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[2′-(2-Formyl-4-nitroanilino)ethyl]dimethylammonium Fluoroborate

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Abstract. $C_{11}H_{16}NO_3^+BF_4^-$, monoclinic, $P2_1/n$, $a = 7{\cdot}958\ (5)$, $b = 16{\cdot}277\ (8)$, $c = 11{\cdot}267\ (5)$ Å, $\beta = 98{\cdot}41\ (4)°$, $U = 1444$ Å^3, $Z = 4$, $D_x = 1{\cdot}50$ g cm^{-3}. The structure was solved by direct methods and refined to an R of 0·039 for 1381 unique two-circle diffractometer data.

Introduction. The structure of the title compound was undertaken in connexion with studies on the conformation of the organic residue. In its unprotonated state it can be regarded as a novel ligand capable of complexing with transition-metal ions (Hughes, 1975).

3646 reflexions were measured with an automated two-circle Stoe diffractometer, Mo $K\alpha$ radiation, graphite monochromator and two crystals 0·20 × 0·15 × 0·08 mm (layers h0–16l) and 0·25 × 0·18 × 0·10 mm (layers hk0–8). After interlayer scale factors had been found by linear least squares, equivalent reflexions were merged to yield 1381 unique data with $F > 5\sigma(F)$ based on counting statistics. Absorption corrections were not applied ($\mu = 0{\cdot}96$ cm^{-1}). Cell dimensions were found by a least-squares fit to 512 diffractometer ω angle measurements for the $h0l$ and hk0 zero-layer reflexions.

All the non-hydrogen atoms were found by multi-solution Σ_2 sign expansion, and the structure was refined by full-matrix least squares with all non-hydrogen atoms anisotropic. H atom coordinates were calculated geometrically; those attached to C(11), N(2) and N(3) were refined freely, whilst the rest were constrained so that the C–H vectors remained constant,

Table 1. *Atom coordinates* (× 10^4)

	x	y	z
C(1)	2407 (4)	5702 (2)	1202 (3)
C(2)	2522 (4)	5036 (2)	479 (3)
C(3)	1696 (3)	4302 (2)	689 (3)
C(4)	772 (3)	4245 (2)	1678 (3)
C(5)	663 (4)	4951 (2)	2386 (3)
C(6)	1453 (4)	5665 (2)	2149 (3)
C(7)	−844 (4)	3401 (2)	2944 (3)
C(8)	−2681 (4)	3683 (2)	2659 (3)
C(9)	−3047 (5)	3955 (2)	4774 (4)
C(10)	−5508 (4)	3663 (3)	3260 (4)
C(11)	1878 (4)	3639 (2)	−127 (3)
O(1)	3012 (4)	7060 (2)	1595 (3)
O(2)	4210 (3)	6469 (2)	229 (3)
O(3)	1269 (3)	2949 (1)	−55 (2)
N(1)	3267 (3)	6460 (2)	996 (3)
N(2)	21 (3)	3530 (2)	1916 (3)
N(3)	−3672 (3)	3497 (2)	3652 (2)
B(1)	2570 (5)	3649 (2)	6486 (4)
F(1)	1554 (3)	3187 (1)	7130 (2)
F(2)	3432 (3)	4217 (1)	7251 (2)
F(3)	3673 (3)	3135 (2)	6032 (2)
F(4)	1579 (4)	4063 (2)	5582 (2)
H(1)	3252 (4)	5075 (2)	−255 (3)
H(2)	−55 (4)	4927 (2)	3127 (3)
H(3)	1340 (4)	6201 (2)	2695 (3)
H(4)	−814 (4)	2756 (2)	3171 (3)
H(5)	−211 (4)	3750 (2)	3694 (3)
H(6)	−3270 (4)	3375 (2)	1856 (3)
H(7)	−2701 (4)	4338 (2)	2510 (3)
H(8)	−3020 (5)	4611 (2)	4643 (4)
H(9)	−1775 (5)	3735 (2)	5074 (4)
H(10)	−3848 (5)	3812 (2)	5442 (4)
H(11)	−5693 (4)	4309 (3)	3070 (4)
H(12)	−6242 (4)	3484 (3)	3951 (4)
H(13)	−5918 (4)	3312 (3)	2456 (4)
H(14)	2465 (46)	3778 (21)	−802 (36)
H(15)	189 (46)	3111 (23)	1462 (36)
H(16)	−3581 (44)	2911 (23)	3877 (34)

Table 2. *Anisotropic temperature factors* (Å^2 × 10^3)

The temperature factor exponent takes the form: $[-2\pi^2(U_{11}h^2a^{*2} + \cdots + 2U_{12}hka^*b^*)]$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	34 (1)	37 (2)	46 (2)	3 (2)	1 (1)	−5 (1)
C(2)	32 (1)	43 (2)	37 (2)	3 (1)	2 (1)	1 (1)
C(3)	31 (1)	36 (1)	36 (2)	−2 (1)	7 (1)	2 (1)
C(4)	31 (1)	36 (1)	33 (2)	−1 (1)	4 (1)	3 (1)
C(5)	36 (2)	45 (2)	40 (2)	−6 (1)	9 (1)	2 (1)
C(6)	41 (2)	40 (2)	45 (2)	−10 (2)	1 (2)	0 (1)
C(7)	39 (1)	41 (2)	39 (2)	6 (1)	15 (1)	5 (1)
C(8)	40 (1)	38 (1)	33 (2)	7 (1)	13 (1)	3 (1)
C(9)	62 (2)	75 (2)	49 (2)	−10 (2)	22 (2)	−11 (2)
C(10)	37 (2)	90 (3)	73 (3)	20 (2)	12 (2)	10 (2)
C(11)	42 (2)	46 (2)	42 (2)	−2 (2)	14 (2)	2 (1)
O(1)	77 (2)	49 (1)	108 (2)	−20 (2)	19 (2)	−22 (1)
O(2)	62 (2)	68 (2)	63 (2)	11 (1)	11 (1)	−21 (1)
O(3)	70 (2)	42 (1)	56 (2)	−12 (1)	30 (1)	−7 (1)
N(1)	44 (2)	49 (2)	61 (2)	2 (2)	−1 (2)	−11 (1)
N(2)	47 (1)	35 (1)	43 (2)	−1 (1)	20 (1)	3 (1)
N(3)	36 (1)	36 (1)	37 (2)	5 (1)	8 (1)	0 (1)
B(1)	52 (2)	41 (2)	45 (3)	0 (2)	15 (2)	7 (2)
F(1)	86 (2)	65 (1)	92 (2)	−3 (1)	50 (1)	0 (1)
F(2)	97 (2)	70 (1)	81 (2)	−2 (1)	−4 (1)	−12 (1)
F(3)	98 (2)	75 (2)	94 (2)	16 (1)	59 (2)	32 (1)
F(4)	102 (2)	81 (2)	69 (2)	9 (1)	−11 (1)	23 (1)

Table 3. *Bond lengths* (Å)

C(2)–C(1)	1·367 (5)	C(3)–C(2)	1·400 (5)
C(4)–C(3)	1·425 (5)	C(5)–C(4)	1·407 (5)
C(6)–C(5)	1·366 (5)	C(6)–C(1)	1·399 (5)
N(2)–C(4)	1·354 (5)	N(2)–C(7)	1·447 (5)
C(8)–C(7)	1·521 (6)	N(3)–C(8)	1·490 (5)
N(3)–C(9)	1·489 (5)	N(3)–C(10)	1·488 (5)
N(1)–C(1)	1·446 (5)	N(1)–O(1)	1·222 (5)
N(1)–O(2)	1·224 (5)	C(11)–C(3)	1·439 (5)
O(3)–C(11)	1·230 (5)	F(1)–B(1)	1·385 (6)
F(2)–B(1)	1·376 (6)	F(3)–B(1)	1·365 (5)
F(4)–B(1)	1·371 (6)	H(14)–C(11)	0·976 (41)
H(15)–N(2)	0·873 (41)	H(16)–N(3)	0·987 (39)

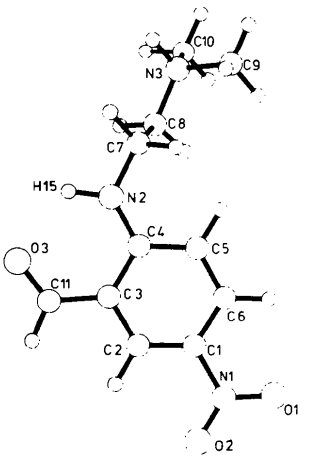

Fig. 1. The [2′-(2-formyl-4-nitroanilino)ethyl]dimethylammonium cation.

Table 4. *Bond angles* (°)

C(6)–C(1)–C(2)	120·7 (4)	F(4)–B(1)–F(1)	109·9 (4)	C(10)–N(3)–C(9)	110·7 (4)
C(4)–C(3)–C(2)	119·7 (4)	F(4)–B(1)–F(2)	108·4 (4)	N(1)–C(1)–C(6)	118·9 (4)
C(6)–C(5)–C(4)	121·0 (4)	H(14)–C(11)–C(3)	115·4 (22)	O(2)–N(1)–C(1)	118·5 (4)
N(2)–C(4)–C(3)	120·2 (4)	H(15)–N(2)–C(4)	116·5 (26)	C(11)–C(3)–C(2)	116·0 (4)
C(7)–N(2)–C(4)	123·9 (4)	H(16)–N(3)–C(8)	111·2 (23)	O(3)–C(11)–C(3)	124·4 (4)
N(3)–C(8)–C(7)	112·2 (3)	H(16)–N(3)–C(10)	106·4 (21)	F(3)–B(1)–F(1)	108·7 (4)
C(10)–N(3)–C(8)	110·2 (4)	C(3)–C(2)–C(1)	120·2 (4)	F(3)–B(1)–F(2)	110·9 (4)
N(1)–C(1)–C(2)	120·4 (4)	C(5)–C(4)–C(3)	118·1 (4)	F(4)–B(1)–F(3)	110·8 (4)
O(1)–N(1)–C(1)	118·2 (4)	C(5)–C(6)–C(1)	120·2 (4)	O(3)–C(11)–H(14)	120·1 (22)
O(2)–N(1)–O(1)	123·3 (4)	N(2)–C(4)–C(5)	121·6 (4)	H(15)–N(2)–C(7)	119·2 (26)
C(11)–C(3)–C(4)	124·3 (4)	N(2)–C(7)–C(8)	110·1 (4)	H(16)–N(3)–C(9)	105·2 (23)
F(2)–B(1)–F(1)	108·2 (4)	C(9)–N(3)–C(8)	112·9 (3)		

but the C atoms were free to move. The common isotropic H atom temperature factor refined to 0·063 (3) Å^2. Complex neutral atom scattering factors were employed, with the weighting scheme $w = 1{\cdot}0/[\sigma^2(F) + 0{\cdot}015F_o^2]$ which led to a flat analysis of variance. The final value of $R' = \Sigma w^{1/2}\Delta/\Sigma w^{1/2}|F_o|$ was 0·045 and R, 0·039. Final atomic coordinates are given in Table 1 and temperature factors in Table 2; the bond lengths and angles are in Tables 3 and 4.* Fig. 1 shows the cation.

Discussion. The crystal structure determination confirms the proposed structure and hence the synthetic route employed in the preparation of the title compound. The nitro and aldehyde groups are virtually coplanar with the aromatic ring, and there is a possibility of intramolecular hydrogen bonding between the amino and aldehyde groups [O(3)···H(15) 2·04 Å]. The side chain is fully staggered, and the plane of the *trans* N(2)–C(7)–C(8)–N(3) moiety makes an angle of 86° with the mean plane of the aromatic ring. The fluoroborate group is tetrahedral, with a mean B–F length of 1·374 Å.

We are grateful to the Science Research Council for providing the diffractometer, and for financial support to JJG. The calculations were performed on the Cambridge University IBM 370/165 computer with programs written by GMS, and the figure was drawn with *PLUTO* written by Dr W. D. S. Motherwell.

Reference

HUGHES, M. (1975). Ph.D. Thesis, Univ. of Cambridge.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32071 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.