uncoupled. The Mo—Mo bond distance is 2·906 (1) Å, shorter by 0·33 Å than in analogous $(\eta^5\text{-}C_5H_5)_2Mo_2(CO)_6$ complexes (Adams, Collins & Cotton, 1974) and 0·05–0·08 Å shorter than in alkyne complexes of the latter (Bailey, Chisholm, Cotton & Rankel, 1978). These trends compare favorably with the contracted W—W distance of 2·987 Å in $(\eta^5\text{-}C_5H_5)_2W_2(CO)_4C_2H_2$ (Ginley, Bock, Wrighton, Fischer, Tipton & Bau, 1978) and the W—W distance of 3·347 Å of an alkyne-deficient complex, hexacarbonyl-μ-(1–5:1′–5′-η-fulvalene)-ditungsten (Abrahamson & Heeg, 1985).

The bridging alkyne C—C distance of 1·374 (4) Å and the singly bound alkyne C—C distance of 1·265 (4) Å are consistent with 4e$^-$- and 2e$^-$-donating ligands. This is also supported by the similarity between the average terminal and average bridging Mo—C(alkyne) distances. Interestingly, the bridging alkyne ligand is not symmetrically oriented with respect to the dinuclear framework; it is displaced slightly towards the singly coordinating alkyne, in the more sterically hindered direction.

The crystal structure analysis was performed by the authors at the UC Berkeley X-ray Diffraction Facility (CHEXRAY) under the supervision of Dr F. J. Hollander, Professor K. Raymond, Professor S. H. Kim and Mr R. Scarrow as part of a requirement for Chemistry 295.

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cis-Dichlorobis(tetrahydrothiophene 1-oxide)platinum(II), $[PtCl_2(C_4H_8OS)_2]$

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Abstract. $M_r = 474{\cdot}34$, monoclinic, $P2_1/c$, $a = 11{\cdot}273$ (5), $b = 19{\cdot}548$ (7), $c = 9{\cdot}885$ (4) Å, $\beta = 142{\cdot}61$ (2)°, $V = 1322{\cdot}9$ (9) Å^3, $Z = 4$, $D_x = 2{\cdot}381$, $D_m = 2{\cdot}38$ (1) Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 11{\cdot}407$ mm^{-1}, $F(000) = 896$, $T = 295$ K, $R = 0{\cdot}034$ for 2547 unique observed reflections. Pt has *cis* square-planar coordination to two Cl and to two S atoms of the tetrahydrothiophene 1-oxide ligands. The bond distances are Pt—Cl = 2·309 (3), 2·312 (3) Å and Pt—S = 2·228 (2), 2·248 (3) Å. The packing consists of layers of molecules parallel to the *ab* plane and centered at $z = 0$.

Introduction. For several years, we have been involved in a study of Pt–Me_2SO compounds. The aqueous reaction of K_2PtCl_4 with Me_2SO gives *cis*-$[PtCl_2(Me_2SO)_2]$ (Melanson & Rochon, 1975). The corresponding Pd^{II} complex is the *trans* isomer (Bennett, Cotton, Weaver, Williams & Watson, 1967). With other sulfoxide ligands (L), IR data have shown that the $[PdCl_2L_2]$ complexes have the *trans* S-bonded configuration. In the corresponding Pt^{II} compounds, IR data support a *cis* configuration except when L is a very bulky ligand like bis(3-methylbutyl) sulfoxide (Price, Williamson, Schramm & Wayland, 1972). The $(d\text{–}d)\pi$

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bonding is probably more effective in the Pt complexes and *cis* structures are obtained. In the Pd compounds the enhanced $(d\text{–}d)\pi$ bonding in the *cis* configuration is probably insufficient to overcome the repulsions between the two sulfoxide ligands and all the complexes have the *trans* geometry.

The preference of Pt^{II} and Pd^{II} for the S donor site is shown by the fact that all neutral complexes $[MX_2L_2]$ are exclusively S-bonded in the solid state. The cationic complexes of the type $[ML_4]X_2$ ($X = BF_6^-$ and ClO_4^-) where steric influences are important, are believed to contain both S- and O-bonded ligands in a *cis* configuration in the solid state (Price *et al.*, 1972).

Recently, we have started a study on the reactions of Pt compounds with sulfoxide ligands other than Me_2SO. We have synthesized as starting materials several compounds of the type $[PtCl_2L_2]$. In order to characterize well all the compounds involved in these reactions, we have decided to determine the crystal structure of a few of these $[PtCl_2L_2]$ compounds. Up to now, only $[PtCl_2(TMSO)_2]$ (TMSO = tetrahydrothiophene 1-oxide) gave suitable crystals for X-ray diffraction. Its structure is reported below.

Experimental. *Synthesis of cis-*$[PtCl_2(TMSO)_2]$. 2·2 mmol of TMSO (bought from Aldrich) (10% excess) was added to 1 mmol of K_2PtCl_4 dissolved in water. The mixture was stirred at room temperature for 18 h. The white precipitate was filtered, washed several times with hot water and dried under vacuum. Yield: 90%, m.p. 467 K. Elemental analysis: calc. % C: 20·26, % H: 3·40; exp. % C: 20·33, % H: 3·39.

The crystals used for the structure determination were obtained by slow evaporation of the aqueous filtrate obtained in the method described above.

Rectangular plate, dimensions (mm): 0·240 ($100\text{–}\bar{1}00$) × 0·164 ($12\bar{1}\text{–}\bar{1}\bar{2}1$) × 0·124 ($1\bar{2}\bar{1}\text{–}\bar{1}21$); density by flotation in a $CCl_4\text{–}CH_2Br_2$ solution; precession photographs showed $P2_1/c$ space group; Syntex $P\bar{1}$ diffractometer; graphite-monochromatized Mo $K\alpha$ radiation; cell parameters from refined angles of 15 centered reflections; 3866 independent reflections measured up to $2\theta < 60°$ by $\theta\text{–}2\theta$ scan technique; range of *hkl*: $h\, 0\rightarrow 15$, $k\, 0\rightarrow 27$, $l\, -9\rightarrow 8$; standard reflections 300, 042 and 002; variations <3%; reflections with $I_{net} < 2·5\sigma(I)$ unobserved, $\sigma(I)$ calculated as in Melanson & Rochon (1975); absorption correction based on equations of crystal faces, transmission factors from 0·197 to 0·295; data corrected for Lorentz and polarization effects; 2547 unique observed reflections; atomic scattering factors of Cromer & Waber (1965) for Pt, Cl, S, O, C and of Stewart, Davidson & Simpson (1965) for H; anomalous-dispersion terms of Pt, Cl and S from Cromer (1965).

Patterson map showed position of Pt; other atoms (except H) located by structure factors and Fourier-map calculations; isotropic secondary-extinction correction (Coppens & Hamilton, 1970); $w = 1/\sigma^2(F)$; H atoms fixed at calculated positions (C—H = 0·95 Å) with isotropic $B = 6·0$ Å^2. Ratio of maximum least-squares shift to e.s.d. in final refinement cycle (on F): <0·45; $\rho_{max} = 0·66$ (close to Pt), $\rho_{min} = -0·40$ e Å^{-3} in final difference Fourier synthesis. $R = 0·034$, $wR = 0·030$, calculations on a Cyber 171 with programs of Melanson & Rochon (1975).[*]

Discussion. The refined atomic parameters are listed in Table 1. A labeled diagram of the molecule is shown in Fig. 1. The coordination around the Pt atom is square planar. The deviations from the weighted coordination plane are: Pt, −0·0009 (3); Cl(1), 0·001 (2); Cl(2), 0·030 (2); S(1) 0·022 (2); S(2), 0·001 (2) Å. The bond lengths and angles are given in Table 2. The angles around the Pt atom are close to the expected values of 90 and 180°.

Table 1. *Positional parameters* (× 10^4) *with their e.s.d.'s and equivalent isotropic temperature factors* (× 10^4)

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
Pt	−114·0 (3)	1204·8 (1)	1981·8 (4)	177
Cl(1)	−2792 (2)	1896 (1)	−37 (3)	275
Cl(2)	−1431 (3)	409 (1)	2255 (3)	291
S(1)	1123 (2)	1969 (1)	1636 (3)	186
S(2)	2391 (2)	495 (1)	3940 (3)	176
O(1)	949 (8)	1773 (2)	58 (8)	341
O(2)	3836 (8)	709 (2)	4254 (10)	184
C(1)	222 (10)	2829 (3)	1053 (12)	197
C(2)	1956 (12)	3277 (3)	2302 (14)	225
C(3)	3681 (11)	2966 (4)	4572 (13)	364
C(4)	3653 (10)	2202 (4)	4262 (12)	281
C(5)	1692 (10)	−379 (3)	2961 (12)	303
C(6)	2545 (12)	−810 (3)	4857 (14)	249
C(7)	4281 (11)	−436 (3)	7038 (13)	193
C(8)	3600 (10)	291 (3)	6588 (11)	221

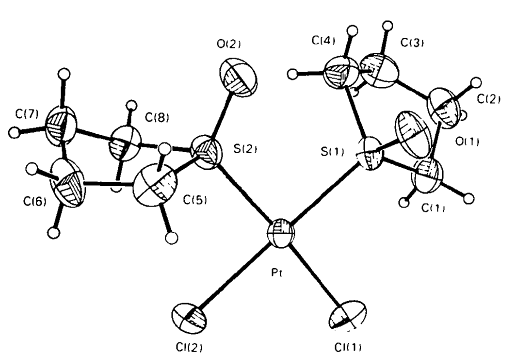

Fig. 1. Labeled diagram of the *cis*-$[PtCl_2(TMSO)_2]$ molecule. The ellipsoids correspond to 50% probability.

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[*] Lists of structure factors, anisotropic thermal parameters, H coordinates, deviations from best planes and equations of weighted least-squares planes, and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42272 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

As expected from IR spectroscopy the compound is the *cis* isomer. The IR spectrum of the compound showed two stretching Pt—Cl vibrations at 336 and 320 cm^{-1} which is typical of *cis* isomers. The Pt—Cl bonds (2·312 and 2·309 Å) are of normal lengths.

The sulfoxide ligands are bonded to Pt through their S atom. The Pt—S bond lengths [2·228 (2) and 2·248 (3) Å, the standard deviations are probably underestimated] are normal and agree well with the values found in Pt–Me_2SO compounds (Melanson & Rochon, 1975, 1977, 1978*a,b,* 1984; Melanson, Hubert & Rochon, 1976) and in a Pt–diisopropyl sulfoxide compound (Lock, Speranzini & Powell, 1976). The S atoms in the sulfoxide molecules are in approximate tetrahedral environment. The Pt—S—O angles (114·5 and 115·9°) are larger than the tetrahedral values as observed in the Pt–sulfoxide structures described above. The Pt—S—C angles are larger than the tetrahedral value in *cis*-$[PtCl_2(TMSO)_2]$ (112·1 to 114·3°) whereas these angles were normal in the above structures. This deformation is probably caused by a considerable strain inside the five-membered ring. This strain is confirmed by the small values of the C—S—C angles (94·0 and 94·3°). The S—O, S—C and C—C bond distances agree well with published values.

The weighted least-squares plane was calculated through each five-membered ring. The atom-to-plane distances are S(1) 0·005 (3), C(1) −0·22 (1), C(2) 0·35 (2), C(3) −0·18 (2), C(4) 0·01 (1) Å for the first ring and S(2) −0·009 (3), C(5) 0·04 (1), C(6) 0·19 (2), C(7) −0·34 (2) and C(8) 0·24 (1) Å for the second ring. The first plane makes an angle of 51° with the Pt plane while the equivalent angle with the second ligand is 96°. The torsion angles have been calculated and the table is part of the supplementary material.* Both TMSO rings in the complex are clearly in an envelope conformation (Figs. 1 and 2). The best least-squares planes were calculated through four atoms in each ring. The deviations are shown in Table 3 (also deposited).* C(2) and C(7) are clearly out of the plane by 0·64 Å and on the same side as O(1) and O(2) respectively. Therefore there are short '1–3 interactions' between O(1) and H(21) of 3·11 Å in ring 1 and between O(2) and H(72) of 3·07 Å in ring 2.

The packing of the molecules is shown on Fig. 2. It consists of layers of molecules parallel to the *ab* plane and centered at $z = 0$. No strong hydrogen bonds are expected in this compound. But there are a few intermolecular distances shorter than van der Waals contacts. The C(4)···Cl(1) distance is 3·51 (1) Å [calculated H(42)···Cl(1) = 2·71 Å] and the C(7)···O(2) distance is 3·26 (1) Å [calculated H(72)···O(2) = 2·49 Å]. The angles C(4)—H(42)···Cl(1) = 141·8 (6) and C(7)—H(72)···O(2) = 140·0 (7)°.

Table 2. *Bond distances* (Å) *and angles* (°)

Pt—Cl(1)	2·312 (3)	S(2)—C(5)	1·806 (7)
Pt—Cl(2)	2·309 (3)	S(2)—C(8)	1·789 (8)
Pt—S(1)	2·248 (3)	C(1)—C(2)	1·51 (2)
Pt—S(2)	2·228 (2)	C(2)—C(3)	1·50 (1)
S(1)—O(1)	1·460 (9)	C(3)—C(4)	1·52 (1)
S(2)—O(2)	1·456 (11)	C(5)—C(6)	1·51 (1)
S(1)—C(1)	1·805 (8)	C(6)—C(7)	1·52 (1)
S(1)—C(4)	1·820 (9)	C(7)—C(8)	1·51 (1)
Cl(1)—Pt—Cl(2)	88·5 (1)	O(1)—S(1)—C(4)	108·5 (5)
Cl(1)—Pt—S(1)	91·3 (1)	C(1)—S(1)—C(4)	94·0 (5)
Cl(1)—Pt—S(2)	176·7 (1)	O(2)—S(2)—C(5)	109·0 (5)
Cl(2)—Pt—S(1)	178·6 (1)	O(2)—S(2)—C(8)	109·2 (5)
Cl(2)—Pt—S(2)	88·2 (1)	C(5)—S(2)—C(8)	94·3 (5)
S(1)—Pt—S(2)	92·0 (1)	S(1)—C(1)—C(2)	104·2 (7)
Pt—S(1)—O(1)	114·5 (3)	S(1)—C(4)—C(3)	106·9 (7)
Pt—S(1)—C(1)	114·0 (3)	S(2)—C(5)—C(6)	106·6 (7)
Pt—S(1)—C(4)	114·0 (3)	S(2)—C(8)—C(7)	105·0 (7)
Pt—S(2)—O(2)	115·9 (3)	C(1)—C(2)—C(3)	106·6 (9)
Pt—S(2)—C(5)	114·3 (3)	C(2)—C(3)—C(4)	107·6 (9)
Pt—S(2)—C(8)	112·1 (3)	C(5)—C(6)—C(7)	109·0 (9)
O(1)—S(1)—C(1)	109·8 (5)	C(6)—C(7)—C(8)	104·8 (9)

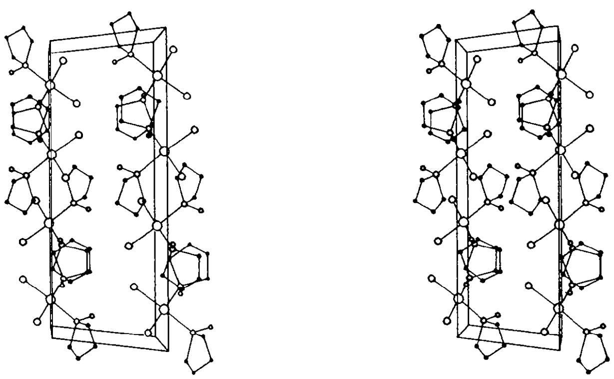

Fig. 2. Stereoscopic diagram of the packing in the *cis*-$[PtCl_2(TMSO)_2]$ crystal (*b* axis vertical, view down *a* axis).

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* See previous footnote.

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Structure of Copper Cimetidine Nitrate

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Abstract. [*N*-Cyano-*N*′-methyl-*N*″-(2-{[(5-methyl-1*H*-imidazol-4-yl)methyl]thio}ethyl)guanidine]copper(II) nitrate, $[Cu(NO_3)_2(C_{10}H_{16}N_6S)_2]$, $M_r = 692{\cdot}22$, monoclinic, $P2_1/n$, $a = 11{\cdot}597$ (3), $b = 10{\cdot}555$ (1), $c = 12{\cdot}256$ (1) Å, $\beta = 97{\cdot}07$ (1)°, $V = 1488{\cdot}8$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}54$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}927$ mm^{-1}, $F(000) = 718$, $T = 295 \pm 1$ K, $R = 0{\cdot}024$ for 2215 observed diffractometer data. The Cu^{2+} ions lie at symmetry centres and are six-coordinated with a distorted octahedral geometry. The structure is characterized by infinite polymeric chains along [010], constructed from [Cu(cimetidine)$_2$] entities which are linked through *N*-cyano groups belonging to symmetry-related molecules. The interchain packing is assured by hydrogen bonds between O atoms from nitrate groups and N atoms from cimetidine.

Introduction. Cimetidine (CM) is an imidazole derivative used as an antihistamine H_2 receptor drug in the treatment of peptic ulcer. The X-ray structural study of the compound in several polymorphic crystalline forms has already been carried out by many authors (Hädicke, Frickel & Franke, 1978; Shibata, Kokubo, Morimoto, Morisaka, Ishida & Inoue, 1983; Párkányi, Kálmán, Hegedüs, Harsányi & Kreidl, 1984).

The structure determination of the title compound was undertaken as part of a continuing research project developed in the Department of Inorganic Chemistry involving the synthesis, structure and properties of metal complexes of cimetidine (Sancho, Borrás, Soto-Tuero, Esteban-Calderón, Martínez-Ripoll & García-Blanco, 1985; Sancho, Soto & Borrás, 1985). Also, it was of particular interest to confirm the molecular structure of this complex previously established by spectroscopic techniques (Soto, Sancho & Borrás, 1985).

Experimental. Brown crystals were obtained from a mixture of aqueous solutions of $Cu(NO_3)_2.3H_2O$ and cimetidine. Crystal 0·3 × 0·3 × 0·3 mm. Enraf–Nonius CAD-4 diffractometer. Lattice parameters from least-squares refinement of 25 medium-angle reflections. Lorentz and polarization corrections applied. No correction for absorption or extinction. Variation of standard reflections ±0·46%. $2° \leq 2\theta \leq 50°$. ω–2θ scan. 2915 measured reflections ($-13 \leq h \leq 13$; $0 \leq k \leq 12$; $0 \leq l \leq 14$), 2215 observed [$I > 2{\cdot}5\sigma(I)$]. Structure solved by direct methods (*MULTAN*11/84: Main, Germain & Woolfson, 1984), using the 262 highest E values; 17 non-H atoms found in first E map. Remaining non-H atoms were located from a subsequent Fourier synthesis. Full-matrix least-squares refinement of the model with isotropic temperature factors minimizing $\sum w(|F_o| - |F_c|)^2$ gave $R = 0{\cdot}095$; $w = 1/\sigma^2(F)$. Inclusion of anisotropic temperature factors reduced R to 0·044. At this stage all H atoms were located from a difference map. R reduced to 0·024 ($wR = 0{\cdot}031$) (245 variables) when refinement was carried out with inclusion of H atoms. All H atoms were given an isotropic temperature factor (U) of 0·05 Å^2, and this overall value was 0·0548 Å^2 in the last cycle. Max. and min. values in final difference density maps 0·32 and −0·25 e Å^{-3} respectively. $(\Delta/\sigma)_{max} = 0{\cdot}07$, $S = 1{\cdot}35$. Atomic scattering factors and corrections for anomalous dispersion taken from *International Tables for X-ray Crystallography* (1974). Calculations performed with *SHELX*76 (Sheldrick, 1976), *PLUTO*78 (Motherwell & Clegg, 1978) and *XANADU* (Roberts & Sheldrick, 1975).

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