study were from the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates Inc., 1985) and *ORTEP*II (Johnson, 1976).

Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s are given in Table 1.* Table 2 contains bond lengths and bond angles. Fig. 1 shows the molecular structure of the chlorpheniramine cation with the crystallographic numbering scheme. Fig. 2 is a stereoview of the unit cell showing hydrogen bonds.

Related literature. The structure of (+)-chlorpheniramine maleate has been reported by James & Williams (1974).

References

B. A. Frenz & Associates, Inc. (1985). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

James, M. N. G. & Williams, G. J. B. (1974). *Can. J. Chem.* **52**, 1872–1879.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

---

* Lists of structure factors, anisotropic temperature factors, H-atom coordinates, molecular dimensions involving H atoms, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52530 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1990). C**46**, 945–947

p-Nitrobenzenediazonium Tetrafluoroborate

By John C. Barnes

*Chemistry Department, The University, Dundee DD*1 4*HN, Scotland*

and Anthony Butler and Lesley Anderson

*Chemistry Department, University of St Andrews, St Andrews, KY*16 9*ST, Scotland*

(*Received* 27 *November* 1989; *accepted* 5 *January* 1990)

Abstract. $C_6H_4N_3O_2^+.BF_4^-$, $M_r = 236{\cdot}92$, orthorhombic, $Fdd2$, $a = 22{\cdot}56\,(5)$, $b = 31{\cdot}09\,(5)$, $c = 5{\cdot}33\,(3)$ Å, $V = 3738{\cdot}4$ Å^3, $Z = 16$, $D_x = 1{\cdot}68$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}26$ cm^{-1}, $F(000) = 1888$, $T = 293$ K, $R = 0{\cdot}081$ for 732 reflexions. The structure was determined because of an anomaly in the solid-state NMR spectrum of the title compound. The dimensions of the cation offer no evidence for a significant contribution from a quinonoid structure, $N{=}N^+$ 1·103 (8) Å. The normal to the mean plane of the nitro group lies at 11·4 (1)° to the normal to the mean plane of the benzene ring. The anion is disordered. Rotation about an F—B bond almost parallel to the *c* axis gives two sets of positions for the three F atoms. The site occupancy of the majority position is 82·3 (1·6)%. The shortest cation–anion contact is 2·621 (7) Å (N···F). There are several N···F and O···F distances in the range 3·1–3·3 Å.

Experimental. Off-white, striated needles were prepared from 4-nitroaniline by the method of Starky (1943) and recrystallized from acetonitrile by slow evaporation of the solvent at room temperature. Crystals deteriorated slowly on the open bench but showed no loss of intensity during data collection when mounted in Lindemann glass capillaries. Stoe Stadi-II diffractometer. Crystal 0·61 × 0·26 × 0·25 mm aligned along **c**. Unit cell refined from 20 precisely set reflexions with $12 < 2\theta < 15°$. 1418 measured reflexions gave 886 unique data ($R_{\text{int}} = 0{\cdot}070$). Index range $-6 \le h \le 27$, $-2 \le k \le 37$, $0 \le l \le 5$, $1 \le 2\theta \le 50°$. 732 reflexions with $F_o > 2\sigma_F$ used in the refinement. Programs used: *SHELXS*86 (Sheldrick, 1986), *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *SHELX*76.

Structure solution by routine direct methods. Refinement minimizing $\sum w|F_o - |F_c||^2$ to $R = 0{\cdot}081$, $wR = 0{\cdot}095$. Difference map showed alternative positions for F(14), F(15), F(16) by rotation about F(13)—B(12), which is almost parallel to **c**. Majority position [82·3 (1·6%)] refined as rigid tetrahedron

0108-2701/90/050945-03$03.00

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* ($Å^2 \times 10^3$) *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	849 (3)	771 (2)	112 (18)	45 (2)
C(2)	1228 (4)	837 (2)	2088 (18)	53 (2)
C(3)	1576 (4)	490 (3)	2803 (20)	57 (2)
C(4)	1528 (3)	106 (2)	1449 (18)	45 (2)
C(5)	1144 (3)	52 (2)	−527 (18)	52 (2)
C(6)	794 (3)	395 (2)	−1204 (18)	53 (2)
N(7)	494 (3)	1125 (2)	−554 (14)	46 (2)
N(8)	223 (3)	1407 (2)	−1066 (20)	73 (2)
N(9)	1914 (3)	−248 (2)	2181 (21)	66 (2)
O(10)	2178 (3)	−222 (2)	4079 (18)	100 (3)
O(11)	1943 (3)	−553 (2)	797 (20)	96 (3)
B(12)	540 (1)	4193 (1)	9046 *	57 (3)
F(13)	555 (1)	4201 (1)	6477 (1)	83 (2)
F(14)	−16 (1)	4075 (1)	9830 (1)	110 (3)
F(15)	671 (1)	4593 (1)	9957 (1)	162 (5)
F(16)	947 (1)	3902 (1)	9918 (1)	197 (6)
F(14*A*)	1105 (1)	4310 (1)	9742 (1)	134 (21)
F(15*A*)	416 (1)	3795 (1)	10001 (1)	212 (34)
F(16*A*)	145 (1)	4487 (1)	9957 (1)	229 (36)

* Fixed coordinate

Table 2. *Interatomic distances* (Å) *and angles* (°) *in the cation*

C(2)—C(1)	1·373 (11)	C(6)—C(1)	1·367 (11)
N(7)—C(1)	1·408 (10)	C(3)—C(2)	1·388 (11)
C(4)—C(3)	1·398 (11)	C(5)—C(4)	1·374 (11)
N(9)—C(4)	1·458 (10)	C(6)—C(5)	1·377 (10)
N(8)—N(7)	1·103 (8)	O(10)—N(9)	1·177 (11)
O(11)—N(9)	1·204 (10)		
C(6)—C(1)—C(2)	125·4 (7)	N(7)—C(1)—C(2)	115·5 (7)
N(7)—C(1)—C(6)	119·2 (7)	C(3)—C(2)—C(1)	116·5 (7)
C(4)—C(3)—C(2)	118·5 (8)	C(5)—C(4)—C(3)	123·4 (7)
N(9)—C(4)—C(3)	117·4 (7)	N(9)—C(4)—C(5)	119·2 (8)
C(6)—C(5)—C(4)	117·8 (8)	C(5)—C(6)—C(1)	118·4 (8)
N(8)—N(7)—C(1)	178·8 (7)	O(10)—N(9)—C(4)	118·7 (9)
O(11)—N(9)—C(4)	117·6 (9)	O(11)—N(9)—O(10)	123·7 (9)

with B—F 1·370 Å. All non-H atoms anisotropic except for minority positions F(14*A*) *etc.* U(H) refined to 0·069 (12) $Å^2$ for H atoms on calculated positions. 141 refined parmeters, $w = 2{\cdot}6856/[\sigma^2(F) + 0{\cdot}000438F^2]$; max. Δ/σ 0·021; max. features on final difference map 0·32, −0·29 e $Å^{-3}$. Final coordinates are given in Table 1, with bond lengths and angles of the cation in Table 2.* The cation is shown in Fig. 1 and a packing diagram in Fig. 2. There is no suggestion of disorder in the cation but the disorder of the anion is probably more complicated than the model used here. The crystals did not diffract strongly and attempts at more elaborate treatments did not succeed with the limited data available.

Related literature. The structure of benzenediazonium tetrafluoroborate has been reported several times with increasing precision and most recently by Cygler, Przybylska & Elofson (1982). The structure does not deviate significantly from the conventional 'aromatic' model whereas in 4-morpholinobenzenediazonium tetrafluoroborate (Alcock, Greenhough, Hirst, Kemp & Payne, 1980) the benzene ring is distorted towards a 'quinonoid' structure. In the present compound the bond lengths in the ring are all within 2σ of their mean (1·380 Å). The ring

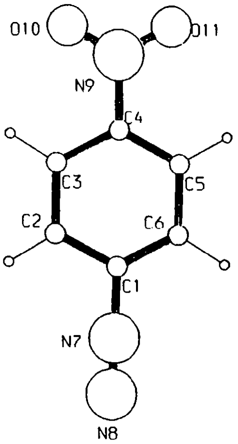

Fig. 1. *p*-Nitrobenzenediazonium cation, showing numbering scheme.

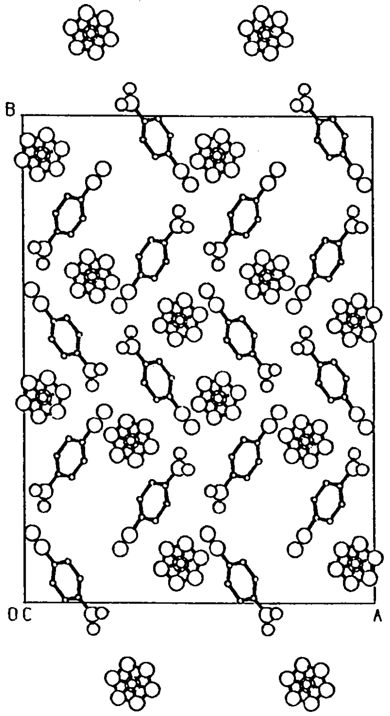

Fig. 2. Packing diagram for *p*-nitrobenzenediazonium tetrafluoroborate.

---

* 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 52579 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angles at C(1) [125·4 (7)°] and C(4) [123·4 (7)°] are significantly larger than the remainder (117·8° av.), as often observed when nitro or diazo groups are attached to a benzene ring. The title compound clearly belongs to the 'aromatic' class of diazo compounds.

In benzenediazonium tetrafluoroborate where the anions are ordered, each N atom has four contacts to F in the range 2·84–3·10 Å. The shortest distance from F to azo N in the present structure is N(8)···F(15*A*), 2·621 (9) Å, significantly shorter than the sum of van der Waals radii (2·85 Å). N(8)···F(13) is 2·914 (9) Å. N(7) has no contacts to F under 3·1 Å. F(14*A*) is close to the nitro group, 2·663 (10) Å to N(9) and 2·86 Å to O(10), O(11).

References

Alcock, N. W., Greenhough, T. J., Hirst, D. M., Kemp, T. J. & Payne, D. R. (1980). *J. Chem. Soc. Perkin Trans* 2. pp. 8–12.

Cygler, M., Przybylska, M. & Elofson, R. M. (1982). *Can. J. Chem.* **60**, 2852–2855.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Roberts, P. & Sheldrick, G. M. (1975). *XANADU*. Program for torsion angle, mean plane and libration correction calculation. Univ. of Cambridge, England.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS86*. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

Starky, E. B. (1943). *Organic Synthesis*, Collective Vol. II, p. 225. New York: John Wiley.

Acta Cryst. (1990). **C46**, 947–948

Structure of 8-Dimethylamino-1-naphthonitrile

By Masood Parvez and Ingeborg I. Schuster

Department of Chemistry, The Pennsylvania State University, University Park, PA 16802, *USA*

(*Received* 8 *September* 1989; *accepted* 8 *December* 1989)

Abstract. $C_{13}H_{12}N_2$, $M_r = 196·25$, monoclinic, Pc, $a = 8·786$ (3), $b = 9·341$ (4), $c = 7·071$ (3) Å, $\beta = 110·24$ (3)°, $V = 544·5$ Å^3, $Z = 2$, $D_x = 1·197$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·0672$ mm^{-1}, $F(000) = 208$, $T = 293$ (1) K, $R = 0·0708$ for 949 observed reflections with $I > 3\sigma(I)$. As a result of the substituents on the C(1) and C(8) atoms, the naphthyl moiety is twisted with deviations of these atoms of −0·071 and 0·099 Å, respectively, from the least-squares plane of the naphthalene ring, the C—C≡N angle in the nitrile group is 171·2 (4)° and the amine N atom is 0·361 (3) Å from the plane of its three bonded C atoms.

Experimental. 1,8-Naphthoic anhydride was converted to 8-bromo-1-naphthoic acid (Bailey, Card & Shechter, 1983) which was reacted with concentrated sulfuric acid and sodium azide to yield 8-bromo-1-naphthylamine. The amine was methylated and then converted to the title compound by the procedure reported by Whitaker & Snyder (1970). The product was recrystallized from hexane by slow evaporation at room temperature. A crystal of approximate dimensions 0·17 × 0·15 × 0·45 mm was cut from a long needle and used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. The cell constants and orientation matrix were determined by least-squares refinement of the setting angles of 21 reflections in the 10–15° range. Intensity data were collected in the range $2 < \theta < 27°$ using the ω–2θ scan method and variable scan speed. The intensities of two standard reflections, monitored at regular intervals, did not show significant variation. 1758 reflections measured, 1650 unique ($R_{\rm int} = 0·007$) (h 0→12, k 0→12, l −9→9) of which 949 with $I > 3\sigma(I)$ were considered observed. Data were corrected for Lorentz, polarization and absorption effects (North, Phillips & Mathews, 1968); the max. and min. correction factors were 0·9986 and 0·9092, respectively.

The structure was solved by direct methods using *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least-squares calculations of F's. H atoms were located from a difference map and included in the refinement with the overall isotropic temperature factor $B_{\rm iso} = 5·0$ Å^2; C and N had anisotropic temperature factors. The refinement converged completely with $R = 0·0708$ and $wR = 0·0932$, where $w = [\sigma^2(F_o) + (0·080F_o)^2]^{-1}$; max. shift/e.s.d. in the last cycle of refinement was < 0·07 and goodness of fit, $S = 1·723$. A final difference map was devoid of significant features with $\Delta\rho$ in the range −0·35 to 0·32 e Å^{-3}. Scattering factors used in the calcula-

0108-2701/90/050947-02$03.00