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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

$T = 93\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

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, $(\text{NH}_4)[\text{Fe}(\text{C}_{23}\text{H}_{24}\text{ClN}_3\text{O}_3)_2]\cdot\text{CH}_4\text{O}$, 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.

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

(NH₄)[Fe(C₂₃H₂₄ClN₃O₃)₂].CH₄O

*M*_r = 937.58

Triclinic, *P*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) Å³

Z = 2

Mo *K*α 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 > 2σ(*F*²)

*R*_{int} = 0.054

Refinement

R [*F*² > 2σ(*F*²)] = 0.054

wR (*F*²) = 0.157

S = 1.00

9404 reflections

578 parameters

H-atom parameters constrained

Δρ_{max} = 0.77 e Å^{−3}

Δρ_{min} = −0.92 e Å^{−3}

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ... <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> — <i>H</i> ... <i>A</i>
N7—H1 <i>A</i> ...O1	1.06	1.69	2.747 (3)	174
N7—H1 <i>B</i> ...O6 ⁱ	0.95	1.96	2.910 (3)	178
N7—H1 <i>C</i> ...O7	0.92	1.84	2.748 (4)	173
N7—H1 <i>D</i> ...O3 ⁱ	0.84	1.92	2.753 (3)	171
O7—H7 <i>O</i> ...O6 ⁱⁱ	0.84	1.89	2.709 (3)	166
N3—H3 <i>N</i> ...O2	0.88	2.09	2.675 (3)	124
N6—H6 <i>N</i> ...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) = 1.2*U*_{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) = 1.2*U*_{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: *ORTEP-III* (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

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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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