

The volume of the sample was 0.2 g. The sample was dried at 105°C for 24 h. The sample was then dissolved in 10 ml of a 10% NaOH solution. The solution was then titrated with 0.1 M HCl. The titration was carried out in a glass beaker with a magnetic stirrer. The titration was stopped when the color of the solution changed from yellow to blue. The volume of the sample was 0.2 g. The sample was dried at 105°C for 24 h. The sample was then dissolved in 10 ml of a 10% NaOH solution. The solution was then titrated with 0.1 M HCl. The titration was stopped when the color of the solution changed from yellow to blue.

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PHLOGOPITE FROM LAMPROPHYRES NEAR ZAWIERCIE (UPPER SILESIA)

Abstract. X-ray, IR spectroscopic and quantitative chemical analyses were carried out on a dark mica separated from minette penetrated by a borehole near Zawiercie. It has been found that it is phlogopite (3T) with the unit cell parameters: $a = 5.317 \pm 0.002$ Å, $c = 30.031 \pm 0.01$ Å, $c/a = 5.648$, and the structural formula: $(K_{1.51}Na_{0.38}Ca_{0.32})(Mg_{3.98}Fe^{2+}_{0.81}Fe^{3+}_{0.77}Ti_{0.54}Mn_{0.01}Li_{0.01}\square_{0.39})[(Si_{5.15}Al_{2.18}Fe^{3+}_{0.18})O_{20}] (F_{1.90}OH_{1.86}O_{0.24})$. These results confirm the earlier observations that dark micas from minettes, kersantites and alkaline rocks are usually phlogopites.

INTRODUCTION

A typical component of lamprophyres (semilamprophyres) occurring in the NE margin of the Upper Silesian Coal Basin is dark mica which therefore, on the basis of microscopic studies, has been referred to as biotite (Małkowski, Karasiński 1928; Śliwiński 1960, 1964; Juskowiak 1971; Ryka 1974). During the petrographic studies of lamprophyres deriving from new boreholes near Zawiercie (Heflik et al., 1985), this mica was separated from minette penetrated by the ZMZ-94 borehole. The mineral was subjected to quantitative chemical analysis, X-ray diffractometric and IR spectroscopic studies.

EXPERIMENTAL

Separation

The rock was ground in the Abbich mortar and sieved on 0.2, 0.4 and 0.6 mm mesh screens. From the 0.4–0.6 mm fraction the heavy fraction enriched in mica was separated in bromoform (specific gravity = 2.98 G/cm³) and purified by hand, in the field of view of a stereoscope, of any subordinate admixtures of other components.

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Quantitative chemical analysis

SiO_2 and Al_2O_3 were determined on melting of about 0.5 g sample with Na_2CO_3 , the former by gravimetric and colorimetric methods, the latter by direct method, using EDTA standard solution and PAN as an indicator. The total Fe content ($\text{Fe}^{2+} + \text{Fe}^{3+}$), as well as the content of Mg, Ca, Mn, Cr, Na, K, Li, Rb and Ti, was determined from about 0.1 g sample decomposed through treatment with hydrogen fluoride. ($\text{Fe}^{2+} + \text{Fe}^{3+}$), Mg, Ca, Mn and Cr were determined with ASA method, while Na, K, Li and Rb by flame photometry with a Pye Unicam SP-90B spectroscope, under the conditions recommended by the manufacturer. Fe^{2+} content was determined by means of manganometry after the decomposition of about 0.1 g sample in concentrated HCl. Ti was determined colorimetrically with the peroxide method, using a Carl Zeiss Jena "Specol" spectrophotometer. H_2O^- and then H_2O^+ were determined on about 0.5 g sample, adopting the generally accepted methods. Fluorine content was determined after 0.5 g sample had been melted with a mixture of $\text{Na}_2\text{CO}_3 + \text{ZnO}$ and distilled off from other components. Distillation was carried out in a mixture with concentrated HClO_4 , supplying water vapour to the bottom of the distillation flask. The determination was made by titration of fluorine and S alizarin complex with $\text{Th}(\text{NO}_3)_4$ solution.

X-ray analysis

X-ray diffraction patterns were recorded with a DRON-1.5 diffractometer. The instrument settings used were: CuK_α radiation ($\lambda_{\text{av.}} = 1.541778 \text{ \AA}$), Ni filter, $I = 20 \text{ mA}$, $V = 36 \text{ kV}$, counter speed $v_1 = 1^\circ/\text{min.}$, chart speed $v_t = 600 \text{ mm/hr}$. The error of measurements of 2θ angles was $\Delta 2\theta = \pm 0.01^\circ$. In order to obtain the most accurate values of d_{hkl} and lattice parameters, corrections were made to eliminate errors arising from the vertical divergence of radiation, Lorentz factors and polarization (Bold, Kosider 1967).

Infrared spectroscopic analysis

Infrared spectra were recorded in the wave number ranges of $3800-3000 \text{ cm}^{-1}$ and $1800-400 \text{ cm}^{-1}$ with a Carl Zeiss Jena UR-10 spectrometer. The analytical conditions were: sample mass 1 mg, KBr discs, chart speed $50 \text{ cm}^{-1}/\text{min.}$, recording breadth $50 \text{ cm}^{-1} = 12 \text{ mm}$.

RESULTS

Macro- and microscopic data

The mica in question makes up 10—25 vol. % of the rock, being the principal component of phenocrysts (Phot. 1). Macroscopically it is black or black-brown, and commonly appears in the form of thick idiomorphic flakes with a hexagonal habit (Phot. 2), varying in size from 0.025 to 4 mm, averaging 0.2—0.5 mm.

Viewed under the microscope, the mineral displays strong pleochroism: α — pale yellow, $\beta = \gamma$ — yellow-brown, of varying intensity even within individual flakes. The optical heterogeneity of flakes becomes more pronounced at crossed polars (Phot. 3). Other optical features of this mineral are: bright, medium interference colours, $n_\beta = n_\gamma = 1.637$, $2V_a = 0$ to a dozen or so degrees, negative optical character. These optical properties correspond best to phlogopite. Most mica flakes are inter-

Table 1
Interplanar spacings (d_{hkl}) of phlogopite from lamprophyre from ZMZ-94 borehole

$d(\text{\AA})$	I	hkl	$d(\text{\AA})$	I	hkl
9.98	100	003	2.257	1	117
5.00	5	006	2.173	5	118
4.60	1	100	2.003	90	00.15
3.924	1	104	1.907	1	11.11
3.657	5	105	1.833	1	11.12; 10.15
3.340	100	009	1.670	25	11.14; 00.18; 215
3.139	10	107	1.537	15	300
2.912	10	108	1.432	20	00.21
2.703	5	109	1.360	10	10.21; 11.19
2.649	1	111	1.329	1	220
2.622	15	112	1.311	1	10.22
2.503	75	114; 00.12	1.306	1	11.20; 224
2.433	5	115	1.253	10	00.24

grown with apatite crystals of varying size (Phot. 4). Occasionally they contain inclusions of opaque minerals (magnetite?).

The micas in question are generally little, altered, although the other rock components have usually been subject to intense alteration. The most common phenomenon is their decoloring due to conversion into hydromica and then into vermiculite. Less common is their replacement by chlorites or light mica with the optical features of muscovite (phengite?) and by carbonate minerals.

X-ray diffraction data

X-ray diffraction patterns obtained for the mica studied are presented in Fig. 1 and Table 1. According to the ASTM classification, they correspond to phlogopite, trigonal polytype 3T.

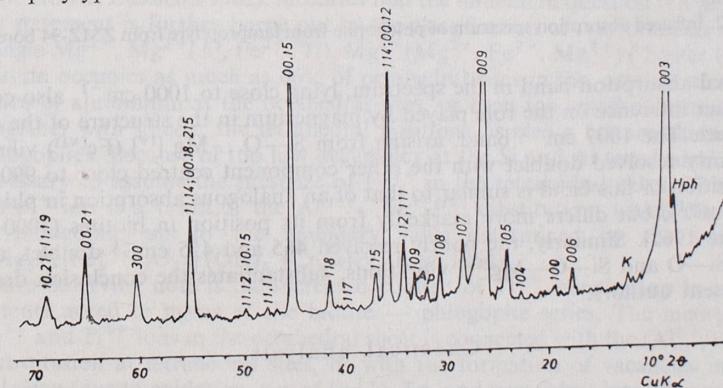


Fig. 1. X-ray diffraction pattern of phlogopite from lamprophyre from ZMZ-94 borehole

Reflections attributed to small admixtures of other minerals: Ap — apatite, Hph — hydrophlogopite, K — kaolinite

The unit cell parameters, calculated from five strongest reflections ($I > 10$) lying in the 2θ angle range between 50 and 80° , are as follows: $a = 5.317 \pm 0.002$ Å, $c = 30.031 \pm 0.01$ Å, $c/a = 5.648$.

Infrared absorption data

A comparison of the infrared spectrum obtained (Fig. 2) with the spectra of trioctahedral micas — biotite, phlogopite (Moenke 1962) — shows that the mica studied has an intermediate character between biotite and phlogopite, yet with the marked dominance of the phlogopite component. The determinant role of magnesium in the structure of the octahedral sheet is indicated by the position of the most intense absorption bands in the IR spectrum, regarding both O—H...Me and Si—O...Me groupings. The most intense OH stretching absorption is centred close to 3700 cm $^{-1}$ and corresponds to OH $^-$ ion situated over the triad consisting of three Mg $^{2+}$ ions. No other bands caused by stretching vibrations of OH $^-$ group which would coordinate the octahedral cations in a different way than the one described above have been recorded. The low absolute intensity of this absorption implies that the analogous structural positions may be partly occupied by F $^-$ or O $^{2-}$ ion.

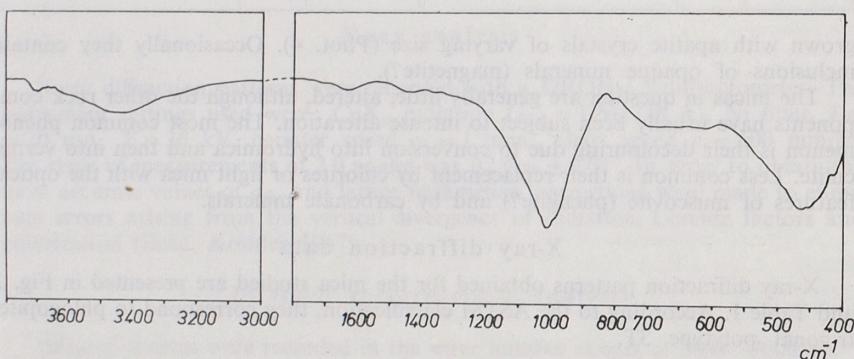


Fig. 2. Infrared absorption spectrum of phlogopite from lamprophyre from ZMZ-94 borehole

The basal absorption band in the spectrum, lying close to 1000 cm $^{-1}$, also confirms the earlier inference on the role played by magnesium in the structure of the octahedral sheet. The 1007 cm $^{-1}$ band, arising from Si—O...Mg $^{[IV]}$ (Fe $^{[VI]}$) vibrations, is a poorly resolved doublet with the other component centred close to 990 cm $^{-1}$. The position of this band is similar to that of an analogous absorption in phlogopite (1010 cm $^{-1}$), but differs more markedly from its position in biotites (1000 cm $^{-1}$) (Moenke 1962). Similarly, the poorly resolved 465 and 455 cm $^{-1}$ doublet, attributed to Si—O and Si—O—Mg $^{[IV]}$ vibrations, substantiates the conclusion drawn by the present authors.

Chemical analysis

The results of quantitative chemical analysis and its recalculations to the chemical formula of mica are presented in Table 2. The resulting structural formula is as follows: $(K_{1.51}Na_{0.38}Ca_{0.32})(Mg_{3.98}Fe^{2+}_{0.81}Fe^{3+}_{0.27}Ti_{0.54}Mn_{0.01}Li_{0.01}\square_{0.39})[(Si_{5.15}Al_{2.68}Fe^{3+}_{0.18})_{20}](F_{1.90}OH_{1.86}O_{0.24})$.

Table 2
Chemical analysis of phlogopite from lamprophyre from
ZMZ-94 borehole

Component	Weight %		
SiO ₂	35.1	K	— 1.514
Al ₂ O ₃	15.5	Na	— 0.375
Fe ₂ O ₃	4.09	Ca	— 0.323
FeO	6.60	Mg	— 3.976
MgO	18.2	Fe ²⁺	— 0.809
MnO	0.06	Fe ³⁺	— 0.274
Cr ₂ O ₃	—	Ti	— 0.537
Li ₂ O	0.01	Mn	— 0.007
TiO ₂	4.87	Li	— 0.006
CaO	2.06	Si	— 5.145
Na ₂ O	1.32	Al	— 2.678
K ₂ O	8.10	Fe ³⁺	— 0.177
Rb ₂ O	—	F	— 1.901
H ₂ O ⁽⁻⁾	0.48	OH	— 1.859
H ₂ O ⁽⁺⁾	1.42	O	— 0.240
F	4.1	O	— 20.000
<hr/>		<hr/>	
—O=F ₂	101.91	Ionic ratio per 24 (O, OH, F)	
	1.73		

It appears from the above data that the mica studied represents one of the members of the isomorphous series annite $K_2Fe_6[Si_6Al_2O_{20}] (OH)_4$ — phlogopite $K_2Mg_6[Si_6Al_2O_{20}](OH)_4$, on the borderline between magnesium biotite — meroxyne and phlogopite. However, the Mg/Fe $^{[VI]}$ ratio of 3.6, exceeding considerably the 2:1 ratio which is generally regarded as a value separating biotites and phlogopites (Deer, Howie, Zussman 1962), indicates that the mineral in question is a phlogopite. This statement is further borne out by the classification of trioctahedral micas in the triangle Mg $^{2+}$, Mg $^{3+}$ (Al, Fe $^{3+}$, Ti), Mg $^{2+}$ (Mg $^{2+}$, Fe $^{2+}$, Mn $^{2+}$) (Foster 1960), as magnesium occupies as much as 66% of octahedral sites in the octahedral sheet. The absence of aluminium at the octahedral sites, or even too small an amount to fill up, together with silicon, the tetrahedral positions, is also a feature characteristic of phlogopites. Because of too low occupancy at the Si and Al tetrahedral sites, it was necessary to assume the presence of Fe $^{3+}$ in the tetrahedral sheet. The presence of Fe $^{3+}$ ion is a result of the Al $^{[IV]} \leftarrow Fe^{3+[IV]}$ substitution, which can take place in phlogopite up to the boundary value determined by the composition $KMg_5Fe[Fe^{3+}Si_5O_{20}](OH)_4$ (Crowley, Roy 1964).

A feature deserving note is the increased content of Ti $^{4+}$, running up to maximum contents noted in micas of the biotite — phlogopite series. The incorporation of Fe $^{3+}$ and Ti $^{4+}$ ions in the octahedral sheet is connected with the (Al $^{3+}/Fe^{3+}$) \rightarrow Si $^{4+}$ substitution at tetrahedral sites, or with the formation of vacancies in the octahedral sheet (due to oxidation, e.g. of Fe $^{2+}$). Tri- and tetravalent ions can occupy, on the average, one out of three available structural positions, the number of cations decreasing (Radoslovich 1963). At the sum of Fe $^{3+}$ and Ti $^{4+}$ ions at the octahedral sites being 0.81, the total content of cations in the octahedral sheet is about 5.61, which is in good agreement with the above observations.

CONCLUSIONS

The above studies have demonstrated unequivocally that dark mica occurring in minette penetrated by the ZMZ-94 borehole near Zawiercie is a 3T phlogopite with the structural formula $(K_{1.51}Na_{0.38}Ca_{0.32})(Mg_{3.98}Fe^{2+}_{0.81}Fe^{3+}_{0.27}Ti_{0.54}Mn^{2+}_{0.01}Li_{0.01}\square_{0.39})[(Si_{5.15}Al_{2.68}Fe^{3+}_{0.18})O_{20}] (F_{1.90}OH_{1.86}O_{0.24})$.

It is feasible that dark micas found in lamprophyres from two other boreholes near Zawiercie (ZMZ-41 and P-8), which were also the object of studies (Heflik et al., 1985), are phlogopites of a similar type, as they exhibit similar optical features and yield identical diffraction lines on X-ray powder patterns.

A mica with a nearly identical chemical composition and optical properties was also described by Pelczar (1973) from minette penetrated by the Borzęta IG-1 bore-hole south of Wieliczka, i.e. about 70 km SE of Zawiercie. The cited author defined it as biotite of a phlogopite type. According to the classification criteria adopted in this paper, this mica should also be referred to as phlogopite. From chemical analyses of biotites from the lamprophyres of the Holy Cross Mountains (Kardymowicz 1964) it appears that their chemical composition corresponds in several cases to phlogopite.

The above conclusions are in accordance with the observations of Velde (1969) and Němec (1972), who found on the basis of a great many chemical analyses that micas from minettes, kersantites and alkaline rocks are as a rule phlogopites.

Translated by Hanna Kisielewska

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FLOGOPIT Z LAMPROFIRÓW OKOLIC ZAWIERCIA (GÓRNY ŚLĄSK)

Streszczenie

Charakterystycznym składnikiem lamprofirów (semilamprofirów), napotkanych w NE obrzeżeniu Górnego Śląskiego Zagłębia Węglowego, jest ciemna mika określana dotychczas na podstawie obserwacji mikroskopowych jako biotyt (Małkowski, Karasiński 1928, Śliwiński 1960, 1964, Juskowiak 1971, Ryka 1974). Łyszczek ten udało się wyseparować z minetty napotkanej w otworze ZMZ-94 koło Zawiercia, poddając go szczegółowym badaniom fazowym (rentgenograficznym i spektroskopowym w podczerwieni) oraz ilościowej analizie chemicznej. Ustalono, że jest to flogopit (3T) o parametrach komórki elementarnej: $a = 5,317 \pm 0,002 \text{ Å}$, $c = 30,031 \pm 0,01 \text{ Å}$ i $c/a = 5,648$ oraz wzorze strukturalnym: $(K_{1.51}Na_{0.38}Ca_{0.32})(Mg_{3.98}Fe^{2+}_{0.81}Fe^{3+}_{0.27}Ti_{0.54}Mn^{2+}_{0.01}Li_{0.01}\square_{0.39})[(Si_{5.15}Al_{2.68}Fe^{3+}_{0.18})O_{20}] (F_{1.90}OH_{1.86}O_{0.24})$.

Można przypuszczać, że flogopitami podobnego typu są ciemne łyszczki występujące w lamprofirach z dwu innych otworów wiertniczych z okolic Zawiercia (Heflik i in., 1985), wykazujące analogiczne cechy optyczne i dające identyczne linie dyfrakcyjne na rentgenogramach jak badana mika. Flogopitem należałoby nazwać również łyszczek opisany przez Pelczar (1973) z minetty napotkanej w otworze Borzęta IG-1, zlokalizowanym około 70 km na SE od Zawiercia, a także szereg biotytów z lamprofirów Górz Świętokrzyskich, analizowanych przez I. Kardymowicz (1964).

Powyższe wnioski są zgodne z obserwacjami Velde (1969) oraz Němec (1972), którzy opierając się na wynikach licznych analiz chemicznych ustalili, że miki z minett, kersantytów i skał alkalicznych są z reguły flogopitami.

OBJAŚNIENIA FIGUR

Fig. 1. Dyfraktogram flogopitu z lamprofiru z otworu ZMZ-94

Refleksy pochodzące od drobnych domieszek innych faz: Ap — apatyt, Hph — hydroflogopit, K — kaolinit

Fig. 2. Widmo absorpcyjne w podczerwieni flogopitu z lamprofiru z otworu ZMZ-94

OBJAŚNIENIA FOTOGRAFIÍ

Fot. 1. Fenoblaszki flogopitu w drobnokrystalicznym tle lamprofiru z otworu ZMZ-94
Nikole skrzyżowane, pow. około 45×

Fot. 2. Blaszki flogopitu wyseparowane z lamprofiru z otworu ZMZ-94
Makrofotografia, pow. około 20×

Fot. 3. Niejednorodna optyczna, skorodowana magmowo fenoblaszka flogopitu z lamprofiru z otworem ZMZ-94
Nikole skrzyżowane, pow. około 50×

Fot. 4. Wrostki apatytu (Ap) we flogopicie z lamprofiru z otworem ZMZ-94
1 nikol, pow. około 90×

**ФЛОГОПИТ ИЗ ЛАМПРОФИРОВ ОКРЕСТНОСТЕЙ
ЗАВЕРЦЯ (ВЕРХНЯЯ СИЛЕЗИЯ)**

Резюме

Характерным компонентом лампрофиров (семилампрофиров), в стреченных в СВ обрамлении Верхнесилезского угленосного бассейна, является темно-цветная слюда, до сих пор на основании микроскопических наблюдений определяемая как биотит (Малковски, Карасиньски 1928; Съливиньски 1960, 1964; деляемая Юсковяк 1971; Рыка 1974). Это слюда удалось выделить из минетты, набуреной скважиной ZMZ-94, возле Заверца, подвергая ее подробным фазовым (рентгенографическим и ИК-спектропресским) исследованиям, а также количественному химическому анализу. Установлено, что это флогопит (3Т) с параметрами элементарной ячейки: $a = 5,317 \pm 0,002 \text{ \AA}$, $c = 30,031 \pm 0,01 \text{ \AA}$ и $c/a = 5,648$ и структурной формулой: $(\text{K}_{1.51} \text{Na}_{0.38} \text{Ca}_{0.32})(\text{Mg}_{3.98} \text{Fe}_{0.81}^{2+} \text{Fe}_{0.27}^{3+} \text{Ti}_{0.54} \text{Mn}_{0.01}^{2+} \text{Li}_{0.01} \square_{0.39})[(\text{Si}_{5.15} \text{Al}_{2.68} \text{Fe}_{0.18}^{3+})\text{O}_{20}] (\text{F}_{1.90} \text{OH}_{1.86} \text{O}_{0.24})$.

Можно предполагать, что подобного типа флогопитами являются темно-цветные слюды, встречающиеся в лампрофирах из двух других буровых скважин в окрестностях Заверца (Хефлик и др., 1985), обнаруживающие аналогичные оптические свойства и дающие идентичные как исследуемая слюда дифракционные линии на рентгенограммах. Флогопитом следовало бы назвать также описанную Пельчар (1973) слюду из минетты, набуренной скважиной Божента ИГ 1 расположенной в около 70 км на ЮВ от Заверца, а также ряд биотитов из лампрофиров Свентокшиских гор, изученных И. Кардымович (1964).

Вышеупомянутые выводы совпадают с наблюдениями Вельде (1969), а также Немеча (1974), которые, исходя из результатов многочисленных химических анализов, установили, что слюды из минетт, керсантитов и щелочных пород как правило представляют собой флогопиты.

ОБЪЯСНЕНИЯ К ФИГУРАМ

Фиг. 1. Дифрактограмма флогопита из лампрофира, набуренного скважиной ZMZ-94
Отражения, происходящие от небольших примесей других фаз: *Ap* — апатит, *Hph* — гидрофлогопит
K — каолинит

Фиг. 2. ИК-абсорбционная кривая флогопита из лампрофира, набуренного скважиной ZMZ-94

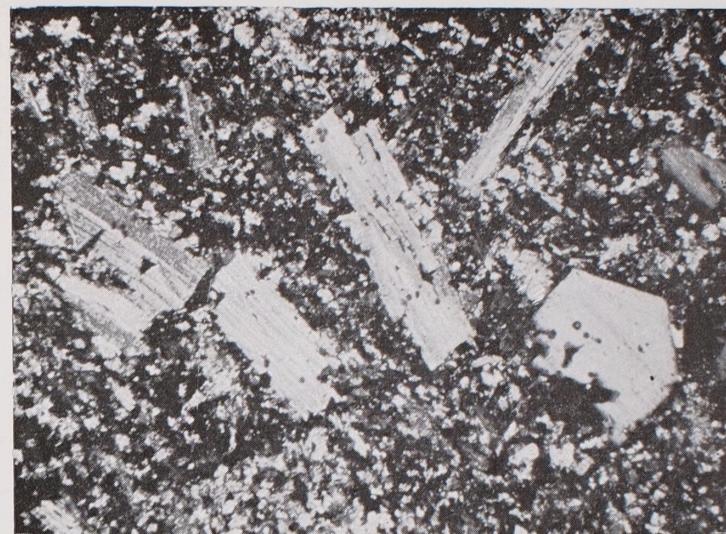
ОБЪЯСНЕНИЯ К ФОТОГРАФИЯМ

Фото 1. Фенопластинки флогопита в мелкокристаллической основной массе лампрофира из буровой скважины ZMZ-94
Скрепленные никели, увелич. $\times 45$

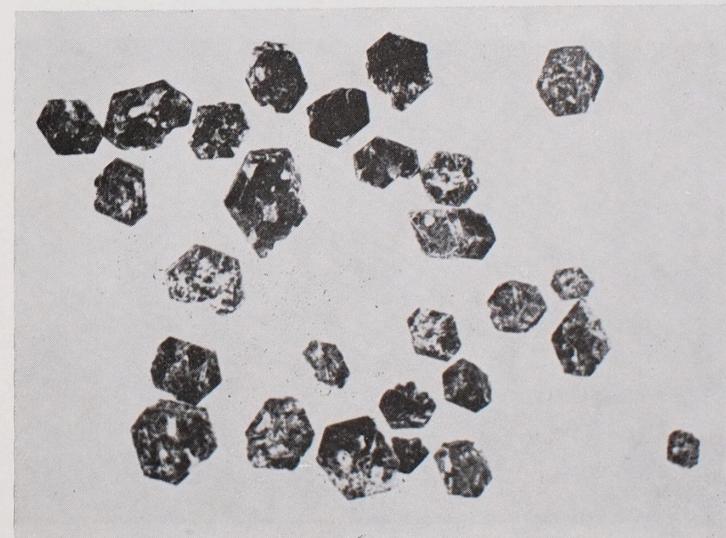
Фото 2. Пластины флогопита, выделенные из лампрофира из буровой скважины ZMZ-94
Макрофотография, увелич. $\times 20$

Фото 3. Оптически неоднородная, магматически корродированная фенопластинка флогопита из лампрофира из буровой скважины ZMZ-94
Скрепленные никели, увелич. $\times 50$

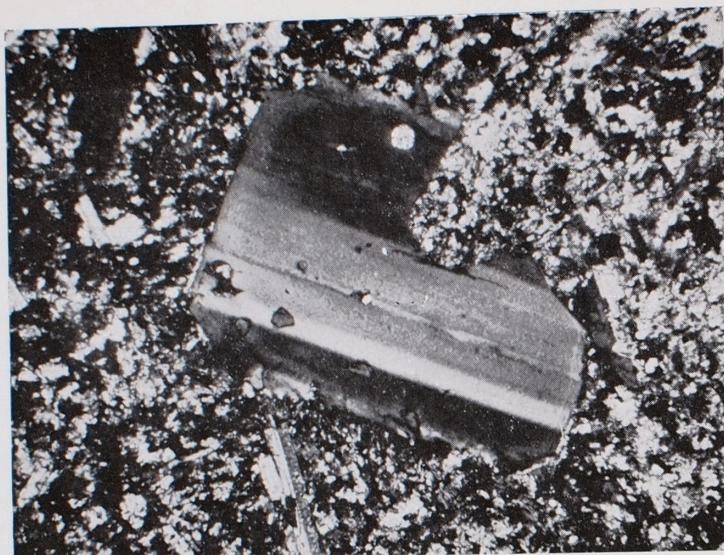
Фото 4. Бростки апатита (*Ap*) во флогопите из лампрофира из буровой скважины ZMZ-94
Один никель, увелич. около $\times 90$



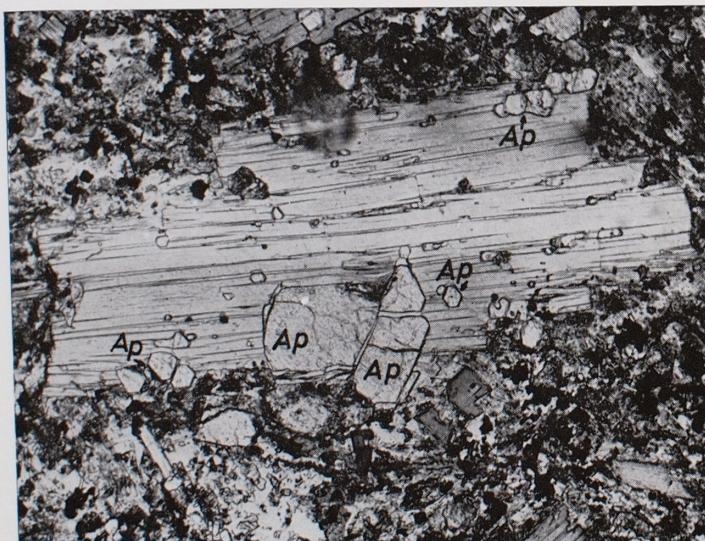
Phot. 1. Phenocrysts of phlogopite in fine-crystalline lamprophyre groundmass from ZMZ-94 borehole
Crossed polars, 45 \times



Phot. 2. Phlogopite flakes separated from lamprophyre from ZMZ-94 borehole
Photomacrophotograph, 20 \times



Phot. 3. Phlogopite phenocryst from lamprophyre from ZMZ-94 borehole, showing optical heterogeneity and magmatic corrosion
Crossed polars, 50×



Phot. 4. Apatite inclusions (*Ap*) in phlogopite from lamprophyre from ZMZ-94 borehole
Plane polarized light, 90×

Marek MUSZYŃSKI, Adam PIECZKA — Phlogopite from lamprophyres near Zawiercie (Upper Silesia)