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Ammonium bis{3-anilinocarbonyl-1-[(5-chloro-2-oxidophenyl)diazenyl]-2-naphtholato}ferrate(III) methanol solvate

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Key indicators

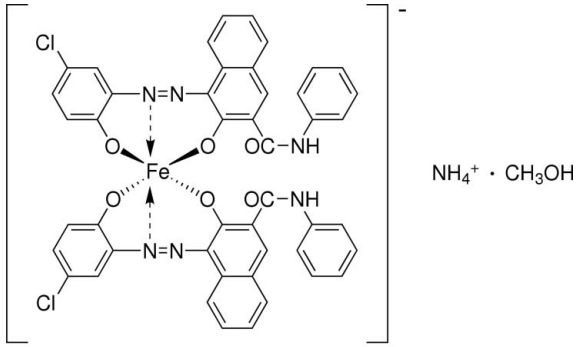
Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.157
Data-to-parameter ratio = 16.3

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

The title compound, $(NH_4)[Fe(C_{23}H_{24}ClN_3O_3)_2]\cdot CH_4O$, is a charge-control agent used in electrophotography. The central Fe^{III} atom is coordinated by four O atoms and two N atoms of two symmetry-independent ligands. The metallic complex has no crystallographic symmetry. The structure features hydrogen-bonded double chains, which run along the *a* axis.

Comment

The title compound, (I), is a methanol-solvated, azo–iron complex used widely as a charge-control agent (CCA) of the negative type in electrophotography (Tanaka, 1995). CCAs are usually added to toners to create a desired charge level and polarity (Nash *et al.*, 2001). However, the charge-control mechanism of CCA is not yet fully understood. An attempt was therefore made to determine the crystal structure as a step towards elucidation of the mechanism. This paper deals with the structure of the methanol-solvated complex; the acetone-solvated complex (Mizuguchi *et al.*, 2007) follows this publication.



(I)

Fig. 1 shows the molecular structure of (I). The anion complex has no crystallographic symmetry, though inversion symmetry (which is not possible) has been implied in literature (for example, Tanaka, 1995). Fig. 2 illustrates the packing arrangement in (I), showing ions related by inversion centers and the hydrogen-bond network (Table 1) linking the metal complexes in double chains running along the *a* axis.

Experimental

Compound (I) was prepared according to the methods previously reported (Yasumatsu *et al.*, 2006). Single crystals of (I) were recrystallized from a methanol solution. After 48 h, a number of black crystals were obtained in the form of blocks.

Crystal data

$(NH_4)[Fe(C_{23}H_{24}ClN_3O_3)_2]\cdot CH_4O$
M_r = 937.58
Triclinic, $P\overline{1}$
a = 10.1107 (4) Å
b = 14.1645 (6) Å
c = 15.3877 (7) Å
α = 103.2240 (11)°
β = 102.8270 (12)°
γ = 94.0860 (12)°
V = 2074.39 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.56 mm^{-1}
T = 93.1 K
0.20 × 0.20 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.896, T_{max} = 0.940
32362 measured reflections
9404 independent reflections
6242 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.157
S = 1.00
9404 reflections
578 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.77 e Å^{-3}
$\Delta\rho_{min}$ = −0.92 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N7—H1A···O1	1.06	1.69	2.747 (3)	174
N7—H1B···O6^{i}	0.95	1.96	2.910 (3)	178
N7—H1C···O7	0.92	1.84	2.748 (4)	173
N7—H1D···O3^{i}	0.84	1.92	2.753 (3)	171
O7—H7O···O6^{ii}	0.84	1.89	2.709 (3)	166
N3—H3N···O2	0.88	2.09	2.675 (3)	124
N6—H6N···O5	0.88	1.95	2.653 (2)	136

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z+1$.

The four H atoms of the ammonium cation were found in difference maps and fixed in position during the least-squares refinement with U_{iso}(H) = 1.2U_{eq}(N). All remaining H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.95 and 0.98 Å, N—H = 0.88 Å, O—H = 0.84 Å, and U_{iso}(H) = 1.2U_{eq}(parent atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2006); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

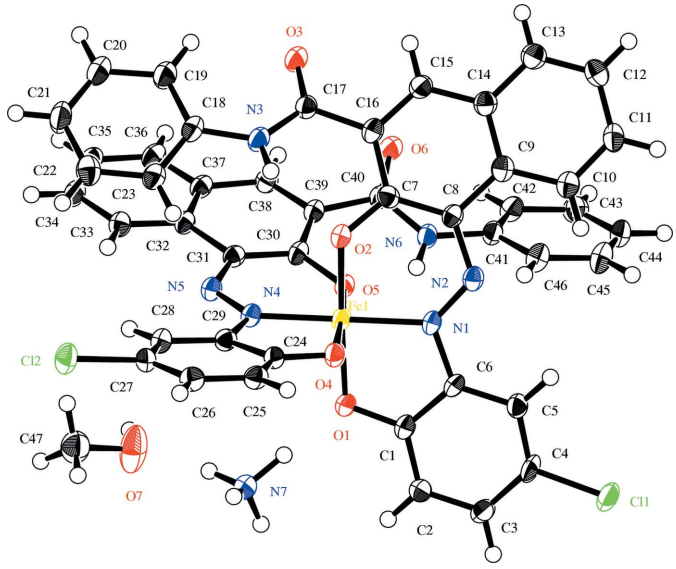


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

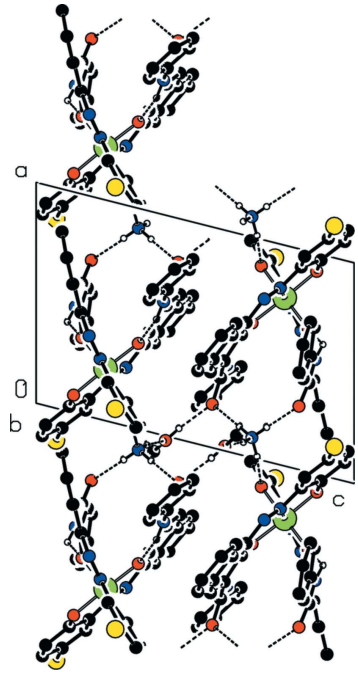


Figure 2
The packing arrangement in (I) showing a double chain. The H atoms, except for the ammonium cation and methanol solvent molecule, have been omitted for clarity. Dashed lines indicate hydrogen bonds.

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