Cleavage of Ge–S and C–H bonds in the reaction of electron-deficient [Os$_3$(CO)$_8$(μ-H)(μ$_3$-Ph$_2$PCH$_2$P(Ph)C$_6$H$_4$)] with Ph$_3$GeSPh: Generation of thiophenol derivatives [Os$_3$(CO)$_8$(μ-H)(μ-SPh)(μ-dppm)] and [Os$_3$(CO)$_7$(μ-H)(μ-SPh)(μ$_3$-SC$_6$H$_4$)(μ-dppm)]

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ABSTRACT

Heating the electron-deficient [Os$_3$(CO)$_8$(μ-H)(μ$_3$-Ph$_2$PCH$_2$P(Ph)C$_6$H$_4$)] (**1**) and Ph$_3$GeSPh in benzene at 80 °C led to the thiolato bridged compounds, [Os$_3$(CO)$_8$(μ-H)(μ-SPh)(μ-dppm)] (**2**) and [Os$_3$(CO)$_7$(μ-H)(μ-SPh)(μ$_3$-SC$_6$H$_4$)(μ-dppm)] (**3**), formed by cleavage of Ge–S and C–S bonds of the ligand, in 40% and 17% yields, respectively. Both compounds **2** and **3** have been characterized by a combination of elemental analysis, infrared and ^{1}H NMR spectroscopic data together with single crystal X-ray crystallography. Compound **3** contains an open triangle of osmium atoms bridged by a SPh and SC$_6$H$_4$ ligands on opposite sides of the cluster with a dppm ligand bridging one of the Os–Os edges. Compound **2** consists of a closed triangular cluster of osmium atoms with a bridging SPh, and a bridging hydride ligand on the same Os–Os edge, and a dppm ligand bridging one of the remaining Os–Os edges.

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1. Introduction

The electron-deficient triosmium cluster [Os$_3$(CO)$_8$(μ-H)(μ$_3$-Ph$_2$PCH$_2$P(Ph)C$_6$H$_4$)] (**1**) derived from the decarbonylation of the decacarbonyl compound [Os$_3$(CO)$_{10}$(μ-dppm)] has attracted considerable interest for many years and its chemistry has been thoroughly investigated during the last decade to reveal its catalytic potential as well as applications in organic synthesis [1–15]. With an electron count of 46, cluster **1** is electron-deficient with respect to the noble gas rule which requires 48 electrons for a closed trinuclear cluster; for this reason it is susceptible to nucleophilic attack and reacts with a wide range of electron donor ligands under mild conditions relative to electron precise complexes.

The reactions of **1** with a wide variety of small inorganic and organic ligands such as CO [4], H$_2$ [5], PR$_3$ [6], P(OR)$_3$ (R = Me, Et, Pr^i, Bu, Ph) [6], PPh$_2$H [7], RC≡CR (R = Ph, C$_6$H$_4$Me, Me, CF$_3$) [8], [Au(PPh$_3$)]PF$_6$ [9], EtSH [10], CH$_3$CH(CH$_3$)SH [10], PhSH [10], pySH [11], HSCH$_2$CH$_2$SH [12], HSCH$_2$CH$_2$CH$_2$SH [12], Se [13], HX (X = Cl, Br, F, CF$_3$CO$_2$, CH$_3$CO$_2$) [14], CH$_2$N$_2$ [15], silane [16] and Ph$_3$SnH [17] that afford many interesting and potentially useful compounds have been investigated (Scheme 1). We have recently reported some unusual bimetallic Ru–Sn, Ru–Ge clusters, e.g. [Ru$_3$(CO)$_8$(μ-SPh)$_2$(μ$_3$-SnPh$_2$)(SnPh$_3$)$_2$] and [Ru$_3$(CO)$_8$(μ-SPh)$_2$(μ$_3$-GePh$_2$)(GePh$_3$)$_2$], from the reactions of Ph$_3$SnSPh or Ph$_3$GeSPh with [Ru$_3$(CO)$_{12}$], respectively [18]. We also reported the Os-Sn compound [Os$_3$(CO)$_9$(μ-SPh)(μ$_3$-SnPh$_2$)(NCMe)(η^1-C$_6$H$_5$)$_2$] from the reaction of [Os$_3$(CO)$_{10}$(NCMe)$_2$] with Ph$_3$SnSPh.
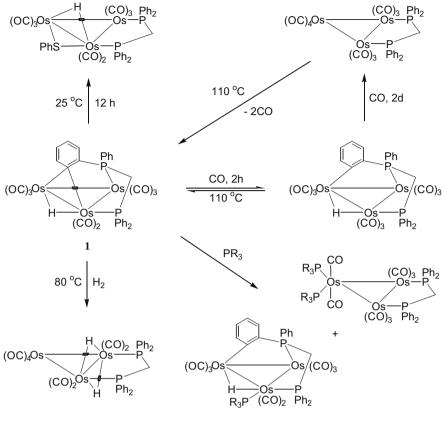
As a part of our studies on bimetallic Os–Ge clusters, we set out to investigate the reactivity of the electron-deficient **1** with Ph$_3$GeSPh. Unfortunately, the reaction does not appear to give any Os–Ge product, instead only the thiolato part of the ligand incorporated into the cluster resulting in the formation of [Os$_3$(CO)$_8$(μ-H)(μ-SPh)(μ-dppm)] (**2**) and [Os$_3$(CO)$_7$(μ-H)(μ-SPh)(μ$_3$-SC$_6$H$_4$)(μ-dppm)] (**3**).

2. Experimental

All the reactions were performed under a nitrogen atmosphere using standard Schlenk techniques. Solvents were dried and distilled prior to use by standard methods. The starting complex [Os$_3$(CO)$_8$(μ-H)(μ$_3$-Ph$_2$PCH$_2$P(Ph)C$_6$H$_4$)] (**1**) and the ligand Ph$_3$GeSPh were prepared according to published procedures [4,19]. Infrared spectra were recorded on a Shimadzu FTIR 8101 spectrophotometer. ^{1}H NMR spectra were recorded on a Bruker DPX 400 spectrometer. Elemental analyses were performed by

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Scheme 1.

2.1. Reaction of [Os₃(CO)₈(μ-H)(μ₃-Ph₂PCH₂P(Ph)C₆H₄)] (1) with Ph₃GeSPh

A benzene (20 mL) solution of **1** (50 mg, 0.042 mmol) and Ph₃GeSPh (35 mg, 0.084 mmol) was heated to reflux for 9 h during which time the color changed from green to yellow. The progress of the reaction was followed by spot TLC. The solvent was removed under reduced pressure and the residue chromatographed by TLC on silica gel. Elution with hexane/CH_2Cl_2 (7:3, v/v) developed three bands. The second band gave the unreacted starting compound **1** (6 mg) while the first and third bands afforded [Os₃(CO)₈(μ-H)(μ-SPh)(μ-dppm)] (**2**) (22 mg, 40%) as orange crystals and [Os₃(CO)₇(μ-H)(μ-SPh)(μ₃-SC₆H₄)(μ-dppm)] (**3**) (10 mg, 17%) as pale yellow crystals after recrystallization from hexane/CH_2Cl_2 at 4 °C. Spectral data for **2**: Anal. Calc. for C₃₉H₂₈O₈Os₃P₂S: C, 36.33; H, 2.19. Found: C, 36.55; H, 2.23%. IR (νCO, CH_2Cl_2): 2067 vs, 2023 m, 1993 vs, 1970 m, 1955 m, 1923 w cm⁻¹. ¹H NMR (CDCl₃): δ 7.66 (m, 2H), 7.53 (m, 3H), 7.42 (m, 5H), 7.32 (m, 6H), 7.13 (m, 9H), 5.73 (dd, 1H, J = 24.2, 12.5 Hz), 4.56 (dd, 1H, J = 24.2, 12.5 Hz), −15.76 (d, 1H, J = 29.6 Hz). ³¹P–{¹H} NMR (CDCl₃): δ −24.1 (br, s), −24.8 (br, s). FAB mass spectrum: *m/z* 1290 (M⁺), 1262 (M⁺−CO), 1234 (M⁺−2CO), 1206 (M⁺−3CO), 1178 (M⁺−4CO), 1150 (M⁺−5CO), 1122 (M⁺−6CO), 1094 (M⁺−7CO), 1066 (M⁺−8CO). Spectral data for **3**: Anal. Calc. for C₄₄H₃₂O₇Os₃P₂S₂: C, 38.59; H, 2.36. Found: C, 38.73; H, 2.41%. IR (νCO, CH_2Cl_2): 2056 s, 2041 vs, 1996 s, 1983 vs, 1953 w, 1940 w cm⁻¹. ¹H NMR (CDCl₃): δ 7.83 (dd, 1H, J = 6.8, 3.6 Hz), 7.64 (dd, 1H, J = 6.0, 3.6 Hz), 7.58 (m, 3H), 7.38 (m, 8H), 7.26 (m, 3H), 7.12 (m, 5H), 6.90 (m, 6H), 6.72 (m, 2H), 2.97 (m, 1H), 1.58 (m, 1H), −15.66 (t, 1H, J = 7.2 Hz). ³¹P–{¹H} NMR (CDCl₃): δ 17.2 (d, J = 94.8 Hz), 21.3 (d, J = 94.8 Hz). FAB mass spectrum: *m/z* 1370 (M⁺), 1342 (M⁺−CO), 1314 (M⁺−2CO), 1286 (M⁺−3CO), 1258 (M⁺−4CO), 1230 (M⁺−5CO), 1202 (M⁺−6CO), 1174 (M⁺−7CO).

2.2. X-ray crystallography

Single crystals were mounted on fibres and diffraction data collected at low temperatures (see Table 1) on Bruker AXS SMART APEX CCD diffractometers using Mo Kα radiation (λ = 0.71073 Å). Data collection, indexing and initial cell refinements were all done using SMART [20] software. Data reduction was done with SAINT [21] software and the SADABS program [22] was used to apply empirical absorption corrections. The structures were solved by direct methods [23] and refined by full matrix least-squares on F^2 [24]. All non-hydrogen atoms were refined anisotropically and hydrogen atoms were included using a riding model. For compound **3** the refinement with just the cluster molecule included converged with R_1 = 0.093. There was considerable diffuse electron density still remaining (2–3 e A⁻³) and PLATON calculated very large voids in the lattice. Obviously very poorly ordered solvent, probably CH_2Cl_2 was present. Since this could not be modeled, it was eliminated using SQUEEZE procedures [25]. After this treatment refinement converged to R_1 = 0.074. The disordered solvent, combined with a small poorly diffracting crystal, meant that refinement was less successful than normal. Only the Os, S, P, and O atoms were refined anisotropically, since several of the carbon atoms gave positive definite ellipsoids if released from isotropic modeling. The hydride ligand could not be located directly but the disposition of the CO ligands suggest strongly that it bridges the Os(1)–Os(2) edge. Scattering factors were taken from International Tables for X-ray

Table 1
Crystal and structural refinement data for [Os$_3$(CO)$_8$(μ-H)(μ-SPh)(μ-dppm)] (**2**) and [Os$_3$(CO)$_7$(μ-H)(μ-SPh)(μ$_3$-SC$_6$H$_4$)(μ-dppm)] (**3**).

Empirical formula	C$_{39}$H$_{28}$O$_8$Os$_3$P$_2$S	C$_{44}$H$_{32}$O$_7$Os$_3$P$_2$S
Formula weight	1289.21	1368.35
T (K)	150(2)	89(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
Space group	$P-1$	$P2_1/n$
a (Å)	11.5072(8)	10.9678(2)
b (Å)	12.3475(8)	28.0191(2)
c (Å)	15.1330(10)	18.5013(3)
α (°)	112.9460(10)	90
β (°)	91.0800(10)	98.894(1)
γ (°)	106.5390(10)	90
V (Å^3)	1877.5(2)	5105.07(14)
Z	2	4
D_{calc} (Mg/m^3)	2.281	1.780[a]
Absorption coefficient (mm^{-1})	10.320	7.735[a]
$F(000)$	1200	2564[a]
Crystal size (mm^3)	0.22 × 0.16 × 0.04	0.22 × 0.10 × 0.05
θ range for data collection (°)	2.77–28.30	1.33–25.72
Index ranges	$-15 \leqslant h \geqslant 15$	$-12 \leqslant h \geqslant 12$
	$-16 \leqslant k \geqslant 16$	$-33 \leqslant k \geqslant 33$
	$-20 \leqslant l \geqslant 20$	$-16 \leqslant l \geqslant 22$
Reflections collected	16053	28287
Independent reflections	8500 (0.0295)	9664 (0.1269)
Completeness to $\theta = 67.10°$	96.7	99.3
Maximum and minimum transmission (R_{int})	0.6830 and 0.2098	0.7014 and 0.2844
Refinement method (%)	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	8500/0/478	6267/0/516
Goodness-of-fit on F^2	0.933	1.004
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0383$, $wR_2 = 0.1333$	$R_1 = 0.0743$, $wR_2 = 0.1528$
R indices (all data)	$R_1 = 0.0479$, $wR_2 = 0.1711$	$R_1 = 0.1343$, $wR_2 = 0.1700$
Largest difference in peak and hole (e Å^{-3})	2.3471and −2.284	2.540 and −2.132

[a] Not including the disordered solvent that was excluded from the refinement using the SQUEEZE method (see Ref. [25]).

Crystallography [26]. All pertinent crystal data and other experimental conditions and refinement details are summarized in Table 1.

3. Results and discussion

Reaction of [Os$_3$(CO)$_8$(μ-H)(μ$_3$-Ph$_2$PCH$_2$P(Ph)C$_6$H$_4$)] (**1**) with Ph$_3$GeSPh in refluxing benzene for 9 h resulted in the formation

of two triosmium compounds, [Os$_3$(CO)$_8$(μ-H)(μ-SPh)(μ-dppm)] (**2**) and [Os$_3$(CO)$_7$(μ-H)(μ-SPh)(μ$_3$-SC$_6$H$_4$)(μ-dppm)] (**3**) in 40% and 17% yields, respectively (Scheme 2). Compound **2** was previously reported from the reactions of thiophenol with [Os$_3$(CO)$_9$(NCMe)(μ-dppm)] [27] and **1** [10] at ambient temperature and with [Os$_3$(CO)$_{10}$(μ-dppm)] [28] at 110 °C and was characterized spectroscopically. Compounds **2** and **3** do not contain any Ph$_3$Ge-derived ligand. Compound **2** does not seem to be a precursor of **3** as attempts to obtain **3** by treating **2** with thiophenol in refluxing benzene for 24 h or in refluxing toluene for 8 h have failed.

The solid-state molecular structure of **2** is shown in Fig. 1, crystallographic data are collected in Table 1, and selected bond lengths and angles are listed in the caption. The structure is based upon a triangular arrangement of three osmium atoms coordinated by eight carbonyl ligands, a bridging bis(diphenylphosphino)methane (dppm) ligand, a bridging phenylsulfido ligand and a bridging hydride. The three osmium atoms define an approximate isosceles triangle of osmium atoms with one long [Os(1)–Os(2) 2.8877(5) Å] and two comparatively short bonds [Os(1)–Os(3) 2.8549(5) Å and Os(2)–Os(3) 2.8588(5) Å]. The eight carbonyl ligands are all terminal, three of which are bonded to each of Os(1) and Os(3) while the other two bond to Os(2). The phenylsulfido ligand spans symmetrically across the Os(1)–Os(2) edge [Os(1)–S(1) 2.412(2) Å and Os(2)–S(1) 2.414(2) Å] with the phenyl ring perpendicularly oriented with respect to the trimetallic plane. The hydride ligand was located crystallographically but not refined and also spans the Os(1)–Os(2) edge but lies on the opposite side of the Os$_3$ plane relative to the phenylsulfido ligand. This observation is consistent with the lengthening of the Os(1)–Os(2) edge and the opening out of the carbonyl ligands along this edge [C(1)–Os(1)–Os(2) 116.8(3)°, C(4)–Os(2)–Os(1) 115.1(3)°, and C(5)–Os(2)–Os(1)
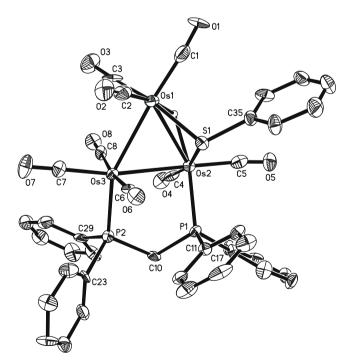

Fig. 1. Molecular structure of [Os$_3$(CO)$_8$(μ-H)(μ-SPh)(μ-dppm)] (**2**) with thermal ellipsoids drawn at the 50% probability level. Ring hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (°): Os(2)–Os(3) 2.8588(5), Os(1)–Os(3) 2.8549(5), Os(1)–Os(2) 2.8877(5), Os(1)–S(1) 2.412(2), Os(2)–S(1) 2.414(2), Os(2)–P(1) 2.317(2), Os(1)–P(2) 2.333(2), Os(3)–Os(1)–Os(2) 59.709(12), Os(1)–Os(3)–Os(2) 83.4(3), Os(3)–Os(2)–Os(1) 59.575(12), S(1)–Os(1)–Os(2) 53.28(5), S(1)–Os(1)–Os(3) 81.16(5), C(1)–Os(1)–Os(2) 116.8(3), C(4)–Os(2)–Os(1) 115.1(3), C(5)–Os(2)–Os(1) 119.7(3), Os(1)–S(1)–Os(2) 73.50(6), P(1)–C(10)–P(2) 113.6(4).

Scheme 2.

119.7(3)°]. The dppm ligand bridges the Os(2)–Os(3) edge slightly asymmetrically [Os(2)–P(1) 2.317(2) Å and Os(3)–P(2) 2.333(2) Å] and the phosphorus atoms of the ligand occupy equatorial sites on both osmium atoms as expected. The Os–S and Os–P bond distances in **2** are similar to those observed in related complexes [10–12] and the overall structural features are similar to the corresponding ruthenium compound [Ru$_3$(CO)$_8$(μ-H)(μ-SPh)(μ-dppm)] [29] and the ethanethiol analog [Os$_3$(CO)$_8$(μ-H)(μ-SC$_2$H$_5$)(μ-dppm)] [10]. The spectroscopic data of **2** are consistent with the solid-state structure and are very similar to those reported previously [10,28].

The solid-state molecular structure of **3** is depicted in Fig. 2, crystallographic data are collected in Table 1, and selected bond lengths and angles are listed in the caption. The molecule consists of an open triangle of three osmium atoms with two distinctly different metal–metal bonds [Os(1)–Os(2) 2.9817(10) Å and Os(2)–Os(3) 2.9160(10) Å], seven carbonyl ligands, a bridging phenylsulfido ligand, a bridging dppm ligand, a triply bridging cyclometallated SC$_6$H$_4$ ligand, and a bridging hydride. The carbonyl ligands are terminally bonded, two are bonded to each of Os(1) and Os(2) and the other three to Os(3). An interesting structural feature of **3** is the *ortho* C–H bond activation of the phenyl ring of one phenylsulfido ligand thus forming a μ$_3$-SC$_6$H$_4$ ligand on the cluster surface, which is spatially oriented such that the plane constituted by the atoms of this ligand is at 73.6° to the Os$_3$ plane. Surprisingly, there is only one previous example of a structurally characterized orthometallated arylthiolate ligand on a cluster, that reported by Adams et al. [30], in which a μ$_3$-SC$_6$H$_3$Me-*p* ligand lies across the face of a closed Os$_3$ triangle. This is in contrast to the many analogous examples of μ$_3$-PC$_6$H$_3$R cyclometallated ligands derived from phosphines. The covalent Os–C distance in **3** [Os(3)–C(12) 2.143(17) Å] is quite similar to those observed in related complexes [10,12,27]. The phenylsulfido ligand which lies on the opposite side to the μ$_3$-SC$_6$H$_4$ ligand asymmetrically bridges the open Os(1)···Os(3) edge [Os(1)–S(2) 2.424(4) Å and Os(3)–S(2) 2.459(5) Å] through the sulfur atom while the μ$_3$-SC$_6$H$_4$ ligand
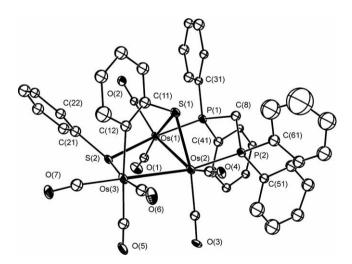
similarly spans across the Os(1)–Os(2) bonding edge [Os(1)–S(1) 2.446(4) Å and Os(2)–S(1) 2.404(4) Å]. The hydride ligand was not located in the structural analysis but clearly bridges the Os(1)–Os(2) edge, *cis* to the Os(1)–C(1) and Os(1)–C(3) bonds, from the lengthening of this edge and widening of the C(1)–Os(1)–Os(2) and C(3)–Os(2)–Os(1) angles [121.6(5)° and 115.6(5)°]. The dppm ligand also bridges the same Os–Os edge and the Os–P bond distances are within the range reported for related compounds [7,10–12,27,28]. The Os–S bond distances in **3** are also in the range found in the literature [10–12]. The C–C bond lengths within the phenyl ring of the μ$_3$-SC$_6$H$_4$ ligand do not vary significantly, suggesting unperturbed benzenoid character in the ring. All other features of the molecule are within the expected range and the molecule is a 50-electron cluster with two formal metal–metal bonds assuming the phenylsulfido and μ$_3$-SC$_6$H$_4$ ligands serve as three and four electron donors, respectively.

The spectroscopic data of **3** support the solid-state structure. The infrared spectrum exhibits absorption bands characteristic for terminally bonded carbonyl ligands. In addition to the usual aromatic resonances for the phenyl protons, the ^{1}H NMR spectrum shows a virtual triplet at δ −15.66 (J = 7.2 Hz) for the bridging hydride ligand while the ^{31}P–{^{1}H} NMR spectrum displays two equal intensity doublets at δ 17.2 (J = 94.8 Hz) and 21.3 (J = 94.8 Hz) due to the phosphorus atoms of the dppm ligand. The FAB mass spectrum shows the parent molecular ion peak at m/z 1370 together with fragmentation peaks due to the sequential loss of all seven carbonyl groups which are also consistent with the solid-state structure.

In summary, the thiolato compounds [Os$_3$(CO)$_8$(μ-H)(μ-SPh)(μ-dppm)] (**2**) and [Os$_3$(CO)$_7$(μ-H)(μ-SPh)(μ$_3$-SC$_6$H$_4$)(μ-dppm)] (**3**) have been synthesized and structurally characterized. Compound **3** provides an interesting unique example of multiple additions of thiolate ligands to a metal carbonyl cluster complex. The formation of an orthometallated thiolate ligand is also rare.

Acknowledgement

Appendix A. Supplementary material

CCDC 705028 and 704328 contain the supplementary crystallographic data for **1** and **2**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2008.12.005.

Fig. 2. Molecular structure of [Os$_3$(CO)$_7$(μ-H)(μ-SPh)(μ$_3$-SC$_6$H$_4$)(μ-dppm)] (**3**) with thermal ellipsoids drawn at the 30% probability level. Ring hydrogen atoms are omitted for clarity. Ring hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (°): Os(1)–Os(2) 2.9817(10), Os(2)–Os(3) 2.9160(10), Os(1)–S(2) 2.424(4), Os(1)–S(1) 2.446(4), Os(3)–S(2) 2.459(5), Os(3)–C(12) 2.143(17), Os(1)–P(1) 2.349(5), Os(2)–P(2) 2.387 (5), Os(3)–Os(2)–Os(1) 83.70(3), S(2)–Os(1)–Os(2) 82.44(11), S(2)–Os(3)–Os(2) 83.27(10), S(1)–Os(2)–Os(1) 52.71(11), S(1)–Os(1)–Os(2) 51.44(10), S(2)–Os(1)–S(1) 92.25(15), C(12)–Os(3)–Os(2) 87.6(5), C(12)–Os(3)–S(2) 91.2(5), C(11)–C(12)–Os(3) 123.0(13), S(2)–Os(1)–Os(3) 75.85(13), Os(1)–S(2)–Os(3) 107.33(17), C(21)–S(2)–Os(1) 109.0(6), C(21)–S(2)–Os(3) 117.0(6), C(11)–S(1)–Os(1) 116.5(6), C(11)–S(1)–Os(2) 109.5(6), C(1)–Os(1)–Os(2) 121.6(5), C(3)–Os(2)–Os(1) 115.6(5), P(1)–C(8)–P(2) 112.7(9).

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