Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
2569 measured reflections
1391 independent reflections
1158 reflections with $I > 2\sigma(I)$
$R_{int} = 0.0202$
$\theta_{max} = 27.50°$
$h = -7 \rightarrow 0$
$k = -18 \rightarrow 13$
$l = 0 \rightarrow 18$
3 standard reflections
every 247 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0263$
$wR(F^2) = 0.0776$
$S = 1.060$
1391 reflections
82 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.123P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.275$ e Å^{-3}
$\Delta\rho_{min} = -0.230$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S1—C2	1.7276 (14)	C3a—C7a	1.414 (2)
S1—C7a	1.7308 (13)	C4—C5	1.373 (2)
C2—C3	1.351 (2)	C5—N6	1.354 (2)
C3—C3a	1.433 (2)	N6—C7	1.332 (2)
C3a—C4	1.402 (2)	C7—C7a	1.397 (2)
C2—S1—C7a	90.76 (6)	N6—C5—C4	124.83 (13)
C3—C2—S1	114.04 (11)	C7—N6—C5	117.57 (13)
C2—C3—C3a	112.08 (12)	N6—C7—C7a	122.08 (13)
C4—C3a—C7a	117.29 (12)	C7—C7a—C3a	119.98 (12)
C4—C3a—C3	131.10 (12)	C7—C7a—S1	128.51 (10)
C7a—C3a—C3	111.60 (12)	C3a—C7a—S1	111.51 (9)
C5—C4—C3a	118.25 (13)		

A rigid-body libration analysis (Schomaker & Trueblood, 1968) gave corrections of +0.004 Å for the bonds C2—S1 and C7a—S1, and +0.003 Å for all other bonds. The *RG* value of 0.039 confirms that the rigid-body approximation is a reasonable one.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: FG1264). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Methylhexamethylenetetramine Fluoride Tetrahydrate, MeHMTAF.4H_2O

DAVID J. NIGHTINGALE, LEROY CRONIN AND JAMES H. CLARK

Department of Chemistry, University of York, Heslington, York YO1 5DD, England. E-mail: jhc1@york.ac.uk

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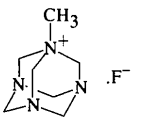
Abstract

The novel title fluoride source, $C_7H_{15}N_4^+.F^-.4H_2O$, was crystallized from methanol. The cations adopt an alternating up–down arrangement and are separated into layers by an extended hydrogen-bonded fluoride–water sheet network.

Comment

In the course of our work producing novel fluoride sources for the fluorodenitration of aromatic compounds,

we synthesized the title compound, MeHMTAF.$4H_2O$ (Clark & Nightingale, 1996). Although structural data are available for a variety of quaternarized hexamethylenetetramine derivatives (Mak, 1984; Chou, Lessinger & Chiang, 1987; Ribár, Mészáros, Vladimirov, Živanov-Stakič & Golič, 1991), none of these examples contains fluoride ions. The structure of the title compound represents the first example of an extended hydrogen-bonded anion–water network involving a hexamethylenetetramine salt, although extended systems with simple tetraalkylammonium fluorides have been reported (Mak, 1985; McLean & Jeffrey, 1967).



MeHMTAF

Within the lattice, the cations adopt an up–down arrangement sandwiched between a fluoride–water hydrogen-bond network [shortest $N^+\cdots F^-$ distance is 4.22 (1) Å]. The network extends in the *ab* plane and consists of fluorides forming four hydrogen bonds with water, the distances ranging between 2.634 (8) and 2.681 (9) Å. In addition, each water molecule makes three hydrogen bonds with other fluoride ions or water molecules. The O···O distances of the water molecules are in the range 2.718 (9)–2.825 (10) Å. Angles between hydrogen-bonded water with fluoride as the central atom range between 86.9 (3) and 136.2 (3)°, whereas angles with oxygen as the central atom range between 93.0 (2) and 120.9 (3)°. The network (Fig. 2) consists of buckled edge-sharing polyhedra, *i.e.* a pentagon containing two fluoride ions, a further pentagon containing a single fluoride and thirdly, a hexagon containing one fluoride.

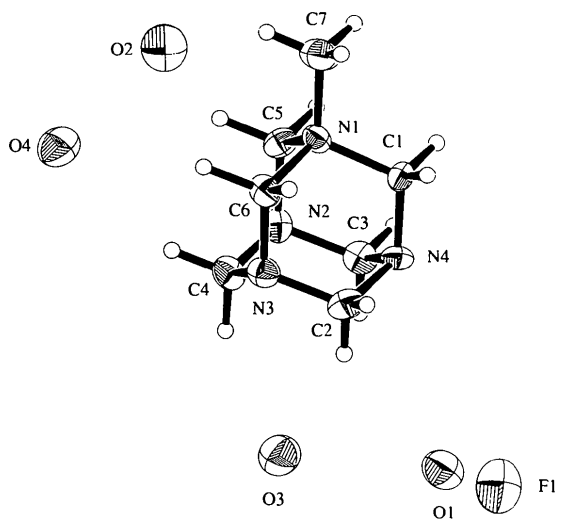


Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids.

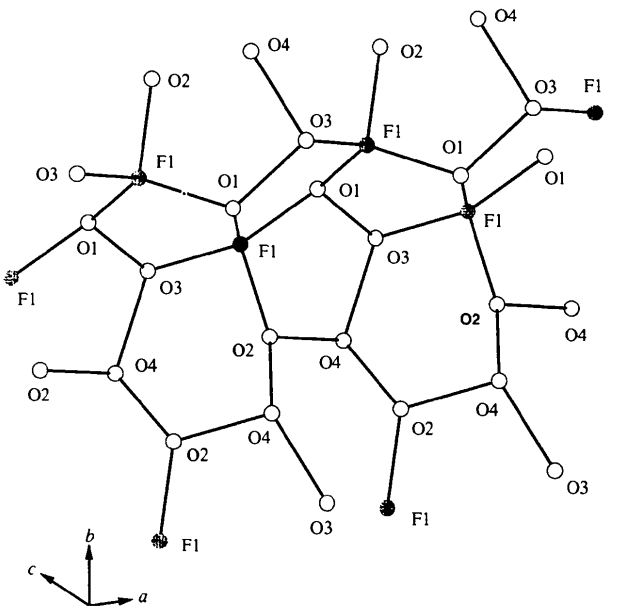


Fig. 2. Schematic diagram showing a section of the hydrogen-bond network. Note the fluoride is four-coordinate within the network of polyhedra.

Bond lengths and angles for the cation are similar to those reported by Ribár, Mészáros, Vladimirov, Živanov-Stakić & Golič (1991) and the largest uncertainty on an N—C bond is 0.010 Å.

Experimental

The synthesis of the title compound was performed as previously reported (Clark & Nightingale, 1996) with methanol as the recrystallization solvent.

Crystal data

$C_7H_{15}N_4^+.F^-.4H_2O$
M_r = 246.29
Monoclinic
$P2_1/c$
a = 9.854 (4) Å
b = 6.352 (4) Å
c = 19.372 (6) Å
β = 90.52 (3)°
V = 1212.4 (9) Å^3
Z = 4
D_x = 1.349 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 20 reflections
θ = 4.63–8.81°
μ = 0.117 mm^{-1}
T = 293 (2) K
Needle
0.4 × 0.1 × 0.1 mm
Colourless

Data collection

Rigaku AFC-6*S* diffractometer
ω–2θ scans
Absorption correction: none
1848 measured reflections
1848 independent reflections
616 reflections with $I > 2\sigma(I)$

θ_{max} = 25°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 7$
$l = -22 \rightarrow 23$
3 standard reflections every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0828$
$wR(F^2) = 0.2541$
$S = 0.993$
1848 reflections
152 parameters
H atoms: riding model

$w = 1/[\sigma^2(F_o^2) + (0.0961P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.16$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

It was not possible to locate the eight water protons and thus unambiguously identify the fluoride ion. We therefore performed five separate refinements with the fluoride in each of the five possible positions. The final model was chosen because it had the lowest s.u.'s (both in the bond lengths and intermolecular contacts) and R value. In addition, the chosen model placed the fluoride such that it made four contacts with solvent molecules at distances of 2.63–2.68 Å; in the four other models, the fluoride made only three contacts with distances of 2.63–2.83 Å. Furthermore, in the alternative refinements, some distances contracted giving O···O distances which were shorter than the F···O distances. It should be noted that as the data set was collected at room temperature and the crystal diffracted weakly, the proportion of observed data was rather low. This has probably contributed to the problems in locating the water H atoms.

Data collection: *TEXSAN* (Molecular Structure Corporation, 1992). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BM1129). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-[(2-Iodophenyl)iminomethyl]phenol

AYHAN ELMALI AND YALÇIN ELERMAN

Department of Physics Engineering, Faculty of Sciences, University of Ankara, 06100 Besevler, Ankara, Turkey.
E-mail: elmali@science.ankara.edu.tr

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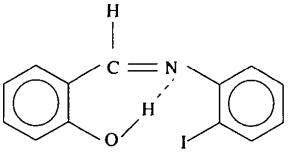
Abstract

Molecules of the title compound, $C_{13}H_{10}INO$, are non-planar and contain an intramolecular O—H···N hydrogen bond.

Comment

Schiff bases have been widely used as ligands in the formation of transition metal complexes (Calligaris & Randaccio, 1987). N-Substituted salicylaldimines are also of interest because of their thermochromism and photochromism in the solid state which may involve reversible proton transfer from the hydroxyl O to the imine N atom (Moustakali, Mavridis & Hadjoudis, 1978; Hadjoudis, Vitterakis & Mavridis, 1987; Xu, You, Sun, Wang & Liu, 1994) and charge transport occurs through intermolecular overlap between π orbitals.

Our structural investigations of Schiff bases (Elerman, Svoboda & Fuess, 1991; Elerman, Paulus, Svoboda & Fuess, 1992; Elerman, Elmali, Kabak, Aydin & Peder, 1994; Elerman, Elmali & Svoboda, 1995; Elmali, Özbey, Kendi, Kabak & Elerman, 1995; Kevran, Elmali & Elerman, 1996) have led us to examine the title compound, (I) (Fig. 1). The space group and lattice constants of the title compound were determined previously (Bernstein, 1967) and are consistent with the corresponding values presented here. It has been proposed that Schiff base molecules exhibiting thermochromy are planar, while those exhibiting photochromy are non-planar (Moustakali *et al.*, 1978). In agreement with this, the title molecule is non-planar and photochromic (Bernstein, 1967); moieties *A* (O1, C1–C7) and *B* (N1, C8–C13, I1) [both planar with a maximum deviation of 0.025 (4) Å] are inclined at an angle of 45.7 (1)° reflecting mainly the twist about N1—C8 [C7—N1—C8—C9 43.8 (6)°]. Clearly this conformation is not suitable for direct coordination to a metal ion.



(I)