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Hexakis(acetonitrile)iron(II) hexafluoroantimonate

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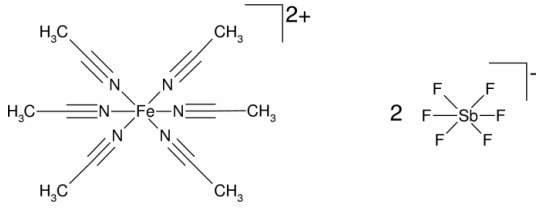
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.024
wR factor = 0.069
Data-to-parameter ratio = 18.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Fe(C_2H_3N)_6][SbF_6]_2$, (I), synthesized by reaction of $AgSbF_6$ with $FeCl_2$ in CH_3CN, is isostructural with the analogous nickel complex [see Leban *et al.* (1987), *Acta Cryst.* C**43**, 1888–1890]. The Fe^{II} centre in the cation occupies a crystallographic $\bar{3}$ site and is octahedrally coordinated by six acetonitrile ligands, with an Fe—N distance of 2.153 (2) Å and an N—Fe—N angle of 88.05 (10)°. The SbF_6^- anion lies on a crystallographic threefold rotation axis.



(I)

Experimental

Caution: exothermic reaction! $AgSbF_6$ (25.00 g, 72 mmol) was slowly added to $FeCl_2$ (9.23 g, 72 mmol) in CH_3CN and allowed to stir overnight. The precipitate was removed by filtration and washed several times with CH_3CN. Removal of the solvent from the filtrate yielded a white powder. Dissolving this solid in a minimal amount of CH_3CN and layering with Et_2O led to a clear colourless microcrystalline solid which became a white powder upon drying under vacuum. Yield: 11.95 g (21.2%). Analysis calculated for $C_{12}H_{18}F_{12}FeN_6Sb_2$ (%): C 18.63, H 2.35, N 10.86, F 29.47; found (%): C 18.42, H 2.21, N 10.57, F 29.20. Crystallographic quality single crystals were obtained by vapour diffusion of Et_2O into a CH_3CN solution of this compound (Fig. 1).

Crystal data

$[Fe(C_2H_3N)_6][SbF_6]_2$
M_r = 773.67
Hexagonal, $R\bar{3}$ (hexagonal axes)
a = 11.3398 (6) Å
c = 17.3584 (11) Å
V = 1933.1 (2) Å^3
Z = 3
D_x = 1.994 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4001 reflections
θ = 2.4–27.5°
μ = 2.73 mm^{-1}
T = 173 (2) K
Block cut from hexagonal prism, colourless
0.40 × 0.30 × 0.30 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995; Sheldrick, 2000)
T_{min} = 0.602, T_{max} = 0.900
5690 measured reflections
996 independent reflections
933 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°
$h = -14 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -22 \rightarrow 22$

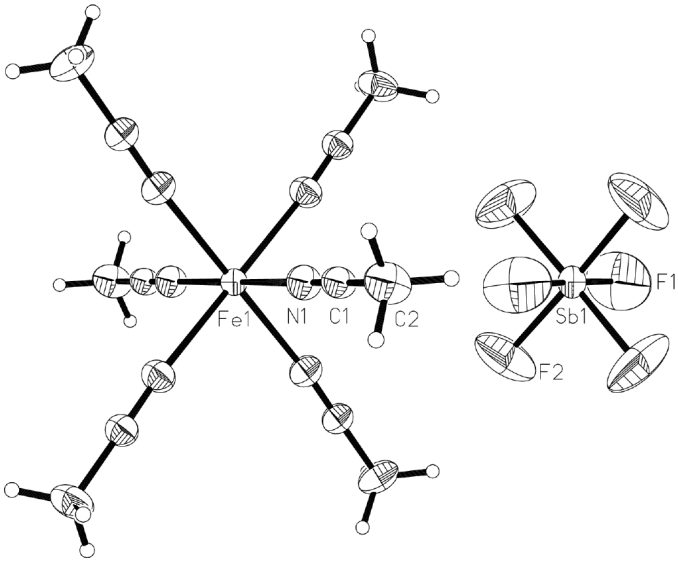


Figure 1
A view of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2 + 4.867P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.024$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.069$	$\Delta\rho_{max} = 0.55$ e Å^{-3}
$S = 1.12$	$\Delta\rho_{min} = -0.47$ e Å^{-3}
996 reflections	Extinction correction: *SHELXL*97
53 parameters	Extinction coefficient: 0.0107 (5)
H-atom parameters constrained	

The positions of the Fe and Sb atoms were found using Patterson methods (Sheldrick, 1990). H atoms were placed geometrically on the CH_3CN ligands optimizing the fit with the diffraction data and then refined as a rigid rotor.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL/PC* and *PLATON* (Spek, 2001).

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