24.1-20 HIGH PRESSURE X-RAY DIFFRACTION STUDY OF ACTINIDES AND ACTINIDE COMPOUNDS BY AN ENERGY-DISPERSIVE METHOD. J. Benedict, CEC Joint Research Centre, European Institute for Transuranium Elements, P.O. Box 2266, D-7500 Karlsruhe 1, and H. Holzapfel, University of Paderborn, Experimentalphysik, Pohlweg 53, D-4790 Paderborn, F.R.G.

Actinide metals and compounds were investigated in a Holzapfel type diamond anvil cell at pressures up to 40 GPa. Measuring time is kept within the range of ten minutes to some hours by the use of a conical slit for the diffracted radiation which makes use of the complete diffraction cone. The pressures were determined from the lattice parameters of the marker substances NaCl and CsCl or with the ruby fluorescence manometer.

Studies of thorium metal (G. Bellussi, U. Benedict, W.B. Holzapfel, J. Less Common Metals 78(1981)147-153) and of stoichiometric uranium dioxide were completed. Investigations of thorium metal (together with I. Birkel) and of other actinide materials is underway. Uranium dioxide gradually transforms to a low symmetry high pressure phase in the range 29 to 38 GPa. On release of pressure, a marked hysteresis to transformation is observed, but complete retransformation to the fluorite type occurs between 15 and 5 GPa. Among the known high density AB₂ structure types, the lead chloride type gives the best agreement with the observed high pressure phase, using the atom coordinates of thorium disulfide in the L2₁/PULVERIX program. Lattice parameters a=5.59, b=3.50, c=6.76 (pm; Pmna setting) are obtained when approximate lattice parameters estimated from the volume compression on transformation are refined by Taupin's indexing program.

Single-pulse fitting of the data obtained for uranium metal up to 6.6 GPa gives a bulk modulus B=122.6 GPa and the elastic constants $c_{11}=3.627$, $c_{22}=2.191$, $c_{33}=1.956$ (1 TPa⁻¹).

24.1-21 TWINNING BY PSEUDO-ORTHORHOMBIC IN (NH₄)₂SnCl₄


Some time ago we tried to refine the structure of (NH₄)₂SnCl₄ in space group P2₁c. Satisfactory agreement could not be achieved. Therefore the correctness of the space group was doubted. Recently, papers on the same compound were published by I. Mikhail (Acta Cryst. (1980) B36, 2126) and by J. Warzevski (Kristall u. Technik (1980), 667). Both authors adopted the orthorhombic space group P2₁cn. The anisotropic thermal parameters estimated by Mikhail show some anomalous values which do not find a reasonable explanation. Warzevski determined the space group photographically and a small difference in general (pseudo-orthorhombic) reflection intensities can easily have been overlooked. This confusion about the space group induced us to examine our data more closely. Systematic extinctions, H00 for $\pi$-values, are obvious from our data set but several H0 reflections with $\pi\neq0$ look significantly different from zero. Six general reflections were accurately remeasured from several crystals. The results clearly prove monoclinic symmetry. The systematic extinctions are consistent with space group P2₁cn or P2₁. The polar space group P2₁ is chosen because of the pyroelectricity effect observed (J. Warzevski, Kristall u. Technik (1980), 667). In some crystals the apparent mm symmetry, due to twinning and to the pseudo-orthorhombic lattice, can lead to an incorrect determination of the space group. All measured crystals exhibit twinning by pseudo-merohedry with symmetry (apparently) equal to zero. (H. Catti and G. Ferraris, Acta Cryst. (1976) A32, 163). The twinning ranges from a small amount to nearly equal fractions of the individual crystals in the twin. A graphical representation of the results is given illustrating this twinning phenomenon.