

## Synthesis and Characterization of a Novel Beryllorphosphate Zeolite

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A novel beryllorphosphate zeolite,  $\text{BePO}_4\text{-CJ}_2$ , has been synthesized hydrothermally, and we conclude that the zeolite framework consists of Be, P, and O atoms rather than Al, P, Si, and O atoms.

Recently many T-atoms other than aluminium and silicon have been used to synthesize novel open-framework molecular sieves.<sup>1-3</sup> Both beryllium and phosphorus are capable of forming stable, tetrahedral frameworks. Phosphorus has been widely investigated, but beryllium has been less thoroughly studied. Some beryllorphosphate minerals have been found to possess framework structures, well above the accepted range for zeolites.<sup>4-6</sup> We report here the synthesis and characterization of a novel beryllorphosphate zeolite,  $\text{BePO}_4\text{-CJ}_2$ .

Hydrothermal preparation of  $\text{BePO}_4\text{-CJ}_2$  was carried out in a Teflon-lined autoclave under autogenous pressure at 150 °C for 7 days, using a reaction mixture with molar composition 1.0  $\text{BeO}$ :0.5  $\text{P}_2\text{O}_5$ :1.8 di-isopropylamine:1.5  $\text{NH}_4\text{F}$ :60  $\text{H}_2\text{O}$ . The crystalline product was separated from amorphous materials by an ultrasonic wave, washed with water, and dried at 100 °C in air.

Under an optical microscope  $\text{BePO}_4\text{-CJ}_2$  appeared to be a pure phase. The product was identified by X-ray powder diffraction (D/MAX III A X-ray diffractometer) and the X-ray powder diffraction data is shown in Table 1. It reveals that  $\text{BePO}_4\text{-CJ}_2$  is a new compound with a novel framework structure. The mole ratio of Be/P in  $\text{BePO}_4\text{-CJ}_2$  was measured by chemical analyses and other elemental analyses were performed on a P-240 C element analyser. The bulk composition of  $\text{BePO}_4\text{-CJ}_2$  is  $(\text{NH}_4)_{23}\text{Be}_{60}\text{P}_{60}\text{O}_{120}\text{H}_{37}\cdot 10 \text{H}_2\text{O}$ . The analyses showed that the Be/P atomic ratio of the framework is 1.0, and that the resulting negative charge is balanced by a sufficient number of cations.

The IR spectrum of  $\text{BePO}_4\text{-CJ}_2$  shows two bands at 1000–1100  $\text{cm}^{-1}$  and at 400–500  $\text{cm}^{-1}$ , which were assigned to asymmetrical stretching and T-O bending of the  $\text{TO}_4$  unit, respectively; IR absorption at 500–600  $\text{cm}^{-1}$  was considered to be due to ring vibration.<sup>7</sup> This indicates that the basic building units of  $\text{BePO}_4\text{-CJ}_2$  are tetrahedral  $\text{TO}_4$  units.

During the synthesis of  $\text{BePO}_4\text{-CJ}_2$ , di-isopropylamine and  $\text{NH}_4\text{F}$  were added, but elemental analysis of the crystalline product shows that only  $\text{NH}_4^+$  is present in the structure. These  $\text{NH}_4^+$  cations can be exchanged with  $\text{Li}^+$  or  $\text{Na}^+$  cations.

TG-DT analysis shows that extra-framework water is present and, on heating, most of the water is lost at around 200–300 °C (Figure 1). It also proves that  $\text{NH}_4^+$  cations in the structure are lost at about 484–570 °C.

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Table 1. X-Ray powder diffraction data for  $\text{BePO}_4\text{-CJ}_2$ .

| $2\theta^\circ$ | $d/\text{Å}$ | $hkl$ | $2\theta^\circ$ | $d/\text{Å}$ | $hkl$ |
|-----------------|--------------|-------|-----------------|--------------|-------|
| 16.694          | 5.31         | 90    | 27.994          | 3.187        | 100   |
| 19.654          | 4.517        | 2     | 31.344          | 2.854        | 10    |
| 21.654          | 4.104        | 2     | 32.934          | 2.720        | 36    |
| 21.954          | 4.409        | 3     | 34.414          | 2.606        | 8     |
| 22.214          | 4.002        | 3     | 35.874          | 2.503        | 26    |
| 23.204          | 3.833        | 2     | 38.634          | 2.330        | 9     |
| 24.144          | 3.686        | 4     | 39.944          | 2.257        | 7     |
| 26.144          | 3.408        | 4     |                 |              |       |

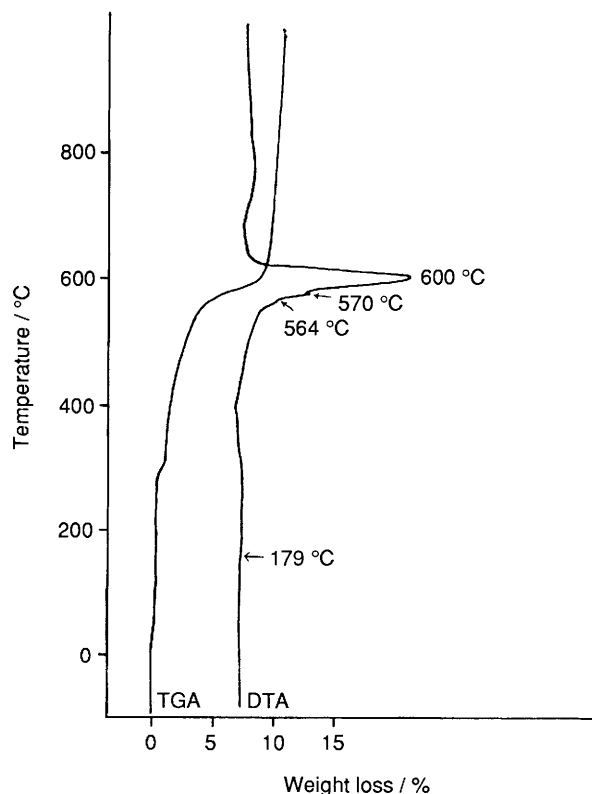


Figure 1. TG-DT analysis in  $\text{N}_2$  of  $\text{BePO}_4\text{-CJ}_2$ .