

Flash Fusion Tests for Economic Minerals

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The author describes

a method of

rapidly identifying

certain minerals

Abstract

Methods are described for the rapid fusion of minerals with fluxes and for the rapid reduction of sulphates and phosphates. A "flash fusion" takes less time than a minute.

The fusions are usually carried out on an inverted Pyrex dish or on asbestos paper. A mixture of the powdered mineral and flux, or sometimes the mineral powder alone, is covered with sodium peroxide and a burning splint applied to the latter. An alternative procedure involves covering the mineral, with or without admixed flux, with sodium peroxide, which in turn is covered with photographer's flash powder. This charge is ignited by means of touch paper.

Reductions are obtained by covering the powdered mineral with flash powder upon which has been placed a little oxidizing agent to permit of easy ignition. The charge is ignited by one of the methods noted above. Subsequent examination of the products by spot or other tests is fully described.

The advantages of flash fusion and reductions as compared with the usual methods are noted.

Introduction

As a preliminary to the application of chemical tests to many minerals of economic importance it is often desirable or necessary for them to be fused with a suitable flux in order to convert them into compounds which are soluble in the commoner and less noxious reagents. The preliminary fusion is usually carried out on a charcoal block, or in a wire loop in a blow-piped flame of a bunsen burner, alcohol or grease lamp, or in a crucible heated by a bunsen burner in the laboratory, or by a "Primus" in the field.

A considerable degree of skill is required if quick and adequate fusions are to be made consistently by means of a blow-pipe. Charcoal blocks, although not without merit, are far from ideal, in that only a small number of tests can be made on any one block without serious risk of contamination. One of the chief disadvantages associated with the use of crucibles is their high destruction rate if not treated with the greatest care.

The methods of fusion described in what follows have certain advantages over those already noted in that there is no risk of contamination by residues from previous tests, no external source of heat is required, and the manipulative technique is extremely simple. Furthermore, the necessary apparatus is cheap and compact and small quantities of material can be treated. Flash fusions are quickly done. Assuming the material to be tested is already in a fine state of division, the apparatus can be set up and the fusion completed in less than a minute.

During the flash fusion only a portion of the mineral sample reacts with the flux, the amount depending on the species under examination. However, as far as the minerals listed later are concerned, sufficient fusion takes place to permit the use of the chemical tests described. There is no reason to believe that any mineral will completely resist fusion by this method, although for the adequate testing of some minerals—*e.g.*, those containing earth acids—fusion in a crucible is necessary.

Only a selection of the economically important minerals has been tested, but the aim has been so to devise the fusions and chemical tests that they shall be, as far as possible, of general application. Thus, for example, the methods utilized for the detection of aluminium in a few minerals can probably be used for the detection of aluminium in all minerals in which it is an essential element.

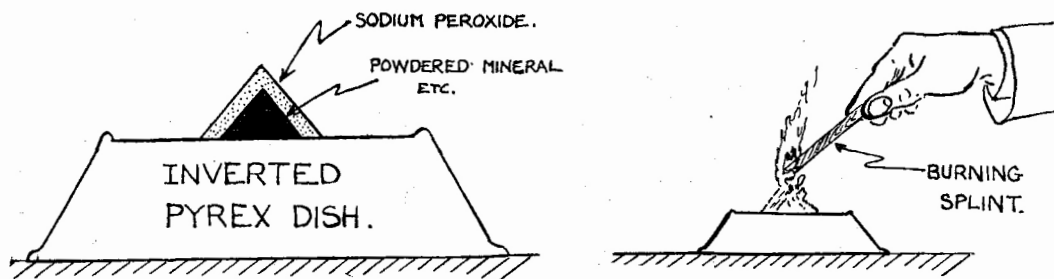
The chemical tests utilized for examining the fused products are either well-known or adaptations which have been made by the author.

Basis of the Methods

The methods described here are based on the following principles:—

(i) If wood or a powdered metal like magnesium is permitted to burn in the

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Figs. 1a and 1b.—Charge Prepared for Fusion and the Process Initiated.

presence of a strong oxidizing agent such as sodium peroxide intense heat is developed which may be utilized to bring about rapid reactions between finely-powdered minerals and suitable fluxes.

(ii) If a powdered metal such as magnesium is ignited in close contact with certain powdered minerals the latter will be reduced. Thus it is possible to reduce sulphates to sulphides and phosphates to phosphides.

Methods and Procedures

(1) *The Pyrex Dish Method*

In this method fusions and reductions are carried out on an oval Pyrex pie-dish, about 5½ in. long, which is inverted to form a "table." It forms an admirable base for the tests described for the following reasons:—

(i) Neither the heat generated nor the reagents used affect it to any marked extent.

(ii) The dish is robust—a most desirable property for apparatus used in the field.

(iii) The products of a given fusion or reduction may be examined by the direct application of liquid reagents either to the whole mass or to isolated portions of it. The raised rim permits of the application of several ml. of reagent.

(iv) The colours of reaction products can be emphasized by placing white, or suitably coloured, paper beneath the Pyrex table.

(v) If necessary the dish may be transported to a microscope stage and the products examined by microchemical means.

(vi) As the products of a given test are readily removed cleaning is very easy.

A normal fusion is carried out as follows:—

A cone from 5 mm. to 7 mm. diameter of finely-powdered mineral, or in some cases a 1 : 1 mixture of mineral and flux, is placed on the Pyrex table. The cone is covered with

double its volume of sodium peroxide and fused by applying a burning splint to the peroxide and maintaining it in contact with the assay until all the oxidizing agent has been consumed (Figs. 1a and 1b).

Occasionally it is necessary to make up the charge in a different manner to that described; in such cases it is clearly stated in the appropriate section.

To carry out a reduction, cover the mineral cone with about twice its volume of magnesium powder, or, better, photographer's flash-powder. Such material burns somewhat slower and less violently than the finer magnesium powder usually used in the laboratory. A little potassium chlorate, or sodium peroxide, depending on the test to be made, is placed on top of the powdered metal in order that the latter may be easily ignited. Ignition may either be made by touching the oxidizing agent with a burning splint, or by using a piece of touch paper supported by an asbestos-paper roof in the manner described below.

(2) *The Asbestos-Paper Method*

Alternatively, certain fusions can be carried out on a square inch of asbestos paper supported on an unglazed tile or an asbestos mat.

In the first procedure the charges are made up exactly as in the Pyrex-dish method and the method of ignition and fusion is the same.

In the second the procedure is as follows:—Cut out two pieces of asbestos paper, each about 1 in. square. Bend one along the central line and in the middle of it cut a square hole of about ½ in. in size. This is termed the roof. Place the uncut square on a tile or asbestos mat and in the centre of it a cone of the powdered mineral, or of mineral and flux, as in the Pyrex-dish method. Cover the cone with twice its volume of sodium

peroxide and place over the latter a volume of flash powder equal to half that of the peroxide. Place the asbestos roof over the charge and insert a piece of touch paper 1 in. long by $\frac{1}{8}$ in. wide through the "chimney" and into the charge folded longitudinally. Ignite the touch paper¹ with a match (Fig. 2).

When carrying out a reduction the charge is made up as in the Pyrex-dish method and either method of ignition may be used.

Chemical examination of the products of fusion or reduction on asbestos paper is best done on a spot plate. For these tests it is wise to cut out the portions of obviously fused material with scissors. Care should be taken to limit as far as possible the amount of asbestos paper taken, as this swells and disintegrates when wetted and may obscure results of the chemical tests.

Observations on the Processes

The Pyrex-dish method is superior to the asbestos-paper methods in that it permits of the easier testing of the products of fusion and there is no danger of introducing iron or magnesium ions into the product. Furthermore, the inconvenience associated with disintegrating asbestos paper is not encountered.

On the other hand the asbestos-paper methods possess the following advantages:—

- (i) Asbestos paper is cheap, sufficient for a great number of tests, and takes little space.
- (ii) Since a fresh piece of paper is used for each test there is no risk of contamination from previous test material and there is no fusion apparatus to clean.
- (iii) The roof and touch-paper method minimizes the flash and reduces charge dispersion during the moment of fusion.

¹ Touch paper is supplied with each bottle of flash powder.

When there is a possibility that ions liberated by partial fusion of the asbestos will interfere with subsequent tests a sheet of a suitable metal is used as a base. For example, sheet nickel makes an adequate base when testing for iron. Under such conditions the fusion products must be scraped on to a spot plate for subsequent testing.

Use of Sodium Peroxide

Sodium peroxide must be kept in a well-stoppered container and the stopper must be replaced *immediately* after use. The chemical is dangerous in that if water or acids come into contact with it the resultant reaction is very violent and fires may easily be started. Furthermore, as it reacts with water, forming sodium hydroxide (caustic soda), it should be kept well away from the skin. However, as only a small quantity of this chemical is used in any one test, any which may be left over after a given fusion may be treated with water or acids with practically no attendant danger.

Tests for Specific Elements and Radicals

- (i) The products of fusion are most readily dissolved by adding the attacking reagent and then disintegrating the solid in it by means of a glass rod.
- (ii) The safest procedure is to dispense small quantities of the organic spot reagent as and when they are required, as many of them are unstable.
- (iii) Intimate mixing of fluxes and minerals is best achieved by grinding the powders together in an agate mortar.
- (iv) In the majority of cases further information concerning the chemical tests utilized is to be found in the book by Feigl (1947)¹

¹ Reference at the end of this article.

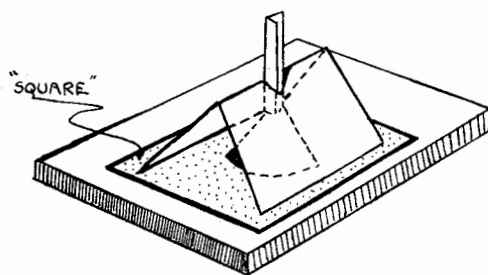
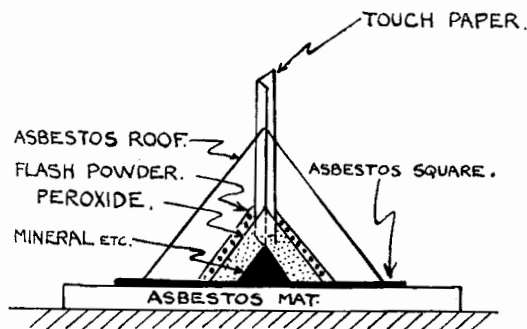


Fig. 2.—Fusion by the Asbestos-Paper Method.

and therefore for convenience of reference it is cited, whenever possible, rather than the original papers.

(v) Many minerals, although attacked by common acids, are decomposed very slowly, even when heat is applied. Some of these may readily be tested by subjecting them to a preliminary fusion, followed by treatment of the melt with cold reagent. Relevant examples have been incorporated in some of the following sections.

Aluminium

Minerals Tested.—Orthoclase and microcline, KAlSi_3O_8 ; albite, $\text{NaAlSi}_3\text{O}_8$.

Fusion Process.—Pyrex-dish method. Cover a 1:1 mixture of mineral and KHSO_4 with Na_2O_2 and fuse.

Test (Feigl, pp. 142, 143).—Dissolve the product in 5N NaOH and transfer some of the solution to a filter paper by dipping an edge of the latter into it and allowing time for a $\frac{1}{4}$ -in. band of solution free of solid particles to develop. Place first a drop of 5N acetic acid immediately beyond this band and then a drop of a saturated solution of Morin in methyl alcohol. Examine under either a long- or short-wave ultra-violet lamp. Aluminium is indicated by the presence of a green fluorescent band.

Interfering Elements.—Be, Zn, Ga, and Sc also form fluorescent compounds with Morin.

Other Notes.—Asbestos-paper methods of fusion should not be used, but the burning-splint method of fusion may be conducted on a nickel sheet.

Bauxite has not been included because the presence of aluminium in this substance may easily be shown by other simple methods.

Although KHSO_4 does not help when decomposing the above minerals it has been included because it increases the decomposition of other aluminium minerals.

A 0.1% aqueous solution of Pontachrome Blue Black R may be substituted for Morin in the above test (Feigl, p. 147). An orange-red fluorescence indicates aluminium and the test is specific.

Barium and Strontium

Minerals Tested.—Baryte, BaSO_4 ; celestine, SrSO_4 .

Reduction Process.—Pyrex-dish method. Cover the powdered mineral with flash powder and on top of the latter place a little Na_2O_2 . Ignite with a touch-paper or a burning splint.

Test (Feigl, pp. 165-167).—Divide the reduced mass into two portions and to each add one or two drops of sodium rhodizonate solution. The development of a brownish-red colour indicates the presence of either barium or strontium. This, however, is not always very apparent. To one such treated portion add a few drops of 5N HCl. A rose-red colour indicates the presence of barium. Should no colour develop, add a few drops of N acetic acid to the other portion. A pink colour indicates the presence of strontium.

Interfering Elements.—White lead minerals such as cerussite and anglesite are the only substances likely to cause confusion as these react in a similar manner to the above. However, the presence of lead in such minerals is easily established by streaking the mineral on a portion of unglazed tile and adding

in succession to the streak a drop of 10% KI solution, then a drop of 1:7 HNO_3 . The streak immediately becomes yellow.

Reagent.—A freshly prepared 0.2% aqueous solution of sodium rhodizonate.

Other Notes.—Asbestos-paper methods of reduction may be used.

As a barium flame is not readily obtained from baryte the above method is specially useful for detecting this mineral in the field.

Beryllium

Mineral Tested.—Beryl, $\text{Be}_3\text{Al}_2\text{Si}_6\text{O}_{18}$.

Fusion Process.—Pyrex-dish method. Cover a 1:1 mixture of mineral and KHSO_4 with Na_2O_2 and fuse.

Test (Feigl, pp. 149, 150).—Add a few drops of 5N NaOH to the fused mass and disintegrate the latter with a glass rod. Place two drops of p-nitrobenzeneazoorcinol on a double thickness of spot-reaction paper and on the same spot place a drop of 25% KCN (very poisonous). On this wet orange-coloured spot place a drop of the test solution. The presence of beryllium is indicated by the development of a pink to peach-red colour.

Interfering Elements.—None.

Reagent.—Dissolve 0.025 g. of p-nitrobenzeneazoorcinol in 100 ml. of 4% NaOH. Fresh solutions should be prepared fortnightly.

Other Notes.—The pink Be spot disappears as the paper dries.

The asbestos-paper burning-splint method may be used for the preliminary fusion.

The test is of considerable value in the field as it permits of the rapid differentiation between massive beryl and other somewhat-similar minerals such as quartz and feldspar.

Chromium

Mineral Tested.—Chromite, $\text{FeO} \cdot \text{Cr}_2\text{O}_3$.

Fusion Process.—Pyrex-dish method. Cover a 1:1 mixture of the mineral and Na_2CO_3 with Na_2O_2 and fuse. The distinct yellow colour of the product indicates chromium, but the element should be confirmed as follows:—Dissolve a portion of the yellow product in a minimum of 5N acetic acid, but ensure that the final solution is acid. Place a fragment of silver nitrate in it. The presence of chromium (as chromate) is indicated by the crystal becoming coated with pink silver chromate.

Other Notes.—Asbestos-paper methods of fusion may be used.

Iron

Minerals Tested.—Magnetite, Fe_3O_4 ; ilmenite, $\text{FeO} \cdot \text{TiO}_2$; chromite, $\text{FeO} \cdot \text{Cr}_2\text{O}_3$; franklinite, $(\text{Fe}, \text{Mn}, \text{Zn})\text{O} \cdot (\text{Fe}, \text{Mn})_2\text{O}_3$; wolframite, $(\text{Fe}, \text{Mn})\text{WO}_4$; columbite, $(\text{Fe}, \text{Mn})(\text{Nb}, \text{Ta})_2\text{O}_6$; hematite, Fe_2O_3 ; glauconite, essentially a hydrated silicate of iron and aluminium.

Fusion Process.—Pyrex-dish method. Cover the mineral with Na_2O_2 and fuse.

Test.—Dissolve a portion of the fused mass in 5N HNO_3 and ensure that the resulting solution is acid. Add a drop of ammonium mercuric thiocyanate to it. The development of a red solution indicates iron.

Interfering Elements.—Certain elements—such as, Co, Zn, and Cu—react with the reagent producing precipitates, but these do not obscure the red colour due to iron.

Reagent.—Dissolve 8g. mercuric chloride and 9 g. ammonium thiocyanate in 100 ml. water. Allow to stand several days before using. The reagent is stable.

Other Notes.—As an alternative the fusion may be carried out on aluminium or nickel sheet and either method of ignition and fusion may be used.

The presence of white feathery cruciform crystals, seen only under the microscope, in the red franklinite solution, confirms the presence of zinc.

Manganese

Minerals Tested.—Pyrolusite, MnO_2 ; psilomelane, a hydrated oxide; braunite, Mn_2O_3 ; manganite, $\text{MnO}(\text{OH})$; franklinite, $(\text{Fe}, \text{Mn}, \text{Zn})\text{O}(\text{Fe}, \text{Mn})_2\text{O}_3$; wolframite, $(\text{Fe}, \text{Mn})\text{WO}_4$; manganotantalite and columbite, $(\text{Fe}, \text{Mn})(\text{Nb}, \text{Ta})_2\text{O}_6$; rhodonite, MnSiO_3 ; rhodochrosite, MnCO_3 .

Fusion Process.—Pyrex-dish method. Cover the mineral with Na_2O_2 and fuse. The presence of manganese is indicated by the development of a green or blue-green fusion product. Further confirmation, though usually unnecessary, may be obtained by adding a drop of 5N HNO_3 to the fused mass. This results in the development of a transient reddish-purple solution.

Other Notes.—Asbestos-paper methods of fusion are quite satisfactory.

This test constitutes the quickest method available for confirming the presence of manganese in those minerals in which it occurs as an essential constituent. For this reason minerals such as pyrolusite and rhodochrosite, which are readily soluble in acids, have been included.

Molybdenum

Mineral Tested.—Molybdenite, MoS_2 .

Fusion Process.—Pyrex-dish method. Cover the mineral with Na_2O_2 and fuse.

Test (Feigl, p. 93).—Dissolve a portion of the fused mass in 5N acetic acid and ensure that the solution is acid. Add a small fragment of potassium ethyl xanthate. The presence of molybdenum (as molybdate) is indicated by the solution becoming purple.

Interfering Elements.—None.

Other Notes.—Asbestos-paper methods of fusion are satisfactory alternatives to the above method.

Phosphates

Minerals Tested.—Apatite, $3\text{Ca}_3\text{P}_2\text{O}_8\cdot\text{CaF}_2$; pyromorphite, $3\text{Pb}_3\text{P}_2\text{O}_8\cdot\text{PbCl}_2$; plumbogummite, $\text{PbAl}_2(\text{OH})_2\text{P}_2\text{O}_7$; libethenite, $\text{Cu}_2(\text{OH})\text{PO}_4$; tarbuttite, $\text{Zn}_3\text{P}_2\text{O}_8\cdot\text{Zn}(\text{OH})_2$; vivianite, $\text{Fe}_2\text{P}_2\text{O}_8\cdot 8\text{H}_2\text{O}$; wavellite, $4\text{AlPO}_4\cdot 2\text{Al}(\text{OH})_3\cdot 9\text{H}_2\text{O}$.

Reduction Process.—Pyrex-dish method. Cover the mineral with flash powder and on the latter place a comparatively-small volume of Na_2O_2 in order to aid ignition. Ignite with a burning splint.

Test.—Place a drop or two of water on the reduced product. A mixture of hydrides of phosphorus is liberated, the characteristic odour of which confirms the presence of phosphate in the mineral under test.

Other Notes.—The reduction may be carried out on asbestos paper.

Sulphates

Minerals Tested.—Barite, BaSO_4 ; celestine, SrSO_4 ; gypsum, $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$; anglesite, PbSO_4 .

Reduction Process.—As for phosphates, excepting that KClO_3 takes the place of Na_2O_2 .

Test.—Add a few drops of sodium azide/iodine solution to the product of reduction. The marked evolution of bubbles of nitrogen confirms the presence of sulphate ion in the original mineral (Feigl, pp. 227–229).

Interfering Substances.—All mineral sulphides react with the reagent liberating bubbles of nitrogen.

Reagent.—Dissolve 3 g. sodium azide in 100 ml. 0.1N iodine solution. The reagent is stable.

Other Notes.—The reduction process may be carried out on asbestos paper.

Phosphides derived from the reduction of phosphates will react with the water of the reagent liberating bubbles of hydrides of phosphorus. These will not be mistaken for bubbles of nitrogen as the former have a characteristic odour.

The addition of dilute HCl to the product of reduction usually results in the generation of sufficient H_2S for it to be recognized by its odour. Therefore this test may be substituted for the more sensitive sodium azide/iodine test.

Titanium

Minerals Tested.—Ilmenite, $\text{FeO}\cdot\text{TiO}_2$; rutile, TiO_2 ; sphene, $\text{CaO}\cdot\text{TiO}_2\cdot\text{SiO}_2$.

Fusion Process.—Pyrex-dish method. Cover a 1:1 mixture of the powdered mineral and KHSO_4 with an equal volume of Na_2O_2 and fuse.

Test (Feigl, pp. 151–152 and p. 440).—Add a few drops of hot stannous chloride/hydrochloric acid solution to a portion of the fused mass and disintegrate the latter with a glass rod. Add one or two drops of a freshly-prepared 5% aqueous solution of the sodium salt of chromotropic acid to the acid test solution. The presence of titanium is indicated by the solution becoming red-brown. The colour may be more clearly appreciated if some of the solution is taken up on a piece of spot-reaction paper.

Interfering Elements.—None.

Reagent.—The stannous chloride/hydrochloric solution is best prepared by boiling 2g. to 3g. of tin in about 5 ml. of hydrochloric acid for about five minutes. Leave the undissolved tin in the solution, and on subsequent occasions reboil for two minutes before using.

Other Notes.—Fusion may also be carried out on asbestos paper provided that the burning-splint method is used.

Tungsten

Mineral Tested.—Wolframite, $(\text{Fe}, \text{Mn})\text{WO}_4$.

Fusion Process.—Pyrex-dish method. Cover the mineral with Na_2O_2 and fuse.

The blue-green colour of the product indicates the presence of manganese.

Test.—To a portion of the fused mass add a few drops of warm stannous chloride/hydrochloric acid solution, at the same time disintegrating the mass with a glass rod. The development of a blue precipitate indicates the presence of tungsten.

Interfering Elements.—When a more complete fusion is carried out in a crucible Ti and Nb interfere by forming coloured solutions when the stannous chloride is added, but these elements do not interfere when the test is done in the manner described above.

Reagent.—The preparation of the stannous chloride/hydrochloric acid solution is described under titanium.

Other Notes.—Either of the asbestos-paper methods may be used for the fusion.

Tungsten in any mineral may be readily confirmed by the following rapid-fusion method:—Mix a little of the powdered mineral with twice its volume of ammonium hypophosphite and place it in an open tube and near to one end. Heat the charge with a match and whilst the former is hot add a drop or two of water. The development of a deep blue to purple solution is a certain indication of the presence of tungsten (De Ment and Dake, 1946, p. 204).

Uranium

Mineral Tested.—Pitchblende — essentially $2\text{UO}_3 \cdot \text{UO}_2$.

Fluorescence Test.

Fusion Process.—Pyrex-dish method. Cover a cone composed of equal volumes of mineral, NaF, and KHSO_4 with about half its volume of Na_2O_2 and fuse. Examine the product under either a short-wave or long-wave ultraviolet light. The presence of uranium is indicated by the appearance of brilliant-yellow fluorescent spots.

Other Notes.—Either asbestos-paper method of fusion may be utilized.

Nigerian columbite, when similarly treated, results in a product which fluoresces a pale yellow.

Potassium Ferrocyanide Test.

Mineral Tested.—Pitchblende.

Fusion Process.—Pyrex-dish method. Cover the mineral with Na_2O_2 and fuse.

Test.—Dissolve a portion of the fused mass in 1 : 1 HNO_3 and ensure that the resulting solution is acid. Make two radial cuts about one-third of an inch apart in a 10 cm. disc of Whatman No. 1 filter-paper. Bend the tail so formed at right-angles to the paper. At the intersection of the tail and the main body of the paper place a drop of the test solution. Half fill a small crucible with a 1 : 5 mixture of nitric acid and ethyl acetate and put into a deep Petri dish of diameter slightly less than that of the filter paper. Rest the paper on the Petri dish

so that the tail dips into the crucible. Cover the paper with an inverted Petri dish of diameter the same as the one mentioned above. Hold in place with a sufficiently heavy weight to ensure that the apparatus is gas-tight. When the liquid has diffused over an area of the paper equal to that of a five-shilling piece remove the paper and spray it with a 1.0% aqueous solution of potassium ferrocyanide. (It is preferable but by no means necessary to dry the paper before spraying). The presence of uranium is indicated by a brown ring separated by a colourless zone from a central spot, which is often blue due to the presence of iron.

Interfering Elements.—None.

Other Notes.—Either asbestos-paper method may be used.

Zirconium

Minerals Tested.—Zircon, ZrSiO_4 ; baddeleyite, ZrO_2 .

Fusion Process.—Pyrex-dish method. Cover the mineral with Na_2O_2 and fuse.

Test (Feigl, pp. 155–157).—Dissolve a portion of the fused mass in 2N HCl. Place a drop of the acid-test solution on a piece of spot-reaction paper which has been previously impregnated with the organic reagent and dried. Wash the paper for one minute in hot (c. 60° C.) 2N HCl. The presence of zirconium is indicated by the appearance of an orange to brown spot or ring.

Interfering Substances.—Molybdates, tungstates, and titanium salts may interfere.

Reagent.—Dissolve 0.1 g. p-dimethylaminoazophenylarsonic acid in a mixture composed of 95 ml. ethyl alcohol and 5 ml. conc. HCl.

Other Notes.—The asbestos-paper burning-splint method of fusion may be used.

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South African Asbestos Fibres

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Asbestos minerals and
their impurities are
reviewed and fibre
grading discussed

(Concluded from the August issue, p. 83)

Part 2.—Determination of the Impurities and the Grading of Fibre.

(1) *The Nature of the Impurities.*—Fibre seams as recovered in mining are partly broken up into aggregates of loosened fibre bundles and finely-broken rock and partly into larger lumps consisting of country rock

with portions of the asbestos seams lying between or attached thereto. Depending on fibre width, large lumps are separated into portions for hand-cobbing or milling, while the loosened mass of fibre goes straight to milling processes.

The ore is crushed in the mill and the fibre "opened-up" (fiberized) and freed from the