Related literature. The title compound catalyzes asymmetric oxidation of methyl phenyl sulfide to give (*R*)-methyl phenyl sulfoxide (Nakajima, Sasaki, Kojima, Aoyama, Ohba, Saito & Fujita, 1987). As far as we know, the structure of no other binuclear Ti–Schiff-base complex has been determined. The Ti^{III}–(Pasquali, Marchetti, Landi & Floriani, 1978) and Ti^{IV}–(Gilli, Cruickshank, Beddoes & Mills, 1972) Schiff-base complexes reported are mononuclear. The geometry of the Fe—O—Fe bridge in binuclear Fe^{III}–Schiff-base complexes was discussed by Davies & Gatehouse (1973).

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Structure d'une Nouvelle Variété de Di-μ-chloro-bis[(aza-4 azonia-1 bicyclo[2.2.2]octane)dichloromanganèse(II)]

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Abstract. [Mn$_2$Cl$_6$(C$_6$H$_{13}$N$_2$)$_2$], $M_r = 548 \cdot 96$, orthorhombic, *Pbca*, $a = 12 \cdot 078$ (2), $b = 14 \cdot 218$ (2), $c = 12 \cdot 169$ (2) Å, $V = 2089 \cdot 6$ (9) Å^3, $Z = 4$, $D_m = 1 \cdot 78$ (2), $D_x = 1 \cdot 745$ Mg m^{-3}, λ(Mo Kα) = 0·7107 Å, $\mu = 1 \cdot 94$ mm^{-1}, $F(000) = 1112$, $T = 294$ (1) K, $R = 0 \cdot 024$ for 1837 independent observed reflections. This compound is a modification of the monoclinic $P2_1/n$ complex [Viossat, Khodadad & Rodier (1988). *Acta Cryst.* C44, 825–828]. Its dimeric molecule also has a centre of symmetry, two Mn—Cl—Mn bridges and the coordination polyhedron of each Mn atom is a trigonal bipyramid. It differs from the molecule of the monoclinic variety by having larger distances from the N atoms to the plane of the two Mn—Cl—Mn bridges and by having greater N—C—C—N torsional angles, which prevents its description in terms of C_{2h} symmetry. In both forms, the molecules are linked together by N—H⋯Cl and C—H⋯Cl hydrogen bonds.

Partie expérimentale. Cristallisation lente obtenue par diffusion de vapeurs d'acétone dans la solution méthanolique. Masse volumique par flottaison. Cristal parallélépipédique: 0,25 × 0,35 × 0,40 mm. Dimensions de la maille déterminées sur monocristal avec 25 réflexions telles que $9,41 \le \theta \le 20,62°$. Diffractomètre Enraf–Nonius CAD-4. Scan $\theta/2\theta$ d'amplitude $s(°) = 0,65 + 0,35$ tgθ; $2 \le \theta \le 28°$; $0 \le h \le 15$, $0 \le k \le 18$, $0 \le l \le 16$. Réflexions de contrôle: 362, $\overline{4}\overline{4}2$ et $\overline{6}2\overline{3}$. $\sigma(I)/I$ moyen (contrôle): 0,0030. Diminution de *I*: 1,2%. 2514 réflexions indépendantes mesurées, 677 réflexions inobservées $(F_o \le F_{o,\text{max}}/30)$. Corrections d'absorption selon Walker & Stuart (1983). Valeurs maximale et minimale du coefficient de correction: 1,144 et 0,787. Programme *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) et série de Fourier des ΔF. Affinement basé sur les *F*. Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974) corrigés des *f'* et *f''*. Paramètres affinés: *x*, *y*, *z* de tous les atomes et β_{ij} de Mn, Cl, N et C. *B* de chaque H laissé fixe et pris égal à $B_{\text{éq}}$ de l'atome porteur augmenté de 1 Å^2. $R = 0,024$, $wR = 0,019$, $w = 3F_o/F_{o,\text{max}}$ lorsque $F_o <$

Tableau 1. *Coordonnées atomiques relatives, facteurs de température isotropes équivalents et écarts-type*

$$B_{\acute{e}q} = \tfrac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab\cos\gamma + \beta_{13}ac\cos\beta + \beta_{23}bc\cos\alpha).$$

	x	y	z	$B_{\acute{e}q}(\text{Å}^2)$
Mn	0,36439 (3)	0,46411 (3)	0,43163 (3)	2,059 (5)
Cl(1)	0,44597 (5)	0,45977 (6)	0,61424 (5)	3,95 (1)
Cl(2)	0,38896 (5)	0,34616 (5)	0,29148 (5)	2,79 (1)
Cl(3)	0,25206 (6)	0,59298 (5)	0,38070 (5)	2,98 (1)
N(1)	0,2129 (1)	0,3820 (1)	0,5082 (2)	1,77 (4)
N(2)	0,0491 (2)	0,2936 (2)	0,5896 (2)	2,70 (5)
C(3)	0,2501 (3)	0,2949 (2)	0,5650 (2)	2,73 (5)
C(4)	0,1337 (2)	0,3535 (2)	0,4214 (2)	2,83 (5)
C(5)	0,1542 (2)	0,4409 (2)	0,5896 (2)	2,73 (5)
C(6)	0,1525 (2)	0,2401 (2)	0,6119 (3)	4,05 (6)
C(7)	0,0327 (2)	0,3038 (2)	0,4691 (2)	3,32 (6)
C(8)	0,0571 (2)	0,3874 (2)	0,6426 (2)	3,60 (6)

Tableau 2. *Distances interatomiques (Å), angles des liaisons (°) et écarts-type*

Mn—Cl(1)	2,432 (1)	N(1)—C(5)	1,478 (3)
Mn—Cl(1^i)	2,594 (1)	N(2)—C(6)	1,487 (4)
Mn—Cl(2)	2,410 (1)	N(2)—C(7)	1,487 (4)
Mn—Cl(3)	2,363 (1)	N(2)—C(8)	1,484 (4)
Mn—N(1)	2,362 (2)	C(3)—C(6)	1,523 (4)
N(1)—C(3)	1,488 (3)	C(4)—C(7)	1,524 (4)
N(1)—C(4)	1,482 (3)	C(5)—C(8)	1,539 (4)

Cl(1)—Mn—Cl(1^i)	81,34 (2)	Mn—N(1)—C(5)	110,9 (1)
Cl(1)—Mn—Cl(2)	125,39 (3)	C(3)—N(1)—C(4)	107,3 (2)
Cl(1)—Mn—Cl(3)	119,47 (3)	C(3)—N(1)—C(5)	107,8 (2)
Cl(1)—Mn—N(1)	86,60 (5)	C(4)—N(1)—C(5)	108,9 (2)
Cl(1^i)—Mn—Cl(2)	91,68 (3)	C(6)—N(2)—C(7)	110,0 (2)
Cl(1^i)—Mn—Cl(3)	97,30 (3)	C(6)—N(2)—C(8)	109,0 (2)
Cl(1^i)—Mn—N(1)	167,19 (5)	C(7)—N(2)—C(8)	110,4 (2)
Cl(2)—Mn—Cl(3)	115,13 (3)	N(1)—C(3)—C(6)	111,5 (2)
Cl(2)—Mn—N(1)	91,78 (5)	N(1)—C(4)—C(7)	111,8 (2)
Cl(3)—Mn—N(1)	92,39 (5)	N(1)—C(5)—C(8)	111,5 (2)
Mn—Cl(1)—Mn^i	98,66 (3)	N(2)—C(6)—C(3)	108,7 (2)
Mn—N(1)—C(3)	111,1 (2)	N(2)—C(7)—C(4)	108,3 (2)
Mn—N(1)—C(4)	110,7 (1)	N(2)—C(8)—C(5)	108,2 (2)

Code de symétrie : (i) $1-x$, $1-y$, $1-z$.

$F_{o,\text{max}}/3$ et $w = F_{o,\text{max}}/3F_o$ lorsque $F_o \geq F_{o,\text{max}}/3$. $S = 1,12$. $(\Delta/\sigma)_{\text{max}} < 0,01$, $|\Delta\rho|_{\text{max}} = 0,27$ (6) e Å^{-3}. Programmes de calcul du système *SDP* (Frenz, 1982). Fig. 1: programme *ORTEP*II (Johnson, 1976). Ordinateur DEC PDP 11/44.

Les coordonnées atomiques relatives sont rassemblées dans le Tableau 1, les distances interatomiques et les angles des liaisons dans le Tableau 2.* La Fig. 1 indique les numéros utilisés pour désigner les atomes contenus dans l'unité asymétrique. La Fig. 2 est une vue de la structure selon [001].

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* Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope, des paramètres des atomes d'hydrogène, des distances intermoléculaires, des liaisons hydrogène, des distances des atomes aux plans moyens et des angles de torsion ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 44883: 16 pp.). On peut en obtenir des copies en s'adressant à : The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

Littérature associée. Di-μ-chloro-bis[(aza-4 azonia-1 bicyclo[2.2.2]octane)dichloromanganèse(II)] (Viossat *et al.*, 1988). Perchlorate d'aza-4 azonia-1 bicyclo-[2.2.2]octane (Głowiak, Sobczyck & Grech, 1975). Di-μ-chloro-hexacarbonylbis(tétrahydrofuranne)-dimanganèse (Vanderveer & Burlitch, 1980). Dichloro-[oxo-4 (pyridyl-2)-2 (pyridyl-2 méthyl)imino-3 tétra-hydro-1,2,3,4 quinazoline]manganèse(II) (Pelizzi & Pelizzi, 1974).
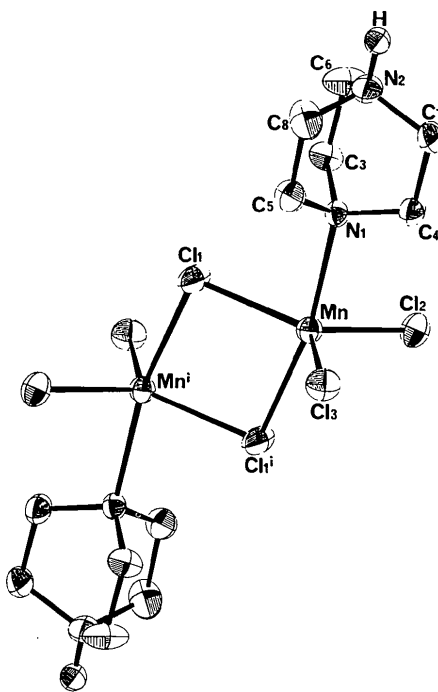

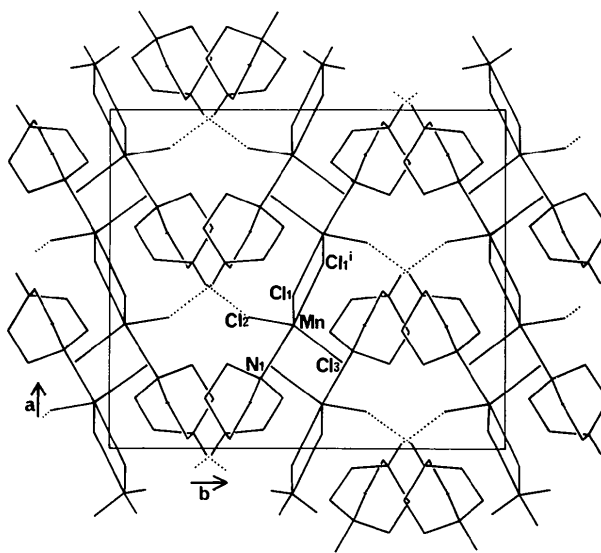Fig. 1. Vue de la molécule en perspective et numéros attribués aux atomes de l'unité asymétrique.

Fig. 2. Dessin de la structure vue selon [001]. Les traits en pointillé représentent les liaisons hydrogène N—H···Cl.

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The Structure of [(C₆H₅)CH₂P(C₆H₅)₃][Au(C₆F₅)Cl]: an Organometallic Gold(I) Pentafluorophenyl Complex Useful as an Ylide Precursor

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Abstract. [(C₆H₅)CH₂P(C₆H₅)₃][Au(C₆F₅)Cl], $M_r =$ 752·9, triclinic, $P\bar{1}$, $a = 17\cdot808$ (3), $b = 18\cdot419$ (3), $c = 9\cdot725$ (1) Å, $\alpha = 97\cdot68$ (1), $\beta = 94\cdot32$ (1), $\gamma = 62\cdot36$ (1)° [(0 0 1, -1 0 0, 0 -1 0) is the transform which converts the chosen cell to a standard: $a = 9\cdot725$ (1), $b = 17\cdot808$ (3), $c = 18\cdot419$ (3) Å, $\alpha = 62\cdot36$ (1), $\beta = 82\cdot32$ (1), $\gamma = 85\cdot62$ (1)°], $V = 2800\cdot3$ (8) Å³, $Z = 4$, $D_x = 1\cdot78$ g cm⁻³, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 54\cdot5$ cm⁻¹, $F(000) = 1456$, $T = 295$ K. Convergence to final conventional R values of $R = 0\cdot0535$ and $wR = 0\cdot0501$ was obtained using 283 variable parameters and 3545 reflections with $F_o^2 > 3\sigma(F_o^2)$. The structure of (benzyl)(triphenyl)phosphonium chloro(pentafluorophenyl)gold(I) consists of an anion–cation pair. The anionic [Au(C₆F₅)Cl]⁻ part is composed of a two-coordinate Au atom bonded to a pentafluorophenyl ring and a Cl atom, and is the first structurally characterized arylgold(I) halide. The counter ion is a benzyltriphenylphosphonium.

Experimental. The title complex (Usón, Laguna & Laguna, 1988; Usón, Laguna, Laguna & Usón, 1983), along with the Au(C₆F₅)(SC₄H₈) starting material (Usón, Laguna & Vicente, 1977; Usón, Laguna, Laguna & Fernandez, 1980), were prepared according to the literature procedure. Benzyltriphenylphosphonium chloride was obtained from Alfa Products and used as received. Crystals suitable for X-ray analysis were obtained by recrystallization from an ethyl alcohol/hexane solution cooled to 278 K.

A single multi-faceted white crystal of approximate dimensions 0·25 × 0·15 × 0·10 mm was mounted in a random orientation on a glass fiber. Triclinic symmetry was suggested on the basis of interaxial angles and confirmed by a Delaunay reduction. Axial lengths were checked by comparison with the interlayer spacings observed in axial photographs. Refined cell parameters were obtained from the setting angles of 25 reflections with $25 < 2\theta < 31°$. Data collection was carried out at room temperature using the ω scanning technique in bisecting geometry. (Nicolet $R3m/E$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation.) Intensities were measured for 3545 unique reflections ($h < 20$, $|k| < 20$, $|l| < 11$) with $0 < 2\theta < 45°$, and no symmetry-equivalent reflections were collected. Scan rate was variable, 3–30° min⁻¹; scan range $-0\cdot8$ in ω from $K\alpha_1$ to $+0\cdot8$ from $K\alpha_2$. Backgrounds were estimated from a 96-step peak profile. Three low-angle standards ($\bar{1}2\bar{1}$, $\bar{3}11$, $\bar{2}5\bar{1}$) measured every 97 data. No decay in these check reflections was noted through completion of data collection. Corrections for absorption were applied empirically on the basis of azimuthal scans of nine reflections spanning a range of 2θ values (minimum transmission, 0·304 and 0·475, respectively). Structure solution and refinement were carried out using the *SHELXTL* collection of crystallographic software (Sheldrick, 1981). The Au position was determined from a sharpened Patterson map; all remaining non-hydrogen atoms located on difference

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