chlorocuprate(II) salts range from 110·6 (4) (Clay, Murray-Rust & Murray-Rust, 1973) to 159 (3)° (Anderson & Willett, 1971).

The diisobutylammonium cation is linked to Cl atoms of $CuCl_4^{2-}$ anions by hydrogen bonds: Cl(1)···H(1) = 2·144 (2) and Cl(2)···H(2) = 2·248 (2) Å. The N—H(1)···Cl(1) angle is 173·2 (4)° and the N—H(2)···Cl(2) angle is 165·8 (4)°, with N···Cl(1) = 3·240 (8) and N···Cl(2) = 3·285 (7) Å. The N···Cl interatomic distances for tetrachlorocuprates span the range 3·11 (Lamotte-Brasseur, Dupont & Dideberg, 1973) to 3·55 Å (Russell & Wallwork, 1969).

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Acta Cryst. (1988). C**44**, 815–818

Structure of Tetrakis(biuret)samarium(III) Perchlorate

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(*Received* 9 *September* 1987; *accepted* 12 *January* 1988)

Abstract. $[Sm(C_2H_5N_3O_2)_4](ClO_4)_3$, $M_r = 861{\cdot}02$, monoclinic, $P2_1/c$, $a = 11{\cdot}90\,(2)$, $b = 12{\cdot}41\,(2)$, $c = 19{\cdot}93\,(4)$ Å, $\beta = 92{\cdot}5\,(1)^\circ$, $V = 2940\,(16)$ Å^3, $Z = 4$, $D_m = 2{\cdot}010$, $D_x = 1{\cdot}945$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 172$ cm^{-1}, $F(000) = 1700$, ambient temperature, $R = 0{\cdot}107$ for 1313 reflections with $\sin^2\theta > 0{\cdot}1$. All atoms in general positions. The four neutral biuret ligands chelate *via* carbonyl O atoms to Sm^{III} in a rather planar *trans–trans* configuration with a mean twist of 9·4 (2·7)° about the oxygen–oxygen line. The six-membered rings formed with Sm are folded by a mean value of 11·4 (6·3)° about the oxygen–oxygen line. The mean chelate bite angle is 69·9 (1·0)°. The four bidentate ligands span opposite edges of the two rectangular faces of an approximate square antiprism (D_{4d}). The three perchlorate moieties are ionic but deviate significantly from tetrahedral geometry, possibly due to hydrogen bonding, and exhibit rather high thermal parameters.

Introduction. Series of alkaline-earth and lanthanide halides, nitrates and perchlorate complexes with biuret were prepared by us and the crystal structures of tetrakis(biuret)strontium(II) perchlorate (Haddad & Gentile, 1975) and tetrakis(biuret)samarium(III) nitrate (Haddad, 1987) reported. The structure determination of tetrakis(biuret)samarium(III) perchlorate was undertaken as a prototype of lanthanide(III) perchlorate complexes with biuret. As indicated by preliminary X-ray photographs of the *h*0*l* zones all lanthanide(III) perchlorate complexes with biuret prepared under the same conditions are isostructural with tetrakis(biuret)samarium(III) perchlorate and belong to space group $P2_1/c$.

To our knowledge other X-ray structures of biuret and its complexes reported are those of biuret hydrate (Hughes, Yakel & Freeman, 1961), bis(biuret)cadmium(II) chloride (Cavalca, Nardelli & Fava, 1960), bis(biuret)copper(II) chloride (Freeman, Smith & Taylor, 1961), and bis(biuret)zinc(II) chloride (Nardelli, Fava & Giraldi, 1963). All of the above complexes of biuret are with transition metals.

Experimental. Biuret, $NH_2CONHCONH_2$, and hydrated samarium(III) perchlorate in the molar ratio of 6:1 were refluxed in minimum 95% ethanol for two days. An equal volume of *n*-butanol was then added and the mixture allowed to evaporate slowly in warm (320 K) dry atmosphere. Clear white crystals suitable for X-ray diffraction work separated as a second crop after several days. Dried *in vacuo*. M.p. 490 K (dec.). Analysis calculated for $Sm(C_2H_5N_3O_2)_4(ClO_4)_3$: C 11·16, N 19·52, H 2·34, Sm 17·46%; found: C 11·26, N 19·55, H 2·40, Sm 17·37%.

Density measured by flotation in carbon tetrachloride and methylene iodide. Unit-cell translations were determined from rotation photographs about *a*, *b* and *c* by extrapolation to a Bragg angle of 90° with σ of the intercept reported. β was determined from a Weissenberg photograph of the *h*0*l* zone with σ of the

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ac(\cos\beta)\beta_{13}].$$

	x	y	z	B_{eq}(Å^2)*
Sm	0·1379 (2)	0·1853 (2)	0·3477 (1)	2·20 (4)
O(11)	0·2742 (20)	0·2771 (22)	0·2852 (16)	4·1 (9)
O(12)	0·2521 (24)	0·0592 (23)	0·2912 (14)	4·2 (8)
N(11)	0·4049 (34)	0·3344 (42)	0·2198 (26)	6·8 (14)
N(12)	0·3652 (23)	0·1522 (37)	0·2208 (16)	3·7 (10)
N(13)	0·3510 (32)	−0·0365 (35)	0·2212 (19)	7·0 (15)
C(11)	0·3436 (28)	0·2565 (37)	0·2463 (19)	4·1 (12)
C(12)	0·3152 (34)	0·0552 (43)	0·2441 (26)	3·6 (12)
O(21)	0·0318 (20)	0·2862 (23)	0·2661 (14)	3·2 (7)
O(22)	0·0082 (24)	0·0722 (26)	0·2858 (14)	3·9 (8)
N(21)	−0·1094 (38)	0·3578 (39)	0·2018 (21)	6·8 (15)
N(22)	−0·1204 (34)	0·1850 (39)	0·2337 (20)	5·3 (11)
N(23)	−0·1498 (33)	0·0001 (28)	0·2415 (21)	5·4 (13)
C(21)	−0·0631 (52)	0·2780 (46)	0·2304 (25)	5·5 (17)
C(22)	−0·0806 (46)	0·0906 (44)	0·2516 (27)	3·9 (12)
O(31)	0·1574 (20)	0·3582 (22)	0·3922 (19)	4·2 (8)
O(32)	−0·0348 (18)	0·2424 (26)	0·3963 (14)	3·8 (8)
N(31)	0·1279 (31)	0·5309 (33)	0·4290 (22)	5·2 (12)
N(32)	−0·0227 (28)	0·4218 (29)	0·4072 (18)	4·3 (11)
N(33)	−0·1959 (22)	0·3426 (33)	0·4061 (22)	6·3 (15)
C(31)	0·0946 (37)	0·4331 (52)	0·4148 (21)	5·4 (16)
C(32)	−0·0834 (53)	0·3352 (51)	0·4040 (21)	3·5 (12)
O(41)	0·2791 (22)	0·1710 (34)	0·4314 (16)	4·5 (9)
O(42)	0·1074 (25)	0·0291 (22)	0·4097 (18)	4·3 (9)
N(41)	0·4162 (28)	0·1153 (35)	0·5116 (15)	4·8 (12)
N(42)	0·2540 (31)	0·0115 (32)	0·4969 (24)	4·9 (15)
N(43)	0·1006 (36)	−0·0979 (39)	0·4902 (23)	6·0 (14)
C(41)	0·3167 (51)	0·1041 (51)	0·4793 (27)	5·5 (15)
C(42)	0·1524 (42)	−0·0096 (31)	0·4619 (28)	4·5 (14)
Cl(1)	0·6114 (11)	0·1150 (12)	0·3396 (8)	6·0 (4)
O(51)	0·5780 (50)	0·2154 (65)	0·3391 (27)	11·1 (20)
O(52)	0·5307 (34)	0·0394 (40)	0·3732 (30)	8·1 (14)
O(53)	0·7136 (36)	0·1053 (35)	0·3780 (25)	9·1 (16)
O(54)	0·6223 (35)	0·0748 (53)	0·2731 (20)	7·8 (13)
Cl(2)	−0·3682 (14)	0·2302 (18)	0·1009 (9)	6·6 (4)
O(61)	−0·3426 (41)	0·3243 (73)	0·1358 (42)	12·7 (22)
O(62)	−0·2981 (61)	0·1476 (58)	0·1315 (32)	12·2 (20)
O(63)	−0·4798 (26)	0·1971 (53)	0·1086 (17)	8·3 (14)
O(64)	−0·3547 (41)	0·2537 (54)	0·0306 (22)	13·4 (24)
Cl(3)	0·1458 (12)	0·3002 (14)	0·5900 (6)	6·1 (4)
O(71)	0·0561 (35)	0·3163 (88)	0·5430 (21)	14·6 (24)
O(72)	0·1210 (47)	0·3552 (39)	0·6442 (26)	10·0 (17)
O(73)	0·1696 (87)	0·2002 (52)	0·6148 (39)	13·0 (21)
O(74)	0·2366 (50)	0·3444 (113)	0·5597 (28)	13·7 (24)

* At $R = 0·13$.

mean reported. The crystal used for data collection by the multi-film equi-inclination Weissenberg technique had the dimensions 0·11 × 0·09 × 0·07 mm ($b \times a \times c$) and was mounted along **b**. Nickel-filtered copper radiation. $h0l$–$h10l$. 1496 nonintegrated reflections were observable and were estimated visually. One fourth of the reciprocal lattice up to h, k, $\pm l$ of 15, 10, ±24. The raw intensities were reduced to structure amplitudes by applying Lp and cylindrical absorption corrections (Snyder & Spatola, 1968). Interlayer scales were set initially at unity with final values employed, towards the end of the refinement, equating $\sum|F_o|$ and $\sum|F_c|$ within each $h0l$–$h10l$ layer. Scattering curves for H, C, N, O and Cl from self-consistent wave functions (Cromer & Waber, 1965), while for Sm from Thomas–Fermi–Dirac statistical model (Thomas & Umeda, 1957). Scattering factors for Sm and Cl were corrected for f' and f'' (Cromer, 1965).

Structure determination, by the heavy-atom method, and refinement, by block-diagonal least squares, were performed on an IBM360 employing NRC programs (Ahmed, Hall, Pippy & Huber, 1973). The function minimized in least squares was $\sum w(|F_o| - |F_c|)^2$ with $w = 1·00$. The final residual is 0·107 for 1313 reflections with $\sin^2\theta > 0·1$, and 0·121 for all 1496 observable reflections. Mean $\Delta/\sigma = 0·40$. All non-H atoms were refined anisotropically; however, anisotropic refinement reduced R by only 0·02. No attempts were made to locate the H atoms. The highest peak in the final difference map was 0·7 e Å^{-3}. The final positional parameters and isotropic thermal parameters at $R = 0·13$ are listed in Table 1.*

Discussion. Fig. 1 shows the asymmetric and molecular unit, $[Sm(C_2H_5N_3O_2)_4](ClO_4)_3$, H atoms excluded, and indicates the labeling scheme. As in the strontium(II) perchlorate analogue (Haddad & Gentile, 1975), and its samarium(III) nitrate analogue (Haddad, 1987), the four biuret ligands chelate to the metal cation *via* the carbonyl O atoms in a rather planar *trans–trans* configuration. The mean dimensions $\{\sigma(\bar{x}) = [\sum(x-\bar{x})^2/(n-1)]^{1/2}\}$ for the bonded biuret in the three structures are given in Fig. 2. It is expected that the higher ionic potential of Sm^{III} compared with Sr^{II} should lead in the Sm^{III} cases to longer C—O bonds with more single-bond character and shorter C—N bonds with more double-bond character. This seems to be generally true although the data are not statistically very significant. The mean C—O bond length in the two Sm^{III} cases is 1·241 (5) and 1·27 (5) Å, respectively, compared with 1·22 (3) Å in the Sr^{II} case. The mean C—N(terminal) bond length in the two Sm^{III} cases is 1·320 (8) and 1·33 (5) Å, respectively, compared with 1·37 (2) Å in the Sr^{II} case.

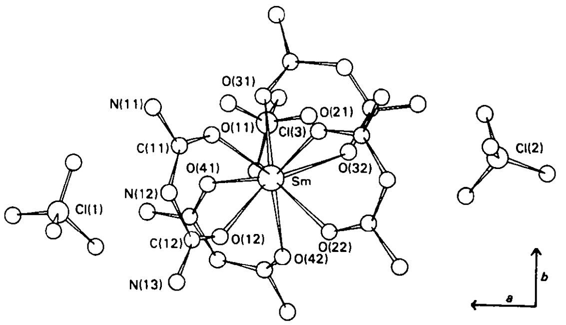

Fig. 1. Schematic drawing of the molecular and asymmetric unit, $[Sm(C_2H_5N_3O_2)_4](ClO_4)_3$, H atoms excluded, projected on the ab plane.

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* Lists of structure factors, anisotropic thermal parameters, least-squares planes, schematic drawing showing distances within the coordination polyhedron and another showing angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44682 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The twist about the O(1)···O(2) line, defined as the dihedral angle between the best planes O(1), C(1), N(1), N(2) and O(2), C(2), N(3), N(2) (Table 5, deposited material), is for the four biuret ligands, i = 1–4, 6·72, 8·84, 13·12, and 9·00° with a mean value of 9·4 (2·7)°. The fold about the O(1)···O(2) line, defined as the dihedral angle between the best ligand's plane and the O(1), Sm, O(2) plane (Table 5, deposited material), is for the four biuret ligands, i = 1–4, 7·24, 16·20, 17·39, and 4·96° with a mean value of 11·4 (6·3)°. The mean chelate bite angle O(1)—Sm—O(2) is 69·9 (1·0)°, while the mean chelate bite along the O(1)···O(2) line is 2·71 (1) Å.

The perchlorate moieties are ionic but seem to deviate significantly from tetrahedral geometry. The very high thermal parameters of the perchlorate O atoms render the determination of the perchlorate dimensions inaccurate. The perchlorate O atoms are extensively hydrogen-bonded to the amide N atoms (Table 2) as supported by the infrared spectrum.

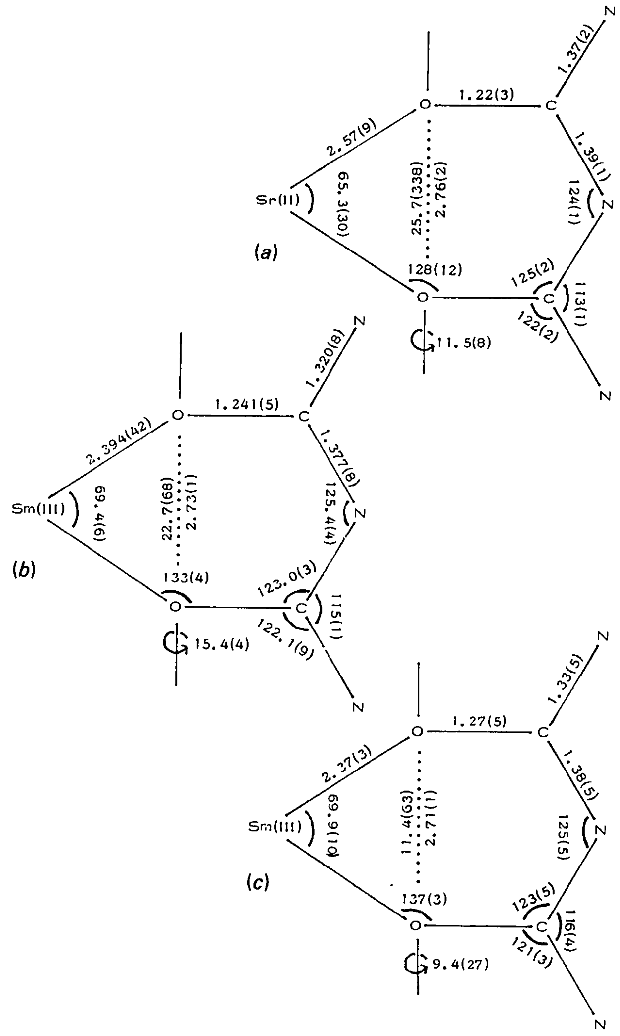

Fig. 2. Schematic drawing showing the mean distances (Å) and angles (°) in the chelated biuret: (*a*) tetrakis(biuret)strontium(II) perchlorate, (*b*) tetrakis(biuret)samarium(III) nitrate, (*c*) tetrakis(biuret)samarium(III) perchlorate.

Table 2. *Bond lengths* (Å), *bond angles* (°), *and hydrogen-bond lengths* (Å) *with e.s.d.'s in parentheses*

	$i = 1$	$i = 2$	$i = 3$	$i = 4$
Sm–O(i1)	2·38 (3)	2·37 (3)	2·33 (3)	2·32 (3)
Sm–O(i2)	2·39 (3)	2·39 (3)	2·42 (2)	2·34 (3)
C(i1)–O(i1)	1·19 (5)	1·31 (6)	1·29 (6)	1·33 (7)
C(i2)–O(i2)	1·23 (5)	1·25 (6)	1·30 (7)	1·25 (6)
C(i1)–N(i1)	1·33 (6)	1·26 (7)	1·30 (7)	1·33 (7)
C(i2)–N(i3)	1·30 (7)	1·40 (7)	1·35 (5)	1·39 (6)
C(i1)–N(i2)	1·42 (6)	1·34 (7)	1·40 (6)	1·42 (7)
C(i2)–N(i2)	1·43 (7)	1·31 (7)	1·30 (7)	1·40 (6)
O(i1)–Sm–O(i2)	69·6 (9)	69·1 (9)	69·5 (10)	71·4 (11)
Sm–O(i1)–C(i1)	139 (3)	139 (3)	139 (3)	141 (3)
Sm–O(i2)–C(i2)	140 (3)	133 (3)	134 (3)	134 (3)
O(i1)–C(i1)–N(i1)	121 (4)	122 (5)	125 (5)	123 (5)
O(i2)–C(i2)–N(i3)	121 (4)	114 (4)	121 (4)	118 (4)
O(i1)–C(i1)–N(i2)	125 (4)	118 (5)	119 (4)	121 (5)
O(i2)–C(i2)–N(i2)	119 (4)	127 (5)	119 (4)	133 (4)
N(i1)–C(i1)–N(i2)	114 (4)	119 (5)	114 (4)	112 (3)
N(i3)–C(i2)–N(i2)	118 (4)	118 (5)	120 (4)	110 (4)
C(i1)–N(i2)–C(i2)	125 (4)	127 (5)	130 (4)	119 (4)

O(61)···N(21)	3·05 (7)	N(41)···O(52^{ii})	3·04 (7)
O(62)···N(22)	2·91 (8)	N(42)···O(53^{ii})	2·90 (7)
O(71)···N(32)	3·11 (7)	N(23)···O(54)	2·96 (6)
N(21)···O(12^{i})	3·03 (5)	N(11)···O(54^{i})	3·01 (8)
N(23)···O(21^{i})	3·01 (4)	N(31)···O(62^{i})	2·81 (8)
N(21)···O(22^{i})	2·93 (6)	N(12)···O(63)	3·01 (4)
N(43)···O(32^{ii})	3·02 (5)	N(31)···O(71^{ii})	2·97 (9)
N(21)···O(42^{i})	3·08 (6)		

Symmetry code: (i) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $-x, -y, -z$.

Although full shape parameters (Muetterties & Guggenberger, 1974) were not computed for the eight-coordination polyhedron, it is obvious through model inspection that the coordination polyhedron is closer to the square antiprism (D_{4d}) than the dodecahedron (D_{2d}). All of the faces of the dodecahedron are equilateral triangles, but the spatial arrangements of the group of atoms O(11), O(12), O(21), O(22) and the group of atoms O(31), O(32), O(41), O(42) are much closer to two squares than to four equilateral triangles (dimensions given in Figs. 3 and 4, deposited material). The internal angles in the two approximately square faces are, respectively, 86·9 (11), 92·4 (12), 93·0 (12), 87·2 (12)° and sum to 360°, and 88·8 (13), 89·9 (12), 96·4 (14), 82·2 (12)° and sum to 358°, indicating arrangements close to planarity. On the other hand, the sum of internal angles in the two theoretically planar dodecahedral *BAAB* trapezoids O(12), O(11), O(31), O(32) and O(21), O(22), O(42), O(41) are 346 and 352°, respectively, indicating significant deviation from planarity in at least the first *BAAB* trapezoid.

In the crystal structures of tetrakis(biuret)strontium(II) perchlorate (Haddad & Gentile, 1975) and tetrakis(biuret)samarium(III) nitrate (Haddad, 1987) as well as in this structure, tetrakis(biuret)samarium(III) perchlorate, biuret ligands span opposite edges of the square faces of the square antiprism, D_{4d}. In the first two structures the metal is on a crystallographic 2 axis, while in the the third the metal is in a general position. This seems to indicate that for these tetrakis complexes the metal's site symmetry is not the deciding factor in the choice of the coordination polyhedron.

Data collection and reduction were performed at The University of Jordan, and structure determination and refinement at Fordham University, Bronx, NY, to which we are grateful. We are also grateful to D. D. Cavalluzzo for her help in the final stages of refinement.

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Acta Cryst. (1988). C**44**, 818–820

Structure of 1,2,2,2,3,4,4,4-Octacarbonyl-1,2;3,4-di-μ-hydrido-1,3-bis(tricyclohexylphosphine)-*tetrahedro*-diplatinumdiosmium(4*Pt–Os*)(*Os–Os*)

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(*Received* 24 *August* 1987; *accepted* 1 *February* 1988)

Abstract. $[\mathrm{Os_2Pt_2}(\mu\text{-H})_2(\mathrm{CO})_8\{\mathrm{P(C_6H_{11})_3}\}_2]$, $M_r = 1557{\cdot}6$, monoclinic, $P2_1/a$, $a = 19{\cdot}713\,(2)$, $b = 10{\cdot}482\,(4)$, $c = 24{\cdot}146\,(6)$ Å, $\beta = 92{\cdot}25\,(1)°$, $V = 4985\,(2)$ Å^3, $Z = 4$, $D_x = 2{\cdot}08$ g cm^{-3}, $\lambda(\mathrm{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 108{\cdot}5$ cm^{-1}, $F(000) = 2936$, $T = 298$ K, $R = 0{\cdot}036$ for 4515 observed reflections. The dihedral butterfly angle of the metal framework is 84·2 (1)°, and the Pt(1)···Pt(2) distance is 3·230 (1) Å, compared with corresponding magnitudes of 82·9° and 3·206 (1) Å found in the related triphenylphosphine derivative.

Introduction. The dihedral 'butterfly' angle in several tetraruthenium clusters has been found to vary widely (Carty, MacLaughlin, Wagner & Taylor, 1982), and for $[\mathrm{AuFe_3}(\mu_3\text{-HC{=}NBu^t})(\mathrm{CO})_9(\mathrm{PPh_3})]$ this angle has values of 110·9 and 132·1° for the two molecules within the same asymmetric unit (Bruce & Nicholson, 1983). Molecular-dynamical calculations on $[\mathrm{Fe_4}(\mu_4\text{-C})(\mathrm{CO})_{12}]^{2-}$ also suggest that the deformation of this angle is a soft mode (Bogdan, Horwitz & Shriver, 1986). The title butterfly cluster complex (1) has been examined structurally to compare parameters with those of the previously determined triphenylphosphine analogue (2) (Farrugia, Howard, Mitrprachachon, Stone & Woodward, 1981).

Experimental. Thin yellow plates from hexane solution: crystal dimensions 0·30 × 0·29 × 0·016 mm; systematic absences: $h = 2n + 1$ in $h0l$; $k = 2n + 1$ in $0k0$; Enraf–Nonius CAD-4F diffractometer; graphite monochromator; $\theta/2\theta$ scan mode; cell parameters refined by least-squares method on basis of 25 independent θ values, $11 < \theta < 13°$; intensities measured to $\theta = 25{\cdot}0°$ over range of hkl 0 to 23, 0 to 12, −28 to +28; $11,2,\bar{2}$, 402, $20\bar{2}$ measured every 2 h with nonlinear decay corrected by two linear sections, corresponding to 8% decay over 150 h data collection; 9573 reflections measured, 8757 independent data with 4515 having $I > 2{\cdot}0\sigma(I)$ considered observed and used in structure determination and refinement; R_{int} before absorption correction 0·20, after correction 0·097; corrected for Lorentz/polarization, absorption [Gaussian grid (Coppens, 1970) using 512 grid points; range of transmission factors on F^2, 0·061 to 0·829]; solved by direct methods (*MITHRIL*; Gilmore, 1984) and subsequent full-matrix least squares; isotropic thermal parameters for cyclohexyl-ring C atoms, anisotropic for remaining non-H atoms; H atoms included at fixed calculated positions [cyclohexyl C—H = 1·0 Å: hydrides (*HYDEX*; Orpen, 1980) Os—H, Pt—H *ca* 1·85 Å], with fixed isotropic ($U = 0{\cdot}05$ Å^2) thermal parameters; $w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; max.

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