

COMPOSITION AND PROPERTIES OF SOME NEW ZEALAND GLAUCONITES

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ABSTRACT

Seven new analyses of pure glauconites separated from sediments of Upper Cretaceous and Tertiary age, have been analyzed and their optical data determined. From this it has been possible to construct curves showing the variation of α , γ and $\gamma-\alpha$, with increasing content of Fe_2O_3 . Finally it is shown that the analyses fit the structural formula derived for glauconite by Gruner.

During an investigation of the distribution and quantity of greensands of the Upper Cretaceous and Tertiary formations in New Zealand, a considerable number of samples was collected with a view to a mineralogical study of glauconite. Of the specimens collected, seven have been used in the present study.

The samples were crushed and passed through a 60-mesh sieve, and then concentrated to some extent by shaking on a 100-mesh; material having a grain-size greater than 60-mesh or less than 100-mesh was rejected as too impure. The glauconite in the sand of this grade was then further concentrated by electro-magnetic treatment and subsequently purified by flotation in bromoform or bromoform-benzene mixtures. Clerici solution was not used in preparing any samples of glauconite for analysis, for Gruner (1935, p. 704) has found experimentally that in the case of a sample of glauconite boiled in thallium nitrate solution for four hours, about 25% of the K ions were replaced by thallium ions, with resultant increase in the specific gravity from 2.81 to 3.02. The behavior of glauconite in Clerici solution is similar to that of the stilpnomelane minerals, for Holzner (1933) found that thallium was absorbed into the structure to such an extent that analysis showed 9.83% Tl_2O , while K_2O was reduced from 2.20 to 0.29%. Centrifuging was necessary in two cases, while one specimen of glauconitic greensand, rich in coarse biotite, was separated from the latter constituent by the simple procedure of vibration on cardboard, the mica remaining on the board and the glauconite rolling off. Microscopic inspection was carried out before chemical analysis was undertaken.

The glauconite content of the samples ranges between wide limits, from about 10% in *P.* 4640,* a glauconitic limestone from Milburn

* Numbers *P.* 4640 etc. refer to specimens in the mineral and rock collections of the New Zealand Geological Survey, Wellington.

Quarry, Otago, to about 90% in *P.* 4895, a greensand from Makerewau Creek, Mangatu Survey District. The other samples used in this study were glauconitic sandstones and siltstones, containing approximately 40–60% of glauconite. This group is typical of many of the greensand deposits of New Zealand, which in places reach a thickness possibly in excess of 200 feet.

The glauconite grains were rounded, spongy, concertina-shaped, or moulded into coprolites in the manner described by Galliher (1935*a* and *b*). The individual grains are made up of microcrystalline aggregates of platelets of glauconite, occasionally with a regular radiate arrangement, though more frequently with some suggestion of optical continuity as

TABLE I. ANALYSES OF PURE GLAUCONITES

	1.	2.	3.	4.	5.	6.	7.
SiO ₂	49.07	52.64	49.29	43.33	48.54	47.42	47.15
Al ₂ O ₃	10.95	5.78	3.17	7.27	7.82	7.19	6.20
Fe ₂ O ₃	15.86	17.88	21.72	24.87	17.50	22.64	21.50
FeO	1.36	3.85	3.19	2.90	3.07	3.39	3.76
TiO ₂	0.15	0.16	0.12	0.20	0.10	0.10	0.14
CaO	0.07	0.12	0.74	0.10	0.68	0.27	0.46
MgO	4.49	3.43	3.85	2.95	3.26	2.28	2.80
Na ₂ O	0.13	0.18	0.12	0.02	0.22	0.05	0.13
K ₂ O	7.51	7.42	6.02	6.00	5.87	7.46	6.98
MnO	tr.(?)	tr.	tr.	tr.	tr.	tr.(?)	tr.(?)
P ₂ O ₅	0.19	0.18	0.32	0.15	0.14	0.22	0.19
Cr ₂ O ₃	0.07	n.d.	n.d.	n.d.	0.03	0.04	0.05
S	0.06	n.d.	n.d.	n.d.	0.05	0.05	0.06
BaO	nt.fd.	n.d.	n.d.	n.d.	nt.fd.	nt.fd.	0.04
ZrO ₂	nt.fd.	n.d.	n.d.	n.d.	nt.fd.	nt.fd.	nt.fd.
H ₂ O+	6.63	5.86	7.21	6.22	6.00	6.07	6.47
H ₂ O—	3.66	2.83	4.60	5.94	6.71	3.01	4.20
	100.20	100.33	100.35	99.95	99.99	100.19	100.13
Sp. Gr.	2.687	2.708	2.580	2.735	2.555	2.790	2.770

1. Lower band, Milburn Lime and Cement Co.'s Quarry, Milburn, Table Hill Survey District, Otago Land District. *P.* 4640.
2. From thin band, Makerewau Creek, Mangatu Survey District, Gisborne Land District. *P.* 4895.
3. Whare Flat, East Taieri Survey District, Otago Land District. *P.* 6034.
4. Abandoned railway cutting, Elephant Hill Survey District, Canterbury Land District. *P.* 5392.
5. Kakaho Creek, Otepopo Survey District, Otago Land District. *P.* 6035.
6. North end of Otepopo railway tunnel, Otepopo Survey District, Otago Land District. *P.* 5621.
7. Northern bridge, Waianakarua, Otepopo Survey District, Otago Land District. *P.* 5618.
A trace of CO₂ was present.

shown by inspection after insertion of a gypsum plate. The diameter of the platelets averages approximately 0.01 mm. though in *P.* 6035, platelets up to 0.05 mm., with an average of 0.02 mm. were observed. Single crystals, however, are exceedingly rare and long and careful searching was necessary to find these, except in the case of *P.* 6035; a preparation of this specimen showed a number of crystal fragments.

It was not found possible to determine the hardness with any accuracy but grinding tests certainly showed considerable differences. The specific gravities of the analyzed specimens ranged from 2.55–2.79.

The complete chemical analyses of seven specimens of glauconite are given in Table I. Inspection of the analyses in this table reveals a noteworthy range in the percentage of SiO_2 , and the figure 52.64% in No. 2 (*P.* 4895) would suggest that a little quartz might have been present as an impurity, though microscopic examination did not reveal this. There is considerable alumina in No. 1, but this is not sufficiently high to warrant comparison with the aluminous glauconite, skolite (Table II, analysis 11), described by Smulikowski (1936). The iron in all examples is mainly in the ferric condition, with ferrous iron not exceeding 3.85%. TiO_2 is persistently low but fairly constant in amount. It should be noted that Allen (1937, p. 1181) observed rutile needles in two glauconites from Monterey Bay, California, thus accounting for 0.59% and 0.30% of TiO_2 found in the analyses. Microscopic examination of the New Zealand glauconites, however, failed to reveal any trace of rutile as a possible source of the titanium.

Lime is consistently low and does not in any example exceed 0.74% but is usually much less than this figure. After allotting sufficient CaO for the P_2O_5 determined in each analysis, slight excess of CaO remains in three cases only, viz. No. 3 (0.35%), No. 5 (0.45%) and No. 7 (0.2%). The mode of occurrence of the tri-calcium phosphate is not at all clear. An experiment was carried out as follows, in order to determine if grains of apatite were associated with the glauconite: the granular glauconite was treated with dilute HNO_3 (2%) and warmed for ten minutes on the water-bath. The liquid was then decanted and the glauconite well washed, crushed and again treated with HNO_3 . The glauconite was filtered off and on test the second filtrate gave a strong phosphate reaction. At present it can only be suggested that the phosphatic solutions percolating through the sediments have, to some extent, intimately penetrated the crystalline glauconite aggregates or granules and deposited films of $\text{Ca}_3(\text{PO}_4)_2$ over the surfaces of the minute platelets. All good analyses of carefully prepared glauconite show a low content of CaO and apparently as Gruner (1935, p. 713) points out, the crystal structure would allow only a fraction of one per cent of calcium to be present.

TABLE II

	8.	9.	10.	11.	12.	13.	14.
SiO ₂	48.66	49.05	55.95	49.09	48.12	50.58	49.47
Al ₂ O ₃	8.46	7.96	11.56	18.17	9.60	6.72	5.59
Fe ₂ O ₃	18.80	19.66	9.99	6.42	19.10	19.50	19.46
FeO	3.98	0.75	2.02	2.56	3.47	2.96	3.36
TiO ₂	nt.dt.*	nt.dt.	nt.dt.*	0.21	nt.dt.	nt.dt.	nt.dt.
CaO	0.62	2.34	3.95	1.03	0.76	0.34	0.60
MgO	3.56	1.17	6.77	3.10	2.36	4.10	3.96
Na ₂ O	nil	0.78	0.61	0.23	0.22	0.04	0.16
K ₂ O	8.31	6.18	4.12	5.62	7.08	8.26	8.04
MnO	0.01	nt.dt.	nt. dt.	tr.	nt.dt.	nt.dt.	nt.dt.
P ₂ O ₅	0.12	nt.dt.	0.18	tr.	nt.dt.	0.27	1.06
CO ₂	nt.dt.	nt.dt.	nt.dt.	nt.dt.	nt.dt.	0.30	0.56
H ₂ O+	4.62	11.79	3.22	13.47	10.06	7.76	8.54
H ₂ O-	1.94						
	99.08	99.68	99.97	99.90	100.77	100.83	100.80

8. St. Joseph Lead Co. Mines, St. Joseph, Mo., U.S.A. G. V. Brown, *analyst* (Ross, 1926, p. 10, Table II, No. 11).

* TiO₂=0.10 determined by R. T. Rolufs (see V. T. Allen, 1937, p. 1181).

9. Huntington, Oregon, E. P. Henderson, *analyst* (Ross, 1926, p. 10, Table II, No. 13). Note that the figures for Al₂O₃, Fe₂O₃, Na₂O and K₂O were probably transposed; they have been corrected by Gruner, 1935, p. 706, Table 3.

10. Firm type, Monterey Bay, California, A. A. Hanks, *analyst* (Gallihier, 1935b, p. 1359).

* TiO₂=0.59% determined by R. T. Rolufs (see V. T. Allen, 1937, p. 1181).

11. Skolite, a new mineral of the glauconite group (K. Smulikowski, 1936).

12. Lewes, Sussex, E. G. Radley, *analyst* (see A. F. Hallimond, 1922, p. 331).

13. Sewell, N. J., Hornerstown marl, glauconite grains separated magnetically from the residues of washed greensand, R. K. Bailey, *analyst* (see G. R. Mansfield, 1922, p. 128).

14. Elmwood Road, N. J.; as in No. 13 above.

The figure for magnesia is very constant and comparison with other analyses substantiates the evidence that it never becomes an important constituent. In all the New Zealand specimens, Na₂O is quite unimportant and the analyses do not provide any further evidence of the existence of a soda-bearing glauconite, which Hallimond (1922, pp. 332-333) suggests may occur. Ross (1926, p. 10, Table II) quotes four analyses of glauconite containing over 1% Na₂O, No. 13 in his table being shown as containing 6.18% Na₂O; it seems reasonable to suppose, however, that in this latter case, the Na₂O and K₂O figures have been transposed (this paper, Table II, analysis 9).

The determination of small amounts of Cr₂O₃ in four of the New Zealand glauconites is of interest, but owing to the failure of most analysts to estimate the quantity of this constituent, no further comment can be made now.

The amount of water evolved at temperatures greater than 105°C. is remarkably constant but there is considerable variation in that given off at temperatures below that figure. Ross (1926, p. 8) has carefully investigated the water content of an analyzed glauconite from Bonnetterre, Mo., U.S.A., at different temperatures, and the dehydration curve thus determined clearly shows a break at 280°C., 3.30% H₂O having been given off below that temperature and 3.26% H₂O above it.

The writers have carried out a series of experiments in order to discover the solubility of glauconite in acids of different concentrations. Granular glauconite, that is uncrushed material, when treated with 1% HCl solution on the water-bath for 15 minutes, was considerably attacked and further, such action appeared to be selective. Quantitative data were obtained by treating a sample of slightly impure glauconite obtained by electro-magnetic separation from the Waianakarua greensands, with different strengths of hydrochloric acid; the solutions so obtained were then analyzed for the main constituents (see Table III).

TABLE III

	3%	6%	12%	24%
SiO ₂	5.64	7.60	8.06	6.00
Al ₂ O ₃	1.58*	2.42*	3.82*	5.12*
Fe ₂ O ₃ } FeO }	7.48	14.28	21.34	27.64
TiO ₂	0.03	0.10	0.14	0.20
CaO	0.68	0.68	0.70	0.70
MgO	0.96	1.24	1.78	2.42
Na ₂ O } K ₂ O }	1.20	2.02	3.60	5.36
	<hr/>	<hr/>	<hr/>	<hr/>
	17.57	28.34	39.44	47.44
Insol. res.	70.90	60.32	49.32	40.94

* Includes any soluble P₂O₅ and Cr₂O₃.

A sufficient quantity of pure Waianakarua glauconite (Table I, No. 7) was available in order to determine its solubility with one concentration of HCl (24%), and the amounts of the main oxides dissolved are given in Table IV.

In all the above tests the finely powdered samples were heated with acid solutions for two hours at 100°C. No attempt was made to dissolve out with alkali from the residue any silica which had gelatinized during the acid treatment. However, it is evident from inspection of Tables III and IV that, especially in the case of the stronger acids, most of the insoluble residues must consist of gelatinous silica.

At this stage it might be pertinent to examine critically a method used by Schneider (1927, pp. 301-302) for the preparation of a sample of pure glauconite. In order to effect a separation of glauconite from a mixture of that mineral and quartz, he suggests digestion with HCl and then NaOH. The colloidal suspension of "glauconite"* is then made slightly acid, coagulation results and the precipitate of "glauconite" is then washed, dried at 110°, powdered and analyzed. Now the writers' work has shown conclusively that dilute HCl selectively attacks glauconite,

TABLE IV. OXIDES DISSOLVED FROM PURE GLAUCONITE

	24% HCl
SiO ₂	7.30
Al ₂ O ₃	5.66
Fe ₂ O ₃ } FeO }	24.80
TiO ₂	0.13
CaO	0.46
MgO	2.66
Na ₂ O	0.08
K ₂ O	6.68
	<hr/>
	47.77
Insol. res.	42.06

dissolving a portion of each oxide present in varying amounts. How Schneider's separation was carried out remains incomprehensible to the present writers. If Schneider uses the term glauconite (1927, first line on p. 302) loosely for a solution of glauconite in HCl, it is still not clear how he was able to estimate K₂O, if this precipitate was washed as he states. Again it is not clear how H₂O could be determined in this preparation, for a temperature of 110° could not dehydrate the gelatinous silica and the R₂O₃ precipitate. The base exchange or zeolitic properties of glauconite are well known and in view of this, it is most surprising that Schneider's "glauconite" prepared in this way, should not have had a considerable amount of the potash replaced by soda.

Shaking tests carried out for periods up to 8 hours bear out the work of Mansfield (1922, pp. 116-118) on the cause of the cloudiness developed in the liquid when grains of glauconite are agitated or stirred in pure water. Pure samples of glauconite granules were shaken with water in a rotary shaker, revolving at 35 revolutions per minute. A period of five minutes was sufficient to produce considerable cloudiness and after a period of one hour, a very dense cloudiness was produced. Shaking was continued for a further two hours and then the fine suspension was de-

* The quotation marks are the present authors'.

canted and the liquid evaporated to dryness on a water-bath. Microscopic inspection proved that this cloudiness was entirely caused by a suspension of finely comminuted glauconite. The glauconite granules were recovered from this first shaking, washed and shaken with water for another period of three hours, with similar results. It appears to the writers that this process could be continued until the granules were reduced to a grain-size where the cushioning effect of the water would prevent mutual attrition.

OPTICAL PROPERTIES OF GLAUCONITE

Because the glauconite granules are composed of aggregates of very minute platelets determination of the optical properties was not easily accomplished. All the optical data that could be obtained for the seven analyzed glauconites are given in Table V.

The α and γ refractive indices were determined in sodium light but owing to the nature of the material dealt with, it is stressed that the accuracy will be of the order of ± 0.005 . Further, in view of the small optic axial angle and the fineness of the material it was impossible to measure β as distinct from γ . The figures given for the optic axial angle, $2V$, likewise are not more accurate than $\pm 5^\circ$. In all examples a fair cleavage parallel to the γ -vibration direction was observed; extinction appeared to be straight with reference to this direction.

TABLE V. OPTICAL PROPERTIES OF GLAUCONITES.

	1.	2.	3.	4.	5.	6.	7.
$\alpha =$	1.601	1.602	1.592	1.610	1.600	1.602	1.604
$\gamma =$	1.615	1.618	1.614	1.634	1.621	1.627	1.624
$\gamma - \alpha =$	0.014	0.016	0.022	0.024	0.021	0.025	0.020
$2V =$	10°	$10-20^\circ$	10°	10°	13°	12°	$10-20^\circ$
Opt. sign:	neg.	neg.	neg.	neg.	neg.	neg.	neg.
Pleochroism, α :	yellowish-green	bright green	yellowish-brown	yellowish-green	pale yellowish-green	yellowish-green	very pale green
γ :	deeper yellowish-green	bright bluish-green	olive green	bright green	deep green	olive green	olive green
Absorption:	$\gamma > \alpha$	$\gamma > \alpha$	$\gamma > \alpha$	$\gamma > \alpha$	$\gamma > \alpha$	$\gamma > \alpha$	$\gamma > \alpha$
Elongation:	positive	positive	positive	positive	positive	positive	positive

The optical properties correspond closely with the data obtained by Ross (1926, pp. 3-4) and attempts to correlate the Fe_2O_3 content with the

refractive index data have been moderately successful (Fig. 1). It will be noticed that the refractive indices of specimen No. 3 are somewhat different from the other values plotted, though the analysis reveals no obvious cause for this discrepancy. Except for No. 3, the γ values all lie in a fair position but this happy state was not attained so completely for the α values. It is suggested that the determination of γ is more readily and accurately carried out than the determination of α , on

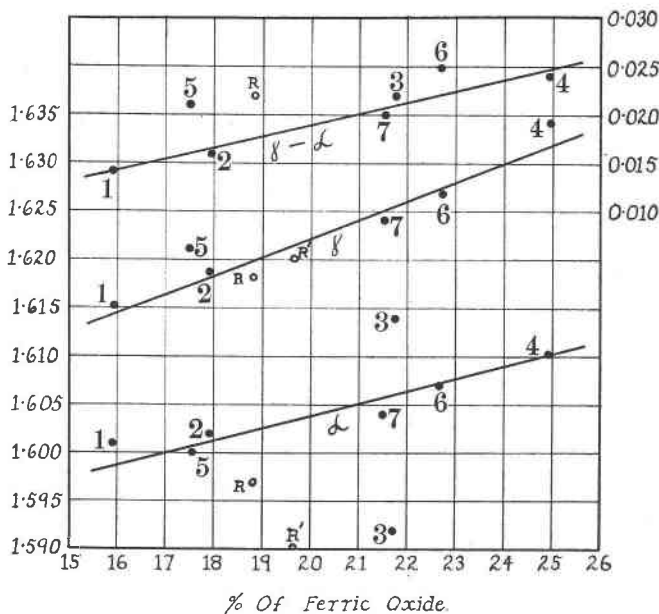


FIG. 1. Relation between refractive indices and content of Fe_2O_3 in glauconite.

account of the micaceous nature of the mineral. Hence, the poorer alignment of the α points will probably be due to the difficulty of determining the minimum value for the refractive index ellipsoid. Data taken from the literature have been added to those determined by the writers; these are the points R and R' (Ross, 1926, p. 4). The values for R fit the curves quite well but unfortunately Ross only gave the optical constants for R' to the second place, so that plotting these values on Fig. 1 will be somewhat inaccurate. The birefringence for R' was not plotted. The curves certainly suggest that a series exists, grading from the normal ferruginous glauconites to the aluminous glauconites; the latter are classed by Smulikowski (1936) as pholidoides, and in this group he includes his mineral skolite.

It must be pointed out that these curves, based on so few data, can only be provisional in the meantime, but the writers consider that the data obtained so far should be plotted and curves drawn so that as new information is obtained, this may be added and the curves corrected. It is proposed to name the series the glauconite series and the two end-members are to be termed aluminous glauconite (AlGl) and glauconite (Gl).

FORMULA OF GLAUCONITE

The derivation of a formula for glauconite has occasioned considerable discussion. The formulae suggested by some authors are given below:

Clarke (1908):

$\text{Fe}^{\text{III}}\text{KSi}_2\text{O}_6 + aq$, in which some iron is replaced by aluminum and other bases partly replace potassium.

Hallimond (1922):

$\text{R}_2\text{O} \cdot 4(\text{R}_2\text{O}_3, \text{RO}) \cdot 10 \text{SiO}_2 \cdot n \text{H}_2\text{O}$.

Ross (1926):

From $2\text{H}_2\text{O} \cdot \text{K}_2\text{O} \cdot 2(\text{Mg}, \text{Fe})\text{O} \cdot 2(\text{Fe}, \text{Al})_2\text{O}_3 \cdot 10\text{SiO}_2 + 3\text{H}_2\text{O}$ to
 $2\text{H}_2\text{O} \cdot \text{K}_2\text{O} \cdot (\text{Mg}, \text{Fe})\text{O} \cdot 3(\text{Fe}, \text{Al})_2\text{O}_3 \cdot 10\text{SiO}_2 + 3\text{H}_2\text{O}$.

Schneider (1927):

$(\text{K}, \text{Na})(\text{Fe}, \text{Mg})(\text{Fe}, \text{Al})_3 \cdot \text{Si}_6\text{O}_{18} \cdot 3\text{H}_2\text{O}$.

Hadding (1932):

$\text{K}_2\text{O} \cdot (\text{Mg}, \text{Fe})\text{O} \cdot \text{Fe}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3 \cdot 8\text{SiO}_2 \cdot 3\text{H}_2\text{O}$.

Gruner (1935):

$(\text{OH})_{6-10} \cdot \text{K}_{2-3}(\text{Mg}, \text{Fe}^{\text{II}}, \text{Ca})_{1-3}(\text{Fe}^{\text{III}}, \text{Al}, \text{Si})_{3-6}(\text{Si}_{13-14}, \text{Al}_{2-3})\text{O}_{38-40}$.

Smulikowski (1936):

$\text{H}_4\text{K}(\text{Mg}, \text{Fe}^{\text{II}}, \text{Ca})(\text{Al}, \text{Fe}^{\text{III}})_3\text{Si}_6\text{O}_{20} \cdot 4\text{H}_2\text{O}$.

The writers agree with Gruner (1935, p. 705) that the various formulae suggested are all very close to the truth. Gruner's formula, however, appears to fit the existing analyses very well indeed and has a further advantage that it has been derived after consideration of the chemical composition and crystal structure of glauconite. Nevertheless Smulikowski's formula differs very little from that derived by x-ray analysis by Gruner. One of the analyses of New Zealand glauconite (No. 6) has been recalculated on the basis of 48 (O, OH) atoms to the unit cell and the derived formula fits Gruner's suggested formula reasonably well (Table VI). However, SiO_2 is rather higher than the theoretical figure 14. Nevertheless Gruner (1935, p. 713) points out that the excess of SiO_2 over Al_2O_3 gives rise to a higher Si:Al ratio in the $(\text{Al}, \text{Si})_4\text{O}_{10}$ group than in mica, and that this might even result in substitution of a few Si ions for Fe^{III} or Al in positions with a covalency of 6. The writers considered it justifiable to use only 4% of $\text{H}_2\text{O} > 105^\circ\text{C}$. in the calculation of the formula, after consideration of Ross's dehydration curve (1926, p. 8).

TABLE VI. CALCULATION OF FORMULA OF GLAUCONITE (ANAL. NO. 6)

	Wt. %	mols.	48 (O, OH)	
SiO ₂	47.42	0.790	14.444	14.444
Al ₂ O ₃	7.19	0.070	2.558	} 2.000 0.558
Fe ₂ O ₃	22.64	0.142	5.190	
FeO	3.39	0.046	0.840	} 5.748
TiO ₂	0.10	0.001	0.018	
CaO	0.27	0.004	0.072	} 1.972
MgO	2.28	0.057	1.042	
K ₂ O	7.46	0.079	2.888	2.888
H ₂ O	4.00	0.222	8.116	8.116

Formula: (OH)_{8.1} · K_{2.8} · (Mg, Fe'', Ca)_{1.9} · (Fe''', Al)_{6.7} (Si_{14.4}, Al₂)O₄₀.

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