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Atmospheric Pressure Synthesis for β -LiAlO₂¹

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In the course of a study of the preparation of the various compounds in the Li₂O–Al₂O₃ system, a new synthesis for the compound β -LiAlO₂ was found. The two common forms of LiAlO₂ are the high-density (3.4 g/cm³) α form stable at low temperature and the low-density (2.6 g/cm³) γ form stable at high temperature. The β form had been unknown until Chang and Margrave's report of its preparation by a high-pressure method.² In this report, a method is given for conveniently preparing β -LiAlO₂ at atmospheric pressure.

The β form was prepared from a mixture of 3.11 g of LiOH (0.13 mol) and 5.08 g of α -Al₂O₃ (0.05 mol in the form of the Alcoa A-2 brand). The reactants were ground under benzene, dried, loaded into a gold crucible, and heated to constant weight in a 500 °C furnace (4.5 days). The product was washed with water, separated by centrifugation ten times, and then dried overnight at 240 °C. Analysis by the x-ray powder diffraction method indicated it to be a clean, one-phase preparation of β -LiAlO₂, identical with the compound de-

scribed by Chang and Margrave. The 6.4 g of product represented a 97% yield based on Al₂O₃. Heating the material at 900 °C converted it to the high-temperature, low-density form, γ -LiAlO₂. The density of β -LiAlO₂ was found to be 2.61 g/cm³ by the gas pycnometric method.

LiOH appeared to be a more effective reagent than Li₂O for producing β -LiAlO₂. For example, with Li₂O and α - or γ -Al₂O₃, the product was a mixture of γ -LiAlO₂ and β -LiAlO₂, in variable proportions. With LiOH and AlOOH (Dispal brand from Conoco), the major product was β -LiAlO₂, with a minor amount of α -LiAlO₂. The results presented here indicate that high pressures are not needed to synthesize the "high-pressure" forms of LiAlO₂.

Nomenclature for the α and γ forms of LiAlO₂ used in this paper is that prevailing in the bulk of the literature, which differs from the "new" version proposed by Chang and Margrave, who discussed, in their paper, the rationale for their position.

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Registry No. β -LiAlO₂, 12003-67-7; LiOH, 1310-65-2; α -Al₂O₃, 1344-28-1.

References and Notes

- (1) This work was performed under the auspices of the U.S. Energy Research and Development Administration.
- (2) C. H. Chang and J. L. Margrave, *J. Am. Chem. Soc.*, **90**, 2020 (1968).