

**PREPARATION OF  $M^{1+}$ -( $M^{2+}$ -) $M^{3+}$ -SILICATES  
( $M^{1+}$  = Na, K, Rb, Cs;  $M^{2+}$  = Sr, Ba;  $M^{3+}$  = Sc, V, Cr, Fe, In, Y, Yb)  
BY HYDROTHERMAL AND FLUX GROWTH METHODS: FIRST RESULTS**

**Wierzbicka, M., Kolitsch, U. & Tillmanns, E.**

Institut für Mineralogie und Kristallographie, Universität Wien, Geozentrum, Althanstr. 14, A-1090 Wien,  
Austria

e-mail: maria.wierzbicka@univie.ac.at

A study of the system  $M^{1+}$ -( $M^{2+}$ -) $M^{3+}$ -Si-O-H has been started with the aim to synthesize novel microporous and small-pore mixed-framework silicates. The syntheses were carried out by two different methods thus far, hydrothermal growth (220°C, 7 d) and flux growth (1150-850°C, cooling rates 1-2 K/h); the latter method yielding the best results to date. The following compounds could be grown as mostly well developed crystals (\* = crystal structure has been determined/refined from single-crystal X-ray intensity data):  $\text{Cs}_3\text{YSi}_8\text{O}_{19}$  (\*, isotypic with  $\text{Cs}_3\text{ScSi}_8\text{O}_{19}$ , reported by KOLITSCH & TILLMANN, 2004),  $\text{CsFeSi}_2\text{O}_6$  (\*, cubic, isotypic with leucite; see below for details),  $\text{Na}_3\text{YbSi}_2\text{O}_7$  (\*, isotypic with other  $\text{Na}_3\text{REESi}_2\text{O}_7$ ) and  $\text{NaScSi}_2\text{O}_6$  (synthetic jervisite, a clinopyroxene).

Several presently unidentified compounds include an *I*-centred cubic Yb silicate, a pseudo-rhombohedral Cs(?) -Sc silicate, an *F*-centred cubic Sr-Fe compound and others. Interesting byproducts of the hydrothermal syntheses are the following silicates and fluorides:  $\text{RbSi}_2\text{O}_4(\text{OH})$  (BULL & PARISE, 2003),  $\text{KSi}_2\text{O}_4(\text{OH})$ ,  $\text{K}_2\text{NaScF}_6$ ,  $\text{K}_3\text{FeF}_6$ ,  $\text{K}_3\text{CrF}_6$  and  $\text{K}_2\text{SiF}_6$ . Byproducts of the flux growth experiments were mainly molybdates, including  $\text{Rb}_5\text{In}(\text{MoO}_4)_4$  (\*, isotypic with  $\text{Rb}_5\text{Er}(\text{MoO}_4)_4$  and related to the mineral palmierite,  $\text{K}_2\text{Pb}(\text{SO}_4)_2$ ,  $\text{K}_5\text{Y}(\text{MoO}_4)_4$ ,  $\text{Rb}_2\text{Mo}_4\text{O}_{13}$  (\*, isotypic with the triclinic modification of  $\text{K}_2\text{Mo}_4\text{O}_{13}$ ),  $\text{KYb}(\text{MoO}_4)_2$  (isotypic with  $\text{KY}(\text{MoO}_4)_2$ ), as well as several well-known  $\text{REE}_2\text{SiO}_5$ - and  $\text{REE}_2\text{Si}_2\text{O}_7$ -silicates, and  $M^{1+}$ -stabilised, micro-twinned tridymite.

It is noteworthy that well-developed crystals of leucite-type  $\text{CsFeSi}_2\text{O}_6$  could be grown by both preparation methods. The hydrothermally grown crystals ( $a = 13.827(2)$  Å) show an average (Si,Fe)-O bond length of 1.664 Å, which differs considerably from that of material synthesized at 1400°C by a dry solid-state method (1.682 Å; BELL & HENDERSON, 1994), and have a cell edge much longer than that of previous hydrothermally synthesized crystals ( $a = 13.66(3)$  Å; KOPP et al., 1963). The corresponding values of the flux-grown crystals ( $a = 13.839(2)$  Å,  $\langle(\text{Si,Fe})\text{-O}\rangle = 1.670$  Å) are slightly larger than those of our hydrothermally grown crystals. These differences can be attributed to the temperature and other conditions of formation (higher temperatures cause longer cell edges and (Si,Fe)-O bonds).

Financial support by the Austrian Science Foundation (FWF) (Grant P17623-N10) is gratefully acknowledged.

**References**

- BELL, A. M. T. & HENDERSON, C. M. B. (1994): Acta Crystallogr., C50, 1531-1536.  
BULL, I. & PARISE, J. B. (2003): Acta Crystallogr., C59, i100-i102.  
KOLITSCH, U. & TILLMANN, E. (2001): Mineral. Mag., 68, 677-686.  
KOPP, O. C., HARRIS, L. A., CLARK, G. W. & YAKEL, H. L. (1963): Am. Mineral., 48, 100-109.