

The first Cp*Os half-sandwich complexes containing chalcogenido ligands¹

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Abstract

The reactions of the pentamethylcyclopentadienyl-osmium compound Cp*Os(NO)Br₂ (**1**) with Na₂S₄ and (NEt₄)₂Se₆ in dimethylformamide solution lead to mononuclear tetrachalcogenido complexes Cp*Os(NO)(E₄) (E = S (**2a**), Se (**2b**)), whereas binuclear products Cp₂*Os₂(NO)₂(μ-E)₂ (E = S (**3a**), Se (**3b**), Te (**3c**)) are obtained from the reactions of **1** with Li₂E in tetrahydrofuran solution. Dechalcogenation of **2a,b** using tri(n-butyl)phosphane gives **3a,b**. The molecular geometry of Cp*Os(NO)(Se₄) (**2b**) has been determined by an X-ray crystal structure analysis which confirms the presence of a non-planar cyclotetraselenido chelate ligand.

Keywords: Osmium; Sulfur; Selenium; Tellurium; Pentamethylcyclopentadienyl half-sandwich compounds; Chalcogenide complexes; Crystal structure

1. Introduction

Owing to their thermal stability and solubility in organic solvents, the mononuclear pentamethylcyclopentadienyl (Cp* M) half-sandwich compounds of the third-row transition metals (M = W, Re, Ir) have been used extensively as models for reactivity studies. Typical complexes containing oligochalcogenido chelate ligands (E_n) are presented in Table 1.

Comparable Cp*Os complexes were so far unknown. We now report the synthesis of the mononuclear tetrachalcogenides Cp*Os(NO)(E₄) (E = S (**2a**), Se (**2b**)) and their facile dechalcogenation to the binuclear chalcogen-bridged derivatives Cp₂*Os₂(NO)₂(μ-E)₂ (E = S (**3a**), Se (**3b**)).

2. Results and discussion

A suitable educt for our Cp*Os complexes is Cp₂*Os₂Br₂(μ-Br)₂, recently described by Girolami and coworkers [13]. Treatment of this compound with either

NO gas or “diazald” (4-MeC₆H₄-SO₂N-(NO)Me) gave the violet nitrosyl complex Cp*Os(NO)Br₂ (**1**), which was reacted with anionic oligochalcogenide sources such as Na₂S₄, (NEt₄)₂Se₆ [14] and (NⁿBu₄)₂Te₅ [15] in warm dimethylformamide (DMF) solution (Scheme 1).

Whereas the mononuclear tetrachalcogenides Cp*Os(NO)(E₄) (E = S (**2a**), Se (**2b**)) could be easily obtained, only the binuclear product Cp₂*Os₂(NO)₂(μ-E)₂ (E = Te (**3c**)) was isolated from the reaction of **1** with an excess of the pentatelluride (NⁿBu₄)₂Te₅ in DMF solution. The binuclear complexes **3a–c** are best prepared, however, by treating **1** with a limited amount of monochalcogenide, Li₂E, in THF solution. The silyl-substituted chalcogenanes E(SiMe₂R)₂ (E = S, Se, R = Me; E = Te, R = ⁿBu) have also been successfully used as precursors of the monochalcogenide bridges in **3a–c**.

The mononuclear complexes **2a,b** and their binuclear counterparts **3a,b** can be interconverted (Scheme 1). Elimination of chalcogen from **2a,b** takes place in CH₂Cl₂ solution in the presence of tri(n-butyl)phosphane (PⁿBu₃) to give EPⁿBu₃ (E = S, Se). In contrast, compounds **3a,b** take up chalcogen from the oligochalcogenide salts Na₂S₄ and (NEt₄)₂Se₆ respectively to reform the mononuclear compounds **2a,b**. The incor-

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¹ Dedicated to Professor Rudolf Taube on the occasion of his 65th birthday.

Table 1

Mononuclear pentamethylcyclopentadienyl (Cp* M) half-sandwich complexes of tungsten, rhenium, osmium and iridium containing oligochalcogenido ligands

| Cp* W | Cp* Re | Cp* Os | Cp* Ir |
|---|--|---|---|
| Cp* W(L)(X)(E ₂) L = O; E = S X = Me [1], I [2], Cl, OMe [3] L = S; E = S X = Me [1], I [2], CH ₂ SiMe ₃ [1], Cl, OMe [3] L = N ^t Bu; E = S, Se X = Cl [4] | Cp* Re(CO) ₂ (E ₂) E = S [6], Se Cp* ReCl ₂ (E ₃) E = S, Se [7] Cp* Re(S ₃ XS ₄) [8] Cp* Re(L)(E ₄) L = O, E = S [7–10], Se [7,9] L = N ^t Bu; E = S, Se [7] | Cp* Os(NO)(E ₄) ^a E = S, Se | Cp* Ir(PMe ₃)(E ₂) E = Se [11], Te Cp* Ir(COX)(S ₄) [12] Cp* Ir(PMe ₃)(E ₄) E = S, Se [11] Cp* Ir(PMe ₃)(E _n) E _n = S ₅ , S ₆ [11] |
| Cp* W(NO)(E ₅) E = S, Se [5] | | | |

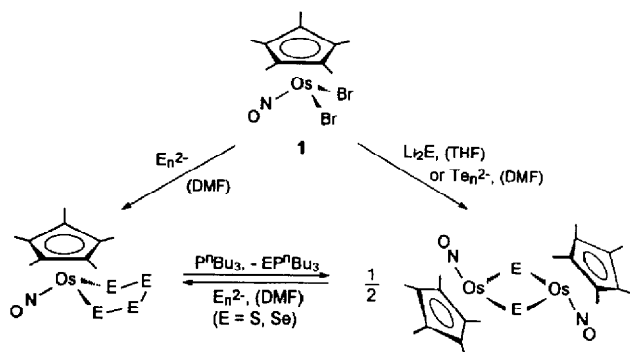
^a Present work.

poration of sulfur and selenium into **3a,b** from the elements proceeds considerably more slowly.

The spectroscopic data of the new complexes **1**, **2a,b** and **3a–c** are summarized in Table 2. The nitrosyl stretching frequency $\nu(\text{NO})$ increases if the chalcogen is varied in a given complex type in the order S < Se < Te, indicating that the heavier chalcogens are better acceptor ligands than sulfur. The EI mass spectra consistently contain the molecular ion; the most prominent fragment ions are Cp* OsE₂⁺ in the case of **2a,b** and Cp₂* Os₂E₂⁺ in the case of **3a–c**. The molecular geometry of Cp* Os(NO)(Se₄) (**2b**) has been determined by a single-crystal X-ray structure analysis. The proposed molecular structure of the binuclear compounds is consistent with all available spectroscopic data and is also supported by an X-ray structure analysis of Cp₂* Ru₂(NO)₂(μ -Te)₂ [16], which is analogous to **3c**.

2.1. Molecular structure of Cp* Os(NO)(Se₄) (**2b**)

The asymmetric unit of the triclinic cell ($Z = 4$) contains two symmetry-independent but chemically equivalent molecules, A and B. As an example, Fig. 1 presents the geometry of one Cp* Os(NO)(Se₄) molecule (**2b-A**) with the numbering system. Table 3 gives selected intramolecular distances and angles for both



Scheme 1.

molecules, **2b-A** and **2b-B**. The molecular dimensions are similar in A and B, although the atoms of the Cp* ligand have large vibrational ellipsoids and the standard deviations of all distances and angles related to the Cp* rings are relatively high (Table 4).

Complex **2b** possesses basically a trilegged half-sandwich Cp* Os structure. The five-membered metallacycle OsSe₄ has a distorted envelope conformation similar to the MSe₄ rings found in Cp* Re(O)(Se₄) [7], Cp* Re(N^tBu)(Se₄) [7] and Cp* Ir(PMe₃)(Se₄) [11]. The bite angle of the cyclotetraselenido chelate ligand (Se–Os–Se 94.6(1)°) is in the range 94–97°, which is typical for half-sandwich Cp* M(Se₄) compounds [7,11]. The nitrosyl ligand deviates slightly from linearity with Os–N–O angles of 170.4(18)° (A) and 173.4(24)° (B).

3. Experimental details

3.1. Syntheses

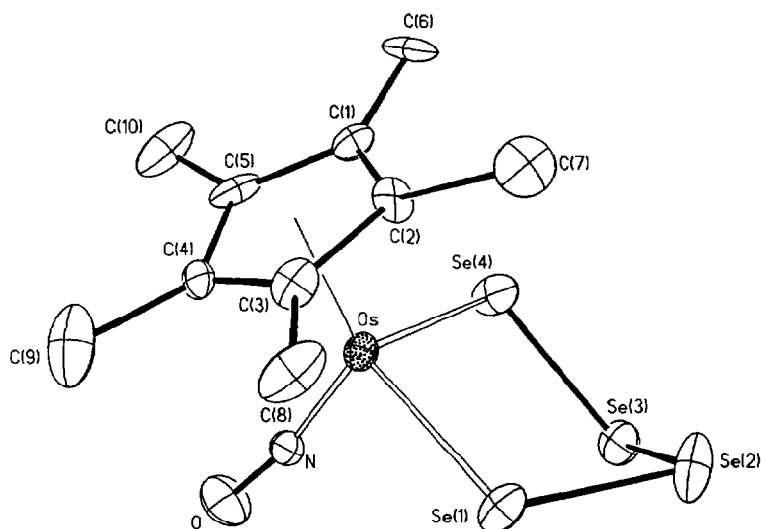
3.1.1. Cp* Os(NO)Br₂ (**1**)

NO gas was bubbled through a suspension of 1 g (1.03 mmol) [Cp* OsBr₂]₂ [13] in 80 ml of CH₂Cl₂ for 30 min. The violet solution was then stirred under NO atmosphere for 2 h at room temperature. The solvent was removed in vacuo, and the residue separated by column chromatography on silica (Merck, Kieselgel

Table 2
Spectroscopic characterization

| Complex | IR ^a $\nu(\text{NO})$ | ¹ H NMR ^b $\delta(\text{CH}_3)$ | ¹³ C NMR ^b $\delta(\text{C}_5\text{Me}_5)$ | $\delta(\text{C}_5\text{Me}_5)$ |
|---|-------------------------------------|--|---|---------------------------------|
| Cp* Os(NO)Br ₂ (1) | 1765 | 2.04 | 10.2 | 105.4 |
| Cp* Os(NO)(S ₄) (2a) | 1724 | 2.05 | 9.9 | 105.7 |
| Cp* Os(NO)(Se ₄) (2b) | 1740 | 2.10 | 10.6 | 104.6 |
| Cp ₂ * Os ₂ (NO) ₂ (μ -S) ₂ (3a) | 1676 | 2.04 | 10.3 | 105.5 |
| Cp ₂ * Os ₂ (NO) ₂ (μ -Se) ₂ (3b) | 1685 | 1.99 | 9.9 | 102.8 |
| Cp ₂ * Os ₂ (NO) ₂ (μ -Te) ₂ (3c) | 1707 | 2.13 | 10.9 | 99.7 |

^a CsI pellet [cm⁻¹]. ^b CDCl₃ solution.

Fig. 1. Geometry of $\text{Cp}^* \text{Os}(\text{NO})(\text{Se}_4)$ molecule **2b-A** with atom numbering scheme.

60). Elution with neat CH_2Cl_2 developed a violet zone, and recrystallization from $\text{CHCl}_3/\text{hexane}$ at -25°C gave 0.85 g (80%) violet crystals of **1**, no decomp. up to 250°C . EI-MS (70 eV): m/e 515 (M^+ , 36%), 485 ($\text{M}^+ - \text{NO}$, 17%), 436 ($\text{M}^+ - \text{HBr}$, 35%), 405 ($\text{Cp}^* \text{OsBr}^+$, 100%).

3.1.2. $\text{Cp}^* \text{Os}(\text{NO})(\text{E}_4)$ ($\text{E} = \text{S}$ (**2a**), Se (**2b**))

A solution of 0.30 g (0.58 mmol) **1** and 0.17 g (0.98 mmol) Na_2S_4 in 20 ml of DMF was stirred at $70\text{--}80^\circ\text{C}$ for 3 h. After removal of the solvent the crude product **2a** was purified by column chromatography on silica (using neat CH_2Cl_2 for elution). Recrystallization from

Table 3
Selected interatomic distances [pm] and angles [$^\circ$] in $\text{Cp}^* \text{Os}(\text{NO})(\text{Se}_4)$ (**2b**)

| Molecule A | | Molecule B | |
|-------------------------|-----------|-----------------------------|-----------|
| <i>Distances</i> | | | |
| Os–Se(1) | 246.7(3) | Os'–Se(1') | 247.1(3) |
| Os–Se(4) | 247.1(3) | Os'–Se(4') | 247.1(3) |
| Os–N | 173.7(21) | Os'–N' | 174.8(20) |
| N–O | 119.3(30) | N'–O' | 116.8(26) |
| Os–centre Cp* | 193.0 | Os'–centre Cp** | 192.5 |
| Se(1)–Se(2) | 237.2(5) | Se(1')–Se(2') | 234.5(4) |
| Se(2)–Se(3) | 229.6(4) | Se(2')–Se(3') | 230.9(6) |
| Se(3)–Se(4) | 234.5(4) | Se(3')–Se(4') | 235.0(5) |
| <i>Angles</i> | | | |
| Se(1)–Os–Se(4) | 94.6(1) | Se(1')–Os'–Se(4') | 94.5(1) |
| Se(1)–Os–N | 99.2(6) | Se(1')–Os'–N' | 96.9(8) |
| Se(4)–Os–N | 95.1(6) | Se(4')–Os'–N' | 98.6(8) |
| Os–N–O | 170.4(18) | Os'–N'–O' | 173.4(24) |
| Os–Se(1)–Se(2) | 108.3(1) | Os'–Se(1')–Se(2') | 108.3(1) |
| Se(1)–Se(2)–Se(3) | 97.6(2) | Se(1')–Se(2')–Se(3') | 97.2(1) |
| Se(2)–Se(3)–Se(4) | 96.3(4) | Se(2')–Se(3')–Se(4') | 96.7(2) |
| Se(3)–Se(4)–Os | 107.4(1) | Se(3')–Se(4')–Os' | 107.8(1) |
| <i>Torsion angles</i> | | | |
| Se(4)–Os–Se(1)–Se(2) | 13.0 | Se(4')–Os'–Se(1')–Se(2') | 16.8 |
| Os–Se(1)–Se(2)–Se(3) | –45.4 | Os–Se(1')–Se(2')–Se(3') | –47.9 |
| Se(1)–Se(2)–Se(3)–Se(4) | 56.8 | Se(1')–Se(2')–Se(3')–Se(4') | 56.8 |
| Se(2)–Se(3)–Se(4)–Os | –52.7 | Se(2')–Se(3')–Se(4')–Os | –50.1 |
| Se(3)–Se(4)–Os–Se(1) | 23.6 | Se(3')–Se(4')–Os'–Se(1') | 20.1 |

Table 4
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

| Atom | x | y | z | U_{eq}^* |
|--------|-----------|------------|-----------|-------------------|
| Os | 4813(9) | 12865.5(7) | 6156.3(7) | 32(1) |
| Os' | 1655.5(9) | 8768.6(8) | 8906.1(7) | 37(1) |
| Se(1) | 2817(2) | 13617(2) | 6263(2) | 51(1) |
| Se(2) | 1604(3) | 12234(3) | 6755(3) | 73(2) |
| Se(3) | 1723(2) | 10301(2) | 5625(2) | 52(1) |
| Se(4) | 4042(3) | 10769(2) | 6217(2) | 56(1) |
| Se(1') | 2792(3) | 7027(2) | 8984(2) | 56(1) |
| Se(2') | 1178(4) | 5070(2) | 8255(3) | 95(2) |
| Se(3') | -160(3) | 5805(3) | 9163(2) | 73(2) |
| Se(4') | -524(3) | 7492(3) | 8607(2) | 60(1) |
| N | 4373(16) | 12244(16) | 4801(15) | 40(9) |
| O | 4257(20) | 11905(18) | 3903(14) | 80(11) |
| N' | 2184(21) | 9647(18) | 10263(16) | 55(11) |
| O' | 2585(20) | 10336(17) | 11154(16) | 78(11) |
| C(1) | 6657(21) | 13180(19) | 7617(18) | 36(11) |
| C(2) | 6028(22) | 14131(19) | 7873(16) | 37(10) |
| C(3) | 6069(22) | 14913(18) | 7304(19) | 42(11) |
| C(4) | 6646(21) | 14348(18) | 6583(18) | 35(11) |
| C(5) | 7039(21) | 13245(20) | 6766(18) | 43(11) |
| C(6) | 6986(24) | 12257(20) | 8096(18) | 52(12) |
| C(7) | 5525(27) | 14458(20) | 8797(19) | 63(14) |
| C(8) | 5595(23) | 16118(18) | 7416(21) | 60(13) |
| C(9) | 6973(29) | 14877(23) | 5848(22) | 84(17) |
| C(10) | 7865(23) | 12470(23) | 6299(22) | 76(15) |
| C(1') | 1597(49) | 8372(27) | 7266(24) | 83(21) |
| C(2') | 2770(33) | 9244(46) | 7975(38) | 100(29) |
| C(3') | 2440(26) | 10359(23) | 8514(19) | 52(13) |
| C(4') | 1063(24) | 10149(21) | 8153(20) | 43(13) |
| C(5') | 574(30) | 8933(29) | 7343(21) | 68(15) |
| C(6') | 1524(77) | 7115(35) | 6395(34) | 445(105) |
| C(7') | 4206(36) | 9301(50) | 8173(42) | 288(76) |
| C(8') | 3400(47) | 11616(33) | 9384(29) | 244(39) |
| C(9') | 397(46) | 11147(34) | 8511(32) | 168(40) |
| C(10') | -778(36) | 8361(39) | 6663(32) | 204(35) |

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

$\text{CH}_2\text{Cl}_2/\text{CHCl}_3/\text{hexane}$ at -25°C gave 0.19 g (67.3%) red crystals of **2a**. EI-MS: m/e 485 (M^+ , 38%), 455 (M^+-NO , 60%), 421 (M^+-2S , 20%), 391 ($\text{Cp}^*\text{OsS}_2^+$, 100%), 357 ($\text{Cp}^*\text{Os}(\text{NO})^+$, 60%).

An analogous procedure led to **2b**. Starting from 0.30 g (0.58 mmol) **1** and 0.80 g (1.09 mmol) $(\text{NEt}_4)_2\text{Se}_6$ [14], product **2b** (0.22 g, 56.1%) was isolated as dark-red crystals, m.p. 224°C . EI-MS: m/e 673 (M^+ , 17%), 643 (M^+-NO , 15%), 515 (M^+-2Se , 33%), 485 ($\text{Cp}^*\text{OsSe}_2^+$, 100%), 403 (Cp^*OsSe^+ , 18%), the most intense peak of the isotope pattern is listed.

3.1.3. $\text{Cp}_2^*\text{Os}_2(\text{NO})_2(\mu\text{-E})_2$ ($\text{E} = \text{Se}$ (**3a**), Se (**3b**), Te (**3c**))

Lithium chalcogenides Li_2E were prepared by reaction of elemental chalcogen with $\text{Li}[\text{HB}(\text{Et})_3]$ (1:2) in THF solution. A mixture of ca. 0.80 mmol Li_2E and 0.30 g (0.58 mmol) **1** in 40 ml of THF was then stirred at room temperature for two to three days. The solvent

THF was evaporated, and the crude product chromatographed over silica. Neat CH_2Cl_2 was used to elute complexes **3a–c**, which were finally recrystallized from either THF/hexane or toluene/hexane at -25°C to give dark-red prismatic crystals (no decomp. up to 250°C).

$\text{Cp}_2^*\text{Os}_2(\text{NO})_2(\mu\text{-S})_2$ (**3a**, 0.14 g, 62.1%). IR: $\nu(\text{Os-SOs})$ 357 cm^{-1} . EI-MS: m/e 774 (M^+ , 2%), 744 (M^+-NO , 2%), 714 (M^+-2NO , 100%), 580 ($\text{M}^+-2\text{NO-Cp}^*$, 40%), 357 ($(\text{M}-2\text{NO})^{2+}$, 36%).

$\text{Cp}_2^*\text{Os}_2(\text{NO})_2(\mu\text{-Se})_2$ (**3b**, 0.16 g, 63.3%). IR: $\nu(\text{OsSeOs})$ 367 cm^{-1} . EI-MS: m/e 870 (M^+ , 20%), 840 (M^+-NO , 8%), 810/808 (M^+-2NO , 90/100%), 790 (M^+-Se , 14%), 405 ($(\text{M}-2\text{NO})^{2+}$, 59%).

$\text{Cp}_2^*\text{Os}_2(\text{NO})_2(\mu\text{-Te})_2$ (**3c**, 0.20 g, 71.2%). EI-MS: m/e 968 (M^+ , 46%), 938 (M^+-NO , 7%), 908 (M^+-2NO , 100%), 454 ($(\text{M}-2\text{NO})^{2+}$, 80%). Anal. Found: C, 25.11; H, 3.32; Os, 39.6; Te, 26.6. $\text{C}_{20}\text{H}_{30}\text{N}_2\text{O}_2\text{Os}_2\text{Te}_2$ (966.07) Calc.: C, 24.87; H, 3.13; Os, 39.38; Te, 26.42%.

3.2. Chalcogen elimination and insertion reactions

3.2.1. Dechalcogenation of $\text{Cp}^*\text{Os}(\text{NO})(\text{E}_4)$ (**2a,b**)

A solution of **2a** (0.20 g, 0.41 mmol) and tri(*n*-butyl)phosphane (0.29 g, 1.44 mmol) in 40 ml of CH_2Cl_2 was stirred for 1 h at ambient temperature. The solvent was then evaporated and the residue separated by column chromatography over silica. Elution with neat CH_2Cl_2 gave an oily mixture of **3a** and $^n\text{Bu}_3\text{PS}$. The mixture was treated with toluene/hexane (1:2) and the crude product **3a** recrystallized from THF/hexane. Yield 0.07 g, 40.3%, dark-red crystals.

An analogous reaction of **2b** (0.24 g, 0.36 mmol) with excess P^nBu_3 (0.24 g, 1.26 mmol) afforded 0.09 g (57.7%) dark-red crystals of **3b**.

3.2.2. Chalcogenation of $\text{Cp}_2^*\text{Os}_2(\text{NO})_2(\mu\text{-E})_2$ (**3a,b**)

A solution containing **3a** (0.12 g, 0.16 mmol) and Na_2S_4 (0.08 g, 0.47 mmol) in 20 ml DMF was stirred at 70°C for 90 min. After evaporation of the solvent, the residue was chromatographed on silica and the product **2a** (0.10 g, 66.7%) eluted with CH_2Cl_2 .

The corresponding reaction of **3b** (0.14 g, 0.16 mmol) with excess $(\text{Et}_4\text{N})_2\text{Se}_6$ (0.30 g, 0.40 mmol) in 30 ml DMF at 80°C gave 0.15 g (69.1%) **3b** as dark-red crystals.

3.3. Crystal structure determination of $\text{Cp}^*\text{Os}(\text{NO})(\text{Se}_4)$ (**2b**) [17]

A dark-red crystal ($0.05 \times 0.23 \times 0.25\text{ mm}^3$) was mounted with epoxy cement on a thin glass fiber. $\text{C}_{10}\text{H}_{15}\text{NOOsSe}_4$ (671.3), triclinic, space group $P\bar{1}$; $a = 1095.6(3)$, $b = 1134.9(4)$, $c = 1459.1(6)$ pm; $\alpha = 109.99(3)$, $\beta = 108.05(3)$, $\gamma = 96.66(2)^\circ$; $V = 1570(1)$

$\times 10^6 \text{ pm}^3$; $Z = 4$. $D_{\text{calc.}} = 2.839 \text{ g cm}^{-3}$; $\mu(\text{MoK } \alpha) = 17.376 \text{ mm}^{-1}$; 296 K.

Data collection: Siemens P4, graphite monochromator, MoK α radiation ($\lambda = 71.073 \text{ pm}$), 2θ scan range $4.0\text{--}43.0^\circ$. Collected reflections 3401, independent reflections 3231, independent observed reflections [$F_o \geq 5\sigma(F_o)$] 2056.

Solution and refinement: direct methods, completed by subsequent difference Fourier syntheses and refined by full-matrix least-squares procedures; semi-empirical laminar absorption correction, all data with a glancing angle of not more than 3° to the prominent face [001] were rejected, affecting 347 reflections. Siemens SHELXTL PLUS (4.2) program libraries, $R(F) = 3.82\%$, $R(wF) = 4.50\%$, Δ/σ (max) = 0.034; $\Delta(\rho) = 1.43 \times 10^{-6} \text{ e pm}^{-3}$, $N_o/N_v = 6.7$; GOF 1.03.

All non-hydrogen atoms were refined with anisotropic displacement parameters, whereas hydrogen atoms were treated as idealized contributions. Table 4 contains atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients.

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