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Oxidative addition of 2,3,4-benzotrithiepin 1-oxide to platinum(0) compounds: a convenient synthetic route to platinum(II) thiosulfinato complexes ^{1,2}

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Abstract

The oxidative addition of 2,3,4-trithiepin 1-oxide 2 to $(Ph_3P)_2Pt(C_2H_4)$ 5 gives the platinum(II) thiolato thiosulfinato complex *cis*-{ $(Ph_3P)_2Pt[S-S(O)-CH_2-C_6H_4-CH_2-S]$ } 6. The structure of 2 was established by X-ray structural analysis. 2 crystallizes in the monoclinic space group $P2_1/c$ with a = 878.52(10), b = 1263.4(2), c = 936.95(10) pm, $\beta = 117.818(9)^\circ$, Z = 4. The 2,3,4-trithiepin 2-oxide 4 reacts with 5 to form the diplatinum(II) thiolato complex 8. Formally, 1 equiv. SO has been eliminated during this reaction. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Trithiepin 1-oxide; Thiosulfinato complexes: Crystal structure; Platinum complexes; Thiosulfinic acid anions

1. Introduction

The synthesis and investigation of thiosulfinic S-esters R– S(O)–S–R' (thiosulfinates) have received much attention in recent years. The impetus stems from the observation that some of these compounds cause inhibition of the enzymes cyclooxygenase and 5-lipoxygenase [2]. Thiosulfinates can be viewed as the S esters of the unknown thiosulfinic acid R– S(O)–SH. Some relatively stable salts of thiosulfinic acids containing bulky substituent groups have been synthesized by Mikołajczyk et al. [3]. The stability of these salts is attributed to steric protection by bulky groups bonded to the central sulfinyl sulfur atom.

Complexes of the type $L_mMSS(O)R$ containing the thiosulfinato ligand are scarcely known. Shaver and Plouffe [4] and we [1,5] have recently prepared for the first time stable thiosulfinato complexes of ruthenium(II) by reaction of *N*-(alkyl- and arylsulfinyl)phthalimides phth–S(O)–R (phth=phthalimido) with C₅H₅Ru(L)(PPh₃)(SH) (L= CO, PPh₃); here a new sulfur–sulfur bond has been formed



(Scheme 1, A). The second type of synthesis (Scheme 1, B) involves the oxidative addition of reagents R-S(O)-S-X (X = phth, SR) already containing the thiosulfinato unit, e.g. N-[(alkyl- and arylsulfinyl)thio]-phthalimides phth-S-S(O)-R [6] or trisulfide 1-oxides to low-valent platinum complexes.

We now describe the preparation of novel (bisphosphane)Pt(II) complexes containing thiosulfinato ligands by oxidative addition of 2,3,4-trithiepin 1-oxide **2** to $(Ph_3P)_2$ -Pt(C₂H₄) **5**. A crystal structure is presented for **2**.

2. Experimental

The experiments yielding **2–8** were performed under dry, oxygen-free nitrogen in distilled solvents. IR (recorded in KBr, Nicolet ZDX 5), ¹H NMR (Jeol EX 400, internal standard TMS), ¹³C NMR (Jeol EX 400, internal standard TMS),

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¹ Dedicated to Professor Ivano Bertini.

² Metal complexes of functionalized sulfur-containing ligands, Part 13. For Part 12 see Ref. [1].

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³¹P NMR (Jeol GSX 270, external standard 85% aqueous H_3PO_4), and FAB-MS (VG-ZAB-VSEQ spectrometer [7]) measurements were taken. 2,3,4-benzotrithiepin 1 [8], 2,3,4-benzotrithiepin 1-oxide 2 [8], α, α' -dimercapto-*o*-xylol 3 [8b,9], and (Ph₃P)₂Pt(C₂H₄) 5 [10] were prepared by literature procedures. K₂PtCl₄ was a gift from Degussa AG, Werk Wolfgang.

2.1. Preparation of 2 [8]

Colorless crystals. Yield 78%. M.p. 134–136°C (135– 137°C [8]). IR: ν (SO) 1075 s cm⁻¹. ¹H NMR (δ , CDCl₃): 4.08/4.46 (2H, CH₂S, AB spin system, J=11.8 Hz), 4.19/ 4.59 (2H, CH₂S(O), AB spin system, J=12.7 Hz), 7.14– 7.37 (m, 4H, Ph). ¹³C{¹H} NMR (δ , CDCl₃): 39.4 (s, CH₂S), 61.3 (s, CH₂S(O)), 128.9/129.4/130.3/130.4/ 130.5/132.1 (s, Ph).

2.2. Preparation of 4 [11]

A solution of 0.4 ml of thionyl chloride (660 mg, 5.6 mmol) in 20 ml of ether was added dropwise and with stirring during 1 h to an ice-cold solution of 3 [8,9] (950 mg, 5.6 mmol) in 200 ml of the same solvent. After a few minutes this solution changed its color from yellow to dark brown, and over a period of 30 min a black material precipitated. This precipitate was centrifuged off and the brown solution was washed three times with a saturated, aqueous solution of NaHCO₃. The organic phase was dried with MgSO₄ and the solvent was removed. Purification of the brownish crude product by chromatography on silica gel (dichloromethane:hexane 1:4) gave 4 as colorless solid. Yield 145 mg (12%). M.p. 116-118°C ([11]), dec. Anal. Calc. for $C_8H_8OS_3$ (216.34 g mol⁻¹): C, 44.41; H, 3.73; S, 44.46. Found: C, 44.25; H, 3.81; S, 44.52%. IR: v(SO) 1081 s cm⁻¹. ¹H NMR (δ, CDCl₃): 4.18/4.88 (4H, CH₂S, AB spin system, J = 14.8 Hz), 7.16 (m_c, 4H, Ph). ¹³C{¹H} NMR (δ , CDCl₃): 50.0 (s, CH₂S), 127.9/130.4/140.4 (s, Ph).

2.3. Preparation of 6

A solution of **5** (149 mg, 0.2 mmol) in 10 ml of toluene was cooled to -60° C. To this solution 45 mg (0.21 mmol) of **2** were added with stirring over a period of 15 min. After stirring the bright yellow solution for an additional 10 h at room temperature a pale yellow solid began to separate. The precipitate was collected by centrifugation, washed twice with 10 ml of ether and dried in vacuo. Yield 187 mg (89%). M.p. 176–177°C, dec. Anal. Calc. for C₄₄H₃₈OP₂PtS₃ (935.9 g mol⁻¹): C, 56.46; H, 4.09; S, 10.28. Found: C, 55.32; H, 4.16; S, 10.26%. IR: ν (SO) 1034 s cm⁻¹. ¹H NMR (δ , CD₂Cl₂): 3.08/3.60 (4H, CH₂S, AB spin system, *J*=12.3 Hz), 4.47/4.83 (4H, CH₂S(O), AB spin system, *J*=12.9 Hz), 6.7–7.1 (m, 34H, Ph). ³¹P{¹H} NMR (δ , CDCl₃): 18.96/21.49 (AB spin system, *J*(PP) = 17.2, *J*(PPt) = 2876/3079 Hz). MS (pos-FAB), *m/z* (relative intensity $\begin{array}{l} (\%) : 935 \ (85) \ (M+H)^+; 903 \ (27) \ [M^{-32}S]^+; \ 887 \ (22) \\ [M^{-32}SO]^+; \ 784 \ (67) \ [(Ph_3P)PtS_2]^+; \ 719 \ (100) \\ [(Ph_3P)_2Pt]^+. \end{array}$

2.4. Reaction of 6 with dppe (detection of 7)

A dichloromethane solution of 180 mg (0.19 mmol) of 6 and 147 mg (0.4 mmol) of dppe was stirred at room temperature for 2 h. After removal of the solvent the resulting yellow solid was washed three times with 10 ml of ether. After drying in vacuo the yellow product was identified as 7. Yield 136 mg (92%). M.p. 185-187°C, dec. Anal. Calc. for C₃₄H₃₂- OP_2PtS_2 (777.8 g mol⁻¹): C, 52.50; H, 4.15; S, 8.24. Found: C, 51.74; H, 4.27; S, 8.04%. IR: ν (SO) 964 s cm⁻¹. ¹H NMR (δ, CD_2Cl_2) : 3.17-3.28 $(dd, {}^2J(HH) = 13.4, {}^4J(HP) =$ 3.4, ${}^{3}J(\text{HPt}) = 32$ Hz, 1H, 1- H_{eq}^{2}), 3.60–3.79 (dd, ${}^{2}J(\text{HH}) = 12.3, {}^{4}J(\text{HP}) = 10.0, {}^{3}J(\text{HPt}) = 54 \text{ Hz}, 1\text{H}, 4\text{-}$ H_{eq}^4), 4.03–4.12 (dd, ²J(HH) = 12.4, ⁴J(HP) = 3.7, $^{3}J(HPt) = 22$ Hz, 1H, 4-H_{ax}³), 4.73-4.90 (dd, $^{2}J(HH)$ $= 13.4, {}^{4}J(\text{HP}) = 11.2, {}^{3}J(\text{HPt}) = 54 \text{ Hz}, 1\text{H}, 1\text{-}H_{\text{av}}).$ ¹³C{¹H} NMR (δ , CDCl₃): 26.85 (s, CH₂S), 59.71 (s, CH₂S(O)), 125.4/126.6/129.3/129.8/134.7/144.7 - (s, Ph). ${}^{31}P{}^{1}H{} NMR (\delta, CH_2Cl_2): 33.06/47.49 (AB spin)$ system, J(PP) = 9.7, J(PPt) = 2139/3127 Hz).

Solvent was removed from the combined etheral solutions; after drying in vacuo the resulting oily residue was identified by its ³¹P NMR spectrum as a mixture of Ph₃P=S (δ =41.3) and dppe.

2.5. Reaction of 4 with 5 (detection of 8)

To a stirred solution of **5** (100 mg, 0.13 mmol) in 10 ml of toluene an equimolar amount of **4** (29 mg, 0.13 mmol) was added. After stirring for 1 h a yellow solid was separated by centrifugation. The microcrystalline powder was washed twice with 10 ml of ether; traces of sulfur were removed by treating the crude product with 5 ml of a mixture of dichloromethane:carbon disulfide (1:1). The resulting yellow powder, which was dried in vacuo, is only slightly soluble in common organic solvents. No satisfactory elemental analysis could be obtained of compound **8**. Yield 40 mg (47%). M.p. 249–253°C, dec. *Anal.* Calc. for C₅₂H₄₆P₂Pt₂S₄ (1251.3 g mol⁻¹): C, 49.91; H, 3.70; S, 10.23. Found: C, 51.42; H, 3.88; S, 9.86%.

2.6. X-ray structural analysis of 2

Crystal and refinement data are summarized in Table 1. Single crystals of **2** were obtained by layering a methylene chloride solution with hexane followed by cooling to 0°C. Cell parameters were obtained by least-squares refinement of 25 reflections in the range $10^{\circ} < \theta < 13^{\circ}$. The data collection was performed, at room temperature 293(2) K, using a Nonius CAD4 diffractometer, ω -scans, scan width (1.21 + 0.95 tan θ)° and a maximum counting time of 45 s. Three standard reflections were measured every 2 h and showed an

Table 1 Crystallographic data collection parameters of **2**

Formula	$C_8H_8OS_3$
Formula weight	216.32
Crystal size (mm)	$0.27 \times 0.47 \times 0.55$
Crystal system	monoclinic
Space group	$P2_1/c$ (No. 14)
a (pm)	878.5(1)
<i>b</i> (pm)	1263.4(2)
<i>c</i> (pm)	936.9(1)
β (°)	117.818(9)
$V(pm^3)$	$919.7(2) \times 10^{6}$
Z	4
$D_c (g cm^{-3})$	1.56
Radiation	graphite-monochromatized Mo Ka
λ (pm)	71,069
Absorption coefficient μ (cm ⁻¹)	7.212
2θ range (°)	5-45
Index ranges	$0 \le h \le 9, 0 \le k \le 13, -10 \le l \le 9$
c	

intensity decay of 1.8%. The corrections for *Lp*, linear decay and absorption ($T_{min} = 0.9117$, $T_{max} = 0.9982$) were applied on a total of 1365 reflections, 1273 independent ($R_{int} = 0.0088$) and 1099 with $I^{3}2\sigma_{I}$. All non-hydrogen atoms were refined anisotropically. The final *R*1 was 0.0300 (wR2 = 0.0820) for 1099 reflections and 109 variables and R1 = 0.0373 (wR2 = 0.0879) for all 1273 data. The structure was solved using SHELXS-86 [12] and refined by SHELXL-93 [13] with $|F^{2}|$. The extrema of the final difference Fourier synthesis were $+0.269/-0.145 \text{ e} \times 10^{-6} \text{ pm}^{-3}$. Plotting programs were XPMA. ZORTEP [14].

3. Results and discussion

3.1. Synthesis of 2 and 4; X-ray structure of 2

Whereas the chemical and dynamic properties of (a)cyclic trisulfides have been intensively investigated [15], very little is known about the chemistry of their sulfur oxide derivatives. The regiospecific oxidation of (a)cyclic trisulfides with 1 equiv. peroxyacid gives the 1-oxides [8]. The electrophilic oxidation takes place exclusively at the more electron-rich sulfur atom bound to the alkyl group. The preparation of the corresponding 2-oxide **4** was achieved by reaction of the dithiol **3** with thionyl chloride in highly diluted solution



(Scheme 2). Whereas the linear trisulfide 1-oxides exhibit low stability (their decomposition mechanisms have been carefully studied [16]), the 2,3,4-benzotrithiepin 1-oxide 2 is a remarkably stable compound. Thus crystals of X-ray quality were obtained and 2 was characterized by X-ray diffraction (Table 1, Section 2). The atomic positional parameters are listed in Table 2 and selected bond length and angles are given in Table 3. As can be seen from the small dihedral angle C(1)-C(2)-C(7)-C(8) of 3°, the molecule exhibits an explicit boat conformation (Fig. 1). The presence of the oxygen atom at the terminal sulfur atom has a great cffect on the sulfur-sulfur bonds and there is a difference of 14 pm between the lengths of S(O)-S(214.8(1) pm) and the S-S bond (200.9(1) pm). Similar differences have been established in S_8O [17] and $tBu-S(O)_2-S-S-tBu$ [18] (11 pm). A comparable value for the S(O)-S distance is found in $tBu-S(O)_2-S-S(O)-tBu$; the S(O)-S bond is 4 pm longer than the $S(O)_2$ -S bond [19]. The sulfur-oxygen bond length (147.2(2) pm) is similar to that in $tBu-S(O)_2-S-$ S(O)-tBu [19] and in the thiosulfinate tol-S(O)-S-tol [1,20]. In the structure of 2, the oxygen atom bound to the sulfur atom is located at the axial position which agrees well

Table 2

Fractional atomic coordinates for non-hydrogen atoms and equivalent isotropic thermal parameters ($Å^2 \times 10^3$) with e.s.d.s in parentheses for **2**

Atom	x	у	ĩ.	$U_{ m eq}$
S1	0.44030(9)	-0.02858(6)	0.67163(9)	0.0438
S2	0.41667(10)	0.13990(6)	0.63784(9)	0.0459
S3	0.47338(11)	0.19565(7)	0.85792(10)	0.0510
0	0.3137(3)	-0.0688(2)	0.7195(3)	0.0609
C1	0.7106(4)	0.1946(3)	0.9733(4)	0.0501
C2	0.8043(4)	0.1443(2)	0.8920(3)	0.0437
C3	0.9265(4)	0.2036(3)	0.8719(4)	0.0589
C4	1.0190(5)	0.1601(4)	0.8028(5)	0.0730
C5	0.9923(5)	0.0573(4)	0.7503(5)	0.0748
C6	0.8711(4)	-0.0033(3)	0.7695(4)	0.0607
C7	0.7770(4)	0.0393(2)	0.8408(3)	0.0439
C8	0.6444(4)	-0.0275(2)	0.8561(4)	0.0477

Table 3		
Selected bond distances	(pm), bond angles and	torsion angles (°) of ${\bf 2}$

Bond distances			
S(1)-O	147.9(2)	S(1) - S(2)	214.8(1)
S(2)-S(3)	200.9(1)	S(1)-C(8)	181.9(3)
S(3) - C(1)	184.6(3)	C(1)-C(2)	149.9(5)
C(7)-C(8)	149.9(4)		
Bond angles			
C(7)-C(8)-S(1)	112.3(2)	S(1)-S(2)-S(3)	103.6(2)
C(8)-S(1)-O	105.2(2)	S(2)-S(1)-O	110.6(1)
C(8)-S(1)-S(2)	110.7(1)	S(2)-S(3)-C(1)	105.7(1)
C(2)-C(1)-S(3)	116.3(2)		
Torsion angles			
O=S(1)=S(2)=S(3)	62.9		
C(8)-S(1)-S(2)-S(3)	-46.2		
C(1)-C(2)-C(7)-C(8)	3.5		



Fig. 1. Molecular structure of 2 in the crystal.

with the corresponding sulfur-oxygen position observed for [1,2,3] trithiolo [h] benzo-penta-thiepin monoxide [21].

3.2. Reactions of 2,3,4-benzotrithiepin 1-oxide (2) and 2-oxide (4) with 5

A solution of platinum(0) complex 5 in toluene was treated with the 2,3,4-benzotrithiepin 1-oxide 2 at -70° C and then the mixture was warmed up to room temperature. After a period of 10 h the reaction is complete. 5 inserts regiospecifically *via* oxidative addition into the sulfur–sulfur bond achieving the thiolato thiosulfinato platinum(II) complex 6 (Scheme 3); however, an insertion into the S(O)–S bond could not be detected. This result is confirmed by IR and ³¹P NMR spectroscopic data.

The stretching mode of the sulfoxide group occurs at 1034 cm⁻¹ which is in agreement with the values observed for the ruthenium(II) [5b,c] and platinum(II) thiosulfinato complexes [6a]. In the case of insertion of platinum(0) species into the S(O)-S bond a disulfano sulfenato complex (Ph₃P)₂Pt-S(O)-CH₂-C₆H₄-CH₂-S-S would result; the S=O stretching modes from compounds containing a sulfenato group occur at significantly lower wavenumbers between 960 and 990 cm⁻¹ [1,7,22]. The ³¹P NMR spectrum of **6**



shows the expected AB spin pattern for two *cis*-phosphines *trans* to two ligands having differing *trans* influences. The two values of ${}^{1}J(PPt)$ are rather similar at 2876 and 3079 Hz, and these are assigned to phosphines *trans* to thiolato sulfur atoms. Note, however, that the *trans* influence of a sulfenato group is higher than that of the thiolato and thiosulfinato ligand respectively [1,6,7]; thus, the value of ${}^{1}J(PPt)$ (sulfenato *trans* to phosphine) should be smaller than those observed for thiolato and thiosulfinato ligands *trans* to phosphine. This is an important evidence for the cleavage of the sulfur–sulfur bond instead of the sulfur–sulf-oxide linkage.

Complex 6 shows a relatively intensive protonated molecular peak at m/z=935. There are also two additional weak peaks at m/z=903 and 887. These are assigned to the complex fragments $[M-^{32}S]^+$ and $[M-^{32}SO]^+$ respectively.

When complex **6** was treated with an excess of dppe in the first step the triphenyl phosphine ligands were substituted (Scheme 3). This complex undergoes a partial desulfurization reaction with the uncoordinated triphenyl phosphines to produce Ph₃P=S and the sulfenato thiolato platinum(II) complexes **7**. Here the value of ${}^{+}J(PPt)$ for the phosphine *trans* to the sulfenato ligand (2139 Hz) is very different from that *trans* to the thiolato ligand (3127 Hz). **7** has also been produced by an alternative method through oxidative addition of 1,4-dihydro-2,3-benzodithiin-S-oxide to dppePt(C₂H₄) [22].

Unexpectedly, the reaction of the benzotrithiepine 2-oxide 4 with 5 produces the bimetallic dithiolato platinum(II) complex 8 (Scheme 4). It seems that sulfur monoxide SO has formally been eliminated from 4. A similar elimination was observed when the reaction was carried out with benzotrithiol 2-oxide and 5 [1,23]. These unexpected metal-mediated

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[(Ph<sub>3</sub>P)Pt(\mu-S-CH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>-CH<sub>2</sub>-S)]<sub>2</sub>
8
Scheme 4.
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eliminations of sulfur monoxide are not yet understood in detail.

4. Supplementary material

Further details of the crystal structure may be obtained upon request from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information GmbH, D-76344 Eggenstein-Leopoldshafen (Germany), given reference to the depository number CSD 408136 and citing the authors and this paper.

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