ORGANIC COMPOUNDS

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1-Adeninium 2-Fluorobenzoate Monohydrate

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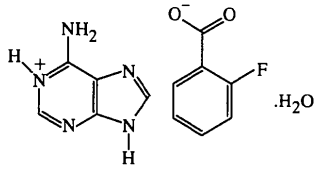
Abstract

The title compound, $C_5H_6N_5^+.C_7H_4FO_2^-.H_2O$, is the product of the acid–base reaction of 2-fluorobenzoic acid and adenine in water. A layered structure held together by interionic N—H···O and N—H···N bonds is formed. Water molecules, which link adjacent layers, participate in two O—H···O bonds and one O···H—N bond.

Comment

The compound, (I), formed from 2-fluorobenzoic acid and adenine was expected to have a large number of hydrogen bonds. The structure was determined in order to see if the F atom participates in the hydrogen-bond network.



(I)

Crystals grown from aqueous solution proved to contain one molecule of water for each acid–base pair (Fig. 1). Six hydrogen bonds are formed (see Table 2), but none involves the F atom. All possible donors participate in the hydrogen bonding, while two acceptor sites are unused; the water O atom and one of the carboxylate O atoms could each accept a second proton.

The hydrogen-bond network is complicated and three-dimensional; part of it is shown in Fig. 2. All ions are approximately parallel and are arranged in layers. Adeninium ions form dimers around inversion centers; the two cations of the dimer, together with the attached fluorobenzoate ions, form a four-ion packing unit. The water molecules, each of which participates in three hydrogen bonds, link packing units both within and between layers.

There is a shorter than average contact between the O1*W* and F2*B* atoms of 3.021 (2) Å, but this same O1*W* atom is substantially closer to the nearby O2*B* atom [2.805 (2) Å].

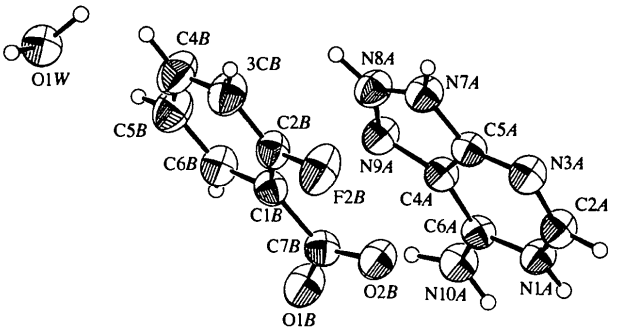


Fig. 1. A formula unit of 1-adeninium 2-fluorobenzoate monohydrate. The displacement ellipsoids are at the 50% probability level.

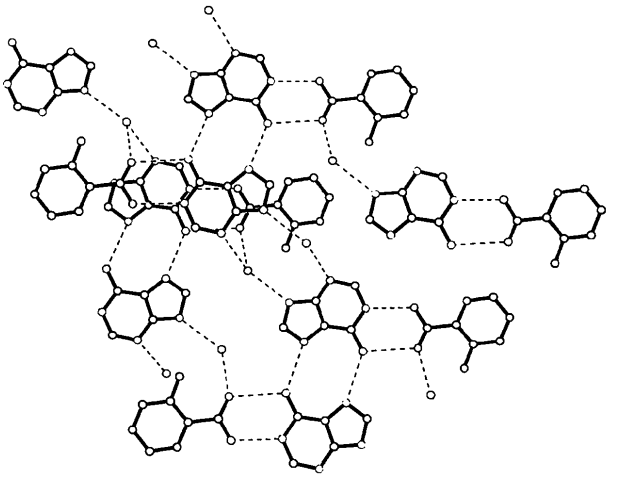


Fig. 2. Diagram showing part of the hydrogen-bond network.

Experimental

2-Fluorobenzoic acid (0.26 g) was dissolved in water (20 ml) and adenine (0.24 g) was added. The mixture was stirred and heated to 338 K. After about 10 min, the solution became clear, whereupon it was filtered and transferred to an evaporating dish for crystallization.

Crystal data

$C_5H_6N_5^+.C_7H_4FO_2^-.H_2O$ — Mo $K\alpha$ radiation
M_r = 293.27 — λ = 0.71069 Å

† Deceased, 1994.

Triclinic
$P\bar{1}$
a = 7.2790 (10) Å
b = 8.455 (2) Å
c = 11.408 (2) Å
α = 67.730 (10)°
β = 85.150 (10)°
γ = 84.51 (2)°
V = 645.9 (2) Å^3
Z = 2
D_x = 1.508 Mg m^{-3}
D_m = 1.52 Mg m^{-3}
D_m measured by flotation in benzene/1-bromo-2-chlorobenzene

Cell parameters from 25 reflections
θ = 11.8–18.8°
μ = 0.122 mm^{-1}
T = 293 (2) K
Block
0.56 × 0.31 × 0.28 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.89, T_{max} = 0.97
2399 measured reflections
2399 independent reflections

2002 reflections with $I > 2\sigma(I)$
θ_{max} = 26.96°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 10$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 240 min
intensity decay: −0.2%

Refinement

Refinement on F^2
$R(F)$ = 0.045
$wR(F^2)$ = 0.124
S = 1.05
2399 reflections
192 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.1525P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1A—C2A	1.344 (2)	O1B—C7B	1.283 (2)
N1A—C6A	1.358 (2)	O2B—C7B	1.227 (2)
C2A—N3A	1.313 (3)	C7B—C1B	1.495 (3)
N3A—C5A	1.347 (2)	C1B—C6B	1.387 (3)
C4A—N9A	1.379 (2)	C1B—C2B	1.389 (3)
C4A—C5A	1.384 (2)	C2B—F2B	1.354 (2)
C4A—C6A	1.400 (3)	C2B—C3B	1.367 (3)
C5A—N7A	1.362 (2)	C3B—C4B	1.376 (3)
C6A—N10A	1.320 (2)	C4B—C5B	1.379 (3)
N7A—C8A	1.349 (3)	C5B—C6B	1.367 (3)
C8A—N9A	1.314 (3)		
C2A—N1A—C6A	121.2 (2)	O2B—C7B—O1B	123.9 (2)
N3A—C2A—N1A	127.1 (2)	O2B—C7B—C1B	121.9 (2)
C2A—N3A—C5A	111.7 (2)	O1B—C7B—C1B	114.2 (2)
N9A—C4A—C5A	111.0 (2)	C6B—C1B—C2B	116.2 (2)
N9A—C4A—C6A	131.5 (2)	C6B—C1B—C7B	120.2 (2)
C5A—C4A—C6A	117.5 (2)	C2B—C1B—C7B	123.6 (2)
N3A—C5A—N7A	128.0 (2)	F2B—C2B—C3B	117.3 (2)
N3A—C5A—C4A	126.8 (2)	F2B—C2B—C1B	119.9 (2)
N7A—C5A—C4A	105.2 (2)	C3B—C2B—C1B	122.8 (2)
N10A—C6A—C4A	125.1 (2)	C2B—C3B—C4B	119.0 (2)
N1A—C6A—C4A	115.6 (2)	C3B—C4B—C5B	120.3 (2)
C8A—N7A—C5A	106.6 (2)	C6B—C5B—C4B	119.3 (2)
N9A—C8A—N7A	114.0 (2)	C5B—C6B—C1B	122.4 (2)
C8A—N9A—C4A	103.2 (2)		

Table 2. *Contact distances (Å)*

N1A···O1B^{i}	2.560 (2)	O1W···N3A^{iii}	2.895 (2)
N7A···O1W^{ii}	2.751 (2)	N10A···O2B^{i}	2.867 (2)
O1W···O2B^{iii}	2.805 (2)	N10A···N9A^{iv}	2.994 (2)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XPMA* and *ZORTEP* (Zsolnai, 1995).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BK1179). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany
Zsolnai, L. (1995). *XPMA* and *ZORTEP. Interactive Graphics Programs*. University of Heidelberg, Germany.

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N,N'-Bis(pentafluorophenyl)urea

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Abstract

In the crystal structure of the title compound, $C_{13}H_2F_{10}N_2O$, there is a close contact of a H atom and an F atom as a three-centered hydrogen-bonding interaction.

† Deceased, 1994.