THE PREPARATION AND CRYSTAL AND MOLECULAR STRUCTURE OF (SELENITO-0,0')BIS(TRIPHENYLPHOSPHINE)PLATINUM(II)-BENZENE(1/1)*

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Summary

The crystal structure of the compound (selenito-O, O')bis(triphenylphosphine)platinum(II)-benzene(1/1) has been determined by a singlecrystal X-ray analysis. The compound crystallizes in the triclinic space group $P\bar{1}$ with a = 10.651(2) Å, b = 12.812(2) Å, c = 15.473(2) Å; $\alpha =$ $85.76(1)^{\circ}$, $\beta = 73.39(1)^{\circ}$, $\gamma = 68.52(1)^{\circ}$ and z = 2. The structure has been refined to an R value of 0.036 using 4795 observed $I > 3\sigma(I)$ intensities measured on an automatic diffractometer. The platinum has a squareplanar coordination geometry with the selenito-ligand bonded in a bidentate fashion through the oxygen atoms with Pt-O = 2.049(5) Å and 2.052(5) Å. Coordination of the pyramidal SeO₃²⁻ ion leads to a divergence of the Se-O bond lengths with the bonds to the coordinated oxygen atoms taking values of 1.726(5) Å and 1.746(5) Å and the bond to the uncoordinated oxygen taking the value of 1.602(7) Å.

The synthesis of this compound from the zero-valent compounds $Pt(PPh_3)_3$ or $Pt(PPh_3)_2(C_2H_4)$ and SeO_2 is also discussed.

1. Introduction

The recent interest shown in the coordination chemistry of sulphur dioxide, particularly with the platinum metals [1], prompted us to investigate the possibility that SeO_2 might form related complexes. Although a literature search revealed that no SeO_2 complexes of the transition metals had previously been prepared, we were encouraged by a recent report that SeO_2 inserts into transition metal carbon σ bonds in a manner reminiscent of SO_2 to form SeO_2Me complexes [2]. Unlike SO_2 , SeO_2 is a polymeric infinite solid, and consequently has only a low solubility in non-polar organic solvents. This makes it less convenient as a starting material for the formation of SeO_2 complexes. Furthermore, commercially available SeO_2

^{*}Dedicated to Professor J. D. Corbett on the occasion of his 60th birthday.

is extensively hydrated and requires sublimation before use [3]. This paper describes some attempts to prepare SeO_2 complexes of the platinum metals, and a single-crystal X-ray analysis which has shown that the product resulting from the addition of SeO_2 to zero-valent platinum metal complexes is actually an O, O'-selenito-complex rather than a SeO_2 complex.

2. Results and discussion

When a freshly sublimed sample of SeO₂ was stirred in suspension with a solution of $Pt(PPh_3)_3$ in dry benzene under nitrogen for 3 h a pale yellow crystalline solid separated from the solution, and on the basis of carbon, hydrogen and selenium analyses was formulated as $Pt(SeO_2)_{1.5}(PPh_3)_2$. Recrystallization of the sample from dichloromethane, or the preparation of the compound from $Pt(C_2H_4)(PPh_3)_2$ and SeO_2 resulted in a compound with a similar appearance, but which on the basis of carbon, hydrogen and selenium analyses was formulated as $Pt(SeO_2)(PPh_3)_2$. The IR spectra of the two samples described above proved to be almost identical. In addition to those bands characteristic of triphenylphosphine they showed a strong band at 900 cm⁻¹ and a much weaker band at 885 cm⁻¹ which were assigned to the coordinated SeO₂ moiety in view of the fact that the SeO₂ molecule in the gas phase has ν_s and ν_{asym} at 920 cm⁻¹ and 967 cm⁻¹ respectively [4]. The $\{{}^{31}P-{}^{1}H\}$ nuclear magnetic resonance (NMR) spectrum of the complexes before and after recrystallization were identical and showed a multiplet at +4.58 ppm relative to trimethylphosphate characteristic of two equivalent and *cis*-phosphine ligands coordinated to platinum with ${}^{1}J(Pt-P) = 3670$ Hz [5].

From this evidence it was concluded [1, 6] that the two species described above were essentially identical and best formulated as $Pt(SeO_2)$ -(PPh_3)₂·0.5SeO₂ and $Pt(SeO_2)(PPh_3)_2$, *i.e.* with and without SeO₂ of crystallization, and the following structures were proposed:



Under higher resolution the ³¹P{¹H} NMR spectrum showed fine structure which could be attributed to the presence of ⁷⁷Se $(I = \frac{1}{2}, 7.5\%)$ relative abundance) in the compound. The four isotopomeric species which arise from the distribution of the ³¹P, ¹⁹⁵Pt and ⁷⁷Se magnetically active nuclei are shown below for the O, O'-isomer. (See opposite page.)

The satellites in the ${}^{31}P{}^{1}H$ spectrum of the compound shown in Fig. 1(a) can be attributed to isotopomer (III) with $J({}^{31}P{}^{-77}Se) = 4.5$ Hz; the corresponding splittings in the platinum satellites arising from isotopomer (IV) were not observed, presumably because of the lower relative abundance. The ${}^{77}Se{}^{1}H$ NMR spectrum of the compound shown in Fig. 1(b) consists of a triplet due to ${}^{31}P{}^{-77}Se$ coupling with $J({}^{31}P{}^{-77}Se) = 4.5$ Hz.



where * denotes a nucleus other than phosphorus with nuclear spin $\frac{1}{2}$.



Fig. 1. (a) The $^{31}P\{^1H\}$ NMR spectrum of $Pt(SeO_3)(PPh_3)_2$ and (b) the corresponding $^{77}Se\{^1H\}$ spectrum.

The platinum satellites associated with this spectrum associated with isotopomer (IV) were not located. The low value of $J({}^{31}P-{}^{77}Se)$ led us to favour the O,O' structure shown above in preference to the alternative seleniumbonded structure.

Single crystals of the complex were grown by slowly diffusing dilute solutions of SeO₂ in benzene through a sinter tube into a solution of Pt(PPh₃)₂(C₂H₄) also in benzene. An X-ray analysis of these crystals, details of which are described in the experimental section, showed the complex *actually* to be the O, O'-selenito-complex of platinum(II) shown below.



The coordinated SeO₃ and the associated oxidation of platinum from the (0) to the (II) oxidation state must have originated from the greater oxidizing ability of SeO₂ compared with SO₂. Attempts to locate lower valent selenium species either in the form of Ph₃PSe [7] or elemental selenium proved to be negative and therefore we were forced to conclude that even after sublimation there are traces of water in the SeO₂ leading to the following oxidative reaction:

$$(Ph_3P)_2Pt(C_2H_4) + H_2SeO_3 \longrightarrow Pt(SeO_3)(PPh_3)_2 + H_2$$

Indeed IR spectra of freshly sublimed samples of SeO_2 have shown that they contain traces of H_2SeO_3 [8].

3. Description of the structure

The molecular structure of $Pt(SeO_3)(PPh_3)_2$ is shown in Fig. 2, which also gives the atom numbering scheme used. Important bond lengths and angles are given in Tables 1 and 2. The geometry about the platinum is essentially square planar, with the distortions away from square planarity imposed by the small bite angle of the bidentate selenito- ligand $(74.3(2)^\circ)$ and a concomitant opening up of the angle between the bulky triphenylphosphine ligands $(98.4(1)^\circ)$. The platinum-phosphine and platinum-oxygen bond lengths fall in the range expected for *cis*-platinum(II) triphenylphosphine complexes with oxygen derived ligands [9 - 11].

The coordination chemistry of the sulphito ligand is well documented structurally and examples of both S- and O- bonded monodentate and bidentate complexes have been reported [12, 13]. The structural chemistry



Fig. 2. The coordination geometry around the platinum atom in $Pt(SeO_3)(PPh_3)_2$. For reasons of clarity the phenyl rings on the phosphine ligands have been omitted.

Pt(1) - P(1)	2.246(2)	Pt(1) - P(2)	2.219(2)
Pt(1) - O(1)	2.052(5)	Pt(1) - O(2)	2.049(5)
Se(1) - O(1)	1.726(5)	Se(1) - O(2)	1.746(5)
Se(1) - O(3)	1.602(7)	P(1) - C(111)	1.816(8)
P(1)-C(121)	1.819(8)	P(1) - C(131)	1.813(8)
P(2)-C(211)	1.825(7)	P(2) - C(221)	1.825(8)
P(2) - C(231)	1.816(8)		
Pt(1) - Se(1)	2.806(1)	O(1) - O(2)	2.476(8)
O(1) - O(3)	2.671(9)	O(2) - O(3)	2.682(8)
,			

TABLE 1 Intramolecular interatomic distances (Å)

TABLE 2

Intramolecular bond angles (deg)

P(1) - Pt(1) - P(2)	98.35(7)	O(1) - Pt(1) - O(2)	74.29(21)
O(1) - Pt(1) - P(1)	91.74(16)	O(2) - Pt(1) - P(2)	95.62(15)
O(1)-Se(1)-O(2)	90.97(24)	O(1) - Se(1) - O(3)	106.70(33)
O(2) - Se(1) - O(3)	106.40(34)	Pt(1) - Se(1) - O(3)	125.83(28)
Pt(1) - O(1) - Se(1)	95.56(23)		
Pt(1) - O(2) - Se(1)	95.03(24)		
Pt(1) - P(1) - C(111)	110.97(26)	Pt(1) - P(2) - C(211)	113.38(23)
Pt(1) - P(1) - C(121)	123.21(26)	Pt(1) - P(2) - C(221)	112.78(25)
Pt(1) - P(1) - C(131)	107.27(26)	Pt(1) - P(2) - C(231)	113.47(26)
C(111) - P(1) - C(121)	105.36(36)	C(211) - P(2) - C(221)	102.90(34)
C(111) - P(1) - C(131)	104.60(35)	C(211) - P(2) - C(231)	111.10(34)
C(121) - P(1) - C(131)	103.81(36)	C(221) - P(2) - C(231)	102.17(34)

of the selenito- ligand is much less well established and limited to a recent investigation of $[Co(NH_3)_5(SeO_3)]^+$ [14], which has a monodentate O-bonded $SeO_3^{2^-}$ ligand. Therefore, the present structural study of Pt(SeO₃)-(PPh₃)₂ represents the first example of a bidentate selenito- ligand.

In SeO₃²⁻ salts, e.g. MgSeO₃·6H₂O [15], the selenite anion is pyramidal with O—Se—O equal to 101° and the Se–O bond length is 1.69 Å. In Pt(SeO₃)(PPh₃)₂ the SeO bond lengths to the coordinated oxygen atoms are 1.726 and 1.746 Å, and the third Se–O bond length is 1.602 Å. This divergence in bond lengths is a common characteristic of oxyanions which coordinate to transition metals and can be readily rationalized in terms of the loss of some multiple bond character from those oxygen atoms which coordinate to the metal [16]. In $[Co(NH_3)_5(SeO_3)]^+$ a similar pattern has been noted with Se–O(Co) equal to 1.666 Å and the uncoordinated Se–O bond lengths equal to 1.630 Å.

4. Experimental details

The reactions were performed under anaerobic conditions, and the solvents were dried and purified before use. The selenium dioxide was sublimed before use using the recommended procedure [3].

IR spectra were recorded using a Perkin Elmer 457 Spectrometer as nujol mulls and calibrated using a polystyrene film. The $\{^{31}P^{-1}H\}$ NMR spectra were recorded using a Bruker WH 90 spectrometer with CD_2Cl_2 as the solvent and trimethylphosphate as the standard. Microanalyses were obtained in these laboratories and Butterworths Microanalytical Laboratories.

4.1. Reaction of $Pt(PPh_3)_3$ with SeO_2

Tris(triphenylphosphine)platinum(0) [17] was dissolved in dry, degassed benzene (30 cm³), and freshly sublimed SeO₂ was added with stirring under a nitrogen atmosphere. After stirring for 3 h a pale yellow solid precipitated out which was filtered and washed thoroughly with benzene, the solid was dried *in vacuo* and identified as selenito-O, O'-bis(triphenylphosphine)platinum-hemi(selenium dioxide), melting point, 204 - 205 °C. (Found: C, 47.7%; H, 3.7%; Se, 15.2%. C₃₆H₃₀O₄Se_{1.5}Pt requires: C 48.0%; H, 3.30%; Se 13.0%).

4.2. Preparation of sample of $Pt(SeO_3)(PPh_3)_2$ used in crystallographic analysis

Bis(triphenylphosphine)(ethylene)platinum(0) (0.25 g) was dissolved in dry, degassed benzene under a nitrogen atmosphere and SeO₂ which had been freshly sublimed was introduced into the solution through a fine sintered tube, which allowed a dilute solution of SeO₂ in benzene to diffuse into the solution of the platinum complex. After three days pale yellow crystals of Pt(SeO₃)(PPh₃)₂·C₆H₆ formed which were suitable for the crystallographic analysis described below, yield 0.1 g. (Found: C, 55.8%; H, 3.9%).

5. Crystallographic analysis

Crystals used for the X-ray work were sealed in Lindemann capillaries 0.5 mm in diameter. Unit cell parameters were initially determined from oscillation and Weissenberg photographs and later refined using setting angles for 25 high angle reflections automatically centred on a CAD4 diffractometer. The crystal data and details of the data collection and structure refinement are summarized in Table 3. Intensity data were recorded on the CAD4 diffractometer using graphite monochromated Mo K α radiation and an $\omega - 2\theta$ scan technique, in the manner described previously [18]. During data collection the crystals showed no indications of decomposition. The data were corrected for Lorentz and polarization effects and absorption.

TABLE 3

Crystal data and details of structure analysis

Crystal data	
Formula	$Pt(SeO_3)(PPh_3)_2 \cdot C_6H_6$
М	924.57
Crystal system	Triclinic
a (Å)	10.651(2)
b (Å)	12.812(2)
c (Å)	15.473(2)
α (deg)	85.76(1)
β (deg)	73.39(1)
γ (deg)	68.52(1)
$U(A^{-3})$	1881.7
Space group	$P\bar{1}$
Z	2
$D_{\rm c} ({\rm g}{\rm cm}^{-3})$	1.632
D_0 (by flotation)	1.63(1)
F(000)	908
Linear absorption	
coefficient (cm ⁻¹)	50.88 (Μο Κα)
Crystal size	$0.8 \times 0.3 \times 0.3$ mm
Data collection	
$\theta_{\min}, \theta_{\max}$ (deg)	1.5, 27.5
Scan width parameters A, B	
in width $(A + B \tan \theta)$	1, 0.35
Observed data with	
$F_{\rm o} > 3\sigma(F_{\rm o})$	4795
Refinement	
Number of parameters	231
Weighting scheme	
$\omega = [\Sigma A_r T_r^*(x)]^{-1}$	Three-term Chebyshev polynomial [19]
$x = F_{0}/F_{0}$ (max)	
Final $\overline{R} = \overline{\Sigma} \Delta F / \overline{\Sigma} (F_o)$	0.036
$R' = (\Sigma \omega \Delta F^2 / \Sigma \omega F_0^2)^{1/2}$	0.046

The structure of the compound was solved via Patterson and electron density Fourier syntheses and refined by full matrix least squares techniques, anisotropic temperature factors being assigned to the non-carbon atoms. Hydrogen atoms were not refined, but were positioned in idealized positions (C-H = 1.0 Å) and assigned isotropic temperature factors of 0.05 Å² before each cycle of refinement.

A difference electron density Fourier synthesis also indicated the presence of a disordered benzene molecule of crystallization in a vacant region of the asymmetric unit. Attempts to refine the disordered configuration using multiple sites of occupation were unsuccessful and thus only the dominant configuration was refined with unit occupation factors. This accounts for the rather high temperature factors associated with these atoms (see Table 4).

TABLE	4
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Fractional coordinates (×10⁴) and isotropic temperature factors (Å² × 10⁴) for Pt(SeO₃)-(PPh₃)₂·C₆H₆

Atom	x/a	y/b	z/c	U(iso)
Pt(1)	1854.4(3)	1252.2(2)	2147.7(2)	
Se(1)	915.5(8)	-536.2(7)	2696.4(6)	_
P(1)	3144(2)	1872(2)	2753(1)	
P(2)	1239(2)	2665(1)	1248(1)	-
O(1)	2216(6)	-166(4)	2893(4)	
O(2)	812(6)	363(5)	1793(4)	
O(3)	1659(8)	-1787(5)	2266(5)	_
$C(111)^{a}$	4987(8)	981(6)	2394(5)	445(17)
C(112)	6067(10)	1394(8)	2280(7)	611(22)
C(113)	7459(11)	668(9)	2056(7)	702(26)
C(114)	7769(11)	-478(9)	1959(7)	691(25)
C(115)	6702(11)	-871(9)	2072(7)	684(25)
C(116)	5315(10)	-157(8)	2285(7)	608(22)
C(121)	3120(8)	3304(7)	2651(5)	480(17)
C(122)	2382(11)	4048(9)	3388(7)	700(26)
C(123)	2329(14)	5183(11)	3274(9)	906(34)
C(124)	3037(14)	5489(11)	2484(9)	916(35)
C(125)	3761(12)	4765(9)	1765(8)	743(27)
C(126)	3812(10)	3647(8)	1840(6)	593(22)
C(131)	2614(8)	1705(6)	3961(5)	440(17)
C(132)	3584(10)	1418(8)	4469(6)	580(21)
C(133)	3145(12)	1313(9)	5391(7)	733(27)
C(134)	1745(12)	1524(10)	5813(8)	771(28)
C(135)	756(11)	1844(9)	5327(7)	702(25)
C(136)	1190(10)	1905(8)	4393(6)	588(21)
C(211)	2675(7)	2663(6)	262(5)	385(15)
C(212)	2449(9)	3436(7)	-405(6)	532(20)
C(213)	3580(11)	3399(8)	-1164(7)	651(24)
C(214)	4875(10)	2572(8)	-1251(7)	624(23)
C(215)	5094(9)	1778(7)	-591(6)	562(21)
C(216)	3983(8)	1828(7)	164(5)	476(16)
C(221)	-96(8)	2610(6)	749(5)	416(16)
C(222)	221(9)	1636(7)	220(6)	523(19)
C(223)	-771(10)	1603(8)	-181(6)	596(22)
C(224)	-2034(11)	2468(8)	-77(7)	697(27)
C(225)	-2331(12)	3409(10)	438(8)	769(28)
C(226)	11362(10)	3482(8)	857(6)	564(21)
C(231)	393(8)	4028(6)	1834(5)	451(17)
C(232)	-585(10)	4055(8)	2661(7)	643(23)
C(233)	-1356(13)	5082(10)	3143(8)	843(32)
C(234)	-1133(13)	6050(11)	2785(9)	855(32)
C(235)	-167(13)	6018(10)	1989(8)	808(30)
C(236)	612(9)	5002(7)	1484(6)	561(21)
Disordered benz	ene of crystallize	ation		
C(1)	6430(23)	3038(18)	4057(15)	1420(63)
C(2)	6402(23)	2065(18)	5703(14)	1383(60)
C(3)	5386(23)	3523(18)	4806(15)	1445(65)
C(4)	5430(25)	3077(20)	5617(17)	1606(77)
C(5)	7370(21)	1547(16)	4960(14)	1357(59)
C(6)	7375(24)	2097(19)	4090(15)	1511(69)

^aC(1x1) to C(1x6) refer to phenyl rings attached to P(1) if x = 1, 3 and P(2) if x = 4, 6.

The final cycle of least squares refinement led to an R value of 0.0363. The weighting scheme based on a modified three-term Chebyshev polynomial was used [19] of the form $\omega = [\Sigma A_r T_r^*(x)]^{-1}$ where $x = F_o/F_{o \max}$ to give flat agreement analyses.

Fractional atomic coordinates are given in Table 4. Calculated hydrogen atom positions and anisotropic thermal parameters are given in Appendixes A and B. Lists of observed and calculated structure factors have been deposited with the Editor.

Neutral scattering factors were taken from ref. 20 (platinum) and ref. 21 (oxygen, selenium and CO) and corrected for anomalous dispersion using $\Delta f'$ and $\Delta f''$ values from ref. 20. All the calculations were performed on the Oxford University ICL 1906A computer using the CRYSTALS computer programmes [22].

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Appendix A

Atom	x/a	y/b	z/c
H(112)	5837	2217	2359
H(113)	8299	893	1923
H(114)	8726	-1073	1852
H(115)	6932	-1695	1998
H(116)	4480	-386	2411
H(122)	1905	3801	3978
H(123)	1766	5752	3780
H(124)	3024	6275	2431
H(125)	4256	5020	1187
H(126)	4341	3104	1313
H(132)	4117	1384	4915
H(133)	4019	1305	5517
H(134)	1435	1445	6481
H(135)	-953	2488	5643
H(136)	554	2161	5013
H(212)	2123	3998	102
H(213)	3440	3973	-1636
H(214)	5670	2539	-1796
H(215)	5811	1567	-246
H(216)	5002	1647	101
			(continued

Hydrogen coordinates (×10⁴) ($U(iso) = 0.05 \text{ Å}^2$) for Pt(SeO₃)(PPh₃)₂·C₆H₆

Atom	x/a	y/b	z/c	
H(222)	371	1868	-422	
H(223)	-1523	2351	-188	
H(224)	-2602	2205	-362	
H(225)	-3249	4046	512	
H(226)	-1934	3742	1490	
H(232)	-902	3497	2454	
H(233)	-2059	5119	3740	
H(234)	-1700	6784	3124	
H(235)	-458	6091	1421	
H(236)	1320	5375	1300	
HÌI)	5961	3672	3694	
H(2)	6625	1563	6208	
ม (่3)	5147	4013	4298	
H(4)	4850	3317	6253	
H(5)	7954	825	5172	
H(6)	8131	1738	3530	

Appendix B

Anisotropic temperature factors $(\text{\AA}^2 \times 10^4)^a$

Atom	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Pt(1)	275(2)	315(2)	404(2)	58(1)	-102(1)	-115(1)
$\hat{Se(1)}$	462(4)	456(4)	627(5)	102(4)	-131(4)	-233(4)
P(1)	339(9)	351(9)	459(11)	33(8)	-114(8)	-134(8)
P(2)	297(9)	342(9)	423(10)	61(8)	-87(8)	-100(7)
O(1)	541(33)	465(30)	618(36)	166(26)	-292(29)	-257(26)
O(2)	478(32)	478(31)	698(36)	136(26)	-259(28)	-263(26)
O(3)	869(49)	472(35)	1059(57)	26(35)	-376(44)	-294(26)

^aTake the form $\exp[-2\pi^2(h^2a^{*2}U_{11}\dots 2klb^*c^*U_{23}\dots)].$

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