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Notes on the Micro-chemical Analysis of Rock-making Minerals.

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Part 1.—General Remarks.

ALL petrologists who have worked much with the microscope must have felt the want of a system of micro-chemical analysis at once simple and reliable. Having devoted some time to the study of micro-chemical reactions, I think it may save the time and labour of students who propose to take up the subject if I give them the result of the experience I have gained up to date.

In order to shorten this paper as far as possible, I have limited myself to the bases that commonly enter into the composition of rock-making minerals, and have excluded those which relate to ores.

Micro-chemical analysis is not only an essential adjunct to micro-petrological research, but it is capable of rendering some aid to the ordinary chemist. Not only does it afford a ready means of testing precipitates, but it is even more essential as a method for testing the purity of reagents.

Until I took up this inquiry I had no idea of the *extent* to which the reagents supplied by some good firms contain chemical impurities. I was astonished to find, for instance, that my bichloride of platinum invariably showed the presence of potash in every mineral I tested with it; that my fluosilicic acid always indicated the existence of an appreciable amount of soda; and my aluminium sulphate, delusively labelled "purified," contained so much potash that it crystallised on my glass slides as an alum. I mention these cases as mere illustrations. I have succeeded in obtaining some chemicals sufficiently pure to work with; but in other cases I have failed to do so. Pure uranium acetate, for instance, is said to belong to the rhombic system and to crystallise in long prisms; but though I have had uranium acetate specially made for me, all that I have obtained crystallises in the cubic system in forms identical with those of the double salt of acetate of sodium and uranium. As a test for soda, therefore, by the microscopic method, it is worse than useless.

In micro-chemical analysis I have found the following system the most convenient to pursue. I convert the bases present in the minerals under examination into sulphates,[1] and, with one or two exceptions to be noted further on, work with these sulphates in aqueous solution without the aid of heat. There is considerable advantage, it seems to me, in working with salts in the form of sulphates; most of them are soluble in water; they are all insoluble in absolute alcohol, and Canada balsam diluted with benzole or chloroform does not corrode them. Scarcely any of those entered in Part II. are deliquescent; whilst the alums are the only ones that crystallise in the cubic system.

All the sulphates can be mounted in Canada balsam with the greatest ease and preserved for any number of years for future study. The microscopist therefore can dispense with the aid of figured illustrations; he can form, what is far better, an imperishable collection of typical specimens of the salts themselves to be always at hand for ready reference.

Other salts besides the sulphates can be mounted in Canada balsam,

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[1] Silicates, insoluble in sulphuric but soluble in hydrofluoric acid, I convert into sulphates by dissolving them in a state of fine powder in hydrofluoric acid in a platinum vessel with the aid of heat. I then add sufficient sulphuric acid to convert the bases into sulphates. On evaporation to dryness the silica is driven off in the form of fluosilicic acid and the sulphates are left behind. These can now be dissolved in water, or, in the case of barium or strontium, in hot sulphuric acid. A similar process can be used to test the purity of the hydrofluoric acid employed, viz. add concentrated sulphuric acid to the *HF* aq. and evaporate to dryness. If the HF contains bases in any appreciable quantity they will be left behind as sulphates and may be detected under the microscope. In the case of minerals not soluble in hydrofluoric acid they must be decomposed by fusion with carbonate of soda or some other flux. See Zircon, Part III.

but the mounting and preservation of some of them are extremely difficult. Many of the chlorides, for instance, deliquesce, rendering the use of heat in some form necessary; many are corroded by Canada balsam; and many crystallise in the cubic system, rendering their identification more difficult. Potassium chloride, for instance, cannot be distinguished from sodium chloride under the microscope.

Having obtained a solution of a sulphate, or a mixture of sulphates, I place a drop of the solution on a glass slide and allow it to evaporate spontaneously in a place free from wind, draught or dust. I find a shelf in a glass book-case a suitable place. Five or six slides should be prepared at the same time. The next day two or three of the most perfect of these should be selected for mounting, but it is material to preserve an unmounted specimen for subsequent comparison with the mounted ones. The unmounted slide enables us to observe whether the salt effloresces, deliquesces, becomes opalescent, or has a silky lustre. Information regarding its refraction can also be obtained, for salts, like gypsum, whose index of refraction is very close to that of balsam will, when mounted in that medium, become invisible, or nearly so, in ordinary transmitted light.

The material I use for mounting is Canada balsam diluted with chloroform until it flows freely. I keep it in a small stoppered bottle, and find one of about half an oz. capacity very convenient. I pour this balsam very carefully, drop by drop, on the crystals to be mounted, place a thin cover glass over the balsam and allow the latter to consolidate by the spontaneous evaporation of the chloroform without subjecting the preparation to pressure or heat. From one to three days is sufficient, in ordinary cases, for the consolidation of the balsam.

I have found it convenient to divide this paper into three parts. The present part (Part I.) is devoted to remarks of an explanatory character. Part II. contains, in a tabular form, a list of sulphates with the most important or best known of their hydrates and double salts. It contains chemical information gleaned from various standard works on chemistry. The optical system to which each salt belongs, and its chemical formula, are given in cols. 1 and 4 on the authority of either Fock[1] or Roscoe and Schorlemmer.[2] Col. 5 indicates the form in which the salt appears when crystallised on a glass slide by the method above described, and embodies in every case the result of my own observations.

The forms described in col. 5 will not be found to agree, in every case,

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[1] *Krystallographisch-Chemische Tabellen*, 1890.

[2] *A Treatise on Chemistry*, Vol. II. The Metals. New Edn.

with the crystals figured in mineralogical or ordinary chemical works. The crystals figured in such works are often natural minerals formed in the laboratory of nature under conditions that cannot be reproduced by the micro-chemical student; in other cases they are typical examples of exceptionally perfect crystals selected from a large number obtained by the crystallisation of a considerable quantity of material from saturated solutions. For our purposes we must be content with the results that can be obtained by allowing a drop of an aqueous solution to crystallise on a glass slide by spontaneous evaporation. The conditions under which crystallisation takes place in the latter case differ from those which obtain in the cases above referred to, and the results are therefore sometimes considerably modified. The material at the disposal of the microscopist is often extremely small, and his methods, to be of any value, must be capable of dealing with small quantities.

Cols. 6, 7 and 8 contain optical observations for which I am responsible. Cols. 6 and 7 require a few words of explanation. I have given the order of colours exhibited by the crystals according to Newton's scale,[1] as I think this affords a rough mode of determining the double refraction of these salts which is of some practical value. Colours, no doubt, depend not only on the strength of the double refraction, and the direction taken by the light in its passage through a crystal, but also on the thickness of the crystal. But in dealing with the results of the spontaneous evaporation of an aqueous solution on a glass slide I think it will be generally found that each salt has a habit of its own in respect of the general size and general thickness of its crystals as well as of the faces on which they commonly lie, and that therefore they will generally yield fairly similar results as regards colours in polarised light. It will be found that whilst certain salts habitually yield colours as high as the 7th order, others never give colours higher than those of the 1st, 2nd or 3rd order, as the case may be. By discarding from consideration specially thin and attenuated crystals deposited when nearly the whole of the material in the drop had been exhausted, and by selecting for observation the crystals that exhibit the strongest double refraction, we obtain data of some value. Treated in this way the colours afford a rough practical guide to aid us in determining the identity of the salt. The order of colour recorded in Col. 6 is the maximum observed by me.

In Col. 7 the direction of the major axis of elasticity has been deter-

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[1] For the mode of ascertaining the order see my paper on Double Refraction of Minerals, *Geol. Mag.* 1888, p. 548. Also p. 65 of *Rosenbusch's Microscopical Physiography*, by Iddings.

mined with the aid of the quartz wedge. When it is said to be " || to *e* " it is intended to imply that when the axis of the quartz wedge is parallel to the axis of elongation of the crystal the phenomenon of *thinning* is produced. I have employed (*e*)[1] as an abbreviation for the ordinary direction of elongation because it excludes any pretence of absolute precision. Some salts habitually lie on one face on the glass slide and are elongated in one direction. In practice I find the direction of the major axis, as indicated in Col. 7, an aid to the determination of salts of great practical value.

In Col. 8 I have alluded to a phenomenon observed when the polariser is used without the analyser which is usually attributed to "absorption." Calcite, and some other colourless minerals, when examined in transmitted light with the polariser alone, exhibit dark lines, shadows and dark markings, in a certain position of the polariser (or when the polariser is rapidly revolved) which disappear altogether when the plane of vibration of the nicol is altered relatively to the plane of vibration of the light passing through the mineral. Some salts possess this property in a high degree—others in a much less degree or not at all. When a colourless salt, or mineral, possesses this absorption conspicuously it marks it off from other minerals that do not display the property. We have here therefore a mode of distinguishing colourless minerals from one another which I have endeavoured to utilise in Col. 8.

In a previous page I assumed that we had reduced the chemical constituents of a mineral to the form of soluble sulphates, and had crystallised a mixture of these sulphates on glass slides for microscopic examination. An inspection of these slides, with the information contained in Part II. before us, will often give us important information regarding the character of the mineral under examination. For instance, if we have treated a piece of serpentine in the manner described it will be at once seen that the principal chemical constituent of the mineral is magnesia, for unmistakable crystals of magnesium sulphate will occupy a prominent position on the slide. But supposing that we wish to confirm this determination, or to ascertain what other sulphates are contained in our solution, we can pass on to the reactions based on Part II., or to those detailed in Part III.

The double sulphates, recorded in Part II., supply us with a valuable means of detecting the presence of many metallic salts. Suppose, for instance, that we are seeking for evidence of the presence of sodium, we

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[1] I have occasionally given *c* when the vertical crystallographic axis is intended.

examine our original slides to see whether they contain any cubic crystals of alum. If they do not do so there is a strong probability that the mineral under examination does not contain either aluminium or chromium as well as sodium. We then place a few drops of the original aqueous solution on several glass slides and add two or three drops of an aqueous solution of aluminium sulphate to some of the slides, and two or three of chromic sulphate to the other slides, and allow these to evaporate spontaneously without the application of heat. If soda be present in the mineral crystals of alum will be found on the slides.

Then we can further test for soda (without going beyond methods based on Part II.) by employing, in the way described, the sulphates of calcium, magnesium or manganese, with each of which sodium sulphate forms double salts.

The above illustration refers to the detection of sodium only, but a reference to Part II. will show that the sulphates of many other metals may be detected by the employment of sulphates that form double salts with them. Cold solutions, combined with spontaneous evaporation, have yielded me the best results.

In Part III. I have given some selected tests *supplementary* to those suggested by Part II. Most of these reactions, due originally to Haushofer, Behrens, Boricky, and others, have been taken from the works of Klement and Renard,[1] and Behrens.[2] I have tried, and can recommend, all the reactions entered in Part III. In one or two cases I have given my reasons for rejecting some reactions that have been suggested by various authorities, but I think it unnecessary to specify my grounds for excluding other reactions which I have tried, but which, from their requiring great heat, great expertness on the part of the operator, or reagents that I could not obtain sufficiently pure, or for some other reason, were found unsuitable for ordinary use.

In Part III. I have noted facts that seem to me of interest regarding some of the salts alluded to therein, and I will now conclude with a few observations of more general character.

Several interesting facts will be apparent on a consideration of Part II. The first is that the addition of a single molecule of water to the formula of a hydrate is sufficient, in some cases, to completely alter the physical properties of the salt, involving not only a change in its optical properties, and in its solubility, but in some cases even a change of colour.

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[1] *Réactions Microchimiques*, par C. Klement et A. Renard, 1886.

[2] *Contributions to Micro-chemical Analysis*, by H. Behrens. *Chemical News*, 1891.

Conversely the loss of water, even when heat has not been employed, may result in a change of molecular structure, as in the interesting instance noted in Part III., Iron, where a salt changes from a glass into a crystal on losing some of its water.

The formation of one hydrate rather than another depends in some cases on the temperature at which crystallisation takes place, and in others on other considerations—solubility being probably a dominating factor. That is to say, as evaporation proceeds, and the volume of water present is reduced, one hydrate may be thrown down first and another later on.

The student may sometimes be disconcerted by the diversity of forms presented to him by micro-chemical reactions; but fundamental diversity of form in such cases is, I suspect, more often the result of minute difference in chemical composition than an instance of polymorphism. Hydrates, basic salts or double salts, of which little or nothing is yet known, may be formed under special conditions. These salts reveal themselves under the microscope in forms that are new to the student, and invite him to future excursions into unexplored fields of research.

Another thing that has interested me as a student of igneous rocks is the presence in these salts of aqueous origin of twinning, zonal structure and cleavage lines.

Many kinds of twinning—penetration twins, ordinary binary twins and polysynthetic twinning, combined with macles analogous to those on the pericline plan,—are to be found in crystals formed on glass slides in the manner above described, and they appear to be congenital in origin. They are by no means confined to crystals formed round the margin of a drop, where it might possibly be alleged that they had suffered strains from molecular tension, or from crowding; but they appear quite as commonly in discrete crystals standing by themselves in the centre of the area originally covered by the liquid.

The following abbreviations are used in Part II. :—

R.L.	for	right angle.
$\parallel$	,,	parallel.
e	,,	direction of elongation.
c.p.l.	,,	converging polarised light.
Q.W.	,,	quartz wedge.
C.	,,	cubic.
T.	,,	tetragonal.
H.	,,	hexagonal.
R.	,,	orthorhombic.
M.	,,	monoclinic.
Tr.	,,	triclinic.

PART II.

TABLE OF SULPHATES.

It will be convenient and will save space, to preface the Table of Sulphates with the general formula of the *alums.* Omitting the selenates and mixed selenates and sulphates, the general formula is $M_2 3SO_4 . N_2SO_4 . 24H_2O$; where

$M =$ Al, Cr, Mn, Fe, In or Ga; and $N =$ Na, K, Rb, Cs, NH_4, Ag or Tl.

The best known combinations are—.

M	with	N.
Al	,,	Na, K, NH_4, Rb, Cs, $NH_3(C_2H_5)$, Ag or Tl.
Cr	,,	Na, K or NH_4.
Mn	,,	— K or NH_4.
Fe	,,	— K or NH_4.
Ga or In	,,	— — NH_4.
M_2	,,	
CrAl	,,	— K or NH_4.

The *alums* are strictly isomorphous. They crystallise in forms belonging to the cubic system, usually in cubes or octahedra, or in octahedra combined with cubes.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Characters.	Remarks.
ALUMINIUM.								
$Al_2 (SO_4)_3$	1							
$Al_2 (SO_4)_3 + 18 H_2O$	2	Alunogen	M	(*a*) In six-sided tablets (*b*) Radiating tufts like horse tails and grasses	1st order	About 45° from *e*	Boundaries of platy crystals marked by fine dark lines	No. 2. Dissolves in two parts of water. The tablets overlap each other and form platy masses, in which six-sided micaceous crystals can be distinguished. Two angles of 92° and four of 134°. When not mounted in Canada balsam the crystals have a peculiar pearly gloss. The pearly appearance is produced by a granular salt white in reflected and opaque in transmitted light, reminding one of the beginning of kaolinisation in felspar. Query, is this one of the basic sulphates $Al_2O (SO_4)_2 + H_2O$, or $Al (OH) SO_4$?
Double Salts.		*See alums ante*						
BARIUM.								
$Ba SO_4$	3	Barytes	R	(*a*) Modified rhombic and rectangular prisms (*b*) X shaped skeleton crystals resembling ice crystals	1st order	(*a*) ‖ to *e*	(*b*) Extinguish at 45° from the arms of skeleton	No. 3. Almost insol. in H_2O (sol. 1 in 400,000). The p.p. formed by adding H_2SO_4 to an aqueous solution of a Ba salt does not yield definite crystals under the microscope. A Ba salt heated with concentrated H_2SO_4 dissolves in part, and deposits on cooling crystals of $H_2SO_4 + BaSO_4$. If exposed to moist air crystals of $H_2SO_4 + BaSO_4 + 2 H_2O$ are deposited. See Part III.
(Ba Sr) SO_4	4	*See Sr.*						

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Characters.	Remarks.
Beryllium. $BeSO_4 + 4 H_2O$	5		T	(*a*) Prisms with pyramidal terminations	7th order	(*a*) *R.L.* to *e*	Axial sections in *c.p.l.* cross well seen. Negative	Beryllium readily forms basic sulphates. They may be obtained by boiling the normal sulphate (No. 5) with beryllium carbonate. They form gelatinous masses. The normal salt dissolves at the ordinary temperature in its own weight of water.
				(*b*) In radiating and star-shaped arborescent forms		(*b*) ∥ to fibres		
$BeSO_4 + 7 H_2O$	6	—	M	In prisms, sometimes twinned	3rd orde	At a high angle to *e* & to twinning plane		No. 6 separates out from an acid solution of the normal salt.
$BeSO_4 K_2SO_4 + 2H_2O$	7	—	—	Globulites either isolated or in perlitic looking base	1st order	In quadrants at *R.L.* to *Q.W.*		No. 7. The outer margins of the globulites are hard black lines. They do not possess a radiate structure. The centres are often white in reflected and brown and opaque in transmitted light. The ground-mass may be either glassy or micro-granular.
Calcium. $CaSO_4$	8	Anhydrite	R	Usually combinations of the prism, pinacoids and basal planes. Microscopic crystals often boat-shaped	4th order. In micro-crystals, usually not higher than 2nd order.	∥ to *e*	Interference fig. in *c.p.l.* is seen when prism lies on its macropinacoidal face	No. 8 forms when concentrated H_2SO_4 is used. With dilute H_2SO_4 No. 9 forms.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
CALCIUM. $CaSO_4 + 2\ H_2O$	9	Gypsum	M	(*a*) Acicular prisms often in radiating groups (*b*) Swallow-tailed twins (*c*) Short prisms with rhombic outline. Acute angle about 50°. See Part III.	1st order	(*a*) About 38° from *e*, measured in direction of acute angle of rhomb	Refraction nearly the same as Canada balsam, viz, Gypsum $n=1{\cdot}52$. Balsam $n=1{\cdot}54$.	No. 9. See Part III.
$CaSO_4\ K_2SO_4 + H_2O$	10	Syngenite	M	Small tabular crystals generally showing 6 or 8 sides in outline	1st order	About 3° from (100)	No. 10 Refraction not far from balsam.	No. 10. Sol. in water 1 in 400 with separation of some gypsum. Slide shows numerous needles and prisms of gypsum with stumpy crystals of syngenite, roughly speaking resembling augite in habit. Twinning very common. Extinction between twins 15°—24°, sometimes less. Of the natural mineral Dana says it is elongated in the direction of vertical axis, and that the plane (100) always predominates.
$CaSO_4\ Na_2\ SO_4$	11	Glauberite	M	(*a*) Crystals of approximately rhombic outline with a strong rib down centre radiating like the petals of a flower (*b*) Radiating fibrous groups	(*a*) Blue of 2nd order (*b*) Double refraction feeble	(*a*) At a high angle to rib (*b*) Sometimes at high sometimes at low angle to fibre		No. 11. The crystals (*a*) have one of the acute angles pointing outwards: the other is at the centre of radiating group. The rib approximately bisects the acute angles. Many of the crystals strongly recall fig. 528 Dana's system 5th Edn. The rib is apparently the junction of $-1 : -1$ and $1 : 1$.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Characters.	Remarks.
CERIUM. Ce $(SO_4)_2 + 7H_2O$	12	Ceric Sulp.	—	Reniform mass	—	—	—	No. 12 is obtained by dissolving the dioxide in dilute H_2SO_4 and evaporating in a vacuum over H_2SO_4. Ceric sulphate is decomposed by water with formation of basic salts, which vary in composition according to the quantity of water present. It forms double salts with sulphates of the alkali metals, such as $Ce(SO_4)_2 + 2 K_2SO_4 + 2H_2O$ small yellow monoclinic crystals.
$Ce_2 (SO_4)_3$	13	Cerous Sulp.	—	Small crystals and long needle-shaped prisms badly terminated	—	—	No. 13. Straight extinction	No. 13. Separates from a warm solution. It dissolves in 6 parts of cold and 60 parts of hot water. Also in hot H_2SO_4 which on cooling throws down small crystals and long needle-shaped prisms, which are re-dissolved after standing for some hours.
$Ce_2 (SO_4)_3 + 8H_2O$	14	,,	M	Prisms terminated by clino-domes	7th order	At a very high angle to plane of twinning and to *c*	No. 14. Prisms extinguish 25° from vertical edge Some crystals exhibit absorption with single nicol	No. 14 separates from a concentrated solution. It occasionally exhibits multiple twinning.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
CERIUM. $Ce_2(SO_4)_3 + 9H_2O$	15	Cerous Sulp.	H	(*a*) Prisms with pyrl. terminations (*b*) Radiating fibres like spherulites (*c*) Radiating tables with the habit of a zeolite	1st order	(*a*) and (*b*) at *RL* to *e* (*c*) ‖ to *e*		No. 15 separates principally from dilute solution, but generally both 14 and 15 will be found on the same slide. Sol. 1 in 6. Cerous Sulp. forms several potassium cerous double salts, as $3K_2SO_4 + Ce_2(SO_4)_3$ $Ce_2(SO_4)_3 + 2K_2SO_4 + 3H_2O$ and $Ce_2(SO_4)_3 + K_2SO_4$. Sodium and ammonium sulphates also form double salts with cerous sulphate.
COBALT. $CoSO_4 + 4H_2O$	16		—	A peach-blossom coloured powder				No. 16. Deposited when a concentrated solution of Co. Sulp. is passed into H_2SO_4.
$CoSO_4 + 6H_2O$	17		M	In crystals				
$CoSO_4 + 7H_2O$	18	Bieberite or Cobalt Vitriol	M	Crystalline crusts made up of obscurely shaped granular crystals, with occasional 4- or 6-sided tabular crystals	2nd order	About 30° from *e*	Dichroic. Rose pink to amber. Relief sharp	No. 18. Sol. in 24 parts of cold water. Rose colour deepens on heating; crystal becomes opaque.
$CoSO_4\ K_2SO_4 + 2H_2O$	19	—	Tr					

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
COBALT.								
$CoSO_4.K_2SO_4 + 6H_2O$	20	—	M	Large tabular crystals with 6, 8 or more faces, 2 or more corners often truncated; edges sometimes bevelled	So high that a double Q.W. is sometimes required	(a) Within a few degrees of R.L. to *e* in approximately rectangular prisms (b) Within a few degrees of a line bisecting the acute angles in prisms of sphenic habit	Extinction in (*a*) within a few degrees of *e*, in (*b*) within a few degrees of line bisecting acute angles. Dichroic in rose pink and amber colours. Zonal structure. Twinning not uncommon	No. 20. On heating the rose coloured crystals become reddish violet. Some of the rose red crystals show a positive bisectrix.
$CoSO_4 (NH_4)_2SO_4 + 6H_2O$	21	—	M	Large stumpy prisms	Some as high as 6th, but generally 3rd order	No. 21. Within about 8° of *e* in the few cases in which the crystals are elongated	No. 21. Absorption with single nicol strong	No. 21. On heating changes from rose red to violet: on cooling rose colour returns. Traces of a very oblique bisectrix, apparently positive.
CHROMIUM.								
$CrSO_4$	22	Chromous Sulp.						No. 22. Only known in solution.
$Cr_2 (SO_4)_3$	23	Chromic Sulp.	—	Crystalline				No. 23. A *red* salt formed by heating No. 24 to 370°. Insol. in water, alcohol or acids.
$Cr_2 (SO_4)_3 + 5H_2O$	24		—	Non-crystalline				No. 24. A *green* salt. Sol. in alcohol. Formed by boiling No. 25, or by heating it to 100°.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
CHROMIUM. $Cr_2(SO_4)_3 + 15H_2O$	25	Chromic Sulp.	—				Green in transmitted light	No. 25. A *violet* salt. Insol. in alcohol.
$2Cr\,SO_4(OH)_4 + Cr_2(OH)_6 + 5H_2O$	26		—					No. 26. A basic salt. *Chromates* and *Dichromates*, see K and Na.
Double Salts. With Sulphates of K, Na and (NH_4)	—	*See Alums*	—	—	—	—	—	
CÆSIUM. Cs_2SO_4	27		R	Prisms with numerous faces	2nd order	*R.L.* to *e*		
$CsH\,SO_4$	28		R					
Double Salts. $Cs_2SO_4MgSO_4 + 6H_2O$	29	*See Mg.*	M	—	—	—	—	
With Sulp. of Al.	—	*See Alums*		—	—	—	—	
IRON. $FeSO_4 + 4H_2O$	30	Ferrous Sulp.	R M					$FeSO_4$ under ordinary conditions crystallises in the form of No. 32, but it takes the orthorhombic form if its supersaturated solution is touched with a crystal of zinc sulp. and the triclinic form (No. 31) if a crystal of copper sulp. is placed in a concentrated solution. When a solution of $FeSO_4$ containing free H_2SO_4 is allowed to evaporate in a vacuum, No. 32 separates first, then No. 31, and lastly No. 30. When the ferrous sulp. is exposed to the air it is changed into a compound containing ferric sulp. and ferric hydroxide, or a basic ferric sulp. $Fe_2(SO_4)_3(OH)_3$.
$FeSO_4 + 5H_2O$	31	"	Tr					
$FeSO_4 + 7H_2O$	32	Melanterite or green vitriol	M	When evap. on slide generally in rhombs or tabular prisms	7th order	Generally ‖ to a line dividing the obtuse angle of rhomb into two unequal portions		

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
IRON.								
$Fe_2(SO_4)_3$	33	Ferric Sulp.	—	In oval & rounded disks, often with radiating fibrous structure and in fibrous patches	*1st order*	$\parallel$ *to* axis of elongation	*Extinction* coincides with axis of elongation. Decided absorption with single nicol	Ferric Sulp. (No. 33) readily forms basic salts the composition of which is not positively known. It dissolves slowly in water, and is decomposed by heating into ferric oxide and sulphur trioxide. Cols. 5 to 8 is the anhydrous salt dissolved in water and re-crystallised.
$Fe_2(SO_4)_3$	34		R	Radiating needle-shaped prisms[1]	Blue of 2nd order	$\parallel$ to *e*		No. 34 is formed by adding No. 32 to boiling H_2SO_4.
$Fe_2(SO_4)_3 + 9H_2O$	35	Coquimbite	H	Prisms usually with terminal edges replaced				
$Fe_3(SO_4)_4 + 12H_2O$	36	Ferrous-ferric sulp.	M	Yellow crystals				No. 36. The two sulphates of iron form various double salts, of which this (Roemerite) is one.
FeS_2O_7	37	Ferrous disulp.	—	Green microscopic prisms				No. 37 is formed by adding iron sulp. to several times its volume of concentrated H_2SO_4. It decomposes on contact with water.
$FeSO_4(OH)_4 + Fe(OH)_6 + H_2O$	38	Vitriol Ochre	—	Massive, earthy, stalactitic				
$Fe_2(SO_4)_3 + Fe_2(SO_4)_2(OH)_2 + 10H_2O$	39	Copiapite	H ?	In six-sided crystalline scales	Red of 2nd order	$\parallel$ to *e*	Basal sections give cross on *bluish ground* in *c.p.l.*	
$2Fe_2(SO_4)_2(OH)_2 + Fe_2SO_4(OH)_4 + 24H_2O$	40	Fibroferrite	M ?	In delicate fibres				No. 40. Pale yellow to white, pearly or silky.

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[1] See Remarks, Part 3.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
IRON. *Double Salts.* $FeSO_4 Na_2SO_4 + 6H_2O$[1]	41		M	Globulites, spherulites and radiating fibrous crystals	2nd order	About 45° to longer axis of fibres	Sometimes has a concentric as well as radiating structure, and resembles Chalcedony	
$FeSO_4 K_2SO_4 + 6H_2O$	42		M	Large tabular crystals	7th order and higher	Variable	Absorption with single nicol, very strong	
$FeSO_4 (NH_4)_2 SO_4 + 6H_2O$	43		M	Large stout prisms variously terminated. Also in skeleton crystals	5th order	Within a few degrees of || to *e*	Strong absorption with single nicol. Zonal structure. *In c.p.l.* either an optic axis, or a more or less oblique bisectrix emerges	
With Sulp. of K & NH_4		*See Alum*						
POTASSIUM. K_2SO_4	44	Normal Sulp.	R	Flat prisms elongated in one direction: terminations various	2nd order	Generally ||, sometimes *RL* to *e*	Relief strong when mounted in balsam; when unmounted faces at angle to glass cover are black	No. 44. Sol. 1 in 10 in cold and in much smaller quantity of boiling water. Occurs in Apthitalite and Kainite. The diff. in appearance of mounted and unmounted specimens is a striking feature, and one useful for identification

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[1] See Remarks, Part 3.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
POTASSIUM.								
$KHSO_4$	45	Acid Sulp.	M R	(*a*) Six-sided tabular prisms (*b*) Needle-shaped ditto, also fibrous (*c*) Irregular shaped tabular crystals	6th order	Low angle to *e*	Absorption with single nicol. Mottled appearance under crossed nicols seldom extinguishing uniformly; unmounted deep black margin; strong relief; shagreened irregular surface marked with black lines. Penetration twins often seen	No. 45 occurs native in the *grotto del solfo*, near Naples, in the form of silky needles. Dissolves readily in water: is decomposed by alcohol into No. 44 & H_2SO_4. When 45 is re-crystallised from aqueous sol. No. 44 separates out first, then crystals of a salt having composition of $K_2SO_4 + KHSO_4$, and finally No. 45 crystallises out.
$K_2S_2O_7$	46	Disulphate	—	In long slender needles				No. 46 is obtained by heating No. 44 in H_2SO_4 until the mass fuses quietly. Decomposes on contact with water.
Double Salts.								
With Sulp. of Al, Cr, Mn, Fe, &c.	—	*See Alums*						
With Sulp. of Be	–	*See Be*						
,, ,, Fe	—	*See Fe*						
,, ,, Mg	—	*See Mg*						
,, ,, Mn	—	*See Mn*						
LITHIUM.								
Li_2SO_4	47		R					
$Li_2SO_4 + H_2O$	48		M					Li HSO_4 is also known.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
LITHIUM.								
$Li_2SO_4 + 3H_2O$	49		H R	(*a*) 6-sided prisms, elongated in one direction; some tabular, some radiated (*b*) Stars of radiating blades	White of 7th order	*R.L.* to *e*	Some of the crystals show a negative bisectrix in *c.p.l.*, almost all of (*a*) show traces of one	
$Li\,K\,SO_4$	50		H	Prisms with pyramidal terminations. Sometimes basal plane truncates the pyramid	Sometimes so low as to look like an *isotropic* mineral, sometimes pink of 2nd order	‖ to *c*	Relief good. Margins dark and sharp. Absorption with single nicol	
$Li\,Na\,SO_4$	51		H	Spherulitic	3rd order	Various	Relief good. Sharp dark margins Slight absorption.	Some of the spherulites have a radial structure, some have not, and others show a fixed dark cross when revolved between crossed nicols. In the former the cross is very irregular, owing to overlapping. Sometimes opaque centres. In *c.p.l.* the spherulites show traces of lemniscates.
$Li\,NH_4\,SO_4$	52		H	Large six-sided micaceous-looking plates	Generally very low, sometimes as high as blue of 2nd order	*R.L.* to *e*	(*n*) Cannot be far from that of Canada balsam	

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
LITHIUM.								
Li Rb SO_4	53		H	(*a*) Hexagonal tabular crystals (*b*) Rectangular four sided plates (*c*) Fibrous or stalk-like	Very feeble	*R.L.* to *e*	Outline well defined	No. 53. The hexagonal tables differ from those of $RbSO_4$. Latter are transparent, sharply outlined, and much twinned. 53 are often only translucent, never in my specimens clear or glassy.
MAGNESIUM.								
Mg SO_4	54	—	—					No. 54. When this salt is dissolved in H_2SO_4 the solution on standing deposits $MgSO_4$ + H_2SO_4 in 6 sided tables, which soon absorb moisture from the air and decompose.
Mg SO_4 + H_2O	55	Kieserite	M	In prisms, or in granular masses				No. 55. This salt is as difficult of solution in water as gypsum.
Mg SO_4 + $6H_2O$	56	—	M					
Mg SO_4 + $7H_2O$	57	Epsom Salt	R	(*a*) Needle-shaped prisms in radiating groups (*b*) Stout prisms	(*a*) 2nd order (*b*) Sometimes as high as 7th order	(*a*) *R.L.* to *e* (*b*) Sometimes *RL* to *e*, sometimes ‖ to *e*	Absorption with single nicol	No. 57. At the ordinary temperature 100 parts of water dissolve 125 of salt. Epsom salt on heating melts in its water of crystallisation, and loses 6 molecules of water at 150°. The last molecule is not driven off until 200° is reached. The prisms are usually 6 sided, with complex to rounded terminations. 57 (*b*) commonly contain liquid cavities with bubbles; also endo-prisms. There is conclusive evidence that the latter are not negative crystals.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
MAGNESIUM. Mg SO_4 Na_2 SO_4 + $6H_2O$[1]	58	Blödite	M	(a) Crystals like primrose leaves radiating from a centre (b) Elongated irregular shaped prisms radiating from a centre (c) Spherulitic radiating groups	(a) 1st order (b) 7th order (c) Blue of 2nd order	(a) About 45° from axis of quartz wedge (b) High angle to *e* (c) About *R.L.* to *Q.W.*	Absorption with single nicol	
Mg $SO_4K_2SO_4$ + $6H_2O$	59		M	Large irregular crystals	7th order, sometimes higher	—	Absorption here and there. Internal structure often not homogeneous, giving dusty appearance	
Mg SO_4 $(NH_4)_2$ SO_4 + $6H_2O$	60		M	(a) Prisms with complex terminations elongated in one direction (b) Stumpy prisms, not elongated	(a) 3rd order (b) 7th order	(a) Within 8° or 9° of ‖ to *e*	Strong absorption with single nicol zonal structure. (a) Extinction 5° to 8° from (*e*) In *c.p.l.* an optical axis emerges from (*a*).	No. 60. Liquid cavities with bubbles very common. Often arranged in zones.
Mg SO_4Cs_2 SO_4 + $6H_2O$	61		M	Large tabular crystals; shapes varied and irregular, rarely well formed prisms	Sometimes as high as 6th order	—	Absorption with single nicol	No. 61. Liquid cavities with bubbles rather numerous.

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[1] I have followed Roscoe and Schorlemmer. Fock gives $4H_2O$ for this salt.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
MAGNESIUM.								
Mg $SO_4 Rb_2 SO_4 + 6H_2O$	62		M	Generally in rhombs, sometimes with two corners truncated	5th order	High angle to *e* when crystal is elongated	Absorption with single nicol	If the concentrated solution of Manganous Sulphate is allowed to evaporate *below* 6°, rhombic crystals of No. 66 are deposited. Between 7° and 20° (viz. at the ordinary temperature), crystals of No. 65 are formed. Between 20° and 30° No. 64 is deposited. (Roscoe calls them "quadratic"); at the same time a crystalline crust containing $3H_2O$ is formed. When the hydrates are heated to 200°, or their concentrated solution is boiled, the anhydrous salt $MnSO_4$ is deposited in a reddish yellow powder. It decomposes at a bright red heat. Manganous Sulp. is insol. in absolute alcohol but it removes a portion of the water from the hydrates.
MANGANESE.								
Mn $SO_4 + H_2O$	63		M					
Mn $SO_4 + 4H_2O$	64		R M					
Mn $SO_4 + 5H_2O$	65			A platy mass of felted blade-like crystals, sometimes radiated, sometimes like the shafts and barbs of a bird's wing	7th order	High angle to *e*	Strong absorption with single nicol. Some crystals show a negative bisectrix	
Mn $SO_4 + 7H_2O$	66		R M					
Mn $SO_4 Na_2SO_4 + 2H_2O$	67		?					
Mn $SO_4 K_2SO_4 + 2H_2O$	68		Tr					
Mn SO_4 HK$SO_4 + 2H_2O$	69		Tr					

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
MANGANESE.								
Mn SO_4 Na_2 SO_4 + $4H_2O$	70		M	(*a*) Almond shaped granules (*b*) Spherulitic radiating groups (*c*) Short prisms of cubic aspect	(*a*) (*b*) Pink of 2nd order	(*a*) *R.L.* to *e* (*b*) *R.L.* to axis of *Q.W.* (*c*) Uncertain		Nos. 70-73. The salts described under these Nos. are those obtained by evaporating a mixture of $MnSO_4$ with Na_2SO_4, K_2SO_4 and $(NH_4)_2$ SO_4. I know of no test applicable to microscopic specimens on glass slides for determining whether the resulting double salts contain 2, 4 or 5 molecules of water
Mn SO_4 K_2 SO_4 + $4H_2O$	71		M	In (*a*) spherulites and (*b*) confused matted masses	Blue of 2nd order	(*a*) ‖ to axis of *Q.W.*		
Mn SO_4 $(NH_4)_2$ SO_4 + $6H_2O$	72		M	Large stumpy prisms of complex shape	3rd order	—	Strong absorption with single nicol. Refraction probably near that of balsam. Small crystals cannot be seen without polarised light	No. 72. Some crystals have well marked zonal lines. Others are so full of minute liquid and air bubbles as to present a dark dusty appearance under low powers. Many of the crystals show an optic axis in *c.p.l.*
Mn SO_4 K_2 SO_4 + $6H_2O$	73		M					
Mn_2 $(SO_4)_3$, with Sulphates of K and NH_4		See *Alums*						
SODIUM.								
Na_2 SO_4	74	Salt Cake	R					
Na_2 SO_4 + $2H_2O$	75		R					

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
SODIUM.								
$Na_2SO_4 + 3H_2O$	76		H R					
$Na_2SO_4 + 10H_2O$	77	Glauber's Salt	M	Short, stumpy prisms, showing many faces	Extremely feeble: from grey of 1st order to blue of 2nd order	Low angle to *e*	Crystals bordered by broad dark lines. Retraction probably high	No. 77. Glauber's Salt (mirabilite) effloresces on exposure to the air. It forms from aqueous solution between 12° and 33°. Between 33° and 40° the anhydrous salt (74) is deposited. Solubility in water is greatest at 33°, after that it declines. Between 12° a salt with 7 molecules of water forms. The anhydrous salt also forms if glauber's salt is allowed to remain exposed to the air. The anhydrous salt occurs native in thenardite and in solution in sea water, and in some springs.
$NaHSO_4$	78		Tr[1]	(*a*) Long blade-like prisms (*b*) In granules or stumpy prisms with many faces	Sometimes as high as 6th or 7th order	Varies greatly	Twinning not uncommon. In (*a*) the twins form penetration twins, taking the form of a cross. Outlines sharp	
Na_2SO_4 with $Al_2(SO_4)_3$ and $Cr_2(SO_4)_3$		See *Alums* See *Alums*						
$Na_2SO_4 CaSO_4$		See *Ca*						
$Na_2SO_4 MgSO_4 + 6H_2O$		See *Mg*						

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[1] I have followed Roscoe and Schorlemmer. Fock does not notice $NaHSO_4$, but gives $NaHSO_4 + H_2O$ as a Monoclinic Salt.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
SODIUM.								
$Na_2 SO_4 Mn SO_4 + 2H_2O$		See *Mn*						
$Na_2 SO_4 Mn SO_4 + 4H_2O$		See *Mn*						
$Na Li SO_4$		See *Li*						
AMMONIA.								
$(NH_4)_2 SO_4$	79	Mascagnite	R	Long flat prisms Laths with rectangular tops common	Some as high 6th order	*R.L.* to *e*		No. 79. Long rod-like liquid cavities containing air bubbles running with the length of the crystal are common. This salt is said to be isomorphous with K_2SO_4 but they may readily be distinguished under the microscope. There is a striking difference between them in their refraction; their double refraction is different, and the crystals of $(NH_4)_2 SO_4$ are much larger and flatter than those of K_2SO_4. No. 79 dissolves at 0° in 71 and at 100° in 97·5 parts of water.
$(NH_4) H SO_4$	80		R	Stumpy prisms; complex shapes, six-sided outlines not uncommon. Twinning common	Very high, probably near Calcite	*R.L.* to *e* when elongated. ∥ to twinning plane of one twin, sometimes of both		No. 80. Very deliquescent. Aqueous solution deposits crystals, but does not completely evaporate in the open air at the ordinary temperature.

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
AMMONIA.								
$(NH_4)_2SO_4$ with Al, Cr, &c.	—	See *Alums*						
$(NH_4)LiSO_4$	—	See *Li*						
$(NH_4)_2SO_4MnSO_4 + 6H_2O$	—	See *Mn*						
$(NH_4)_2SO_4MgSO_4 + 6H_2O$	—	See *Mg*						
RUBIDIUM.								
Rb_2SO_4	81		R	(*a*) 6-sided plates of hexagonal habit (*b*) Lath-shaped with ragged ends (*c*) Regular well formed crystals with complex shapes round edge of drop	4th order	(*b*) *R.L.* to *e* (*c*) Various	(*c*) Absorption with single nicol. Multiple twinning almost invariable	See Remarks No. 53.
$Rb_2SO_4Al_2(SO_4)_3 + 24H_2O$	—	See *Alums*	R					
$Rb_2SO_4MgSO_4 + 6H_2O$	—	See *Mg*						
$RbLiSO_4$	—	See *Li*						
STRONTIUM.								
$SrSO_4$	82	Celestine	R	Globulites, round and almond-shaped disks, and in prisms	White of 1st order	In almond-shaped disks ‖ to longer axis		No. 82. Solubility in water between $CaSO_4$ and $BaSO_4$; viz. 0·015 parts dissolve in 100 of water at 60° F. It is less soluble in boiling than in cold water. When $SrSO_4$ is dissolved in hot concentrated H_2SO_4 crystals of celestine are deposited on cooling. See Part 3

	No.	Name.	System.	Form.	Double Refraction shows Colours of.	Direction of Major Axis.	Other Optical Properties.	Remarks.
STRONTIUM. (Sr Ba) SO_4	83	Baryto-Celestine	R					
TITANIUM. Ti $(SO_4)_2$	84	Normal Sulp.	—					
(Ti O) SO_4	85	Basic Sulp.	—	White hard mass				No. 85. Obtained by dissolving dry titanic acid in boiling H_2SO_4 and evaporating
$Ti_2 (SO_4)_3 + 3H_2O$	86		—	A resinous transparent yellowish deliquescent mass				No. 86 is formed by the oxidation of the sesqui-sulphate with Nitric Acid and evaporating
$Ti_2 (SO_4)_3 + 8H_2O$	87		—	Crystals in tufts				No. 87 is obtained by dissolving the metal in dilute H_2SO_4
ZIRCONIUM. $Zr (SO_4)_2 + 4H_2O$	88		R	Spherulites, elongated lozenges, and 6-sided micaceous looking thin plates. Spherulites often constricted in the middle like dumb-bells	3rd Order	‖ to *e* Spherulites ‖ to axis of *Q.W.*	Absorption with a single nicol. The elongated lozenges are deeply scored with numerous fine dark lines, which give them the appearance of bundles of micaceous plates	Swells up on heating with alum. Decomposes at a red heat

PART III.

Aluminium.[1]

Cæsium chloride is usually recommended as a test for the salts of aluminium, the reaction yielding cæsium-aluminium-alum (cubic), but inasmuch as CsCl itself crystallises in the cubic system, there is serious danger of the reagent being mistaken for the desired product. The sulphates of Cs or Rb are more suitable (Cs_2SO_4 is recommended), as they both crystallise in the rhombic system and cannot therefore be mistaken for cæsium-alum. See alum *ante.*

It is essential that samples of the sulphates obtained from the mineral under examination should be crystallised on glass slides prior to the addition of Cs_2SO_4, or Rb_2SO_4, in order to be sure that the mineral does not yield an alum without the addition of the reagent. When Mg as well as Al is present, as $MgSO_4$ forms monoclinic double salts with Cs_2SO_4 and Rb_2SO_4 (see Nos. 61 and 62, Part II.), sufficient of the reagent must be used to satisfy any $MgSO_4$ that may be present and leave sufficient over to convert the Al-sulphate into Cs- or Rb-alum. The advantage of Cs_2SO_4, or Rb_2SO_4, is that neither of these reagents forms alums with the sulphates of other metals (see alums).

Barium.

The sulphate of barium is almost insoluble in water (1 in 400,000), and barium precipitated by H_2SO_4 has no crystalline form under the microscope. The sulphate, however, dissolves to a considerable extent in hot concentrated H_2SO_4 and deposits on cooling crystals, having the composition $H_2SO_4 + BaSO_4$ and $H_2SO_4 + BaSO_4 + 2H_2O$. Very frequently both are deposited together, the character of the deposit depending a good deal on the condition of the atmosphere at the time. If the air is damp the hydrous salt may predominate; if it is dry the anhydrous salt may be the one principally found on the glass slide. $H_2SO_4 + BaSO_4$ is in microscopic four-sided[2] transparent crystals (*a*) as shown in figs. 20, 21.

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[1] The bases in Part III. are arranged alphabetically with reference to their *symbols*; soda, for instance, being under the letter *N*.

[2] By this and similar expressions it is intended that as seen in transmitted light, the crystal has a four-sided *outline*.

One of the four sides is very commonly a little longer than the others, and this side is frequently curved inwards like a concave lens. These small transparent crystals have straight extinction. More rarely crystals of hexagonal and more complex outline are formed. When the hot acid is exposed to a current of air, especially damp air, large skeleton crystals (*b*) are formed which bear considerable resemblance to negative ice crystals. These are the skeleton forms of tabular rhombic crystals, and every transitional form up to a perfect rhomb may be seen. Some of these are depicted at figs. 19, 22-25. The skeletons extinguish in a line bisecting two arms, as indicated by the dotted lines and the letters N.S. in the illustration (figs. 22 to 25). This is a very interesting fact, it seems to me, which throws light on the process of crystal building. Each molecule of these embryo crystals (figs. 22, 23) appears to be orientated not with reference to its present, but with reference to the ultimate form of the perfect crystals (fig. 25). Each molecule appears to have been endowed with (what for want of a better term we must call) polarity, and to have attached itself to its neighbour in a way that needed no subsequent modification as the mineral grew from a crystallite into a perfect crystal.[1] The fact that some embryo crystals of doubly refracting minerals show very little or no double refraction is, I think, due to the fact that owing to their excessive thinness they do not show colours between crossed nicols.

The sulphuric acid test is a delicate and sure one. It may be combined with ordinary testing in the following way: Place some of the liquid to be tested in a test tube or watch glass. If H_2SO_4 gives a white p.p., evaporate to dryness. Add concentrated H_2SO_4; heat for some time; draw off some of the clear, hot, H_2SO_4 with a pipette and place a few drops on a warm slide. If anhydrous crystals (*a*) be desired allow the H_2SO_4 solution to cool gradually in a warm dry room. If the hydrous salt (*b*) be desired, allow the H_2SO_4 to cool in or near an open window or exposed to steamy air. If the preparation is to be mounted in Canada balsam allow the slide to stand for 24 hours, then cautiously drain off the free H_2SO_4 with strips of filter paper.

(2.) If barium is present as sulphate I convert it into the chloride by the following process:—Evaporate solution to dryness; mix powder in agate mortar with sufficient charcoal to make the mixture dark grey in colour. Heat in small covered earthenware crucible strongly for one hour over a Bunsen burner. This converts the sulphate into the sul-

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[1] For another illustration of the same principle see Calcium (2).

phide. Dissolve in dilute HCl and heat until all the sulphuretted hydrogen is given off; filter, evaporate to dryness; dissolve in water and allow a few drops to evaporate on a glass slide. The crystals of $BaCl_2+2H_2O$ (*Monoc.*) thus obtained are in square prisms very much resembling those of augite in shape. They almost always exhibit a very characteristic polysynthetic twinning closely resembling the albite-baveno and albite-pericline twins of felspar. Two sets of these twins may sometimes be seen. Each set of multiple twins is apparently parallel to a pinacoidal face and they intersect at about 90°. A third and fourth set may also be seen, which appear to be parallel to the prismatic faces.

(3.) With a solution of tartar-emetic barium chloride yields tabular rhombs with two acute angles of about 50° each. Sometimes one or both of the acute angles are cut off, giving six-sided forms. They extinguish parallel to a line bisecting the acute angles, and the major axis is parallel to that line. They polarise brilliantly in colours of the 1st and 2nd order. These crystals very commonly exhibit multiple twinning, the twinning plane being parallel to a line bisecting the obtuse angles of the rhomb.

Beryllium (*Glucinum*).

The principal reaction mentioned in Klement and Renard's *Réactions Micro-chimiques* is the bichloride of platinum, but I do not think this is to be relied on when the beryllium to be tested is in the form of sulphate, for this salt and the chloroplatinate of beryllium both yield crystals belonging to the tetragonal system and consequently one might be mistaken for the other.

(1.) The monoclinic salt may be obtained by dissolving the normal salt (tetragonal) in H_2SO_4. See Part II.

(2.) The double salt with K_2SO_4 is very characteristic. See Part II.

(3.) On the addition of KOH solution a p.p. of white flocculent $Be(OH)_2$ forms which dissolves in an excess of the precipitant or on application of heat. A p.p. reforms on addition of a few drops of ammonium chloride. The p.p. formed by KOH, on being allowed to crystallise spontaneously, deposits crystals of K_2SO_4 imbedded in $Be(OH)_2$. The latter appears in radiating sheaves, apparently monoclinic. Extinction $0°-25°$ to c, and this is also the major axis. Double refraction very high, reminding one of calcite.

(4.) By treating a solution of the sulphate with a solution of ammonium carbonate very remarkable crystals of beryllium carbonate may be obtained, which I think have not yet been described. If too much of the ammonium carbonate is used the mixture will yield a white opaque amorphous mass; if too little is used the result will be a structureless glass; but if the right proportion of the reagent is added a carbonate is obtained that gives crystals which resemble bivalve shells in a remarkable manner (see figs. 7, 8 and 9). This resemblance is increased by the fact that they project, sometimes standing up on edge, sometimes lying more on their sides, from a ground-mass of granular ill-formed crystals that look extremely like a matrix of broken shells. The large and perfect crystals have two discoidal faces (resembling the upper and lower valves of shells) joined by a sharp edge, which is very frequently truncated at one side in a way to simulate the hinge of a bivalve shell. (See fig. 8.) Straight extinction: viz. || to axis of elongation. Major axis || to *e*. Between crossed nicols these crystals polarise in the blue of the 2nd order. Twinning is not uncommon. The combinations presented are varied, but that depicted at fig. 10 is a common type. After mounting in Canada balsam the crystals gradually become more or less corroded.

(5.) The reagent recommended by Behrens is potassium oxalate, but the objection to this is that the results vary greatly with the proportion of the reagent to the beryllium salt. I obtained with one drop of the reagent a mass of globulites showing a cross under crossed nicols, and with from 2 to 6 drops: (*a*) massive crystals; (*b*) dendritic arrangements bristling with very sharply pointed spear heads (rhombs with one of the acute angles projecting); (*c*) lenticular crystals (with rather sharp terminations) shaped like a Zulu shield; and (*d*) rosette-like groups of (*c*) composed of two or more individuals. Extinction in (*b*) oblique; in (*c*) and (*d*) straight, viz. || to *e*. Double refraction: (*b*), (*c*), and (*d*) polarise brilliantly in the blues and reds of the 2nd order. Refraction strong. I have not observed any twinning in (*c*) or (*d*); the groups of crystals (*d*) are not in my specimens penetration twins, but thin plates overlapping each other.

Calcium.

Gypsum ($CaSO_4 + 2H_2O$), the form in which the sulphate crystallises from aqueous solution under ordinary conditions, is soluble in the proportion of one part in 500 parts of water at the ordinary temperature. The maximum solubility is attained at about 35°, after which it declines. I find in practice that by boiling powdered gypsum in water for from 10 to 15 minutes sufficient of the sulphate is taken up to give good typical crystals when a few drops of the solution are allowed to evaporate on a glass slide. The crystals usually lie on the 010 face, and are commonly elongated in one direction (apparently 110). Gypsum prisms are often very slender and radiate from a common centre like the rays of a star. This grouping of slender needle-shaped prisms is very characteristic of the mineral, but it must not be accepted as conclusive evidence of its identity, as several other salts have a similar habit, and some of these have an oblique extinction similar to gypsum.

More important, to my mind, is the presence of very peculiar twins, which have always been present when I have allowed an aqueous solution to evaporate spontaneously on a glass slide. See figs. 26 to 31. The re-entering angles appear to be formed by the prism faces (110), as a fibrous striping (110 cleavage) is very distinct on almost every twin crystal. Sometimes this has a curious resemblance to polysynthetic twinning. The twin crystals depicted at figs. 26 to 31 disappear when they are mounted in Canada balsam. Owing to their thinness and the consequent feebleness of their double refraction, they rarely show even between crossed nicols.

At figs. 27, 31 a curious notching of the faces may be observed. When this is deep, and the crystals are wider in proportion to their length than those shown in the illustration, the twins assume the appearance of a star or rosette. The sides are usually curved, as shown in figs. 26, 27, and 29.

Another very common and characteristic form is that of a prism shaped like fig. 36. These prisms are usually isolated from other crystals, but sometimes they appear in combination with other forms, as, for instance, in (*c*) (*d*) of figs. 26, 29-31. The major axis of elasticity, *ma* of fig. 36, is theoretically 37°28′ from the axis of elongation, measured towards the acute angle of the rhomb, but it varies, I find, from 33° to 43°.

The above characteristic forms are often seen together on the same glass slide.

(2.) If a few drops of calcium sulphate in aqueous solution are placed on a glass slide and a couple of drops of ammonium carbonate are added, the solution becomes clouded. On spontaneous evaporation numerous grains and congeries of grains of calcite are left on the glass. Long filamentous crystals, or strings of grains, are not uncommon, and these occasionally form skeleton rhombs, the obtuse angle of which varies from 105° to 108°. The high refraction and double refraction of calcite makes the detection of calcite crystals very easy. Being thin they usually polarise in brilliant colours. I have not succeeded in making magnesium carbonate in the above way, otherwise the latter might be mistaken for $CaCO_3$.

(3.) Concentrated H_2SO_4 raised to the boiling point dissolves $CaSO_4$ to a certain extent, and deposits, on cooling, a salt which, according to Roscoe and Schorlemmer,[1] consists "of microscopic prisms which have the composition $CaSO_4 + H_2SO_4$." The results obtained by me are as follows: a little of the hot acid solution allowed to cool slowly on a glass slide, and to stand for some time, deposits embryonic crystals consisting of round and oval disks and moss-like clumps, the latter of which, under high powers, are seen to be composed of spicules sticking out in all directions. More rarely microscopic crosses consisting of four arms and obscurely shaped crystals may be seen. Straight extinction, viz. parallel to the longer axis of the oval disks and to the arms of the cross. Major axis at right angles to the longer axis of the oval disks; relief good; the disks polarise in the yellow of the first order. The round disks in transmitted light are rather suggestive of silkworms' eggs; in reflected light they are a dull dead white. Sometimes I have obtained a quantity of well-shaped crystals of the form shown at fig. 5; the sharp bows of these boat-shaped prisms meet at an angle of about 62°. At other times I have obtained crystals of the shapes shown at figs. 1-4. That at fig. 1 is not a thin platy crystal, but owing to the action of free H_2SO_4 on the balsam, and other causes, it is very difficult to make out the precise shapes of the side faces. Sometimes the top seems to be four-sided (like fig. 2), and the side faces to be six-sided like the outline of fig. 5; but they cannot be made out exactly. All these crystals (figs. 1-5) appear to belong to the orthorhombic system, and the mineral I doubt not is anhydrite ($Ca\ SO_4$).

Cerium.

Klement and Renard put in the foreground as tests oxalic acid and ammonium oxalate, but I have not obtained any very reliable results from these. I prefer ammonia as a test. This does not yield crystals, but its results are striking. If a drop of $CeSO_4$ solution is placed on a glass slide and a drop of liquid ammonia is placed near it, the vapour from the latter, even when the drops are not allowed to touch each other, causes the $CeSO_4$ drop to assume a beautiful opalescent appearance. If the drops are allowed to evaporate side by side, or if ammonia be added to the $CeSO_4$, an isotropic deposit is formed which shrinks considerably as evaporation proceeds and becomes scored with cracks that give it the appearance of a

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[1] Vol. II. (1), p. 203.

slice of serpentine. When unmounted the dried deposit generally exhibits, like opal, a blue and red sheen.

(2.) With ammonium formate crystals of cerium formate, $Ce_2\ (Co_2H)_6$, are deposited in rounded, or six-sided, generally opaque white clumps, made up of micro-prisms which usually project from the edge of the mass. They have straight extinction. Direction of major axis doubtful. On mounting in balsam a few become translucent and show colours up to the blue of the second order. They look like radiating bunches of feathers, each feather with a central shaft and side barbs.

(3.) With K_2SO_4 (see remarks Part II.) cerous sulphate forms double salts. Under the microscope these form transparent hexagonal disks, microscopic crystals of complicated structure, some of which have pyramidal terminations, globulites, and irregular hexagonal disks made up of overlapping or irregularly grouped crystals. The apparently heterogeneous internal structure is increased by numerous inclusions of opaque matter. When the globulites are densely packed together so as to form an opaque crust, they often appear in reflected light like branching stalks of coral. Refraction high ; double refraction never higher than blue of 2nd order.

(4.) With Na_2SO_4 and $Ce_2(SO_4)_3$ in the right proportion we obtain small four-sided prisms terminated by sharp pyramids at both ends, or lenticular granules with sharp ends often arranged in groups like flowers with four petals. Extinction || to *e*. Major axis at right angles to *e*.

(5.) With potassium ferrocyanide, cerous sulphate yields (*a*) hexagonal disks ; (*b*) short hexagonal prisms lying on their sides ; and (*c*) stars with six rays, each star being an embryo hexagonal disk. The passage of the star into the hexagonal disk may often be seen, the almost complete forms being a hexagonal disk with a dark line running from the centre of each face towards the centre of the crystal. Other forms also occur, viz.: (*d*) four-rayed stars passing into: (*e*) short prisms of cube like aspect; (*f*) bundles of bud-shaped crystals ; (*g*) rounded complexes of small crystals suggestive of marigold flowers ; and some variations of the skeletal form (*d*). (*a*) and (*c*) remain dark between crossed nicols ; (*b*) and (*e*) have straight extinction ; (*d*) polarises parallel to a line bisecting the rays of the star, and (*f*) to the axis of bundle. Major axis || to length of prism in (*b*) and at right angles to longer axis of (*f*).

Cobalt.

(1.) If potassium nitrite solution be added to the aqueous solution of $CoSO_4$ and then acetic acid to strong acid reaction, and the mixture gently warmed, a beautiful yellow precipitate (resembling the colour of chrome yellow) is formed. Under the microscope this yellow deposit ($K_6Co_2N_{12} + 8H_2O$) is seen to be made up of very small isotropic crystals in the shape of four-rayed stars or crosses with short arms. These are said to be the skeletons of crystals belonging to the cubic system. They require a $\frac{1}{8}$ inch objective and 2nd eye-piece for their definition.

(2.) Solutions of cobalt that do not contain nickel, as an impurity, are said in Klement and Renard's work to give with oxalic acid the oxalate of cobalt ($CoC_2O_4 + 2H_2O$) in rectangular flat prisms (straight extinction) which are often grouped as rosettes, and one is referred to Plate VI., fig. 11, of their work for the form of these rosettes. Oxalic acid (straight extinction) itself, however, sometimes occurs in radiating groups of fine prisms exactly as those represented, and it seems possible that, in this case, the reagent may have been mistaken for the oxalate of cobalt. In my experiments this reaction has yielded white opaque globules with rough edges suggestive of a resemblance to the spores of ferns. Among these there are also numerous slender crystals of oxalic acid, some of which are in radiating star-shaped groups.

Chromium.

(1.) For the following test the chromium salt should be converted into an alkaline chromate by ignition with potassium nitrate and an alkaline carbonate. Dissolve the resulting chromate in water and then neutralise the alkaline solution with acetic acid. Add a drop or two of a solution of nitrate of silver. The liquid turns blood red. On spontaneous evaporation microscopic crystals of chromate of silver are deposited (Ag_2CrO_4). Sometimes these crystals are in very minute and short rods, and remind one of a clotted mass of some species of microbe, at other times they are of larger size and more irregular shape. They are then seen to be of blood red colour in transmitted light, and to be transparent or translucent. In any case they require a $\frac{1}{8}$ inch objective and the 2nd eye-piece. As the chromate of silver is insoluble in water all trace of the chromate of potash can be removed by washing. The residue dissolves readily on the addition of a few drops of ammonia to the slide, and on evaporation thin, branching, moss- or lichen-like clumps and tufts of $Ag_2CrO_4 + 4NH_3$ are deposited. These tufts are opaque to translucent, the latter being of blood red colour in transmitted light. Powerful absorption with a single nicol (blood red to nearly black), but between crossed nicols the double refraction is so feeble as to be nearly nil. In reflected light the crystals have a brilliant metallic lustre. These crystals are said (Haushofer, quoted by Klement and Renard, p. 44) to occur in elongated rhombic plates, but I have not obtained any that exhibit a definite crystallographic form.

(2.) To the dichromate add a drop or two of barium chloride. On evaporation the chromate of barium ($BaCrO_4$) will be found on the slide. This is insoluble in water, and is of yellowish white colour and of granular texture. If the solution was strong the deposit will be opaque and dotted over with the cubic crystals of potassium chloride. If very dilute the barium chromate commonly yields skeleton crystals in the form of four-armed crosses. The arms of these crosses are varied and irregular in shape, but they all more or less resemble the fronds of a fern with transverse lobes. They require $\frac{1}{5}$ inch objective and have only a feeble action on polarised light. See fig. 6.

Cæsium (Cs).

Stannic chloride[1] gives with Cs_2SO_4 (orthorhombic system) a multitude of micro-crystals belonging to the cubic system (principally octahedrons) and star-shaped skeleton crystals, shaped like negative ice crystals, but consisting usually of three (occasionally four) rays.

Cæsium chloride gives with the above reagent small octahedrons and colourless crystals of the cubic system shaped like fig. 11. The latter usually predominate. Cæsium chloride itself crystallises in the cubic system (Cs_2SO_4 is therefore to be preferred), but its habit is different from Cs_2SnCl_6. I have always found cæsium chloride crystallise in cubes like common salt.

The bichloride of platinum gives with the salts of NH_3, K, Cs, and Rb crystals similar in shape to those of Cs_2SnCl_6.

Iron (Fe).

The ferro- and ferri-cyanides of potassium give such delicate and characteristic reactions that hardly any other tests are required to detect the presence of iron. Basic sulphates, about which little seems to be known from a microscopic point of view, are numerous, and they form on the application of heat or on a solution being allowed to oxidise in the air, and this renders the determination of iron with the aid of the microscope very difficult. Of microscopic tests the following may be mentioned:—

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[1] $SnCl_4 + 3H_2O$ (in needles); $SnCl_4 + 5H_2O$ (opaque acute prisms); $SnCl_4 + 8H_2O$ large transparent crystals. These hydrates all crystallise in the monoclinic system.

(1.) With oxalic acid ferrous sulphate gives greenish yellow crystals of ferrous oxalate ($FeC_2O_4 + 2H_2O$, solution 1 in 3,800). They are of microscopic size, belong to the orthorhombic system, and have their major axis of elasticity at right angles to *e*. They are ordinarily rectangular in shape but are sometimes prisms with six sides terminated by domes. They polarise rather brilliantly.

(2.) With ferrous sulphate, ammonia throws down a whitish p.p. which at first turns green and ultimately a dull red colour. When dried on the glass slide it closely resembles a drop of dull red sealing wax on which a plain seal has been impressed. It is only translucent here and there, being in transmitted light of bright orange-red colour. With ferric sulphate ammonia throws down at once a foxy red p.p., which forms on evaporation an amorphous translucent glass of much brighter red than that formed by $FeSO_4$.

(3.) When ferric sulphate is dissolved in boiling water several salts seem to be formed. When allowed to evaporate on glass slides my specimens contained: (*a*) needle-like prisms in groups radiating from an opaque amorphous centre; these seem to be No. 34, Part II.; (*b*) hexagonal scaly plates of micaceous habit; these seem to agree with No. 39, Part II.; and (*c*) spherulite rounded disks composed of radiating platy crystals resembling two open fans arranged so as to form a circle. They polarise brilliantly and show radiating stripes of colour. Major axis || to axis of quartz wedge. It is difficult to say whether these disks are No. 34 or No. 39 of Part II., or some other basic salt.

(4.) The ferrous sodium double salt (Part II., No. 41) on first crystallising forms an isotropic structureless glass. After standing for some little time it parts with some of its water and passes into the crystalline condition, forming masses of long prisms radiating from different centres, which polarise brilliantly. After further exposure to the air, and further loss of water, the salt gradually becomes a white opaque mass. This process, which is attended with loss of weight, takes place in a somewhat capricious manner, drops from the same solution evaporated side by side behaving differently. Some take a short cut to the white opaque stage at once; others take as much as from 6 to 10 days to accomplish it. The process may be stopped by mounting in Canada balsam.

(5.) The ferrous and the ferric sulphates treated with ammonium fluoride yield numerous small octahedra, cubes and other forms of the cubic system, the octahedra being by far the most numerous. Interesting cases of penetration twinning frequently occur.

Potassium (K).

A very delicate test is the reaction with the bichloride of platinum ($Pt\,Cl_4$). It is absolutely indispensable that this reagent should be pure, and that its purity should be tested by evaporation on a glass slide and microscopic examination before it is used. The bichloride commonly sold contains potash and yields crystals of chloroplatinate of potash described below. The pure bichloride (that is to say the purest I have been able to obtain) crystallises in long slender prisms like blades of grass. Extinction takes place nearly parallel to the length of the blade. Major axis parallel to *e*. The blades polarise brilliantly, usually in the blue and pink of 2nd order. The reagent must not be kept in glass bottles. I use a gutta-percha one. If a drop or two of this reagent be added to a few drops of the sulphate of potassium (or to a neutral or feebly acid solution of any other K-salt) yellow crystals of chloroplatinate of potash ($K_2Pt\,Cl_6$, solution 1 in 100) are formed. These are either octahedrons or combinations of the octahedron and dodecahedron, or some other form of the cubic system. The complex forms, however, almost always exhibit a six-sided face (a triangle with the corners cut off) of deep orange yellow colour, the other faces of the crystal at various angles to the plane of the glass slide, owing to the high refraction of the mineral, remain dark in transmitted light. See fig. **11**. Sometimes skeleton crystals in trefoil form, or with four arms arranged as a cross, are formed.

An excess of free H_2SO_4 is unfavourable to this reaction.

The salts of ammonium, cæsium and rubidium give with the bichloride of platinum crystals belonging to the cubic system similar in shape to those of $K_2\,Pt\,Cl_6$.

Lithium.

(1.) Phosphate of soda produces a precipitate of phosphate of lithium $Li_3PO_4 + H_2O$, (solution 1 in 2500) which forms in dagger-shaped crystals (two short transverse arms representing the hilt), or in star-shaped crystals composed of four or five rays. These crystals are interspersed among numerous globulites, which give a dark cross between crossed nicols. Most of these globulites are divided into approximately two halves (see *e f*, fig. 32); and when resolved between crossed nicols, in parallel light, the cross opens up into two hyperbolic branches like axial sections of some biaxial crystals in converging polarised light (see fig. 34). The curvature is greatest when the axis of the hyperbola is at right angles to the dividing line (fig. 34). In elongated individuals the result between crossed nicols is an elongated cross with two transverse arms, one on either side of the dividing line. See fig. 35. These elongated twins are very suggestive of the multiplication of organic cells by fission. Indeed the resemblance is so close that one may readily obtain on the same slide illustrations of this supposed fission at every stage. The optical phenomena noted above are very curious, but, pending the completion of some investigations which I am carrying on, I think it better to postpone any attempt to explain them.

The crystals of lithium phosphate possess strong refraction, which shows itself in the dark boundaries of the globulites, and in the strongly wrinkled surface of the crystals. The latter have straight extinction. The addition of acetic acid increases the number of globulites.

(1.) With the reagents mentioned below (see Magnesium, No. 2) crystals isomorphous with those of struvite are obtained. They also exhibit similar optical properties. A little warmth assists the reaction and the phosphate of soda should be added in crystalline form. Manganese salts also yield similar results with these reagents.

Magnesium.

(1.) With ammonia the sulphate yields a gelatinous precipitate which on spontaneous evaporation forms a colourless mass with a silky gloss on it. This is the monoclinic double salt $MgSO_4\ (NH_4)_2\ SO_4 + 6H_2O$ (see Part II.). Prepared on a glass slide this salt usually appears in crystals arranged like the shaft and web of a bird's feather. Mounting in Canada balsam appears to decompose the double salt. In every instance in which I have tried the experiment the monoclinic crystals of the double salt have broken up into needle-shaped orthorhombic prisms, presenting the appearance and optical properties of $MgSO_4 + 7H_2O$.

(2.) Add a *little* ammonium chloride (cubic) to a dilute solution of $MgSO_4$ (orthorhombic) in water. Place a few drops of this mixture on a glass slide. Place a drop of an aqueous solution of sodium phosphate (monoclinic) on the slide near it, and to the latter add one or two drops of ammonia. Then join the two drops. On evaporation struvite crystals will be found on the glass slide. As hemimorphism is common the shapes of the crystals are varied, but those shown at figs. 12 to 15 are often seen. That depicted at fig. 12, usually spoken of as the coffin-shaped form, is especially characteristic. The struvite crystals, $Mg\ (NH_4)\ PO_4 + 6H_2O$ (solution 1 in 15,000), belong to the orthorhombic system. They polarise brilliantly in the colours of the 1st and 2nd order. Major axis *RL* to *e*.

Most writers recommend the gentle warming of the drops prior to mixing, but I have not found it necessary. Behrens adds the phosphate of soda solid. This may be useful in some cases. The reaction requires a little practice.

Crystals similar in shape and optical properties are also produced by the action of the above reagents on the sulphates of lithium and manganese.

(3.) On adding some drops of the aqueous solution of potassium antimonate to the aqueous solution of $MgSO_4$ (the proportion of the latter to the antimonate should not be too great) numerous microscopic crystals of the acid pyro-antimonate of magnesia ($MgH_2Sb_2O_7 + 9H_2O$) are formed on the slide. These occur, for the most part, in six-sided disks lying on their basal planes, but also, not unfrequently, on one of the sides of the hexagonal prism. They also occur in cube-like prisms (sometimes slightly elongated in one direction), and in the form of little flowers, like primroses, with four (occasionally five) uniform petals. The hexagonal disks sometimes pass into globulites, and the latter are doubtless the immature form of the former. Penetration twins are not uncommon and their forms are very varied. Double refraction is not strong and sometimes it is nil. Fock refers the crystals of pyro-antimonate of magnesia to the hexagonal system.

Manganese.

The blowpipe and ordinary chemical tests are the most reliable for the detection of manganese.

(1.) Manganous sulphate (or other manganous neutral or feebly acid salt) treated with oxalic acid yields crystals of oxalate of manganese ($MnC_2O_4 + 3H_2O$) in radiating groups of elongated prisms with straight extinction; but as oxalic acid itself forms similar prisms, and groups of prisms with straight extinction,[1] this reaction is not recommended.

(2.) Ammonia produces a white p.p., which on exposure to the air becomes of dark brown colour (chloride of ammonium prevents this precipitation). On allowing this to evaporate spontaneously on a glass slide, the crust formed is partly transparent and partly opaque. Under the microscope in transmitted light the crust becomes a very beautiful object, and is seen to be made up of fern-like fronds arranged in groups

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[1] Oxalic acid is said to crystallise in the monoclinic system, but the prisms obtained on a glass slide have straight extinction. Double refraction very high. Major axis || to *e*.

on a rich yellow-brown background. The transparent portions (sometimes the fronds are transparent—sometimes opaque) extinguish in a direction oblique to the stems of the fern-like forms.

(3.) With phosphate of soda $MnSO_4$ gives the same results as Li_2SO and $MgSO_4$. For details see Mg, Part III. No. 2.

Sodium (Na).

The blowpipe and flame reactions are principally to be relied on for the detection of soda. Szabo's well known method is a most useful one. The following is the process for microscopic testing which I follow. It is good but a somewhat troublesome method.

(1.) Reduce the mineral to a fine powder in an agate mortar; ignite in platinum crucible; regrind in mortar; ignite; add hydrofluoric acid and stir; heat gently for some time in platinum dish or crucible (agitating the liquid from time to time and adding more HF aq. if necessary) until the powder, or as much of it as is soluble in HF aq., is dissolved. Cool, add water, and then heat again. This operation converts the soda and silica present into sodium fluo-silicate (Na_2SiF_6). Should the mineral operated on not contain silica, pure silica must be added. A glass slide must now be prepared by giving the central portion of it a thin coating of hardened Canada balsam. A few drops of the liquid are then removed by means of a platinum spoon (all the platinum employed must be pure from all surface trace of soda) and transferred to the prepared glass slide. If the liquid does not completely evaporate in 48 hours the surplus can be removed by bringing a strip of filter paper into contact with the outer edge of the drop. When this has been done the crystals left on the slide can be mounted in the usual way.

The most characteristic crystals of sodium fluo-silicate are six-sided prisms; sometimes short and terminated with rather flat pyramids (in which case they much resemble micro-crystals of quartz); sometimes comparatively long and terminated at both ends by flat basal planes. Penetration twins are not uncommon, and other forms also occur. Before mounting the crystals sometimes lie with the apex of the flat terminal pyramid turned towards the eye of the observer. In the process of mounting they get turned over on their sides. Straight extinction. Major axis parallel to length of prism. Double refraction—white of the 1st order.

Crystals of fluo-silicate of soda are isomorphous with those of the fluo-titanate of soda.

Rubidium.

(1.) The reaction of the salts of rubidium with platinum bichloride is the same as that of the salts of potassium, cæsium and ammonium. See potassium.

(2.) For reaction with fluoride of titanium see titanium, No. 2, Part III.

Silica.

The reaction for silica is the same as that for sodium (see *ante*). If the mineral to be tested does not contain soda, some soda must be added. Crystals of fluo-silicate of soda are isomorphous with those of fluo-titanate of soda.

Strontium.

Strontium sulphate dissolves in hot concentrated H_2SO_4 like $BaSO_4$ (see *ante*). On cooling embryo crystals are deposited, the lowest form of which is the micro-globulite. More advanced forms present themselves in the shape of disks, oval crystals, and acicular prisms (often forked) which sometimes arrange themselves in rosettes. The prisms and almond-shaped bodies occasionally develop radiating tufts at one or both ends. The most developed is the arrangement of almond shaped bodies in the form of a short cross with oval arms (see fig. 16) which gradually grows, by the filling up of the re-entering angles, into a four-sided tabular crystal. Not unfrequently we get either a combination of lenticular crystals, or two disks at right angles to each other, as shown at fig. 17. The suppression of one half of one of the disks produces a hat-like form (fig. 18) which is not uncommon. In all these crystals the unit is clearly a globulite. Even the most advanced forms (the four-sided tabular crystals) may sometimes be distinctly seen to be built up of globulites.

The double refraction is feeble, showing only the grey white of the 1st order. Major axis parallel to *e* in oval disks. Straight extinction, viz. parallel to *e* in oval disks. In the cross-like forms extinction is parallel and at *R.L.* to each arm. The more gradual the cooling the more the cross-like forms abound. After mounting in balsam I have obtained comparatively large crystals of celestine (orthorhombic).

Klement and Renard state on the authority of Haushofer that a mixture of equal parts of the sulphates of Sr and Ba dissolved in hot concentrated H_2SO_4 gives on cooling crystals in the form of $SrSO_4$ only; Behrens says the same. My experiments do not confirm this. I have under the above circumstances obtained typical crystals of $BaSO_4$.

Titanium.

(1.) Titanium dioxide dissolved in hydrofluoric acid[1] gives with the salts of potassium monoclinic crystals of fluotitanate of potassium ($K_2TiFl_6 + H_2O$) in rectangular crystals, rhombs, and elongated six-sided prisms. The truncation of the corners of the former give six- and eight-sided outlines. Polarisation feeble. The best results are obtained by placing a crystal of the K-salt in contact with the acid solution. Crystals of fluoride of potassium cannot be mistaken for those of the fluotitanate, as the extinction of the former is straight and the latter decidedly oblique.

(2.) Strong solutions of rubidium chloride (cubic) and cæsium chloride (cubic) both give similar reactions.

(3.) The fluotitanate of sodium Na_2TiF_6 (obtained by melting the substance to be analysed in a bead of sodium fluoride, treating with cold water first, and afterwards with hydrofluoric acid, and crystallising on a glass slide) is isomorphous with sodium fluosilicate (see sodium *ante*). This reaction, therefore, is of no use when silica is present.

Zirconium.

(1.) Two or three drops of an aqueous solution of potassium binoxalate added to a solution of zirconium sulphate on a glass slide yield, on evaporation, crystals probably tetragonal (Behrens); they are in: (*a*) rectangular and six-sided prisms, principally the former, lying apparently on their basal plane with the apex of a pyramid turned towards the eye of the observer; (*b*) crystals showing a six-sided face parallel to the plane of the glass slide and bounded by faces that remain black in transmitted light. These crystals present a *general* resemblance to those depicted at fig. 11, but the arrangement of the side faces is not quite the same. (*a*) Remain dark between crossed nicols. (*b*) The central portion polarises and exhibits a limpid gentian blue of very beautiful tone; at the margin of the drop, where the crystals are massive, the blue passes through violet into red. None of the above crystals will bear mounting in Canada balsam.

(2.) Zircon (and other varieties of the silicate of zirconium) is decom-

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[1] When HF aq., or a fluoride, is evaporated on a glass slide the glass must be protected by a thin coat of hardened Canada balsam, as otherwise Ca, K, and other bases, would be extracted from the glass.

posed by fusion[1] with about five times its weight of pure anhydrous sodium carbonate. A little hydrous sodium carbonate (pure) should be added when fusion is nearly complete. After fusion treat the mass with cold water for the removal of soluble silica; dissolve the insoluble residue with hydrochloric acid; evaporate to dryness; dissolve in water acidulated with hydrochloric acid; the solution contains nearly pure zirconium chloride. Oxalic acid throws down zirconium oxalate. The crystals I have obtained in this way appear, like those described under No. 1, to belong to the tetragonal system. They present rectangular outlines with pyramids pointing to the eye of the observer truncated by a plane, the outline of which is a rhomb, the diagonals of which bisect the faces of the rectangle. Two of the edges of the pyramids are sometimes truncated towards their apex by planes which are apparently the pyramids of the 2nd order, and these planes also truncate the acute angles of the rhomb producing a six-sided outline. The rhomb alluded to polarises in the whitish yellow of the 1st order. Major axis parallel to a line bisecting the acute angles of the terminal rhomb. Extinction parallel to the sides of the rectangle.

List of Illustrations.

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[1] Behrens recommends fusion with sodium fluoride and evaporating down with sulphuric acid.