

X-RAY STUDIES ON CRYSTALLINE ROCKS OF THE ÓFALU GROUP, MECSEK MOUNTAINS, HUNGARY

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SUMMARY

Plagioclase, chlorite, biotite and muscovite component minerals have been separated from the metamorphic rock members of the Ófalu Group, Mecsek Mountains, S-Hungary. The mineral fractions were studied by X-ray powder diffraction methods. The *plagioclases* have composition of (low) albite to oligoclase (peristerites). Chlorites and biotites were separated from greenstones derived from basic volcanic rocks. *Chlorite* formula corresponds to the composition of ripidolites according to the classification of HEY (1954). *Biotites* contain 2.7 (Fe + Mn) and 2.9 (Mg + Al) in six octahedral positions. *Micas* separated from mica schists revealed special characteristics: there is a double 060 reflection at 1.503 and 1.522 Å, respectively, probably due to a mixture of *muscovite with a transitional dioctahedral-trioctahedral type mica* similar to that, which was synthesized by CROWLEY and ROY [1964]. According to the authors' knowledge, no natural analogues of this letter phase which have been reported so far.

Standard mixtures of the separated minerals were prepared for the purposes of a quantitative X-ray diffraction analysis. Determinative graphs were constructed for hornblende, biotite, chlorite, plagioclase and quartz using metallic Cu as internal standard. Results of the application of the present method for the quantitative analysis of the Ófalu Group greenstones were compared with those of the application of the BRISTOL [1967] method and with chemical mesonorm of the rocks.

INTRODUCTION

The Ófalu Group is a sequence of regionally metamorphosed eugeosynclinal volcanic and sedimentary rocks [SZEDERKÉNYI and GHONEIM, 1977]. The grade of metamorphism ranges from the quartz-albite-epidote-biotite subfacies of greenschist facies [TURNER and VERHOOGEN, 1960] up to the greenschist-amphibolite transition facies [TURNER 1968]. The metavolcanic rock members included in the Ófalu Group are greenstones derived from a basic volcanic suit of andesitic basalt to basaltic composition. The greenstones alternate with metasedimentary mica schists. It was already stated by GHANEM and RAVASZ—BARÁNYAI [1969] that the crystalline schists of the area "seem to derive from mostly basic products of an initial magmatism connected with the ancient geosyncline".

Aim of the present study is to apply some current techniques of the X-ray diffraction analysis to the mineralogical characterization of the component minerals of these metamorphic rocks as well as to develop and apply a quantitative method to the X-ray determination of the composition of the rock samples. This work represents a part of the detailed petrographical investigation of the Ófalu Group [GHONEIM, 1977].

X-RAY IDENTIFICATION AND CHARACTERIZATION
OF MINERALS SEPARATED FROM THE ROCKS

Methods of separation and X-ray diffraction analysis

For a detailed X-ray characterization of the individual mineral phases as well as for the preparation of standard mixtures to the purposes of the quantitative analysis, pure mineral phases were necessary.

The selection of rock samples for separation was preceded by a microscopic and X-ray diffraction analysis in order to check the amount and alteration degree of the component under consideration. By means of a preliminary qualitative X-ray analysis of the individual rock types the following minerals were found (components separated during the present work are italicized):

— in greenstones: *plagioclase, hornblende, chlorite, biotite*; a few quartz, epidote, carbonates and secondary potash feldspar (in some exceptional cases),

— in mica schists: *muscovite, quartz, plagioclase, potash feldspar*; a few chlorite and carbonates.

Mineral fractions were obtained with a Frantz isodynamic magnetic separator in conjunction with a separation in bromoform. The most effective grain size varied from one sample to another in the range of 0.3 to 0.06 mm. Plagioclase and quartz were separated from the light fraction while hornblende, biotite and chlorite from the heavy one. Calcite was removed from the light fraction by treatment with 1:1 HCl. Quartz and plagioclase were picked under a petrographic microscope and then checked for the possible presence of impurities. A slide slope of 20° with magnet set at 0.8 amps in the Frantz isodynamic separator were the best extraction conditions for hornblende, biotite and chlorite. Magnetic separation was repeated several times.

Biotite, chlorite and muscovite are adhesive to some extent. This property was used to a further purification by shaking them carefully on a coarse paper.

Oriented prepares for the X-ray analysis were made by filling sample holder and pressing the powder with a glass plate from above. Non-oriented X-ray prepares were obtained by sieving the fine powder of the material on a coarse paper put under the opening of a sample holder and removing the surplus of the material without the slightest pressure on the sample. The sample holder was then turned over and the coarse paper removed. X-ray analysis was carried out with a Mueller Micro 111-Philips type diffractometer using Ni filtered Cu radiation at 26 kV and 36 mA.

Results

Plagioclase

It was attempted to determine the composition of plagioclases on the basis of the separation of the $131/1\bar{3}1$ and $241/2\bar{4}1$ reflections, respectively [BAMBAUER *et al.*, 1967]. The following ranges of variation of these characteristic parameters were found (expressed as $2\theta^\circ$ for $\text{CuK}_{\alpha 1}$ radiation):

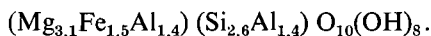
TABLE 1

Petrographic name	Number of analyses	$2\theta_{131} - 2\theta_{1\bar{3}1}$ (°)	$2\theta_{241} - 2\theta_{2\bar{4}1}$ (°)
greenstones	18	1,0—1,55	1,4—1,7
mica schists	4	1,1—1,3	1,4

All these characteristic values fall into the domain of peristerites of the low-plagioclase series. No exact determination of the anorthite content from powder data is possible in this interval of composition. It can be concluded, however, that it is the range of albite to oligoclase.

Chlorite

Chlorites of the greenstones are IIB monoclinic polytypic modifications [BROWN, BAILEY, 1962], as it is normally found in metamorphic rocks. The octahedral iron content ("heavy atom content") equals 1.5 ± 0.5 as determined by the PETRUK [1964] method considering six octahedral sites. The "degree of asymmetry" of the iron distribution is +0.1. Octahedral aluminum contents were estimated from the exact *d*-positions of the 004 and 005 reflections using the *d*/001/ — Al diagram published by BRINDLEY [1961, Fig. VI. 7]. The chemical formula of the chlorites of the samples No. 49 and SA-2 obtained is as follows:



This formula corresponds to the composition of *ripidolites* in the classification scheme of HEY [1954, see BRINDLEY 1961, Table VI. 3].

Biotite

The octahedral ion content of the biotites of the greenstones was determined by a method of FRANZINI and SCHIAFFINO [1965] recommended by RADOSLOVICH [1975]. No individual ions, only groups of ions of similar atomic scattering properties can be determined by this method. The data obtained for the biotite of the sample No. SA-6 are as follows:

$$Fe^{2+} + Fe^{3+} + Mn = 2.7,$$

$$Mg + Al + 2Ti = 2.9$$

(for six octahedral positions, supposing that 0.4 octahedral sites are vacant). The accuracy of these figures is slightly less than ± 0.5 .

Muscovite + transitional di-trioctahedral mica (?)

White micas were separated from the mica schists (sample No. 78). X-ray data obtained are as follows:

Polytypism: 2M

$$d(002) = 9.975 \pm 0.005 \text{ \AA}$$

$$d(060) = \begin{cases} 1.503 \pm 0.0005 \text{ \AA} \\ 1.522 \pm 0.0005 \text{ \AA} \end{cases}$$

$$KUBLER's \text{ degree of crystallinity} = 2.75 \pm 0.25 \text{ mm} = 0.205 \pm 0.015^\circ 2\theta(CuK_{\alpha 1})$$

$$\frac{I_{004}}{I_{002}} = 0.43 \text{ (I = peak height)}.$$

The parameters listed above are widely used for the X-ray characterization of potassic white micas.

The sharpness of the 002 reflection as expressed by the low value of the KUBLER's degree of crystallinity is in good agreement with the metamorphic origin of these micas.

There are however different opinions concerning the crystal chemical and petrological interpretation of the values $d(002)$ and $d(060)$.

$d(002)$ was determined in oriented preparates as the average of the spacings $4.d(008)$ and $5.d(0010)$ measured on 3 parallel X-ray patterns with a correction using the 4.26 Å reflection of low quartz. The values of $d(060)$ were determined in non-oriented preparates on 5 parallel X-ray patterns with a correction using the 1.541 Å line of low quartz.

From a crystal chemical point of view, it seems to be true that no direct determination of the chemical composition of micas from structural data similar to those listed above is possible. This is mainly due to the fact that the various formulas expressing the relationship between cell dimensions and chemical composition, contain too many chemical variables [e.g. “ b_0 -axis formula”, ZUSSMAN, 1967, RADOSLOVICH, 1975].

Petrological experience has shown, however, that a reliable empirical relationship can be found between some structural and chemical variables considering only the range of variation of the chemical composition frequently occurring under normal natural conditions. In addition, X-ray data can be interpreted directly in terms of various characteristics of metamorphism. It was attempted earlier to relate $d(002)$ values to the Na content an $d(060)$ or b_0 values to the octahedral Fe+Mg content of micas. More recent works consider both variables simultaneously pointing out that high contents of octahedral Fe+Mg (*i.e.* phengitic compositions) influence strongly the $d(002)$ — Na relationship [CIPRIANI *et al.*, 1968; KOTOV *et al.*, 1969; MILKEVICH and KOTOV, 1972; DUNOYER DE SEGONZAC and HICKEL, 1972; SEKI, 1973].

Fortunately, the $d(002)$ value of the Ófalu mica (9.975 Å) lies in the range where the influence of the octahedral substitutions is weak: using the diagrams of the authors mentioned it can be concluded that the $\frac{\text{Na}}{\text{Na} + \text{K}}$ ratio of this mica is

not higher than 10% (according to the formulas of CIPRIANI *et al.*, 1968: $\frac{\text{Na}}{\text{Na} + \text{K}} = 4\%$ considering only the $d(060) = 1.503$ Å reflection).

By means of a statistical study SASSI [1972] divided the dioctahedral mica series into the fields of muscovites *s. str.* and of phengites at $b_0 = 9.025$ Å, *i.e.* at $d(060) \approx 1.504$ Å. According to this boundary, one of the micas under consideration ($d(060) = 1.503$ Å) is a muscovite *s. str.* It is remarkable, however, that its $d(060)$ value falls just into the interval of statistical minimum between muscovites and phengites.

The relatively strong reflection at 1.522 Å (*Fig. 1*) deserves special attention. This reflection cannot be attributed to any impurity found in the separated mica fraction (quartz, feldspars). It was therefore considered to be a 060 reflection of another type of mica present in the same sample. Indeed, by means of microscopic analysis two different types of “muscovite” have been found in these rocks: the first one occurs as well crystallized muscovite sheets from 0.6×0.25 mm to 1.5×1.0 mm large, while the other forms cryptocrystalline shreds and flakes [GHONEIM, 1977].

According to the authors' knowledge, no $d(060)$ values near 1.522 Å have been reported so far for natural micas. This figure is substantially higher than $d(060)$ spacings measured in phengitic micas and celadonites (≈ 1.51 Å) and much lower than those found for trioctahedral micas (≈ 1.53 Å). The mineral characterized by this intermediate $d(060)$ value can be interpreted preliminarily as having a transitional

di-trioctahedral composition. KWAK [1971] synthesized micas transitional between dioctahedral and trioctahedral compositions having b_0 parameters occasionally as low as 9.13 Å (corresponding to about $d(060)=1.52$ Å). These phases, however,

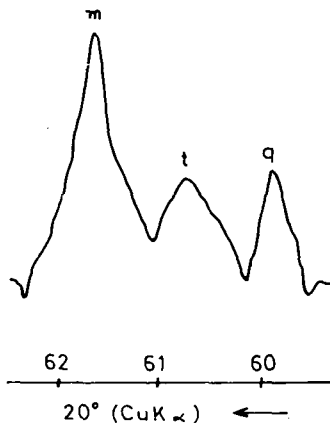


Fig. 1. 060 reflections of the mica fraction of sample No. 78 (mica schist, Ófalu Group). m=muscovite, t=transitional di-trioctahedral mica (?), q=quartz. Conditions of the X-ray diffraction analysis: non-oriented prepare, scanning speed: 1/2°/min, registration speed: 800 mm/h, slits: 1°—0,2—1°, time constant: 8 sec, intensity factor: 1×4

probably have no natural analogues, being pure Fe-Mg-micas that contain no Al. X-ray data of the Ófalu micas agree better with those obtained for some synthetic Mg-Al-mica phases described by CROWLEY and ROY [1964]. According to these data, micas synthesized in the transitional compositional rang ebetween phlogopite and muscovite revealed two distinct 060 spacings almost identical with those found in the Ófalu material (Table 2).

TABLE 2

Material	Reference	$2\theta(0,60)(^\circ)$ (CuK $_{\alpha 1}$)	
synthetic mica	CROWLEY, ROY [1964, Fig. 9]	61,72	60,73
synthetic mica	CROWLEY, ROY [1964, Fig. 9]	61,77	60,82
natural mica	Ófalu, present study	61,65	60,79

CROWLEY and ROY considered that the spacings listed above represent two different mica phases each of which being a crystalline solution that “must involve some of the trioctahedral-dioctahedral type substitution” (p. 358). This may be true especially for the phase characterized by $2\theta(060) \approx 61.7^\circ$.

The unusual “basic” character of these potassic white micas seems to be in accordance with the geological nature of the rock series: it is supposed that the Ófalu mica schists have derived from basic rocks [GHONEIM, SZEDERKÉNYI, 1977].

In addition, however, to this preliminary interpretation, further investigations (e.g. electron microprobe analysis) are needed in order to elucidate the nature of these micas in more detail.

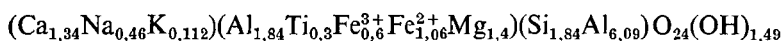
QUANTITATIVE X-RAY DIFFRACTION ANALYSIS

Principles of the method

Unlike sediments, metamorphic rocks are relatively rarely analysed by quantitative X-ray diffraction methods. For the study of the greenstones of the Ófalu Group a method developed by BRISTOL [1967] was adopted with some essential modifications. This is an internal standard method with metallic Cu as reference material. The main deviation from BRISTOL's original method consists in the selection of the components of the artificial "background" mixtures. BRISTOL prepared these "background" mixtures from pure minerals belonging to the same metamorphic facies but taken from different sources while the present authors separated these minerals from the rocks to be analysed.

The importance of the approximate selection of the standard minerals used to the quantitative X-ray analysis has been pointed out by a number of authors dealing with the problem. This is mainly due to the fact that the minerals included into the same mineralogical species or group (e.g.: chlorite, amphibole, plagioclase etc.) may substantially differ from the point of view of their X-ray diffraction intensities [see e.g.: VAN DER MAREL, 1966, RISCHÁK, VICZIÁN, 1974]. This is why it has been frequently proposed to use standard minerals extracted from the rocks themselves that are to be analysed [GIBBS, 1967].

"Background" mixtures were prepared from the components biotite, hornblende, chlorite, quartz and plagioclase. These minerals are present in high amounts in the rocks under consideration. It was attempted to cover the compositional range that may occur in the reality in these rocks. A correction of the "theoretical" composition of these mixtures was necessary in order to take the amount of the impurities of the separated minerals into consideration. Percentages of quartz and plagioclase were determined by a dilution with pure quartz. The actual compositions of the "pure" fractions of biotite, hornblende and chlorite were determined by an approximation using the uncorrected determinative graphs obtained directly from the measurements on our "background" mixtures. These "pure" fractions contained the same minerals that were described in the first part of this work. Hornblende has the formula:



[SZEDERKÉNYI and GHONEIM, 1977].

Methods of preparation and measurement

To 1.00 g of each mixture 0.05 g fine-grained metallic Cu was added. The whole mixture was then homogenized in a rotating mortar in 25 ml of acetone, for about 2 hours.

X-ray prepreparates were made by filling a Philips rotating sample holder from above and pressing the powder by a glass plate. This preparation is similar to that described by BRISTOL, though producing a somewhat higher degree of preferred orientation. Five parallel X-ray measurements were made for each mixture. Instrument settings were as follows: scanning speed: 1°/min, registration speed: 1600 mm/h, slits: 1°—0.2—1°, angular range: 2 to 50°; 2θ(CuK_{α1} radiation).

X-ray intensities were measured as the heights of the reflections above the base line. Base lines were drawn somewhat more flexibly than by BRISTOL (not a completely

straight line across the whole pattern was drawn). This permitted us to obtain zero intensity in each case when the corresponding concentration was also equal to zero. Reflections selected for intensity measurements are listed in Table 3.

TABLE 3

Mineral	$d(\text{\AA})$	$2\theta^\circ(\text{CuK}\alpha_1)$	hkl	JCPDS Card No.
Hornblende	8.40	10.52	110	20—481
Plagioclase (low) (Oligoclase)	3.18	28.04	040	9—457
Chlorite (Ripidolite)	7.07	12.52	002	7—76
Biotite	10.1	8.74	001	2—0045
Quartz	4.26	20.82	100	5—0490
Cu (standard)	2.088	43.36	111	4—0836

Results

The measured intensities of the selected reflections of the minerals (Table 3) were divided by the intensity of the 111 reflection of the Cu standard. The intensity ratios $I_{i(hkl)}/I_{\text{Cu}(111)}$ were plotted as a function of the weight per cent of the mineral in the standard mixture. Determinative graphs were constructed by "least squares" calculation (Fig. 2). No significant deviations from the linearity could be observed in any case.

The scattering of the points representing the individual measurements around the best fit lines satisfactorily low except for the case of chlorite. This may be due to difficulties of pure separation as well as probably to the different degree of preferred orientation of the preparates containing chlorites.

The agreement with the determinative graphs of BRISTOL is sufficiently good for quartz, biotite and chlorite considering the differences in the choice of standard minerals, methods and apparatus. The graph obtained by us for plagioclase (low) having a composition that corresponds to that of peristerites (albite to oligoclase) lies between the lines proposed by BRISTOL for "albite" and "plagioclase (An_{30})". The largest deviation is that of the graph of hornblende from the line proposed by BRISTOL for actinolite. This may be due to greater differences in mineralogy and grain shape, which are reflected also by the degree of preferred orientation.

APPLICATION OF THE METHOD TO THE STUDY OF THE GREENSTONES OF THE ÓFALU GROUP

The present standard determinative graphs were used for the mineralogical analysis of the Ófalu greenstones. The data obtained by this method were compared with those obtained using the standard graphs of BRISTOL [1967]. Since the determination of the actual modal composition of the Ófalu greenstones by microscopic analysis was not possible because of the fine grain size and high degree of alteration, particularly for chlorite, biotite and hornblende, the X-ray modes were compared with a chemical mesonorm as a reference to the actual percentage of the minerals in the rocks (Table 4).

In chemical mesonorm-calculations parts of biotite and hornblende were transformed into chlorite. Because they were difficult to assess the limits of this trans-

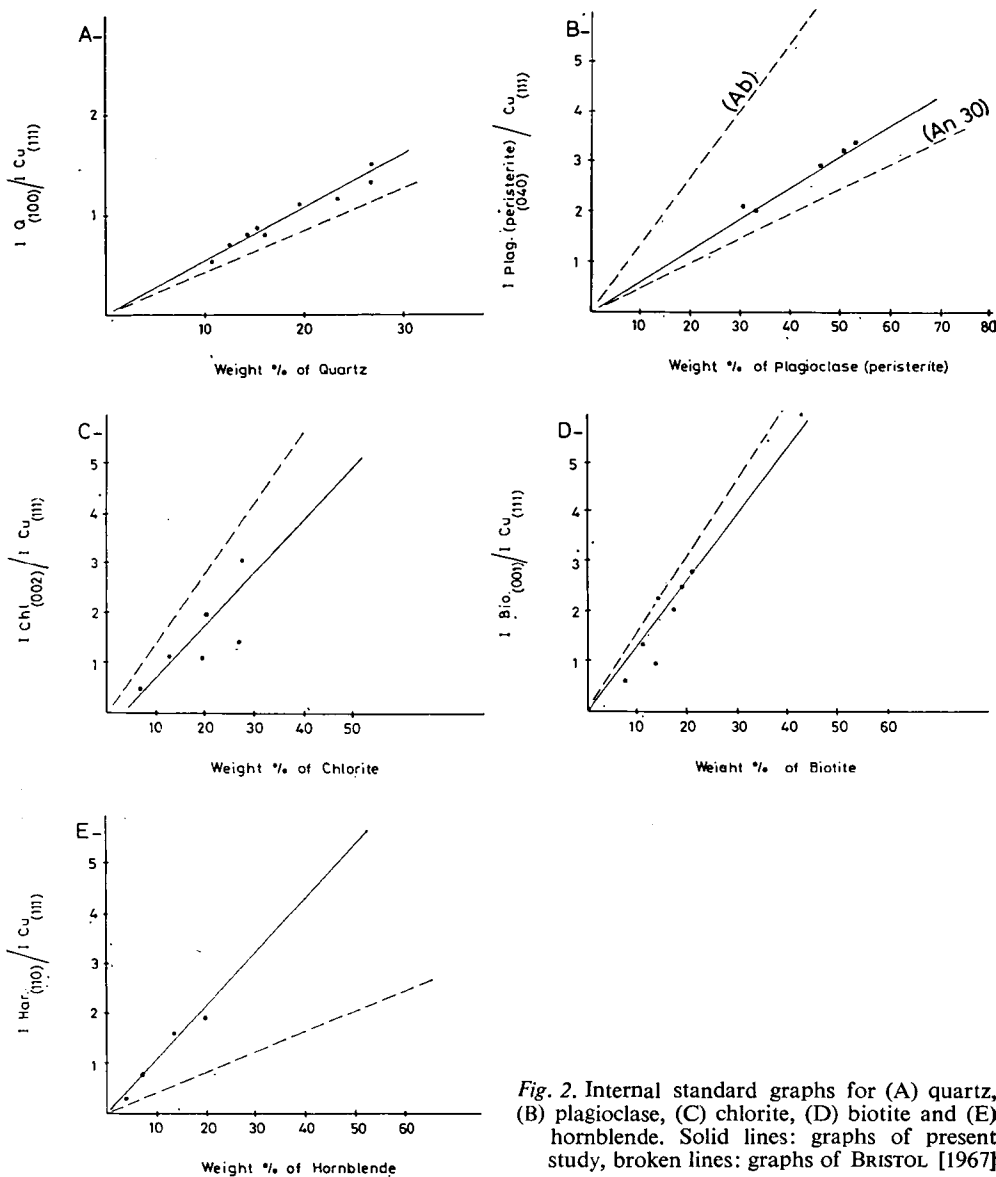


Fig. 2. Internal standard graphs for (A) quartz, (B) plagioclase, (C) chlorite, (D) biotite and (E) hornblende. Solid lines: graphs of present study, broken lines: graphs of BRISTOL [1967]

formation, the samples for chemical analysis were microscopically selected. Three cases can be distinguished:

1. Sample No. Sc. 17 contains a minimum quantity of chlorite and no biotite, but a considerable amount of hornblende.

2. Samples No. 49 and 94 contain a minimum of biotite, plenty of chlorite and no hornblende.

3. Sample No. SA-5 contains equal proportions of biotite and chlorite, but no hornblende.

TABLE 4

*Quantitative mineral composition of some Ófalu greenstones
Comparison of data obtained by different methods*

Sample No.	Petrographic name	Mineral	X-ray mode		Chemical mesonorm
			BRISTOL's graph	present graph	
Sc. 17	Amphibolite	hornblende chlorite plagioclase quartz	80*** 10** 7 (26)* 11	28,5 17 20 8	34,0 19,5 22,4 8,1
49	Andesitic basalt	chlorite biotite plagioclase quartz	16 5 17 (50)* 0	20 6 39 2	11,3 5,0 38,25 3,0
SA—5	Andesitic basalt	chlorite biotite plagioclase quartz	15 11,5 12 (41,5)* 4	19 12 35 5,5	13,0 8,0 44,0 8,1
94	Calcareous phyllitic tuffs	chlorite biotite plagioclase quartz	15 3,5 6 (25)* 17	20 3 19 15	24,9 4,0 19,85 21,5

* In brackets: values calculated from the „plagioclase (An₃₀)” standard graph. Values in normal position: calculated from the „albite” standard graph.

** Uncorrected value.

*** Determined using the graph of actinolite of BRISTOL.

In addition to the components listed in the table there are accessories in each sample: calcite, magnetite, sphene, rutile, apatite, epidote, hematite, goethite, orthoclase (secondary).

Table 4 shows that the X-ray modes determined using the standard graphs described here in general agreement with those determined using BRISTOL's standard graphs with some exceptions. As revealed by a comparison of the standard graphs, the largest difference consists in the determination of amphibole (Sample Sc. 17.). The weight per cent of hornblende determined using the present standard graph is nearer chemical mesonorm than so determined as actinolite by BRISTOL's method.

In the quantitative analysis of plagioclases using BRISTOL's method we considered the standard graphs of both “albite” and “plagioclase (An₃₀)” since the plagioclase of Ófalu greenstones contains less than 30 per cent anorthite. The values determined with the “albite” graph were far from those calculated by chemical mesonorm. Some difficulties also arose with the use of the standard “plagioclase (An₃₀)” graph of BRISTOL (sample No. 49). The data obtained from the present standard plagioclase (albite-oligoclase) graph seem to be near the actual percentages of plagioclase as expressed by the chemical mesonorm values.

It is to be mentioned that details of the calculation of the chemical mesonorm are to be found in the work of GHONEIM [1977].

CONCLUSIONS

In conclusion, it can be stated that the internal standard method as proposed by BRISTOL [1967] proved to be a suitable tool in determining the quantitative composition of metamorphosed volcanic rocks. The published standard determinative graphs, however, either those of BRISTOL, or of the present work, are to be adopted only with great precaution in the case of minerals which may have variable compositions (e.g. amphiboles, plagioclases). The separation of standard minerals from the rocks to be analysed, as described here, seems to be an appropriate way to overcome most of these difficulties.

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