Hydrothermal Synthesis and Characterization of $KSn_2(PO_4)_3$

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$KSn_2(PO_4)_3$ is synthesized hydrothermally from the K_2O–SnO_2–P_2O_5–H_2O system, and characterized by X-ray powder diffraction, Raman and IR spectroscopy.

Thermally stable $KSn_2(PO_4)_3$, which has a Nasicon-type structure (space group *R*3*c*) with a three-dimensional network of PO_4 tetrahedra corner-shared with SnO_6 octahedra, has been prepared previously by solid-state reactions.[1] However, there have been no reports of a hydrothermal synthesis so far. Here, we report a novel hydrothermal crystallization for the synthesis of $KSn_2(PO_4)_3$.

Hydrothermal crystallization of $KSn_2(PO_4)_3$ was carried out in a stainless steel autoclave with a Teflon liner under autogenous pressure. SnO_2(AR), 85% orthophosphoric acid, and KOH solution (2 mol dm^{-3}), a mineralizer, were mixed in the molar ratio $5K_2O : SnO_2 : 3P_2O_5$ in the Teflon liner. The autoclave was sealed and heated in an oven at 250 °C for 5–7 days. After cooling, the product was filtered, washed with distilled water, and dried at ambient temperature.

The crystalline product was identified by means of X-ray powder diffraction (Fig. 1) recorded with a Rigaku D/max-IIIA diffractometer using Cu-Kα (λ = 1.5418 Å) radiation and is similar to that of the material previously prepared by the solid state reaction.[2] The product is free of impurities. The Raman spectrum (Fig. 2) was recorded on a Ramanor SPEX

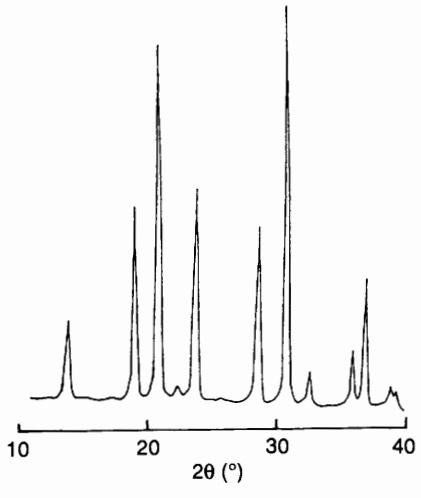

Fig. 1 X-Ray powder diffraction pattern of the product

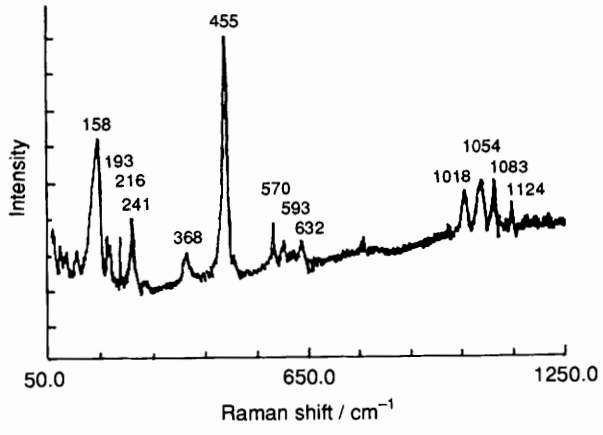

Fig. 2 Raman spectrum of the product

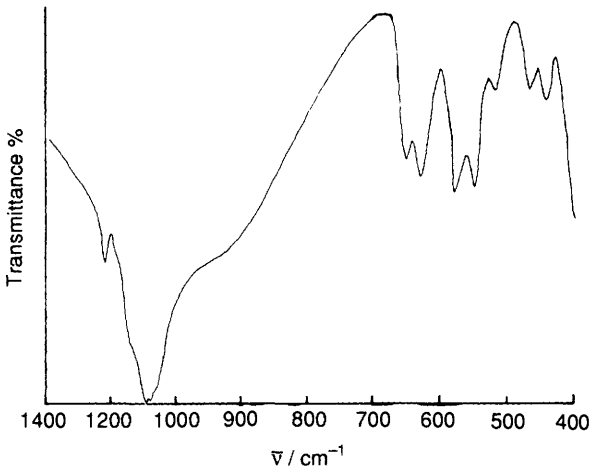

Fig. 3 IR spectrum of the product

1403 double spectrometer with an argon-ion laser (488.0 nm line, 100 mW). The spectrum is unique and quite characteristic of $KSn_2(PO_4)_3$ with two strong peaks at 158 and 455 cm^{-1} and weaker peaks at 1018–1124 cm^{-1}.[3] The IR spectrum (Fig. 3) shows absorption bands at 500–650 cm^{-1} and 900–1200 cm^{-1}, which are attributed to PO_4 bending and stretching vibrations.[4,5] DTA–TG (differential thermal analysis–thermogravimetric analysis) show that $KSn_2(PO_4)_3$ synthesized hydrothermally was stable at 1200 °C.

Received, 24th March 1993; Com. 3/01709E

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