trois H [HB(11), HB(21) et HB(41)] sont en moyenne à 2 Å de l'U, trois autres [HB(12), HB(22) et HB(42)] à 2,5 Å et les trois derniers à 2,8 Å. Un hydrogène du BH_4 bidenté est aussi à 2,1 Å de l'atome d'uranium, ce qui porte à quatre le nombre d'hydrogènes très proches de l'uranium. Une étude de diffraction de neutrons serait toutefois nécessaire pour confirmer la position de ces hydrogènes et vérifier en particulier la régularité du tétraèdre BH_4 lié, prise dans ce travail comme hypothèse.

La comparaison des structures du complexe $U(BH_4)_4[OP(C_6H_5)_3]_2$ et de son solvate avec le benzène met aussi en évidence des différences dans la géométrie des ligands oxyde de phosphine. Les distances U—O(1) et U—O(2) de 2,33 (2) et 2,24 (2) Å sont proches de celle obtenue dans le complexe solvaté [U—O 2,27 (1) Å], les angles O—U—O étant pratiquement identiques (180°) dans les deux complexes. Par contre, l'angle U—O—P de 170,8 (7)° dans le solvate est très différent de l'un des angles U—O(1)—P(1) et U—O(2)—P(2), respectivement de 156 (1) et 168 (1)°, ce qui correspond à une configuration très dissymétrique.

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Structure of a Complex of Platinum(II) with 2-(2-Chloro-5-nitrostyryl)benzothiazole

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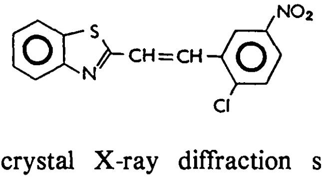
Abstract. Tetraethylammonium tribromo[2-(2-chloro-5-nitrostyryl)benzothiazole]platinate(II), $[(C_2H_5)_4N][PtBr_3(C_{15}H_9ClN_2O_2S)]$, $M_r = 881\cdot8$, monoclinic, $P2_1/c$, $a = 9\cdot454$ (4), $b = 20\cdot137$ (10), $c = 15\cdot071$ (7) Å, $\beta = 99\cdot25$ (3)°, $V = 2831\cdot9$ Å^3, $D_x = 2\cdot07$ Mg m^{-3}, $Z = 4$, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu(\text{Mo } K\alpha) = 9\cdot39$ mm^{-1}, $F(000) = 1680$, $T = 169$ K, final $R = 0\cdot050$ for 2286 observed reflections. The $[PtBr_3(nsb)]^-$ unit has square-planar geometry about the Pt, with the nsb coordinated to the Pt through the N of the thiazole ring. The ligand is non-planar, with a dihedral angle of 60·1 (4)° between the benzothiazole and the nitrobenzene rings.

Introduction. Platinum(II) complexes with imidazole, thiazole and other *N*-donor heterocyclic ligands have been studied for their potential antitumor properties (Dehand & Jordanov, 1975, 1976; Van Kralingen & Reedijk, 1978; Van Kralingen, Reedijk & De Ridder, 1979). Some Schiff bases derived from thiazoles and benzothiazoles (Dash, Patra & Praharaj, 1980) and several derivatives of the styrylbenzothiazoles (Cox, Jackson, Vargas, Baez, Colon, Gonzalez & de Leon, 1982) have also shown biological activity. The complex $[PtBr_3(nsb)]^-$ where nsb is 2-(2-chloro-5-nitrostyryl)benzothiazole, is of particular interest because of the possible synergism between the nsb ligand, shown below, and the metal ion.



A single-crystal X-ray diffraction study of this complex has been undertaken for two principal reasons: (1) to determine unequivocally through which atom, N or S, the ligand is coordinated to the platinum atom; and (2) to ascertain the full three-dimensional structural configuration of the complex, in order to study the structure–activity relationship of platinum complexes with antitumor activity.

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Experimental. 5 ml of an acetone solution of 0·056 g (0·178 mmol) of 2-(2-chloro-5-nitrostyryl)benzothiazole were added slowly to 5 ml of an acetone solution of 0·100 g (0·088 mmol) of $[NEt_4]_2[Pt_2Br_6]$. The reaction mixture was heated and stirred for 30 min and left to evaporate at room temperature. A yellow powder was obtained which yielded 67% product which was recrystallized from nitromethane (Cadiz, 1985).

Diffraction data were collected by Molecular Structure Corp., and refined in this laboratory. Yellow needle crystal, D_m not determined, 0·15 × 0·07 × 0·07 mm, Enraf–Nonius CAD-4 diffractometer, monochromated Mo $K\alpha$ radiation, ω–θ scan, scan speed 2–20° min^{-1} in ω, scan width 0·7 + 0·35 tanθ°, $2\theta_{\max} = 50°$, lattice parameters from least-squares fitting of 25 reflections in the range $3 < \theta < 7°$. Systematic absences $0k0$ ($k = 2n+1$), and $h0l$ ($l = 2n+1$); $\pm h + k + l$ with $|h| \le 11$, $k \le 23$, $l \le 17$; three standard reflections, intensities constant within experimental error; total unique reflections 4994, 2708 'observed' reflections, 2286 with $F^2 > 2\sigma(F_o)^2$ used in structure refinement, $\sigma(F_o)^2 = [\sigma(I_{\text{raw}})^2 + (0{\cdot}04F_o{}^2)^2]^{1/2}$. Absorption corrections from ψ scans, relative transmission factors 0·569–1·000. Structure was solved with the Enraf–Nonius *Structure Determination Package* (Frenz, 1986), heavy-atom and difference-Fourier methods; isotropic then anisotropic refinement on F of all non-hydrogen atoms, H atoms calculated [C—H 0·95 Å, $U(\text{H}) = 1{\cdot}3U(\text{C})$]; 307 parameters refined, $R = 0{\cdot}050$, $wR = 0{\cdot}056$, $w = 4(F_o)^2/[\sigma(F_o)^2]^2$, $S = 1{\cdot}1$; difference syntheses showed a max. density of 0·94 e Å^{-3} (min. −0·97 e Å^{-3}); max. $\Delta/\sigma = 0{\cdot}06$. Neutral-atom scattering factors were used, except for Pt^{II} [all from *International Tables for X-ray Crystallography* (1974)], and were corrected for anomalous dispersion (Cromer & Liberman, 1970). No correction for secondary extinction. Other computer program used: *SHELX*76 (Sheldrick, 1976).

Discussion. The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1, and bond lengths and angles in Table 2.* An *ORTEP* plot (Johnson, 1976) of the complex anion and the atom numbering is shown in Fig. 1, and the packing of the molecules (Motherwell & Clegg, 1976), in Fig. 2.

The $[\text{PtBr}_3(\text{nsb})]^-$ unit has square-planar geometry about the Pt with the nsb coordinated through the N of the thiazole ring, and a Pt—N bond length of 2·01 (1) Å. No atom deviates from the least-squares plane of coordination by more than 0·034 (2) Å [$\chi^2 = 5{\cdot}0$]. The nsb is a potentially ambidentate ligand with either the N or S atom available for coordination. Although Pt—S coordination might be expected to be more stable according to Pearson's 'hard and soft acids and bases' principle (Pearson, 1963), it is evident from this study that nsb is coordinated through the N atom. This behavior has also been observed in a Pd^{II} benzothiazole complex (Churchill, Wasserman & Young, 1980).

The average C—N, C—S and C—C distances [1·35 (4), 1·73 (1), 1·40 (3) Å] and bond angles [120 (2) and 108 (11)°] for the coordinated benzothiazole are similar to those found in the Pd^{II} complex. It should be mentioned that the large size of the S atom compared with the N atom results in a reduction of the C(6)—S—C(7) angle [89·6 (7)°] compared with the C(7)—N(1)—C(1) angle [111 (1)°] and the other internal angles [113 (3)°] within the thiazole ring. This suggests that the S atom might be using unhybridized p orbitals for bonding.

The nsb is non-planar with a dihedral angle of 60·1 (4)° between the benzothiazole and nitrobenzene rings. The twist occurs at C(9)—C(10). Possible factors which might contribute to this deviation from planarity include: (1) loss of aromaticity as the N coordinates to the metal ion; (2) packing of the molecules in the unit cell; (3) electronic repulsions between the H atoms of

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

	x	y	z	B_{eq}
Pt	0·78186 (7)	0·65748 (3)	0·26575 (4)	2·43 (1)
Br(1)	0·5624 (2)	0·71394 (8)	0·2888 (1)	3·56 (4
Br(2)	0·7687 (2)	0·57509 (9)	0·3815 (1)	4·34 (4)
Br(3)	0·9971 (2)	0·60486 (9)	0·2301 (1)	4·51 (4)
N(1)	0·795 (1)	0·7268 (6)	0·1713 (7)	2·4 (3)
C(1)	0·835 (2)	0·7918 (6)	0·1895 (8)	2·1 (3)
C(2)	0·875 (2)	0·8195 (7)	0·277 (1)	2·8 (3)
C(3)	0·913 (2)	0·8859 (7)	0·284 (1)	3·3 (4)
C(4)	0·913 (2)	0·9238 (7)	0·207 (1)	3·3 (4)
C(5)	0·873 (2)	0·8989 (6)	0·123 (1)	2·6 (3)
C(6)	0·832 (2)	0·8323 (6)	0·1140 (9)	2·3 (3)
S	0·7830 (5)	0·7848 (2)	0·0177 (2)	2·71 (8)
C(7)	0·769 (2)	0·7161 (7)	0·0839 (9)	2·6 (3)
C(8)	0·727 (2)	0·6517 (7)	0·0454 (8)	2·7 (3)
C(9)	0·650 (2)	0·6391 (7)	−0·0355 (9)	2·8 (3)
C(10)	0·626 (2)	0·5706 (7)	−0·0692 (8)	2·9 (3)
C(11)	0·726 (2)	0·5215 (8)	−0·0458 (9)	3·2 (4)
C(12)	0·697 (2)	0·4571 (7)	−0·0759 (9)	2·8 (3)
C(13)	0·571 (2)	0·4392 (7)	−0·131 (1)	4·5 (4)
C(14)	0·468 (2)	0·4889 (7)	−0·156 (1)	3·8 (4)
C(15)	0·498 (2)	0·5525 (8)	−0·1236 (9)	3·5 (4)
Cl	0·3612 (6)	0·6107 (3)	−0·1497 (3)	5·5 (1)
N(2)	0·805 (2)	0·4062 (6)	−0·0482 (8)	4·1 (3)
O(1)	0·920 (1)	0·4210 (6)	−0·0026 (7)	4·8 (3)
O(2)	0·772 (2)	0·3491 (5)	−0·0665 (9)	6·2 (4)
N(3)	0·299 (1)	0·6464 (6)	0·5158 (8)	3·3 (3)
C(16)	0·295 (2)	0·7141 (7)	0·467 (1)	3·1 (4)
C(17)	0·196 (2)	0·6468 (7)	0·580 (1)	3·4 (4)
C(18)	0·455 (2)	0·6384 (8)	0·565 (1)	3·9 (4)
C(19)	0·252 (2)	0·5905 (8)	0·450 (1)	4·2 (4)
C(20)	0·146 (2)	0·7312 (8)	0·414 (1)	4·4 (4)
C(21)	0·214 (2)	0·7004 (9)	0·651 (1)	4·6 (4)
C(22)	0·484 (2)	0·577 (1)	0·622 (2)	6·7 (6)
C(23)	0·342 (2)	0·5824 (9)	0·372 (1)	5·0 (5)

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as:

$$B_{\text{eq}} = \tfrac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

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* Lists of structure factors, anisotropic thermal factors, H-atom parameters and least-squares-planes information have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43782 (57 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°)

Pt—Br(1)	2·439 (2)	C(10)—C(15)	1·40 (2)
Pt—Br(2)	2·425 (2)	C(11)—C(12)	1·39 (2)
Pt—Br(3)	2·430 (2)	C(12)—C(13)	1·39 (2)
Pt—N(1)	2·01 (1)	C(12)—N(2)	1·46 (2)
N(1)—C(1)	1·38 (2)	C(13)—C(14)	1·41 (2)
N(1)—C(7)	1·32 (2)	C(14)—C(15)	1·38 (2)
C(1)—C(2)	1·42 (2)	C(15)—Cl	1·74 (2)
C(1)—C(6)	1·40 (2)	N(2)—O(1)	1·23 (2)
C(2)—C(3)	1·38 (2)	N(2)—O(2)	1·21 (2)
C(3)—C(4)	1·39 (2)	N(3)—C(16)	1·55 (2)
C(4)—C(5)	1·36 (2)	N(3)—C(17)	1·48 (2)
C(5)—C(6)	1·40 (2)	N(3)—C(18)	1·55 (2)
C(6)—S	1·74 (1)	N(3)—C(19)	1·52 (2)
S—C(7)	1·72 (1)	C(16)—C(20)	1·54 (2)
C(7)—C(8)	1·45 (2)	C(17)—C(21)	1·51 (2)
C(8)—C(9)	1·34 (2)	C(18)—C(22)	1·51 (3)
C(9)—C(10)	1·48 (2)	C(19)—C(23)	1·56 (3)
C(10)—C(11)	1·37 (2)		
Br(1)—Pt—Br(2)	94·22 (7)	C(9)—C(10)—C(11)	121· (1)
Br(1)—Pt—Br(3)	175·22 (7)	C(9)—C(10)—C(15)	121· (1)
Br(1)—Pt—N(1)	85·9 (4)	C(11)—C(10)—C(15)	117· (1)
Br(2)—Pt—Br(3)	90·12 (6)	C(10)—C(11)—C(12)	120· (1)
Br(2)—Pt—N(1)	178·9 (4)	C(11)—C(12)—C(13)	123· (1)
Br(3)—Pt—N(1)	89·8 (4)	C(11)—C(12)—N(2)	118· (1)
Pt—N(1)—C(1)	124·4 (8)	C(13)—C(12)—N(2)	119· (1)
Pt—N(1)—C(7)	125· (1)	C(12)—C(13)—C(14)	118· (1)
C(1)—N(1)—C(7)	111· (1)	C(13)—C(14)—C(15)	118· (1)
N(1)—C(1)—C(2)	125· (1)	C(10)—C(15)—C(14)	124· (1)
N(1)—C(1)—C(6)	115· (1)	C(10)—C(15)—Cl	120· (1)
C(2)—C(1)—C(6)	120· (1)	C(14)—C(15)—Cl	116· (1)
C(1)—C(2)—C(3)	119· (1)	C(12)—N(2)—O(1)	120· (1)
C(2)—C(3)—C(4)	120· (1)	C(12)—N(2)—O(2)	117· (1)
C(3)—C(4)—C(5)	123· (1)	O(1)—N(2)—O(2)	122· (1)
C(4)—C(5)—C(6)	118· (1)	C(16)—N(3)—C(17)	110· (1)
C(1)—C(6)—C(5)	121· (1)	C(16)—N(3)—C(18)	106· (1)
C(1)—C(6)—S	109· (1)	C(16)—N(3)—C(19)	111· (1)
C(5)—C(6)—S	130· (1)	C(17)—N(3)—C(18)	111· (1)
C(6)—S—C(7)	89·6 (7)	C(17)—N(3)—C(19)	107· (1)
N(1)—C(7)—S	115· (1)	C(18)—N(3)—C(19)	113· (1)
N(1)—C(7)—C(8)	123· (1)	N(3)—C(16)—C(20)	113· (1)
S—C(7)—C(8)	122· (1)	N(3)—C(17)—C(21)	117· (1)
C(7)—C(8)—C(9)	127· (1)	N(3)—C(18)—C(22)	116· (1)
C(8)—C(9)—C(10)	121· (1)	N(3)—C(19)—C(23)	116· (1)

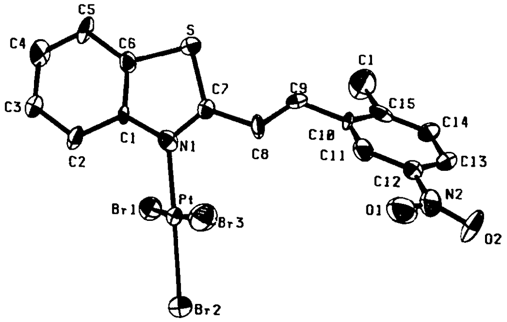

Fig. 1. Labeling of atoms in the $[PtBr_3(nsb)]^-$ anion (50% probability ellipsoids).

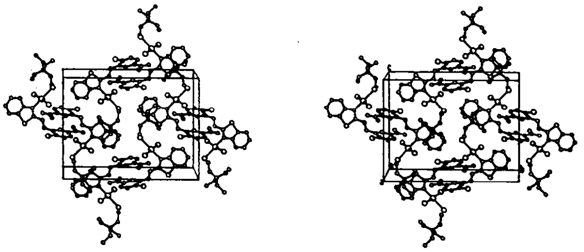

Fig. 2. Packing diagram of $[NEt_4][PtBr_3(nsb)]$ molecules in unit cell.

C(8) and C(11). From the packing diagram it can be seen that the nitrobenzene rings are stacked in a parallel but staggered form. The distance between these rings is 3·53 (1) Å. The deviation from planarity facilitates the approach of these rings to a distance which permits interaction of the π electrons without losing packing efficiency.

Within the nitrobenzene ring no atom deviates significantly from the least-squares plane [$\chi^2 = 5{\cdot}9$]. The benzothiazole moiety is essentially planar with no atom deviating from the plane by more than 0·034 (15) Å [$\chi^2 = 9{\cdot}0$]. The dihedral angles between the $PtNBr_3$ plane and the benzothiazole and nitrobenzene planes are 73·9 (2) and 79·3 (3)° respectively. The closest non-bonding distance involving the non-hydrogen atoms of the nsb ligand is 2·23 (2) Å [C(1)···C(7)].

Distances and angles within the tetraethylammonium cation are all normal. The closest intramolecular contact involving the non-hydrogen atoms is 2·40 (2) Å [C(17)···C(19)].

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Structures of Bis(aqua)bis(isoquinoline)copper(II) Perchlorate and of Tetrakis(isoquinoline)bis(trifluoromethanesulfonato)copper(II)

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Abstract. Compound (1): $[Cu(C_9H_7N)_2(H_2O)_2](ClO_4)_2$, $M_r = 556{\cdot}5$, triclinic, $P\bar{1}$, $a = 7{\cdot}487\,(2)$, $b = 7{\cdot}621\,(2)$, $c = 10{\cdot}271\,(4)$ Å, $\alpha = 100{\cdot}82\,(2)$, $\beta = 108{\cdot}33\,(3)$, $\gamma = 99{\cdot}03\,(2)°$, $V = 531{\cdot}6\,(7)$ Å^3, $Z = 1$, $D_m = 1{\cdot}60$ (flotation in *n*-octane + 1,2-dibromoethane), $D_x = 1{\cdot}74$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0{\cdot}71070$ Å) = 1·359 mm^{-1}, $F(000) = 277$, $T = 294$ K, $R = 0{\cdot}041$ and $wR = 0{\cdot}035$ for 2741 significant reflections. Octahedral copper environment: two water molecules in *trans* positions and two isoquinolines in *trans* positions for basal plane, and two perchlorate anions in apical positions. Each perchlorate anion connected to three different copper atoms through semicoordinated O and through two hydrogen bonds. Compound (2): $[Cu(C_9H_7N)_4(CF_3SO_3)_2]$, $M_r = 877{\cdot}5$, triclinic, $P\bar{1}$, $a = 10{\cdot}882\,(3)$, $b = 18{\cdot}219\,(8)$, $c = 22{\cdot}384\,(3)$ Å, $\alpha = 67{\cdot}49\,(2)$, $\beta = 88{\cdot}64\,(2)$, $\gamma = 83{\cdot}34\,(3)°$, $V = 4071\,(4)$ Å^3, $Z = 4$, $D_m = 1{\cdot}30$ (flotation in *n*-octane + 1,2-dibromoethane), $D_x = 1{\cdot}43$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0{\cdot}71070$ Å) = 0·643 mm^{-1}, $F(000) = 1568$, $T = 294$ K, $R = 0{\cdot}071$ and $wR = 0{\cdot}049$ for 3540 significant reflections. Octahedral copper environment: four isoquinolines in basal plane and two monodentate anions semicoordinated through O in apical positions.

Introduction. A large number of studies have been carried out on complexes of copper(II) with pyridine. Some work involved isoquinoline, a monodentate aromatic amine of larger size than pyridine, but with similar donor properties. For example, from spectroscopic measurements, copper(II) complexes $[CuL_2Cl_2]$ (L = quinoline or isoquinoline) are supposed to have polymeric octahedral structures involving halogen bridges as for pyridine complexes (Patel & Ramana Rao, 1966). Also copper(II) complexes $[Cu(isoquin)_n(CF_3CO_2)_2]$ (where n = 2 or 4) have been studied by spectrometric methods (Agambar & Orrell, 1969). They are described as six-coordinated with trifluoroacetate (either mono- or bidentate). While looking for dinuclear copper complexes, we have synthesized and studied by X-ray diffraction $CuL_2(ClO_4)_2$ [compound (1)] and $CuL_4(CF_3SO_3)_2$ [compound (2)] with L = isoquinoline.

Experimental. Compound (1): $1{\cdot}4 \times 10^{-4}$ mol isoquinoline poured into 4 ml of a solution of $CuClO_4.6H_2O$ in propanone. Precipitated white powder filtered after 24 h. Blue solution slowly evaporated 10 days under nitrogen, giving bright green air-stable crystals. Crystal: 0·5 × 0·2 × 0·1 mm; precessions and Laue: $P\bar{1}$; Nonius CAD-4 diffractometer, graphite monochromator; Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71070$ Å); unit cell: least squares on 25 reflections, $8 < \theta < 28°$; intensity measurements by θ–2θ scan; $(\sin\theta)/\lambda$ range: 0·04–

Table 1. *Atomic parameters for compound* (1) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Cu	0·0000	0·0000	0·0000	2·75 (1)
O	0·1669 (2)	0·2297 (2)	−0·0037 (2)	3·70 (5)
N	0·0343 (2)	0·1116 (2)	0·2011 (2)	3·07 (4)
C(1)	−0·1128 (3)	0·0913 (3)	0·2451 (2)	3·15 (5)
C(2)	−0·1005 (3)	0·1652 (3)	0·3844 (2)	3·12 (5)
C(3)	−0·2639 (4)	0·1388 (4)	0·4259 (3)	4·08 (7)
C(4)	−0·2463 (4)	0·2123 (4)	0·5621 (3)	4·62 (8)
C(5)	−0·0657 (5)	0·3188 (4)	0·6608 (3)	4·73 (9)
C(6)	0·0939 (4)	0·3463 (4)	0·6244 (3)	4·44 (7)
C(7)	0·0815 (3)	0·2693 (3)	0·4846 (2)	3·40 (6)
C(8)	0·2371 (3)	0·2878 (4)	0·4343 (3)	4·13 (7)
C(9)	0·2111 (3)	0·2113 (3)	0·2982 (3)	3·73 (6)
Cl	0·34891 (6)	−0·26676 (6)	0·05836 (5)	2·86 (1)
O(1)	0·3092 (3)	−0·0884 (2)	0·0917 (2)	4·46 (5)
O(2)	0·4390 (3)	−0·2734 (4)	−0·0458 (3)	5·97 (3)
O(3)	0·4712 (4)	−0·3023 (4)	0·1815 (3)	6·62 (9)
O(4)	0·1688 (2)	−0·3984 (2)	−0·0014 (3)	5·20 (7)

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