Some new data concerning kornerupine and its chemistry.

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SOME years ago, when working through large parcels of mixed faceted gem-stones in a search for blue spinels[1] of high refractive index, several unusual specimens were encountered, two of which were later identified as kornerupine by Dr. G. F. Claringbull, using X-ray methods. The other stones in these parcels were all quite typical products of the Ceylon gem gravels, consisting mainly of zircons, garnets, and spinels. Since kornerupine is so rare a mineral, and had not previously been reported from Ceylon, it was considered worth while to attempt to place the new occurrence beyond doubt by searching through rough gem gravel imported direct from Ceylon. This was made possible by the kindness of Mr. Hans Van Starrex, who sent a consignment weighing several pounds from the Matale district (Anderson and Payne, 1940).

Rapid sorting was carried out by means of a direct-vision prism spectroscope, with which the characteristic absorption bands of the zircons constituting the bulk of the parcel could at once be discerned in the red and orange regions of the spectrum. Kornerupine, on the other hand, has rather indistinct absorption bands in the blue and violet, as detailed later. By great good fortune a piece of this mineral was encountered only an hour after the search was begun, and a second very similar specimen shortly afterwards. No further kornerupines could be found in the remainder of the parcel, and none at all were found in a subsequent consignment of rough gravel. One could wish for the opportunity to visit Ceylon in order to make a more systematic search and

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[1] B. W. Anderson and C. J. Payne, Magnesium-zinc-spinels from Ceylon. Min. Mag., 1937, vol. 24, pp. 547–554.

a more detailed description of the exact localities where the mineral is to be found. The identity of the rough pieces was confirmed by their characteristic pleochroism, flotation in methylene iodide, which they almost match in density, and (when one had been faceted) by optical examination.

In addition to these undoubted Sinhalese kornerupines, other Ceylon-cut specimens of the mineral of rather different type have been encountered on several occasions, which are unusual in being optically pseudo-uniaxial. In colour they are very similar to the stones mentioned above, being brownish-green or dull leaf-green. So far as their appearance goes in the cut state they would pass amongst dealers as chrysoberyl, green zircon, or tourmaline. Though Ceylon is also probably the locality

TABLE I. Optical properties and specific gravities of some gem kornerupines.
(B. W. A. and C. J. P.)

	Source.	Colour.	Carats.	D_4^{18}.	Ref. ind. (Na) (± 0.002).	2V.	Pleochroism.
1.	[Madagascar?]	pale sea-green	1·56	3·31 ± 0.01	α 1·665 β 1·677 γ 1·678	$16\frac{1}{4}°$ $r > v$	distinct
2.	Ceylon (rough)	brownish-green	2·15	3·342 ± 0.003	—	—	—
3.	Ceylon	brownish-green	0·55	3·354 ± 0.003	α 1·671 γ 1·684	25° $r < v$ weak	α pale brown β yellow γ dark green
4.*	Ceylon	brownish-green	0·91	3·335 ± 0.005	α 1·669 β 1·681 γ 1·682	$20\frac{1}{4}°$ $r < v$ weak	α green β pale brownish-yellow γ pale brownish-green
5.	Ceylon	brownish-green	1·00	3·330 ± 0.003	—	—	—
6.	Ceylon	brownish-green	2·52	3·350 ± 0.003	α 1·671 β 1·683 γ 1·684	21°	—
7.	[Ceylon?]	brownish-green	9·182	3·325 ± 0.005	α 1·666 β 1·678 γ 1·678	5°	α pale brown β dark green γ brown
8.*	[Ceylon?]	brownish-green	0·734	3·330 ± 0.005	α 1·668 β 1·681 γ 1·681	8°	—
9.*	[Ceylon?]	leaf-green	3·50	3·330 ± 0.003	α 1·669 β 1·681 γ 1·681	3°	—
10.	[Ceylon?]	leaf-green	1·48	3·330 ± 0.003	α 1·669 β 1·681 γ 1·681	—	—

* Analysed, see table II.

of these pseudo-uniaxial stones it cannot yet be asserted with complete certainty. The largest known cut kornerupine (9·182 carats) belongs to this type, and was in the British Museum collection (B.M.75946) as 'idocrase' and later as 'enstatite' before its true identity was realized. Another sizable specimen was purchased from a jeweller in York, and weighed 6·74 carats before part of the stone was slit off for micro-analysis—the re-cut stone weighing 3·50 carats.

The properties of all such specimens so far examined are given in table I, together with those of a specimen of Malagasy type rescued from a jeweller's junk-box by Major H. J. Beadnell in 1936. The facets of this particular stone are sufficiently flat, and the orientation such, that determinations of β were possible for different wave-lengths by the minimum deviation method, which gave:

	Li (6708).	Na (5893).	Tl (5350).	Ca (4226).	Estimated dispersion.
β	1·6722	1·6764	1·6798	1·6914	B–G 0·018

Specimens 4, 8, and 9 were used for analysis, and correspond to I, II, and III, respectively, in table II. Refractive indices were measured in sodium-light on the Tully, Rayner, and Special Rayner (spinel and diamond) direct-reading refractometers, and densities were determined either by exact suspension in Clerici solution, followed by refractive index measurement of the solution in a hollow prism, or by hydrostatic weighing in ethylene dibromide.[1]

Specimen 7 revealed several absorption bands which were measured with a Beck 'wave-length' spectrometer as follows:

α and γ.	β.
5500 very weak.	5400 very weak.
5030 moderately weak.	5030 moderately weak.
4540 very weak.	4630 very weak.
4440 moderately weak.	4460 strong.
4300 very weak.	4300 very weak.

The measurements are in Ångströms, and on account of the diffuse and weak nature of the bands can only be taken as accurate to about ± 20 Å. In none other of the specimens was the absorption spectrum so fully observed, but in all of them the band at 5030 Å. could be seen, and this may therefore have some slight diagnostic value.

The inclusions in these kornerupines show certain similarities. The Sinhalese specimens contain 'feathers' made up of small dark crystals arranged in one plane, and many scattered small zircon crystals were seen, in some cases with pleochroic haloes. In nos. 5, 6, and 9 there are numerous short rod-like inclusions parallel to one direction, which are

[1] B. W. Anderson, Gemmologist, London, 1940, vol. 9, p. 141. [M.A. 8–67.]

of similar refractive index to the parent stone, while in nos. 7 and 8 relatively large inclusions of mica were found.

Very shortly after this new occurrence of kornerupine had been established, J. E. de Villiers (1940) published a description and analysis of kornerupine from another new locality, Port Shepstone, Natal; his material was much richer in iron than any previously analysed.

These new occurrences of a mineral, the composition of which was known to be variable but of which only two satisfactory analyses had been made, prompted an optical, X-ray, and chemical study of all available material. We have studied four specimens in detail, three of them cut gems from Ceylon (two of the unusual pseudo-uniaxial type and one optically normal) and one a prismatine from Waldheim, Saxony, kindly supplied by Prof. C. E. Tilley for this work, and have obtained additional data on the Madagascar and Natal material. We were unable to obtain suitable material from the type locality, Fiskernæs, Greenland, for a detailed examination.

Chemical and X-ray data.

The presence of boron in kornerupine was established by A. Lacroix and A. de Gramont (1921), and invalidates the four earlier analyses, while B. Gossner and F. Mussgnug (1928) appear to have overlooked Lacroix's work, and omitted to examine their material for boron; accordingly, only two trustworthy analyses of kornerupine were available when this work was begun, and it soon became clear that even these were imperfect, the ferrous iron determinations being unreliable; fortunately, specimens presented by Lacroix and by de Villiers were available in the British Museum collections, and it was possible to complete these two analyses by new ferrous iron determinations.

The chemical analyses proved unusually difficult; they were necessarily on a micro-scale, as the weights of the three gem specimens were all near 0·1 gram, and the yield of pure prismatine from the Waldheim specimen was of the same order after crushing, hand-picking, and purification with heavy liquids (bromoform and Clerici solution) and the electromagnet.

The most serious difficulties arose from the presence of boron; this had to be determined and it had to be eliminated before the main analysis, both troublesome operations on the micro-scale. For the boron determination, the method of E. Schulek and G. Vastagh[1] was slightly modified: it was found preferable to avoid transferring the sodium carbonate fusion to the distillation flask with water; instead, the fusion was carried out in a micro-crucible, and the crucible, fusion cake, and lid dropped into the flask[2] without any attempt to detach the fusion cake.

[1] E. Schulek and G. Vastagh, Zeits. Anal. Chem., 1931, vol. 84, p. 167; 1932, vol. 87, p. 165.

[2] Some difficulty was experienced in obtaining a distillation apparatus with

Further, it was found that with some specimens of methyl alcohol there is considerable reduction of the sulphuric acid during the second distillation, and the sulphur dioxide evolved may be enough to render the distillate acidic and occasion serious losses; the addition of a little phenol red (which, unlike phenolphthalein, does not fade on standing in strongly alkaline solution) to the alkali in the receiver enables a check to be kept on its alkalinity. Finally, it was not found possible to obtain anything like the precision claimed by Schulek and Vastagh in the titration of small quantities of boric acid with 0·01 N alkali using methyl red and phenolphthalein as indicators; phenolphthalein has too long a transition interval, and even when a reference solution is used the end point is not sharp enough; much greater accuracy was obtained by using a 1 : 1 mixture of bromthymol blue and bromcresol purple as indicator both for the neutralization before addition of mannitol and for the titration proper (the indicator used has a transition range of pH 6·0 to 6·8; this is high enough to be fully sensitive to mannito-boric acid and low enough to be insensitive to free boric acid); but even with this an accuracy of $\pm 0·10$ c.c. of 0·01 N alkali, equivalent to 0·035 mg. B_2O_3, could not be consistently surpassed in titrations of 10 c.c. of a borax solution containing 40 mg. B_2O_3 per litre.[1] This accuracy corresponds to a probable error of $\pm 0·35$ %, or roughly 10 % of the amount present, due to the titration error alone in the determination of the boron in a 10 mg. portion of kornerupine. Further experiments were made with a boron-free rock plus small amounts of added borax, taken through the whole procedure, and showed that the losses in the rest of the process are negligible. The results of the boron determinations (carried out in duplicate, with generally good agreement) are accordingly regarded as reliable within at least $\pm 0·5$ %, or about 15–20 % of the amount present.

Owing to the presence of boron, the sodium carbonate fusion for the main analysis had to be treated with methyl alcohol and acid to volatilize the boron before the dehydration of the silica; this raised unexpected difficulties. On the micro-scale, dehydration of the silica by double evaporation with hydrochloric acid, following the usual macro-procedure, has several disadvantages; in particular, the silica from the two evaporations has to be weighed separately, with a double weighing error, and the weighing error in micro-analysis is too large to neglect. An attempt was made to use perchloric acid for the dehydration, after expulsion of the boron with methyl alcohol and hydrochloric acid, but traces of organic matter left from the methyl alcohol reduced the perchloric acid and led to solution of notable amounts of platinum from the crucible. Good results were finally obtained by expelling the boron with methyl alcohol and sulphuric acid, and dehydrating with sulphuric acid. The crude silica carries very considerable amounts of basic sulphates of alumina and iron, and needs thorough ignition for their complete decomposition; if hydrofluoric and sulphuric acids were used for the volatilization of the silica, vigorous ignition would again be needed to decompose the residual sulphates, with risk of loss of weight of the crucible by volatilization of platinum; this is avoided by the use of hydrofluoric and oxalic acids to volatilize the silica. The residue after expulsion of the silica was dissolved by a bisulphate fusion and incorporated with the main solution.

ground joints, made entirely in soda-glass; the resistance glasses commonly employed for this class of apparatus are borosilicate glasses and would not be safe.

[1] The alkali was standardized at a strength of 0·05 N against borax, and then diluted to 0·01 N.

No difficulties were encountered in the main analysis after the determination of the silica. Iron and aluminium were precipitated together by 8-hydroxyquinoline in acetic acid solution at pH 5 to 6, and after weighing the precipitate was dissolved in hydrochloric acid and iron precipitated with cupferron.[1] Magnesium was precipitated in the filtrate from the iron and alumina by the addition of further 8-hydroxyquinoline and excess ammonia, and the filtrate from the magnesium was examined for lime. The magnesia-bearing filtrate in one analysis (II) was rendered useless by the failure of a filter-pump, and as no more material was available, magnesia is reported by difference for this specimen.

Alkalis were determined on a separate portion by the Lawrence Smith method in the normal way, except that, since thorough mixing is difficult, instead of using ammonium chloride to form the necessary calcium chloride as flux, a mixture of calcium chloride and calcium carbonate was used, prepared by adding hydrochloric acid to calcium carbonate moistened with alcohol and drying the mixture on the sand-bath.

The determination of ferrous iron proved a particularly difficult problem; as noticed by J. Uhlig (1910) and by C. J. van der Walt (in J. E. de Villiers, 1940), kornerupine is only slowly attacked by hydrofluoric and dilute sulphuric acids. A variety of methods designed to avoid air oxidation during a prolonged acid attack were tried without success, but a modification of the macro-method of H. P. Rowledge was successfully adapted to the micro-scale, and is described fully in a separate communication.[2] With this method, the following results were obtained with the four specimens selected for analysis: I, 4·8 (also 3·1, probably low); II, 7·0; III, 4·7, 5·3; IV, 9·0, 9·3 %. Further, since previous ferrous iron determinations on this mineral must be suspect, specimens from Madagascar (V) and Natal (VI) were examined: V gave 2·8, 3·0 and VI 12·0, 12·3 % FeO; these results agree closely with the values obtained for the total iron (calculated as FeO) by F. Raoult and by C. J. van der Walt, and indicate that in their analyses the iron should all be reported as ferrous; the figures given under V and VI in table II are Raoult's and van der Walt's analyses modified accordingly. Additional confirmation that the iron in the Natal material is wholly ferrous was obtained by a total iron determination made by the following method on 10 mg., which gave 11·8 % total iron as FeO (probably slightly low): the mineral was fused with sodium carbonate, dissolved in 5 c.c. of dilute hydrochloric acid, reduced with metallic zinc and filtered through an Emich filter-stick into 5 c.c. of 0·005 M (molar) iodine monochloride, the flask washed with 8 c.c. of strong hydrochloric acid, and the solution titrated with 0·001 M potassium iodate, using carbon tetrachloride as indicator.

Special tests were made on specimens V and VI for fluorine, with negative results. Spectroscopic examination of specimens I and II show that no constituent present in significant amounts has been overlooked; there are traces of Ca, Mn, Cr, and P.

In order to avoid any assumptions in the calculation of the unit-cell contents X-ray measurements were made on all four analysed specimens,

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[1] For this filtration, and for that of the silica and all the filtrations in the Lawrence Smith procedure, the filter-stick devised by Miss C. C. Miller (Journ. Chem. Soc. London, 1939, p. 1962) was found eminently satisfactory.

[2] M. H. Hey, this vol., p. 116.

TABLE II. Chemical analyses of kornerupine. (M. H. H.)

			I.	II.	III.	IV.	V.	VI.
SiO_2...	...	...	30·5	30·1	29·3	30·5	31·09	29·53
B_2O_3	...	...	2·8	3·6	2·5	3·0	3·59	3·5
Al_2O_3	...	...	37·6	42·9	40·0	35·8	38·17	40·97
Fe_2O_3	...	...	3·3	nil	nil	3·2	nil	nil
FeO...	...	...	4·8	7·0	5·0	9·1	2·55	12·22
MgO...	...	...	20·7	[16·4]	21·0	17·6	22·51	10·90
Na_2O	...	...	trace	n.d.	0·1	1·6	1·36	0·70
K_2O...	...	...	trace	n.d.	0·6	trace	0·08	—
Total	...	...	99·9*	[100·0]	98·5	100·8	99·96†	99·13§
D_4^{18} ...	...	...	{ 3·335	3·330	3·330	3·370	3·27‡	3·449‖
			±0·005	±0·005	±0·003	±0·010		

* Including ignition loss 0·2.   † Including CaO 0·51, H_2O 0·10.   ‡ This value may be low; a determination on B.M. 1920,424 gave D_4^{18} 3·290 (B. W. A.). § Including MnO 0·08, TiO_2 0·44, P_2O_5 0·12, H_2O 0·67.   ‖ J. E. de Villiers's value, corrected for 0·84 % ilmenite and 0·67 % H_2O.

I. Ceylon, normal type. Optics, table I, no. 4.

II. Ceylon, optically pseudo-uniaxial type. Optics, table I, no. 8.

III. Ceylon, optically pseudo-uniaxial type. Optics, table I, no. 9.

IV. 'Prismatine', Waldheim, Saxony. B.M. 1940,21. α 1·675, β and γ 1·687 ±0·004 (Na).

V. Itrongay, Madagascar. Analysis by F. Raoult, in A. Lacroix (1922), corrected in accordance with a new FeO determination on B.M. 1920,424 (a specimen presented by A. Lacroix). α 1·6650, β 1·6766, γ 1·6770, 2V 19½°.

VI. Port Shepstone, Natal. Analysis by C. J. van der Walt, in J. E. de Villiers (1940), corrected in accordance with a new FeO and total iron determination on B.M. 1940,39 (a specimen presented by J. E. de Villiers). α 1·682, β 1·696, γ 1·699, 2V 48°.

TABLE III. Empirical unit-cell contents of kornerupine (Analyses I–VI).

			I.	II.	III.	IV.	V.	VI.
Si	...	...	14·9	14·7	14·6	15·0	15·00	15·36
B	...	...	2·4	3·0	2·1	2·5	2·98	3·14
Al	...	...	21·8	24·8	23·5	20·8	21·68	25·12
Fe''' ...	...	...	1·2	—	—	1·2	—	—
Fe'' ...	...	...	2·0	2·9	2·1	3·8	1·03	5·13
Mg	...	...	15·1	[11·9]	15·5	12·9	16·14	8·46
Na	...	...	trace	—	0·1	1·5	1·26§	0·70‖
K	...	...	trace	—	0·4	—	0·05	—
O	...	...	85·0	86·0	85·4	84·2	85·07	87·10
ΣR*...	...	...	40·1	39·6	41·6	40·2	40·42	39·44
Si+B	...	...	17·3	17·7	16·7	17·5	17·98	18·50
$\Sigma(R, S)$†	...	...	57·4	57·3	58·3	57·7	58·40	57·94
F‡ ...	...	...	{ 29·40	29·34	29·82	29·56	28·94¶	31·30
			±0·29	±0·29	±0·28	±0·34	±0·4	±0·4

* Sum of all atoms except Si, B, and O.   † Sum of all atoms except O. ‡ Conversion factor, $VD/1·649S$ (see M. H. Hey, Min. Mag., 1939, vol. 25, p. 403).   § Also Ca 0·26.   ‖ Also Mn 0·03.   ¶ This value and all the above cell-contents may be low by 0·7 %, see footnote to table II.

and also the British Museum specimens from Madagascar and Natal (V and VI); no variation in the cell-sides could be detected. The dimensions found, a 13·68±0·04, b 15·95±0·04, c 6·68±0·02 Å., are all smaller than those (a 13·86, b 16·02, c 6·78) found by B. Gossner and F. Mussgnug (1928); while it is possible that the latter are correct for the unusually alkali-rich material examined by them it appears more probable that their results were less accurate than they supposed, and that the unit-cell dimensions of kornerupine do not show appreciable variations. Gossner and Mussgnug found that the unit-cell of kornerupine has one face only centred, namely (001), but did not determine the space-group;[1] Dr. G. F. Claringbull will continue his X-ray work, and expects to determine the space-group at least.

When the empirical unit-cell contents (table III) were calculated from the six available complete analyses and their correlated X-ray and specific gravity measurements, it was clear that the data show regularities which J. E. de Villiers (1940) could not discover for lack of reliable ferrous iron determinations. The average oxygen per unit cell from the data in table III is 85·4, with a mean departure from the mean of 0·6; the corresponding figures for (Si+B) are 17·6 and 0·4; for ΣR, the sum of all atoms other than O, Si, and B, 40·2 and 0·5; and for $\Sigma(R, S)$, the sum of all atoms other than O, 57·8 and 0·4. The tendency of ΣR to 40 and of $\Sigma(R, S)$ to 58, implying (Si+B) 18, are clear, but it is not clear whether the oxygen content approaches 85 or 86; the assessed probable error of 0·9 to 1·2 % in the conversion factor, F, arising from errors in the physical determinations would allow of either.

That the true oxygen content is probably 86 is indicated by a consideration of the probable errors of the chemical analyses. The general effects of analytical errors have been discussed by one of the authors;[2] some account of the extension of these general investigations to a particular case may be useful. The first step is to consider the maximum probable errors in the oxygen content and in ΣR, $\Sigma(R, S)$, and (Si+B). We may probably assume that no single oxide reported in the analysis is in error by more than 1 % (i. e. 1 % of the sum, not of the amount of that oxide present), and there is evidence that no essential constituent present in any significant amount has been overlooked; water has not been determined in analyses II, III, and IV, for lack of material. As water is regarded as an impurity (see below), its only effect is to depress
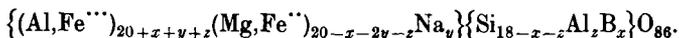
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[1] J. Uhlig (1910) found that the symmetry of etch-figures on prismatine crystals agrees with the requirements of the didigonal equatorial class, D_{2h}.

[2] M. H. Hey, Min. Mag., 1939, vol. 25, p. 402.

the density and hence the observed value of the conversion factor F by $\frac{2}{3}$ % for every 1 % of water present; there is no reason to suppose the 0·2 % H_2O determined in analysis I is seriously low (0·0 to 0·7 % are probable limits in this case), and the high summation of analysis IV suggests that there is little water here; but analysis III has a low sum, and up to 2 % of water might be present and would involve an increase in F of 1·3 %. A consideration of the analytical procedure suggests that any error in B, Si, or Fe is likely to be a loss, and in Mg an excess; Al is probably one of the most accurate values in the analysis, and like Na, is as likely to be high as low. Taking these facts into consideration, and allowing an error up to 1 % in each constituent, it may readily be shown that the true oxygen content is very unlikely to be lower than the 'observed' value by more than 0·3 or higher by more than 0·7 in consequence of analytical errors in constituents other than water; the corresponding figures for ΣR are 1·1 and 0·2, for $\Sigma(R, S)$ 0·5 and 0·6, and for (Si+B) 0·5 and 1·1. Combining these results with the maximum probable errors in F due to non-determination of water and to errors in the physical determinations, the maximum probable errors in cell-contents as determined from the four new analyses may be calculated, with the results shown in table IV. These figures show that the analytical results are compatible with a general formula

$$(\text{Al},\text{Mg},\text{Fe}^{\cdot\cdot},\text{Fe}^{\cdot\cdot\cdot},\text{Na})_{40}(\text{Si},\text{B})_{18}\text{O}_{86},$$
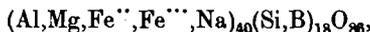
with perhaps a small replacement of Si by Al; this may be expanded to

$$\{(\text{Al},\text{Fe}^{\cdot\cdot\cdot})_{20+x+y+z}(\text{Mg},\text{Fe}^{\cdot\cdot})_{20-x-2y-z}\text{Na}_y\}\{\text{Si}_{18-x-z}\text{Al}_z\text{B}_x\}\text{O}_{86}.$$

Any division of the $40\Sigma R$ must await a determination of the space-group. B. Gossner and F. Mussgnug (1928) found that the Bravais

TABLE IV. Minimum and maximum probable values of the oxygen and other contents of the unit cell of kornerupine.

			I.	II.	III.	IV.
O	...	... { minimum	83·9	84·9	84·3	82·9
		maximum	86·5	87·5	86·9	86·0
ΣR	...	... { minimum	38·6	38·1	40·1	38·7
		maximum	40·7	40·2	42·2	40·8
(Si+B)	...	{ minimum	16·6	17·0	16·0	16·8
		maximum	18·6	19·0	18·0	18·8
$\Sigma(R, S)$	...	{ minimum	56·3	56·2	57·2	56·5
		maximum	58·6	58·5	59·5	59·0

lattice was the end-centred Γ'_o; if this is correct, then unless there are vacancies in the oxygen lattice, the space-group must be D_{2h}^{19},

the only one having this lattice which possesses the necessary two-fold positions to accommodate a number of oxygen atoms which is not a multiple of four.

It is of interest to consider the five available analyses in which the boron content was overlooked. When an analysis of a boron-bearing mineral is carried out by the usual methods, the presence of boron not being recognized, three things may happen to the boron: a part is usually lost by volatilization during the dehydration of the silica; a part is weighed with, volatilized with, and counted as silica; and the remainder is weighed with and counted as alumina; in other respects the analysis should be of normal accuracy. Now the percentage of boron may be taken as between 2·2 and 3·8 %; its non-estimation will affect the 'apparent' number of oxygen atoms per unit cell, obtained by accepting the analysis as correct, differently according as it is lost, weighed with the alumina, or weighed with the silica, always giving a low 'apparent' value, but lowest when the boron is lost and highest when it is weighed with the silica. A short calculation shows that the true value for the oxygen atoms per unit cell must be between 0·6 and 1·5 higher than the apparent value. Similarly, the effect of the neglect to determine the boron on ΣR, $\Sigma(R, S)$, and $(Si + B)$ may be calculated; the 'apparent' value of ΣR will be the same as the true value if the boron is all weighed as silica, but if it is lost ΣR will be high, and if weighed as alumina higher, by as much as 2·2; $(Si + B)$ and $\Sigma(R, S)$ will be low, the former by from 1·3 to 3·2, the latter by from 0·8 to 1·4.

Applying these considerations to the five incomplete analyses, the atomic contents per 1458 $Å^3$ were calculated from the analytical figures and densities (table V); these values will be the actual unit-cell contents if the cell-dimensions are really invariable within the accuracy of working, as is probably the case. If the cell-sides found by B. Gossner and F. Mussgnug are adopted for the material they analysed (X), values 3·5 % higher result (Xa).

It is clear that the four early analyses, VII, VIII, IX, and XI are in agreement with the above repeat formula when account is taken of the neglected boron. For if the percentage of boron is between 2·2 and 3·8 %, the 'apparent' number of oxygen atoms per 1458 $Å^3$ should be between 84·5 ± 1·0 and 85·4 ± 1·0 to comply with the above repeat formula (the ± 1·0 represents the probable error in the conversion factor, F, assuming that the densities given in table IV are accurate to ± 0·01), while ΣR should be between 42·2 ± 0·5 and 40·0 ± 0·5. The 'observed' values of ΣR suggest that the boron was probably mainly caught with the alumina, which is in accord with the usual experience of analysts; assuming that 3·2 % boron was present and was wholly caught with the alumina, the oxygen contents given under $O_{prob.}$ in table V were calculated; these values are regarded as the results of the most probable interpretation of the analyses, and will be seen to agree well with the proposed repeat formula.

Analysis X (B. Gossner and F. Mussgnug, 1928) offers more difficulties, for if Gossner's cell-dimensions are accepted, the oxygen content is reasonable but unless the boron content is abnormally low (not above 1·3 atoms B per unit cell) the sum $(Si + B)$ will exceed 18, which seems improbable; and if it is assumed that the cell-dimensions of kornerupine do not vary appreciably, as seems more probable, the 'observed' number of oxygen atoms falls below the minimum probable figure of 83·5, and it appears that the bulk of the boron must have been weighed with the silica, which is unusual. Gossner and Mussgnug mention the presence of quartz, tourmaline, mica, and rutile (yet TiO_2 does not appear in the analysis) in their

material in appreciable amounts, and it is possible that their density may be low, but a definite interpretation of their results has not been possible.

TABLE V. Interpretation of some incomplete analyses of kornerupine.

Chemical data and atomic contents per 1458Å³, indicating the probable fate of the neglected boron.

	VII.	VIII.	IX.	X and Xa.		XI.
SiO_2...	30·90	30·89	30·61	33·20		31·35
Al_2O_3	46·79	43·06	42·11	42·36		41·20
FeO*	1·82	6·28	6·50	6·08		2·04
MgO	19·46	15·08	15·23	11·76		23·80
Na_2O	—	2·04	1·85	5·07		0·60
K_2O...	—	0·79	0·85	0·83		0·24
H_2O...	1·30	1·36	2·08	1·60		0·64
Total	100·27	99·50	99·53†	100·90		99·87
$D_{obs.}$	3·23‡	3·341	3·345	3·28		3·27
$D_{corr.}$§	3·26	3·371	3·383	3·31		3·28
Si	15·0	15·6	15·7	16·3	16·9	15·3
Al	26·7	25·7	25·5	24·5	25·3	23·6
Fe··	0·7	2·7	2·7	2·5	2·6	0·8
Mg	14·0	11·4	11·6	8·6	8·9	17·2
Na	—	2·0	1·8	4·8	4·9	0·6
K	—	0·5	0·6	0·5	0·5	0·2
O	84·8	85·0	85·2	83·2	86·1	84·3
ΣR	41·4	42·3	42·2	40·9	42·3	42·4
Boron mainly ...	caught with Al_2O_3	caught with Al_2O_3	caught with Al_2O_3	caught with SiO_2	caught with Al_2O_3	caught with Al_2O_3
$O_{prob.}$	86·0	86·4	86·5	84·2	87·4	85·6

* Total iron calculated as FeO; summations adjusted accordingly. † Including TiO_2 0·30. ‡ N. V. Ussing (1889) found for Fiskernæs material, D 3·273. § Densities corrected for water (assumed to be an impurity) and in anal. IX for TiO_2 (taken as ilmenite).

VII. Fiskernæs, Greenland. J. Lorenzen, 1884.

VIII. 'Prismatine', Waldheim, Saxony. A. Sauer, 1886.

IX. 'Prismatine', Waldheim, Saxony. J. Uhlig, 1910. α 1·671, β 1·683, γ 1·684, 2V 29°.

X. 'Prismatine', Waldheim, Saxony. B. Gossner and F. Mussgnug, 1928.

Xa. Analysis X, calculated to empirical unit-cell contents, using Gossner and Mussgnug's cell-dimensions.

XI. Betroka, Madagascar. A. Lacroix, 1912 (F. Pisani, analyst). α 1·6613, β 1·6733, γ 1·6742, 2V 20°.

In all the above discussion of the repeat formula of kornerupine, it has been assumed that the water is an unessential impurity. Several workers have regarded it as an essential constituent, since it is only lost at a fairly high temperature. If it is accounted essential, the number of oxygen atoms per unit cell will be increased (the increase will be $0·2x$ if

$x \%$ of water is present), while the number of all other atoms will be decreased (by $1 \cdot 7x \%$ for $x \%$ of water). The data are not sufficient to decide whether the water is essential or not; if it is the repeat formula must be modified to:

$$\{(Al, Fe^{\cdots})_{20+x+y+z-w}(Mg, Fe^{\cdot\cdot})_{20-x-2y-z+w}Na_y\}$$
$$\{Si_{18-x-z}Al_zB_x\}\{O_{86-w}(OH)_w\}.$$
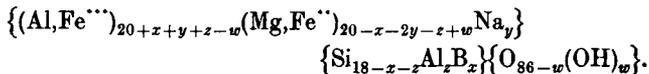
Summary.

The occurrence of kornerupine in the gem gravels of Ceylon has been established, and specimens are described together with examples of an optically pseudo-uniaxial variety of the mineral. Micro-chemical analyses have been carried out on the new material and on 'prismatine' from Saxony, and previously published analyses are reconsidered in the light of new knowledge. The repeat formula of kornerupine approximates to $Al_{20}Mg_{20}Si_{18}O_{86}$, with considerable replacements of Al and Mg by Fe and Na, and of Si by B and perhaps Al. The boron content is fairly constant, but the iron varies widely. The refractive indices, birefringence, and density increase with the iron content. The methods of chemical analysis are described, and include modifications in the micro-determination of boron.

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