Data collection
Siemens P4 diffractometer
ω scans
Absorption correction: none
2569 measured reflections
1391 independent reflections
1158 reflections with
I > 2σ(I)
Rint = 0.0202

Refinement
Refinement on F2
R[F2 > 2σ(F2)] = 0.0263
wR(F2) = 0.0776
S = 1.060
1391 reflections
82 parameters
H atoms riding

Table 1. Selected geometric parameters (Å, °)

<table>
<thead>
<tr>
<th>Bond</th>
<th>Distance (Å)</th>
<th>Angle (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1-C2</td>
<td>1.7276 (14)</td>
<td>1.433 (2)</td>
</tr>
<tr>
<td>C1-C2</td>
<td>1.433 (2)</td>
<td>1.351 (2)</td>
</tr>
<tr>
<td>C2-C3</td>
<td>1.402 (2)</td>
<td>1.402 (2)</td>
</tr>
<tr>
<td>C3-C4</td>
<td>1.332 (2)</td>
<td>1.354 (2)</td>
</tr>
<tr>
<td>C4-C5</td>
<td>1.373 (2)</td>
<td>1.373 (2)</td>
</tr>
<tr>
<td>C5-N6</td>
<td>1.354 (2)</td>
<td>1.354 (2)</td>
</tr>
<tr>
<td>C7-N6</td>
<td>1.332 (2)</td>
<td>1.332 (2)</td>
</tr>
<tr>
<td>C7-N6</td>
<td>1.397 (2)</td>
<td>1.397 (2)</td>
</tr>
</tbody>
</table>

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.

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References


Methyloxamethylenetetramine Fluoride Tetrahydrate, MeHMTAF.4H2O

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

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

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Abstract
The novel title fluoride source, C7H15N4+·F−·4H2O, 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·4H₂O (Clark & Nightingale, 1996). Although structural data are available for a variety of quaternized hexamethylene-tetramine derivatives (Mak, 1984; Chou, Lessinger & Chiang, 1987; Ribár, Mészáros, Vladimirov, Živanov-Stakić & 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).

Within the lattice, the cations adopt an up–down arrangement sandwiched between a fluoride–water hydrogen-bond network [shortest Nw–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.

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**

\[
\begin{align*}
C_7\text{H}_{15}\text{N}_4\text{F}^-\cdot4\text{H}_2\text{O} \\
M_r &= 246.29 \\
\text{Monoclinic} \\
P2_1/c \\
a &= 9.854 (4) \text{ Å} \\
b &= 6.352 (4) \text{ Å} \\
c &= 19.372 (6) \text{ Å} \\
\beta &= 90.52 (3)° \\
V &= 1212.4 (9) \text{ Å}^3 \\
Z &= 4 \\
D_t &= 1.349 \text{ Mg m}^{-3} \\
D_m &\text{ not measured}
\end{align*}
\]

Mo Kα radiation
\[
\begin{align*}
\lambda &= 0.71069 \text{ Å} \\
\theta_{\text{max}} &= 25° \\
h &= 0 \rightarrow 10 \\
k &= 0 \rightarrow 7 \\
l &= -22 \rightarrow 23
\end{align*}
\]

**Data collection**

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

Colourless
**References**


