

THE RELATIVE STABILITY OF ELPIDITE AND VLASOVITE: A P-T INDICATOR FOR PERALKALINE ROCKS

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ABSTRACT

The univariant reaction $\text{Na}_2\text{ZrSi}_6\text{O}_{15} \cdot 3\text{H}_2\text{O}$ (elpidite) = $\text{Na}_2\text{ZrSi}_4\text{O}_{11}$ (vlasovite) + $2\text{SiO}_2 + 3\text{H}_2\text{O}$ passes through the points 1000 bars, $595 \pm 4^\circ\text{C}$ and 2000 bars, $644 \pm 4^\circ\text{C}$. The reaction curve is unaffected by the presence of excess Na in the vapor phase, but the substitution of 1 N HCl for water decreases the stability field of elpidite by about 20°C . Elpidite is unstable relative to keldyshite $\text{Na}_2\text{ZrSi}_2\text{O}_7$ in highly chlorinated assemblages. Synthetic elpidite has smaller cell-dimensions and a different space-group than natural elpidite, but synthetic vlasovite appears essentially identical to the natural mineral. Elpidite forms from high-temperature hydrothermal fluids, whereas vlasovite forms as a magmatic accessory or by metamorphism of elpidite. The more common occurrence of elpidite, relative to vlasovite, suggests that an aqueous phase separates from peralkaline granitic magma only when crystallization is virtually complete.

Keywords: elpidite, vlasovite, reaction curve, pressure-temperature indicator, peralkaline granite.

SOMMAIRE

La réaction univariante $\text{Na}_2\text{ZrSi}_6\text{O}_{15} \cdot 3\text{H}_2\text{O}$ (elpidite) = $\text{Na}_2\text{ZrSi}_4\text{O}_{11}$ (vlasovite) + $2\text{SiO}_2 + 3\text{H}_2\text{O}$ est en équilibre aux points 1000 bars, $595 \pm 4^\circ\text{C}$ et 2000 bars, $644 \pm 4^\circ\text{C}$. La courbe de réaction n'est pas affectée par la présence d'un excès de sodium dans la phase vapeur, mais le champ de stabilité de l'elpidite décroît d'environ 20°C quand on remplace l'eau par une solution de HCl (1N). L'elpidite est instable vis-à-vis la keldyshite ($\text{Na}_2\text{ZrSi}_2\text{O}_7$) dans les mélanges très riches en chlore. L'elpidite synthétique possède une maille plus petite que celle de l'elpidite naturelle, et un groupe spatial différent, mais la vlasovite synthétique ressemble beaucoup au minéral naturel. L'elpidite se forme à partir de solutions hydrothermales à haute température, tandis que la vlasovite provient d'une phase magmatique accessoire, ou par métamorphisme de l'elpidite. L'elpidite est plus répandue que la vlasovite, ce qui fait penser qu'une phase aqueuse ne se sépare des magmas granitiques hyperalcalins que lorsque la cristallisation tire à sa fin.

Mots-clés: elpidite, vlasovite, courbe de réaction, indicateur de pression et de température, granite hyperalcalin.

INTRODUCTION

In most rocks, the molecular ration $(\text{Na} + \text{K})/\text{Al}$ is less than or equal to 1, and Zr occurs mainly in

accessory zircon. In peralkaline rocks, the ratio $(\text{Na} + \text{K})/\text{Al}$ exceeds 1, zircon may be rare or absent, and Zr appears in a bewildering variety of minerals, including in amphiboles and pyroxenes (up to several percent ZrO_2). Among minerals that may appear as Zr-bearing phases in peralkaline rocks, vlasovite $\text{Na}_2\text{ZrSi}_4\text{O}_{11}$ and elpidite $\text{Na}_2\text{ZrSi}_6\text{O}_{15} \cdot 3\text{H}_2\text{O}$ are of special interest because for stoichiometric compositions, elpidite = vlasovite + 2 quartz + 3 water. Therefore, elpidite would be expected in environments with relatively high activities of silica and water, and vlasovite in environments with relatively low activities of silica and water. However, both minerals, although never found together, apparently occur in similar environments, namely high-level peralkaline granites and peralkaline nepheline syenites. Elpidite occurs relatively commonly in peralkaline granites, for example at Lac Brisson, Quebec (Zajac *et al.* 1984) and Mount Champlain, New Brunswick (R.F. Martin, pers. comm. 1983) in Canada, and also in peralkaline syenites at Mont Saint-Hilaire, Quebec (Chao 1967) and Lovozero, U.S.S.R. (Semenov 1967), as well as in mafic quartz-bearing fenites (Kapustin 1966). Vlasovite has been reported in blocks of high-level peralkaline granite found as inclusions in trachyte on Ascension Island (Fleet & Cann 1967), and from metamorphosed agpaite rocks of amphibolite grade at Kipawa, Quebec (Gittins *et al.* 1973), as well as from nepheline syenites of the Lovozero complex (Semenov 1967). The similarity of the parageneses suggests that some feature other than silica or water activity must control the mutually exclusive character of these minerals. If this controlling factor is pressure or temperature, then the comparatively simple chemical character of the minerals (Semenov 1967) suggests that their relations might give a useful P-T indicator. In order to test this hypothesis, an experimental study was undertaken to determine the relative stabilities of elpidite and vlasovite.

EXPERIMENTAL METHOD

Both elpidite and vlasovite can be readily synthesized (Christophe-Michel-Lévy 1961, Baussy *et al.* 1974), but previous studies produced little useful information on the relative or absolute stabilities of the minerals. Since the natural parageneses suggest that silica activity is not the major factor control-

ling the relations of the minerals, we worked mainly with materials of stoichiometric elpidite composition. We used different mixtures only to evaluate the degree of peralkalinity required to produce Na-Zr silicates rather than zircon in peralkaline compositions.

In all cases the starting materials were dried gels prepared from reagent-grade zirconyl chloride ($ZrOCl_2 \cdot 8 H_2O$) as a source of Zr, sodium-stabilized colloidal silica sol as a source of SiO_2 , and either reagent-grade $NaHCO_3$ or NaCl as a source of sodium. Gels were prepared by dissolving the appropriate weight of zirconyl chloride in 0.1 N HCl and adding the silica sol to it while stirring. For the initial experiments, weighed amounts of NaCl were dissolved in this solution, and the material gelled by adding NH_4OH . Hydrothermal experiments on this material consistently produced keldyshite $Na_2ZrSi_2O_7$ rather than elpidite or vlasovite. We therefore prepared a chloride-free gel. The solution of zirconyl chloride and silica was gelled by adding excess NH_4OH . The gel was dried at $90^\circ C$ for 12 hours and fired at $900^\circ C$ for one hour, which quantitatively removed Cl by volatilization as NH_4Cl . $NaHCO_3$ was then added to the chloride-free mix, and the mixture shaken in an agitator with plastic grinding balls for 30 minutes and heated at $550^\circ C$ for 4 hours to drive off CO_2 . Gels were made up so that the calculated weight of the dried products would be about 15–20 grams. The products were rejected if their weight departed from the calculated weight by more than 0.02 grams. X-ray-diffraction analysis of the chloride-free starting material showed weak lines of baddeleyite. Wet-chemical analysis of this material by S. Courville (Table 1) showed that it corresponded to the stated Zr:Na:Si ratio within analytical error.

The experiments were conducted with standard hydrothermal techniques. About 0.2 grams of starting material were loaded into thin-walled (0.1 mm) gold tube, about 0.02 grams of aqueous phase (distilled water, 1 N HCl, 1 N NaOH) added, and the capsule welded and weighed. Experiments were made in René 41 test-tube bombs with graphite or stainless steel filler-rods. Temperature was measured by chromel–alumel thermocouple inserted into drilled wells in the bombs, and is believed to be maintained within $2^\circ C$ during experiments. Pressure was measured on a 30-cm Heise bourdon gauge, and is

TABLE 1. CHEMICAL COMPOSITION OF STARTING MATERIALS

	Weight	Mol
SiO_2	59.03 %	6.006
Na_2O	10.25	1.010
Zr:O ₂	20.17	1.000
H_2O	10.36	3.514
Cl	0.04	0.006

S. Courville, analyst. Molecular proportions based on Zr = 1.000

TABLE 2. RESULTS OF HYDROTHERMAL EXPERIMENTS

(a) Fluid phase pure water				
Run	P(bars)	T(C)	Reactants	Products
5-3	2026	680	gel	vi + qtz
7-2	2026	660	gel	vi + qtz
9-7	2026	650	gel	vi + qtz
5-4	2026	640	gel	el(+qtz)
X17	2026	635	* el	el(+qtz)
X18	2026	640	* el	el(+qtz)
X20	2026	645	* el	el + vi + qtz
X21	2026	650	* el	el(+qtz)
X22	2026	648	* vi + qtz	vi+qtz
X23	2026	643	* vi + qtz	el + vi + qtz
X24	2026	638	* vi + qtz	el
6-4	1519	640	gel	vi + qtz
8-3	1519	630	gel	vi + qtz
6-3	1519	620	gel	vi(el)
8-2	1519	615	gel	vi(+qtz + el)
8-1	1519	600	gel	el(+vi + qtz)
6-5	1519	600	gel	el(+vi + qtz)
X12	1519	615	* el	el(+qtz)
X13	1519	620	* el	el
X14	1519	625	* el	el + vi + qtz
X16	1519	630	* el	vi + qtz
X26	1519	628	* vi + qtz	el + vi + qtz
X27	1519	623	* vi + qtz	el + vi + qtz
X29	1519	618	* vi + qtz	el(+qtz)
3-6	1013	600	gel	vi(+qtz + el)
4-6	1013	600	gel	vi + qtz
5-8	1013	580	gel	el(+qtz)
5-7	1013	560	gel	el(+qtz)
4-5	1013	550	gel	el(+vi + qtz)
5-6	1013	540	gel	el(+qtz)
3-4	1013	500	gel	el(+qtz)
4-4	1013	500	gel	el(+qtz)
3-2	1013	400	gel	el(+qtz)
4-2	1013	400	gel	el(+qtz)
X5	1013	585	* el	el
X7	1013	590	* el	el(+qtz)
X8	1013	595	* el	el + vi + qtz
X9	1013	599	* el	vi + qtz
X30	1013	600	* vi + qtz	vi + qtz
X31	1013	593	* vi + qtz	el + vi + qtz
X33	1013	588	* vi + qtz	el(+qtz)
6-8	507	580	gel	vi + qtz(+el)
9-2	507	580	gel	el(+qtz)
9-3	507	560	gel	el(+qtz)
7-1	507	560	gel	el(+qtz)
6-6	507	540	gel	el(+qtz)
X1	507	546	* el	el(+qtz)
X2	507	550	* el	el
X3	507	554	* el	el + vi + qtz
X4	507	559	* el	vi + qtz
X35	507	550	* vi + qtz	el
X34	507	557	* vi + qtz	vi + qtz

(b) Fluid phase 1 N NaOH				
Run	P(bars)	T(C)	Reactants	Products
4-10	1013	600	gel	vi + qtz(+el)
3-10	1013	550	gel	el
4-9	1013	550	gel	el
3-9	1013	500	gel	el(+qtz)
4-7	1013	450	gel	el(+qtz)
3-7	1013	450	gel	el(+qtz)

(c) Fluid phase 1 N HCl				
Run	P(bars)	T(C)	Reactants	Products
9-6	2026	700	gel	vi + qtz
8-5	2026	660	gel	el(+vi + qtz)
7-9	2026	640	gel	el(+vi + qtz)
7-7	2026	620	gel	el(+vi + qtz)
X38	2026	640	* el	vi + qtz
X39	2026	630	* el	vi + qtz
X51	2026	625	* el	el + vi + qtz
X40	2026	620	* el	el(+qtz)
X41	2026	640	* vi+qtz	vi + qtz
X42	2026	630	* vi + qtz	vi + qtz
X43	2026	620	* el	el
8-8	1013	660	gel	vi(+el + qtz)
9-4	1013	640	gel	vi + qtz
9-1	1013	620	gel	vi + qtz(+el)
7-5	1013	600	gel	el(+qtz)
X44	1013	590	* el	vi + qtz
X45	1013	580	* el	vi + qtz
X46	1013	570	* el	el(+qtz)
X47	1013	590	* vi + qtz	vi + qtz
X48	1013	58	* vi + qtz	vi + qtz
X53	1013	575	* vi + qtz	el + vi + qtz
X49	1013	570	* vi + qtz	el(+qtz)

(d) Chlorinated mix, fluid phase pure H₂O

Run	P(bars)	T(C)	Reactants	Products
1-1	1013	500	Cl-gel	kl + qtz
1-3	1013	530	Cl-gel	kl + qtz
1-4	1013	540	Cl-gel	kl + qtz
1-7	1013	570	Cl-gel	kl + qtz
1-14	1013	580	Cl-gel	kl + qtz
1-16	1013	600	Cl-gel	kl + qtz
1-18	1013	620	Cl-gel	kl + qtz
1-22	1013	660	Cl-gel	kl + qtz
1-24	1013	700	Cl-gel	vl + kl + qtz
1-26	1013	740	Cl-gel	vl + qtz

Symbols: vl vlasovite, el elpidite, kl keldyshite, qtz quartz. Minor and trace amounts (in parentheses) may be metastable. * denotes an equilibrium experiment.

believed accurate to within 5 bars. At the end of an experiment the bombs were air-quenched. After quenching the capsules were weighed, slit with a razor, and the contents mounted on glass slides for optical and X-ray examination. Only results from capsules that exuded water on puncturing are reported in Table 2. Although elpidite can be optically recognized, we were unable to consistently recognize vlasovite optically, and the identifications given in Table 2 are all based on X-ray powder diffraction. Products of a few experiments were also analyzed by SEM by David Walker to check the Na:Zr ratio of synthetic elpidite relative to natural elpidite from Mont Saint-Hilaire. The ratio is essentially identical in natural and artificial elpidite.

EXPERIMENTAL RESULTS

In order to establish approximately the phase

boundary, a number of synthesis experiments were made by running the starting mix at P-T for 168 hours (Fig. 1). Elpidite and vlasovite can be readily synthesized in neutral, alkaline or acid aqueous fluid phases (Table 2). The equilibrium phase-boundary (Fig. 2) was established by first converting the starting material to elpidite (500° at 1 kilobar for 168 hours) or to vlasovite + quartz (650° at 500 bars for 168 hours). These materials were then used as starting materials for experiments to establish the reversed equilibrium phase-boundary.

The elpidite produced consistently coexists with a variable but small amount of quartz, suggesting either that the mineral is slightly nonstoichiometric, or that the material from which it crystallized departs slightly from elpidite composition. Since the starting mix contains Na₂O, the solid phase will depart from elpidite stoichiometry because of solution in the aqueous phase. The appearance of quartz rather than zircon in the products suggests that Zr must also be quite soluble in the aqueous phase. Watson (1979) concluded that alkali zirconosilicate complexes play a major role in the enhanced solubility of Zr and zircon in peralkaline melts. His conclusions may also be applicable to the peralkaline aqueous solutions of our experiments.

The X-ray diffraction powder-pattern of our synthetic vlasovite agrees well with that in the ASTM file, and there appears to be no difference between the natural and synthetic material. Although synthetic elpidite is easily identified on X-ray-diffraction records, it exhibits either a broad or doublet peak at a *d* of 3.227 Å that is not mentioned in the ASTM

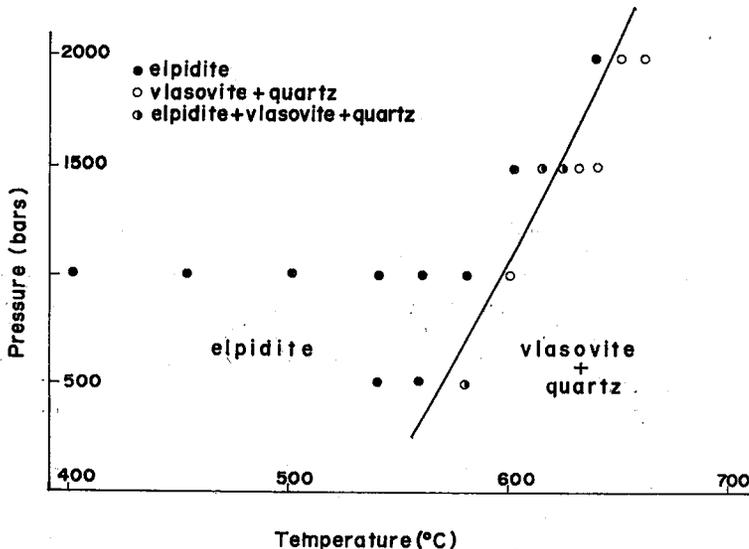


FIG. 1. Data on the synthesis of elpidite and vlasovite from gel of elpidite composition. The curve dividing the fields of elpidite and vlasovite + quartz is taken from Figure 2. Excess water or 1 N NaOH was added.

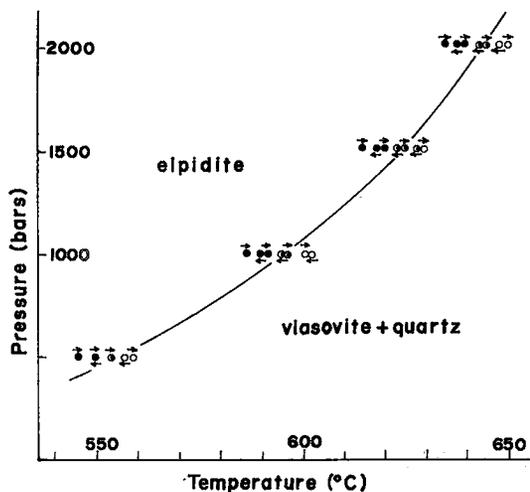


FIG. 2. Experimental data on the univariant reaction elpidite = vlasovite + quartz + water. Symbols are the same as in Figure 1. The arrows indicate starting assemblages, with right-pointing arrows indicating elpidite, and left-pointing arrows indicating vlasovite + quartz. Excess water was added.

powder-data file. A preliminary examination of our data and those of PDF 29-1294 (synthetic elpidite) by J.L. Jambor strongly suggests that the indexing and calculated cell-dimensions of synthetic elpidite in the PDF are in error. The indexing by Jambor is given in Table 3, and the calculated cell-dimensions in Table 4. The d values of our material agree well with the PDF values for synthetic elpidite, but all parameters are significantly smaller than those for natural elpidite. The larger cell-dimensions of natural elpidite may be due to the presence of Ca, as suggested by Zajac *et al.* (1984) and Jambor (pers. comm. 1984) for the Lac Brisson material, although Chao (1967) did not report Ca in the Mont Saint-Hilaire material. Both our synthetic elpidite and the PDF reference material exhibit a diffraction line at 2.82 Å that violates the space-group conditions determined for natural elpidite. The presence of this line indicates that the symmetry of synthetic elpidite differs from that of natural elpidite as determined by Canillo *et al.* (1973). It would be of interest to undertake a structure determination on synthetic elpidite, but we have been unable to grow a crystal sufficiently large for this purpose.

We have synthesized elpidite without difficulty at 400°C, but have not observed catapleite $\text{Na}_2\text{ZrSi}_3\text{O}_9 \cdot 2\text{H}_2\text{O}$, gaidonnayite, dimorph of catapleite, or hilaireite $\text{Na}_2\text{ZrSi}_3\text{O}_9 \cdot 3\text{H}_2\text{O}$, all of which have the same Na:Zr ratio as elpidite. Since experiments and natural parageneses suggest that silica activity is not the controlling factor in the appearance of Na-Zr silicates, these phases must presuma-

TABLE 3. INDEXING OF POWDER-DIFFRACTION DATA FOR ELPIDITE

Synthetic elpidite				Natural elpidite			
2θ	d(Å)	I	hkl	2θ	d(Å)	I	hkl
12.54	7.050	60	100	12.36	7.161	50	100
13.61	6.496	50	021	13.44	6.580	50	021
17.36	5.104	60	022	17.08	5.189	60	022
18.54	4.781	40	121	18.36	4.828	30	121
19.64	4.517	5	013*				
21.52	4.125	40	122	21.22	4.102	40	122
22.38	3.969	10	023				
22.86	3.863	30	131*				
24.89	3.575	30	040	24.28	3.662	5	040
27.53	3.237	50	042	27.16	3.280	90	042
27.58	3.231	50	140	27.32	3.260	90	140
27.93	3.192	50	024				
28.30	3.150	20	202	27.85	3.201	10	202
28.86	3.091	60	221	28.51	3.129	50	221
30.65	2.914	50	142	30.06	2.970	20	142
31.68	2.821	30	203*				
33.62	2.663	20	143	32.88	2.721	10	143
35.37	2.536	40	044	34.52	2.597	10	044
				35.06	2.557	30	240
				37.24	2.412	30	242
43.83	2.063	40	244	43.08	2.098	20	244
45.32	1.999	30	064	44.42	2.037	40	064
47.12	1.927	40	164	46.30	1.959	30	164
48.98	1.858	20	065*				
52.48	1.742	20	264	51.79	1.774	40	264
58.38	1.579	40	441	57.58	1.594	30	441

* Indices marked with this symbol are not permitted in the structure for natural elpidite determined by Canillo *et al.* (1973). Indexing by J.L. Jambor on synthetic elpidite produced in experiment 4-4 and natural elpidite from Lac Brisson on the Quebec-Labrador border.

TABLE 4. CALCULATED CELL-DIMENSIONS FOR ELPIDITE

Synthetic	Natural				
	Run 4-4	PDF29-1294	Lac Brisson	Mt. St-Hilaire	
a (Å)	7.04 (0.012)	7.06	7.12 (0.009)	7.14	
b	14.55 (0.021)	14.54	14.67 (0.017)	14.68	
c	14.22 (0.028)	14.21	14.67 (0.026)	14.65	

Data for synthetic elpidite calculated for product of experiment 4-4 and taken from ASTM Powder-Diffraction File 29-1294. Data for natural elpidite from Lac Brisson by J.L. Jambor (pers. comm.) and for mont St-Hilaire from Canillo *et al.* (1973). Standard error in parentheses.

bly be low-temperature minerals, difficult to nucleate, or controlled by factors not present in the simplified experimental system (or combinations of these). The low-temperature stability limit of elpidite was not defined by the experiments reported here. The upper stability-limit of elpidite is well defined, lying between 550 and 557°C at 500 bars, 591 and 599°C at 1000 bars, 620 and 630°C at 1500 bars, and 640 and 648°C at 2000 bars. Vlasovite and elpidite occur together over a range of about 5° near the phase boundary. This may be due to small fluctuations in temperature ($\pm 2^\circ$), or to slight solid-solution and nonstoichiometric phenomena. We did not investigate the stability of the assemblage vlasovite + quartz beyond 700°, but Gittins *et al.* (1973) reported vlasovite to be stable to at least 800°C.

The formula of elpidite is written showing molecu-

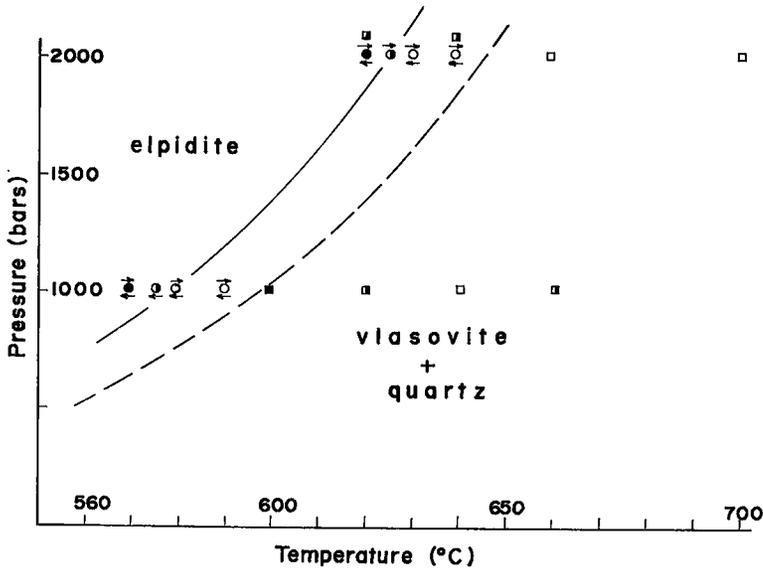


FIG. 3. Experimental data on the relative stability of elpidite and vlasovite + quartz in the presence of 1 N HCl. Circles indicate products made from elpidite or vlasovite + quartz starting materials (same symbols as Fig. 2). Squares indicate products made from gel. The broken line is the equilibrium curve from Figure 2. The solid line is the same curve transposed 20°C to lower temperature. Note the discrepancy between synthesis and equilibrium experiments at 1013 bars.

lar water, not hydroxyl ion. The effect of other molecules in the water site may be a significant factor affecting the stability of elpidite. Substitution of 1 N HCl for pure water raises the maximum temperature at which elpidite appears in synthesis experiments by about 20°C (Fig. 3) but depresses the equilibrium curve by about the same amount. This depression could be qualitatively accounted for by depression of the activity of water due to dilution with HCl. The discrepancy in behavior between synthesis and equilibrium suggests that this dilution also has significant kinetic effects.

Use of strongly chlorinated mixes using NaCl as a source of Na rather than NaHCO_3 completely suppressed the appearance of elpidite in favor of keldyshite + quartz. A transition from keldyshite + quartz to vlasovite occurs at some poorly defined temperature above 700°C. Since keldyshite contains no site for chlorine, and can be synthesized from anhydrous starting materials (Baussy *et al.* 1974), the appearance of keldyshite in these experiments must be related to some control of silica or water activity exercised by the Cl ion. The presence of excess Na in the fluid has no obvious effect on the stability relations (Table 1), but mixes with Na:Zr less than 2 produce zircon in addition to sodium zirconosilicates, whereas mixes with Na:Zr less than about 1.3 produce only zircon.

IMPLICATIONS FOR PETROGENESIS

The stability range of stoichiometric elpidite lies below the water-saturated solidus of granitic rocks, except at pressures above 2 kilobars. Since most known examples of natural elpidite come from relatively high-level plutons, the experimental data imply that elpidite develops at the hydrothermal stage, after solidification of the magma. Our experimental data agree with observations that most natural elpidite occurs in miarolitic cavities or vug-like masses. Our data are not directly applicable to the temperature of formation of Ca-rich elpidite, which may possibly form at magmatic temperatures. Elpidite underwent alteration, to hilaireite, gaidonnayite and lemoyneite at Mont Saint-Hilaire, and to gittinsite $\text{CaZrSi}_2\text{O}_7$, at Lac Brisson. These minerals must have stability fields at lower temperature than elpidite. Our data imply that these stability fields lie below 400°C.

Since vlasovite + quartz forms the high-temperature equivalent of elpidite, vlasovite might be expected to appear as a magmatic accessory phase in elpidite-bearing peralkaline granites. Such an occurrence has never been reported. This apparent anomaly can be explained in several ways. Firstly, vlasovite may have simply been overlooked where it occurs in accessory or trace amounts because it is

difficult to recognize optically. We believe this explanation to be unlikely. Secondly, vlasovite may have been originally present, but have since been converted to elpidite. We are aware of no evidence, such as elpidite pseudomorphic after vlasovite, or elpidite with a vlasovite core, which would support such a mechanism. We believe that the available evidence strongly suggests that vlasovite rarely or never occurs in elpidite-bearing granites. Low concentration and high solubility of Zr in peralkaline melts (Watson 1979) could retard nucleation of a Zr-rich phase until the separation of an aqueous phase relatively rich in Zr. The almost exclusive presence of elpidite in peralkaline granites suggests that such a free aqueous phase only separated at temperatures on or near the solidus. The rare occurrence of vlasovite requires either unusually high concentrations of Zr in a peralkaline melt or metamorphism of an elpidite-bearing assemblage.

The easy synthesis of Na-Zr silicates, their apparent independence of silica activity, and their widespread occurrence in peralkaline rocks suggest that they offer the possibility of a detailed subdivision of P-T-f(H₂O) space. We intend to continue experimental investigation of this subdivision.

ACKNOWLEDGEMENTS

We are indebted to J.L. Jambor, not only for indexing the powder patterns, but also for helpful discussions on the occurrence of elpidite. G.M. LeCheminant assisted with the X-ray powder patterns. J. Graves made some initial experiments.

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Received December 17, 1984, revised manuscript accepted May 3, 1985.