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PARAKELDYSHITE FROM NORWAY

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ABSTRACT

Parakeldyshite from nepheline syenite pegmatites in foyaite in Lågendalen, southern Norway, is triclinic, *a* 5.419(1), *b* 6.607(2), *c* 8.806(2)Å, α 71.50(2), β 87.15(3), γ 85.63(2)°, *V* 298.0(3)Å^3, *Z*=2. The strongest lines of the indexed X-ray powder pattern are 3.963(10)($1\bar{1}0$), 2.913(9)(121), 4.179(8)(002), 4.234(7) (110), 2.703(7) (200), 6.00(6)(011), 2.718Å (6)($\bar{1}21$). The hardness is 5½-6, sp. gr. (meas.) 3.39, *D* (calc.) 3.40 g/cm^3. The mineral shows a strong cream fluorescence in short-wave UV light.

Parakeldyshite has three perfect pseudo-rhombohedral cleavages {001}, {110} and {$1\bar{1}0$}, and a good cleavage {011}. It shows several sets of polysynthetic twinning, one of which is probably on (100). $2V_\alpha$ (meas.) 84°, α' 1.670, β' 1.692, γ' 1.713 (measured on cleavage fragments). Chemical analysis gives a formula very close to $Na_2ZrSi_2O_7$ with some substitution of H_3O^+ for Na^+. The mineral alters very easily to a secondary phase which is depleted in Na^+ and enriched in H_3O^+ relative to parakeldyshite. The infrared spectrum and DTA data are given.

SOMMAIRE

La parakeldyshite, minéral des pegmatites de syénite néphélinique qui recoupent la foyaite de Lågendalen, dans le Sud de la Norvège, est triclinique. *a* 5.419(1), *b* 6.607(2), *c* 8.806(2)Å, α 71.50(2), β 87.15(3), γ 85.63(2)°, *V* 298.0(3)Å^3, *Z*=2. Le dépouillement du diagramme de poudre donne, pour les raies les plus intenses, les espacements suivants: 3.963(10) (110), 2.913(9) (121), 4.179(8) (002), 4.234(7) (110), 2.703(7) (200), 6.00(6)(011), 2.718Å (6) ($\bar{1}21$). Dureté 5½-6, poids spécifique: 3.39, *D*(calc.) 3.40. En lumière ultraviolette à ondes courtes, le minéral est fortement fluorescent en jaune crème. La parakeldyshite a trois clivages parfaits {001}, {110}, {$1\bar{1}0$}, simulant un rhomboèdre, et un bon clivage {011}. Elle montre plusieurs macles polysynthétiques dont l'une est probablement sur (100). Optiquement on a mesuré sur fragments de clivage: $2V_\alpha$ (mes.) 84°, α' 1.670, β' 1.692, γ' 1.713. A l'analyse, la composition chimique est proche de $Na_2ZrSi_2O_7$ avec substitution de H_3O^+ à Na^+. La parakeldyshite s'altère très facilement en une phase secondaire appauvrie en Na^+ et enrichie en H_3O^+. Les données du spectre à l'infrarouge et de l'ATD sont fournies.

(Traduit par la Rédaction)

INTRODUCTION

Keldyshite was described as a new mineral (Gerasimovskii 1962) from the Lovozero alkaline massif, Kola Peninsula, USSR. The mineral is triclinic, and the formula was given as $(Na,H)_2ZrSi_2O_7$, but no single-crystal data could be obtained. Khomyakov *et al.* (1969) gave new data on keldyshite; H_2O was not found and the analysis corresponded to $Na_2ZrSi_2O_7$. X-ray powder data were close to those previously given. They also described a second phase, occurring with keldyshite, with similar indices of refraction and specific gravity, but with somewhat different X-ray powder data. This new phase also had triclinic symmetry, with *a* 6.66, *b* 8.83, *c* 5.42Å, α 92°45′, β 94°13′, γ 72°20′, and Na, Zr and Si were detected by spectral analysis (Voronkov *et al.* 1970).

The crystal structure of the second phase was described by Voronkov *et al.* (1970), and it was claimed to be another polymorphic form of $Na_2ZrSi_2O_7$. Keldyshite and its supposed polymorph, which remained unnamed, have been repeatedly described by Russian authors both from the Lovozero and Khibina massifs (Bussen *et al.* 1972; Khomyakov & Voronkov 1973; Sizova *et al.* 1974; Sizova *et al.* 1975).

It was recently found (Khomyakov *et al.* 1975; Khalilov *et al.* 1975) that the mineral described by Gerasimovskii is a mixture of two different triclinic phases, one having the composition $Na_2ZrSi_2O_7$ (Phase II) and the other probably $(Na,H_3O)_2ZrSi_2O_7$ (Phase I). The paper by Khomyakov *et al.* (1975) gives much new data for these minerals, and reviews earlier work. The name parakeldyshite has been proposed by Khomyakov for the phase $Na_2ZrSi_2O_7$, and this was approved by the Commission on New Minerals and Mineral Names, IMA, in January 1976, although the name had appeared in the paper by Sizova *et al.* (1975). The naming of the other phase is not yet settled by the IMA commission.

Parakeldyshite occurs in fair abundance in nepheline syenite pegmatites near Larvik, southern Norway. A description of this material is the purpose of the present paper. The mineral was first noticed by Raade in 1970, but the find

was not published because at that time the distinction between keldyshite and its supposed polymorph was not clear, and a comparison of X-ray powder patterns was difficult because the published data had been obtained only with cameras having a diameter of 57.3 mm. Specimens of Norwegian parakeldyshite are deposited in the Mineralogical-Geological Museum, University of Oslo.

Occurrence

Numerous nepheline syenite pegmatites transect a trachytoidal foyaite at Bratthagen, Lågendalen, near Larvik, southern Norway. The foyaite belongs to the igneous rock complex of the Oslo Region. The mineralogy of the pegmatites was described by Oftedal & Sæbø (1963) and Sæbø (1966a,b). Parakeldyshite occurs as irregular, cleavage masses up to several cm in size. It is an early crystallization product, probably contemporaneous with alkali feldspar and nepheline, all of which are in general later than aegirine. Other associated phases include pyrophanite, loparite and biotite. Astrophyllite, catapleiite, ramsayite, analcime and late-formed zeolites are typical of other parts of the pegmatite dykes. Also present are minor boehmite, genthelvite, eudialyte, barylite, hilairite, ancylite and some as yet unidentified phases.

Parakeldyshite is inconspicuous on a freshly broken surface, appearing, with its white color and polysynthetic twinning, very much like plagioclase. It is, however, easily detected by its fluorescence and a very pronounced weathering on exposed surface, as described below.

X-Ray Crystallography

Weissenberg and precession studies showed triclinic symmetry for parakeldyshite, space group probably $P\bar{1}$. Indexed X-ray powder data are given in Table 1, together with the latest powder data for parakeldyshite from Khibina (Khomyakov *et al.* 1975). The doublet at 2.913/2.905Å was barely resolved on a Guinier film, and will appear as a single line of strongest intensity on ordinary powder photographs. The indexing was performed partly with the aid of the single-crystal photographs and partly through repeated cycles of cell constant refinements and calculations of theoretical *d*-values.

The refined cell parameters based on 45 unequivocally indexed lines are a 5.419(1), b 6.607(2), c 8.806(2)Å, α 71.50(2)°, β 87.15(3)°, γ 85.63(2)°, V=298.0(3)Å^3. The unit cell chosen by us, before the identity of the Norwegian mineral with parakeldyshite was known, is a Dirichlet reduced cell with all angles acute

TABLE 1. X-RAY POWDER DIFFRACTION DATA FOR PARAKELDYSHITE

Lågendalen, Norway[1]				Khibina, Kola, USSR[2]	
hkl	d_{calc}Å	d_{obs}Å	I_{est}	d_{obs}Å	I/I_o
001	8.35	8.37	2	8.33	8
011	5.99	6.00	6	5.99	11
100	5.40	5.40	1		
				4.65	5
101	4.592	4.601	3	4.59	16
$\bar{1}01$	4.479	4.483	2	4.48	5
$0\bar{1}1$	4.385	4.380	2	4.38	9
110	4.223	4.234	7	4.22	50
002	4.173	4.179	8	4.17	100
012	4.121	4.127	3	4.12	13
$1\bar{1}0$	3.964	3.963	10	3.96	70
$\bar{1}\bar{1}1$	3.456	3.458	4	3.46	13
021	3.287	3.292	2	3.30	2
				3.20	4
$\bar{1}12$	3.173	3.175	2	3.17	6
020	3.126	3.129	2	3.13	5
$0\bar{1}2$	3.055	3.055	2	3.05	9
022	2.995	2.989	1	2.98	4
121	2.910	2.913	9	2.91	45
013	2.904	2.905	5		
120	2.784	2.785	4	2.784	13
$\bar{1}21$	2.715	2.718	6	2.718	25
122	2.713				
200	2.701	2.703	7	2.704	35
$\bar{1}\bar{1}2$	2.672	2.676	5	2.671	36
$1\bar{1}2$	2.646	2.646	4	2.645	28
113	2.622	2.623	1		
201	2.590	2.589	1	2.591	2
211	2.540	2.543	1	2.541	2
103	2.501	2.503	2	2.499	9
$\bar{1}03$	2.446	2.450	2	2.444	11
$\bar{1}\bar{2}1$	2.434	2.438	1		
$2\bar{1}1$	2.269	2.270	3	2.268	12
$\bar{1}23$	2.211	2.212	1		
014	2.198	2.200	4	2.197	18
$\bar{2}12$	2.191	2.192	1		
031	2.190				
221	2.171	2.173	2	2.172	7
220	2.111	2.111	1	2.111	5
004	2.087	2.086	3	2.085	14
131	2.084				
030	2.084				
024	2.061	2.062	1		
132	2.059				
213	2.037	2.041	1	2.036	4
$\bar{1}14$	2.000	2.005	1	2.013	7
033	1.996	1.999	1		
$\bar{2}20$	1.982	1.983	5	1.982	19
104	1.964	1.965	2	1.965	9
$\bar{2}\bar{2}1$	1.942	1.943	1	1.942	4
133	1.924	1.925	3	1.926	11
$\bar{2}13$	1.923				
$1\bar{3}0$	1.904	1.906	1		
$03\bar{1}$	1.887	1.889	2	1.887	7
034	1.781	1.782	1	1.782	3
301	1.770	1.770	1		
311	1.764	1.765	1	1.767	4
015	1.756	1.757	1	1.756	7
232	1.754				
214	1.749	1.749	1	1.746	8
$\bar{2}\bar{1}3$	1.747				
$\bar{1}23$	1.730	1.730	1	1.730	6
$\bar{2}\bar{2}2$	1.728				
025	1.714	1.716	1	1.717	7
$3\bar{1}0$	1.701	1.704	4	1.704	12
$3\bar{1}1$	1.684	1.685	1		
$2\bar{2}2$	1.677	1.678	1	1.678	4
233	1.671	1.669	2	1.668	9
302	1.670				
005	1.669				
$03\bar{2}$	1.666				
042	1.643	1.644	1		
$\bar{2}31$	1.643				
321	1.633	1.635	2	1.633	5
				+ 17 lines to 1.337	

1. Guinier quadruple focusing camera, 22.9 cm diameter, quartz monochromator, Fe$K\alpha$ radiation (λ = 1.93728 A), lead nitrate as internal standard, d_{calc} obtained from a = 5.419, b = 6.607, c = 8.806 A, α = 71.50, β = 87.15, γ = 85.63°.
2. Khomyakov *et al.* 1975. Diffractometer, 0.5 deg/min, Fe radiation, Mn filter.

TABLE 2. CELL PARAMETERS FOR PARAKELDYSHITE WITH TRANSFORMATION MATRICES

	1	2	3
a	6.66 A	9.31 A	5.419(1) A
b	8.83	5.42	6.607(2)
c	5.42	6.66	8.806(2)
α	92°45'	94°15'	71.50(2)°
β	94 15	115 20	87.15(3)
γ	72 20	89 35	85.63(2)
V		302.8 A^3	298.0(3) A^3
Z	2	2	2
Space group	$P\bar{1}(P1)$	$P\bar{1}$	$P\bar{1}(P1)$

Transformation matrices, 1 to 2: $\bar{1}10/001/100$
1 to 3: $001/\bar{1}00/0\bar{1}0$
2 to 1: $001/101/010$
2 to 3: $010/00\bar{1}/\bar{1}0\bar{1}$
3 to 1: $0\bar{1}0/00\bar{1}/100$
3 to 2: $01\bar{1}/100/0\bar{1}0$

1. Lovozero, Kola, USSR. Khomyakov *et al.* 1969, Voronkov *et al.* 1970 (β = 94°13'); Khomyakov & Voronkov 1973 (β = 94°15'); b and c interchanged in Bussen *et al.* 1972.
2. Lovozero, Kola, USSR. Sizova *et al.* 1974 with a and b interchanged; Khalilov *et al.* 1975, Khomyakov *et al.* 1975.
3. Lågendalen, Norway. The chosen cell has all angles acute.

(Balashov 1956; Balashov & Ursell 1957). This is only slightly different from the first unit cell quoted for parakeldyshite from Lovozero (Table 2). However, a new 'rational' orientation was preferred in later descriptions of the mineral (Table 2), one which is more logically related to the structure and displays the relationship with khibinskite, monoclinic $K_2ZrSi_2O_7$ (Sizova *et al.* 1974; Khomyakov *et al.* 1975; Chernov *et al.* 1970). We have not found it expedient to change our original choice of unit-cell orientation. Transformation matrices between the different systems are given in Table 2.

The structure consists of double-tetrahedra $Si_2O_7^{6-}$ coupled with isolated octahedra of Zr, with Na atoms in eight-coordination occupying holes in the three-dimensional network (Voronkov *et al.* 1970; Sizova *et al.* 1974; Sizova *et al.* 1975).

Physical Properties

Parakeldyshite is white with a slight bluish tinge, translucent, and has a vitreous lustre. It shows a strong cream fluorescence in short-wave UV light, only a very weak cream fluorescence in long-wave UV light, and no discernible phosphorescence. The hardness is 5½-6. Measurements of specific gravity gave 3.37(2) with a Berman balance, and 3.39 with a pycnometer using 1.5 g which were also used for the chemical analysis. The latter figure is probably the more accurate.

Three cleavage directions are prominent, resulting in pseudo-rhombohedral cleavage fragments. This is consistent with the trigonal pseudo-symmetry displayed by the structure (Sizova *et al.* 1974; Khomyakov *et al.* 1975). The cleavage is perfect along $\{001\}$ and somewhat less perfect along $\{110\}$ and $\{1\bar{1}0\}$. The acute angles between these three cleavage planes were roughly 74-80° on a reflection goniometer, the calculated values being: $(00\bar{1}) \wedge (110) = 76.48°$, $(001) \wedge (1\bar{1}0) = 79.64°$, $(110) \wedge (1\bar{1}0) = 81.63°$. There also seems to be a good $\{011\}$ cleavage. These four cleavage directions are consistent with those reported by Khomyakov *et al.* (1975).

Universal-stage measurements of the four cleavages and at least three sets of polysynthetic twinning planes were most intricate and could not be resolved satisfactorily. Khomyakov *et al.* (1975) have described polysynthetic twinning on (011), $(\bar{3}21)$, $(1\bar{1}0)$, and for synthetic material, $(\bar{1}12)$ (all indices refer to our axial system). These composition planes do not seem to fit with our measurements. Polysynthetic twinning on (100), however, is in our case thought to be well-established.

Parakeldyshite is biaxial negative, $2V$ (meas.) = 84°. The optical orientation is Y near c, X near b and Z near a. The refractive indices α' 1.670, β' 1.692, γ' 1.713 (all ±0.002) were measured in white light on cleavage fragments, and because of the optical orientation they are probably close to the true values for α, β and γ. $2V\alpha$ calculated from these indices is 88°.

Chemical Composition

Three wet-chemical analyses and the empirical formulae of parakeldyshite are given in Table 3. Based on 2(Si+Al) the formulae are close to the ideal $Na_2ZrSi_2O_7$. The small amount of H_2O^+ in analysis 3 (Table 3) is attributed to hydronium ions substituting for sodium, as indicated by the infrared spectrum. With Z=2 the calculated density for composition 3 is 3.40 g/cm^3, in good agreement with the measured specific gravity of 3.39. The density calculated from the Gladstone-Dale relationship (Larsen & Berman 1934) is 3.45 g/cm^3, but this is with inaccurate refractive indices (see above).

Infrared Spectrum and DTA

The infrared spectrum is shown in Figure 1. Strong absorptions due to the $Si_2O_7^{6-}$ group occur at 1085, 940 and 875 cm^{-1}. The numerous absorptions at lower frequencies cannot be easily

TABLE 3. CHEMICAL ANALYSES OF PARAKELDYSHITE (WT.%)

	Khibina, Kola, USSR	Lågendalen, Norway	
	1	2	3
SiO_2	38.80	38.88	39.22
Al_2O_3			0.21
ZrO_2	38.70	40.50	40.07
TiO_2	tr.	0.19	0.13
Fe_2O_3	0.29		0.18
MgO			0.03
CaO	3.50	1.55	0.27
Na_2O	17.97	18.72	19.33
K_2O	1.13	0.31	0.25
H_2O^+	0.00		0.48
H_2O^-	0.00		0.00
Total	100.39	100.15	100.17

1. Analyst: M.E. Kazakova (Khomyakov *et al.* 1975; Phase II).

2. Analyst: M.E. Kazakova (Khomyakov, pers. comm. Oct. 1975).

3. Analyst: B. Bruun, Mineralogisk-Geologisk Museum, Oslo.

Chemical compositions based on 2(Si+Al):

1. $(Na_{1.796}Ca_{0.193}K_{0.074})_{2.063}(Zr_{0.973}Fe_{0.006})_{0.979}Si_{2.000}O_{7.091}$

2. $(Na_{1.867}Ca_{0.085}K_{0.020})_{1.972}(Zr_{1.016}Ti_{0.007})_{1.023}Si_{2.000}O_{7.076}$

3. $(Na_{1.899}H_3O_{0.054}K_{0.016}Ca_{0.015})_{1.984}$
$(Zr_{0.990}Fe_{0.007}Ti_{0.005}Mg_{0.002})_{1.004}(Si_{1.987}Al_{0.012})_{1.999}O_{6.995}$

interpreted. Most interesting are the small but distinct double absorption peaks at 3580, 3500 cm^{-1} and 1650, 1560 cm^{-1}. This feature must be interpreted either as resulting from water molecules in two different environments, or the low frequency absorptions may be due to hydronium ions substituting for sodium (Wilkins *et al.* 1974).

DTA (DuPont 900 Thermal Analyzer, heating rate 10 deg/min in air) indicates that water is given off below ca. 350°C. Three small endothermic peaks occur at 155, 205 and 310°C. X-ray diffraction of the heated product shows that parakeldyshite is stable up to at least 1200°C.

Alteration Product

Parakeldyshite is extremely susceptible to alteration. The fresh mineral exposed in road cuts for just a few years has acquired a snow-white coating of a secondary product, making the mineral very conspicuous. Parakeldyshite in pegmatites which have been jointed along their strike by tectonic movements (*cf.* Sæbø 1966b, p. 337), is completely altered to the secondary phase.

This secondary product is similar to Phases I-III-IV described by Khomyakov *et al.* (1975) from Khibina. A chemical analysis of the Norwegian mineral by M. E. Kazakova (Khomyakov, pers. comm. Oct. 1975) gave: ZrO_2 40.40, SiO_2 39.73, TiO_2 0.00, CaO 1.57, Na_2O 10.05, K_2O 0.39, H_2O^+ 7.93, H_2O^- 0.00, total 100.07 wt. %. Compared to parakeldyshite, it is obvious that the alteration has involved removal of Na and addition of H_2O, probably in the form of H_3O^+. A more complete description of the secondary phase will be the subject of a separate paper.

As to the presence of 0.48% H_2O^+ in the analysis of parakeldyshite (Table 3), care was taken to select only fresh cleavage fragments under the binocular microscope. Since it is unlikely

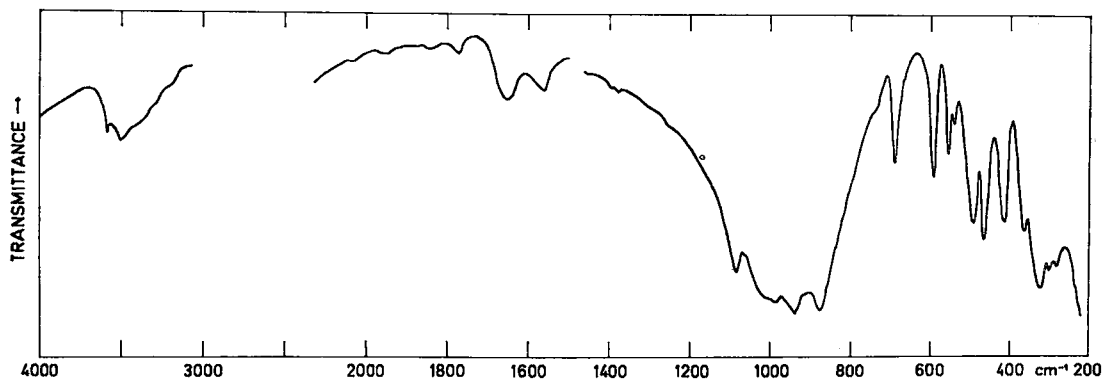

FIG. 1. Infrared spectrum of parakeldyshite from Lågendalen, Norway. Right side (<1500 cm^{-1}): 1.2 mg per 200 mg KBr. Left side (>1500 cm^{-1}): Nujol mull, high sensitivity. Note change in scale at 2000 cm^{-1}.

that the analytical substance was contaminated with 6% of the secondary mineral, it is concluded that the small amount of water must be in parakeldyshite.

The decomposition of parakeldyshite under hydrothermal conditions at 250°C was investigated by Polezhaev *et al.* (1975), who found that the products were tetragonal ZrO_2 and zircon.

Discussion

The synthesis of parakeldyshite has been reported by several authors; apparently the first report is by D'Ans & Löffler (1930). Phase 'Z4' of Christophe-Michel-Lévy (1961), synthesized at 530-550°C, is identical to parakeldyshite. Baussy *et al.* (1974) reported the hydrothermal synthesis of parakeldyshite at 550-700°C and 700 bars in a slightly silicic medium; at lower temperatures catapleiite was formed.

Parakeldyshite is confined to foyaite rocks both in Norway and the USSR, and is in both cases associated with ramsayite, $Na_2Ti_2Si_2O_9$. Parakeldyshite seems to be typical of strongly sodic agpaitic rocks. In more potassium-rich rocks from Khibina, parakeldyshite occurs as inclusions in khibinskite, $K_2ZrSi_2O_7$ (Khomyakov *et al.* 1974).

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