Cu—N3	2.035 (2)	C5—C6	1.409 (4)
Cu—N4	2.042 (2)	C6—C7	1.434 (4)
O1—C1	1.193 (3)	C6—C14	1.396 (3)
Cu^{i}—N1	2.481 (2)	C7—C8	1.346 (4)
O2—C2	1.196 (4)	C8—C9	1.428 (4)
N1—C1	1.147 (3)	C9—C10	1.405 (4)
N2—C2	1.119 (4)	C9—C13	1.398 (3)
N3—C3	1.329 (3)	C10—C11	1.363 (4)
N3—C14	1.356 (3)	C11—C12	1.385 (4)
N4—C12	1.330 (3)	C13—C14	1.430 (3)
N1—Cu—N2	95.27 (9)	C7—C6—C14	118.6 (2)
N1—Cu—N3	91.70 (8)	C6—C7—C8	121.1 (2)
N1—Cu—N4	172.26 (8)	C7—C8—C9	121.6 (2)
N2—Cu—N3	167.98 (9)	C8—C9—C10	124.6 (2)
N2—Cu—N4	92.35 (9)	C8—C9—C13	118.3 (2)
N3—Cu—N4	80.56 (7)	C10—C9—C13	117.0 (2)
Cu—N1—C1	143.3 (2)	C9—C10—C11	119.2 (3)
Cu—N2—C2	166.3 (2)	C10—C11—C12	120.3 (3)
Cu—N3—C3	128.8 (2)	N4—C12—C11	122.4 (3)
Cu—N3—C14	113.3 (2)	N4—C13—C9	123.3 (2)
C3—N3—C14	117.8 (2)	N4—C13—C14	116.3 (2)
Cu—N4—C12	129.2 (2)	C9—C13—C14	120.4 (2)
Cu—N4—C13	112.8 (1)	N3—C14—C6	123.6 (2)
C12—N4—C13	117.7 (2)	N3—C14—C13	116.4 (2)
O1—C1—N1	176.7 (3)	C6—C14—C13	120.0 (2)
O2—C2—N2	178.3 (3)	Cu^{i}—N1—Cu	92.59 (8)
N3—C3—C4	122.5 (2)	Cu^{i}—N1—C1	118.9 (2)
C3—C4—C5	120.0 (2)	N1^{i}—Cu—N1	87.41 (8)
C4—C5—C6	119.1 (2)	N1^{i}—Cu—N2	99.49 (9)
C5—C6—C14	117.0 (2)	N1^{i}—Cu—N3	90.58 (8)
C5—C6—C7	124.3 (2)	N1^{i}—Cu—N4	92.73 (7)

Symmetry code: (i) $-x, -y, 2-z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP* (Johnson, 1965) in *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *MolEN*.

We gratefully acknowledge financial support for this study from the EWU Foundation and the EWU Northwest Institute for Advanced Studies.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Julve, M., Verdaguer, M., De Munno, G., Real, J. A. & Bruno, G. (1993). *Inorg. Chem.* **32**, 795–802.

Lloret, G., Julve, M., Faus, J., Ruiz, R., Castro, I., Mollar, M. & Philoche-Levisalles, M. (1992). *Inorg. Chem.* **31**, 784–791.

Mauro, A. E., Klein, S. I., Saldana, J. S., de Simone, C. A., Zukerman-Schpector, J. & Castellano, E. E. (1990). *Polyhedron*, **9**, 2937–2939.

Parker, O. J. & Breneman, G. L. (1993). *Polyhedron*, **12**, 891–895.

Sheldrick, G. M. (1990). *SHELXTL/PC*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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Acta Cryst. (1996). C**52**, 1091–1093

cis-(*R*,*S*)-Bis(benzyl methyl sulfoxide)dichloroplatinum(II)

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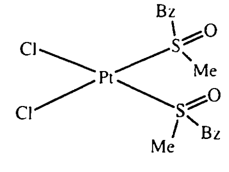
(*Received 10 July 1995; accepted 20 November 1995*)

Abstract

The coordination around the Pt atom in the title compound, $[\mathrm{PtCl_2(C_8H_{10}OS)_2}]$, is pyramidally distorted from *cis* square-planar; two chloride ions form Pt—Cl bonds of 2.310 (3) and 2.307 (3) Å and two benzyl methyl sulfoxide groups form Pt—S bonds of 2.241 (2) and 2.233 (2) Å.

Comment

There should be three isomers of the complex *cis*-bis(benzyl methyl sulfoxide)dichloroplatinum(II): the (*S*,*S*), (*R*,*R*) and *meso* forms. The crystal structure of the (*S*,*S*) form as a methanol solvate have been determined by X-ray methods (Antolini, Folli, Iarossi, Schenetti & Taddei, 1991). We have crystallized and determined the structure of the *meso* form, (I).



(I)

The title compound is composed of neutral *cis*-$[\mathrm{PtCl_2(MeBzSO)_2}]$ complexes (Fig. 1). The closest Pt···Pt distance is 7.274 (1) Å. Both the benzyl methyl sulfoxide molecules are bonded *via* their chiral S atoms. The structure analysis reveals that the two S atoms have different chiralities, *R* and *S*, giving the *meso* form of the platinum complex. The S atoms are in approximately tetrahedral environments with angles ranging from 100.1 (5) (C—S—C) to 117.1 (3)° (Pt—S—O). The S—O and S—C bond distances are close to those found in other sulfoxides (Almeida, Hubbard & Farrell, 1992; Antolini *et al.*, 1991; Melanson & Rochon, 1975, 1987; Melanson, Chevrotire & Rochon, 1985).

Acta Crystallographica Section C
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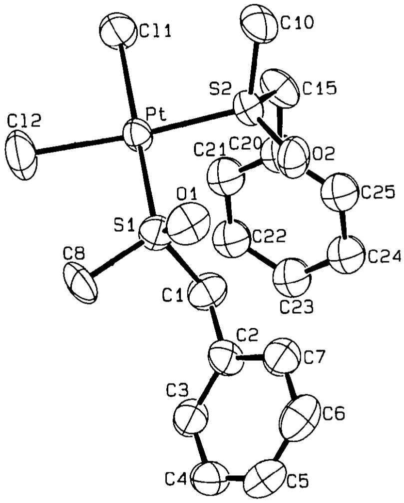

Fig. 1. Molecular structure and atomic numbering of *cis*-$[PtCl_2(MeBzSO)_2]$. Displacement ellipsoids are drawn at the 50% probability level. Only one of three locations of the disordered phenyl group is shown.

One of the benzyl groups was found to be disordered (see *Experimental* and Table 1). The bond distances and angles in the other phenyl ring have close to expected values (Table 2). The dihedral angles between the disordered benzyl groups and the coordination plane are 61 (2)°; for the other benzyl group the dihedral angle is 33 (1)°. The largest deviation from the least-squares plane through the pyramidally distorted Cl_2PtS_2 core is shown by Cl(1) [−0.054 (3) Å]. The non-rigidly refined benzyl group is planar within 0.02 (1) Å.

The Pt—S and Pt—Cl distances are normal compared with those found in similar compounds (see references cited above). The Cl—Pt—Cl angle seems to be the most rigid angle in compounds of the type *cis*-$[PtCl_2(sulfoxide)_2]$, only varying between 87.2 (1) and 88.5 (1)°, while the other angles around the Pt atom are more affected by the orientation of the sulfoxide group and steric and packing effects of the different types of sulfoxides.

Experimental

Benzyl methyl sulfoxide (racemic mixture) reacts with the platinum salt K_2PtCl_4 in aqueous ethanol solution to give the expected product *cis*-$[PtCl_2(MeBzSO)_2]$ as a pale yellow precipitate. The crude material is a mixture of diastereomers, as evidenced by two peaks in the ^{195}Pt NMR spectrum ($CDCl_3$) at −3509 and −3498 p.p.m. relative to Na_2PtCl_6 in D_2O as external standard. The ^1H NMR spectrum confirmed the presence of diastereomers with two separate peaks for methyl resonance and two doublets of doublets for the diastereotopic benzyl protons of the sulfoxide ligand. The peaks are shifted downfield upon coordination, as expected (Farrell, Kiley, Schmidt & Hacker 1990). Recrystallization from EtOH gave colourless crystals of one diastereomer: $\delta(Pt) = -3498$; $\delta(S—CH_3) = 3.31$ (singlet) and $\delta(S—CH_2) = 4.95$ p.p.m. (doublets of doublet); $\nu(SO) = 1103\ cm^{-1}$ {*c.f.* values found in $K[PtCl_3(MeBzSO)]$ (Almeida, Hubbard & Farrell, 1992): $\delta(Pt) = -3011$ (d_6-acetone); $\delta(S—CH_3) = 2.95$ (singlet) and $\delta(S—CH_2) = 4.87$ p.p.m. (doublets of doublet); $\nu(SO) = 1099\ cm^{-1}$}.

Crystal data

$[PtCl_2(C_8H_{10}OS)_2]$
$M_r = 574.439$
Monoclinic
$P2_1/c$
$a = 8.4502\ (9)$ Å
$b = 12.616\ (2)$ Å
$c = 18.219\ (3)$ Å
$\beta = 92.304\ (9)°$
$V = 1940.7\ (8)$ Å^3
$Z = 4$
$D_x = 1.959$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 23 reflections
$\theta = 8.8–24.7°$
$\mu = 7.794$ mm^{-1}
$T = 295$ K
Prism
0.17 × 0.15 × 0.13 mm
Pale yellow

Data collection

CAD-4 diffractometer
$\omega–2\theta$ scans
Absorption correction: numerical by integration from crystal shape
$T_{min} = 0.333$, $T_{max} = 0.436$
6055 measured reflections
5895 independent reflections
3574 observed reflections [$I > 1.5\sigma(I)$]

$R_{int} = 0.037$
$\theta_{max} = 30°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 24$
3 standard reflections monitored every 60 reflections
intensity decay: 0.90%

Refinement

Refinement on F
$R = 0.0529$
$wR = 0.0521$
$S = 1.776$
3574 reflections
178 parameters
H atoms: see below
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = 0.0495$
$\Delta\rho_{max} = 1.91$ e Å^{-3}
$\Delta\rho_{min} = -2.39$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}/U_{iso}
Pt	0.62149 (4)	0.18058 (3)	0.85723 (2)	0.0405 (1)
Cl1	0.4117 (3)	0.1413 (2)	0.7766 (2)	0.063 (1)
Cl2	0.6032 (4)	0.0098 (2)	0.9014 (2)	0.082 (3)
S1	0.8169 (3)	0.2217 (2)	0.9391 (1)	0.050 (1)
S2	0.6215 (3)	0.3450 (2)	0.8118 (1)	0.041 (1)
O1	0.7835 (8)	0.3104 (5)	0.9893 (4)	0.062 (4)
O2	0.7380 (8)	0.4181 (5)	0.8430 (3)	0.057 (4)
C1	0.996 (1)	0.2483 (9)	0.8891 (5)	0.055 (6)
C2	1.132 (1)	0.2816 (7)	0.9382 (5)	0.047 (5)
C3	1.249 (1)	0.2126 (7)	0.9612 (6)	0.054 (6)
C4	1.375 (1)	0.2475 (10)	1.0066 (6)	0.061 (6)
C5	1.385 (1)	0.3513 (10)	1.0289 (7)	0.066 (8)
C6	1.266 (1)	0.4215 (9)	1.0089 (6)	0.067 (8)
C7	1.140 (1)	0.3888 (8)	0.9623 (6)	0.057 (6)
C8	0.878 (1)	0.1110 (9)	0.9945 (7)	0.084 (9)
C10	0.426 (1)	0.4022 (8)	0.8189 (6)	0.062 (6)
C15	0.635 (1)	0.3417 (8)	0.7135 (5)	0.052 (6)

C20†	0.794 (2)	0.305 (2)	0.698 (2)	0.058 (6)
C21†	0.833 (3)	0.199 (2)	0.687 (2)	0.058 (6)
C22†	0.988 (3)	0.172 (2)	0.670 (1)	0.058 (6)
C23†	1.103 (2)	0.251 (3)	0.666 (1)	0.058 (6)
C24†	1.064 (2)	0.356 (3)	0.678 (1)	0.058 (6)
C25†	0.909 (2)	0.384 (2)	0.694 (2)	0.058 (6)
C30‡	0.813 (2)	0.333 (2)	0.696 (2)	0.050 (6)
C31‡	0.874 (3)	0.233 (2)	0.679 (1)	0.050 (6)
C32‡	1.034 (3)	0.222 (2)	0.665 (1)	0.050 (6)
C33‡	1.133 (2)	0.311 (2)	0.667 (1)	0.050 (6)
C34‡	1.072 (2)	0.410 (2)	0.683 (1)	0.050 (6)
C35‡	0.911 (2)	0.421 (2)	0.698 (1)	0.050 (6)
C40§	0.793 (2)	0.289 (2)	0.693 (2)	0.055 (6)
C41§	0.792 (2)	0.179 (2)	0.683 (2)	0.055 (6)
C42§	0.933 (3)	0.126 (2)	0.669 (1)	0.055 (6)
C43§	1.074 (2)	0.183 (2)	0.666 (1)	0.055 (6)
C44§	1.075 (2)	0.292 (2)	0.676 (2)	0.055 (6)
C45§	0.934 (3)	0.345 (2)	0.690 (2)	0.055 (6)

† Occupancy = 0.40 (3); $U_{\rm iso}$. ‡ Occupancy = 0.32 (3); $U_{\rm iso}$. § Occupancy = 0.30 (3); $U_{\rm iso}$.

Table 2. *Selected geometric parameters (Å, °)*

Pt—Cl1	2.310 (3)	S2—O2	1.448 (7)
Pt—Cl2	2.307 (3)	S2—C10	1.81 (1)
Pt—S1	2.241 (2)	S2—C15	1.800 (9)
Pt—S2	2.233 (2)	C1—C2	1.49 (1)
S1—O1	1.479 (7)	C15—C20	1.45 (2)
S1—C1	1.828 (10)	C15—C30	1.55 (2)
S1—C8	1.79 (1)	C15—C40	1.55 (2)
Cl1—Pt—Cl2	87.8 (1)	Pt—S2—C10	109.3 (3)
Cl1—Pt—S1	177.31 (10)	Pt—S2—C15	110.4 (3)
Cl1—Pt—S2	88.56 (9)	O2—S2—C10	109.0 (4)
Cl2—Pt—S1	92.4 (1)	O2—S2—C15	109.5 (4)
Cl2—Pt—S2	176.0 (1)	C10—S2—C15	100.1 (5)
S1—Pt—S2	91.19 (9)	S1—C1—C2	112.8 (7)
Pt—S1—O1	115.7 (3)	S2—C15—C20	106 (1)
Pt—S1—C1	108.2 (3)	S2—C15—C30	108 (1)
Pt—S1—C8	112.6 (4)	S2—C15—C40	109 (1)
O1—S1—C1	110.6 (5)	C20—C15—C30	14 (1)
O1—S1—C8	107.5 (5)	C20—C15—C40	7 (1)
C1—S1—C8	101.4 (5)	C30—C15—C40	21 (1)
Pt—S2—O2	117.1 (3)		

It was possible to resolve three positions of the disordered phenyl group (see Table 1) using a rigid phenyl group in the refinement with C—C 1.40, C—H 0.95 Å and C—C—C 120°. An occupancy factor for each group was refined without restrictions: 0.40 (3), 0.32 (3) and 0.30 (3) resulted, giving a total occupancy for the phenyl group of 1.02 (5), which is an acceptable value. The H atoms of the disordered phenyl group were included in the rigid group and therefore refined with the whole group. The H atoms on C8, C10 and C15 were not included. The H atoms of the ordered benzyl group were included in calculated positions and included in the structure-factor calculations. The largest residual maxima and minima in the $\Delta\rho$ map were 0.92 Å from S1 and 1.55 Å from Cl(1), respectively.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *SET*-4 (Enraf–Nonius, 1989). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (direct methods) (Gilmore, 1983). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

Financial support from Swedish Natural Science Research Council and the Crafoord Foundation is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1300). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Almeida, S. G., Hubbard, J. L. & Farrell, N. (1992). *Inorg. Chim. Acta*, **193**, 149–157.

Antolini, L., Folli, U., Iarossi, D., Schenetti, L. & Taddei, F. (1991). *J. Chem. Soc. Perkin Trans. 2*, pp. 955–961.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Farrell, N., Kiley, D. M., Schmidt, W. & Hacker, M. P. (1990). *Inorg. Chem.* **29**, 397–403.

Gilmore, C. J. (1983). *MITHRIL. Computer Program for the Automatic Solution of Crystal Structures from X-ray Data*. Department of Chemistry, University of Glasgow, Scotland.

Melanson, R. & Rochon, F. D. (1975). *Can. J. Chem.* **53**, 2371–2374.

Melanson, R. & Rochon, F. D. (1987). *Acta Cryst.* C**43**, 1869–1872.

Melanson, R., Chevrotire, C. & Rochon, F. D. (1985). *Acta Cryst.* C**41**, 1428–1431.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Acta Cryst. (1996). C**52**, 1093–1095

(Isothiocyanato)[(1*RS*,4*RS*,8*SR*,11*SR*)-1,4,8,11-tetraazacyclotetradecane]copper(II) Thiocyanate, [Cu(NCS)(cyclam)](SCN)

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(Received 13 July 1995; accepted 6 December 1995)

Abstract

The crystal structure of the title compound, [Cu(NCS)($C_{10}H_{24}N_4$)](SCN), has been determined by X-ray diffraction. A mirror plane passes through the metal atom and bisects the complex with the isothiocyanato ligand lying on the plane. The Cu^{II} ion is five-coordinate in a distorted square-pyramidal environment, with four amine N atoms in equatorial positions and the isothiocyanate N atom in an axial position; its coordination ge-

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