mized was $\sum w(|F_o| - |F_c|)^2$ with $w = 3.3421/\sigma_F^2$. Final $R = 0.030$, $wR = 0.023$ for 39 refined parameters ($R = 0.052$, $wR = 0.024$ for all reflections), $(\Delta/\sigma)_{\max} < 0.002$. Residual electron density in final $\Delta\rho$ map was within $-0.54$ to $0.45$ e Å$^{-3}$. Scattering factors including corrections for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Figures were drawn with program *Graphics* from the *SHELXTL* program system (Sheldrick, 1990). All calculations were performed with an IBM PC/AT.

Final atomic parameters and isotropic temperature factors are reported in Table 1.* Selected interatomic distances and bond angles are given in Table 2. A stereodrawing of the unit-cell contents is shown in Fig. 1. Fig. 2 shows the layer consisting of two planes of the four-membered rings, linked by bridging O(1) atoms.

**Related literature.** The title crystal is the third barium copper silicate obtained during our studies on the chemical reactivity of the '1-2-3' type superconductors. Results on the synthesis and crystal structure of $BaCu_2[Si_2O_7]$ and $Ba_2Cu_2[Si_4O_{12}]$ have been published previously (Janczak, Kubiak & Głowiak, 1990; Janczak & Kubiak, 1992). The structure $BaCu[Si_4O_{12}]$ is isostructural with $BaFe[Si_4O_{10}]$, $CaCu[Si_4O_{10}]$ and $SrCu[Si_4O_{10}]$, reported by Pabst (1954, 1959). The compound $CaCr[Si_4O_{10}]$ is also apparently isostructural, Belsky (1984). The $KCa_4[Si_8O_{20}]$ structure contains $[Si_4O_{10}]^{4-}$ groups as in $BaCu[Si_4O_{10}]$ (Ståhl, Kvick & Ghose, 1987). The Si—O distances in $BaCu[Si_4O_{10}]$ are comparable with those in other tetrasilicates.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54946 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0258]

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# Structure of $KErTe_2$

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**Abstract.** Potassium erbium ditelluride, $KErTe_2$, $M_r = 461.56$, trigonal, $R\bar{3}m$, $a = 4.410\,(2)$, $c = 24.27\,(2)$ Å, $V = 408.8\,(8)$ Å$^3$, $Z = 3$, $D_x = 5.626$ g cm$^{-3}$, $\lambda(\text{Mo}\ K\alpha_1) = 0.7093$ Å, $\mu = 267$ cm$^{-1}$, $F(000) = 567.3$ (including anomalous dispersion), $T = 111$ K, $R(F) = 0.034$ for 202 unique observations with $F_o^2 > 3\sigma(F_o^2)$. $KErTe_2$ adopts the $\alpha$-$NaFeO_2$ structure, an NaCl superstructure where the cations are ordered in alternating layers between cubic close-packed anions.

**Experimental.** $KErTe_2$ was prepared through the use of a flux consisting of $K_2Te_3$ and Te in a ratio of 1:3. $K_2Te_3$ was prepared by the reaction of K (Alfa, 99%) and Te (Aldrich, 99.8%) in the stoichiometric ratio of 2:3 in an evacuated quartz tube at 923 K for three days. In a dry box under an Ar atmosphere, 0.249 g $K_2Te_3$, 0.045 g Er (REacton, 99.9%), and 0.206 g Te were ground together and then loaded into a quartz tube. The tube was evacuated, heated at 873 K for 4 d, and then ramped to 1173 K to heat for 4 d. The tube was cooled at the rate of 3 K h$^{-1}$ to 648 K and then to room temperature at 99 K h$^{-1}$. Black air-stable hexagonal plates were obtained. The presence of the three elements K, Er and Te was confirmed by EDAX (energy-dispersive analysis by X-rays) measurements with a Hitachi S570 scanning electron microscope.

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The crystal used for data collection was a hexagonal plate of dimensions 0.11 × 0.11 × 0.116 mm (hexagonal face) × 0.019 mm (thickness) bound by {100} and {001}. The unit-cell parameters were determined from a least-squares refinement of 17 reflections in the range $25 < 2\theta(\text{Mo } K\alpha_1) < 30°$, automatically centered on a Picker FACS-1 diffractometer. Intensity data were collected by the $\theta/2\theta$ scan technique with a scan range of 1.0° in $2\theta$ below $K\alpha_1$ to 1.9° above $K\alpha_2$ at a scan rate of $2° \text{ min}^{-1}$. Background intensities were counted for 10 s at each end of the scan range. Reflections for which $F_o^2 \leq 3\sigma(F_o^2)$ were rescanned and the two scans were combined. Six representative reflections (024, $\bar{1}\bar{3}4$, $3\bar{1}4$, $\bar{3}1\bar{4}$, $0\bar{2}\bar{4}$, $1\bar{3}4$), measured every 100 reflections during the course of data collection, exhibited no significant variations. Data were collected between $3 \leq 2\theta(\text{Mo } K\alpha_1) \leq 64°$ ($\lambda^{-1} \sin\theta \leq 0.7471$ Å$^{-1}$). A total of 1900 reflections, with indices in the range $-6 \leq h \leq 6$, $-6 \leq k \leq 6$, $-36 \leq l \leq 36$, were obtained. They were corrected for absorption by the analytical method (de Meulenaer & Tompa, 1965); minimum and maximum transmission factors were 0.130–0.596. The $R$ index for merging $(\sum|\langle F^2\rangle - F^2|/\sum F^2)$ 1900 observations was 0.140 (226 unique observations). Of these, 202 had $F_o^2 > 3\sigma(F_o^2)$, where a value of $p$ of 0.04 was used in the calculation of $\sigma(F_o^2)$.

The initial K, Er and Te positions in space group $R\bar{3}m$ were taken from the presumed isostructural compound $NaCrS_2$ (Engelsman, Wiegers, Jellinek & Van Laar, 1973). Conventional atomic scattering factors (Cromer & Waber, 1974) were used and anomalous-dispersion corrections (Cromer, 1974) were applied. All calculations were performed on a Harris 1000 computer with programs and methods standard to this laboratory (Waters & Ibers, 1977). Weights were assigned as $[\sigma^2(\text{counting statistics}) + (0.04F_o^2)^2]^{-1}$. The final isotropic least-squares refinement on $F_o^2$ resulted in values of $R(F_o^2)$ of 0.066, of $wR(F_o^2)$ of 0.080, and of $S$ of 1.19 (six variables, 226 observations) with maximum $\Delta/\sigma$ of 0.065. The $R(F)$ index for those 202 reflections having $F_o^2 > 3\sigma(F_o^2)$ was 0.034. There were no features with a height greater than 2.5% the height of an Er atom in the final difference electron density map ($\Delta\rho_{\max} = 7.6$, $\Delta\rho_{\min} = -2.5$ e Å$^{-3}$).

Final values of the positional and isotropic thermal parameters are given in Table 1.* The structure of $KErTe_2$ is drawn in Fig. 1 and the bond angles and distances are presented in Table 2. The structure consists of close-packed Te layers in a pseudo-cubic structure with a distortion along [111]. The K and Er atoms occur in alternating layers and have distorted octahedral coordination. The Er-centered octahedra share edges with the K-centered octahedra between layers. Within the same layer, both the K- and Er-centered octahedra share edges with themselves.

Table 1. *Positional parameters* and isotropic thermal parameters* ($B = 8\pi^2U^2$) *for* $KErTe_2$

| | Wyckoff position | $x$ | $y$ | $z$ | $B$ (Å$^2$) |
|---|---|---|---|---|---|
| K | $b$ | 0 | 0 | $\frac{1}{2}$ | 0.91 (6) |
| Er | $a$ | 0 | 0 | 0 | 0.46 (2) |
| Te | $c$ | 0 | 0 | 0.263533 (29) | 0.48 (2) |

* In addition, an isotropic extinction parameter (Zachariasen, 1968) was refined. Its final value is 2.4 (4) × $10^{-8}$.

Table 2. *Distances* (Å) *and angles* (°) *in* $KErTe_2$

| | | | |
|---|---|---|---|
| K—Te | 3.466 (2) × 6 | Te—K—Te | 100.97 (5) × 6 |
| Er—Te | 3.058 (1) × 6 | Te—Er—Te | 92.28 (4) × 6 |
| Te—Er—Te | 87.72 (5) × 6 | Te—K—Te | 79.03 (5) × 6 |

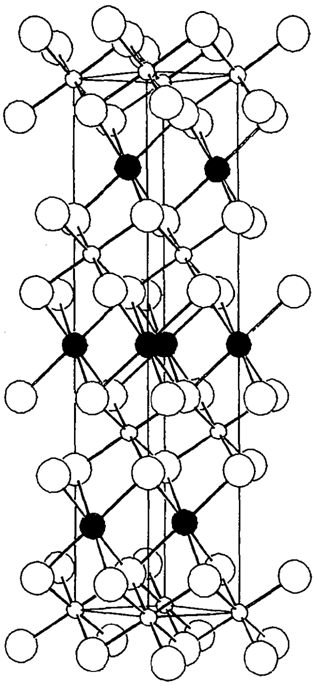

Fig. 1. View of the structure of $KErTe_2$ with the hexagonal unit cell outlined. The large open circles are Te, the medium filled circles are K, the small open circles are Er. The $c_{\text{hex}}$ axis extends from top to bottom.

**Related literature.** Compounds of the type $ALnS_2$ ($A$ = alkali metal, Ln = rare earth) are known for $A$ = Li, Na, K for most of the lanthanides, and for $A$ = Rb, Cs for a few. Ballestracci & Bertaut (1964),

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* A list of structure amplitudes has been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54908 (2 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0551]

Ballestracci (1965), and Bronger, Elter, Maus & Schmitt (1973) determined these structures from X-ray powder-diffraction data. The structures of the $TlLnQ_2$ (Q = S, Se, Te) system (Kabré, Julien-Pouzol & Guittard, 1974) have been characterized by X-ray powder-diffraction measurements with the structure of $TlErS_2$ being determined from Weissenberg data. The majority of these compounds crystallize with the rhombohedral $\alpha$-$NaFeO_2$ structure; a few are cubic. The single-crystal X-ray determination of the structure of $KCeS_2$ (Plug & Verschoor, 1975) resolved the assignment of the space group ($R\bar{3}m$, not $R3m$ or $R32$) and the cation coordination (distorted octahedral, not trigonal prismatic) of this rhombohedral structural type. To our knowledge, $KCeS_2$ was the only structure in this series determined by a single-crystal X-ray diffraction experiment. $KErTe_2$ represents the first alkali metal–lanthanide–telluride whose structure has been determined in this family of compounds.

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# Structure of {*ad*,*be*-Bis[(8-dimethylamino-κ*N*)naphthyl-κ*C*$^1$]}-*f*-ethyl-*c*-iodoplatinum(IV)

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**Abstract.** $[Pt(C_{12}H_{12}N)_2(C_2H_5)I]$, $M_r = 691.51$, monoclinic, $P2_1/n$, $a = 14.902$ (1), $b = 10.815$ (4), $c = 14.941$ (1) Å, $\beta = 103.29$ (1)°, $V = 2343.3$ (3) Å$^3$, $Z = 4$, $D_x = 1.960$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71073 Å, $\mu = 73.7$ cm$^{-1}$, $F(000) = 1320$, $T = 294$ K, $R = 0.030$ for 4204 unique observed diffractometer data with $I > 2.5\sigma(I)$. The platinum(IV) cation is coordinated by two C,N-chelate bonded 8-dimethylamino-1-naphthyl (dman) monoanionic ligands as well as the ethyl and iodo anions which are mutually in *cis* orientation. As oxidative addition reactions of primary alkyl halides to organometal-$d^8$ complexes generally yield *trans*-(alkyl)(halide) products, the observed formation of the *cis* isomer in the reaction of $Pt(dman)_2$ with EtI is an exception: the reaction with MeI affords the *trans*-(Me)(I) product.

**Experimental.** Data were collected at 294 K on an Enraf–Nonius CAD-4F diffractometer for a brownish block-shaped crystal (0.45 × 0.38 × 0.25 mm) glued on top of a glass fibre. Unit-cell parameters were determined from a least-squares

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