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HYDROTHERMAL SYNTHESIS AND X-RAY DIFFRACTION ANALYSIS OF CERTAIN BARIUM SILICATES

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The $BaO-SiO_2-H_2O$ system has long attracted attention as a result of the binder properties of barium silicates [1]. In contrast to the analogous calcium system, which has been investigated rather frequently, particularly under hydrothermal conditions [2], only one study has been made of this barium system during the past 30 years [3]. Production of barium silicates by sintering [4] and by synthesis from aqueous solutions [5, 6] has been reported, but neither technique has yielded crystals suitable for x-ray diffraction analysis, without which research on the system cannot be regarded as complete, even for technological purposes.

The barium silicates were synthesized in autoclaves with a temperature gradient (ΔT = 20-30°C). The initial reagents were chemically pure barium hydroxide and x-ray amorphous silica. An aqueous $Ba(OH)_2$ solution placed in the autoclave served simultaneously as the solvent for the silica and as the barium source. Table 1 gives the results of a series of experiments (T = 450°C, P = 800-2000 atm).

The following succession of phases could be quite clearly traced as the $Ba(OH)_2$ concentration was raised at BaO/SiO_2 molar ratios of 0.5-1.5: $BaSi_2O_5 \rightarrow Ba_2Si_3O_8 \rightarrow \alpha$-$BaSiO_3$. A similar phase sequence was previously observed for silicates of the $Na_xMe_ySi_pO_q$ type [7], where, as the NaOH concentration was increased, the silicates that crystallized presumably had fewer associated silicate radicals $[Si_2O_6]_\infty \rightarrow [Si_2O_7] \rightarrow [SiO_4]$.

We naturally expected that silicates containing simpler radicals would crystallize when the $Ba(OH)_2$ concentration was raised, but the difference in the chemism of the two solvents [8] did not permit us to anticipate a full analogy. According to Douglass

TABLE 1

Exp. No.	$Ba(OH)_2$ conc., wt.%	BaO/SiO_2 molar ratio	Results of synthesis
1	5	0,1 : 1	$BaSi_2O_5 + Ba_2Si_3O_8 + SiO_2$
2	12	0,1 : 1	$BaSi_2O_5 + Ba_2Si_3O_8 + SiO_2$
3	15	0,35 : 1	$BaSi_2O_5 + Ba_2Si_3O_8 + SiO_2$
4	32	0,7 : 1	$BaSi_2O_5 + \alpha$-$BaSiO_3$
5	32	2,5 : 1	α-$BaSiO_3 + B$
6	33	3 : 1	α-$BaSiO_3 + C$
7	40	1,4 : 1	$Ba_2Si_3O_8 + \alpha$-$BaSiO_3$
8	40	2,1 : 1	α-$BaSiO_3 + Ba_2Si_3O_8 + C$
9	40	4 : 1	α-$BaSiO_3 + C$
10	45	1 : 4	$BaSi_2O_5 + SiO_2$
11	45	2,5 : 1	α-$BaSiO_3 + B$
12*	57	1,4 : 1	$Ba_2Si_3O_8$
13	57	4 : 1	α-$BaSiO_3 + C$
14	60	5 : 1	α-$BaSiO_3 + C$
15	70	1,75 : 1	α-$BaSiO_3 + Ba_2Si_3O_8$
16	70	3 : 1	α-$BaSiO_3 + C$
17	90	2,5 : 1	α-$BaSiO_3 + A$

* The solutions with a $Ba(OH)_2$ concentration of 57% or more were prepared at 60-90°C.