

# *cis*- and *trans*-Influence on Bond Distances. Synthesis and Crystal Structures of the Bis(trifluoromethanesulfonates) of Tetrakis(dimethyl sulfide)- and Tetrakis(1,4-thioxane)platinum(II)

Zivadin Bugarcic,\* Bertil Norén,† Åke Oskarsson, Claes Stålhandske and Lars I. Elding

Inorganic Chemistry, Chemical Center, University of Lund, P.O. Box 124, S-221 00 Lund, Sweden

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Crystals of  $[\text{Pt}\{(\text{CH}_3)_2\text{S}\}_4](\text{CF}_3\text{SO}_3)_2$ ,  $M_r = 741.76$ , and  $[\text{Pt}(\text{C}_4\text{H}_8\text{OS})_4](\text{CF}_3\text{SO}_3)_2 \cdot \text{H}_2\text{O}$ ,  $M_r = 927.93$ , were prepared by dissolution of platinum(II) hydroxide in solutions of trifluoromethanesulfonic acid in dimethyl sulfide and 1,4-thioxane, respectively. The structures were determined from X-ray intensity data (Mo  $K\alpha$ ,  $\lambda = 0.7107 \text{ \AA}$ ) collected at room temperature with a CAD-4 diffractometer. The space group for the dimethylsulfide compound is  $P2_1/c$  with  $a = 8.5630(8)$ ,  $b = 8.692(1)$ ,  $c = 16.360(3) \text{ \AA}$ ,  $\beta = 97.064(9)^\circ$ ,  $V = 1208.4(2) \text{ \AA}^3$ ,  $Z = 2$ ,  $D_x = 2.038 \text{ Mg m}^{-3}$ ,  $F(000) = 720$  and  $\mu = 6.43 \text{ mm}^{-1}$ . The refinements converged to  $R = 0.025$  for 1922 reflections with  $I > 3\sigma(I)$ . The 1,4-thioxane compound crystallizes in the space group  $C2/c$  with  $a = 14.438(2)$ ,  $b = 13.016(2)$ ,  $c = 21.696(3) \text{ \AA}$ ,  $\beta = 126.32(1)^\circ$ ,  $V = 3285.0(6) \text{ \AA}^3$ ,  $Z = 4$ ,  $D_x = 1.876 \text{ Mg m}^{-3}$ ,  $F(000) = 1832$  and  $\mu = 4.76 \text{ mm}^{-1}$ . The refinements converged to  $R = 0.035$  for 2268 reflections with  $I > 3\sigma(I)$ . The coordination around platinum is square-planar with four sulfur donor ligands in both cases. In the dimethyl sulfide complex the two unique  $(\text{CH}_3)_2\text{S}$  groups are disordered. The sulfur atoms occupy two different positions with coinciding methyl carbon atoms. The platinum–sulfur distances are between 2.317(3) and 2.321(4)  $\text{ \AA}$  in the dimethylsulfide and between 2.318(2) and 2.321(2)  $\text{ \AA}$  in the thioxane complex. Packing forces thus have a negligible influence on these Pt–S distances, and the mean Pt–S distance of 2.319(2)  $\text{ \AA}$  is a suitable reference distance for calculation of the *cis*- and *trans*-influence by other donor atoms on the Pt–S bond in other complexes.

The present work is part of an investigation aiming to correlate kinetic, thermodynamic and structural properties of square-planar complexes of palladium(II) and platinum(II) containing sulfur–donor ligands. The *trans*-influence,<sup>1–3</sup> expressed as the extent to which a ligand L in a metal complex lengthens or shortens a bond M–X *trans* to itself,<sup>3</sup> is expected to run approximately parallel to the *trans*-effect of L, as displayed by the substitution rate of X.<sup>4</sup> In fact, Bürgi<sup>5</sup> has recently demonstrated that even small structural changes in the ground state will affect the reaction rates dramatically for related compounds undergoing the same type of reaction.

In aqueous 1 M perchloric acid, both dimethyl sulfide and 1,4-thioxane form very strong complexes with similar stability,<sup>6</sup> indicating that all four thioxane ligands are coordinated to the platinum via the sulfur atoms. In the solid state, thioethers are known to act as bridging ligands via the sulfur atoms in dimeric platinum(II) halide complexes.<sup>7–12</sup> Also, monomeric complexes of the type  $\text{PtL}_2\text{X}_2$  (where L is dimethyl sulfide or 1,4-thioxane and X is a

coordinating anion such as chloride or bromide), are known to exist in the solid state.<sup>13–18</sup> There seems to be no reported crystal structure for a platinum complex with four thioether ligands. We here report the synthesis of two such complexes with the non-coordinating counter-ion trifluoromethanesulfonate. The Pt–S distances in these complexes is only affected by intermolecular forces, since the *cis*- and *trans*-influences are constant (and by definition zero, cf. below). The average of the Pt–S distances will therefore be a natural reference for calculating *cis*- and *trans*-influences by other donor atoms on Pt–S by comparison with bonding distances in other compounds.<sup>3</sup>

## Experimental

*Synthesis of  $[\text{Pt}(\text{Me}_2\text{S})_4](\text{CF}_3\text{SO}_3)_2$  ( $\text{Me}_2\text{S}$  = dimethyl sulfide).* Freshly precipitated platinum(II) hydroxide, prepared as described previously,<sup>19,20</sup> was dissolved directly on the filter by addition of 1.25 ml of a cooled solution obtained by mixing 2 ml  $\text{Me}_2\text{S}$  (Merck, for synthesis) and 1 ml  $\text{CF}_3\text{SO}_3\text{H}$  (Fluka, purum) and stirring. Crystallization started immediately. The precipitate was washed twice with 1 ml ice-cold water, then with ethanol and acetone. The white crystals were dissolved in 10 ml hot water, and the

\* On leave from the Department of Chemistry, Svetozar Marcovic University, P.O. Box 60, Yu-340 00 Kragujevac, Yugoslavia.

† To whom correspondence should be addressed.

Table 1. Crystal data, collection and reduction of intensity data, and least-squares refinement.

	[Pt(Me <sub>2</sub> S) <sub>4</sub> ](CF <sub>3</sub> SO <sub>3</sub> ) <sub>2</sub>	[Pt(tx) <sub>4</sub> ](CF <sub>3</sub> SO <sub>3</sub> ) <sub>2</sub> · H <sub>2</sub> O
Mol. wt.	741.74	927.93
Space group	P2 <sub>1</sub> /c	C2/c
a/Å	8.5630(8)	14.438(2)
b/Å	8.692(1)	13.016(2)
c/Å	16.360(3)	21.696(3)
β/°	97.064(9)	126.32(1)
V/Å <sup>3</sup>	1208.4(2)	3285.0(6)
Z	2	4
D/g cm <sup>-3</sup>	2.038	1.876
Crystal size/mm	0.14 × 0.38 × 0.10	0.08 × 0.17 × 0.10
Radiation(graphite monochromatized)	Mo K <sub>α</sub>	Mo K <sub>α</sub>
θ-interval/°	3–28	3–30
ω–2θ scan width, Δω/°	0.7 + 0.5 tan θ	0.8 + 0.5 tan θ
Maximum recording time/s	180	180
μ/cm <sup>-1</sup>	64.3	47.6
Range of transmission factor	0.38–0.58	0.57–0.74
No. of reflections measured	2891	4945
No. of reflections used in the final refinements [ <i>I</i> > 3σ( <i>I</i> )], <i>m</i>	1922	2268
No. of parameters refined, <i>n</i>	153	197
C1 in weighting function <sup>a</sup>	0.020	0.035
C2 in weighting function <sup>a</sup>	0.75	15.0
$R = \sum   F_o  -  F_c   / \sum  F_o $	0.025	0.035
$R_w = [\sum w( F_o  -  F_c )^2 / \sum w F_o ^2]^{1/2}$	0.033	0.050
$S = [\sum w( F_o  -  F_c )^2 / (m - n)]^{1/2}$	0.98	0.84
δ <i>R</i> – plot, slope	1.12	1.36
δ <i>R</i> – plot, intercept	–0.04	–0.05

$$^a w^{-1} = \sigma/4|F_o|^2 + (C1|F_o|)^2 + C2.$$

colourless solution was allowed to cool in an open evaporation dish. White single crystals of [Pt(Me<sub>2</sub>S)<sub>4</sub>](CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub> precipitated. These were stable at room temperature (m.p. 229–233 °C).

**Synthesis of [Pt(tx)<sub>4</sub>](CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub> · H<sub>2</sub>O (tx = 1,4-thioxane).** The platinum(II) hydroxide on the filter was dissolved in 1.25 ml of a cooled solution obtained by mixing 2 ml 1,4-thioxane (Lancaster synthesis Ltd.) and 1 ml CF<sub>3</sub>SO<sub>3</sub>H (Fluka, purum) and stirring for a few minutes. As before, the precipitate was washed twice with 1 ml ice-cold water, then with ethanol and acetone. The white crystals were dissolved in 10 ml nitromethane (Fluka, purum). The solution was filtered and slowly evaporated at room temperature. Single crystals of [Pt(tx)<sub>4</sub>](CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub> · H<sub>2</sub>O, stable at room temperature, precipitated (m.p. 230–232 °C).

**Structure determination.** An Enraf-Nonius CAD-4-four-circle single-crystal diffractometer was used for all X-ray diffraction measurements. Information on the collection and reduction of the data is given in Table 1. The structures were solved by standard Patterson and difference Fourier methods and refined by full-matrix least-squares calculations employing programs amended by Lundgren.<sup>21</sup> Three standard reflections, recorded at regular intervals, showed a small (< 4 %) decrease in intensities in both data collections. The intensity data sets were therefore rescaled with the fitted linear equations. The quantities *I* and σ(*I*) were

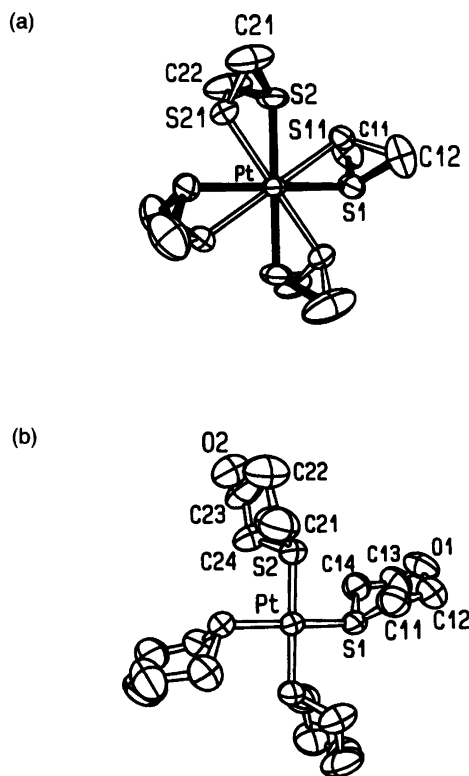


Fig. 1. Views and numbering schemes of [Pt(Me<sub>2</sub>S)<sub>4</sub>]<sup>2+</sup> (a) and [Pt(1,4-thioxane)<sub>4</sub>]<sup>2+</sup> (b).

corrected for Lorentz, polarization and absorption effects, the latter calculated by numerical integration. Scattering factors with corrections for anomalous dispersion were taken from Ref. 22.

In the  $[\text{Pt}(\text{Me}_2\text{S})_4](\text{CF}_3\text{SO}_3)_2$  structure the two unique  $\text{Me}_2\text{S}$  groups are disordered. The sulfur atom occupies two different positions, whereas the methyl carbon atoms almost coincide and could not be resolved, cf. Fig. 1. All non-hydrogen atoms were refined with anisotropic temperature factors in the final refinement, which did not include the hydrogen atoms, owing to the fact that they were not found in the difference map. The occupancy factors for the sulfur atoms were refined to 0.588(3) for S1 and S2 and to

0.412(3) for S11 and S21. An isotropic extinction correction was made with the coefficient  $g = 0.51(3) \times 10^4$ , corresponding to a mosaic spread of  $11.4''$ . The maximum correction on  $F_o(1\ 0\ 2)$  was 1.31.

The  $[\text{Pt}(\text{tx})_4](\text{CF}_3\text{SO}_3)_2 \cdot \text{H}_2\text{O}$  structure was first refined assuming no water content.  $\Delta Q$  maps showed two residual peaks of  $\sim 3 \text{ e } \text{\AA}^{-3}$  located on the two-fold axis  $(0, y, 1/4)$ . IR spectra and elementary analysis proved the structure to be a hydrate, and the peaks were therefore assigned to oxygen atoms of a disordered water molecule and were refined with half-occupied positions. The final refinement was performed with anisotropic thermal parameters for the non-hydrogen atoms and with the hydrogen atoms at calcu-

Table 2. Atomic coordinates and equivalent isotropic temperature factor coefficients with standard deviations.

Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	$U_{\text{eq}}/\text{\AA}^2$ <sup>a</sup>
$[\text{Pt}(\text{Me}_2\text{S})_4](\text{CF}_3\text{SO}_3)_2$				
Pt	0.00	0.00	0.00	0.0396(1)
S1	0.17893(33)	-0.19756(30)	0.03145(19)	0.0593(9)
S11	0.23368(41)	-0.10809(44)	-0.03029(23)	0.0529(11)
S2	0.12250(34)	0.06828(35)	-0.11340(16)	0.0580(9)
S21	-0.00611(48)	0.15801(55)	-0.11474(23)	0.0638(14)
C11	0.1916(10)	-0.3088(10)	-0.0567(6)	0.0935(35)
C12	0.3696(9)	-0.1227(11)	0.0545(6)	0.0940(36)
C21	0.1684(10)	0.2641(10)	-0.1122(4)	0.0826(29)
C22	-0.0133(13)	0.0511(13)	-0.2024(4)	0.1126(41)
S3	0.29872(21)	0.17035(21)	0.62985(10)	0.0617(5)
O31	0.1826(9)	0.0646(10)	0.6005(6)	0.1409(37)
O32	0.3830(9)	0.2377(11)	0.5722(4)	0.1385(37)
O33	0.2469(9)	0.2719(8)	0.6875(4)	0.1218(31)
C3	0.4418(10)	0.0535(11)	0.6917(5)	0.0783(28)
F31	0.5009(9)	-0.0500(9)	0.6474(6)	0.1468(35)
F32	0.3851(11)	-0.0167(10)	0.7489(6)	0.2006(50)
F33	0.5616(8)	0.1311(9)	0.7226(5)	0.1641(37)
$[\text{Pt}(\text{tx})_4](\text{CF}_3\text{SO}_3)_2 \cdot \text{H}_2\text{O}$				
Pt	0.00	0.00	0.00	0.0356(2)
S1	0.11176(16)	0.00806(22)	-0.04443(12)	0.0391(8)
S2	-0.02420(19)	0.17675(16)	-0.01170(17)	0.0464(17)
O1	0.2382(7)	0.1724(5)	-0.0735(4)	0.056(4)
O2	-0.1464(7)	0.3476(5)	0.0155(5)	0.063(5)
C11	0.2387(8)	0.0822(7)	0.0262(5)	0.047(4)
C12	0.3036(9)	0.1082(8)	-0.0085(7)	0.059(6)
C13	0.1469(10)	0.1182(9)	-0.1347(6)	0.059(6)
C14	0.0519(8)	0.0963(7)	-0.1238(6)	0.048(5)
C21	0.0207(10)	0.2333(8)	0.0790(7)	0.065(6)
C22	-0.0241(11)	0.3426(8)	0.0639(7)	0.068(7)
C23	-0.1937(11)	0.3180(9)	-0.0616(7)	0.067(7)
C24	-0.1763(8)	0.2039(7)	-0.0667(6)	0.051(5)
S3	0.27822(27)	0.01102(37)	0.24501(20)	0.0806(16)
O31	0.1744(10)	0.0390(15)	0.1848(13)	0.274(15)
O32	0.2999(17)	-0.0124(12)	0.3173(11)	0.178(15)
O33	0.3307(23)	-0.0621(15)	0.2305(12)	0.250(22)
C3	0.3638(20)	0.1199(19)	0.2681(10)	0.125(13)
F31	0.4609(12)	0.1093(16)	0.3252(10)	0.273(13)
F32	0.3526(13)	0.1593(11)	0.2097(8)	0.191(12)
F33	0.3241(26)	0.1963(12)	0.2882(12)	0.288(27)
OW1	0.00	0.1098(22)	0.25	0.116(26)
OW2	0.00	0.3051(23)	0.25	0.105(23)

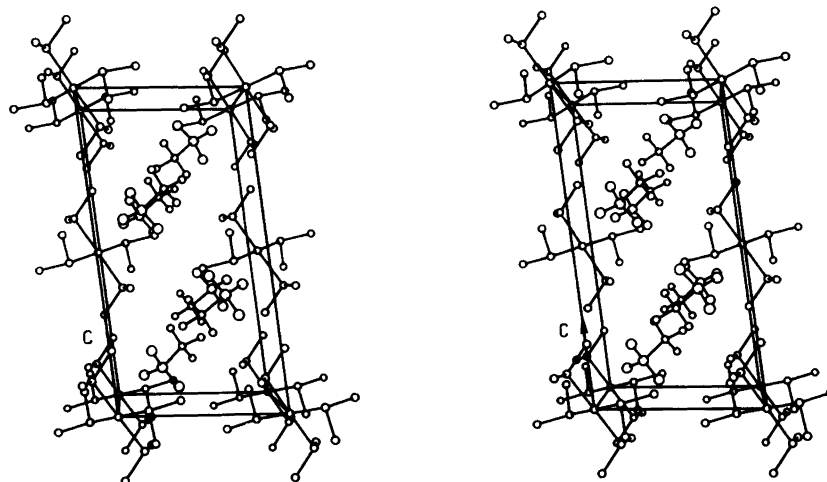
<sup>a</sup> $U_{\text{eq}} = (1/3) \sum \sum U_{ij} a_i^* a_j^* a_i \cdot a_j$ .

lated positions ( $C-H = 1.00 \text{ \AA}$ ) with  $U_{iso}$  fixed at  $0.08 \text{ \AA}^2$ . No extinction correction was needed. A final  $\Delta\rho$  map gave a maximum value of  $1.1 \text{ e \AA}^{-3}$  on the twofold axis between the water molecules, indicating possible further disorder. Cell dimensions were determined by least-squares methods from the  $\theta$  angles of 75 and 50 reflections for  $[Pt(tx)_4](CF_3SO_3)_2 \cdot H_2O$  and  $[Pt(Me_2S)_4](CF_3SO_3)_2$ , respectively. The final positional and thermal parameters are given in Table 2. Lists of observed and calculated structure factors are available from one of the authors (B.N.).

## Results and discussion

Both compounds contain discrete complexes  $[Pt(Me_2S)_4]^{2+}$  and  $[Pt(tx)_4]^{2+}$ , respectively, with four thioether sulfurs equatorially bonded to the platinum in a square-planar arrangement. The shortest Pt–Pt distance is  $8.563(1) \text{ \AA}$  in the dimethyl sulfide compound and  $9.719(1) \text{ \AA}$  in the 1,4-thioxane compound. The structures of the two cations are shown in Fig. 1 and packing diagrams of the unit cell contents in Fig. 2. Selected interatomic distances and angles

(a)



(b)

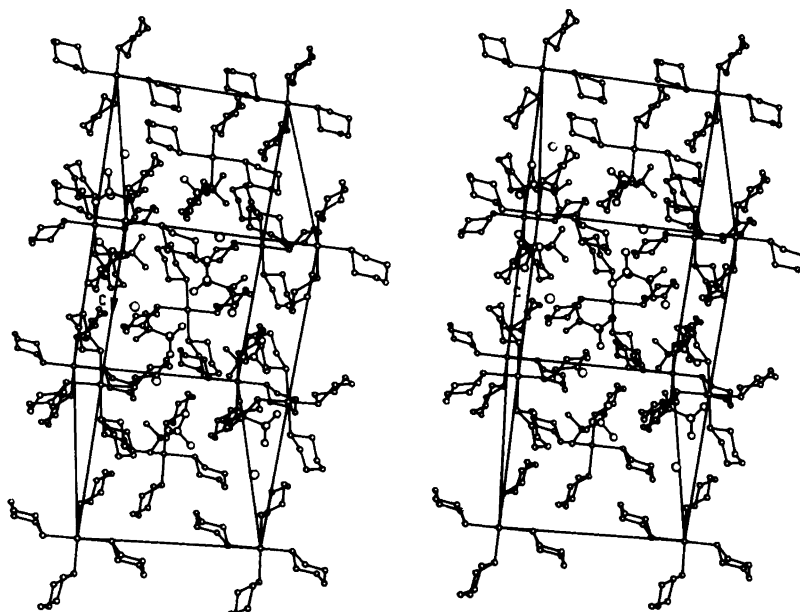


Fig. 2. Packing diagrams of  $[Pt(Me_2S)_4](CF_3SO_3)_2$  (a) and  $[Pt(1,4-thioxane)_4](CF_3SO_3) \cdot H_2O$  (b). For clarity, the atoms of the  $SO_3$  group in  $CF_3SO_3^-$  are enlarged. Single atoms correspond to hydrate oxygens.

Table 3. Selected interatomic distances (in Å) and angles (in °), with estimated standard deviations.

[Pt(Me <sub>2</sub> S) <sub>4</sub> ](CF <sub>3</sub> SO <sub>3</sub> ) <sub>2</sub>			
Pt–S1	2.317(3)	S1–Pt–S2	90.9(1)
Pt–S2	2.318(3)		
Pt–S11	2.319(4)	S11–Pt–S21	90.1(1)
Pt–S21	2.321(4)		
S1–C11	1.75(1)	C11–S11–C12	103.4(5)
S1–C12	1.76(1)		
S11–C11	1.82(1)	C11–S11–C12	102.6(5)
S11–C12	1.70(1)		
S2–C21	1.75(1)	C21–S2–C22	102.5(5)
S2–C22	1.75(1)		
S21–C21	1.75(1)	C21–S21–C22	104.4(5)
S21–C22	1.70(1)		
[Pt(tx) <sub>4</sub> ](CF <sub>3</sub> SO <sub>3</sub> ) <sub>2</sub> · H <sub>2</sub> O			
Pt–S1	2.321(2)	S1–Pt–S2	90.4(1)
Pt–S2	2.318(2)		
S1–C11	1.82(1)	C14–S1–C11	98.1(4)
C11–C12	1.55(1)	S1–C11–C12	107.8(7)
C12–O1	1.41(1)	C11–C12–O1	112.1(8)
O1–C13	1.39(1)	C12–O1–C13	110.5(8)
C13–C14	1.55(2)	O1–C13–C14	111.9(9)
C14–S1	1.81(1)	C13–C14–S1	107.2(7)
S2–C21	1.82(1)	C24–S2–C21	98.6(5)
C21–C22	1.52(2)	S2–C21–C22	108.3(8)
C22–O2	1.42(2)	C21–C22–O2	112.7(9)
O2–C23	1.44(1)	C22–O2–C23	111.9(9)
C23–C24	1.52(1)	O2–C23–C24	111.2(9)
C24–S2	1.81(1)	C23–C24–S2	109.3(7)

for the complexes are given in Table 3. Bond distances and angles within the trifluoromethanesulfonate ions are normal in both compounds.<sup>23,24</sup> Also, the geometries of the dimethyl sulfide and 1,4-thioxane molecules agree with those found previously when these thioether ligands are coordinated to Pd(II) or Pt(II).<sup>12,18,25–27</sup>

The tetrakis(dimethyl sulfoxide) complexes of platinum (II) and palladium(II) are *cis*-isomers with O-bonded ligands *trans* to the S-bonded ones.<sup>28,29</sup> Thioxane is also expected to be capable of ambidentate coordination in a similar arrangement. In the present compound, however, all four ligands are coordinated via the thioether sulfur. As concluded from stability constants and NMR spectra, the coordination is similar in solution.<sup>6,30</sup>

The Pt–S distances in the two complexes are equal within experimental errors, varying between 2.317(3) and 2.321(4) Å. Thus packing effects in the case of the more bulky thioxane do not influence the coordination geometry. The thioxane ligands have a chair conformation and are coordinated in a propeller-like arrangement to minimize steric repulsions within the coordination sphere. The least-squares plane through the four carbon atoms makes an angle of 84° with the coordination plane. The Pt–S distances are all similar and almost the same as those observed in *trans*-[Pd(tx)<sub>2</sub>Cl<sub>2</sub>], where the Pd–S distance is 2.314(3) Å.<sup>27</sup>

The distribution of bond lengths Pt–L in a series of square-planar complexes, [PtLXYZ], is affected by intramolecular forces (*cis*- and *trans*-influence and steric hindrance within the complex) and intermolecular forces, in addition to the experimental errors in the bond lengths. In a complex [PtL<sub>4</sub>] the *cis*- and *trans*-influence operates on each of the four Pt–L bonds to exactly the same extent. Therefore, the distribution, calculated as  $\sigma = [\sum_n (d_n - \bar{d})^2 / (n-1)]^{1/2}$ , of the six Pt–S distances in the two investigated compounds, 0.002 Å, is only affected by experimental errors and packing forces. Obviously, the effect on the bond lengths of differences in packing is small and of the same order of magnitude as the random errors. The distribution of Pt–S distances in compounds of the type *trans*-[PtS<sub>2</sub>XY] (S = thioether sulfur), on the other hand, is as large as 0.013 Å (nine distances),<sup>3</sup> which is a measure of the span of the *cis*-influence on the Pt–S bond length. In the same way the difference in distribution of the Pt–S bond length in compounds of the type [PtSXYZ] (26 distances) and *trans*-[PtS<sub>2</sub>XY], 0.042–0.013 = 0.029 Å,<sup>3</sup> is a measure of the span of the *trans*-influence on the Pt–S bond length. As expected, the following order of importance of the factors influencing the Pt–S bond length is thus obtained: experimental errors and packing forces < *cis*-influence < *trans*-influence. However, even if the *cis*-influence is smaller than the *trans*-influence, it is certainly not negligible. Therefore, previous definitions of the *trans*-influence<sup>2,3</sup> can be generalized to include the *cis*-influence also: *The cis- and trans-influence of X, Y, Z on the bond length M–L in a square-planar complex [MLXYZ] is the shortening or lengthening of the M–L distance as compared to the distance M–L in [ML<sub>4</sub>].* The average value of the Pt–S distance, 2.319(2) Å, obtained for the two structures reported here, may thus be used as a reference distance to calculate *cis*- and *trans*-influences by other donor atoms on the Pt–S bond.

It is possible to discriminate between the *cis*- and *trans*-influence in certain cases. In *trans*-[Pt(THT)<sub>2</sub>I<sub>2</sub>] (THT = tetrahydrothiophene) the Pt–S distances are 2.309(1) and 2.310(1) Å,<sup>3</sup> while in *trans*-[Pt(tx)<sub>2</sub>Br<sub>2</sub>] the Pt–S distance is 2.281(3) Å.<sup>18</sup> The *cis*-influence of two iodides is thus 2.310–2.319 = –0.009 Å, while the *cis*-influence of two bromides is even more negative, 2.281 – 2.319 = –0.038 Å. For estimations of the *trans*-influence, data on complexes of the type [PtS<sub>3</sub>X] are needed. Such work is in progress.

The crystal structures of a few other square-planar complexes in which platinum is coordinated to four sulfur atoms have been described previously.<sup>31–35</sup> In these cases, the sulfur-bonded ligands are of various types. The following mean values of the Pt–S distances are found: 2.313(9) Å for tetra(thiourea)platinum(II) chloride;<sup>31</sup> 2.321(6) Å for tetrakis(1-methyl-4-imidazoline-2-thione)platinum(II) dichloride dihydrate;<sup>32</sup> 2.323(9) Å for [Pt(tep)<sub>2</sub>][Pt(SCN)<sub>4</sub>] · 2DMF (tep = diphenyl(*o*-phenyltellurenyl)phosphine, DMF = dimethylformamide);<sup>33</sup> 2.317(7) Å for tetrakis(dithioacetato)diplatinum(II);<sup>34</sup> and 2.324(13) Å for linear-chain (μ-diido)tetrakis(dithioacetato)diplatinum.<sup>35</sup> The deviations

of these Pt–S distances from the mean value of the present investigation, 2.319(2) Å, are surprisingly small. Evidently, the nature of the sulfur ligands has a rather small influence on the Pt–S distances in square-planar tetrakis complexes whether these are monomeric or polymeric.

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