

## Determination of the High-Low Inversion in Berlinite (AlPO<sub>4</sub>) to 6 kbar

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### Abstract

The high-low inversion in berlinite has been determined to 6 kbar by differential thermal analysis in hydrostatic apparatus. From near 584°C at 1 bar, the transition temperature rises linearly with pressure at the rate of  $26.0 \pm 0.5$  deg kbar<sup>-1</sup>.

### Introduction

Because berlinite is structurally so closely related to quartz, data on AlPO<sub>4</sub> polymorphism might be useful for models for the much more important SiO<sub>2</sub> isotype. In particular, the high-low quartz inversion has been extensively investigated and much of the relevant thermodynamic and other data near that transition have been interrelated (Cohen and Klement, 1967; Klement and Cohen, 1968). For the high-low berlinite inversion, there has been no investigation of the variation of transition temperature with pressure; the thermodynamic data available for predicting the trajectory of this transition are also insufficient. Thus this investigation was undertaken.

### Experiments and Results

AlPO<sub>4</sub> of 99.5 percent purity, obtained from Research Organic/Inorganic Chemical Company, was moistened with ~ 4M H<sub>3</sub>PO<sub>4</sub> and then converted to the berlinite structure by hydrothermal treatment for ~1 day at ~400 bars, ~280°C in a sealed gold capsule. The resulting crystals, of ~0.2 mm maximum dimensions, were verified to be berlinite by comparison with the X-ray diffraction data of Swanson *et al* (1960). In experiments under pressure, the possibility that grain-to-grain contact might result in unsymmetric stresses on the crystals which could smear out the transition is reduced by admixing a soft, chemically inert material of high thermal conductivity, but without phase transition in the *p-T* range of interest. Boron nitride powder (-325 mesh) from Atlantic Equipment Engineers was chosen. A mixture of calcined BN powder and

the berlinite crystals, in equal portions by weight, were mixed at least 30 minutes without grinding in a WIG-L-BUG. Some of this mixture was then packed to ~5 mm length in a 1.59 mm O.D. platinum tube of 0.13 mm wall thickness, which was then sealed in an oxyacetylene flame. The arrangement of sample capsule and chromel-alumel thermocouples and the hydrostatic pressure apparatus using argon will be described elsewhere (Cohen *et al*, in preparation).

In the differential thermal analysis (*dta*) runs, temperature was varied at 0.3 to 0.8 deg sec<sup>-1</sup> and the maximum temperature difference between the measuring and reference thermocouples was always less than 2°. The temperature corresponding to the peak of the differential signal was observed on heating and cooling, and two or more cycles were made at each pressure. In the runs, data were obtained on increasing pressure and then corroborated by data taken on decreasing pressure. Pressures were read to ±5 bar on a Heise bourdon pressure gauge. The temperatures plotted in Figure 1 represent the average of the temperatures obtained on heating and on cooling, the temperature on heating always being less than 2° higher than that obtained on cooling. This "hysteresis" in temperature did not vary with pressure, within the present precision. The reproducibility in average temperature at any point was within 1°; the data from the different runs appear to agree within 1°.

Standard chromel-alumel thermocouple tables were used, and the manufacturer's calibration corrections for the reels of wire used in this investigation were applied. An *in situ* calibration of the