its original Na. K losses were also markedly different as the bead enclosed in a capsule lost 7.8 % of its original K whereas the bead not enclosed in a capsule lost 23.8 % of its original K. It is unknown why the bead enclosed in the capsule loses a higher percentage of K than Na.

Due to the small sample weight (7 mg) and small sample:metal ratio ($\sim$0.4) used, Fe losses were very high (22.2–22.8 % of the original Fe was lost). The bead size used in future experiments must be much larger such that the percentage of total Fe lost is considerably less. Corrigan and Gibb (1979) using beads with weights of approximately 60 mg showed that the percentage of total Fe lost during a 40-hour run would be about 6 % and could be even further reduced to less than 2 % if larger sample:metal ratios of around 10 were used. Also increasing the bead size in a capsule would mean that the percentage loss of initial alkalis would be even further reduced as Corrigan and Gibb (1979) showed that the percentage of original Na lost from a basaltic melt decreased as the mass of the sample increased. There are, however, practical limitations to the bead size if it has to go in a capsule, which might prevent attainment of acceptably small Fe losses unless very large capsules (and furnaces) were used. It has been assumed that enclosing a sample bead in a capsule will still permit f_{O_2} equilibration, however, it is possible that the sample may not achieve the correct Fe^{2+}/Fe^{3+} ratio. As the two samples showed no difference in their colour it seems likely that enclosure of a bead in a capsule (which is only crimped) does not affect the Fe^{2+}/Fe^{3+} ratio. The samples were too small for the post experimental determination of this ratio by wet chemical methods.

Conclusions. The experimental results indicate that alkali losses from silicate melts during experiments using the wire-loop technique can be reduced by running the sample bead in a crimped container. It is, therefore, recommended that when using the wire-loop technique on alkali-bearing samples the beads are run inside Pt or $Ag_{40}Pd_{60}$ capsules using sample:metal ratios of at least 10 to keep Fe losses to acceptable levels (Corrigan and Gibb, 1979).

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Synthesis of $Li_2ZrSi_6O_{15}$, a zektzerite-related phase

CONSIDERABLE recent interest has been shown in a group of double-chain silicate phases which includes the new mineral zektzerite, $LiNaZrSi_6O_{15}$ (Dunn *et al.*, 1977; Ghose and Wan, 1978), and a family of isotypic phases (Marr and Glasser, 1979*a* and *b*). These phases are closely related structurally to $Na_2Mg_2Si_6O_{15}$ (Cradwick and Taylor, 1972), tuhualite, $(Na,K)_2Fe_2^{2+}Fe_2^{3+}Si_{12}O_{30}.H_2O$ (Merlino, 1969), and emeleusite, $Na_2LiFeSi_6O_{15}$ (Upton *et al.*, 1978). All contain a corrugated, double-chain silicate anion, $Si_6O_{15}^{6-}$ and with a variety of possible cation combinations to balance the charge on the anion, viz.

$Li^+ + Na^+ + Zr^{4+}$	zektzerite
$2Na^+ + 2Mg^{2+}$	$Na_2Mg_2Si_6O_{15}$
$(Na,K)^+ + Fe^{2+} + Fe^{3+}$	tuhualite
$2Na^+ + Li^+ + Fe^{3+}$	emeleusite

All have similar-sized, *C*-centred orthorhombic unit cells.

Here we report the synthesis of $Li_2ZrSi_6O_{15}$. It is monoclinic, $\beta = 89.5°$, but has a pseudo-

TABLE I. *X-ray powder diffraction data for* $Li_2ZrSi_6O_{15}$

$d_{obs.}$(Å)	$d_{calc.}$(Å)	$I_{vis.}$	*hkl*	$d_{obs.}$(Å)	$I_{vis.}$
8.635	8.579	24	$\bar{1}01$	2.472	12
7.197	7.166	64	101	2.426	10
6.579	6.551	17	$\bar{1}11$	2.407	5
5.541	5.528	21	002	2.390	5
5.485	5.472	24	$\bar{2}00$	2.367	17
4.858	4.854	86	012	2.358	21
4.822	4.816	83	210	2.343	17
4.615	4.611	29	021	2.305	7
4.370	4.367	100	$\bar{1}21$	2.182	50
4.290	4.289	79	$\bar{2}02$	2.171	14
4.143	4.140	90	121	2.155	17
3.954	3.951	17	$\bar{2}12$	2.140	24
3.670 b	3.675	31	$\bar{1}22$	2.125	17
	3.664		$\bar{3}01$	2.095	14
3.582	3.583	52	202	2.088	17
3.473	3.474	50	$\bar{1}13$	2.068	10
3.445	3.446	47	$\bar{3}11$	2.051	10
3.308 b	3.318	31	103	2.033	12
	3.294		301	2.013	33
3.233	3.234	83	031	2.007	26
	3.231		130	1.973	31
3.174	3.177	57	$\bar{2}13$	1.919	10
3.156	3.154	83	113	1.902	26
3.131	3.133	83	311	1.895	43
3.037		12	?	1.885	14
2.987	2.988	31	$\bar{1}23$	1.869	10
2.970	2.970	19	$\bar{3}21$	1.856	19
2.925	2.927	69	222	1.834	24
2.878 b	2.885	52	032	1.799	10
	2.877		230	1.788	29
2.858	2.859	19	$\bar{3}03$	1.777	21
	2.856		$\bar{1}32$	1.768	12
2.791	2.793	24	$\bar{2}23$	1.736	31
2.780 b	2.783	26	$\bar{3}22$	1.729	29
	2.777		123	1.712	40
2.761 b	2.764	14	004	1.688	10
	2.763		321	1.679	10
2.726 b	2.728	10	132	1.670	12
	2.728		213	1.644	19
2.698	2.700	7	$\bar{1}14$	1.619	12
2.663 b	2.667	50	014	1.613	12
	2.665		$\bar{2}04$	1.607	17
2.655	2.656	33	$\bar{2}32$	1.585	10
2.639	2.641	43	410	1.552	12
2.536	2.537	38	040	1.533	10
2.521		26		1.527	21
2.489		38		1.521	10
				1.507	10

orthorhombic unit cell of similar size to the orthorhombic cell of $LiNaZrSi_6O_{15}$. It appears to be structurally related to zektzerite and represents yet another variant on this large family of phases.

The new phase, $Li_2ZrSi_6O_{15}$, was synthesized during a study of the phase diagram Li_2O-ZrO_2-SiO_2, full details of which are given elsewhere (Quintana and West, 1981). It crystallized in the presence of liquid as irregularly shaped laths of moderately low birefringence.

From single-crystal rotation and Weissenberg photographs, $Li_2ZrSi_6O_{15}$ appeared initially to have a *B*-centred orthorhombic unit cell of dimensions *a* 14.39, *b* 10.09, and *c* 17.27 Å. Two slightly mis-set crystals appeared to be present, as shown by the doubling of certain spots on the photographs. However, it was not possible to completely index the X-ray powder pattern of $Li_2ZrSi_6O_{15}$ on this unit cell since, for certain pairs of lines, only one set of Miller indices was available. Re-examination of the single-crystal photographs showed the true crystal symmetry to be monoclinic and the doubling of spots to be caused by twinning. This led to a *B*-centred monoclinic cell of dimensions, *a* 14.39, *b* 10.09, *c* 17.27 Å, *β* 89.5°, but as this cell did not meet space group requirements, a smaller, primitive cell was chosen by redefining *a* and *c* to be equivalent to the vectorial directions [220] and [$\bar{2}$20] of the *B*-centred cell. The systematic absences gave a space group $P2_{1/n}$. Indexed powder data are given in Table I. Final cell parameters, determined by least-squares refinement of the first 15 lines of the powder pattern, are *a* 11.121 ± 0.006, *b* 10.146 ± 0.006, *c* 11.235 ± 0.007 Å, *β* 100.26 ± 0.06°.

$LiNaZrSi_6O_{15}$ has a *C*-centred orthorhombic unit cell of dimensions 14.306(5), 17.330(4), and 10.140(3) Å (Dunn *et al.*, 1977) which, after transposing *b* and *c*, are similar to the values of pseudo-orthorhombic $Li_2ZrSi_6O_{15}$. This similarity suggests a close relation between the two structures but a full structure determination would be needed to locate the cations in $Li_2ZrSi_6O_{15}$. Assuming that Zr^{4+} is six-coordinated and that one Li^+ ion is tetrahedral, as in zektzerite, it is not obvious which site is occupied by the second Li^+ ion; in zektzerite the Na^+ ion is in a ten-coordinate site but such a large coordination number is not possible for Li^+. The reduction of the symmetry to monoclinic in $Li_2ZrSi_6O_{15}$ is probably associated with a distortion of the structure so as to provide suitable sites for the Li^+ ions.

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