

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

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Translated from Kristallografiya, Vol. 15, No. 4,
pp. 863-865, July-August, 1970
Original article submitted December 8, 1969;
revision submitted February 11, 1970

The BaO-SiO₂-H₂O 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 ($\Delta T = 20-30^\circ\text{C}$). 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 ($T = 450^\circ\text{C}$, $P = 800-2000$ atm).

The following succession of phases could be quite clearly traced as the Ba(OH)₂ concentration was raised at BaO/SiO₂ molar ratios of 0.5-1.5: BaSi₂O₅ → Ba₂Si₃O₈ → α-BaSiO₃. 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 [Si₂O₆]_∞ → [Si₂O₇] → [SiO₄].

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 ₂ O ₅ + Ba ₂ Si ₃ O ₈ + SiO ₂
2	12	0,1:1	BaSi ₂ O ₅ + Ba ₂ Si ₃ O ₈ + SiO ₂
3	15	0,35:1	BaSi ₂ O ₅ + Ba ₂ Si ₃ O ₈ + SiO ₂
4	32	0,7:1	BaSi ₂ O ₅ + α-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 ₂ Si ₃ O ₈ + α-BaSiO ₃
8	40	2,1:1	α-BaSiO ₃ + Ba ₂ Si ₃ O ₈ + C
9	40	4:1	α-BaSiO ₃ + C
10	45	1:4	BaSi ₂ O ₅ + SiO ₂
11	45	2,5:1	α-BaSiO ₃ + B
12*	57	1,4:1	Ba ₂ Si ₃ O 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 ₂ Si ₃ O 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.