

**Refinement**

Refinement on $F^2$	$(\Delta/\sigma)_{\max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$\Delta\rho_{\max} = 0.43 \text{ e } \text{Å}^{-3}$
$wR(F^2) = 0.095$	$\Delta\rho_{\min} = -0.23 \text{ e } \text{Å}^{-3}$
$S = 1.05$	Extinction correction: none
3237 reflections	Scattering factors from
338 parameters	<i>International Tables for</i>
H atoms: see below	<i>Crystallography</i> (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.1868P]$	Absolute structure: Flack
where $P = (F_o^2 + 2F_c^2)/3$	(1983)
	Flack parameter = 0.00 (2)

Table 1. Selected geometric parameters (Å, °)

Co—N3	1.878 (3)	O5—C11	1.217 (4)
Co—N1	1.884 (3)	O6—C11	1.334 (5)
Co—N4	1.884 (3)	O6—C12	1.441 (6)
Co—N2	1.888 (3)	C9—C10	1.525 (6)
Co—C10	2.112 (4)	C10—C11	1.478 (6)
Co—P	2.330 (1)		
C10—Co—P	176.3 (1)	C9—C10—Co	115.2 (3)
C11—O6—C12	116.1 (3)	O5—C11—O6	121.9 (4)
C11—C10—C9	112.4 (4)	O5—C11—C10	125.6 (5)
C11—C10—Co	108.6 (3)	O6—C11—C10	112.5 (3)

H atoms were refined using a riding model, except for the H atoms of the hydroxy groups, the coordinates and displacement parameters of which were fixed.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1992a). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992b). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SV* (Nemoto & Ohashi, 1993). Software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1314). Services for accessing these data are described at the back of the journal.

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**A Polymorph of Bis(2-pyridylmethyl)amine Iron(III) Chloride**

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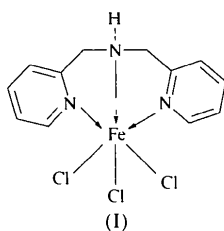
**Abstract**

The structure of the orthorhombic modification of an iron(III) chloride complex of bis(2-pyridylmethyl)amine, a potential tridentate N-donor ligand, has been determined, *i.e.* [bis(2-pyridylmethyl)amine- $\kappa^3N$ ]-trichloroiron(III), [FeCl<sub>3</sub>(C<sub>12</sub>H<sub>13</sub>N<sub>3</sub>)]. The iron(III) has a rhombically distorted octahedral coordination environment made up of all three N atoms of the capping tridentate ligand and three Cl<sup>-</sup> ions.

**Comment**

Iron(III) complexes incorporating pyridyl and phenolate donors have been synthesized in order to unravel the structure–function relationships of iron(III) tyrosinate proteins (Cox & Que, 1988; Que, 1983). Recently, we reported a series of such iron(III) complexes possessing interesting spectral and structural properties (Viswanathan *et al.*, 1996).

In the crystal of the title compound, (I), there are two crystallographically independent complex molecules in the asymmetric unit, which exhibit the same coordination geometry but slightly different bond lengths and angles.



Each complex has distorted octahedral coordination geometry. The equatorial plane is defined by two *cis*-coordinated pyridine N atoms and two Cl<sup>-</sup> ions. The secondary amine N atom is axial to this plane and *trans* to the third Cl<sup>-</sup> ion, resulting in folding and hence facial coordination of the bis(pyridylmethyl)amine ligand. The average Fe—N(pyridine) bond lengths in the two molecules are almost equal [2.182 (5) and 2.188 (5) Å]. The Fe—N(secondary amine) distance is longer by 0.015 Å on average when compared with the Fe—N(pyridine) interactions. The Fe—Cl bonds *trans* to the Fe—N(pyridine) bonds are longer than the Fe—Cl bonds *trans* to the Fe—N(amine) bond. Also, the approximately linear N(amine)—Fe—Cl angles are wider by about 3.5° than the N(pyridine)—Fe—Cl angles. All four five-membered chelate rings adopt half-chair conformations and the bite angles range from 75.8 (2) to 76.5 (2)°. The pyridine rings are planar. All other distances and angles are comparable with values found for related metal complexes obtained from pyridine- and benzimidazolyl-derived ligands (Butcher & Addison, 1989; Adams *et al.*, 1990), including the monoclinic modification of the present structure (Viswanathan *et al.*, 1996).

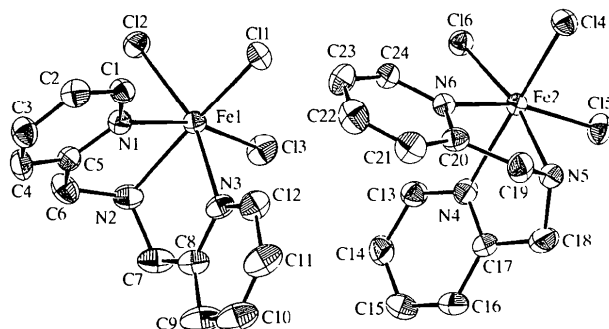


Fig. 1. The structures of the two independent complex molecules in the asymmetric unit (40% probability displacement ellipsoids, H atoms omitted for clarity).

## Experimental

The reaction of iron(III) chloride in methanol with bis(2-pyridylmethyl)amine in methanol in a 1:1 molar ratio produced the title compound in 80% yield.

### Crystal data

[FeCl<sub>3</sub>(C<sub>12</sub>H<sub>13</sub>N<sub>3</sub>)]  
M<sub>r</sub> = 361.45

Mo Kα radiation  
λ = 0.71073 Å

### Orthorhombic

*Pna*2<sub>1</sub>

*a* = 15.575 (5) Å

*b* = 8.455 (5) Å

*c* = 22.745 (5) Å

*V* = 2995 (2) Å<sup>3</sup>

*Z* = 8

*D<sub>x</sub>* = 1.603 Mg m<sup>-3</sup>

*D<sub>m</sub>* = 1.611 Mg m<sup>-3</sup>

*D<sub>m</sub>* measured by flotation in  
CCl<sub>4</sub>/CH<sub>2</sub>Br<sub>2</sub>

Cell parameters from 25  
reflections

θ = 12.5–17.8°

μ = 1.530 mm<sup>-1</sup>

*T* = 293 (2) K

Prism

0.35 × 0.25 × 0.18 mm

Yellow

### Data collection

Enraf–Nonius CAD-4

diffractometer

ω–2θ scans

Absorption correction: none

5254 measured reflections

5254 independent reflections

4318 reflections with

*I* > 2σ(*I*)

θ<sub>max</sub> = 25.0°

*h* = 0 → 18

*k* = 0 → 10

*l* = -27 → 27

3 standard reflections

every 100 reflections

intensity decay: <1%

### Refinement

Refinement on *F*<sup>2</sup>

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.033

w*R*(*F*<sup>2</sup>) = 0.148

*S* = 1.109

5236 reflections

343 parameters

H atoms constrained

w = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0473*P*)<sup>2</sup>  
+ 3.3599*P*]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = -0.001

Δρ<sub>max</sub> = 0.252 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.333 e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from

*International Tables for  
Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Fe1—N3	2.180 (5)	Fe2—N6	2.181 (4)
Fe1—N1	2.183 (4)	Fe2—N4	2.193 (5)
Fe1—N2	2.194 (5)	Fe2—N5	2.205 (5)
Fe1—Cl1	2.276 (2)	Fe2—Cl6	2.276 (2)
Fe1—Cl3	2.283 (2)	Fe2—Cl5	2.284 (2)
Fe1—Cl2	2.312 (2)	Fe2—Cl4	2.319 (2)
N3—Fe1—N1	78.9 (2)	N6—Fe2—N4	79.9 (2)
N3—Fe1—N2	75.8 (2)	N6—Fe2—N5	76.5 (2)
N1—Fe1—N2	76.5 (2)	N4—Fe2—N5	76.4 (2)
N3—Fe1—Cl1	93.7 (2)	N6—Fe2—Cl6	95.09 (14)
N1—Fe1—Cl1	94.24 (14)	N4—Fe2—Cl6	92.70 (15)
N2—Fe1—Cl1	167.05 (15)	N5—Fe2—Cl6	167.16 (14)
N3—Fe1—Cl3	92.18 (14)	N6—Fe2—Cl5	164.50 (13)
N1—Fe1—Cl3	164.52 (13)	N4—Fe2—Cl5	92.44 (14)
N2—Fe1—Cl3	89.1 (2)	N5—Fe2—Cl5	88.67 (15)
Cl1—Fe1—Cl3	98.99 (7)	Cl6—Fe2—Cl5	98.73 (6)
N3—Fe1—Cl2	162.38 (15)	N6—Fe2—Cl4	87.02 (13)
N1—Fe1—Cl2	87.13 (14)	N4—Fe2—Cl4	163.42 (15)
N2—Fe1—Cl2	90.6 (2)	N5—Fe2—Cl4	90.74 (15)
Cl1—Fe1—Cl2	98.05 (8)	Cl6—Fe2—Cl4	98.59 (8)
Cl3—Fe1—Cl2	98.87 (7)	Cl5—Fe2—Cl4	97.77 (6)

The H atoms were fixed at calculated positions riding on their parent atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1990). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1072). Services for accessing these data are described at the back of the journal.

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## (Trifluoroacetato-*O,O'*)bis(triphenylphosphine-*P*)silver(I)

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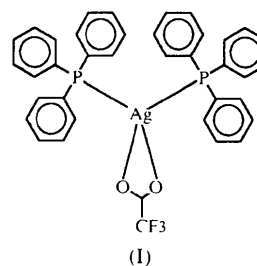
## Abstract

The Ag atom in the title complex,  $[\text{Ag}(\text{C}_2\text{F}_3\text{O}_2)(\text{C}_{18}\text{H}_{15}\text{P})_2]$ , is bonded to two triphenylphosphine ligands and a chelating trifluoroacetate anion in a distorted tetrahedral geometry.

## Comment

Silver(I) acetate reacts with triphenylphosphine to form dimeric bis[acetato(triphenylphosphine)silver(I)],

as well as monomeric acetatobis(triphenylphosphine)silver(I). The latter crystallizes with two independent molecules, *A* and *B*, in the unit cell. The Ag atoms in the monomeric compound show a distorted tetrahedral coordination: the acetate anion chelates in an isobidentate manner in molecule *A* [ $\text{Ag}-\text{P} = 2.433(1)$  and  $2.448(1) \text{ \AA}$ ;  $\text{Ag}-\text{O} = 2.420(2)$  and  $2.438(2) \text{ \AA}$ ], but in an anisobidentate manner in molecule *B* [ $\text{Ag}-\text{P} = 2.426(1)$  and  $2.461(1) \text{ \AA}$ ;  $\text{Ag}-\text{O} = 2.379(3)$  and  $2.510(3) \text{ \AA}$ ] (Ng & Hamid Othman, 1997). The Ag atom in the (trifluoroacetato-*O,O'*)bis(triphenylphosphine-*P*)silver(I) analog, (I), is carboxylate-chelated in a distorted tetrahedral geometry, but whereas the silver–phosphorus distances [ $\text{Ag}-\text{P} = 2.423(1)$  and  $2.445(1) \text{ \AA}$ ] are similar to those in molecule *A* of acetatobis(triphenylphosphine)silver(I), the silver–oxygen distances [ $\text{Ag}-\text{O} = 2.526(3)$  and  $2.542(4) \text{ \AA}$ ] are significantly longer, in agreement with the observation that the trifluoroacetato ion is a weaker Lewis base than the acetato ion.



An analysis of the structural features of several acetates and trifluoroacetates has found that the  $\text{O}-\text{C}-\text{O}$  angles of trifluoroacetates exceed those of acetates (Brown, 1980); this feature is also observed for the (trifluoroacetato-*O,O'*)bis(triphenyl-

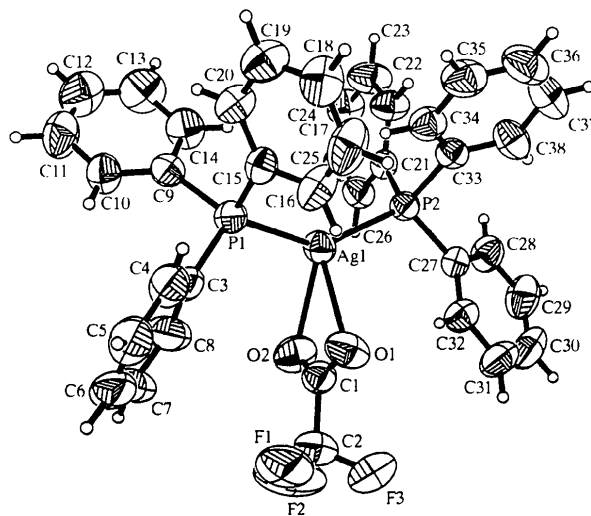


Fig. 1. ORTEP (Johnson, 1976) plot at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.