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TETRAETHYLAMMONIUM TRICHLORO(DIPHENYL SULFOXIDE)PLATINATE(II): A RARE EXAMPLE OF A STRUCTURALLY CHARACTERIZED COMPLEX CONTAINING AN S-COORDINATED DIARYL SULFOXIDE LIGAND

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TETRAETHYLAMMONIUM TRICHLORO(DIPHENYL SULFOXIDE)PLATINATE(II): A RARE EXAMPLE OF A STRUCTURALLY CHARACTERIZED COMPLEX CONTAINING AN S-COORDINATED DIARYL SULFOXIDE LIGAND

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The $(\text{Et}_4\text{N})[\text{Pt}(\text{Ph}_2\text{SO})\text{Cl}_3]$ complex was obtained by the interaction of $[\text{PtCl}_4]^{2-}$ and Ph_2SO in a $\text{MeCN}-\text{H}_2\text{O}$ mixture and also resulted from the bridge-splitting reaction of $(\text{Et}_4\text{N})_2[\text{Pt}_2(\mu\text{-Cl})_2\text{Cl}_4]$ using a stoichiometric amount of diphenyl sulfoxide. The $(\text{Et}_4\text{N})[\text{Pt}(\text{Ph}_2\text{SO})\text{Cl}_3]$ compound crystallizes in a monoclinic $P2_1/n$ space group with $a = 11.525(4)$, $b = 11.078(3)$, $c = 19.106(7)$ Å, $\gamma = 102.02(3)^\circ$, $V = 2386(3)$ Å³, $Z = 4$, $\rho_{\text{calcd}} = 1.76$ g cm⁻³. This complex is a rare example of a structurally characterized compound containing a S-coordinated diaryl sulfoxide ligand.

Key words: Diaryl sulfoxide; diphenyl sulfoxide; Pt(II) sulfoxide complexes; synthesis of Pt(II) sulfoxide complexes; X-ray structure; S-coordinated diphenyl sulfoxide.

INTRODUCTION

The complexes of the $\text{Q}[\text{Pt}(\text{R}_2\text{SO})\text{Cl}_3]$ type are known to be widely used in the Pt chemistry as starting materials for the synthesis of: the cis- and trans- $[\text{Pt}(\text{R}_2\text{SO})\text{LCl}_2]$ ($\text{L} = \text{R}_2\text{SO}^1$; $\text{L} = \text{sp}^3$, sp^2 and sp N-donor ligands of different type²); the bridge-type compound $[\text{Pt}(\text{Me}_2\text{SO})(\mu\text{-Cl})\text{Cl}]_2$ ³ and thioether Pt(IV) derivatives of $[\text{Pt}(\text{R}_2\text{S})\text{Cl}_5]^-$ type.^{4,5} The $\text{Q}[\text{Pt}(\text{R}_2\text{SO})\text{Cl}_3]$ complexes are applied also to the determination of the trans-effect^{6–8} and trans-influence^{6,9} of the innerspheric sulfoxides. It has been fairly recently shown that $(\text{Ph}_3\text{PCH}_2\text{Ph})[\text{Pt}(\text{R}_2\text{SO})\text{Cl}_3]$ catalyzes the reduction of suitable sulfoxides via CO .¹⁰

The above data refer mainly to the platinum complexes containing lower aliphatic sulfoxides. There is just a few published data concerning the bulky lipophilic ligands of R_2SO (in particular with diaryl sulfoxides). This paper describes a preparation technique and discusses results of the X-ray structure study of the $(\text{Et}_4\text{N})[\text{Pt}(\text{Ph}_2\text{SO})\text{Cl}_3]$ complex containing an S-bound molecule of diphenyl sulfoxide.

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RESULTS AND DISCUSSION

Synthesis of the $(Et_4N)[Pt(Ph_2SO)Cl_3]$ Complex

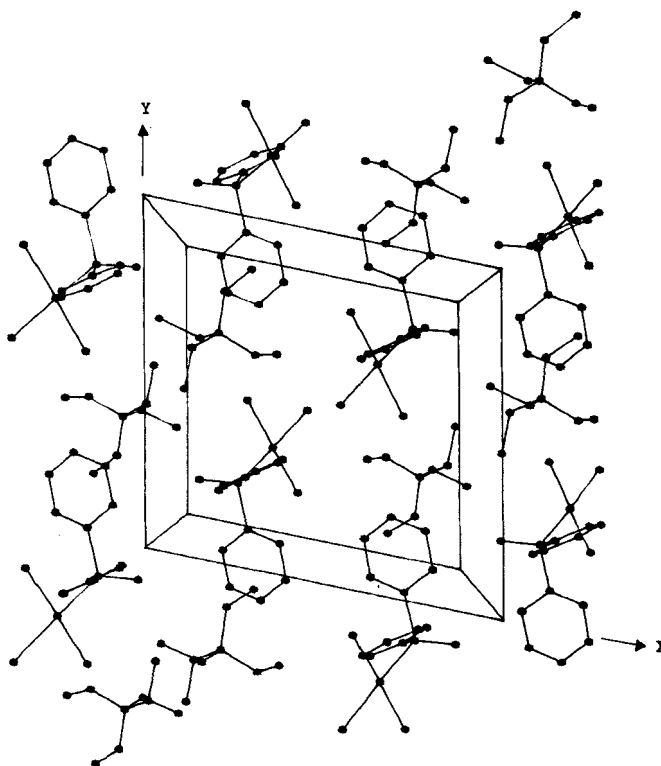
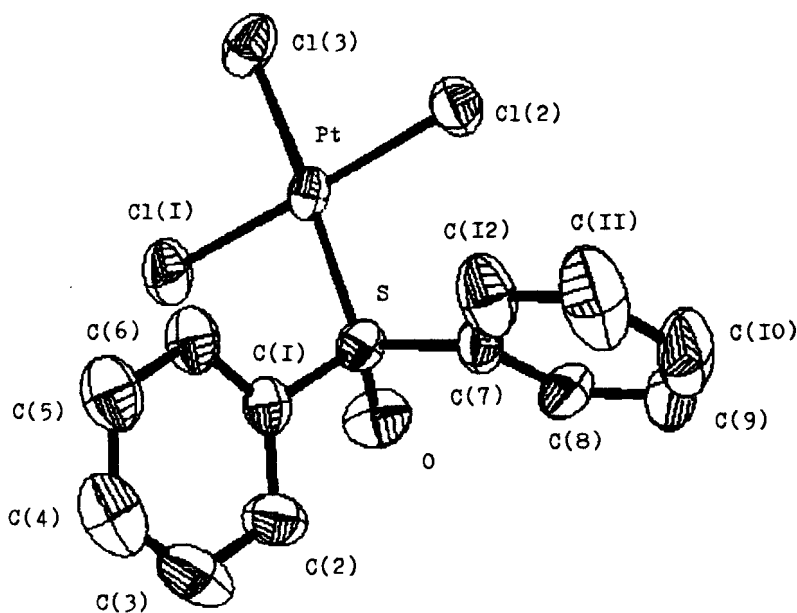
The complexes of the $[PtLCl_3]^-$ type are known to result from the interaction between the $[PtCl_4]^{2-}$ and L in water,¹⁵ by the bridge-splitting reaction of $[Pt_2(\mu-Cl)_2Cl_4]^{2-}$ compounds with a stoichiometric amount of L,¹⁶ as a result of L substitution in the complexes of $[PtL_2Cl_2]$ type for chloride ion in non-aqueous solutions,¹⁷ by reduction of $[PtLCl_3]^-$ complexes,¹⁵ and also as a result of reductive substitution in the $[PtCl_6]^{2-}$ ion.¹⁸ Among the methods listed the simplest is the substitution in the $[PtCl_4]^{2-}$ ion. This very synthesis has been used earlier for the preparation of the $K[Pt(R_2SO)Cl_3]$ ($R_2SO = Me_2SO, Et_2SO, Pr_2SO, (CH_2)_4SO$)^{1,7} complexes. Its difficult to synthesize complexes with bulky lipophilic ligands R_2SO in such a way due to their low solubility in water. The only platinum complex with the coordinated diphenyl sulfoxide $(Et_3NH)[Pt(Ph_2SO)Cl_3]$ has been earlier prepared through the interaction between $(Et_3NH)_2[PtCl_4]$ and Ph_2SO in the ethanol solution for 2 days.¹⁹

We have established that the $(Et_4N)[Pt(Ph_2SO)Cl_3]$ complex can be easily, fastly and with a good yield be prepared in two ways: (i) as a result of the interaction of the $K_2[PtCl_4]$ with an almost equivalent amount of Ph_2SO in a mixture of water and acetonitrile followed by the addition of $(Et_4N)Cl$ to the mixture, complete removal of the solvent and separation of $(Et_4N)[Pt(Ph_2SO)Cl_3]$ from KCl with extraction in acetone. It is noteworthy that performing the reaction in a water-acetonitrile mixture furnishes solubility of starting compounds and proceeding of the reaction in a homogenous liquid phase; (ii) as a result of bridge-splitting reaction of the $(Et_4N)_2[Pt_2(\mu-Cl)_2Cl_4]$ with 2 M of diphenyl sulfoxide occurs in a mixture of nitroethane with acetone. Both procedures have been earlier used to obtain the $(Et_4N)[Pt(R_2SO)Cl_3]$ complexes with different *dialkyl* sulfoxides.^{5,20}

Description of the $(Et_4N)[Pt(Ph_2SO)Cl_3]$ X-ray Structure

Tetraethylammonium trichloro(diphenyl sulfoxide)platinate(II) in the solid phase consists of discrete ions - cation $(Et_4N)^+$ and anion $[Pt(Ph_2SO)Cl_3]^-$ (Fig. 1). The coordination polyhedron of the platinum is slightly distorted square-planar. The deviation of Pt, Cl(1), Cl(2), Cl(3) and S atoms from the plane passed through these atoms are: 0.011, -0.014, -0.014, 0.008 and 0.008 Å. The angles around Pt are close to 90 and 180°. A dihedral angle between the coordination plane and that passed through S, O and Pt atoms is equal to 65.1°.

In the complex studied, diphenyl sulfoxide is coordinated through sulfur (Fig. 2). The Pt—S bond length in $(Et_4N)[Pt(Ph_2SO)Cl_3]$ within 3σ coincides with those in the $Q[Pt(R_2SO)Cl_3]$ complexes containing S-coordinated dialkyl sulfoxides (Table V). The geometry of the innerspheric Ph_2SO changes slightly in coordination. The S=O and S—C bond lengths, 1.462(6) and av.1.785(11) Å, respectively, of the S-coordinated diphenyl sulfoxide actually coincides with those [1.47(3) and av.1.76(5) Å] in a free Ph_2SO molecule.²⁵ It should be mentioned that the S=O bond length in Me_2SO [1.531(5) Å]⁶ decreases considerably upon coordination to Pt(II) (Table V).

FIGURE 1 Unit cell view of $(\text{Et}_4\text{N})[\text{Pt}(\text{Ph}_2\text{SO})\text{Cl}_3]$.FIGURE 2 View of the structure of the $[\text{Pt}(\text{Ph}_2\text{SO})\text{Cl}_3]^-$ ion with the atomic numbering scheme.

For the coordinated Ph_2SO the C—S—O angle in fact does not change while the C—S—C angle increases by about 2° . The dihedral angle between two phenyl rings for coordinated and free diphenyl sulfoxide is 88.5 and 75.8° , respectively. The phenyls in the coordinated Ph_2SO are planar; the maximum deviation of the C atoms from planes is $\pm 0.02 \text{ \AA}$.

The $\text{Pt}_{\text{trans}}\text{Cl}$ bond length [$2.314(2) \text{ \AA}$] exceeds those of $\text{Pt}_{\text{cis}}\text{Cl}$ [$2.306(2)$ and $2.299(2) \text{ \AA}$]. It implies that the trans-influence of diphenyl sulfoxide is somewhat higher than the trans-influence of the chloride ion. It is noteworthy that the change of substituents in R_2SO does not considerably affect the strength of the sulfoxide trans-influence in the Pt(II) complexes (Table V).

In conclusion, tetraethylammonium trichloro(diphenyl sulfoxide)platinate(II) provides a rare example of a structurally characterized compound containing a *S*-coordinated diaryl sulfoxide (see footnote "b" in Table V). On the other hand, it should be mentioned that there are published data on the complexes with *O*-coordinated diaryl sulfoxide (see, for example ²⁶).

TABLE I
Crystal data and data collection parameters for $(\text{Et}_4\text{N})[\text{Pt}(\text{Ph}_2\text{SO})\text{Cl}_3]$

cryst. system	monoclinic
space group	$P 2_1/n$
$a, \text{\AA}$	$11.525(4)$
$b, \text{\AA}$	$11.078(3)$
$c, \text{\AA}$	$19.106(7)$
$\beta, \text{deg.}$	$102.02(3)$
$V, \text{\AA}^3$	$2386(3)$
Z	4
$\rho_{\text{calc.}}, \text{g cm}^{-3}$	1.76
No. independent reflections	2523
No. reflections with	2348
$I \geq 3\sigma(I)$	
Max. $2\theta, \text{deg.}$	50
scan technique	$2\theta/\theta$
scan speed, $^\circ\text{min}^{-1}$	3.4 - 12.0
h, k, l	0 to 16, -16 to 16, 0 to 26
μ, cm^{-1}	63.76
crystal size, mm	$0.093 \times 0.055 \times 0.071$
crystal faces	001, $1\bar{1}0$, 110
R	0.025
wR	0.027
S	0.83

EXPERIMENTAL

Syntheses of $(Et_4N)[Pt(Ph_2SO)Cl_3]$. a) A solution of diphenyl sulfoxide (0.071 g, 0.35 mmol) in acetone (10 ml) was added to a solution of $(Et_4N)_2[Pt_2(\mu-Cl)_2Cl_4]$ ¹¹ (0.149 g, 0.17 mmol) in nitroethane (30 ml) and boiled in the open beaker for about 10 min. Toluene (40 ml) was added to the solution left after evaporation and after 10 min the precipitate formed was filtered off and washed with toluene (5×5 ml), and dried in air at 20–25°C. Yield of $(Et_4N)[Pt(Ph_2SO)Cl_3]$ = 0.190 g, 87% based on Pt.

b) A solution of diphenyl sulfoxide (0.51 g, 2.52 mmol) in acetonitrile (30 ml) was added to a solution of $K_2[PtCl_4]$ (1.03 g, 2.48 mmol) in water (10 ml). The mixture was heated at 85–90°C for 10 min and $(Et_4N)Cl$ (0.46 g, 2.78 mmol) in acetonitrile (10 ml) was added. After heating the mixture to boiling the precipitate formed was filtered off. The filtrate was evaporated till an oily residue formed. The $(Et_4N)[Pt(Ph_2SO)Cl_3]$ complex was extracted from the residue with boiling acetone (3×10 ml), the solution was filtered and the solvent was allowed to evaporate to dryness. Yield of $(Et_4N)[Pt(Ph_2SO)Cl_3]$ = 1.22 g, 78% based on Pt.

The $(Et_4N)[Pt(Ph_2SO)Cl_3]$ complex was isolated from acetone as orange rod-like crystals. M.P. = 184°C (Kofler tables). IR (KBr pellet), cm^{-1} : 1130, 1070 $\nu(S=O)$, 336, 316 and 310 (sh.) $\nu(Pt-Cl)$. Molar conductivity is 56 $\Omega^{-1} cm^2 mol^{-1}$ (DMF). For $C_{20}H_{30}Cl_3NOPtS$ Calc. % Cl = 16.8, % Pt = 30.8. Found % Cl = 17.1, % Pt = 31.0.

X-ray crystallographic study of $(Et_4N)[Pt(Ph_2SO)Cl_3]$. Diffraction data were collected on Nicolet P3 diffractometer, using $MoK\alpha$ radiation, β -filter; cell parameters from refined angles of 12 centered reflections with 2θ between 22 and 25°. Standard reflections 200, 020, 004 measured every 100 reflections showed no change with time. Diffractometer data were processed using the program PROFIT¹² with profile analysis of the reflections. The structure was solved by means of Fourier syntheses based upon the Pt-atom coordinates obtained from Patterson synthesis and was refined by full-matrix least squares based on F with weights $w = 1/\sigma^2(F) + 0.000885 F^2$. All non-hydrogen atoms were treated anisotropically. Hydrogen atoms were treated isotropically. Lorentz, polarization and absorption correction were made. Calculations were carried out with SHELXTL¹³ on Nova-3 computer. Scattering factors were obtained from International Tables for X-ray crystallography.¹⁴ Crystal data and data collection parameters are given in Table I, atomic coordinates, bond lengths and bond angles in Tables II–IV.

TABLE II
Atom coordinates ($\times 10^4$) and temperature factors ($\text{\AA}^2 \times 10^3$) for
 $(Et_4N)[Pt(Ph_2SO)Cl_3]$

Atom	X	Y	Z	U
Pt	3302(1)	3270(1)	3457(1)	38(1)
Cl(1)	2485(2)	4739(2)	4017(1)	67(1)
Cl(2)	4121(2)	1851(2)	2859(1)	57(1)
Cl(3)	4461(2)	4805(2)	2789(1)	62(1)
S	2153(2)	1881(2)	4119(1)	42(1)
O	872(5)	1724(5)	4019(3)	62(2)
C(1)	2464(6)	2196(7)	5028(4)	42(3)
C(2)	1641(7)	1632(7)	5510(5)	59(3)
C(3)	1863(9)	1875(9)	6201(5)	72(4)
C(4)	2904(9)	2561(9)	6422(4)	61(4)
C(5)	3722(8)	3072(8)	5949(5)	60(3)
C(6)	3523(7)	2895(7)	5241(4)	50(3)
C(7)	2482(7)	387(6)	4068(4)	48(3)
C(8)	1610(9)	-542(9)	3777(5)	76(4)
C(9)	1834(14)	-1740(11)	3755(8)	106(6)

TABLE II (Continued)

C(10)	2883(18)	-1956(10)	3996(7)	112(8)
C(11)	3725(13)	-1056(11)	4257(6)	105(6)
C(12)	3542(9)	148(8)	4295(5)	66(4)
N	1761(5)	6701(5)	6484(3)	44(2)
C(13)	1950(7)	8067(8)	6347(5)	62(3)
C(14)	2835(9)	8878(9)	6816(7)	97(5)
C(15)	2904(7)	6238(8)	6451(4)	52(3)
C(16)	3555(8)	6418(10)	5779(5)	82(4)
C(17)	1267(8)	6390(8)	7211(5)	60(3)
C(18)	58(8)	6722(9)	7361(5)	72(4)
C(19)	888(7)	6087(7)	5936(4)	53(3)
C(20)	616(9)	4708(9)	5976(6)	83(4)

TABLE III
Bond lengths (Å) for (Et₄N)[Pt(Ph₂SO)Cl₃]

Pt-Cl(1)	2.306(2)	Pt-Cl(2)	2.299(2)
Pt-Cl(3)	2.314(2)	Pt-S	2.207(2)
S-O	1.462(6)	S-C(1)	1.793(8)
S-C(7)	1.777(8)	C(1)-C(2)	1.38(1)
C(1)-C(6)	1.36(1)	C(2)-C(3)	1.36(1)
C(3)-C(4)	1.35(1)	C(4)-C(5)	1.34(1)
C(5)-C(6)	1.38(1)	C(7)-C(8)	1.40(1)
C(7)-C(12)	1.37(1)	C(8)-C(9)	1.40(2)
C(9)-C(10)	1.36(3)	C(10)-C(11)	1.33(2)
C(11)-C(12)	1.40(2)	N-C(13)	1.51(1)
N-C(15)	1.51(1)	N-C(17)	1.51(1)
N-C(19)	1.51(1)	C(13)-C(14)	1.51(1)
C(15)-C(16)	1.48(1)	C(17)-C(18)	1.54(1)
C(19)-C(20)	1.50(1)		

TABLE IV
Bond angles (°) for (Et₄N)[Pt(Ph₂SO)Cl₃]

Cl(1)-Pt-Cl(2)	177.7(1)	Cl(1)-Pt-Cl(3)	89.7(1)
Cl(2)-Pt-Cl(3)	88.4(1)	Cl(1)-Pt-S	87.3(1)
Cl(2)-Pt-S	94.6(1)	Cl(3)-Pt-S	177.0(1)
Pt-S-O	116.9(3)	Pt-S-C(1)	110.7(2)
O-S-C(1)	107.8(3)	Pt-S-C(7)	113.9(3)

TABLE IV (Continued)

O-S-C(7)	106.7(4)	C(1)-S-C(7)	99.3(4)
S-C(1)-C(2)	118.0(5)	S-C(1)-C(6)	121.3(6)
C(2)-C(1)-C(6)	120.5(7)	C(1)-C(2)-C(3)	118.5(7)
C(2)-C(3)-C(4)	121.6(8)	C(3)-C(4)-C(5)	119.5(8)
C(4)-C(5)-C(6)	121.2(8)	C(1)-C(6)-C(5)	118.5(7)
S-C(7)-C(8)	116.6(7)	S-C(7)-C(12)	122.2(6)
C(8)-C(7)-C(12)	121.2(8)	C(7)-C(8)-C(9)	117.5(9)
C(8)-C(9)-C(10)	120.1(9)	C(9)-C(10)-C(11)	122.0(9)
C(10)-C(11)-C(12)	120.1(9)	C(7)-C(12)-C(11)	119.0(9)
C(13)-N-C(15)	112.3(5)	C(13)-N-C(17)	110.9(6)
C(15)-N-C(17)	105.9(6)	C(13)-N-C(19)	105.9(6)
C(15)-N-C(19)	111.4(6)	C(17)-N-C(19)	110.5(5)
N-C(13)-C(14)	115.7(8)	N-C(15)-C(16)	116.4(7)
N-C(17)-C(18)	115.7(7)	N-C(19)-C(20)	113.7(7)

TABLE V
Selected bond lengths in the structures of Q[Pt(R₂SO)Cl₃] complexes

Complex	Bond lengths, Å				Ref.
	Pt— _{trans} Cl	Pt— _{cis} Cl	Pt—S	S=O	
K[Pt(Me ₂ SO)Cl ₃]	2.318(5)	2.296(5) 2.302(6)	2.193(5)	1.476(15)	21
(Ph ₃ PCH ₂ Ph)[Pt(Me ₂ SO)Cl ₃]	2.309(5)	2.271(5) 2.275(6)	2.185(5)	1.46(1)	9
[Pt(NH ₃) ₄][Pt(Me ₂ SO)Cl ₃] ₂	2.321(2)	2.282(3) 2.307(3)	2.200(3)	1.484(8)	22
[Pt(C ₃ H ₁₀ N ₂) ₂][Pt(Me ₂ SO)Cl ₃] ₂	2.312(4)	2.304(4) 2.288(3)	2.189(4)	1.47(1)	23
K[Pt(Et ₂ SO)Cl ₃]	2.331(2)	2.299(2) 2.315(2)	2.209(2)	1.463(5)	20
K[Pt(Me ^p tolSO)Cl ₃] ^a	2.324(4)	2.305(4) 2.311(4)	2.202(4)	1.47(1)	24
	2.320(5)	2.296(4) 2.294(4)	2.204(4)	1.48(1)	
(Et ₄ N)[Pt(Ph ₂ SO)Cl ₃]	2.314(2)	2.306(2) 2.299(2)	2.207(2)	1.462(6)	This work ^b
K[Pt(Ph ₂ SO)Cl ₃] · Me ₂ CO	2.327(3)	2.302(3) 2.302(3)	2.214(3)	1.469(8)	

^a Data are given for two crystallographically independent molecules.^b When the paper was prepared for publication we learned that N. Farrell group had performed synthesis and X-ray structure analysis of the K[Pt(Ph₂SO)Cl₃] · Me₂CO complex (N. Farrell, private communication).

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