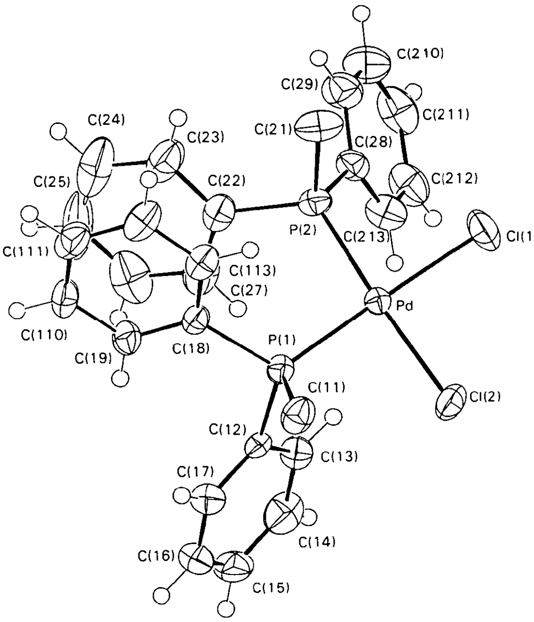

Fig. 1. The molecular structure of the title compound, showing the atomic numbering. Methyl H atoms are omitted for clarity.

Pd—P distance by 0·016 Å. This might be due to the increased steric strain associated with the (presumably) smaller Pd atom, but it can be better explained as resulting from a larger *trans*-effect with Pd than Pt. The Pd—P and Pd—Cl distances fall within the normal range (Palenik, Mathew, Steffan & Beran, 1975).

The title compound exists in solution as a mixture of the *cis*- and *trans*-isomers (Redfield & Nelson, 1973). From solid state ^{13}C NMR measurements, Bodenhausen, Deli, Anklin & Pregosin (1983) have claimed that the solid contains equal amounts of the two isomers. However, the present results show unequivocally that this is a misinterpretation. The NMR inequivalence which leads to the appearance of two sets of phosphine signals must arise because of the crystallographic and therefore environmental inequivalence of the two ligands, which can be seen most markedly in the different Pd—P—C angles noted above. This must also be the correct explanation for the 50:50 ratio of *cis*- and *trans*-isomers of $[PdCl_2(PMe_2Ph)_2]$ claimed by Bodenhausen *et al.* (1983), which is again at variance with X-ray results (Martin & Jacobson, 1971). A similar solid-state inequivalence has been reported for $[PPh_3CuBr]_4$ (Barron, Dyason, Engelhardt, Healy & White, 1984). Clearly any claimed identification of isomers by solid-state NMR in which the ratio of isomers corresponds to the number of chemically equivalent groups in the compound must be treated as suspect until the groups have been shown also to be crystallographically equivalent.

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Structure and Conformation of *cis*-Dichloro[1,1,1-trifluoro-2,3-bis(methylthio)propane]platinum(II), *cis*-$[PtCl_2\{cis\text{-}CH_3SCH_2CH(CF_3)SCH_3\}]$

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Abstract. $C_5H_9Cl_2F_3PtS_2$, $M_r = 456{\cdot}2$, tetragonal, $P4_2/n$ (origin at $\bar{1}$), $a = 17{\cdot}559\ (3)$, $c = 8{\cdot}139\ (3)$ Å, $U = 2509\ (1)$ Å^3, $Z = 8$, $D_x = 2{\cdot}415$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 120{\cdot}5$ cm^{-1}, $F(000) = 1680$, $T = 293$ K, $R = 0{\cdot}029$ for 1298 unique reflections with $I \geq 3\sigma(I)$. The *S*-methyl substituents are mutually *syn*.

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The CF_3 substituent is pseudo-equatorial with respect to the PtS_2C_2 chelate ring. The solid-state structure corresponds to that assigned to the predominant isomer in solution by ^{19}F NMR studies. The Pt coordination geometry is essentially square planar, with bond lengths Pt—S 2·241 (3), 2·231 (4) Å, Pt—Cl 2·303 (4), 2·308 (4) Å.

Introduction. Four isomers of the title complex are observed in solution by ^{19}F NMR at ambient temperature and may be identified spectroscopically by comparison with the analogous complex *cis*-$[PtCl_2\{CF_3SCH_2CH(CH_3)SCF_3\}]$ (Cross, Rycroft, Sharp & Torrens, 1980; Torrens, 1977). This structure analysis confirms the nature of the predominant isomer and the assignments made by spectroscopic techniques.

Experimental. Crystals grown by solvent evaporation from acetonitrile/methylcyclohexane at 278 K; yellow needle, 0·046 × 0·050 × 0·22 mm, exhibiting {100} and {001}; Enraf–Nonius CAD-4F diffractometer, Mo $K\alpha$ radiation, graphite monochromator; cell dimensions by least-squares fit to setting angles of 25 automatically centred reflections with $9 \leq \theta \leq 13°$; intensities of 5841 reflections with $2 \leq \theta \leq 27°$ and $0 \leq h \leq 22$, $0 \leq k \leq 22$, $-10 \leq l \leq 10$, from $\theta/2\theta$ scans of 1·05° in θ; correction for Lp, absorption (Gaussian quadrature, transmission factors on F^2 0·51–0·60) and crystal decomposition (intensity standards 200 and 020 showed a linear decrease of 6% in I during data collection); $R_{\rm int} = 0{\cdot}040$ for 2734 independent reflections measured at least twice; 1451 independent reflections with $I < 3\sigma(I)$ not used in analysis. Structure determined by Patterson and difference syntheses; full-matrix least-squares refinement on F; H atoms positioned geometrically (C—H = 0·96 Å and $U_{\rm H} = U_{\rm C,iso}$) and riding on adjacent C atoms; 118 parameters; $wR = 0{\cdot}037$, $S = 1{\cdot}70$ (on F); $w^{-1} = \sigma^2(F) + 0{\cdot}0025F^2$; mean $\Delta/\sigma = 0{\cdot}009$, max. = 0·04; extreme values in final $\Delta\rho$ synthesis −0·65 to +1·28 e Å^{-3}; no extinction correction necessary; neutral-atom scattering factors and complex anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); *GX* crystallographic program package (Mallinson & Muir, 1985).

Discussion. The crystal packing (Fig. 1) is predominantly of the van der Waals type. However, pairs of centrosymmetrically related molecules are arranged so that the metal coordination planes are antiparallel and the metal–ligand bonds are nearly eclipsed, with a non-bonded Pt···Pt separation of 3·749 (1) Å. Such pairings are a common feature of platinum(II) crystallochemistry and are generally thought to represent a favourable arrangement of molecular dipoles (Manojlović-Muir, Muir & Solomun, 1977).

The platinum atom shows only minor deviations from the expected *cis*-square-planar coordination. The molecule shown in Fig. 2 and defined by the coordinates in Table 1* has a chelate ring of absolute configuration δ [S(1)—C(3)—C(5)—S(2) torsion angle +54·3 (9)°]. The other three chiral centres, S(1), S(2) and C(3), then display absolute configurations R, S and R respectively. The puckered *gauche* chelate ring has approximate C_2 symmetry; ring torsion angles are 16·5 (5) and 6·9 (5)° across Pt—S(1) and Pt—S(2) and −42·4 (9) and −36·8 (9)° across S(1)—C(3) and S(2)—C(5) respectively. The Pt—S(1)—C(3)—C(4) torsion angle of −161·3 (11)° establishes that the CF_3 substituent is pseudo-equatorial with respect to the ring.

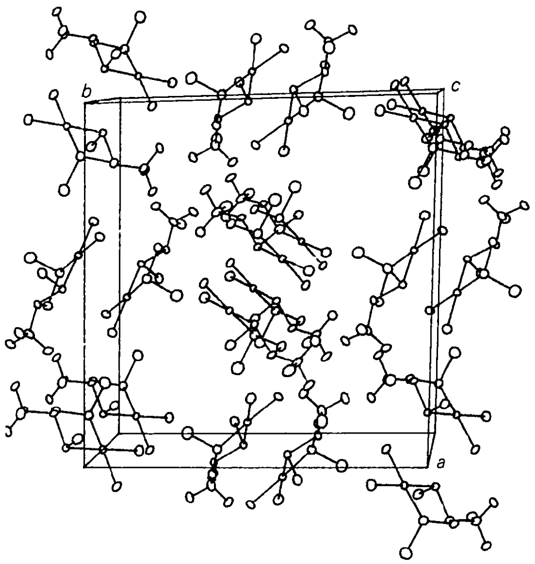

Fig. 1. A view of the molecular packing. Here and in Fig. 2 hydrogen atoms are omitted for clarity.

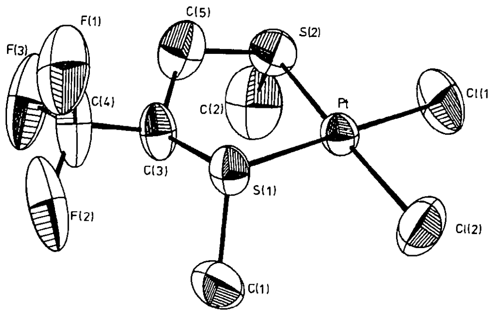

Fig. 2. A view of the *cis*-$[PtCl_2\{CH_3SCH_2CH(CF_3)SCH_3\}]$ molecule showing the atom numbering and 50% vibration ellipsoids.

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42478 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic fractional coordinates and equivalent isotropic displacement parameters* (Å^2)

	x	y	z	U_{eq}*
Pt	0·06238 (3)	0·06724 (2)	0·11786 (5)	0·048
Cl(1)	0·0384 (3)	0·1553 (2)	−0·0853 (5)	0·099
Cl(2)	−0·0218 (2)	0·1278 (2)	0·2922 (5)	0·083
S(1)	0·0869 (2)	−0·0176 (2)	0·3166 (4)	0·051
S(2)	0·1400 (2)	0·0067 (2)	−0·0553 (4)	0·072
F(1)	0·1314 (5)	−0·1887 (5)	0·3129 (13)	0·111
F(2)	0·1950 (6)	−0·1271 (6)	0·4884 (14)	0·133
F(3)	0·2509 (5)	−0·1715 (5)	0·2752 (15)	0·137
C(1)	0·1263 (9)	0·0324 (8)	0·4886 (17)	0·087
C(2)	0·2279 (9)	0·0551 (9)	−0·0540 (2)	0·101
C(3)	0·1728 (6)	−0·0628 (7)	0·2383 (16)	0·062
C(4)	0·1865 (11)	−0·1406 (11)	0·3234 (26)	0·109
C(5)	0·1641 (8)	−0·0767 (8)	0·0587 (18)	0·080

* U_{eq} is the mean latent root of the anisotropic vibration tensor.

Table 2. *Bond lengths* (Å) *and interbond angles* (°)

Pt—Cl(1)	2·303 (4)	Pt—Cl(2)	2·308 (4)
Pt—S(1)	2·241 (3)	Pt—S(2)	2·231 (4)
S(1)—C(1)	1·791 (14)	S(1)—C(3)	1·819 (12)
S(2)—C(2)	1·761 (17)	S(2)—C(5)	1·784 (15)
F(1)—C(4)	1·288 (20)	F(2)—C(4)	1·372 (23)
F(3)—C(4)	1·314 (19)	C(3)—C(4)	1·551 (18)
C(3)—C(5)	1·490 (19)		
Cl(1)—Pt—Cl(2)	90·9 (2)	Cl(1)—Pt—S(1)	179·3 (4)
Cl(1)—Pt—S(2)	88·7 (2)	Cl(2)—Pt—S(1)	89·2 (2)
Cl(2)—Pt—S(2)	177·9 (2)	S(1)—Pt—S(2)	91·2 (2)
Pt—S(1)—C(1)	108·2 (5)	Pt—S(1)—C(3)	101·3 (4)
C(1)—S(1)—C(3)	99·7 (7)	Pt—S(2)—C(2)	107·6 (6)
Pt—S(2)—C(5)	102·0 (5)	C(2)—S(2)—C(5)	100·7 (8)
S(1)—C(3)—C(4)	110·9 (10)	S(1)—C(3)—C(5)	109·3 (9)
C(4)—C(3)—C(5)	108·0 (14)	F(1)—C(4)—F(2)	105·1 (14)
F(1)—C(4)—F(3)	110·8 (18)	F(1)—C(4)—C(3)	115·6 (15)
F(2)—C(4)—F(3)	105·7 (16)	F(2)—C(4)—C(3)	107·6 (17)
F(3)—C(4)—C(3)	111·4 (13)	S(2)—C(5)—C(3)	113·6 (10)

The Pt—S and Pt—Cl bond lengths in Table 2 may be compared with corresponding values in other *cis*-$PtCl_2L$ complexes: Pt—S 2·231 (1), 2·244 (2) Å, Pt—Cl 2·324 (2), 2·316 (2) Å, L = MeSCH(CF_3)CH(CF_3)-SMe (Hunter, Muir & Sharp, 1984); Pt—S 2·240 (3), 2·270 (3) Å, Pt—Cl 2·306 (3), 2·299 (3) Å, L = $MeSCF_2CH_2SMe$ (Cano, Leal, Quintana & Torrens, 1984); Pt—S 2·239 (3), 2·260 (3) Å, Pt—Cl 2·295 (3), 2·290 (4) Å, L = $CF_3SCH_2CH_2SCF_3$ (Manojlović-Muir *et al.*, 1977). Evidently, the Pt—S bond lengths in these complexes are insensitive to the pattern of substitution at the ring S or C atoms. The Pt—Cl(*trans* to SCH_3) distances are 0·01–0·03 Å longer than the Pt—Cl(*trans* to SCF_3) distances, implying a stronger *trans* influence for SCH_3 than for SCF_3, but the effect is small and only barely significant statistically.

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ab,cf-Bis[4-(2-aminoethyl)imidazole-N^3,N^8]-*de*-bis(isothiocyanato)nickel(II)

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Abstract. $[Ni(NCS)_2(C_5H_9N_3)_2]$, M_r = 397·17, monoclinic, $P2_1/c$, a = 8·4903 (5), b = 14·224 (1), c = 14·5746 (9) Å, β = 93·248 (5)°, V = 1757·3 (2) Å^3, Z = 4, D_m = 1·50 (1), D_x = 1·5125 (1) Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 2·80 mm^{-1}, $F(000)$ = 816, T = 293 K, R = 0·0309 for 2349 observed reflections. The six N atoms coordinated to Ni^{2+} form a distorted octahedron. Both histamine ligands are coordinated to Ni^{2+} *via* their N^{π} atoms and amino groups of the side chains. The structure contains a three-dimensional

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