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Dinuclear rhodium–rhodium and rhodium–palladium complexes with different bridging ligands. Crystal structure of $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2]$

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Abstract

Homo- and hetero-dinuclear complexes of general formulae $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(diolefin)_2]$ (diolefin = 1,5-cyclooctadiene (COD) (1), tetrafluorobenzobarrelene (TFB) (2)) and $[RhPd(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(diolefin)(\eta^3\text{-}C_3H_5)]$ (diolefin = COD (4), 2,5-norbornadiene (NBD) (5)), containing one chloride anion and one thiophosphinito group as bridging ligands, have been synthesised by redistribution reactions starting from the corresponding homo-bridged compounds $[Rh_2(\mu\text{-}SPPh_2)_2(diolefin)_2]$, $[Rh_2(\mu\text{-}Cl)_2(diolefin)_2]$ or $[Pd_2(\mu\text{-}Cl)_2(\eta^3\text{-}C_3H_5)_2]$. The structural X-ray analysis carried out for one member of this series, $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2]$, has shown the bridging *P,S*-coordination of the thiophosphinito ligand. The intermetallic separation, 3.291(2) Å, excludes any significant direct metal–metal interaction.

Keywords: Rh; Pd; Sulphides; Dinuclear complexes; Thiophosphinito; X-ray structures; Heteronuclear complexes

1. Introduction

Considerable interest has recently been shown in the coordination properties of hybrid Group 15 and 16 donor ligands [1,2], mostly in connection with the main-fold reactivity of these groups. Among these ambivalent ligands, deprotonated secondary phosphino sulphides (R_2PS^-) constitute an interesting research area. They may coordinate end-on through phosphorus [3] or sulphur atom [4], side-on [5], or may act as a bridge between two metal centres via phosphorus and sulphur [6,7].

In a previous paper from our laboratory [8] we reported the preparation and properties of several thiophosphinito-bridged rhodium(I) complexes of the type $[Rh_2(\mu\text{-}SPPh_2)_2(diolefin)_2]$ prepared by reaction of [Rh(acac)(diolefin)] and $Ph_2P(S)H$ in 1:1 molar ratio. In this type of complex two Ph_2PS^- ligands bridge the two rhodium atoms through their S and P donor atoms.

We report now the preparation of homo- and hetero-dinuclear complexes containing one chloride and one diphenylthiophosphinito as bridging groups. The X-ray structure of the dinuclear complex $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2]$ has also been determined.

2. Results and discussion

Following our studies on the reactivity of thiophoshinito-bridged rhodium complexes, we have observed that monothiophosphinito-bridged derivatives of the type $[RhM(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(L_2)(L'_2)]$ can be prepared by a redistribution reaction, at room temperature, of equimolecular quantities of $[Rh_2(\mu\text{-}SPPh_2)_2(L_2)_2]$ and $[M_2(\mu\text{-}Cl)_2(L'_2)_2]$ (Eq. (1)).

$$[Rh_2(\mu\text{-}SPPh_2)_2(L_2)_2] + [M_2(\mu\text{-}Cl)_2(L'_2)_2] \rightarrow 2[RhM(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(L_2)(L'_2)] \quad (1)$$

M = Rh, $L_2 = L'_2$ = COD(1), TFB(2);

L_2 = COD, L'_2 = TFB(3);

M = Pd, $L'_2 = \eta^3\text{-}C_3H_5$; L_2 = COD(4),

L_2 = NBD(5)

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Table 1
Analytical and spectroscopical data for complexes 1–5

Complex	Anal. Found (Calc.) (%)		Mol. wt. ($CHCl_3$) Found (Calc.)	Yield (%)	ν(PS) (cm^{-1})	^{31}P NMR (J_{RhP} (Hz))
	C	H				
1	49.44 (49.83)	5.33 (5.07)	626 (675)	64	555	71.1 (142)
2	47.63 (47.47)	2.84 (2.43)	862 (911)	64	530	69.1 (139)
3	48.31 (48.47)	3.26 (3.55)	772 (792)	60	535	68.6 (137)
4	45.30 (45.19)	4.53 (4.84)	616 (611)	70	550	63.8
5	44.58 (44.39)	4.12 (3.89)	517 (594)	75	540	63.5

Complexes 1–5 are crystalline and air sensitive materials. Their molecular weights in solution (Table 1) correspond to dinuclear species. The P–S stretching vibrations are observed in the region around 540 cm^{-1}. The lower P–S bond order in these complexes, compared with the uncoordinated diphenylphosphine sulphide (ν(P–S) = 640 cm^{-1}) [9], gives rise to a pronounced shift to lower wave numbers ($\Delta\nu$(P–S) = 100–110 cm^{-1}) (see Table 1).

$^{31}P\{^1H\}$ NMR data for the complexes discussed herein are summarised in Table 1. In the homo-bimetallic complexes 1–3 the bridging μ-$SPPh_2$ ligand appears as a doublet exhibiting coupling to one rhodium nucleus. The $^{31}P\{^1H\}$ NMR spectra of the hetero-metallic complexes 4 and 5 show one singlet at δ 63.8 and δ 63.5 respectively, suggesting that the coordination to the palladium centre is accomplished through the phosphorus atom of the thiophosphinito ligand.

The redistribution reactions were completed in a few hours (1–4 h), as indicated by the $^{31}P\{^1H\}$ NMR spectra of the final solutions, which show only the resonance of the corresponding final compounds [RhM(μ-$SPPh_2$)(μ-Cl)($L_2(L'_2)$]. Thus, the spectra of the reaction mixture leading to 1 in $(CD_3)_2CO$ was periodically run over a period of 4 h, at room temperature, and showed initially the doublet corresponding to the starting material [$Rh_2(\mu$-$SPPh_2)_2(COD)_2$] (δ = 49.3, J_{RhP} = 147 Hz), disappearing while two new doublets at

Table 2
Selected bond distances (Å) and angles (°) for the complex [$Rh_2(\mu$-$SPPh_2)(\mu$-Cl)$(COD)_2$] (1) *

Bond distances			
Rh(1)···Rh(2)	3.291(2)		
Rh(1)–Cl	2.407(2)	Rh(2)–Cl	2.421(2)
Rh(1)–P	2.284(2)	Rh(2)–S	2.370(2)
Rh(1)–C(1)	2.108(7)	Rh(2)–C(11)	2.103(7)
Rh(1)–C(2)	2.110(7)	Rh(2)–C(12)	2.107(7)
Rh(1)–C(5)	2.209(8)	Rh(2)–C(15)	2.124(7)
Rh(1)–C(6)	2.215(7)	Rh(2)–C(16)	2.157(7)
P–C(21)	1.811(7)	P–C(31)	1.826(5)
P–S	2.047(2)		
C(1)–C(2)	1.388(10)	C(11)–C(12)	1.395(11)
C(1)–C(8)	1.519(12)	C(11)–C(18)	1.505(12)
C(2)–C(3)	1.511(11)	C(12)–C(13)	1.536(11)
C(3)–C(4)	1.439(12)	C(13)–C(14)	1.534(11)
C(4)–C(5)	1.504(12)	C(14)–C(15)	1.507(11)
C(5)–C(6)	1.348(12)	C(15)–C(16)	1.379(12)
C(6)–C(7)	1.517(12)	C(16)–C(17)	1.522(12)
C(7)–C(8)	1.434(12)	C(17)–C(18)	1.529(12)
Bond angles			
Cl–Rh(1)–P	85.1(1)	Cl–Rh(2)–S	95.3(1)
Cl–Rh(1)–M(1)	178.9(2)	Cl–Rh(2)–M(3)	173.1(2)
Cl–Rh(1)–M(2)	92.5(1)	Cl–Rh(2)–M(4)	89.3(2)
P–Rh(1)–M(1)	95.0(1)	S–Rh(2)–M(3)	88.7(2)
P–Rh(1)–M(2)	177.4(2)	S–Rh(2)–M(4)	170.9(2)
M(1)–Rh(1)–M(2)	87.4(2)	M(3)–Rh(2)–M(4)	87.5(2)
Rh(1)–Cl–Rh(2)	85.9(1)	Rh(2)–S–P	102.8(1)
Rh(1)–P–S	103.0(1)	S–P–C(21)	109.8(2)
Rh(1)–P–C(21)	115.1(2)	S–P–C(31)	106.6(2)
Rh(1)–P–C(31)	120.1(2)		

* M(1), M(2), M(3) and M(4) are the mid-points of the olefinic bonds C(1)–C(2), C(5)–C(6), C(11)–C(12) and C(15)–C(16) respectively.

$\delta = 59.1$ ($J_{RhP} = 139$ Hz) and $\delta = 71.1$ ($J_{RhP} = 142$ Hz) were observed. This last signal, initially of low intensity, corresponds to the final compound $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD_2)]$ (1). After 4 h of reaction, only compound 1 remained in solution.

The signal observed along the reaction at $\delta = 59.1$ seems to be an intermediate that, unfortunately, we could not isolate. Probably it should be a tetranuclear species similar to those observed in related redistribution reactions involving mixed chloro-mercapto-bridged platinum(II) complexes [10]. Furthermore, we have recently reported the formation of tetranuclear intermediates by interaction of two benzothiazole-2-thiolate dirhodium species, where interchange of bridging ligands takes place [11]. As a whole, the redistribution product observed in this work implies a rhodium–phophorus and rhodium–palladium-chloride bond cleavage. The $^{31}P\{^1H\}$ spectra commented on earlier for compounds 4 and 5, where the palladium centre is clearly coordinated to the phosphorus atom of the bridging ligands, support this proposal. Although a number of mechanisms have been proposed for the redistribution reactions of transition metal complexes [11,12], very few examples of bimolecular mechanisms leading to the formation of tetranuclear intermediates have been described [10–12].

The nature and bonding parameters of the bridging ligands were ascertained by an X-ray analysis of complex 1. Fig. 1 shows a representation of the dinuclear molecule, together with the atom labelling used. Selected bond distances and angles are collected in Table 2.

Complex 1 exhibits two 'Rh(COD)' moieties maintained together through a mixed bridging system formed by a chloride anion and a *P,S*-bridging thiophosphinito ligand, $SPPh_2^-$, bonded to Rh(1) via the phosphorus and through the sulphur atom to Rh(2). Each metal coordination centre completes a slightly distorted square-planar coordination interacting with a 1,5-cyclooctadiene molecule linked to the metal as a quelate through the two olefinic bonds. If M(1), M(2), M(3) and M(4) represent the mid-points of the C(1)–C(2), C(5)–C(6), C(11)–C(12) and C(15)–C(16) double bonds, the greater deviations from the mean metal coordination planes are observed for M(1), 0.034(5) Å, and for M(4), 0.280(7) Å.

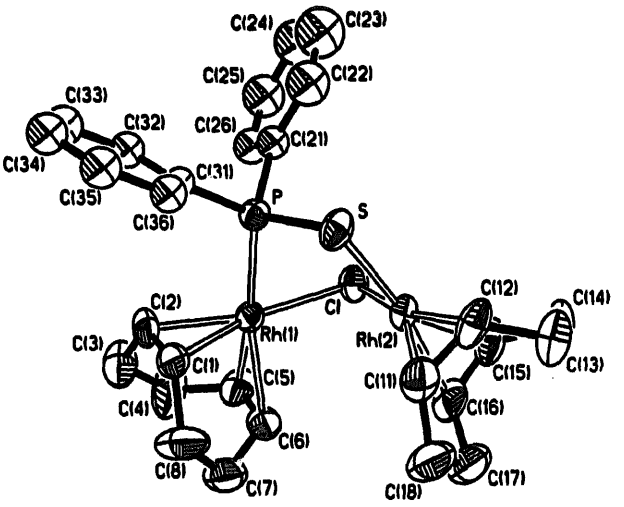

Fig. 1. Molecular representation of $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2]$ (1) showing the numbering scheme.

As a whole, the dinuclear complex resembles the closely related $[Rh_2(\mu\text{-}SPPh_2)_2(COD)_2]$ [8], where one of the bidentate bridging ligands has been substituted by a chloride anion to give the hetero-bridged compound $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2)]$. The asymmetry created in the five-membered metalocycle ring due to the presence of the chloride bridge originates an alternative clear $^1T_5$ twist conformation (Rh(1) up, Cl down) characterised by the Cremer and Pople parameters $Q = 1.239(2)$ Å and $\phi = -17.8(1)°$ [13], significatively different from the symmetric boat conformation observed in the thiophosphinito homo-bridged complex [8]. Associated with this conformation, the metal coordination planes—around Rh(1) and Rh(2)—adopt an almost perpendicular relative disposition, making a dihedral angle of 84.9(1)°. An additional difference between the homo- and hetero-bridged complexes involves the interligand dihedral angle (each plane formed by the two metals and the bridging atoms) 72.4(1)° in 1, which is markedly lower than that reported for $[Rh_2(\mu\text{-}SPPh_2)_2(COD)_2]$, 99.9(1)°, or for the closely related Pt complex $[Pt_2(\mu\text{-}SPPh_2)_2H_2(P^tBu_3)_2]$, 97.5° [6].

The intermetallic separation determined, 3.291(2) Å, excludes any metal–metal interaction. Related to this parameter, it is interesting to note the exceptional flexibility of the thiophosphinito group when acting as a bridging ligand, allowing intermetallic separations from arrangements where a short metal–metal bond is present (2.600(1) Å in $[Pd_2(\mu\text{-}SPPh_2)_2(CNMe)_2]$ [14] or 2.628(1) Å in $[Pt_2(\mu\text{-}SPEt_2)_2\{P(OPh)_3\}_2]$ [15]), to cases with a metal–metal distance as long as 4.351(1) Å, like in $[(COD)Rh(\mu\text{-}SPPh_2)_2Pt(S_2CNEt_2)]$ [16], for instance. In all these complexes the phosphorus atom maintains a tetrahedral environment, with a reasonably fixed S–P–M angle ranging from 105.7 to 122.9°, while the sulphur atom accommodates the M–S–P bond angle (range 81.5–115.4°) to the metal–metal separation, being fundamentally responsible for the observed flexibility of this bridging ligand.

The coordination of the $SPPh_2^-$ ligand in 1 is analogous to those determined in the related dimer $[Rh_2(\mu\text{-}SPPh_2)_2(COD)_2]$, showing Rh(1)–P and Rh(2)–S bond distances (2.284(2) and 2.370(2) Å) similar to those described in the homo-bridged complex, mean 2.287(2) and 2.361(2) Å respectively [8]. As expected from the IR data, the P–S bond length 2.047(2) Å is longer than the corresponding P=S double bond length (1.926–

1.966 Å) [17,18], but shorter than that of a P–S single bond, 2.122(1) Å [17], and compares well with the mean value observed in related dinuclear d^8–d^8 complexes containing two bridging thiophosphinito ligands (mean 2.044(8) Å) [19]. These intermediate P–S distances are clearly indicative of the persistence of some π-bond characteristics for this bond after coordination to the metal [6,20].

However, the chloride anion bridges the two metals in a slightly asymmetrical fashion, with Rh–Cl bond distances, 2.407(2) and 2.421(2) Å, within the normal range observed for this type of bond.

Each cyclooctadiene ring exhibits a boat conformation with Rh–C and C–C bond distances within the normal range for Rh(I)–cyclooctadiene complexes [21]. Interestingly, it is appreciable how the Rh–C lengths are sensitive to the *trans* situated atoms, especially those opposite to the phosphorus atom which show particularly long Rh–C distances, 2.209(8) and 2.215(7) Å, and the shortest olefinic C=C bond, with a value (1.348(12) Å) similar to that observed in the free cyclooctadiene molecule, 1.34 Å [22].

3. Experimental

3.1. General procedures

All reactions were carried out anaerobically by conventional Schlenk techniques. Solvents were deoxygenated and distilled immediately before use. C and H analyses were performed with a Perkin-Elmer 240-B microanalyzer. IR spectra were recorded on a Perkin-Elmer 783 spectrophotometer in the range 4000–200 cm^{-1} with the samples as Nujol mulls and calibration with polystyrene. Molecular weights were determined with a Knauer vapour pressure osmometer. $^{31}P\{^1H\}$ NMR spectra were carried out in $CDCl_3$ solutions, at room temperature, on a Varian XL 200 spectrometer.

Starting materials were prepared by literature procedures as follows: $[Rh_2(\mu\text{-}SPPh_2)_2(diolefin)_2]$ (diolefin = COD, NBD, TFB) [8], $[Rh_2(\mu\text{-}Cl)_2(COD)_2]$ [23], $[Rh_2(\mu\text{-}Cl)_2(TFB)_2]$ [24] and $[Pd_2(\mu\text{-}Cl)_2(\eta^3\text{-}C_3H_5)_2]$ [25].

3.2. Preparation of $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2]$ (1)

To a suspension of $[Rh_2(\mu\text{-}SPPh_2)_2(COD)_2]$ (100 mg, 0.116 mmol) in acetone (20 ml), solid $[Rh_2(\mu\text{-}Cl)_2(COD)_2]$ (57.5 mg, 0.116 mmol) was added. The starting materials dissolved and when the reaction was completed the yellow product $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2]$ precipitated spontaneously from the solution. The resulting suspension was stirred for 4 h and then the solid was isolated by filtration, washed with diethyl ether and vacuum dried (101 mg, 64%).

3.3. Preparation of $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(TFB)_2]$ (2)

Solid $[Rh_2(\mu\text{-}Cl)_2(TFB)_2]$ (66.7 mg, 0.091 mmol) was added to a suspension of $[Rh_2(\mu\text{-}SPPh_2)_2(TFB)_2]$ (100 mg, 0.091 mmol) in acetone (30 ml), giving a red solution. The reaction was continued for 2 h to afford a red suspension of compound 2. The solid was separated by filtration, washed with diethyl ether and vacuum dried (106 mg, 64%).

3.4. Preparation of $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)(TFB)]$ (3)

A suspension of $[Rh_2(\mu\text{-}SPPh_2)_2(COD)_2]$ (100 mg, 0.116 mmol) in acetone (20 ml) was treated with $[Rh_2(\mu\text{-}Cl)_2(TFB)_2]$ (85 mg, 0.116 mmol). The yellow starting materials dissolved immediately and the colour of the solution changed to red. After 2 h of stirring the solvent was evaporated under vacuum to ca. 1 ml and hexane (20 ml) was added. The red solid obtained was filtered off, washed with hexane and vacuum dried (107 mg, 60%).

Compound 3 can alternatively be prepared starting from $[Rh_2(\mu\text{-}SPPh_2)_2(TFB)_2]$ and $[Rh_2(\mu\text{-}Cl)_2(COD)_2]$.

3.5. Preparation of $[RhPd(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(diolefin)(\eta^3\text{-}C_3H_5)]$ (diolefin = 4 COD, 5 NBD)

To a suspension of $[Rh_2(\mu\text{-}SPPh_2)_2(diolefin)_2]$(diolefin = COD, NBD) (0.116 mmol) in acetone (20 ml), solid $[Pd_2(\mu\text{-}Cl)_2(\eta^3\text{-}C_3H_5)_2]$ (42.7 mg, 0.116 mmol) was added. The starting materials dissolved and the resulting yellow solution was stirred for 1 h. The solution was concentrated to ca. 2 ml and addition of hexane (20 ml) led to the precipitation of a yellow solid which was separated by filtration, washed with hexane and vacuum dried. Yield: 4 (100 mg, 70%), 5 (90 mg, 75%).

3.6. Reaction of $[Rh_2(\mu\text{-}SPPh_2)_2(COD)_2]$ with $[Rh_2(\mu\text{-}Cl)_2(COD)_2]$

A sample of $[Rh_2(\mu\text{-}SPPh_2)_2(COD)_2]$, for $^{31}P\{^1H\}$ NMR monitoring, was prepared by dissolving 40 mg (0.046 mmol) in $(CD_3)CO$ (3 ml). The $^{31}P\{^1H\}$ NMR spectrum showed one doublet at $\delta = 49.3$. A stoichiometric amount of $[Rh_2(\mu\text{-}Cl)_2(COD)_2]$ (23 mg, 0.046 mmol) was added. After 1 h, at 20°C, the $^{31}P\{^1H\}$ spectrum was again recorded. The spectrum showed two doublets, one at $\delta = 71.1$ ppm due to $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD_2)]$ and one at $\delta = 59.1$ ppm due to a reaction intermediate (relative ratio 2:1). After 2 h, at 20°C, the $^{31}P\{^1H\}$ spectrum showed only the resonance of the redistribution compound $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2]$.

3.7. X-ray crystal study of $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2]$ (1)

3.7.1. Crystal data

$C_{28}H_{34}ClPRh_2S$; $M = 674.9$; monoclinic, space group $P2_1/c$; $a = 11.1526(6)$, $b = 14.1751(13)$, $c = 17.2884(8)$ Å, $\beta = 97.26(1)°$; $V = 2711.2(3)$ Å^3; $Z = 4$; $D_c = 1.653$ mg m^{-3}; $F(000) = 1360$; λ(Mo Kα) = 0.71069 Å; $\mu = 1.446$ mm^{-1}; $T = 298$ K.

3.7.2. Data collection and processing

A Siemens AED-2 diffractometer with monochromated Mo Kα radiation was used. A yellow prismatic block $0.152 \times 0.445 \times 0.224$ mm^3 was mounted on a glass fibre. 6381 intensities were registered to $2\theta_{max} = 42°$ (ω–2θ scan technique). Averaging equivalents gave 2852 unique reflections, of which 2448 with $F \geq 4.0\sigma(F)$ were used for all calculations (program system SHELX-76) [26]. Cell constants were refined from setting angles of 60 reflections in the 2θ range 20–30°. Three standard reflections were monitored every hour as a check on crystal and instrument stability. Data were corrected for Lorentz and polarization effects. A numerical absorption correction was also applied [27] (transmission factors 0.728, 0.821).

3.7.3. Structure solution and refinement

The structure was solved by Patterson and extended by difference syntheses. Atoms were refined isotropically first, and in subsequent cycles with anisotropic thermal parameters for all the non-hydrogen atoms (excepting those of phenyl groups). Hydrogen atoms were found in difference maps and included in the last cycles of refinement using a riding model with a common thermal parameter. The final R value was 0.0282, with $R_w = 0.0301$. The weighting scheme was $w = k/\sigma^2(F) + gF^2$, with $k = 2.7245$ and $g = 0.00017$; 241 parameters; maximum $\Delta/\sigma < 0.001$; maximum $\Delta\rho = 0.55$ eÅ^{-3}, close to C(7). Atomic scattering factors, corrected for anomalous dispersion, were taken from Ref. [28]. Final atomic coordinates are given in Table 3.

Table 3
Fractional atomic coordinates ($\times 10^4$) for the non-hydrogen atoms of complex $[Rh_2(\mu\text{-}SPPh_2)(\mu\text{-}Cl)(COD)_2]$ (1)

Atom	x	y	z
Rh(1)	6460(1)	336(1)	2109(1)
Rh(2)	7807(1)	−1133(1)	3415(1)
Cl	7410(2)	546(1)	3427(1)
S	5751(2)	−1583(1)	3134(1)
P	4946(1)	−378(1)	2667(1)
C(1)	5915(6)	−259(5)	1003(4)
C(2)	5407(6)	632(5)	1035(4)
C(3)	5826(7)	1509(6)	651(5)
C(4)	6948(8)	1917(7)	998(6)
C(5)	7648(6)	1407(6)	1673(5)
C(6)	8170(6)	558(6)	1607(4)
C(7)	8122(7)	−4(7)	857(5)
C(8)	7010(7)	−480(7)	594(6)
C(11)	8329(7)	−2400(5)	2920(4)
C(12)	8203(7)	−2553(5)	3703(5)
C(13)	9265(8)	−2675(6)	4353(5)
C(14)	9681(8)	−1732(5)	4731(5)
C(15)	9421(6)	−907(6)	4186(5)
C(16)	9708(6)	−837(6)	3435(5)
C(17)	10383(7)	−1616(6)	3065(5)
C(18)	9502(7)	−2282(7)	2585(5)
C(21)	357(6)	300(4)	3423(4)
C(22)	3870(8)	−141(6)	4028(5)
C(23)	3438(10)	447(7)	4610(7)
C(24)	3471(9)	1391(7)	4542(6)
C(25)	3891(8)	1811(7)	3946(5)
C(26)	4346(6)	1263(5)	3380(4)
C(31)	3562(5)	−744(4)	2063(3)
C(32)	2667(6)	−65(5)	1848(4)
C(33)	1623(7)	−297(5)	1374(4)
C(34)	1455(7)	−1198(5)	1081(4)
C(35)	2323(6)	−1871(5)	1280(4)
C(36)	3382(6)	−1644(5)	1768(4)

4. Supplementary material available

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates, thermal parameters, and remaining bond lengths and angles.

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