of washing in the agate mortar. Each single part of pyrites corresponds to 0·533 of sulphur. Several large pieces of the assay-coal should be taken, and broken up into powder, and a couple of trials should be made on separate portions of this. About 500 milligrammes may be taken for each trial, and washed in three or four portions. In the hands of one accustomed to the use of the mortar in

reducing experiments, the results, owing to the lightness of the coal particles, and the consequent ease with which they are floated off, come out surprisingly near to the truth. In travelling, we may dispense with the washing bottle, by employing, in its place, a piece of straight tubing drawn out abruptly to a point. This is to be filled by suction, and the water expelled with the necessary force by blowing down the tube. A tube 6 inches long and the fourth of an inch in diameter, will hold more than a sufficient quantity of water to be used between the separate grindings.* The mortar should be but slightly inclined, and the stream of water must not be too strong; otherwise, especially if the coal be ground up very fine, portions of the pyrites may be lost. The proper manipulation, however, is easily acquired by a little practice.

* In the third edition of his *Probirkunst*, p. 60, PLATTNER alludes to the use of a tube of this kind, as a make-shift for the washing bottle, in reducing operations; but in the new edition, this reference to its use is omitted.

IN PREPARATION:

(With Numerous Woodcuts.)

A Manual of Blowpipe Practice;

OR,

Complete Guide to the use of the Blowpipe in Chemistry and Mineralogy:

COMPRISING:

BLOWPIPE ANALYSIS;

THE DISCRIMINATION OF MINERALS;

AND

BLOWPIPE ASSAYING.

GENERAL SUMMARY OF CONTENTS.

INTRODUCTION—(Containing, amongst other matters, a History of the Blowpipe, far more complete than the very imperfect sketch first given by Berzelius, and copied subsequently, without correction, by so many other authors. The best and most correct account, yet given, of the origin of the Blowpipe as a scientific instrument, is to be found in the valuable *Geschichte der Chemie* of DR. HERMANN KOPP.)

PART I.—BLOWPIPE ANALYSIS, INCLUDING THE DISCRIMINATION OF MINERALS.

Division 1. *Instruments and Appliances*—(In this division various new instruments and adaptations are described, the whole being illustrated by 75 figures, drawn expressly for the present work. The instruments employed in Blowpipe Assaying are described and figured separately, in Part II.)

Division 2. *Reagents.*

Division 3. *Operations*—(Described, in detail, under 36 distinct headings, with 15 woodcuts.)

Division 4. *Reactions*—(This division comprises the largest portion of the work. The elementary bodies are arranged in groups according to a new plan founded on their blowpipe characters; and not only are the reactions of simple substances, oxides, &c., given in detail, but methods for the detection of these in various combinations are also pointed out. The whole subject is methodically arranged under separate paragraphs, each with its special heading, so as to admit of ready reference. Under each metal or metalloid, all the minerals, in which

this occurs, are distributed (for their more ready determination) into groups and sections founded essentially on blowpipe reactions. An attempt is also made, not only here but throughout the entire work, to trace back all discoveries to their primary source, so as to give full credit to their originators.* Nothing, at least, is taken from any source without due acknowledgment.†

Division 5. *Plan of Analysis in the examination of bodies generally*—(The method of analysis given in this division, is entirely original, and applicable both to natural substances and art-products. By its aid, the composition of a body may be arrived at, it is thought, much more rapidly, and with greater certainty, than by the blowpipe method of Rose, or that of Laurent. In Rose's plan, as adopted by Plattner and others, much time is lost, for example, by the same component being detected over and over again, whilst others escape detection altogether. In numerous instances also, by the employment of this method, a substance is subjected to many useless experiments. Laurent's system partially avoids this defect; but in other respects it is exceedingly incomplete, especially as regards the examination of bodies containing several bases.)

PART II. BLOWPIPE ASSAYING.

Division 1.—*Instruments and Appliances*—(Amongst other instruments, a new scale for the measurement of silver globules, is described in this division.)

Division 2. *Reagents.*

Division 3. *Operations.*

Division 4. *Quantitative Examination of Metallic Ores, Coals, and other Assay-Matters*—(This division is necessarily based on the great work of PLATTNER. It will be found to contain, however, no inconsiderable amount of new matter, including many useful hints derived from the author's constant practice in this mode of assaying.)

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* This is too often completely lost sight of. In a modern work, for example—well enough compiled in other respects, and useful as a book of reference—the *Untersuchungen mit dem Löthrohr* of DR. HUGO HARTMANN, many well-known reactions, which were really announced by BERZELIUS or still earlier workers, are credited to PLATTNER. See, for instance, under the Chlorides, p. 18 of the first part of the work, and various other examples.

† The following heads of paragraphs, referring to the element sulphur, will serve to convey a general idea of the method adopted in this portion of the work:

XII. SULPHUR.

226. Conditions of occurrence.

227. Pyrognostic characters of sulphur and sulphur compounds generally (including sulphites, sulphates, &c.)

228. Special methods for the detection of sulphur and its oxygen-acids in bodies of all kinds.

229. Distinction between sulphur and selenium compounds.

230. Detection of sulphur in the presence of selenium.

231. Detection of sulphates in the presence of seleniates.

232. Distinction between sulphides and sulphates (or other oxygen-salts.)

233. Distinction between sulphites and sulphates.

234. Sulphides of natural occurrence—(These are arranged in 4 primary and 26 secondary groups, founded essentially on blowpipe characters.)

235. Sulphates of natural occurrence—(Arranged in 17 determinative groups founded on their blowpipe reactions.)

ADVERTISEMENT.

THE following pages contain brief notices of various blowpipe tests and applications, published or otherwise made known by the writer within the last seventeen or eighteen years. Some of these tests have been appropriated by certain authors without the slightest acknowledgement; others have been incorrectly quoted; and some, again, appear to have escaped the attention of the editors of several recent works in which the applications of the blowpipe come under review. These reasons have led the writer to republish them, in a condensed form, in the present pamphlet, preparatory to the issue of an extended work on Blowpipe Practice, in which various new tests, and a large amount of other original matter, will be incorporated. This work —announced on page 23—will appear, it is hoped, early in 1866.

E. J. C.

UNIVERSITY COLLEGE, TORONTO,
September 1, 1865.