PRELIMINARY CRYSTALLOGRAPHIC DATA OF THE $Ln(PF_6)_3$. 4TDTD.$4H_2O$ COMPLEXES-*

Mueller, R.A. (1), Ulbrich, H.H. (2), Vicentini, G. (1) and Giesbrecht, E. (1)

(1) Instituto de Química and (2) Instituto de Geociências, University of São Paulo, Cidade Universitária, São Paulo, Brazil.

Vicentini et al (J.Inorg.Nucl.Chem., 37, 2021, 1975) synthesized a series of complexes $Ln(PF_6)_3$. 4TDTD . $4H_2O$ (Ln = La-Lu, Y; TDTD = 1,4 - dithiane - 1,4 - dioxide) as very fine powders. Recrystallized single crystals are always complexly twinned and show the same D-S powder pattern as the original powders. Precession, Weissenberg and rotation methods were used to determine the crystallographic characteristics of the compounds with Ln=La, Ce, Yb. The La and Ce compounds are monoclinic with β close to 90^o, and P2, Pm, P2/m as possible space groups (difraction symbol is 2/mP ...). The Yb compound shows orthorhombic symmetry, space group Pnca (Pbcn is the standard orientation). Doubling of two axes increases the unit cell content from 1 in the monoclinic to 4 in the orthorhombic crystals. D-S powder films were made of all the recrystallized material (Cr Kα, Si as internal standard) and a few lines computer-indexed. Cell constants were calculated using conventional least-squares computer methods. All complexes with Ln=Y , La to Tm, have comparable powder patterns and are thus monoclinic, while the Lu pattern is identical to that of the orthorhombic Yb. Cell parameters, chage from a = 9,05(1) , b = 9.12(2), c = 12.44(1) A, V = 1026(2) A^3 (Y compound) to a = 8.93(1), b = 9.31(2) , c = 12.41(2)A, V = 1031(2) A^3 (Tm compound) while β changes from $92^o06'(6)$ to $91^o51'(10)$; corresponding data for the Yb compound are 17.19(4), 17.75(3), 13.37(2) A, V = 1020(3) A^3 (V per molecule). There is, within the low resolution of the cell parameters, an irregular tendency for the cell parameters to decrease, probably controlled by a small overall lanthanide contraction effect (Siekierski, J. Inorg. Nucl. Chem.,33, 377, (1971)).

* Supported by FAPESP