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Key indicators

Single-crystal X-ray study
 $T = 291$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
Disorder in main residue
 R factor = 0.063
 wR factor = 0.164
Data-to-parameter ratio = 18.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Tetrabutylammonium bromotrichloroferrate(III)

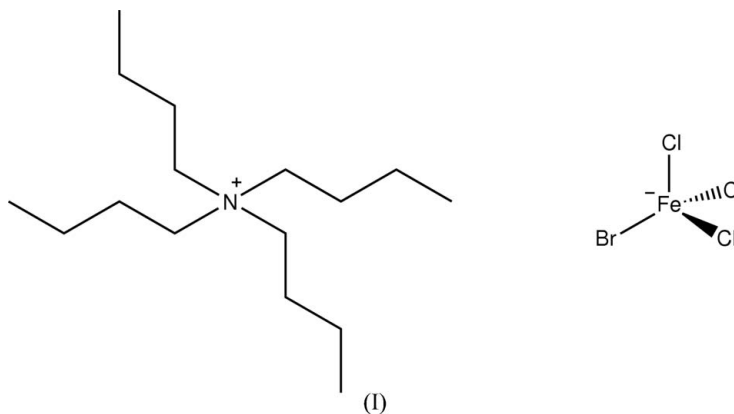
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The Fe^{3+} centre in the complex anion of the title compound, $[(\text{C}_4\text{H}_9)_4\text{N}][\text{FeBrCl}_3]$, is surrounded by three chloride anions and one bromide anion. The Br atom in $[\text{FeBrCl}_3]^-$ is disordered, replacing Cl atoms. The Fe and N atoms lie on a twofold rotation axis.

Comment

Compounds containing high-spin tetrahedrally coordinated Fe ions can constitute simple models for studies of biological systems containing iron–sulfur proteins (Lauher & Ibers, 1975). Tetrahalogenoferrates(III) of quaternary aliphatic amines have frequently been utilized as reactants for the synthesis of this type of model compounds (Smith *et al.*, 2005). Knowledge of the crystal structures of simple tetrahalogenoferrate(III) salts may thus be useful for in-depth interpretation of their physicochemical properties.



The title compound, (I) (Fig. 1), is isostructural with $[(\text{C}_4\text{H}_9)_4\text{N}][\text{FeCl}_4]$ (Hay & Geib, 2005; Wyrzykowski *et al.*, 2006). The Fe^{III} and N atoms lie on a twofold rotation axis. The Br and Cl atoms are mutually disordered, similar to the situation in the $[\text{FeBrCl}_3]^-$ anion of tetraethylammonium bromotrichloroferrate(III) (Evans *et al.*, 1990). The N atom of the tetrabutylammonium cation has an almost ideal tetrahedral arrangement of four *n*-butylammonium groups. Selected geometric parameters of the cation and the anion are shown in Table 1. They are similar to analogous bond lengths and angles reported previously for $[(\text{C}_4\text{H}_9)_4\text{N}][\text{FeCl}_4]$ (Hay & Geib, 2005; Wyrzykowski *et al.*, 2006), $[(\text{C}_2\text{H}_5)_4\text{N}][\text{FeCl}_4]$ and $[(\text{C}_2\text{H}_5)_4\text{N}][\text{FeBrCl}_3]$ (Evans *et al.*, 1990). The shortest $\text{Fe} \cdots \text{Fe}$ distance is 8.211 (1) Å. The cations and anions are packed alternately in the crystal structure (Fig. 2); there are no hydrogen bonds nor any unusual short intermolecular interactions.

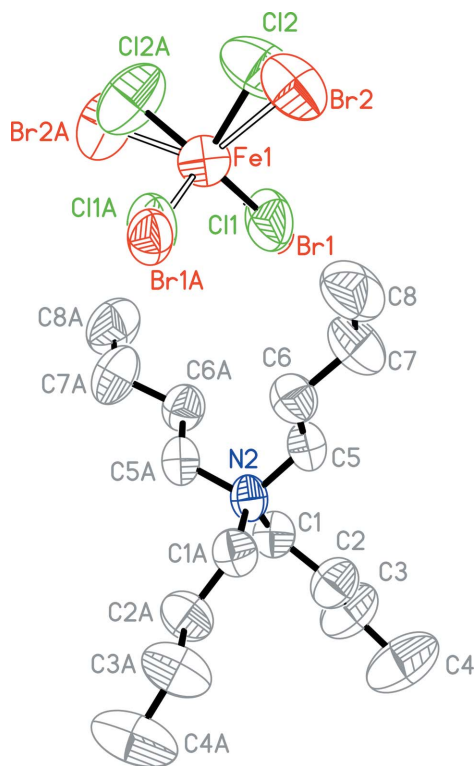


Figure 1
The structure of the ions of the title compound, (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. Both disorder components are shown. [Symmetry code: (A) $x, -y + \frac{1}{2}, -z + \frac{1}{2}$]

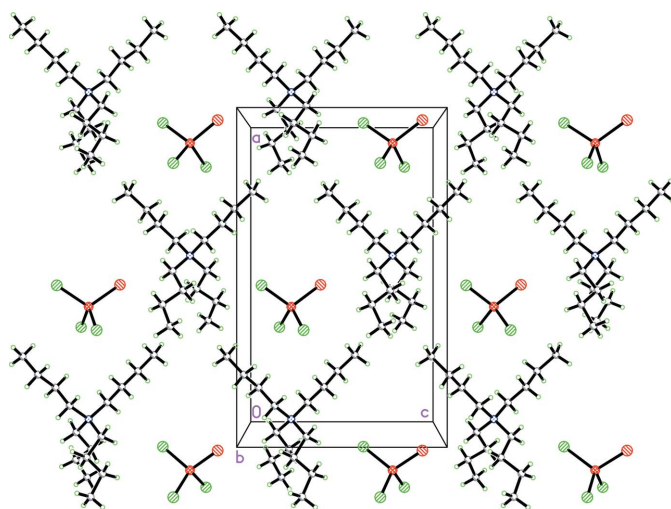


Figure 2
A part of the packing of the title compound, (I), showing the arrangement of cations and anions in the crystal structure. Only one of the four disordered components of the anion is shown.

Experimental

An ethanol solution of ferric chloride was prepared by dissolving *ca* 0.03 mol of FeCl_3 in ethanol (96%). To this solution, a stoichiometric quantity of an ethanol solution of tetrabutylammonium bromide was added. Upon mixing, an amorphous precipitate of $[(\text{C}_4\text{H}_9)_4\text{N}][\text{FeBrCl}_3]$ appeared instantaneously. The precipitate was filtered off,

washed with a small amount of a 1:1 ethanol–diethyl ether mixture, and recrystallized from acetone at ambient temperature. After 7 d, orange crystals appeared. The compound was dried over P_4O_{10} in a vacuum desiccator. Elemental analysis (calculated/found %): C 39.66/39.51, H 7.49/7.47, N 2.89/2.90, Cl 21.95/21.97, Br 16.49/16.44, Fe 11.53/11.34

Crystal data

$(\text{C}_{16}\text{H}_{36}\text{N})[\text{FeBrCl}_3]$
 $M_r = 484.57$
Orthorhombic, $Pnna$
 $a = 18.5191$ (11) Å
 $b = 11.5864$ (7) Å
 $c = 11.4957$ (7) Å
 $V = 2466.6$ (3) Å³

$Z = 4$
 $D_x = 1.305$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 2.55$ mm⁻¹
 $T = 291.2$ (3) K
Prism, orange
 $0.35 \times 0.27 \times 0.14$ mm

Data collection

Kuma KM-4 CCD diffractometer
 ω scans
Absorption correction: numerical
(*X-RED*; Stoe & Cie, 1999)
 $T_{\min} = 0.476$, $T_{\max} = 0.711$

24549 measured reflections
2197 independent reflections
1840 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.089$
 $\theta_{\max} = 25.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.164$
 $S = 1.27$
2197 reflections
121 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0766P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.30$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³

Table 1

Selected bond lengths (Å).

Fe1—Cl1	2.107 (10)	Fe1—Br2	2.337 (6)
Fe1—Cl2	2.172 (6)	Fe1—Br1	2.356 (12)

H atoms were placed in calculated positions ($\text{C—H} = 0.96$ Å) and refined as riding [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{non-methyl C})$ and $1.5U_{\text{eq}}(\text{methyl C})$]. The methyl groups were allowed to rotate about their local threefold axis. The refined occupancy factors of Br and Cl atoms were 0.250 (4) and 0.750 (3), respectively, and at the final refinement cycle, these factors were fixed.

Data collection: *CrysAlis CCD* (UNIL IC & Kuma, 2000); cell refinement: *CrysAlis RED* (UNIL IC & Kuma, 2000); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP in SHELXTL/PC* (Sheldrick, 1990) and *ORTEP-3* (Version 1.062; Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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