

- 3 R. Glauser, U. Hauser, F. Herren, A. Ludi, P. Roder, E. Schmidt, H. Siegenthaler, and F. Wenk, *J. Am. Chem. Soc.*, **95**, 8457 (1973).  
 4 K. Rieder, U. Hauser, H. Siegenthaler, E. Schmidt, and A. Ludi, *Inorg. Chem.*, in the press.

- 5 N. S. Hush, *Progr. Inorg. Chem.*, **8**, 391 (1967).  
 6 M. B. Robin and P. Day, *Advan. Inorg. Chem. Radiochem.*, **10**, 247 (1967).  
 7 D. A. Dows, A. Haim, and W. K. Wilmarth, *J. Inorg. Nucl. Chem.*, **21**, 33 (1961).

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### Preparation and Doping of $\text{CaLaZrTaO}_7$ Pyrochlore

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Belyaev and co-workers<sup>1</sup> recently have described new, complex compounds with pyrochlore structure. Among these materials was  $\text{CaLaZrTaO}_7$  which has been further investigated in these Laboratories.

The synthesis of  $\text{CaLaZrTaO}_7$  consisted of two parts, the first being similar to that described by Belyaev. Lanthanum acetate was reacted with tantalum pentoxide at 1250 °C in air, for ten hours, to yield  $\text{LaTaO}_4$ , as established by X-ray analysis. While Belyaev used calcium zirconate prepared from the oxides, we found multi-phase materials to result. Instead, we prepared a calcium carbonate suspension in water, to which was added a solution of zirconium disulfate. This was then reacted with ammonia, digested (hot) for one hour, filtered and dried. This material was fired at 1300 °C in air, for two hours, ground, and refired for two additional hours, to yield  $\text{CaZrO}_3$ , as established by X-ray analysis.

Equimolar proportions of  $\text{LaTaO}_4$  and  $\text{CaZrO}_3$  were mixed well, and pressed into pellets, either hot-pressing or cold-pressing techniques being used. For hot-pressing, the materials were heated in vacuum, at  $10^4$  psi, at a temperature of 1400°–1450 °C, for four hours. The pressed pellets were then re-fired at 1400 °C in air for four hours, to restore the oxygen stoichiometry.

Materials formed by the above method were examined by both powder (film) X-rays and by diffractometry. Materials formed by hot-pressing were pyrochlore with a lattice constant of about 10.625 Å. A weak second phase, of the order of 5%, was sometimes present, and was probably  $\text{ZrO}_2$ . Belyaev<sup>1</sup> has reported a lattice constant of 10.567 Å for this pyrochlore.

It is of interest that the material formed by cold-pressing contained two pyrochlore phases, in addition to  $\text{ZrO}_2$ . The major pyrochlore phase was the same as the one reported in the hot-pressing case, with a lattice constant of 10.74 Å, and was possibly  $\text{LaTaO}_4$ .

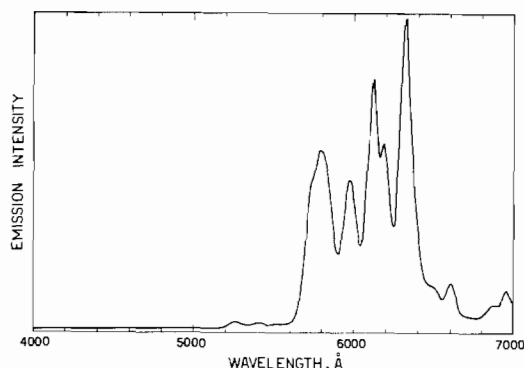


Fig. 1. Spectral distribution of cathodoluminescence emission from  $\text{CaLa}_{0.98}\text{Eu}_{0.02}\text{ZrTaO}_7$ .

A luminescent pyrochlore was obtained by doping with europium. Thus,  $\text{La}_{0.98}\text{Eu}_{0.02}\text{TaO}_4$  was synthesized, and from this and  $\text{CaZrO}_2$ ,  $\text{CaLa}_{0.98}\text{Eu}_{0.02}\text{ZrTaO}_7$  was prepared, as described above. Pellets were pressed, using hot pressing, and the resultant materials were examined under cathode-ray excitation. Figure 1 shows the emission spectra obtained for such a material. Activation by other rare-earth ions and by ions of other transition elements also appears feasible.

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### Reference

- 1 Belyaev, Aver'yanova, Ezhov and Balashov, *Zh. Neorg. Khim.*, **17** (10), 2842 (1972).