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Structure of 1-(1-Phenylethenyl)pyridinium Tetrafluoroborate

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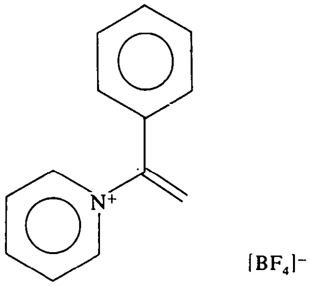
Abstract. $C_{13}H_{12}N^+.BF_4^-$, $M_r = 269{\cdot}048$, orthorhombic, $P2_12_12_1$, $a = 14{\cdot}687\,(1)$, $b = 10{\cdot}327\,(1)$, $c = 8{\cdot}440\,(1)$ Å, $V = 1280{\cdot}1\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}396$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 10{\cdot}371$ cm^{-1}, $F(000) = 552$, room temperature, $R = 0{\cdot}058$ and $wR = 0{\cdot}057$ for 1211 observed reflections of 1265 unique data. The methylene link is well determined and the cation group is not planar, so there is no electronic resonance and the positive charge is localized on the pyridine N atom. The anion displays an irregular tetrahedral geometry as is usual in this group.

Experimental. The title compound was provided by Professor J. Alvarez-Builla, Universidad Alcalá de Henares, Madrid, Spain, who obtained the crystals as part of a general project on the search for new potential antihelmintic agents from pyridinium compounds.



Unit-cell parameters were obtained from least-squares refinement of 53 reflections with $\theta_{\max} = 45°$, measured on a Philips PW1100 four-circle diffractometer, with graphite-monochromated Cu $K\alpha$ radiation.

Intensities were measured from a crystal $0{\cdot}20 \times 0{\cdot}20 \times 0{\cdot}27$ mm on the same diffractometer for $2 < \theta < 65°$, $h = 0$ to 18, $k = 0$ to 12, $l = 0$ to 10. ω–2θ scan technique, detector apertures 1×1 mm. Two standard reflections measured every 90 min, no indication of specimen decay. Of the 1265 independent reflections measured, 1211 were considered as observed with $I > 2\sigma(I)$, σ evaluated from counting statistics.

Lorentz–polarization corrections were applied, absorption ignored; scattering and anomalous-dispersion factors were taken from *International Tables for X-ray Crystallography* (1974); direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and subsequent difference Fourier synthesis calculations and refinement with full-matrix least squares on F_o. All H atoms were located in a difference Fourier map. Refinement using anisotropic and isotropic temperature factors for non-H and H atoms respectively yielded $R = 0{\cdot}058$ and $wR = 0{\cdot}057$ with w calculated by an empirical scheme fitted so as to give no trends in $\langle w\Delta^2F_o\rangle$ *vs* $\langle |F_o| \rangle$ and $\langle (\sin\theta)/\lambda \rangle$. Number of variables 220, max. shift/e.s.d. = 0·015. Highest peak in final difference synthesis 0·20 e Å^{-3}, $S = 1{\cdot}10$.

The computations were performed with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976), *PARST* (Nardelli, 1983) and *PESOS* (Martínez-Ripoll & Cano, 1975) programs on a VAX11/750 computer. The atomic coordinates are given in Table 1 and bond lengths and angles in Table 2.* Fig. 1 shows the structure with the numbering scheme used.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$)

$$U_{\mathrm{eq}} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N1	0·4473 (2)	0·4805 (3)	0·7352 (4)	41 (1)
C2	0·4657 (3)	0·4681 (4)	0·8901 (5)	51 (1)
C3	0·4119 (3)	0·5249 (4)	1·0021 (5)	58 (1)
C4	0·3388 (3)	0·5966 (5)	0·9551 (5)	59 (1)
C5	0·3192 (3)	0·6089 (5)	0·7956 (6)	63 (1)
C6	0·3742 (3)	0·5498 (4)	0·6868 (5)	54 (1)
C7	0·5057 (3)	0·4219 (4)	0·6142 (5)	46 (1)
C8	0·5917 (3)	0·4561 (5)	0·6090 (8)	74 (2)
C9	0·4593 (2)	0·3285 (3)	0·5093 (5)	42 (1)
C10	0·3905 (2)	0·2484 (4)	0·5662 (5)	46 (1)
C11	0·3512 (3)	0·1571 (4)	0·4679 (6)	54 (1)
C12	0·3774 (3)	0·1462 (4)	0·3124 (6)	60 (1)
C13	0·4460 (4)	0·2258 (5)	0·2546 (5)	64 (1)
C14	0·4868 (3)	0·3169 (4)	0·3511 (5)	54 (1)
B	0·7057 (3)	0·2699 (5)	0·1128 (5)	50 (1)
F1	0·6399 (3)	0·3116 (4)	0·0132 (5)	108 (1)
F2	0·7104 (2)	0·3487 (3)	0·2448 (4)	86 (1)
F3	0·6811 (4)	0·1471 (3)	0·1622 (4)	105 (2)
F4	0·7871 (3)	0·2573 (6)	0·0438 (5)	125 (2)

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44530 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. *Molecular geometry*

Bond distances (Å)

N1–C2	1·341 (5)	N1–C6	1·353 (5)
N1–C7	1·465 (5)	C2–C3	1·364 (6)
C3–C4	1·363 (7)	C4–C5	1·383 (7)
C5–C6	1·367 (7)	C7–C8	1·312 (6)
C7–C9	1·476 (5)	C9–C10	1·391 (5)
C9–C14	1·401 (6)	C10–C11	1·382 (6)
C11–C12	1·373 (7)	C12–C13	1·389 (7)
C13–C14	1·381 (7)	B–F1	1·352 (6)
B–F2	1·382 (6)	B–F3	1·384 (6)
B–F4	1·336 (6)		

Bond angles (°)

C6–N1–C7	118·2 (3)	C2–N1–C7	121·5 (3)
C2–N1–C6	120·3 (4)	N1–C2–C3	121·2 (4)
C2–C3–C4	119·2 (4)	C3–C4–C5	119·8 (4)
C4–C5–C6	119·4 (5)	N1–C6–C5	120·1 (4)
N1–C7–C9	114·7 (3)	N1–C7–C8	118·4 (4)
C8–C7–C9	126·9 (4)	C7–C9–C14	119·6 (3)
C7–C9–C10	121·1 (4)	C10–C9–C14	119·2 (3)
C9–C10–C11	120·1 (4)	C10–C11–C12	120·9 (4)
C11–C12–C13	119·4 (4)	C12–C13–C14	120·8 (4)
C9–C14–C13	119·7 (4)	F1–B–F2	110·5 (4)
F1–B–F3	107·0 (4)	F1–B–F4	113·6 (4)
F2–B–F3	108·0 (4)	F2–B–F4	111·4 (4)
F3–B–F4	106·0 (4)		

Some torsion angles (°)

C6–N1–C7–C8	119·9 (5)	C2–N1–C7–C8	−59·1 (6)
C6–N1–C7–C9	−60·2 (5)	C2–N1–C7–C9	120·8 (4)
N1–C7–C9–C10	−35·1 (5)	N1–C7–C9–C14	147·1 (4)
C8–C7–C9–C10	144·9 (5)	C8–C7–C9–C14	−33·0 (6)

Short contacts involving F atoms (Å)

C14···F2(x,y,z)	3·419 (5)	C11···F2($-\frac{1}{2}+x, \frac{1}{2}-y, 1-z$)	3·127 (6)
C2···F1($x,y,1+z$)	3·200 (6)	C12···F2($1-x, -\frac{1}{2}+y, \frac{1}{2}-z$)	3·367 (6)
C6···F3($1-x,\frac{1}{2}+y, \frac{1}{2}-z$)	3·216 (6)		

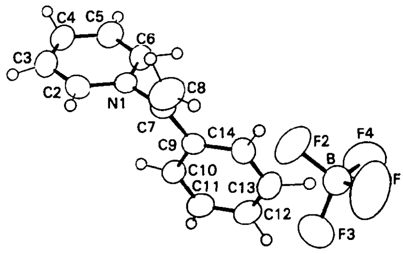

Fig. 1. A view of the molecule with the numbering scheme.

Related literature. No reports on the structure of phenylethenyl derivatives have been found. Previous structure determination of styrylpyridinium salts: Alvarez-Builla, Novella, Galvez, Smith, Florencio, Garcia-Blanco, Bellanato & Santos (1986). The structural geometry of the tetrafluoroborate is comparable with that of Allmam & Waśkowska (1981). Examples of the synthesis of 1-vinylpyridine salts are described by Relles (1973) and Novella & Alvarez-Builla (1985).

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Structure of 1,2:5,6-Di-*O*-isopropylidene-α-D-gulofuranose

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Abstract. $C_{12}H_{20}O_6$, $M_r = 260{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}621$ (2), $b = 12{\cdot}665$ (3), $c = 18{\cdot}680$ (4) Å, $V = 1329{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}300$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}78$ mm^{-1}, $F(000) = 560$, $T = 293$ K, $R = 0{\cdot}039$ for 1145 unique observed reflections [$F > 3\sigma(F)$]. The fused five-membered rings both have envelope conformations with maximum distances from the best least-squares plane for each ring of −0·215 (2) Å for O4 and 0·132 (2) Å for O1 and an angle between the planes of 122·6 (8)°. The largest deviation from the best plane in the third ring is −0·187 (2) Å for O6. The single hydroxyl group

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