the orientation parallel to [010](cannizzarite) is [110]-(galena) of both layers. The structure of phase 3 synthesized by Graham *et al.* (1953) can be proposed from the cannizzarite structure by adding one octahedral layer to the compact close-packed layer *B* in cannizzarite and this is in good agreement with the statistical lattice parameters given by the authors.

The author is grateful to Professor R. Huber and Dr P. Schwager for permission to use the scanning microdensitometer and for assistance during the measurements. All calculations were performed on a Univac 1108 computer of GWD, Göttingen, with programs written by the author, except the intensity reduction program.

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Acta Cryst. (1979). B**35**, 136–137

Ammonium Hexachlorotechnetate(IV)

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(*Received* 4 *August* 1978; *accepted* 12 *September* 1978)

Abstract. $(NH_4)_2TcCl_6$, $Fm3m$, cubic, a = 9·9072 (8) Å, U = 972·42 Å^3, Z = 4, D_x = 2·34 Mg m^{-3}, μ = 2·97 mm^{-1} (Mo $K\alpha$). The dianionic complex of technetium is octahedral with six Cl ligands bonded at a distance of 2·3531 (5) Å. The ammonium ions which occupy the eight tetrahedral sites ($\bar{4}3m$) surrounding each complex anion are rotationally disordered. The structure was refined to an R of 0·033 using 115 independent, observed reflections.

Introduction. As part of an ongoing study of Tc complexes as radiopharmaceuticals, we are precisely determining the structures of a series of Tc complexes.

A nearly octahedral, yellow crystal, with edges ~0·25 mm was mounted on a glass fiber. Intensities were measured using Mo $K\alpha$ radiation on a Syntex $P\bar{1}$ diffractometer equipped with a graphite monochromator. The data supported the choice of space group $Fm3m$ in agreement with the previous work of Schwocau (1964) and Elder, Fergusson, Gaines, Hickford & Penfold (1967) for various salts of hexahalotechnetium dianions. A total of 552 reflections (2·5 < 2θ < 63·8°) were measured. After corrections for absorption, 115 unique reflections were obtained by averaging equivalent reflections. All 115 had $I > 2\sigma(I)$ and were used in the subsequent refinement. Calculated transmission coefficients varied from 0·59 to 0·70.

The choice of space group requires the Tc atoms to be located at the fourfold position (0,0,0), the N atoms at the eightfold position ($\frac{1}{4},\frac{1}{4},\frac{1}{4}$) and the Cl atoms at the 24-fold position of the type (x,0,0). A sharpened, origin-removed Patterson map was used to locate the Cl atoms (0·24,0,0) and confirm the choice of the N-atom positions. Refinement of a scale factor, the Cl positional and anisotropic thermal parameters and Tc and N isotropic thermal parameters (six parameters total) converged with R = 0·033 and R' = $[\sum w(\Delta F)^2/\sum w(F_o)^2]$ = 0·025.

The H atoms could not be located by examination of electron density or difference maps. Various models were tried placing H at the 32-fold position (x,x,x) with x = 0·19 or x = 0·31. Since none of these resulted in improved agreement with the observations, we report here the results for the model without H atoms.* The refined parameters are as follows: Cl, x = 0·23751 (5), U_{11} = 0·0173 (3), U_{22} = U_{33} = 0·0354 (3) Å^2; Tc, U = 0·0172 (2) Å^2; N, U = 0·0315 (8) Å^2. Neutral-atom scattering factors from Cromer & Mann (1968) were corrected for anomalous dispersion (*International Tables for X-ray Crystallography*, 1974). A final difference synthesis was nearly featureless, exhibiting maxima of ~0·4 e Å^{-3} at 0·08, 0·08, 0 and 0·48, 0·12, 0. Calculated positions for H had difference densities of ~0·2–0·1 e Å^{-3}. Minima were observed ~0·1 Å from Cl (−1·4 e Å^{-3}), ~0·6 Å from Tc (−1·0 e Å^{-3}) and at

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33898 (2 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N ($-1\cdot0$ e Å^{-3}). One other minimum was found at 0·25, 0·25, 0 ($-1\cdot4$ e Å^{-3}).

Discussion. The anion consists of six Cl atoms in a regular octahedral arrangement about a central Tc atom. The Tc—Cl bond distance is 2·3531 (5) Å. This value is in close agreement with the value of 2·35 (1) Å found previously for the analogous K salt (Elder *et al.*, 1967). It is significantly less than the value of 2·46 Å predicted from ionic radii (Shannon, 1976). Ammonium ions are known to have low barriers to rotation in many crystals [see, for example, the recent review by Hüller (1977)]. This is presumably the reason why the H atoms in this structure could not be successfully located.

All calculations were performed on an Amdahl 470/V6 computer located at the University of Cincinnati. A locally modified version of XRAY 67 (Elder, 1974) was used for all calculations. This study was supported by the National Institutes of Health (Grant No. HL-21276-01).

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Acta Cryst. (1979). B**35**, 137–139

Structure of Orthorhombic CeS_2 Prepared at 1773 K and 5·5 GPa

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(*Received* 6 *May* 1978; *accepted* 12 *September* 1978)

Abstract. $Pn2_1a$, $a = 8\cdot103$ (2), $b = 16\cdot221$ (2), $c = 4\cdot093$ (1) Å, $Z = 8$ and $D_x = 5\cdot06$ Mg m^{-3}. The structure is roughly expressed as the stacking of three kinds of layers *A*, *B* and *C* parallel to (010) with layer sequence *ABCACBA*. The layer *A* consists of S ions and the layers *B* and *C* of Ce and S ions. Each Ce atom is coordinated to nine S atoms, the average Ce—S distance being 3·01 (2) Å. All S ions in the *A* layer form pairs, the bond length of the pair being 2·18 (2) Å, while the distances between unpaired S atoms are in the range 3·03 to 4·14 Å. The difference between the orthorhombic and monoclinic structures is primarily in the layer sequence.

Introduction. Cerium polysulphide, CeS_2, crystallizes in the monoclinic form with a pseudocubic symmetry (Picon & Patrie, 1956; Flahaut & Guittard, 1956; Marcon & Pascard, 1968). A polymorphic phase, which is isostructural with orthorhombic LaS_2, has been synthesized under pressure (Yanagisawa & Kume, 1973). Single crystals were grown at 1773 K and 5·5 GPa. The size of each grain was approximately 0·2 × 0·2 × 0·1 mm.

The symmetry was orthorhombic and systematic extinctions were $hk0$ with $h = 2n + 1$ and $0kl$ with $k + l = 2n + 1$, indicating that the space group was either $Pn2_1a$ or $Pnma$. A set of three-dimensional intensity data of 688 reflections to $\sin \theta/\lambda = 0\cdot75$ Å^{-1} were collected with a Rigaku Denki four-circle diffractometer using monochromated Mo $K\alpha$ radiation. The intensity data were corrected for Lorentz and polarization factors, but no absorption correction was made. The structure was solved from a Patterson map. Refinement was carried out by the block-diagonal least-squares method

0567-7408/79/010137-03$01.00