

# Miscellaneous New Aids to the Study of Minerals

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## Abstract

THE following new, but unrelated, aids to the study of minerals are described:—Charcoal block — and allied tests — by means of a blow lamp: a "non-destructive" chromatographic method for investigating the components of conducting grains: a simple "confined-spot" test apparatus: a new test for wolframite: the staining of scheelite in thin section: the identification, etc., of platinum-bearing grains.

### 1. Charcoal block — and allied tests — by means of a blow lamp.

Despite the comparatively recent development of numerous powerful aids to mineral identification, charcoal block — and allied tests — are still of considerable value to the mineralogist — particularly when he is conducting field studies in isolated areas. The tests and the apparatus are simple, and the reagents are — for the most part — innocuous solids and so present no transportation problems. The Achilles heel of these methods lies principally in the fact that consistently adequate fusions, etc., can only be made with the aid of a blow pipe after considerable practice. However, experiments conducted at the Camborne School of Metalliferous Mining during the past eighteen months have shown that good results can be readily obtained if a modified Valtock "2000" Blow Lamp is substituted for the blow pipe and alcohol lamp.

The blow lamp — which is 5 ins. high — uses methylated spirit as fuel. It consists of two metal cylinders, and from the top of one of these a blow pipe tube curves over the open top of the other. A flame from the "open" cylinder vapourises the alcohol passing through the blow pipe and so causes a hot pointed flame to issue from the nozzle of the pipe. (Fig. 1.)

To carry out a test the material to be heated is placed in a shallow depression on a charcoal block resting on a bench. If oxidations are to be effected the test substance is heated with the tip of the flame, whilst reductions are carried out by so heating the substance that the flame completely envelopes it. Sublimates are collected on the surface of the block by tilting the latter during the test.

During the initial tests it was found difficult to control the impingement of the flame on the block, but this was overcome by using a hemi-cylindrical, sheet-metal "flame-directing" hood which was clipped to the vertical part of the blow lamp tube: this modification is shown in Fig. 1.

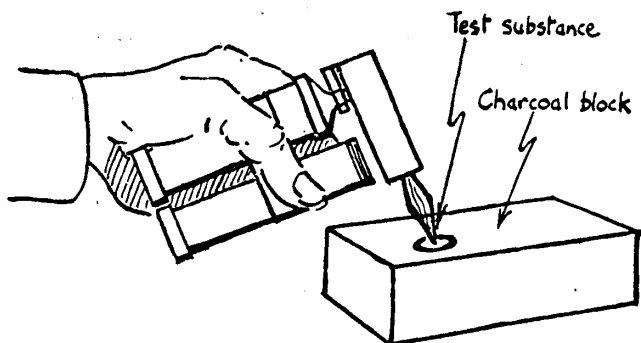
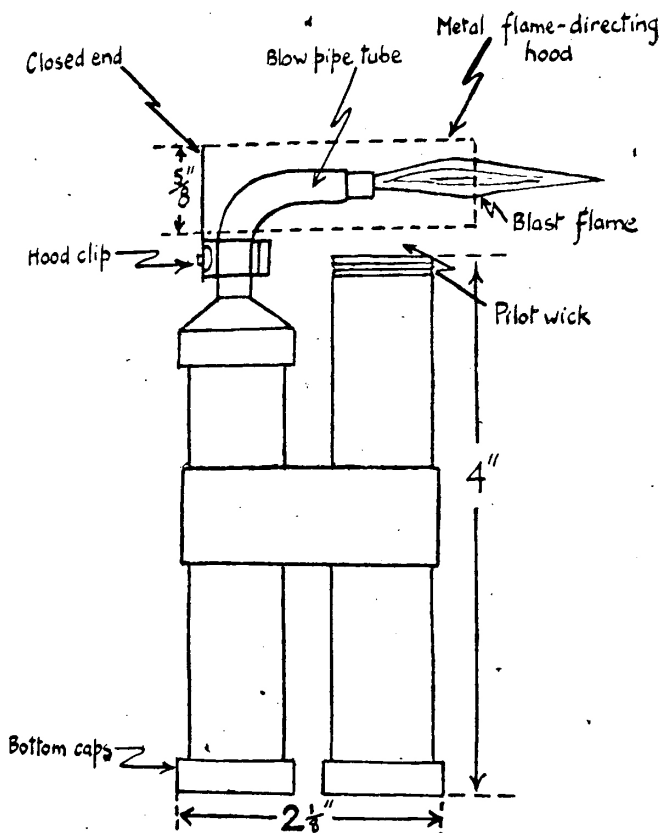


Fig. 1. The modified Valtock "2000" Blow Lamp and the manner of using it when carrying out tests on a charcoal block.

All the usual oxidation, reduction, fusion, bismuth-flux and cobalt nitrate tests have been carried out successfully on charcoal blocks by means of the above technique, and in addition the blow lamp has been used to conduct tests — of the type described elsewhere by the writer — on asbestos paper (Hosking, 1953).

## 2. A "non-destructive" chromatographic method for investigating the components of mineral grains.

In an earlier paper (1956) the writer recorded that the identification of individual conducting grains in mill-products, etc., could be facilitated by employing the current from a 4.5-volt battery to drive the ions of the grain into a square of gelatin-coated paper impregnated with an appropriate attacking reagent and then identifying the captured ions by bathing the paper in a developing reagent.

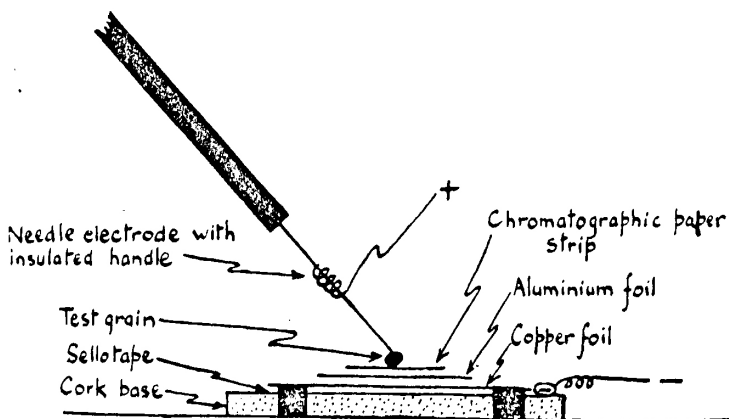


Fig. 2. Electrographic method of obtaining a sample of ions from a conducting grain as a preliminary to their separations by paper chromatographic means.

Subsequent work has shown that, on occasion, ions liberated by the above method can be profitably investigated by chromatographic means. In practice, the initial stage simply involves the use of a strip of Whatman's No. 1 filter-paper instead of the square of gelatin-coated paper. (Fig. 2.) The size of the paper used will depend, largely, on the ease with which the elements to be sought can be separated by chromatographic means. If the  $R_f$  values of the elements are close, then adequate chromatograms will only be obtained by carrying out the separations by ascending or descending means in fairly large gas-jars: under such circumstances strips of 1 in.-wide paper are convenient. If, on the other hand, the  $R_f$  values

of the elements are widely different, rapid results may be obtained by disc chromatography, or by ascending chromatography carried out on a 0.5 in.-wide paper strip dipping into the solvent in a test tube. In the latter case the strip is pinned to a cork which is inserted into the mouth of the tube. If each of a number of grains are to be examined by the same chromatographic method, and if the elements in question are readily separable by chromatographic means, then it is most convenient to carry out the work on panels of Whatman's CRL/1 chromatography paper. Each panel accommodates ten samples, and to effect the necessary separations the panel is bent round in the form of a cylinder, the ends being held together by means of a paper-clip. This cylinder is then placed in a 600-ml. beaker containing the appropriate solvent and is covered with a Petri dish until the solvent front is within an inch of the top of the paper. The paper is then removed, dried, etc., and sprayed with appropriate developing reagents. (For further details of the chromatographic aspect of the method, which is used in geochemical analysis, see Hunt, North and Wells, 1955.)

Conducting minerals, together with suitable attacking reagents, have been reviewed by Williams and Nakhla (1950-51), whilst solvents and developing reagents appropriate to the chromatographic separation of the elements under investigation can usually be found either in the work of Pollard and McOmie (1953) or of Lederer and Lederer (1957). However, the following examples serve to illustrate how the method can facilitate the examination of mineral grains:—

(i) The following procedure may be used to detect any, or all, of the elements iron, nickel, cobalt and copper in single grains of, for example, bornite, carollite, chalcopyrite, chalcocite, chloanthite, gersdorffite, niccolite, pentlandite and pyrrhotite. Place the grain on the chromatographic paper where the latter has been damped with a drop of 1 : 1  $\text{HNO}_3$ . Pass a current from a 4.5-volt dry battery for 30 seconds, then gently dry the paper over a flame, or in a current of hot air. Separate the ions by descending, or disc, chromatographic methods using a solvent of acetone, water and concentrated  $\text{HCl}$  in the ratio, by volume, of 43.5 : 2.5 : 4. Finally, dry the paper somewhat in air, then expose to ammonia vapour and spray with a 1 per cent alcoholic solution of rubeanic acid. The metals are revealed by the development of coloured zones which, taken in order from the point of application of the test ions, are as follows:—Ni (blue); Co (brown); Cu (olive-green); Fe (brown, and at the solvent front).

(ii) The presence of silver (and lead) in a grain of galena may be established by employing the same attack as in the above test, and, having dried to drive off excess  $\text{HNO}_3$ , separating the lead and silver ions by descending, or disc, chromatography using a solvent of 85 parts of industrial ethanol and 15 parts of water, and subsequently spraying the dried paper with a solution of potassium chromate. Silver is revealed by a red zone at the sample application point, whilst a yellow "outer" zone indicates lead.

(iii) A grain suspected of being pitchblende can be tested by using the attack common to the above tests and then separating uranium from other elements by chromatography employing a solvent of ethyl acetate, nitric acid and water in the ratio, by volume, of 100 : 10 : 5. The uranium, which moves with the solvent front, is indicated by the development of a brown band on spraying the dried test paper with a 5 per cent. solution of potassium ferrocyanide.

### 3. A simple "confined-spot" test apparatus.

If an insoluble, characteristically-coloured compound of a component in a given volume of test solution can be precipitated free from interfering substances on a standard area of filter-paper and at a carefully controlled rate, the quantity of the particular component in the solution may be determined semi-quantitatively by comparing the test spot with standard spots similarly prepared.

This analytic technique may be used for the solution of many chemico-mineralogical problems but it has been given particular prominence by those designing rapid tests for the geochemical prospector. Stevens and Lakin (1949) describe an apparatus (called a chromograph) that controls the rate of flow of small volumes (1 ml. or less) of test solution through the confined paper by the pull of a column of water of constant initial height as it passes through a fine-bore capillary. This apparatus works very well but field analysts tend to regard it as being too elaborate.

McCarthy and Stevens (1958) have developed a method by means of which many confined-spot tests may be carried out simultaneously. Their apparatus utilises the increasing pull resulting from the gradual lowering of the water-level in a tank (due to withdrawal by a syphon), to draw a given volume of test solution through the confined spot at a rate which is reproducible.

French Government geologists at Dakar (see Martinet and Genin, 1956) achieve controlled conditions of confined-spot precipitation simply by charging the head of the filter stick with a given volume of test solution whilst the tube below the paper is largely submerged in water in a cylinder, then raising the tube till the water column in it is at a standard height above the level of the water in the cylinder. (Fig. 3.)

In all the above pieces of apparatus the head which holds the filter-paper and test solution is, in effect, a coupling composed of glass and plastic or metal, and although its construction poses no problem to an engineer equipped with a lathe, etc., lack of tools may prevent its manufacture in a simply equipped laboratory. However, a perfectly adequate confined-spot test may be carried out simply by placing a disc of filter-paper between the ground-flat ends of two

glass tubes and making a secure union by binding with Sellotape. (Fig. 3.) The test solution may then be drawn through the confined spot, either by using the Dakar method, or that of McCarthy and Stevens.

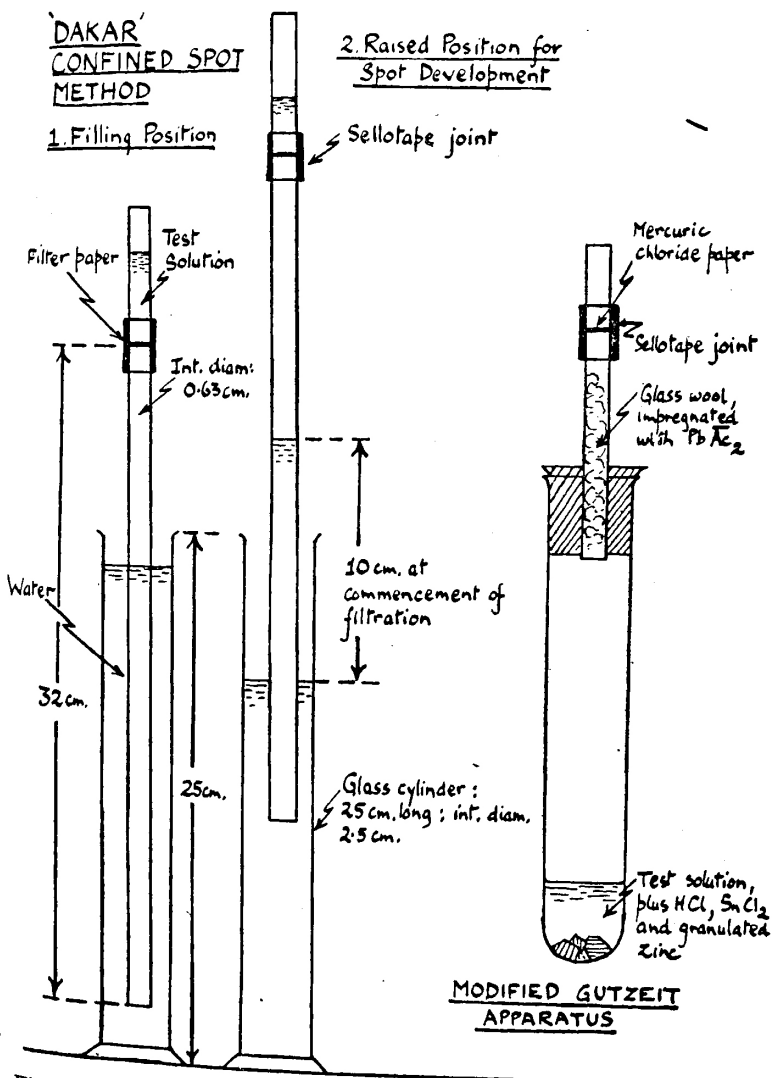


Fig. 3. Diagrams illustrating the employment of the simple head to obtain confined spots by the Dakar Method and arsenic spots by the Gutzeit Method.

The same coupling can be used to hold the mercuric chloride paper when carrying out the Gutzeit arsenic test if the usual metal coupling is unavailable.

Experience has shown that the simple glass "head" can be rapidly assembled if one operator holds the paper firmly between the two glass tubes whilst another binds the joint with tape.

#### 4. A new test for "wolframite".

The following new test is primarily of value in that it enables rapid differentiation to be made in the field between a member of the hübnerite - wolframite - ferberite series and other, somewhat similar, dark species. It may also be used to detect the presence of dominant tungsten in other species, and molybdenum in molybdic ochre and molybdates (but *not* in molybdenite).

**Procedure** :—Place such a quantity of the finely-powdered test mineral, such as would form a regular cone of c. 0.4 mm. diameter, into a 3-in.  $\times$   $\frac{5}{8}$ -in. test tube, then add c. 4 volumes of ground ammonium fluoride. Mix well by rotating and tapping the tube, and heat over a *small* flame — that from a cigarette lighter is admirable. Continue heating for c. 30 seconds after the contents of the tube have become quite dry. Wolframite, unlike such dark minerals as ilmenite, sphene, rutile and columbite yields a strong sky-blue product on a white background.

As tungstic ochre, scheelite, stolzite, molybdic ochre and wulfenite also yield a certain quantity of blue product the test cannot distinguish between these tungsten and molybdenum species, but if still may be of value during the solution of problems involving some of them.

Whilst the chemistry of the reaction has not been fully investigated it seems probable that the blue products are due to the reduction of tungstate or molybdate ions by ammonia.

#### 5. The staining of scheelite in thin section.

The following method of staining scheelite occurring as small white aggregates disseminated throughout a Portuguese greisen considerably facilitated study of the rock in thin section.

**Procedure** :—Grind the section to the normal thickness, having first mounted it on a glass slide with Lakeside cement. Then place the whole in a Petri dish and add sufficient 1 : 1  $H_2O$ /aqua regia, at c. 75° C., to cover the slice. Gently agitate for 5 to 10 minutes, or until the scheelite is a strong lemon-yellow, then decant and wash several times with water. (*Gentle* warming during the agitation phase, accelerates colour development.) Finally, dry and cover with a cover slip in the usual way.

The stained scheelite shows up well, both under the binocular and the petrological microscope, but the contrast between it and the matrix may be still further accentuated by placing a piece of red or purple cellophane immediately under the slide: then the scheelite appears as yellow to orange "islets" in a red or purple "sea". Thus, grain measurements and point counting are facilitated.

**Other notes:**—(i) It is obvious that the above staining method could not be applied to carbonate-rich specimens.

(ii) The writer has found that examination of thin sections generally under the wide field of a binocular microscope—in addition to the usual examination under a petrological microscope—serves to emphasise certain mineral distribution patterns, and enables opaque minerals to be examined much more satisfactorily than they can be under an ordinary petrological microscope. Furthermore, if a section, composed essentially of transparent minerals, is examined under the binocular microscope whilst wet and still quite thick, it is often possible to obtain considerable information about the "three-dimensional" distribution characteristics of some of the species. This technique has, for example, helped considerably in determining the manner in which prismatic crystals of cassiterite are arranged in certain Cornish lodes.

## 6. The identification, etc., of platinum-bearing grains.

Grains of so-called native platinum, osmiridium, platiniridium, palladium, etc., invariably contain varying amounts of all the platinum group of elements, and native platinum may contain several per cent. of iron and so be quite strongly magnetic. Native gold, also, may hold appreciable amounts of the platinum metals. Qualitative and semi-quantitative data concerning the platinum elements in the above species may be obtained reasonably quickly, and very simply, by employing a paper-chromatographic method of separation such as that described by Burstall et. al. (1950). However, when it is sufficient to demonstrate that a given grain contains appreciable amounts of the platinum group it is usually only necessary to employ the following tests which are the simplest and most rapid of all those known to the writer for these elements.

### Test A.

Wherever the domestic supply of coal-gas is as pure as that of Great Britain, the following test—which is a simplification of that proposed by Feigl (1957, 101-103) and which has been used by the writer for six or seven years—constitutes a rapid means of establishing the presence of dominant amounts of Pt, Pd, Ir or Rh in individual grains (e.g., of native platinum).

**Procedure:**—Warm the grain in a small porcelain crucible with a few drops of aqua regia for a few minutes: during this time only a portion of the grain may dissolve. Transfer a drop of the liquid to

the centre of a square of asbestos paper and heat the spot strongly immediately above the blue cone of the bunsen flame. Turn off the gas, rest the paper on the top of the burner with the test spot immediately over the centre of the barrel, and turn on the gas. If appreciable amounts of the metals noted above are present the spot will glow red.

### **Test B. The rubeanic acid test for platinum.**

Dominant amounts of Pt may be rapidly confirmed by attacking the grain with aqua regia — as noted in Test A — and placing a drop of the test liquid in the centre of a disc of Whatman's No. 1 filter-paper, followed by one or two drops of 1 per cent alcoholic solution of rubeanic acid. The presence of platinum is indicated by the development of a rose-pink halo surrounding a brownish central zone where the rubeanates of palladium, etc., have concentrated.

The test is new in that it depends on the hitherto unrecorded fact that under the conditions of the test platinum rubeanate diffuses out of the test zone and hence is not masked by other rubeanates which may be present.

### **Test C. The nickel dimethylglyoxime test for palladium.**

The following test — which is based on that described by Feigl (1954, 131-132) — is very convenient for demonstrating the presence of palladium in grains of native platinum, etc.

**Procedure:**—Attack the grain with aqua regia for a few minutes — as in Test A — and then gently evaporate *just* to dryness. Dissolve the yellow salts thus deposited in the minimum quantity of water and place a drop of this solution on a square of dry nickel dimethylglyoxime paper. Dry the paper in a current of hot air — or, very carefully, over a flame — and bathe it in 1 in 10 HCl. Palladium is indicated by the presence of a red spot which persists after the "unused" nickel dimethylglyoxime has been destroyed by the acid.

**Preparation of reaction paper:**—Whatman's No. 1 filter-paper is bathed in a cold saturated alcoholic solution of dimethylglyoxime and then dried. It is then gently agitated in 2N. nickel nitrate (or sulphate) which has been made just ammoniacal. After the red nickel dimethylglyoxime has deposited on the filter-paper the latter is thoroughly washed in water, then bathed briefly in alcohol, dried and stored.

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### Discarded

He grabbed me round my slender neck ;  
 I could not call or scream.  
 He dragged me to his dingy room  
 Where we could not be seen.  
 He tore away my flimsy wrap  
 And looked upon my form,  
 I was so cold and damp and scared,  
 Whilst he was moist and warm.  
 His fev'rish lips he pressed to mine ;  
 I gave him every drop.  
 He drained me of my very self,  
 I could not make him stop.  
 He made me what I am to-day :—  
 That's why you find me here,  
 A broken bottle cast away  
 That once was full of beer.

Anon.