

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 (Kabr , 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 α - $NaFeO_2$ 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 C¹]}-*f*-ethyl-*c*-iodoplatinum(IV)

BY W. J. J. SMEETS AND A. L. SPEK*

Bijvoet Center for Biomolecular Research, Vakgroep Kristal- en Structuurchemie, University of Utrecht, Padualaan 8, 3584 CH Utrecht, The Netherlands

AND J. A. M. VAN BEEK AND G. VAN KOTEN*

Debye Research Institute, Department of Metal-Mediated Synthesis, University of Utrecht, Padualaan 8, 3584 CH Utrecht, The Netherlands

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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) Å³, $Z = 4$, $D_x = 1.960$ 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

* Authors to whom correspondence should be addressed.

Table 1. Final coordinates and equivalent isotropic thermal parameters (Å²) of the non-H atoms

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Pt	0.22653 (1)	0.53525 (2)	0.47405 (1)	0.0284 (1)
I	0.34173 (3)	0.33643 (4)	0.50521 (3)	0.0554 (2)
N(1)	0.3300 (3)	0.6638 (4)	0.5630 (3)	0.0399 (16)
N(2)	0.2584 (3)	0.5807 (4)	0.3268 (3)	0.0361 (16)
C(1)	0.1494 (4)	0.6895 (5)	0.4557 (3)	0.0323 (17)
C(2)	0.0609 (4)	0.7020 (5)	0.4048 (4)	0.0397 (17)
C(3)	0.0184 (4)	0.8202 (6)	0.3926 (4)	0.050 (2)
C(4)	0.0631 (5)	0.9211 (6)	0.4337 (4)	0.058 (3)
C(5)	0.1524 (5)	0.9122 (6)	0.4896 (4)	0.050 (2)
C(6)	0.2002 (5)	1.0138 (6)	0.5353 (5)	0.066 (3)
C(7)	0.2854 (5)	1.0014 (6)	0.5916 (5)	0.069 (3)
C(8)	0.3289 (5)	0.8862 (6)	0.6020 (4)	0.056 (2)
C(9)	0.2858 (4)	0.7856 (5)	0.5574 (4)	0.0402 (17)
C(10)	0.1968 (4)	0.7944 (5)	0.5008 (3)	0.0370 (17)
C(11)	0.4179 (4)	0.6710 (6)	0.5291 (4)	0.054 (2)
C(12)	0.3594 (5)	0.6228 (7)	0.6604 (4)	0.067 (3)
C(13)	0.1796 (3)	0.5250 (5)	0.2615 (3)	0.0319 (17)
C(14)	0.1666 (4)	0.5459 (5)	0.1686 (3)	0.0440 (17)
C(15)	0.0941 (4)	0.4888 (6)	0.1072 (4)	0.049 (2)
C(16)	0.0357 (4)	0.4096 (5)	0.1373 (4)	0.0450 (17)
C(17)	0.0474 (3)	0.3860 (5)	0.2324 (3)	0.0343 (17)
C(18)	-0.0097 (4)	0.3024 (5)	0.2656 (4)	0.0407 (17)
C(19)	0.0040 (4)	0.2825 (5)	0.3575 (4)	0.0426 (19)
C(20)	0.0722 (4)	0.3461 (5)	0.4211 (4)	0.0418 (17)
C(21)	0.1304 (3)	0.4301 (4)	0.3927 (3)	0.0308 (17)
C(22)	0.1203 (3)	0.4462 (4)	0.2959 (3)	0.0277 (16)
C(23)	0.2589 (4)	0.7167 (5)	0.3081 (4)	0.048 (2)
C(24)	0.3447 (4)	0.5265 (6)	0.3105 (4)	0.0490 (19)
C(25)	0.1636 (4)	0.5086 (5)	0.5846 (3)	0.0419 (19)
C(26)	0.1950 (5)	0.4011 (6)	0.6479 (4)	0.067 (3)

Table 2. Selected bond distances (Å) and bond angles (°)

Pt—I	2.7239 (5)	N(2)—C(23)	1.497 (7)
Pt—N(1)	2.266 (4)	N(2)—C(24)	1.484 (8)
Pt—N(2)	2.406 (4)	C(1)—C(2)	1.369 (8)
Pt—C(1)	2.009 (6)	C(1)—C(10)	1.422 (8)
Pt—C(21)	2.005 (4)	C(9)—C(10)	1.403 (8)
Pt—C(25)	2.098 (5)	C(13)—C(22)	1.407 (7)
N(1)—C(9)	1.466 (7)	C(20)—C(21)	1.388 (7)
N(1)—C(11)	1.511 (8)	C(21)—C(22)	1.430 (6)
N(1)—C(12)	1.488 (7)	C(25)—C(26)	1.504 (8)
N(2)—C(13)	1.472 (6)		
I—Pt—N(1)	93.84 (11)	C(9)—N(1)—C(12)	110.5 (5)
I—Pt—N(2)	93.92 (11)	C(11)—N(1)—C(12)	105.1 (5)
I—Pt—C(1)	175.83 (16)	Pt—N(2)—C(13)	103.1 (3)
I—Pt—C(21)	89.44 (13)	Pt—N(2)—C(23)	112.4 (3)
I—Pt—C(25)	97.77 (15)	Pt—N(2)—C(24)	115.5 (3)
N(1)—Pt—N(2)	99.22 (15)	C(13)—N(2)—C(23)	108.4 (4)
N(1)—Pt—C(1)	82.05 (19)	C(13)—N(2)—C(24)	108.6 (4)
N(1)—Pt—C(21)	176.67 (17)	C(23)—N(2)—C(24)	108.4 (4)
N(1)—Pt—C(25)	89.41 (18)	Pt—C(1)—C(2)	127.3 (4)
N(2)—Pt—C(1)	86.09 (17)	Pt—C(1)—C(10)	113.0 (4)
N(2)—Pt—C(21)	79.94 (17)	C(2)—C(1)—C(10)	119.6 (5)
N(2)—Pt—C(25)	164.98 (18)	N(1)—C(9)—C(10)	116.4 (5)
C(1)—Pt—C(21)	94.7 (2)	C(1)—C(10)—C(9)	122.0 (5)
C(1)—Pt—C(25)	82.9 (2)	N(2)—C(13)—C(22)	118.6 (4)
C(21)—Pt—C(25)	90.74 (19)	Pt—C(21)—C(20)	126.5 (4)
Pt—N(1)—C(9)	106.3 (3)	Pt—C(21)—C(22)	116.0 (3)
Pt—N(1)—C(11)	111.1 (3)	C(20)—C(21)—C(22)	117.5 (4)
Pt—N(1)—C(12)	113.5 (4)	C(13)—C(22)—C(21)	120.9 (4)
C(9)—N(1)—C(11)	110.3 (4)	Pt—C(25)—C(26)	118.1 (4)

treatment of the SET4 setting angles of 25 reflections with $13.9 < \theta < 19.6^\circ$. The unit-cell parameters were checked for the presence of higher lattice symmetry (Spek, 1988). Intensity data for 7102 reflections were collected [h 0:19, k -14:0, l -19:18; maximum $(\sin \theta)/\lambda = 0.65 \text{ \AA}^{-1}$] in $\omega/2\theta$ scan mode with $\Delta\omega = (0.55 + 0.35 \tan \theta)^\circ$ using Zr-filtered Mo $K\alpha$ radiation. Data were corrected for Lp, for a linear decay

(3.1%) of the reference reflections ($\bar{5}75$, $5\bar{7}5$) during the 59 h of X-ray exposure time, and for absorption (DIFABS; Walker & Stuart, 1983), corrections in the range 0.87–1.11, resulting in 4204 unique reflections with $I > 2.5\sigma(I)$. The Pt and I atoms were found with standard Patterson methods (SHELXS86; Sheldrick, 1986), other non-H atoms were located from subsequent difference Fourier analyses. Refinement on F was carried out by full-matrix least-squares techniques (SHELX76; Sheldrick, 1976). H atoms were introduced at calculated positions (C—H = 0.98 Å) and included in the refinement riding on their carrier atoms. All non-H atoms were refined with anisotropic thermal parameters; H atoms with one common isotropic thermal parameter [$U = 0.056(3) \text{ \AA}^2$]. Weights were introduced in the final refinement cycles, convergence with 287 parameters was reached at $R = 0.030$ [$wR = 0.025$, $w = 1/\sigma^2(F)$, $S = 2.27$, $(\Delta/\sigma)_{\max} = 0.0078$]. The final difference Fourier map shows residual densities around Pt and I atoms within the range $-1.12 - 1.14 \text{ e \AA}^{-3}$. Scattering factors from Cromer & Mann (1968), anomalous-dispersion corrections from Cromer & Liberman (1970). The program package EUCLID (Spek, 1982) was used for geometrical calculations and illustrations. All calculations were carried out on a MicroVAX II cluster. Final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.* Bond distances and angles are given in Table 2. Fig. 1 shows the molecular structure with the adopted labelling scheme.

* Lists of anisotropic thermal parameters, H-atom positions, complete lists of bond distances and angles, selected torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54866 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0287]

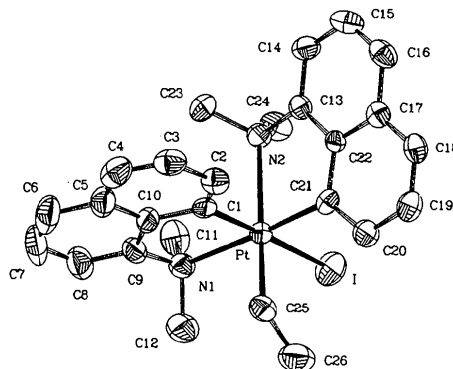


Fig. 1. Thermal ellipsoid plot (50% probability) of the molecular structure, with the adopted atom labelling. H atoms are omitted for clarity.

Related literature. Crystal structures have been reported for the related products bis(8-methoxy-1-naphthyl)methylplatinum(IV) iodide (Wehman, van Koten, Knaap, Ossor, Pfeffer & Spek, 1988) and *a*-chloro-*b*-dichloromethylstannio[*dc,ef*-bis(8-dimethylamino-1-naphthyl)]platinum(IV) (Smeets, Spek, van Beek & van Koten, 1992). Details of the chemistry will be published elsewhere (van Beek, Wehman-Ooyevaar, Grove, Smeets, Spek & van Koten, 1992).

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Bis[2-(2-hydroxyethyl)piperazinium] Hexachloroosmate(IV) Dichloride

BY J. M. ARRIETA*

Dpto de Química Inorgánica, Fac. de Ciencias, Universidad del País Vasco, Apdo 644, 48080 Bilbao, Spain

G. GERMAIN AND M. VLASSI

Unité de Chimie Physique Moléculaire et de Cristallographie, Bâtiment Lavoisier, Bt 35, Place Louis Pasteur 1, B-1348 Louvain-la-Neuve, Belgium

D. G. CRACIUNESCU AND E. PARRONDO IGLESIAS

Departamento de Química Bioinorgánica, Facultad de Farmacia, Universidad Complutense, Madrid, Spain

AND T. DEBAERDEMAEKER

Section für Röntgen und Elektronenbeugung, Universität Ulm, D-7900 Ulm, Germany

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Abstract. $[\text{C}_6\text{H}_{15}\text{N}_2\text{O}]_2[\text{Os}^{\text{IV}}\text{Cl}_6]\text{Cl}_2$, $M_r = 738.2$, monoclinic, $P2_1/a$, $a = 12.252(3)$, $b = 9.176(2)$, $c = 11.974(8)$ Å, $\beta = 115.06(3)^\circ$, $V = 1219(1)$ Å³, $Z = 2$, $D_x = 2.01$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 6.14$ mm⁻¹, $F(000) = 720$, $T = 298$ K, $R = 0.0679$ for 2159 observed reflections. The formula unit consists of the hexachloroosmate(IV) anion, two 2-(2-hydroxyethyl)piperazinium(2+) cations and two chloride ions. The complex is centrosymmetric with the Os atom at the origin. Weak intermolecular hydrogen bonds link the cations with the chloride ions.

Experimental. The complex was obtained under vigorous stirring and heating (363 K) from 1 g $\text{K}_2(\text{Os}^{\text{IV}}\text{Cl}_6)$ dissolved in 50 cm³ 6 M HCl and the corresponding amount of the ligand in 25 cm³ of a 1:1 mixture of ethanol and 6 M HCl. The stirring and heating continued until the solution reached one-third of its initial volume and then was left to form bright prismatic crystals at room temperature. Intensity data were collected from a crystal approximately 0.05 × 0.05 × 0.30 mm. The cell parameters were determined by least squares from 25 automatically centred reflections in the range $10 < 2\theta < 25^\circ$. 3063 independent reflections were measured with ω scans in the range $4 < 2\theta < 56^\circ$ on a Philips PW 1100

* Author to whom correspondence should be addressed.