comment that this is a quite short P—N bond. The P—F bond distance determined in this study, 1·600 (6) Å, is similar to those in $(CH_3)_2NPF_2$ of 1·591 (4) Å and NH_2PF_2 of 1·587 (4) Å. It is longer than those reported for $(CH_3)_2NPF_2B_4H_8$ of 1·555 (4) and 1·555 (4) Å.

Figs. 1 and 2 were drawn using ORTEP (Johnson, 1970). The Chemical Abstracts registry number for 2-fluoro-1,3-dimethyl-1,3,2-diazaphospholidine is [33672-91-2].

References

BRITTAIN, A. H., SMITH, J. E., LEE, P. L., COHN, K. & SCHWENDEMAN, R. H. (1971). J. Am. Chem. Soc. **93**, 6772–6776.

CRUICKSHANK, D. W. J. (1970). Least-Squares Refinement of Atomic Parameters, in Crystallographic Computing, edited by F. R. AHMED, pp. 187–196. Copenhagen: Munksgaard.

DOUGLAS, W. M. & RUFF, J. K. (1971). J. Chem. Soc. A, pp. 3558–3561.

HOBBS, E., CORBRIDGE, D. E. C. & RAISTRICK, B. (1953). Acta Cryst. **6**, 621–626.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 71–98 and 148–151. Birmingham: Kynoch Press.

KILBOURN, B. T., RAEBURN, U. A. & THOMPSON, D. T. (1969). J. Chem. Soc. A, pp. 1906–1909.

JOHNSON, C. K. (1970). ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

LA PRADE, M. D. & NORDMAN, C. E. (1969). Inorg. Chem. **8**, 1669–1673.

MORRIS, E. D. JR & NORDMAN, C. E. (1969). Inorg. Chem. **8**, 1673–1676.

STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, University of Maryland, College Park, Maryland.

Thianthrenegold(III) Chloride (Chloroform Solvate) at −70°C

BY N. W. ALCOCK

Department of Chemistry and Molecular Sciences, University of Warwick, Coventry CV4 7AL, England

AND K. P. ANG, K. F. MOK AND S. F. TAN

Department of Chemistry, University of Singapore, Bukit Timah Road, Singapore

Abstract. $C_{12}H_8AuCl_3S_2$. $CHCl_3$, monoclinic, Cm. At −70°C, $a = 12·887$ (3), $b = 14·275$ (5), $c = 5·331$ (2) Å, $\beta = 103·05$ (2)°, $V = 955·35$ Å^3, $Z = 2$, $D_c = 2·22$, $D_m = 2·17$ g cm^{-3}, μ(Mo $K\alpha$) = 85·26 cm^{-1}. Only one of the two S atoms in the thianthrene ligand coordinates to the Au atom, which is also bonded to three Cl atoms. Au—Cl 2·305 (18), 2·274 (4); Au—S 2·351 (15) Å.

Introduction. The title compound was prepared by reacting chloroauric acid with thianthrene in dry chloroform. Slow recrystallization from chloroform solution in a nitrogen-filled dry box afforded imperfectly formed dark-red crystals which were apparently air-stable but from which at room temperature the solvent molecules slowly escaped. A crystal 0·04 × 0·19 × 0·24 mm was used. Data were collected on a Syntex $P2_1$ diffractometer with graphite-monochromatized Mo $K\alpha$ radiation to a $2\theta_{max}$ of 55°. The crystal was kept at −70°C with the Syntex L-T1 attachment. Three standard reflections were used and showed no significant change in intensity during data collection. 1894 reflections were recorded of which 1258 were considered observed $[I/\sigma(I) \geq 3·0]$ and used in refinement. An absorption correction was carried out with ABSCOR (Alcock, 1970).

Systematic absences were observed for hkl when $h + k = 2n + 1$, indicating possible space groups $C2$, Cm and $C2/m$. However, the presence of chloroform solvent molecules suggested Cm as the most likely space group. The Au atom was placed at $(\frac{1}{2},\frac{1}{2},0)$ and Fourier maps were used to locate all the other non-hydrogen atoms. Least-squares refinement with anisotropic temperature factors for all non-hydrogen atoms led to a final R of 0·034. The solvent molecule was given 50% occupancy. Final difference maps showed the presence of solvent molecules at other positions, each with less than 25% occupancy, but these were ignored. Refinement was carried out with XRAY 76 (Stewart, 1976) run on a Burroughs B6700 computer; scattering factors were from International Tables for

Table 1. *Atomic coordinates* (×10⁴) *with e.s.d.'s in parentheses*

	x	y	z
Au	5000	5000	0
Cl(1)	3737 (14)	5000	−3829 (39)
Cl(2)	4993 (14)	3407 (3)	−13 (43)
S(1)	6188 (12)	5000	4057 (32)
S(2)	7886 (6)	5000	432 (14)
C(1)	7054 (15)	4045 (13)	4038 (34)
C(2)	7777 (15)	4055 (13)	2470 (34)
C(3)	8424 (17)	3267 (15)	2436 (42)
C(4)	8321 (21)	2497 (15)	3966 (50)
C(5)	7633 (26)	2515 (23)	5582 (43)
C(6)	6974 (18)	3283 (14)	5646 (41)
C(7)*	1225 (40)	5000	8370 (134)
Cl(3)*	125 (23)	5000	6421 (58)
Cl(4)*	1227 (48)	3988 (22)	207 (109)
H(3)	8980	3248	1222
H(4)	8794	1897	3918
H(5)	7615	1927	6767
H(6)	6440	3291	6878
H(7)*	1890	5000	7330

* Occupancy 0·5.

Fig. 1. View of the molecule down c, showing the atomic numbering.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Au–Cl(1)	2·305 (18)	Cl(1)–Au–Cl(2)	89·8 (5)
Au–Cl(2)	2·274 (4)	Cl(2)–Au–Cl(2')	179·5 (6)
Au–S(1)	2·351 (15)	Cl(1)–Au–S(1)	175·9 (7)
S(1)–C(1)	1·763 (21)	Cl(2)–Au–S(1)	90·2 (5)
S(2)–C(2)	1·758 (19)	Au–S(1)–C(1)	106·1 (8)
C(1)–C(2)	1·38 (3)	C(1)–S(1)–C(1')	101·3 (12)
C(2)–C(3)	1·40 (3)	C(2)–S(2)–C(2')	100·2 (10)
C(3)–C(4)	1·39 (3)	S(1)–C(1)–C(2)	120·5 (15)
C(4)–C(5)	1·37 (4)	S(1)–C(1)–C(6)	117·5 (17)
C(5)–C(6)	1·39 (4)	C(2)–C(1)–C(6)	122·0 (18)
C(6)–C(1)	1·40 (3)	S(2)–C(2)–C(1)	122·7 (14)
C(7)–Cl(3)	1·56 (6)	S(2)–C(2)–C(3)	118·4 (17)
C(7)–Cl(4)	1·75 (6)	C(1)–C(2)–C(3)	118·9 (18)
		C(2)–C(3)–C(4)	119·3 (22)
		C(3)–C(4)–C(5)	121·0 (24)
		C(4)–C(5)–C(6)	121·0 (26)
		C(5)–C(6)–C(1)	117·8 (23)
		Cl(3)–C(7)–Cl(4)	105·1 (29)
		Cl(4)–C(7)–Cl(4')	111·7 (46)

Fig. 2. View of the molecule almost down b.

X-ray Crystallography (1974) in the analytical form. Atomic coordinates are given in Table 1, bond lengths and bond angles in Table 2.* Figs. 1 and 2 show two views of the molecular structure, and Fig. 3 shows the crystal packing along c.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33788 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. Crystals of thianthrenegold(III) trichloride are so dark red that they appeared almost black when viewed with transmitted light under a microscope. Coupled with the fact that the ligand has two S atoms capable of coordination, this suggests the possibility of a polymeric structure. Another interesting feature of this complex is that the solvated chloroform molecules can readily escape, leaving the solid externally intact but internally so disordered that it does not give a diffraction pattern.

Fig. 1 shows that the Au atom is four-coordinated, with the square-planar geometry normally observed for trivalent Au. The two Au—Cl lengths of 2·305 and 2·274 Å fall within the range observed for a number of Au^III trichloride complexes (Bandoli, Clemente, Marangoni & Cattalini, 1973; Robinson & Sinn, 1975)
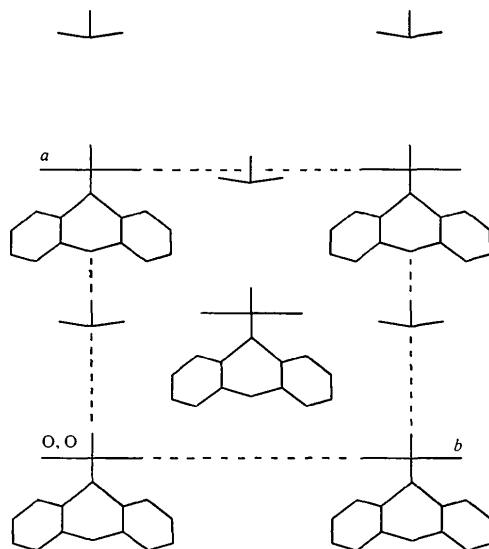
Fig. 3. Packing diagram, viewed down c.

as well as for Au^I chloride complexes (Baenziger, Bennett & Soboroff, 1976; Guy, Jones, Mays & Sheldrick, 1977). Au—Cl(1), which is *trans* to the Au—S bond, is slightly longer than Au—Cl(2). A similar pattern was observed in triphenylphosphinegold(III) trichloride (Bandoli, Clemente, Marangoni & Cattalini, 1973) where the Au—Cl bond *trans* to the Au—P bond was longer at 2·347 (4) Å compared with the other two Au—Cl distances of 2·273 (4) and 2·282 (4) Å.

The Au—S distance of 2·35 Å is within the range 2·28 to 2·36 Å observed for square-planar Au^{III} complexes (Beurskens, Blaaw, Cras & Steggerda, 1968; Beurskens, Cras & van der Linden, 1970; Cras, Noordik, Beurskens & Verhoeven, 1971; McPartlin & Markwell, 1971; Noordik & Beurskens, 1971). The second S atom of the thianthrene ligand is 3·675 (8) Å from the Au atom, which is also 3·817 (18) Å from the Au-bonded S atom of an adjacent molecule. While both these distances are much greater than the covalent Au—S bond, they are shorter than 4·05 Å, the sum of the van der Waals radii (1·85 for S and 2·2 Å estimated for Au by Duckworth & Stephenson, 1969). It is therefore not surprising that the Cl(1)—Au—S(1) linkage deviates significantly from linearity [175·9 (7)°]. The thianthrene molecule is bent, as is normal, with a dihedral angle of 129·5°. This is slightly larger than the

128° observed by Lynton & Cox (1956) and 128·1° by Rowe & Post (1958), both for the free thianthrene molecule. On the other hand it is smaller than the 131·1° observed for uncoordinated 2,7-dimethylthianthrene (Wei, 1971). Bond angles and bond lengths in the thianthrene molecule are normal.

There does not appear to be any interaction between the Au atom and the solvent molecule, with about 4 Å between Au and the H of the chloroform solvate. This lack of interaction probably accounts for the ease with which the solvent molecule escapes.

References

ALCOCK, N. W. (1970). The Analytical Method for Absorption Correction in Crystallographic Computing, edited by F. R. AHMED. Copenhagen: Munksgaard.

BAENZIGER, N. C., BENNETT, W. E. & SOBOROFF, D. M. (1976). Acta Cryst. B32, 962–963.

BANDOLI, G., CLEMENTE, D. A., MARANGONI, G. & CATTALINI, L. (1973). J. Chem. Soc. Dalton Trans. pp. 886–889.

BEURSKENS, P. T., BLAAW, H. J. A., CRAS, J. A. & STEGGERDA, J. J. (1968). Inorg. Chem. 7, 805–810.

BEURSKENS, P. T., CRAS, J. A. & VAN DER LINDEN, J. G. M. (1970). Inorg. Chem. 9, 475–479.

CRAS, J. A., NOORDIK, J. H., BEURSKENS, P. T. & VERHOEVEN, A. M. (1971). J. Cryst. Mol. Struct. 1, 155–160.

DUCKWORTH, V. F. & STEPHENSON, N. C. (1969). Inorg. Chem. 8, 1661–1664.

GUY, J. J., JONES, P. G., MAYS, M. J. & SHELDRICK, G. M. (1977). J. Chem. Soc. Dalton Trans. pp. 8–10.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

LYNTON, H. & COX, E. G. (1956). J. Chem. Soc. pp. 4886–4895.

McPARTLIN, M. & MARKWELL, A. J. (1973). J. Organomet. Chem. 57, C25–C26.

NOORDIK, J. H. & BEURSKENS, P. T. (1971). J. Cryst. Mol. Struct. 1, 339–345.

ROBINSON, W. T. & SINN, E. (1975). J. Chem. Soc. Dalton Trans. pp. 726–731.

ROWE, I. & POST, B. (1958). Acta Cryst. 11, 372–374.

STEWART, J. M. (1976). The XRAY 76 System. Tech. Rep. TR-446. Computer Science Centre, Univ. of Maryland, College Park, Maryland.

WEI, C. H. (1971). Acta Cryst. B27, 1523–1527.