

PETARASITE, A NEW HYDRATED SODIUM ZIRCONIUM HYDROXYCHLOROSILICATE MINERAL FROM MONT ST-HILAIRE, QUEBEC

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ABSTRACT

The new mineral petarasite occurs as irregular grains in a xenolith in the nepheline syenite at Mont St-Hilaire, Quebec. The xenolith is composed predominantly of biotite and microcline with minor amounts of catapleiite, apatite, zircon and other accessory minerals. The mineral is monoclinic, $P2_1/m$, a 10791(4), b 15.505(5), c 6.626(2) Å, β 113.21(3)°. The strongest eight lines of the X-ray diffraction pattern [$d_{hkl}(I)$ (hkl)] are: 7.25(70)(020), 6.09(40)(001), 4.96(15)(200), 4.10(100)(220), 3.220(30)(310, 20 $\bar{2}$, 11 $\bar{2}$), 3.041(30)(002), 2.924(100)(240, 24 $\bar{1}$), 1.729(20)(55 $\bar{1}$, etc.). The mineral is greenish yellow with white streak, transparent to translucent with vitreous lustre. It has perfect {110}, very good {010} and distinct {001} cleavages. Hardness 5-5½; fracture subconchoidal. Density 2.88(1)(meas.), 2.915 g/cm³ (calc.). Optically, the mineral is biaxial (+), α 1.596(1), β 1.598(1), γ 1.632(1) (in Na light), $2V$ meas 29°, calc 27.7°. Dispersion weak $r < v$. Pleochroism: X colorless, $Y=Z$ pale greenish yellow. Absorption formula: $X < Y = Z$. Orientation: $X=b$, $Z \wedge c = +41.5^\circ$. Electron microprobe analyses correspond to $(Na_{4.69}Ca_{0.13}K_{0.05})(Zr_{2.01}Ti_{0.01})Si_6O_{18}[(OH)_{0.60}Cl_{0.48}] \cdot 3.01H_2O$, whereas the ideal structural formula is $Na_5Zr_2Si_6O_{18}(Cl,OH) \cdot 2H_2O$, $Z=2$. Excess water shown in the empirical formula is due to adsorbed water. TGA and DTG show two stages of weight loss at 25-830 and 830-1100°C. The mineral decomposes to parakeldyshite after heating to 1100°C. Infrared absorption spectra confirm the presence of structural and adsorbed water.

Keywords: new mineral, hydrated, sodium, zirconium, hydroxychlorosilicate, Mont St-Hilaire, Quebec.

SOMMAIRE

On trouve la petarasite, espèce nouvelle, en cristaux irréguliers dans un xénolithe de la syénite néphélinique du mont St-Hilaire (Québec). Ce

xénolithe consiste en biotite et microcline, avec quelques minéraux accessoires: catapléiite, apatite, zircon et autres. La petarasite est monoclinique, $P2_1/m$, a 10.791(4), b 14.505(5), c 6.626(2) Å, β 113.21(3)°. Les huit raies les plus intenses du cliché de poudre [$d_{hkl}(I)$ (hkl)] sont: 7.25(70)(020), 6.09(40)(001), 4.96(15)(200), 4.10(100)(220), 3.220(30)(310, 20 $\bar{2}$, 11 $\bar{2}$), 3.041(30)(002), 2.924(100)(240, 24 $\bar{1}$), 1.729(20)(55 $\bar{1}$, etc.). Elle est d'un jaune verdâtre, à la rayure blanche, transparente à translucide et à l'éclat vitreux. Clivages: {110} parfait, {010} excellent, {001} distinct; dureté: 5-5½, fracture subconchoïdale. Densité: 2.88(1)(mes.), 2.915 (calc.). Optiquement, la petarasite est biaxe (+), α 1.596(1), β 1.598(1), γ 1.632(1) en lumière Na; $2V$ 29° (mes.), 27.7° (calc.); faible dispersion $r < v$; pléochroïsme: X incolore, $Y=Z$ jaune verdâtre pâle; formule d'absorption $X < Y = Z$. Orientation: $X=b$, $Z \wedge c = +41.5^\circ$. Les analyses à la microsonde électronique donnent $(Na_{4.69}Ca_{0.13}K_{0.05})(Zr_{2.01}Ti_{0.01})Si_6O_{18}[(OH)_{0.60}Cl_{0.48}] \cdot 3.01H_2O$, tandis que la formule idéale est $Na_5Zr_2Si_6O_{18}(Cl,OH) \cdot 2H_2O$, $Z=2$. Le surplus d'eau dans la formule empirique est dû à l'adsorption. Les spectres ATG et TGD indiquent une perte de poids en deux stades, de 25 à 830 et de 830 à 1100°C; après chauffage à 1100°C, on observe la décomposition en parakeldyshite. Les spectres d'absorption infrarouge confirment la présence de l'eau structurale et de l'eau adsorbée.

(Traduit par la Rédaction)

Mots-clés: espèce minérale nouvelle, hydroxychlorosilicate, hydraté, sodium, zirconium, mont St-Hilaire, Québec.

INTRODUCTION

The new mineral petarasite, $Na_5Zr_2Si_6O_{18}(Cl, OH) \cdot 2H_2O$, formerly known as UK42 (Chao & Baker 1979), was first collected in the summer of 1977 from the Demix quarry, Mont St-Hilaire, Quebec. The mineral occurs in a xenolith in the nepheline syenite as irregular grains up

TABLE 1. PROPERTIES OF PETARASITE AND RELATED MINERALS

	Petarasite Na ₅ Zr ₂ Si ₆ O ₁₈ (Cl,OH) ·2H ₂ O	Catapleite ¹ Na ₂ ZrSi ₃ O ₉ ·2H ₂ O	Gaidonnayite ² Na ₂ ZrSi ₃ O ₉ ·2H ₂ O	Hilaite ³ Na ₂ ZrSi ₃ O ₉ ·3H ₂ O	Parakeldyshite ⁴ Na ₂ ZrSi ₂ O ₇
Space group	<i>P</i> 2 ₁ / <i>m</i>	<i>I</i> 2/ <i>c</i>	<i>P</i> 2 ₁ / <i>n</i> b	<i>R</i> 3̄ <i>m</i> , <i>R</i> 3 <i>m</i> , <i>R</i> 32	<i>P</i> 1, <i>P</i> 1̄
<i>a</i> (Å)	10.791(4)	12.779(9)	11.740(3)	10.556(1)	5.419(1)
<i>b</i>	14.505(5)	7.419(5)	12.820(3)	--	6.607(2)
<i>c</i>	6.626(2)	20.157(8)	6.691(1)	15.851(2)	8.806(2)
α (°)					71.50(2)
β	113.21(3)	90.41(4)			87.15(3)
γ					85.63(2)
Vol. (Å ³)	953.2(4)	1911.0	1007.0	1529.6	298.0 (3)
Z	2	8	4	6	2
D _{meas} (g/cm ³)	2.88(1)	2.80(1)	2.67(1)	2.724(4)	3.39
D _{calc}	2.915	2.79	2.70	2.739	3.40
Hardness	5-5½	5	5	>4	5½-6
α	1.596(1)(Na)	1.588(1)(Na)	1.573(1)(Na)	1.596(1)(Na)	α' 1.670(2)
β	1.598(1)	1.591(1)	1.592(1)		β' 1.692(2)
γ	1.632(1)	1.624(1)	1.599(1)	1.609(1)	γ' 1.713(2)
2 <i>V</i> _z (°)	29(1)	40(1)	121(1)		96
Orientation	X = <i>b</i> ZΔ <i>c</i> = +41.5°	Y = <i>b</i> ZΔ <i>c</i> = 4°	X = <i>a</i> Y = <i>b</i> Z = <i>c</i>		X near <i>b</i> Y near <i>c</i> Z near <i>a</i>
Dispersion	weak r < v				
Pleochroism	X colorless Y pale greenish yellow Z pale greenish yellow	none	none	none	

¹ Mont St-Hilaire, Quebec (Chen & Chao 1973); ² Mont St-Hilaire, Quebec (Chao & Watkinson 1974);
³ Mont St-Hilaire, Quebec (Chao *et al.* 1974); ⁴ Larvik, southern Norway (Raade & Mladek 1977).

TABLE 2. X-RAY POWDER-DIFFRACTION DATA FOR PETARASITE

<i>hkl</i>	<i>d</i> _{calc} Å	<i>d</i> _{obs} Å	<i>I</i> / <i>I</i> ₀	<i>hkl</i>	<i>d</i> _{calc} Å	<i>d</i> _{obs} Å	<i>I</i> / <i>I</i> ₀
020	7.252	7.25	70	442̄	2.032		
001	6.089	6.09	40	003	2.030	2.030	5
111̄	5.889	5.90	5	242	1.901	1.899	10
200	4.959	4.96	15	080	1.813	1.814	5
021	4.664	4.66	10	461̄	1.800	1.800	5
130	4.346	4.34	5	602̄	1.775		
220	4.094	4.10	100	441̄	1.774	1.774	<5
131̄	3.867	3.868	10	443̄	1.755	1.753	<5
311̄	3.449	3.451	10	460	1.731		
310	3.223			621̄	1.731		
202̄	3.222	3.220	30	551̄	1.729	1.729	20
112̄	3.216			133	1.728		
002	3.045	3.041	30	353̄	1.726		
221	2.978	2.981	<5	600	1.653	1.653	5
240	2.927			620	1.612		
241̄	2.916	2.924	100	404	1.611	1.611	10
331̄	2.862	2.861	5	224	1.608		
022	2.807	2.805	5	422	1.593		
401̄	2.698	2.702	5	114	1.593	1.594	10
311	2.482			153	1.560		
400	2.480	2.481	10	082̄	1.558		
402̄	2.453	2.456	5	465	1.543		
151	2.432	2.430	5	553̄	1.543	1.542	5
420	2.346	2.346	10	481̄	1.505		
061̄	2.247			640	1.504	1.505	5
351̄	2.247	2.242	5	643̄	1.491		
203̄	2.208	2.206	<5	024	1.490	1.491	10
260	2.173			551	1.466	1.467	5
261̄	2.169	2.171	10	390	1.449		
441̄	2.164			731̄	1.449	1.447	5
511̄	2.129	2.129	5	192̄	1.448		
223̄	2.112	2.110	5				
440	2.047						
352̄	2.042	2.045	10				

Data obtained with a 114.6 mm camera, using CuK α radiation ($\lambda = 1.5418$ Å) and NBS Si standard. Intensities were estimated visually.

to 10 mm across, in close association with biotite, apatite, catapleite and zircon. The xenolith, measuring approximately 2.5 m in diameter, is

composed mainly of coarse grained biotite and microcline. Locally the biotite content may be as high as 90%. The minor and accessory minerals in the xenolith are natrolite, nepheline, cancrinite, eudialyte, aegirine, altered mosandrite, analcime, fluorite, sodalite, lavenite, astrophylite, pyrochlore, calcite, rhodochrosite, arfvedsonite, galena and pyrrhotite.

The mineral is named after Dr. Peter Tarasoff, an amateur mineralogist from Dollard-des-Ormeaux, Quebec. Both the new mineral and its name have been approved by the Commission on New Minerals and Mineral Names, I.M.A. The type specimens are deposited in the collections at the National Museum of Natural Sciences, Ottawa (43721, 43630, 43631) and at the Royal Ontario Museum, Toronto (M36456, M36457, M36458).

X-RAY CRYSTALLOGRAPHY

Precession and Weissenberg X-ray photographs of petarasite show the mineral to be monoclinic. The systematic extinctions observed are consistent with space groups *P*2₁/*m* and *P*2₁, but a strong pseudo *C*2/*m* symmetry is indicated by the weakness or absence of the *hkl*-type reflections with $h + k = 2n + 1$. The crystal-structure analysis (Ghose *et al.* 1980)

established $P2_1/m$ as the correct space-group. The cell parameters (Table 1) were obtained from single-crystal photographs and were refined by a least-squares method using X-ray powder-diffraction data (Table 2).

PHYSICAL AND OPTICAL PROPERTIES

Petarasite is commonly light greenish yellow (ISCC-NBS color designation No. 101), brilliant greenish yellow (No. 98), dark greenish yellow (No. 103) and less commonly moderate yellow (No. 87), dark yellow (No. 88) and light yellowish brown (No. 76). The streak is white and the lustre is vitreous. Small fragments are transparent; larger fragments are transparent to translucent. The mineral has perfect {110}, very good {010} and distinct {001} cleavages. Fracture is subconchoidal. The hardness is about 5-5½ on the Mohs scale. The density of the mineral measured by flotation in a heavy liquid is 2.88(1) g/cm³. The mineral does not fluoresce under short-wave and long-wave ultraviolet lights.

Optically the mineral is biaxial positive with α 1.596(1), β 1.598(1), γ 1.632(1), $2V(\text{meas.}) = 29^\circ$, $2V(\text{calc.}) = 27.7^\circ$. The dispersion is weak with $r < v$. Petarasite is pleochroic with $X = \text{colorless}$, $Y = Z = \text{pale greenish yellow}$ (ISCC-NBS color designation No. 104). The absorption formula is $X < Y = Z$. The optical orientation is $X = b$, $Z \wedge c = +41.5^\circ$. All optical measurements were made in sodium light at room temperature, using a spindle stage and crystals previously oriented by X-ray goniometry. $2V$ was measured by direct observation of the optic axes using crystals mounted to rotate about the optic normal.

The identification of petarasite presents no difficulties. The mineral can be readily distinguished from other hydrous sodium zirconium silicate minerals such as catapleiteite, gaidonayite and hilairite on the basis of physical and optical properties (Table 1).

CHEMICAL FORMULA

The electron-microprobe analyses of petarasite and the atomic proportions calculated on the basis of six Si atoms are given in Table 3. The average of the analyses corresponds to the empirical formula $(\text{Na}_{4.68}\text{Ca}_{0.13}\text{K}_{0.05})(\text{Zr}_{2.01}\text{Ti}_{0.01})\text{Si}_6\text{O}_{18}[(\text{OH})_{0.60}\text{Cl}_{0.48}] \cdot 3.01\text{H}_2\text{O}$, which may be idealized to $\text{Na}_5\text{Zr}_2\text{Si}_6\text{O}_{18}(\text{OH},\text{Cl}) \cdot 3\text{H}_2\text{O}$. The interpretation of the analyses is, however, complicated by the following findings: (1) only two

TABLE 3. ELECTRON MICROPROBE ANALYSES AND ATOMIC PROPORTIONS OF PETARASITE

	1	2	3	4	5	Mean
SiO ₂	43.0	42.4	43.3	43.2	42.6	42.9
ZrO ₂	29.2	29.1	29.7	30.1	29.5	29.5
TiO ₂	0.21	0.10	n.d.	n.d.	0.06	0.07
CaO	1.15	1.01	0.65	0.68	0.92	0.88
Na ₂ O	17.4	17.3	17.2	17.3	17.2	17.3
K ₂ O	0.17	0.26	0.29	0.28	0.25	0.25
H ₂ O	7.06	7.09	7.12	7.10	7.10	7.09
Cl	2.07	2.04	2.01	2.03	2.03	2.04
O=Cl	0.47	0.46	0.45	0.46	0.46	0.46
Total	99.79	98.84	99.82	100.23	99.20	99.57
Si	6	6	6	6	6	6
Zr	1.99	2.01	2.01	2.04	2.03	2.01
Ti	0.02	0.01			0.01	0.01
Ca	0.17	0.15	0.10	0.10	0.14	0.13
Na	4.71	4.75	4.62	4.66	4.70	4.69
K	0.03	0.05	0.05	0.05	0.05	0.05
H	6.57	6.69	6.58	6.58	6.67	6.61
Cl	0.49	0.49	0.47	0.48	0.49	0.48
O	21.60	21.69	21.51	21.59	21.69	21.61

Analyses performed at 20 kV using a defocused beam. Standards and emission lines used were albite(NaK α), NaNbO₃(NaK α , NbZ α), akermanite(CaK α , SiK α , MgK α), orthoclase(KK α), titanite(CaK α , TiK α), zircon(SiK α , ZrL α), synthetic forsterite(MgK α), NaCl(ClK α). Al, Nb, Mg, F were not detected. H₂O by TGA to 1100°C less Cl. Atomic proportions were calculated on the basis of 6 Si atoms.

water molecules were found in the crystal-structure analysis (Ghose *et al.* 1980); (2) as in the chlorapatites (Hounslow 1968, Mackie *et al.* 1972), Cl and OH occupy slightly different sites in the channels, with occupancy factors of 0.75 and 0.37, respectively. By analogy with the apatites, the ideal structural formula for petarasite may, therefore, be simplified to $\text{Na}_5\text{Zr}_2\text{Si}_6\text{O}_{18}(\text{Cl},\text{OH}) \cdot 2\text{H}_2\text{O}$. The excess water shown in the empirical formula can be accounted for as due to adsorbed water; its presence was confirmed by infrared absorption studies. Furthermore, it is probable that part of the excess water may be trapped in the open channels in the structure.

The density calculated from the empirical formula is 2.915 g/cm³; it is 2.852 g/cm³ if two water molecules *per* formula are assumed. The density calculated from the structural formula with Cl and OH normalized to $[\text{Cl}_{0.67}(\text{OH})_{0.33}]$ is 2.854 g/cm³, whereas the observed density is 2.88 g/cm³.

Calculations based on the Gladstone-Dale relationship (Mandarino 1976, 1979) indicate a good compatibility $(1-K_p/K_c = 0.041)$ for the empirical chemical formula, the calculated density and the observed indices of refraction. However, if the normalized structural formula is utilized, the calculations yield a significantly improved compatibility $(1-K_p/K_c = 0.009)$.

THERMAL STUDY

Thermogravimetric analyses (TGA) of petara-

site were made in air at a heating rate of $10^{\circ}\text{C}/\text{min.}$ with 16–20 mg hand-picked samples, using a Fisher Scientific Series 100A TGA System. TGA curves of petarasite show a two-stage weight loss with a distinct break near 830°C. DTG curves show a very slow increase of the rate of weight loss with temperature, reaching a weak maximum at about 400°C. and a second, somewhat sharper maximum at about 1060°C. Because of the discrepancy in the water contents obtained in the initial TGA study and the crystal-structure analysis, five more TGA runs were made, yielding a range of total weight loss (at 1100°C.) of 9.13–9.81% with a mean of 9.43%.

The minimum value 9.13% was used in the analyses in Table 3. The weight losses at the end of the first stage ($25\text{--}830^{\circ}\text{C.}$) were surprisingly constant, ranging from 6.17 to 6.39% with a mean of 6.24%. The weight losses at the end of the second stage ($830\text{--}1100^{\circ}\text{C.}$) appeared to have a wider spread, from 2.74 to 3.61% with a mean of 3.19%. An additional TGA run programmed to hold the sample at 120°C. for 15 hours and then at 750°C. for 15 hours showed that equilibrium was reached after 6 hours at 120°C. , giving a weight loss of 0.70%, and after one hour at 750°C. , giving a cumulated weight loss of 6.41%. The first stage of weight loss, presumably completed at 750°C. when the mineral was heated isothermally, is interpreted as due to the release of molecular water, structural or adsorbed. This was later confirmed by infrared studies and by electron-microprobe analysis for Cl using a crystal fragment heated to 830°C. Consequently, the second-stage weight loss is presumed to be due to the loss of Cl and OH. The fact that

the second-stage weight losses in all TGA runs are higher than the combined weight of Cl and OH in the empirical formula suggests that the true Cl content of the mineral may be higher than that indicated by the electron-microprobe analyses. The average of the second-stage weight loss, 3.19%, corresponds to $[\text{Cl}_{1.67}(\text{OH})_{0.33}]$, which is more in line with the results from the crystal-structure analysis.

Samples of petarasite treated isothermally at 120°C. for 15 hours and at 250°C. for 2 hours give X-ray powder patterns identical to that of the untreated mineral. The powder patterns of the mineral treated isothermally at 450°C. for 2 hours and at 750°C. for 15 hours are identical and differ only very slightly from the pattern of the untreated mineral (notably in the splitting of the 4.10 and 3.220 \AA lines and a better resolution of the $2.110\text{--}2.129\text{ \AA}$ doublet). One sample treated isothermally at 1100°C. for 15 hours and all the TGA end-products gave X-ray powder patterns identical with that of parakeldyshite, $\text{Na}_2\text{ZrSi}_2\text{O}_7$ (Raade & Mladeck 1977). These results show that the $\text{Zr}_2\text{Si}_6\text{O}_{18}$ framework of petarasite is stable at least up to 750°C. The fact that the framework does not break down until Cl is released suggests that Cl is an essential constituent in the mineral.

INFRARED ABSORPTION STUDY

The infrared absorption spectra of petarasite were obtained using a Beckman 4250 spectrometer and pellets containing 1 mg sample and 400 mg KBr. The general features of the spectrum shown in Figure 1 are basically consistent with those of ring silicates (Plyusnina

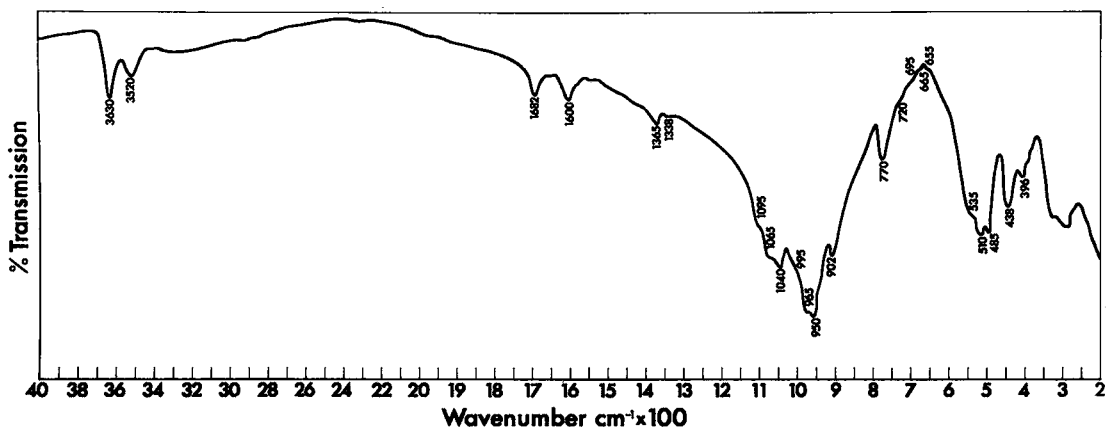


FIG. 1. The infrared absorption spectrum of petarasite.

1961, Strens 1974).

The fairly strong band at 770 cm^{-1} in the petarasite spectrum is well within the range of $740\text{--}830\text{ cm}^{-1}$ for the infrared ring-breathing band characteristic of all ring silicates (Plyusnina 1961, Strens 1974). The absorption bands at 3630 and 3520 cm^{-1} (O-H stretching) and at 1682 and 1600 cm^{-1} (H-O-H bending) confirm the H_2O in the mineral. The broad band centred at about 3300 cm^{-1} is characteristic of the symmetrical and antisymmetrical stretching vibrations of adsorbed water, whereas the lower frequency indicates the formation of moderately strong hydrogen bonds (Strens 1974). Infrared spectra of the mineral heated isothermally for 15 hours at 120 and 750°C , and for 2 hours at 250 and 450°C (Fig. 2) show that (1) most of the adsorbed water is stable against dehydration even at 250°C , and (2) the removal of the molecular water is essentially complete at 450°C . The silicate bands

in the region from 200 to 1200 cm^{-1} are little affected by the heat treatment up to 750°C . The infrared absorption spectrum of the sample heated isothermally at 1100°C for 15 hours shows fundamental differences in both intensities and frequencies of the bands, apparently due to the breaking down of petarasite to parakeldyshite.

Because of the persistence of adsorbed water in the mineral to moderately high temperatures, at which the release of the structural water may already have begun, it is not possible to determine the amount of the adsorbed water quantitatively using the infrared and TGA data.

DISCUSSION

One member of the Commission on New Minerals and Mineral Names, I.M.A., suggested petarasite to be a variety of parakeldyshite. This suggestion is untenable, as the

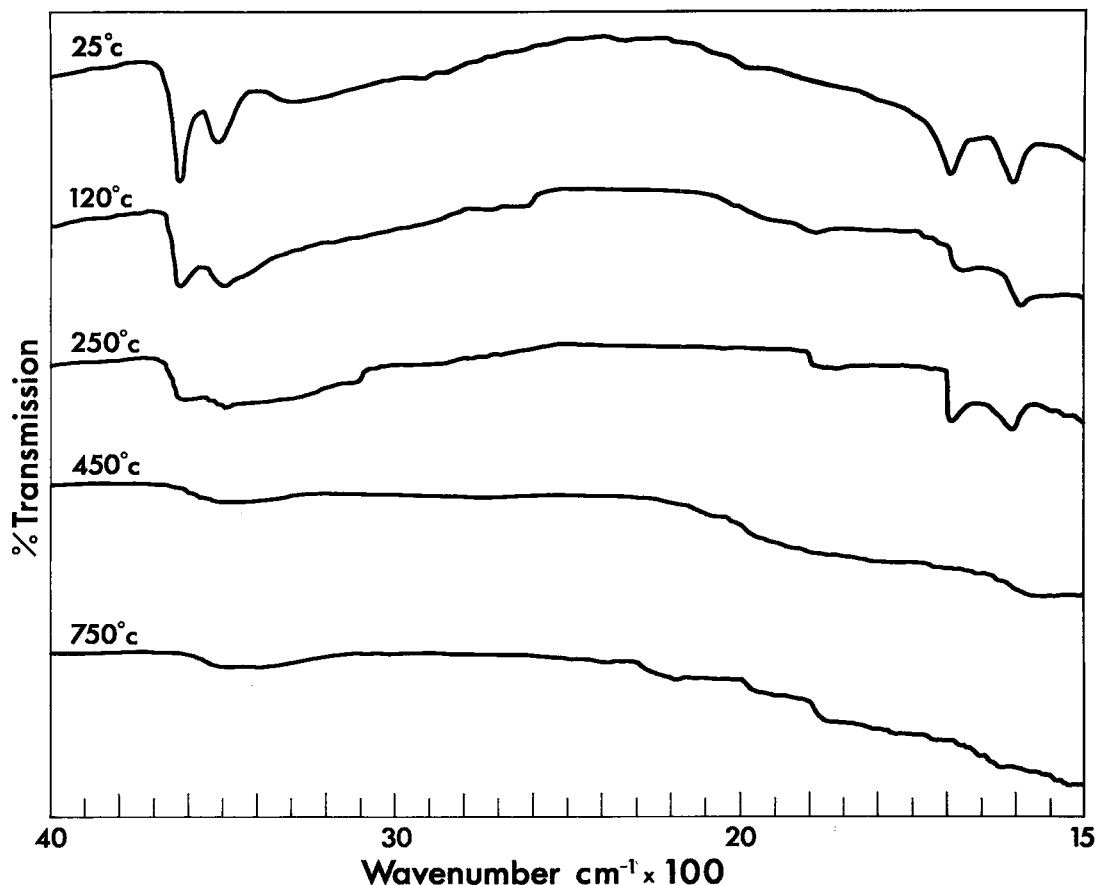


FIG. 2. The infrared absorption spectra of petarasite after heat treatment compared with the spectrum at 25°C .

properties of the two minerals are basically different (Table 2). However, an interesting structural relationship does exist between the two. The framework of the petarasite structure is composed of corner-sharing, six-membered silicate rings linked by ZrO_6 octahedra (Ghose *et al.* 1980), whereas the structure of parakeldyshite is based on layers of mixed six-membered rings formed by two Si_2O_7 groups and two ZrO_6 octahedra (Voronkov *et al.* 1970). The parakeldyshite layers may be derived from the $Zr_2Si_6O_{18}$ framework of petarasite by removing the Si(2) tetrahedra in the six-membered rings (Ghose *et al.* 1980, Figs. 1, 2).

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