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METHODS IN PRACTICAL PETROLOGY

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Methods in Practical Petrology

HINTS ON THE PREPARATION AND EXAMINATION OF ROCK SLICES

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INTRODUCTION.

This volume is intended to act as a practical companion to the standard Petrological text-books.

For this reason, we have omitted all detailed descriptions of rocks, minerals, and structures.

We have tried to indicate the great importance of methodical procedure in the microscopical work, which forms such an important branch of Petrology.

Although the usual University examinations do not require a knowledge of the preparation of rock slices, we have made this an important feature of the book.

We have found that very little literature on this subject is easily accessible to the student, and we have, therefore, described the processes involved in some detail, in the hope that the book may be of use, not only to students, but to those who are starting research, and to others who have been accustomed in the past to send their material to Germany. With this object in view, we have only described methods which we have satisfactorily employed in the Sedgwick Museum at Cambridge, and we do not attempt to deal with elaborate processes suitable for firms operating on a commercial scale.

In the Appendix we have given the methods of preparation of some of the stains employed in microscopical work, so that any chemical student may be able, if necessary, to prepare the small quantities which he may require.

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INTRODUCTION

We particularly desire to thank Prof. T. McK. Hughes for his kindness in placing at our disposal the rock slice department of the Sedgwick Museum, and also Prof. T. G. Bonney, Dr. A. Harker, and other members of the staff for their kind help and assistance throughout our work at the Museum.

> H. B. MILNER. G. M. PART.

TRINITY COLLEGE, CAMBRIDGE, 1916.

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CHAPTER I.

PREPARATION OF ROCK SLICES.

Apparatus necessary—Rock cutting machines—Preparation of material — First grinding — Mounting — Second grinding — Minerals liable to give trouble—Finishing.

Rocks present such a wide variation in texture, hardness, cohesion, composition, and the mutual relations of their constituent minerals, that many methods for the preparation of rock slices have been employed by different workers to suit their particular requirements.

In this chapter we shall describe the methods which we have found to be generally applicable to most kinds of rock, though certain types will necessarily demand special treatment. More particularly is this the case with some coarse varieties where the constituent minerals are loosely intergrown.

Fine textured rocks are in general simpler to deal with, their internal structure materially aiding the production of a thin slice. In general slices should not be more than $\frac{1}{1000}$ inch thick, when quartz and felspar, if present in the rock, will show grey tints of the first order when examined in polarized light.

In the case of very fine textured rocks, it may be advisable to get them even thinner than this if possible, a matter which requires much skill on the part of the operator.

B

METHODS IN PRACTICAL PETROLOGY

The possession of apparatus of good quality is of paramount importance, and will save much time and trouble in the long run. A simple form of microscope, fitted with a low power objective and polarising prisms, will be required ; or a very convenient instrument can easily be devised with a strong pocket lens, on the lines of the simple dissecting microscope familiar to biologists. This consists of a stage to carry the slide, with a mirror underneath, and a vertical pillar supporting a horizontal arm at the end of which the lens and analyser are fitted. The polarizer is placed below the stage. Focussing is effected by moving the arm up or down the pillar.



FIG. I.

A good steel or bronze plate (steel for preference), 1 ft. square $\times \frac{1}{2}$ inch thick, and two glass plates of the same dimensions, will be necessary. The plates should be contained in wooden trays (teak has the advantage of being waterproof) not less than 15 inches square, with a 1 $\frac{1}{2}$ inch beading. This latter keeps the grinding material from splashing on to neighbouring plates, and incidentally this waste from grinding serves to prevent the plates from sliding about in the trays.

There are many kinds of abrasives used, but the one which seems to meet most requirements is an artificial product, *Carborundum*, supplied in many grades, of which the most generally useful are 70 (coarse), 220 (medium), and 30 M.M. (fine).

The mountant used is *Canada Balsam*, the pure material, and not a solution in benzene. The balsam is a thick viscous liquid as supplied, but if heated strongly it melts and sets hard on cooling.

A copper plate heated by gas or methylated spirit lamp is used for the purpose of 'cooking' the balsam; an iron plate may be employed, but we have found that copper provides a more uniform heating surface and does not tend to warp as some iron plates do.

Glass microscope slides of the usual size, 3 inches \times 1 inch, and cover glasses 1 inch $\times \frac{7}{8}$ inch and $\frac{7}{8}$ inch circles, are recommended.

Minor accessories include a blunt knife and a pair of forceps.

Though not by any means an essential from the student's point of view, it is often convenient to employ a ROCK CUTTING APPARATUS. Many forms of this have been devised, of which the most satisfactory is probably that in which the cutting disc rotates in a horizontal plane.



FIG. 2.

In this form of apparatus (fig. 2), a vertical axis (A) carries a circular mild steel cutting disc (B) about 6 inches in diameter and $\frac{1}{3'2}$ inch thick. This is rotated by a gut band from the large driving wheel (C) worked by the treadle (D). The rock to be cut is clamped in a vice (E), movable so that it may be gradually fed up to the disc as the cut deepens. The disc, vice, etc., are contained in a deep metal tray (F).

The lubricant used may be either soapy water or paraffin, kept in a small tank (G) above the cutting disc. The lubricant may be allowed to drip on to the disc by means of a tap, or may be applied with a brush.

The disc is armed with diamond dust. To do this, it is first notched at close intervals round its circumference with a blunt knife. A minute quantity of the diamond dust is mixed to a thin paste with a little machine oil and applied to the edge of the disc with the finger tip. The disc is then rotated against a flint or piece of agate, in order to bed the diamond into the metal. One such arming should be sufficient to cut a considerable number of rocks. Care must be taken not to let the disc become dry during the process of cutting or the diamond will be immediately stripped from its edge.

Very good results can also be obtained from the cutting apparatus in which the disc is carried on a horizontal lathe shaft. The general characters of this type of machine may be gathered from the figure (fig. 3). It may be driven either by a treadle or by an electric motor; the latter power ensures a regular speed, but is not so easily regulated to suit the work in hand as the former. In this apparatus, diamond dust may be employed for arming the cutting disc, as in the machine previously



FIG. 3.

described. But by fitting a specially hardened steel disc, carborundum may be used; this, mixed with sufficient water to form a paste, is applied freely with a brush to the edge of the disc during the process of cutting. Water, which is contained in a trough beneath the disc, is used as the lubricant.

The rock to be cut is clamped in a vice which operates from a back iron stay, parallel with the shaft.

In both these types of rock cutting machine, it is most important that there should be no play in the shaft and that the disc should run absolutely true. A carborundum wheel will be found very useful for rough grinding and should be interchangeable with the cutting disc. It must be used with plenty of water. These wheels are made in a variety of grades. We have found a wheel of 70 grit, grade M, quite satisfactory for all ordinary purposes.

Assuming that the student has got his materials ready, the first thing is to obtain a suitable piece of rock to work with. In the majority of cases, a convenient chip may be obtained by flaking the specimen with a hammer.

When collecting in the field, it is easy to select chips broken off a rock in making a hand specimen, and two or three such chips should always be kept. They should be about an inch square and as thin as can be reasonably obtained. This last condition will vary with different rocks, so that in dealing with some, it is more convenient to cut a piece with the rock cutting apparatus, if one is available. Especially is this the case if the rock has to be cut in a special direction, as when dealing with slates, when it is often necessary to cut a section across the cleavage.

In cases of very friable or porous rocks we have found it advisable to boil the chip for about five to ten minutes in balsam, to which a little *shellac* has been added, before commencing operations. This can be conveniently carried out in a tin lid.

This treatment will fill the cracks or vesicles in the rock and prevent shattering during the subsequent grinding; it is, in addition, particularly valuable during the final stages of the first grinding of such rocks, as a uniform surface cannot in many cases be obtained owing to the tendency of existing holes and cracks . between the crystals to persist.

As examples of rocks which can be advantageously treated in this manner, we may mention :—

Many granites (e.g. Ballachulish, N.B.; Peterhead, Aberdeenshire, N.B.).

Luxulyanite and other coarse Tourmaline rocks (e.g. Cornwall).

Coarse Hornblendic rocks (e.g. Garabal Hill, N.B.). Eclogites (e.g. Bergen, Norway).

Shonkinites (e.g. Monzoni district, Tyrol).

Coarse Pyroxenic rocks (e.g. Cran, Norway).

Coarse Peridotites (e.g. Harrisite, Rum, N.B.).

"Trachytes" of the Melrose type.

All vesicular volcanic rocks, tuffs, etc.

Most Garnetiferous rocks.

Phyllites and Schists.

First Grinding. For this, some of the coarse carborundum (grade 70) is spread on the first (steel) plate and mixed with water.

The chip is then ground down on one side until as large an area as possible, of a uniform character, is obtained. It is then washed to free it of all traces of carborundum and transferred to the second (medium) plate, where a similar grinding with finer carborundum (grade 220) removes the scratches made by the coarse material. (If a carborundum wheel is available, much time will be saved by facing ordinary hard rocks on it preparatory to the first grinding. In many cases if this is done, the use of the coarse plate may be dispensed with, and the chip, after facing, should be washed and transferred to the second plate.) It is most essential that the chip should be well washed to remove all traces of the coarse powder before using the second plate, and the same applies on transferring it from that to the fine plate, where the rock is polished with the 30 grade carborundum, preparatory to mounting. (N.B.—Some workers prefer to use very fine "flour" emery (FFFF grade) instead of carborundum on this third plate.) The finer the surface obtained, the better the rock will adhere to the glass on which it is mounted, and to this end, many of the rocks cited above will repay a final polishing with *putty powder* and water, on a green baize strip stretched on a board. (N.B.—This method may be employed for polishing fossils or rock specimens.)

The chip is now washed and dried on the hot plate.

Mounting. The next operation is to mount the polished chip on a glass slide, and on this, the success of the whole process depends. Some balsam is spread on a glass slide and heated on the copper plate. It is sufficiently 'cooked' when a thread drawn out between the points of a pair of forceps, becomes brittle when cold. Insufficiently cooked balsam will absorb carborundum during the second grinding. Overcooked balsam is too brittle and will break away, thus exposing the edges of the rock chip which it is there to protect.

When the balsam is just right for mounting, the rock chip, which has also been heating meanwhile, is placed, polished surface downwards, on the slide, and firmly pressed down, care being taken to exclude air bubbles. If any of these remain the slide must be re-heated and the chip worked about until the bubbles are pressed out. When cold, the second grinding may be proceeded with. Second Grinding. The chip is now ground on the coarse plate until it is about 1 m.m. thick, the glass slide



FIG. 4.

being used as a convenient holder. It is then well washed and transferred to the medium plate. (N.B.—When a rock-cutting machine of the horizontal type is available the chip may be cut very thin by holding the glass slide, to which it is attached, just below the level of the cutting disc (fig. 4). In this way it is possible to leave only a thin layer of rock adhering to the glass, and, after washing, this may be dealt with on the medium plate, thus avoiding the often tedious coarse grinding on the first plate. It must be understood, however, that this use of the rock-cutting machine requires great skill on the part of the operator, and should not be attempted until the student has had considerable practice in its ordinary manipulation.)

If care is taken, grinding may be continued on the second plate until quartz or felspars, if present in the rock, show first order reds and yellows with polarised light.

At this point it is advisable to wash and transfer to the fine plate. It will sometimes be found that the balsam tends to come away from the edges of the rock a little before this stage is reached. In such cases the slide should be transferred as soon as this happens; or if the balsam behaves very badly in this respect (showing that it has been overcooked) the slide should be washed and dried, and a little freshly cooked balsam applied round the edges of the chip.

Care must be taken to avoid unequal pressure on different parts of the slice during the final stages, and any tendency to thickness in any particular part may be checked by grinding that part on the edge of the plate until a uniform thickness is once more obtained.

The thinness of the slice ultimately attained will depend very largely on the skill of the operator. It is advisable not to use too much water or carborundum on the fine plate, and to check carefully the progress of the operations with the microscope.

When the slice is as thin as desired, it is washed and dried ready for finishing.

Certain constituent minerals of many rocks render the final stages of the grinding a somewhat delicate operation, so that the student has sometimes to choose between having a slightly thick slice and sacrificing some of its minerals. Very good examples of this are Garnetiferous Phyllites, where sections thin enough to show the composition and structure of the main body of the rock will usually be devoid of the garnets, which may drop out even before the section is transparent. Or again, Felspar-Mica Schists, where the soft mica occurring interstitially between the felspar laths will be ground away before the felspar is reasonably thin. The following minerals are always liable to give trouble in this way :—

Hornblende and Augite.

Garnet. Olivine. Leucite. Corundum,

and occasionally quartz, if present in large grains embedded in a soft matrix.

Much trouble of this kind may be avoided by suitable 'cooking' during the first grinding, and by even pressure during the final stages, but in some cases it is almost impossible to avoid losses of this nature.

Frequently, glassy rocks such as Tachylites and Variolites are hard to grind really thin, but provided a section is sufficiently transparent for examination by ordinary light, this will not be of extreme importance.

In the case of rocks which do not contain evident quartz or felspar, other minerals will have to be used as indices of thickness. Augite and Olivine should not exceed a thickness giving higher colours than those of the second order, while the calcite of limestones should show the delicate colours of the third and fourth orders in polarised light.

Finishing of Slide. Having carried the process of the preparation of the rock slice as far as the end of the second grinding, it now remains to complete the slide for examination. To this end it is first dried on the hot plate, but not sufficiently heated to loosen the balsam which cements the rock to the glass. It is then allowed to cool. On another glass slide, some fresh balsam is cooked as previously described, and poured over the rock slice. A cover glass of suitable size is then placed on top of the balsam and the whole transferred to the hot plate. As the temperature rises, the balsam becomes less viscuous, and the cover glass is gently pressed down on to the slice, at the same time expelling any air bubbles present. At this point the surplus balsam can be removed with a knife while still fluid and the slide allowed to cool.

Probably many variations on this method can be employed; for instance the balsam might be heated on the cover glass, but we have found the above method generally satisfactory, as the rock remains firmly fixed to the glass throughout the operations, and there is little tendency for the constituent minerals to disintegrate.

Many workers make a practice of transferring the slice, when finished, to a new glass slide. To do this, the original slide is heated on the hot plate with some fresh balsam ; at the same time some more balsam is heated on a clean glass slide in the usual manner. When the rock slice has been heated sufficiently to melt the balsam which cements the rock to the glass on which it has been ground, a cover glass may be placed on the top of it and then worked off on to the new glass slide, with the rock section adhering to its under-surface.

Owing to the tendency of many rocks to break up during this last operation, the process is not to be recommended, except for the purpose of remounting broken slides.

Microscope slides thus prepared may be conveniently cleaned by soaking in methylated spirit, when the surplace balsam washes off as a milky white precipitate. The slide is then dried with a clean cloth, and is ready for use.

CHAPTER II.

EXAMINATION OF ROCK SLICES.

Method of working—Different stages discussed—Determination of Minerals—Becke's Line—Significance of Rock Structures— Mode of working with Sedimentary Rocks—Hybrid Rocks— Xenoliths.

In this chapter we propose to give an outline of the methods which we have employed in the examination and determination of rocks in thin slices.

Haphazard methods often lead to erroneous conclusions, and much time and trouble may be saved by a methodical process.

At the outset a great deal can be learnt by holding the slide up to the light and examining it with a pocket lens.

If the hand specimen of the rock is available, it should be carefully studied at the same time, for macroscopical details of structure and composition.

The following mode of procedure should now be adopted, though with practice the student will perform the preliminary stages instinctively. (It will be seen that the process involved is one of elimination.)

 Determination as to whether the rock is Igneous or Sedimentary.^x

If sedimentary, see p. 38.



ILLUSTRATING METHOD OF WORKING.

- 2. If Igneous, whether Plutonic, Hypabyssal, Volcanic or Metamorphic.¹
- 3. Whether Acid, Intermediate, Basic or Ultrabasic.
- 4. Determination of Constituent Minerals in order of crystallisation if possible, i.e. Rosenbusch's order of decreasing basicity.²

Frequently deviations from this order may be observed, but in any case it is convenient to determine the minerals as follows :---

- (A) Accessories.
- (B) Ferromagnesian minerals.
- (c) Felspars and Felspathoids.
- (D) Quartz (if present).]
- 5. From the results of (4) whether the rock belongs to the Alkaline or Calcic branch, or is intermediate between these two.³
- 6. Note structures and any special characteristics.⁴

7. Inference as to probable nature of the rock.

We will now proceed to discuss some special points which may arise in each of the above stages; in most cases the method holds good, but it must be clearly understood from the outset that it is merely a means to

¹ In considering metamorphic rocks, it must be remembered that they may originally be of either igneous or sedimentary origin. The metamorphic character of a rock will usually be apparent from stages (4) and (6), and therefore any rock not definitely sedimentary is best treated on the lines suggested above.

²Harker. Natural History of Igneous Rocks, p. 180; also

Petrology for Students, 1908, pp. 23 and 24. 3 Harker. Natural History of Igneous Rocks, Ch. 4. 4 ibid., Ch. 11; also Michel Lévy. Memoire sur les divers modes de structure des roches eruptives, 1875.

an end, and that rocks do not necessarily always accommodate themselves to such an arrangement.

Thus it is sometimes difficult to decide in the case of the Charnockite series of Southern India,^x to what extent a given rock may be described as plutonic or metamorphic.

Again, some of the so-called "Syenites" of Charnwood Forest, Leicestershire, appear to be the common meeting point of Syenites (as ordinarily understood), Diorites and Granophyres (see also under "Hybrids," p. 41).

I. Igneous or sedimentary origin of the rock. In general the igneous or sedimentary origin of a rock will be obvious at a glance, but in some cases confusion may arise. Careful examination of a slice will, however, nearly always show a clastic or fragmental structure, and usually the presence of some cementing material between the grains, in the case of sedimentary rocks. Limestones are easily determined if crystalline calcite or dolomite are present, or if the remains of organisms make up part of the rock. Calcite and dolomite will show their characteristic birefringence colours with polarised light, and generally grey colours of the first order, characteristic of pyrogenetic quartz and felspar will be absent from the slice.

Holocrystalline, porphyritic or glassy structures, so characteristic of different groups of igneous rocks, are not found in *unaltered* sedimentary material.

2. Plutonic Rocks. Plutonic rocks are characterised by a moderate to coarse holocrystalline structure in

Holland. Mem. Geol. Sur. India, Charnockite Series, 1900.

which the main mass of the rock consists of an aggregate of mutually interfering crystals. As a rule, only the accessory minerals, which are the first products of crystallisation, show good idiomorphic outlines.

Porphyritic structure is not common, except in the more acid members of the series.

Glassy structures do not occur.

Hypabyssal Rocks. Many authors object to this division of rocks, and regard them as dyke phases of the Plutonic group, but there are so many rock types which do not conveniently fall into either the Plutonic or the Volcanic groups, that they are best treated as a separate division intermediate between these two.

Hypabyssal rocks are in general characterised by a holocrystalline and porphyritic structure. In many types, the ground mass of the rock is very fine grained and may even be glassy, though some of the larger masses approach the Plutonic group in structure, *e.g.* Dolerites.

Porphyritic structure involves the presence of some of the constituent minerals in two generations, and this will necessitate a slight modification in the method of procedure outlined in (4) (see p. 22). The phenocrysts and the ground mass should be dealt with separately, the order suggested in stage (4) being applied to the minerals of each group *in turn*, though it must be remembered that usually no particular order of crystallisation can be distinguished for the phenocrysts.

Volcanic Rocks. These rocks may be holocrystalline, but commonly contain more or less glass. The main characteristic of those which are not *wholly* glassy, is the porphyritic structure described above, though a few non-porphyritic types occur. Certain minerals such as hornblende, biotite, and muscovite, characteristic of the plutonic, hypabyssal and metamorphic rocks, are not usually found. Hornblende and biotite, if present, are almost always greatly corroded; muscovite will only occur as an alteration product in decomposed rocks.

Many lavas are characterised by the presence of amygdales, often filled with calcite, zeolites and other secondary minerals, and by a parallel orientation of the predominant minerals, due to flow.

Certain of the common types of volcanic rocks occur also as intrusions (sills and dykes), and in the absence of field evidence, cannot be distinguished from effusive lavas (e.g. Basalts, and the Trachytes and related alkaline instrusions of Carboniferous age in Southern Scotland).^I

[Pyroclastic rocks will be dealt with under the sedimentary division (p. 40).]

Metamorphic Rocks. These frequently show traces of crushing (e.g. strain shadows in quartz, or granular structure), and banding, due either to the thermal metamorphism of sediments, whose original stratification has not been obliterated, or to the crushing of igneous rocks subsequent to their consolidation (dynamic metamorphism).²

¹McRobert. Q.J.G.S., 1914, Vol LXX, p. 303 et seq.

² It should be noted that many granites which are of undoubted primary igneous origin (*e.g.* Laurentian of Canada) show a gneissic structure due to flow, but having been formed under conditions of great pressure, they exhibit many of the characters of metamorphic rocks. Metamorphic rocks are also in many cases characterised by the development of certain minerals which require special *mineralising agents*^I for their formation, and by others which are not usually of magmatic origin.

3. Acid Rocks. Acid rocks are characterised by the presence of a quantity of free quartz and alkali felspars.

Quartz sometimes occurs in quite basic types, but never in any quantity, and in such cases, the basic felspars will indicate the nature of the rock (*e.g.*, Quartz Gabbro, Carrock Fell,² and Quartz Dolerite, Whin Sill³); Felspathoids never occur.

Intermediate Rocks. These are characterised by predominant felspar; quartz, at the most, is only an accessory. Ferromagnesian minerals are important in these types, chiefly augite and hornblende. In the absence of quartz, felspathoids may be present.

Basic Rocks. The chief feature of these rocks is the presence of lime-soda felspars and pyroxenes as predominant constituents. Olivine is important in many types, while quartz is typically absent.

In alkaline magmas, alkali felspars and felspathoids may partially take the place of the lime-soda felspars (e.g. Essexites, Monteregian Hills, Quebec).⁴

Iron ore minerals, often titaniferous, are important accessories.

1 Harker. Nat. Hist. Ig. Rocks, Ch. 12.

² Harker. Q.J.G.S., 1894, Vol. 50, p. 311.

3 Teall. Q.J.G.S., 1884, Vol XL., pp. 640-657.

4 Internat. Geol. Congress, 1913, Guide No. 3. Excurs. Monteregian Hills. **Ultra-basic Rocks.** Felspar, if present, is of a basic type, but sometimes is wanting altogether. Except in the anorthite-peridotites (*e.g.* Allivalite, Rum, N.B.)^r felspar is subordinate to the ferromagnesian minerals, the chief of which is olivine.

In alkaline types, felspathoids may be important constituents (e.g. Alnöite, Alnö, Sweden)² and in many cases spinellid minerals are common accessories.

4. Determination of Minerals. We do not propose to discuss here the general properties of rock forming minerals as the student will find these fully set out in the standard works on the subject.³ We shall, however, describe the general mode of procedure in determining minerals and note certain cases where confusion may easily arise. We give, under the various headings, tables containing some useful data for dealing with these.

General method of procedure for determination of minerals in a rock slice.

(a). WITH TRANSMITTED WHITE LIGHT. Note colour, crystalline form, cleavage, clearness of outline and visibility of surface, the two latter giving an idea of the refractive index.⁴ This may be higher or lower than that of the balsam in which the slice is mounted; we may note here that balsam has approximately the same

1 Mem. Geol. Sur. Geol. of Small Isles, 1908, Ch. 7.

² Harker. Petrology for Students, 1908, p. 160.

3 Iddings. Rock Minerals, 1906; also Miers. Mineralogy, 1902.

4 Sorby. On a new method of studying the Optical Properties of Minerals, York Pol. Soc., 1878. refractive index as quartz (1.55). In consequence, clear quartz is nearly invisible in ordinary light, while garnet (refractive index 1.7-1.8) exhibits a very distinct outline combined with a 'rough' surface.

Opaque minerals should be examined with reflected light; this may be done by shielding the light from the mirror below the microscope stage, and reflecting light on to the slide by means of a white card held above it, providing the light is strong enough.

Table of Mean Refractive Indices.							
Fluor	1.43	Muscovite	1.58	Hypersthene	1.70		
Sodalite	1.48	Biotite	1.59	Barkevicite	1.70		
Hauyne	1.49	Dolomite	1.59	Zoisite	1.70		
Analcime	1.49	Topaz	1.62	Arfvedsonite	1.71		
Leucite	1.51	Tremolite	1.62	Idocrase	1.72		
Orthoclase	1.52	Tourmaline	1.63	Magnesite	1.72		
Cancrinite	1.52	Actinolite	1.63	Chloritoid	1.72		
Microcline	1.53	Wollastonite	1.63	Kyanite	1.72		
Albite	1.53	Melilite	1.63	Augite	1.72		
Nepheline	1.54	Aragonite	1.63	Staurolite	1.74		
Oligoclase	1.54	Andalusite	1.64	Chalybite	1.75		
Kaolin	1.54	Apatite	1.64	Garnet 1.76	-1.81		
Cordierite	1.54	Hornblende	1.65	Corundum	1.76		
Quartz	1.55	Fosterite	1.66	Epidote	1.76		
Serpentine	1.55	Sillimanite	1.67	Aegirine	1.78		
Andesine	1.56	Enstatite	1.67	Sphene	1.89		
Labradorite	1.57	Axinite	1.68	Zircon	1.95		
Calcite	1.57	Olivine	1.68	Cassiterite	2.00		
Chlorite	1.58	Diallage	1.68	Perovskite	2.38		
Anorthite	1.58	Diopside	1.68	Anatase	2.53		
Scapolite	1.58	Riebeckite	1.69	Rutile	2.76		

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(b). POLARISER INSERTED BELOW THE STAGE. Note pleochroism. The following minerals when present in rock slices are usually pleochroic :—

Intensely Pleochroic :

Moderately Pleochroic :

Biotite. Hornblende. Riebeckite. Hypersthene. Epidote (varies). Tourmaline. Chloritoid. Kyanite (if coloured). Chlorite. Enstatite (varies). Sphene. Staurolite. Idocrase. Corundum (if coloured). Aegirine.

Slightly Pleochroic :

Titaniferous varieties of Augite.

(c). ANALYSER INSERTED (CROSSED NICOLS). If the mineral remains isotropic on rotation of the microscope stage though 90°, it may be either a glass, a cubic mineral, or a section which has been accidentally cut perpendicular to an optic axis.

If the mineral is birefringent, the birefringence colours should be noted, and also the extinction angle on cleavages or crystal faces (if developed) should be determined.

It will now be convenient to deal with certain minerals which owing to their somewhat similar physical properties, may easily be confused. Conforming to the

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EXAMINATION OF ROCK SLICES

Minerals	arranged	in order of Birefringence.		
UNIAXIAL POSITIVE. UNIAXIAL NEGATIVE.				
Pennine	.003	Idocrase .001		
Quartz	.009	Apatite .004		
Zircon	.062	Nepheline .005		
Cassiterite	.099	Melilite ·005		
Rutile	·287	Corundum .008		
		Tourmaline .017		
		Scapolite -021		
		Cancrinite ·028		
		Anatase .061		
Section 1		Calcite ·172		
	4 18 44	Dolomite ·189		
BIAXIAL POS	SITIVE.	BIAXIAL NEGATIVE.		
Zoisite	.006	Orthoclase .006		
Albite	·008	Microcline ·007		
Enstatite	·009	Andesine .007		
Topaz	•009	Oligoclase .008		
Staurolite	.010	Kaolin ·008		
Chloritoid	.015	Cordierite .008		
Augite	.021	Labradorite .008		
Sillimanite	.021	Axinite .009		
Diallage	.024	Andalusite ·011		
Diopside	•030	Serpentine .011		
Olivine	·036	Anorthite .012		
Sphene	.121	Hypersthene .013		
Brookite	•160	Wollastonite ·014		
Service and the		Kyanite ·016		
		Actinolite ·025		
		Tremolite ·028		
		Muscovite ·038		
		Epidote ·040		
States in the second		Biotite ·044		
30 10 15 1 TO 1		Hornblende 072		
		Aragonite •156		

original order in stage (4) (p. 17), we have firstly the Accessory Minerals.

DISTINCTION BETWEEN IRON ORE AND OTHER OPAQUE MINERALS. With the exception of HAEMATITE and CHROMITE, which are translucent in exceedingly thin sections, all the iron ore minerals are opaque; they are generally distinguished by their colour in reflected light, and in some cases by their crystalline form or characteristic decomposition products. On p. 27 we give a table for the determination of these and certain other opaque minerals.

APATITE may be mistaken for TOPAZ or colourless TOURMALINE, all of which give straight extinction and have somewhat similar refractive indices. Apatite commonly shows an hexagonal outline in cross-section, tourmaline occurs in irregular or acicular forms, while topaz is often granular. Tourmaline has high birefringence (in the coloured varieties this may be masked by the absorption), topaz has a birefringence as high as quartz, while that of apatite is lower. Apatite occurring in fine needles may be confused with RUTILE, which is commonly found included in other minerals (*e.g.* Biotite)^r; but the very high refractive index and double refraction of the rutile serve to distinguish it.

There is a possibility of a confusion between CASSI-TERITE and SPHENE; the latter has a lower refractive index and a higher birefringence than the former, and is often slightly pleochroic, which is not characteristic of cassiterite. Sphene usually occurs in crystals giving a diamond shaped section, cassiterite in tetragonal prisms.

Mennell. Manual of Petrology, 1913, p. 53.

EXAMINATION OF ROCK SLICES

Mineral.	COLOUR IF TRANSLUCENT.	COLOUR IN REFLECTED LIGHT.	COMMON MODE OF OCCURRENCE.
omite ematite ienite	Brown Reddish brown —	Blackish grey Purplish black Black	Octohedra or granular Irregular Irregular or hexagonal flakes
gnetite ites rhotite coxene	1111	Silvery grey Brass yellow Bronze Dull white	Octohedra Cubes Irregular Decomposition product of Ilmenite
phite	1	Dead black	Irregular flakes

Of the Metamorphic minerals CORDIERITE may be easily overlooked owing to its resemblance to QUARTZ in refractive index and birefringence. Cordierite when coloured is pleochroic, but if it is colourless, it can only be distinguished by its biaxial interference figure, using convergent polarised light." A characteristic alteration product is the irregular aggregate of chlorite and muscovite known as ' Pinite.' 2

ANDALUSITE and SILLIMANITE bear considerable. resemblance to one another, but the latter has a higher refractive index and birefringence. If coloured, Andalusite is pleochroic; it commonly occurs in the form "CHIASTOLITE," in which included carbonaceous material tends to group itself in particular directions in the crystal.

WOLLASTONITE³ may be distinguished from the colourless AUGITE with which it occurs in many impure metamorphosed limestones, by its lower refractive index, birefringence and extinction angle.

Some of the alteration products of certain common minerals are liable to cause confusion at times, such as KAOLIN, WHITE MICA, and CALCITE, which occur in the turbid aggregates which discolour many felspars, together with EPIDOTE, ZOISITE and CHLORITE. In these aggregates they can as a rule only be distinguished by chemical means.

Some varieties of CHLORITE, and SERPENTINE, show a similar deep blue birefringence tint, but Chlorite can be distinguished by its greenish colour in ordinary light,

¹ See Miers. *Mineralogy*, 1902. ² Mem. Geol. Sur., 351 and 358, 1907, p. 52. 3 A pyroxene, but more conveniently dealt with as a meta-morphic "accessory."

and by its slight pleochroism with simple polarised light.

EPIDOTE may be mistaken for AUGITE or OLIVINE; it is, however, generally pleochroic, and has a higher refractive index and birefringence, and usually occurs in irregular granular aggregates of a dull greenish-yellow colour.

ZOISITE has a somewhat similar appearance to Epidote, but is paler in colour, and has a low birefringence.

CANCRINITE, a decomposition product of Nepheline, may be mistaken for White Mica, but has a less perfect cleavage and shows birefringence colours of a lower order.

Ferromagnesian Minerals. BIOTITE is often confused with HORNBLENDE, especially the deep brown varieties of the latter (Barkevicite), but its lower refractive index, basal cleavage and straight extinction serve to distinguish it.

Hornblende having an extinction angle normally of about 12° and a cleavage angle of approximately 55°, is readily distinguishable from AUGITE, in which the extinction angle is commonly 40° or more, and the cleavage angle is 87° . Augite *rarely* shows pleochroism to any marked extent.

AEGIRINE may be mistaken for green Hornblende in longitudinal sections, but the marked pleochroism and larger extinction angle of the latter will in most cases be sufficient to distinguish them.

DISTINCTION BETWEEN THE RHOMBIC AND MONO-CLINIC PYROXENES. Rhombic pyroxenes are characterised by straight extinction, and the varieties rich in iron (HYPERSTHENE) show a moderate pleochroism; they also have a lower birefringence than the monoclinic forms.

OLIVINE is readily confused with AUGITE, but the latter's cleavage, slightly lower refractive index and large extinction angle, and the former's straight extinction, irregular fracture and usual alteration to serpentine along the cracks, will serve to distinguish them.

If developed, the crystal shapes of all these ferromagnesian minerals are very characteristic.

The Felspars. For some reason students nearly always appear to have difficulty in determining the nature of the felspars present in a rock slice. In practice, this difficulty is more apparent than real.

MICROCLINE is at once recognisable by its characteristic "cross-hatching"¹ in polarised light.

ORTHOCLASE frequently shows twinning on the Carlsbad law, and this can be recognised by the alternating extinction shown by the two halves of the crystal on rotation. The pure and colourless variety is known as 'ADULARIA,' and that met with in many volcanic rocks is 'SANIDINE,' distinguished from common orthoclase, by its transparency and vitreous lustre.

Orthoclase is frequently turbid owing to decomposition products (see p. 28); it is very commonly intergrown with the alkaline plagioclase felspars, to form 'PERTHITE.'

There remains the isomorphous series of **Plagioclase** Felspars.

These, except in certain metamorphic rocks, invariably show a repeated lamellar twinning, usually on

1 Miers. Mineralogy, 1902, p. 460.

the Albite law, but they may also show twinning on the Pericline law, perpendicular to the Albite lamellae.

This results in a "cross-hatched" appearance distinguishable from that of Microcline by the clearness of the striations, which in the latter mineral are usually "spindle shaped " and somewhat indistinct. In a few cases only the pericline twinning is present (e.g. certain gabbros).

Determination of Plagioclase Felspars.¹ To determine a felspar by means of its extinction angle measured from the albite lamellae, select in the slice, crystals in which the two sets of lamellae give equal extinction angles on opposite sides of a cross wire (fig. 5).



FIG. 5. Plagioclase crystal parallel with cross-wire (B) and in positions of extinction for the two sets of lamellae (A and C), showing equal extinction angles α , β , on each side of the cross-wire.

1 See also Harker. Petrology for Students, 1908, pp. 10-14.

Measure a number of such crystals and select the *maximum* angle obtained. In the case of MICROLITES, it is necessary to measure the extinction angles from their long axes. Obtain a series of several measurements and choose the highest reading determined. Below we give a table of the characteristic extinction angles of the plagioclase felspars for use with the above methods.

Felspar.	Extinction angle measured from the albite lamellae.	EXTINCTION ANGLE MEASURED FROM THE LONG AXES OF MICROLITES.
Albite	6° - 16°	$10^{\circ} - 20^{\circ}$
Oligoclase	0° - 5°	$0^{\circ} - 7^{\circ}$
Oligoclase (basic)	6° - 16°	A STATE OF A STATE OF A
Andesine	$16^{\circ} - 22^{\circ}$	8° - 20°
Labradorite	27° - 45°	$30^{\circ} - 42^{\circ}$
Bytownite	$45^{\circ} - 50^{\circ}$	49° - 56°
Anorthite	50° and over	58° - 64°
		Station Station

It will be seen from the above table that there is an ambiguity between Albite and Andesine in both cases. But examination of the refractive indices of these felspars with reference to quartz or balsam (see p. 33), will do much to decide the point. Further, Albite and Andesine usually occur in rocks of quite different character, and reference to the other constituent minerals will often assist the student in avoiding this difficulty.

In cases where the extinction angles measured on the albite lamellae exceed 40°, there will be an ambiguity between the Labradorite and the more basic

felspars consequent on there being two principal directions of extinction in a crystal section. This, though of theoretical importance, will not cause any difficulty in practice, as we have found that, apart from other considerations, the birefringence and refractive index of Anorthite, combined with its confinement to particular type of rocks in which Labradorite does not *normally* occur, suffice for its determination. On the other hand, if it is desirable to distinguish between these two directions of extinction, use must be made of a quartz wedge or mica plate, for which the student should consult such works as Miers' ' *Mineralogy*,' ^r etc.

It is not safe to assume, in the case of basic felspars, that the lamellar twinning present is necessarily developed on the Albite law, and care should be taken to check this with the cleavage traces in the crystals used for measurement.

In really doubtful cases, the rock should be crushed, and the felspars determined by Schuster's method as described in Chapter IV.

Becke's method for determining relative refractive indices. This will prove of great assistance in dealing with the felspars and many other rock-forming minerals. If the junction of two substances such as quartz and canada balsam be examined under the microscope with ordinary light, it will be seen that in this case it is very indistinct, owing to the similarity of their respective refractive indices. It is found that the greater the difference in refractive index between any two substances so examined, the more distinct the junction becomes.

I Ch. VII.

D

If the objective be slightly raised so as to throw the junction out of focus, it will be observed that a *white line* passes from the substance having the lower refractive index into that having the higher. If the objective be lowered, the reverse takes place, and the greater the difference between the refractive indices the more marked is this effect.

For example, it will be seen from the table (p. 23) that albite and oligoclase have lower refractive indices than quartz (or balsam), and consequently, any felspar having a higher refractive index than these latter, must be andesine, or a more basic variety.

The student will learn by experience that, in general, acid rocks are characterised by acid felspars and basic rocks by basic felspars, and that a rapid examination of the refractive index of a felspar will go a long way to determine its nature, without recourse to more elaborate optical methods.

Felspathoids. These contain a lower percentage of silica than the felspars, and are not formed from magmas in which there is sufficient silica to produce the latter. Consequently they need not be looked for in any rock containing pyrogenetic quartz.

NEPHELINE (a dull variety has been distinguished as ELÆOLITE) is sometimes confused with Orthoclase, but has a refractive index as high as oligoclase and can be distinguished in addition, by its lower birefringence, absence of twinning, and crystal shape if developed. In many rocks, however, nepheline is interstitial, and can often only be determined by microchemical methods (see p. 47).

LEUCITE, usually in icosetetrahedra, often shows

weak anomalous birefringence, and zoning by inclusions, and may be distinguished from nepheline by the latter's higher refractive index and crystalline form, if developed.

MELLITE often shows lath-shaped sections resembling felspars; it has, however, a higher refractive index, and much weaker birefringence. In addition it may show striations perpendicular to the long axis ("*peg structure*")^T and is commonly discoloured. The above characters also serve to distinguish it from nepheline, which it resembles in birefringence.

Of the remaining felspathoids, NOSEAN is easily identified by its characteristic zoning, HAUYNE and SODALITE are often bluish in thin section, the colour being usually irregularly distributed in the case of Hauyne, and very pale in that of Sodalite. For microchemical methods applicable to all the above felspathoids, see Chapter III.

Quartz. In many metamorphic rocks, it is hard to differentiate between quartz and clear recrystallized felspar grains. In such cases the characteristic twinning of plagioclase felspars is frequently not developed, and staining methods may be resorted to, if examination of the refractive indices gives no conclusive result. It may be noted that quartz never occurs with felspathoids and seldom with olivine, and also that the mineral never shows twinning in rock slices.

5. Having determined the mineral constitution of the rock, the student will be in a position to decide whether it belongs to the *Alkaline* or *Calcic* group, or occupies a position *Intermediate* between these two

1 Hatch. Petrology, 1909, p. 102.

(Monzonite types).¹ In the Alkaline group we find that the quartz is generally confined to the acid rocks, that alkali felspars occur more or less throughout the series, while the felspathoids are common in the more basic varieties, and may entirely take the place of felspar. The ferromagnesian minerals are often soda-bearing, *e.g.* Arfvedsonite, Riebeckite, Aegirine, etc.

In the Calcic group, quartz ranges from acid to quite basic rocks, felspathoids are absent, and alkali felspars are generally confined to acid members. Ferromagnesian minerals are typically non-alkaline varieties.

In the monzonite magmas, alkaline and calcic felspars occur in approximately equal amounts.

6. Rock Structures and their significance. In the examination of a rock slice, the student should bear in mind that one of the objects of Petrology is to assist in unravelling the geological history of the earth, and that his object in examining a slice should not be merely to find out whether certain minerals occur in his particular rock, but to interpret as far as the limitations of microscopical methods allow, the history of that rock, both during and after consolidation. In this connection three points are of great importance :—

- (r) The particular association of minerals present in a rock is only one of the possible expressions of its chemical composition.
- (2) This particular mineral association will vary according to the physical conditions (e.g. pressure) under which consolidation has taken place.

^I Harker. Petrology for Students, 1908, pp. 50, 56 and 57; also Hatch, Petrology, pp. 183 and 198. (3) The original character of the minerals present may be partially or entirely changed, during the rock's subsequent history.

From the above, it will be seen that the structure of a rock is, at least, equal in importance to its mineralogical composition.

It is not our province here to describe the characters of various rock structures, as these are dealt with very fully in the standard works on Petrology.¹ It will suffice to say that in general the student will find that certain rocks are characterised by particular types of structure.

7. Inference as to probable nature of the rock. From an intelligent use of all the foregoing considerations the student should now be in a position to determine, within limits, the nature of the rock that he is dealing with.

It must be borne in mind that rock types grade imperceptibly into one another, and that occasionally rocks will be found that are intermediate in this respect. On this account the classification of rocks as at present employed leaves much to be desired,² and one gets cases where existing rock names do not suit the specimens under consideration.³

¹ Harker. Petrology for Students, 1908; also Teall. British Petrography, 1888. Hatch. Petrology, 1909, and Iddings. Igneous Rocks, Vols. 1 and 2, 1909.

² Harker. Nat. Hist. Ig. Rocks, Ch. 15; also Petrology for Students, 1908, p. 20; also Iddings. Igneous Rocks, Vol. 1, p. 334 et seq.; and Lake and Rastall. Text book of Geology, 1910, p. 235 et seq.; and Mennell. Manual of Petrology, 1913, p. 81 et seq.

3 McRobert. Q.J.G.S., Vol. 70, 1914, p. 315.

Method of Working if the rock is Sedimentary. If the rock is of sedimentary origin, it will be necessary to decide whether it has been formed :—

(I) By denudation of land masses.

- (2) By organic agencies.
- (3) From the products of volcanic activity, or by any combination of (I), (2) and (3).

We will consider these in order :----

I. Sedimentary rocks formed by the denudation of land masses, can be divided into two main groups (a) ARENACEOUS, and (b) ARGILLACEOUS.

(a) The chief points to determine in Arenaceous rocks are the minerals which comprise the grains, the cementing material and the structure. Careful study of the grains may indicate sources of origin; it must be remembered, however, that in general only the more stable minerals of igneous and metamorphic origin will be found (see p. 53). The cementing material may be ferruginous, calcareous, silicious or argillaceous. Under the head of "structure," we include such points as coarseness or fineness of the grains, as well as their shapes and mutual relations.

(b) Argillaceous rocks. This group includes the finer sediments such as clays, mudstones, shales and slates. Rock sections of such sediments as shales and slates often present considerable difficulty in complete determination, owing to their general fine texture and the prevalence of decomposition products such as sericitic mica, chlorite, etc. They frequently show a fissile structure due to a parallelism of their constituents; this may be the outcome of original bedding, or of the effects of compression, *i.e.* cleavage in slates.

The high power objective of the microscope will prove of great use in determining these rocks.

2. Rocks due mainly to organic agencies. These may be either (a) CALCAREOUS, (b) SILICIOUS, or (c) CARBONACEOUS.

(a) Calcareous. These are rarely of detrital origin, but are commonly composed of comminuted fragments of the hard parts of such organisms as *mollusca*, *echinodermata*, *actinozoa*, or *foraminfera*, set in a matrix of fine calcareous mud.

Organic structures originally composed of Aragonite (e.g. some *mollusca*), are usually replaced by a granular aggregate of calcite.

The calcareous matrix is often recrystallised by simple pressure, but this does not necessarily imply any great degree of metamorphism.

Special types of concretionary structures such as *oolitic* or *pisolitic* should be noted.

Another important characteristic of many limestones is the replacement of all or part of the calcite by DOLO-MITE,¹ and more rarely by CHALYBITE (ironstones) or SILICA (cherts). Dolomite may usually be distinguished from calcite by its tendency to crystal habit (rhombohedra), and absence of lamellar twinning, but in other cases chemical tests are the only sure means of discrimination (see p. 49).

(b) Silicious. The silicification of limestones to form chert may be due either to infiltration or to the presence of such silicious organisms as Sponges² or

¹ Skeats. Q.J.G.S., LXI, 1905, pp. 97-138 and references there cited. ² Hinde. Phil. Trans. Roy. Soc., 1885. Radiolaria, the remains of which should be looked for in such cases.¹

(c) Carbonaceous. Specially prepared thin sections of coal will show traces of vegetable structures such as stems, fructification, etc., for the study of which we must refer the student to standard Palaeobotanical works.²

3. **Pyroclastic Rocks.** A microscopical study of pyroclastic rocks may throw much light on the nature of the volcanic outburst which gave rise to them.

They are, in general, made up of shattered fragments of pre-existing types of rock, together with varying amounts of usually decomposed glass and other fragments of volcanic origin.

It should be the student's aim to determine the nature of the fragments and of the volcanic matrix which encloses them.

In other instances the rock will be made up wholly of volcanic material. In such cases, this material should be treated as described when dealing with volcanic rocks (p. 19), and according to its nature may be designated as rhyolitic, trachytic, and esitic, or basaltic tuff.

Silicification³ may be looked for in rhyolitic tuffs.

Many tuffs were originally deposited under the sea, and the finer varieties often show a conspicuous bedding; secondary cleavage is quite commonly observed in those varieties which have been subjected to pressure. Where such a cleavage is well marked, secondary minerals,

¹ Howard Fox. Radiolarian Cherts of Cornwall, Trans. Roy. Geol. Soc. Corn., Vol. 12.

² Scott. Studies in Fossil Botany, 1900.

3 Harker and Marr. Q.J.G.S., 1891, p. 303 et seq.

which have been formed as a result of these stresses, should be looked for.

Hybrid Rocks.^I Hybrid rocks may be expected to show many of the characters of metamorphic rocks, but their heterogenous origin will make itself apparent in unusual structures and peculiar mineral associations. These rocks, from the very nature of their composition, will not allow of the methodical microscopic treatment which we have outlined for normal rocks. In general, a very complete knowledge is required of the circumstances of any particular case, both as regards the types of rocks which have contributed to the mixture, and the latter's mode of occurrence in the field.

Consequently a microscopical examination of such rocks, unless supplemented by this knowledge, must necessarily be somewhat arbitrary.

In the absence of circumstantial evidence, the student is advised to make a very thorough determination of the minerals present, and, if possible, to separate those of the original rock from those of the one which has incorporated it.

In many cases superimposed structures will render this impossible, and it is then that microscopical examination alone breaks down. But if the field evidence is available, and the types of rock which have taken part in the mixture can be studied both macro- and microscopically, the student should be in a position to formulate an opinion as to how far intermixing has proceeded in the particular case he is considering.

¹ Harker. Nat. Hist. Ig. Rocks, 1909, ch. 14.; also Mem. Geol. Sur., Tertiary Ig. Rocks Skye, 1904, Ch. 11 and p. 231; and Mem. Geol. Sur., Sheet 60, Scotland, Ch. 9; and Mennell. Manual of Petrology, 1913, p. 209.

Closely allied to this question of Hybridism is the presence in numerous igneous rocks of inclusions (*Xenoliths*)^{\mathbf{r}}; these may be *accidental* and derived from extraneous sources, in which case they differ from the hybrid rocks only in degree, or they may be closely bound up with the formation of the rock which contains them (*cognate xenoliths*).

In the first case a study of such inclusions may be of assistance in considering more advanced stages of admixture, while the occurrence of cognate xenoliths may throw light on the affinities of the rock in which they are found. Unlike the true hybrids, such cognate xenoliths are in nearly all cases amenable to the treatment which we have suggested for normal igneous rocks.

^I Lacroix. Les enclaves des roches volcaniques, 1893; and Harker. Nat. Hist. Ig. Rocks, p. 346 et seq.

CHAPTER III.

MICROCHEMICAL METHODS (STAINING).

Uses of Staining—Apparatus—Preparation of Slide—General Method of Procedure—Stains employed—Special reactions applicable to certain Minerals.

In certain branches of organic microscopy, staining methods, which depend on microchemical reactions, are of great importance, and somewhat analogous methods have been extended to petrological work, with many satisfactory results.

Such methods are used as special or confirmatory tests for certain rock-forming minerals, which are difficult of determination in rock slices by ordinary microscopical means, either on account of their mode of occurrence or resemblance to other minerals. In the following paragraphs we propose to deal with the simplest methods of staining which can be rapidly applied to certain minerals in a rock slice.

In most cases these microchemical tests are modifications of reactions employed when dealing with minerals in bulk, the mode of application being changed to suit the conditions of microscopical examination. It should be noted that the study of these reactions constitutes in itself a wide field for research, and we can only outline a few of the more useful cases.^{*}

¹ See H. Behrens. A Manual of Microchemical Analysis, London, 1894. It will be convenient to deal with the matter under three heads :---

- 1. Apparatus and preparation of slice for chemical tests.
- 2. General method of procedure with regard to reagents employed.
- 3. Special reactions applicable to certain minerals.

I. Beyond the necessary chemicals involved, little extra apparatus is required for these microchemical tests, though a capillary tube or pipette for transference of the reagent to the mineral under examination, or a glass rod for applying it drop by drop, together with a diamond cutter, will be found useful accessories.

The ordinary petrological microscope will be quite efficient for our purpose, though a cheaper pattern or the simple type recommended for use in the manufacture of rock slices (see p. 2), will be an advantage; continued exposure to the presence of mineral acids, even in minute quantities, will not improve the condition of a more expensive instrument.

Slight modifications in the use of canada balsam may be necessary, such as its employment dissolved in carbon di-sulphide or ether, which on evaporation, leave it hard.

If the staining of a particular mineral is to be a permanent feature of the slide, great care must be exercised in carrying out the operations involved.

In the case of slices already finished, it will be first of all necessary to uncover a portion of the rock. This may be done in several ways. Either the slide may be warmed until the balsam is soft, when the cover glass may be partially removed, or a portion of the cover glass may be perforated with a diamond cutter, over that part

of the slice to be experimented upon. A neater method than the latter is to coat the cover glass with fluid beeswax, and when just cool, to scratch a circle through the latter with a sharp point. Apply a small quantity of hydrofluoric acid to this, which will speedily etch through the glass.

In the case of slices in course of preparation, when they have reached the end of the second grinding (see p. 10), they may be covered with a thick layer of balsam dissolved in ether, and a hole made over the part of the slice to be treated. The mounting of the cover glass should be delayed until the staining operations have been completed.

If the subsequent test demands the use of hydrofluoric acid, a glass cover slip for the protection of that part of the slice not to be experimented on, will obviously be useless. In such a case the method of covering the whole slice with a layer of balsam, as outlined above, should be employed.

The part of the rock exposed, is cleaned with methylated spirit or benzene, to free it from balsam.

In the cases where balsam is employed to protect portions of the slice, care must obviously be exercised in the use of benzene or other solvents for cleaning purposes. In this connection a camel-hair brush may be used with advantage.

2. Before minerals can be stained, it is usually necessary to gelatinise them with acids capable of effecting their solution. The etching must be purely superficial and the reaction must not be allowed to go on for longer than absolutely necessary. The success of the subsequent staining operations depends on the complete removal of the acid from the gelatinous film formed. This is usually achieved by washing with a solution of ammonia, care being taken not to wash away the gelatinous material. In many cases the ammonia not only effects the removal of the acid by neutralisation, but materially aids the staining.

When washed and dried, the section is placed in a dish containing the necessary stain.

The stains generally employed are Fuchsine (Magenta), Malachite green, Congo red, Aniline blue and Methylene blue.^I Fuchsine has the disadvantage of not being permanent in the presence of canada balsam, and, moreover, it fades on prolonged exposure to light. Malachite green is a more conspicuous colour, and is permanent; the other dyes mentioned are also quite satisfactory in this respect.

When the staining is complete, the slide is washed; it will be found on examination through the microscope, that all minerals which have gelatinised with the particular reagent employed, will appear coloured. If the staining has not been carried far enough, the process can be repeated.

3. We will now describe the application of the above methods to certain minerals.

The following minerals gelatinise with Hydrochloric acid and take the stain :--

Lime-scapolite. Nepheline. Sodalite. Melilite. Hauyne. Nosean. Lazurite. Anorthite. Olivine. Chlorite. Serpentine. Zeolites.

I See Appendix.

Distinctive tests for some of the above minerals. These should be observed microscopically throughout the entire process.

LIME SCAPOLITE and SODALITE when treated with a solution of silver nitrate in hydrofluoric acid, form a gelatinous film which turns brown when acted on by a photographic developer such as pyro-soda.^I

SODALITE dissolves, without gelatinising, in nitric acid, and crystals of sodium chloride are formed on evaporation of the solution.

NEPHELINE will take the stain, using hydrochloric acid, but its presence is only certain if the stained mineral shows characteristic crystal outline, or by an elimination of other possibilities.

MELILITE, if gelatinised with hydrochloric acid, containing drops of sulphuric acid, will show crystals of gypsum.

HAUYNE gelatinises with hydrochloric acid forming crystals of gypsum.

NOSEAN generally occurs in rocks in association with sodalite. On treatment with 1:3 acetic acid, containing a little barium chloride in solution, nosean will be clouded with a precipitate of barum sulphate, and sodalite, if present, will remain clear.

LAZURITE. If this is treated with a solution of silver nitrate in hydrofluoric acid, a gelatinous film is formed, which turns black owing to deposition of sulphide of silver.²

¹ A few drops of the normal strength used in photographic work will suffice. See Bothamley. *Manual of Photography* (Ilford, London), p. 67.

² For this and the above Lime-Scapolite reaction, see also Weinschenk (Clark), *Petrographic Methods*, 1912, p. 174.

OLIVINE, particularly the varieties rich in iron, gelatinises with hydrochloric and sulphuric acids, slowly with cold, and rapidly with hot acids.

CHLORITE, SERPENTINE, ZEOLITES, etc., are often the cause of the cloudy aggregates of decomposition products which discolour many rock-forming minerals. These can, in general, be completely removed by prolonged digestion in hydrochloric acid.

General methods involving the use of hydrofluoric acid. The following is useful as a distinctive test for FELSPAR when present with QUARTZ in fine recrystallised aggregates, as is often the case in metamorphic rocks.^r

The section exposed is treated for one minute with hydrofluoric acid, when it is found that felspar is superficially altered, becoming cloudy, and quartz, though acted upon, remains clear. It is possible to accentuate this difference by staining. When the etching is complete, the slide is washed and dried, and treated with aniline blue for ten minutes. It is then washed with water, after which it is treated with ethyl alcohol of increasing strength to remove water from the gelatinous material. After a final immersion in absolute alcohol, the slice should be moistened with turpentine or benzene, and finally covered with canada balsam dissolved in benzene (see p. 55). A cover glass may then be placed on the top of the balsam and gently pressed down, and the slice should then be put away to dry.

This somewhat complicated operation is rendered

¹ T. Harada. Das Luganer Eruptivgebiet, Neues Jahrb., B.B., II. (1883), p. 14; also F. Becke. Unterscheidung von Quarz und Feldspath in Dünnschliffen mittelst Farbung, Tschermak's Min., u. Petr., Mitt., Vienna, X (1889), p. 90.

necessary by the desirability of not disturbing the gelatinous material by treatment with liquids of greatly different density.

It is possible by these methods to distinguish the felspars by the depth of colour which they exhibit after staining. But a great deal depends on the skill of the operator in timing the reaction. In general, the lime felspars show a deep blue, the soda varieties being paler in colour and orthoclase only very slightly affected.

APATITE. When this is treated with hot concentrated nitric acid, followed by a solution of ammonium molybdate, the characteristic ammonium phosphomolybdate colour (canary yellow) is obtained.

CARBONATES. Carbonates are often hard to distinguish by ordinary microscopical examination, and in these cases microchemical tests are of extreme value. We will deal first with the separation of CALCITE and DOLOMITE.

Calcite dissolves in cold hydrochloric acid, dolomite being unaffected. The latter is however soluble in hot acid. They may be further separated by *Linck's*¹ method:—20 c.c. of ammonium phosphate are dissolved in 30 c.c. of dilute acetic acid. Treatment with this solution will completely dissolve calcite; dolomite is practically unaffected, though it may be covered with crystal aggregates of magnesium ammonium phosphate.

Lemberg's method 2:- To 30 parts of water, 2 parts of

¹G. Linke. Abh. zur. geol. Spezialkarte von Elsass-Lothringen, III., 1884, 17. ²J. Lemberg. Zeitschr d. deutsch geol. Besell, XL., 1888, 357-359.

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dry aluminium chloride and 3 parts of log wood (haematoxylin) are added. On boiling, a deep violet solution is formed, which is filtered off and allowed to cool. The slice is treated with this stain for about five minutes, and then carefully washed with water. The calcite will be found to be coloured violet, while dolomite remains unchanged.

Heeger's method ¹:—Both these last two methods of separation fail when the carbonates occur as fine interstitial matter. In such a case the following method may be employed with advantage.

It consists of treating the rock slice with a few c.c. of decinormal hydrochloric acid, containing in solution a little potassium ferricyanide. Calcite will give violent effervescence, and on washing, after a few seconds, will be found to be coloured blue. This is due to the fact that nearly all calcite occurring in this manner contains more or less iron. Dolomite is practically unaffected.

SEPARATION OF CALCITE AND ARAGONITE. The application of a cold solution of ferrous ammonium sulphate to calcite and aragonite causes the latter to turn olive green in colour, whilst the former is unaffected.

These minerals may also be separated by *Panebianco's*² method :—Powdered calcite heated with a pure dilute cobalt nitrate solution, turns blue after one minute's boiling, whilst aragonite turns lilac. Continued heating determines the lavender blue colouration of calcite, and the violet colouration of aragonite.

¹ W. Heeger. Centralbl. f. Min., etc., Stuttgart, 1913, 44-51. ² G. Panebianco. Revista di min. crist. Ital. XXVIII. (1902), 5. These last two methods find their principal application in the determination of the composition of organic structures.

Treatment of carbonaceous material. Material containing carbon disseminated throughout its mass can always be clarified by roasting on platinum foil. When it is desired to examine organic structures, as for example. in coal, this method will obviously be inapplicable. In such a case prolonged treatment with chlorous acid will remove the carbonaceous material.

CHAPTER IV.

MOUNTING OF SANDS AND CRUSHED ROCK

MATERIAL.

Method of procedure—Heavy liquids employed—Mounting— Crushed rock material—Schuster's method for the determination of Felspars.

In dealing with loose detrital rocks such as sands, loam, etc., methods of preparation will be required which differ considerably from those employed with rock sections. In general the following mode of procedure will be sufficient for the student's requirements.^{*}

I. The general composition and condition of the material should be ascertained with a lens.

2. The material should now be powdered, if necessary, and passed through a fine sieve, the coarse material being rejected. (A 30-mesh sieve is a convenient grade, though in some cases a 60-mesh may be used with advantage.)

3. Removal of muddy material :---This can be best effected by repeated washing in water; the residue

¹ See also Hatch and Rastall. *Petrology of the Sedimentary Rocks*, 1913, App. by T. Crook ; also Boswell, *Geol. Mag.*, March, 1916 ; and Rastall. *Camb. Phil. Soc.*, March, 1913. should be dried and examined with a lens. In most cases the bulk of this residue will consist of quartz grains with a varying quantity of felspar and a small proportion of the heavier minerals.

4. The next operation is the concentration of the heavier minerals. This can be effected by *panning* and by the use of 'heavy' liquids. If a considerable quantity of material is to be concentrated, it will be convenient to remove most of the lighter quartz and felspar by panning. This is carried out by placing a quantity of the sample in a flat-bottomed dish. It is then covered with water, and by giving the dish a combined to and fro and circular motion, the majority of the heavier grains can be concentrated in the lower, and most of the quartz and felspar in the higher layers. The latter may be washed away and the process repeated until most of the lighter material has been disposed of. If necessary, this latter may be treated again for recovery of the small proportion of heavy material which passed off with it. The concentrate should now be dried and examined.

In many cases, the characters of the grains may be obscured by cementing material. Treatment with dilute hydrochloric acid will remove calcareous material, but ferruginous material will have to be boiled in strong hydrochloric acid. The concentrate should now be dried and treated with a 'heavy' liquid. (In general, the most useful is Bromoform (specific gravity 2.9). If this is not available, Mercury Potassium Iodide (*Thoulet's* solution) is the most suitable of the many solutions which have been used. It has the disadvantage of being highly corrosive, and is also more viscous than Bromoform.^r) For this operation, an ordinary glass funnel, fitted with a stopcock or a piece of rubber tubing and a pinchcock, should be used.

The 'heavy' liquid is first poured into the funnel, and then a quantity of the dried concentrate is mixed with it and well stirred.

Any minerals of greater specific gravity than 2.9 in the case of bromoform, or 3.3 in that of Thoulet's solution, will sink to the bottom, and can be drawn off.

The concentrate from this operation may be conveniently collected in a second funnel lined with a filter paper and fitted into the neck of a bottle (thus avoiding waste of the heavy liquid).

The heavy minerals collected in this manner may now be washed in a watch glass or porcelain dish (using benzene in the case of bromoform, and water in the case of Thoulet's solution), and afterwards dried.

A magnet may be used to extract those which are magnetic, and further separation can be effected, if desired, by the use of liquids of higher specific gravity.² If there is only a small quantity of material to be treated, the panning may be dispensed with.

The heavy minerals concentrated as above may be conveniently mounted in canada balsam. The 'hot balsam method,' as used for rock slices, may be employed, but with this method it is very difficult to avoid air bubbles.

⁴ If necessary these liquids can be diluted, the bromoform with benzene, and *Thoulet's* solution with water, giving liquids of lower specific gravity for use with lighter minerals.

² Hatch and Rastall. Petrology of Sedimentary Rocks, 1913, p. 360.

MOUNTING OF SANDS AND CRUSHED ROCK 55

The following mode of procedure is more satisfactory in this respect, but takes longer time :—Some of the material should be placed on a cover glass and moistened with turpentine. It should then be covered with a few drops of a solution of balsam in benzene (as ordinarily sold for microscopic work) and gently warmed on the hot plate until nearly dry. It may now be covered with fresh balsam solution, and pressed down on to a glass slide. This should now be put away to dry. Surplus balsam can be cleaned off with benzene or methylated spirits.

This method of dealing with loose sediments may also be applied with advantage to the examination of ordinary rocks. By this means it is often possible to isolate accessory minerals which escape detection in a rock slice, owing to their absence in the very small bulk of the rock which such a slice occupies. It is also particularly useful for a rapid determination of the mineral composition of a rock, where the student does not want to go to the trouble of making a thin slice. The latter will, of course, be necessary for a full determination and for a study of the structure.

The mode of procedure is as follows :---

I. A sample of the rock is broken up in an iron mortar, and the powder obtained is passed through a fine sieve; generally a 60-mesh, though for fine-grained rocks a 90-mesh sieve may be required to ensure that each grain contains one mineral only.

2. The fine dust is now removed by washing in water.

3. Some of the residue may be dried and mounted

without further treatment. For a rapid determination of the constituents, this will suffice; but for a fuller examination of the accessories, the heavy minerals will have to be concentrated with bromoform or some other ' heavy ' liquid, and the method of treatment is similar to that employed with sands.

The methods employed in the determination of the minerals present in such microscopical preparations will be essentially the same as those outlined for use with rock slices (see p. 22 et seq.). But the use of convergent light, often difficult to employ satisfactorily with rock slices, may be of considerable assistance in dealing with the minerals present in fragmental material such as we are considering here.

We assume that the microscope which the student possesses is capable of being used for this purpose, *i.e.* that a substage condenser and a *Bertrand* lens can be introduced in addition to the usual petrological accessories. For a detailed description of the use of convergent light ('Interference figures,' etc.) the student is referred to standard text-books on Mineralogy.^r

From the examination in both parallel and convergent light, information as to the isotropic or anisotropic, uniaxial or biaxial nature of the minerals present, should be obtained, together with other physical characters sufficient for identification in all ordinary cases.

¹ Miers. Mineralogy, 1902, Ch. 7; also Mennell. Manual of Petrology, 1913, pp. 22-27; also Introduction to the study of Minerals, British Museum (Natural History) guide, 1914, pp. 43-47; and Lèvy et Lacroix. Les mineraux des roches, 1888, Ch. 5. In connection with this examination of fragmental

material, we may conveniently give here Dr. Schuster's method for the determination of felspars in cleavage flakes." It will facilitate the explanation of this method if we consider a simple felspar crystal (fig. 6). In this we have perfect cleavages parallel to P and M, and a less perfect cleavage parallel to T. Schuster uses the signs + and - for signifying the extinction directions on the two cleavages P and M.



FIG. 6.

If the rock, containing the felspar to be determined, is crushed, and some of the grains are mounted as described above, it will usually be found that the cleavage flakes parallel to M are in greater abundance than those parallel to P.

In general an M flake will be in the form of a parallelogram, bounded by the cleavages parallel to P and T respectively. From the figure it will be seen that when the cleavage fragment has to be rotated in the direction of the obtuse angle of the parallelogram in order to

^I Schuster. K Akad de Wiss, I abth Juli, 1879.

obtain extinction, the sign is +, and when in the direction of the acute angle, the sign is -.

In nearly all cases, P flakes give smaller extinction angles than M flakes, and are determined in the same way.

In the following table the Albite and Anorthite molecules are represented by Ab and An respectively^r:—

Felspar.	Р.	М.
Ab	+ 4° 30'	+ 19° 0'
Ab ₁₂ An ₁	+ 3° 38'	+ 15° 35'
Ab ₈ An ₁	+ 3° 12'	+ 13° 49'
Ab ₆ An ₁	+ 2° 45'	+ 11° 59'
Ab ₅ An ₁	+ 2° 25'	+ 10° 34'
Ab ₄ An ₁	+ 1° 55'	+ 8° 17'
Ab ₃ An ₁	+ 1° 04'	+ 4° 36'
Ab ₂ An ₁	- 0° 35'	- 2° 15′
Ab ₃ An ₂	- 2° 12'	- 7° 58′
Ab ₄ An ₃	- 2° 58'	- 10° 26'
Ab ₁ An ₁	- 5° 10'	- 16° 0'
Ab ₅ An ₆	- 6° 50'	- 19° 12'
Ab ₃ An ₄	- 7° 35'	- 20° 52'
Ab ₁ An ₂	- 12° 28'	- 26° 0'
Ab ₁ An ₃	- 17° 40'	- 29° 28'
Ab ₁ An ₄ .	- 21° 05'	- 31° 10′
Ab ₁ An ₅	- 27° 37'	- 32° 10′
Ab ₁ An ₆	- 27° 33'	- 33° 29'
Ab ₁ An ₈	- 28° 04'	- 33° 40'
Ab ₁ An ₁₂	- 30° 23'	- 34° 19'
An	- 37° 0'	- 36° 0'
Microcline	+ 15° 30'	+ 5° 0'
Orthoclase	0°	$+ 5^{\circ} to + 7^{\circ}$
Soda orthoclase	0°	$+ 9^{\circ} to + 12^{\circ}$
Anorthoclase	+1° 30' to +5° 45'	$+6^{\circ} to + 9^{\circ} 48'$

¹ Rosenbusch. *Mikroskopische Physiographie*, 1892, Vol. I., p. 664.

If a number of flakes of any felspar are examined, two series of approximately similar extinction angles will be obtained, corresponding to P and M flakes respectively, and by the aid of the table the composition of the felspar may be determined with a considerable degree of accuracy.

In the following table we give the six principal plagioclase felspars with their composition :---

Felspar.	Composition.	Silica percentage.
Albite	Ab	68.7%
Oligoclase	Ab to Ab ₃ An ₁	62.0%
Andesine	Ab ₃ An ₁ to An ₁ An ₁	55.6%
Labradorite	Ab ₁ An ₁ to An ₁ An ₃	49.3%
Bytownite	Ab ₁ An ₃ to An	46.6%
Anorthite	An	43.2%
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APPENDIX.

PREPARATION OF STAINS.

It may be convenient to give methods of preparation of stains mentioned in Chapter III. These are mostly aniline dyes.

Preparation of Fuchsine.

Required $\begin{cases} 2 \text{ grs. Aniline.} \\ 2 \text{ grs. o-toluidine.} \\ 2 \text{ grs. p-toluidine.} \\ 12 \text{ grs. Arsenic acid.} \end{cases}$

Method :--Mix the aniline and two toluidines together and boil with the acid in a metal bath for about an hour. The resulting product is soluble in water, and is Fuchsine (Rosaniline or Magenta).

Preparation of Malachite green.

Required Benzaldehyde. Dimethyl aniline. Zinc chloride (solid). Lead peroxide. Hydrochloric acid.

Method :— The benzaldehyde and dimethyl aniline are mixed together in the ratio 1:2 respectively, and heated with excess of solid zinc chloride. The insoluble product is a colourless, crystalline substance (Leucobase of Malachite green). This product is dissolved in dilute hydrochloric acid to which a little peroxide of lead
has been added. It is well shaken, and poured into water, when the dye is obtained.

Preparation of Congo Red. This is a somewhat complicated process and involves the use of Benzidine dves.

Congo red is prepared by the action of sodium salicylate on diphenyltetrazonium chloride.¹

Preparation of Aniline Blue.

(Magenta.

Ammonia and Carbon di-oxide.

Required { Aniline.

Glacial Acetic Acid.

Methylated Spirit.

Method :--- Dissolve some solid magenta in water and pass carbon di-oxide through the solution. Add ammonia while the latter action is in progress. A white precipitate of Rosaniline base will be obtained. One gram of the base is mixed with 5 grs. of Aniline and a little glacial acetic acid. On heating for a quarter of an hour, a blue precipitate is formed. Extract with methylated spirit. This is the 'Aniline Blue' stain.

Preparation of Methylene Blue.

Required Nitrosodimethyl aniline. Ammonium sulphide. Hydrochloric acid. Ferric chloride.

[If Nitrosodimethyl aniline is not available, it should be prepared as follows :- A little dimethyl aniline is dissolved in dilute hydrochloric acid and a clear liquid obtained by violent shaking. When cool, a few crystals

Remsen. Organic Chemistry, 1903, p. 377.

of sodium nitrite are added. Yellowish crystals form, and some of the liquid should then be hydrolised with caustic soda, when a green precipitate is formed, which is Nitrosodimethyl-Aniline base.]

Method :— The nitrosodimethyl aniline is warmed with ammonium sulphide until the former dissolves. When cool, the solution is acidified with hydrochloric acid. Ferric chloride is then added in excess, and a blue colour results which is 'Methylene Blue.'

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