

The crystal structure of benzenediazonium tetrafluoroborate, $C_6H_5N_2^+ \cdot BF_4^-$

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Benzenediazonium tetrafluoroborate, $C_6H_5N_2^+ \cdot BF_4^-$, crystallizes in space group $P2_1/a$ with unit cell dimensions $a = 17.347(2)$, $b = 8.396(1)$, $c = 5.685(1)$ Å, $\beta = 92.14(1)^\circ$, $Z = 4$. The structure was solved by direct phasing methods using the program SHELX 76. The parameters were refined by full-matrix least-squares to a final $R = 0.063$ for 1346 observed reflections. The bond lengths and angles agree very well with those of Rømming for benzenediazonium chloride. The C—N and N≡N bond lengths are 1.415(3) and 1.083(3) Å, respectively, and the bonds of the benzene ring do not show any significant differences as they vary from 1.371(5) to 1.383(4) Å. There are three N...F close contacts of ≤ 2.84 Å and the positive charge appears to be shared between the nitrogen atoms.

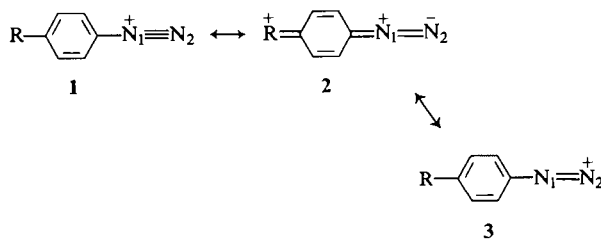
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Le tétrafluoroborate de benzenediazonium $C_6H_5N_2^+ \cdot BF_4^-$ cristallise dans le groupe d'espace $P2_1/a$ avec des dimensions de maille de $a = 17,347(2)$, $b = 8,396(1)$, $c = 5,685(1)$ Å, $\beta = 92,14(1)^\circ$, $Z = 4$. On a résolu la structure par la méthode de phase directe utilisant le programme SHELX 76. On a ajusté les paramètres par la méthode des moindres carrés (matrice complète) jusqu'à une valeur finale de $R = 0,063$ pour 1346 réflexions observées. Les longueurs de liaison et les angles s'accordent bien avec ceux de Rømming relatifs au chlorure de benzenediazonium. Les longueurs de liaison C—N et N≡N sont respectivement de 1,415(3) et 1,083(3) Å et les liaisons du cycle benzène ne présentent aucune différence significative puisqu'elles varient de 1,371(5) à 1,383(4) Å. Il y a trois liaisons N...F très rapprochées $\leq 2,84$ Å et la charge positive semble être partagée entre les atomes d'azote.

[Traduit par le journal]

Introduction

The representation of aryl diazonium salts by resonance forms has concerned organic chemists for over one hundred years (1). Discussion has centred on the relative contributions of canonical structures 1, 2, and 3.



The dominance of structure 1 for the ground state of benzenediazonium chloride was established by Rømming in 1963 (2, 3). His and subsequent X-ray investigations showed that the C(1)—N(1) bond length corresponds to that of a C—N single bond and N(1)—N(2) to a triple bond. Similar results were obtained for a number of substituted diazonium salts with complex anions. In *p*-benzenebisdiazonium tetrachlorozincate (4) in addition to N≡N and N—C bonds of 1.09(1) and

1.42(1) Å, respectively, there is an appreciable deviation from hexagonal symmetry of the benzene ring showing the "antiquinoid" geometry. There is therefore no evidence in these structures for the contribution of 2.

Nevertheless Hammett correlations of certain other parameters such as infrared stretching frequencies (5) and redox properties (6) have continued to use σ^+ constants rather than σ^p constants to obtain good results. These procedures suggested that contributions from the form 2 were important to describe the properties of these compounds.

In the original study of Rømming the interaction of the chlorine ion with hydrogen of the aromatic ring was indicated by the short H(2)...Cl distance of only 2.5 Å, one half of an Ångstrom unit less than the sum of the van der Waals radii of these atoms. Also in our laboratory the polarographic half-wave reduction potential of the aryl diazonium chlorides has been found to be up to 500 mV more negative than that of the tetrafluoroborate, which we interpret as evidence of complex formation (7). Since such an interaction could conceivably have altered some structural parameters it was decided to carry out X-ray analyses of some aryl diazonium tetrafluoroborate salts including *p*-diethylamino benzenediazonium tetrafluoroborate as the low accur-

¹NRCC No. 20552.

acy study of a related compound (8) gave longer N—N and N—C bond lengths than expected.

Experimental

Crystal data are as follows:

$C_6H_5N_2^+ \cdot BF_4^-$ fw = 191.9
Monoclinic, $P2_1/a$, $a = 17.347(2)$, $b = 8.396(1)$, $c = 5.685(1)$ Å,
 $\beta = 92.14^\circ(1)$, $V = 827.4$ Å³, $Z = 4$, $D_x = 1.54$, $D_m = 1.53$ g cm⁻³
(by flotation in toluene and CCl₄ mixture). $F(000) = 384$, μ (Cu K α) = 14.6 cm⁻¹.

The data were collected at room temperature with the Nonius CAD-4 procedures. Three prisms, crystallised from acetonitrile, had to be used as they deteriorated on exposure to light and X-rays. Two of them were almost spherical with diameters varying from 0.32 to 0.37 mm and the third one was $0.24 \times 0.27 \times 0.34$ mm. Reflections in the range $1^\circ < \theta \leq 75^\circ$ were measured using the ω - 2θ technique for $\Delta\omega = (1.20 + 0.15 \tan \theta)^\circ$, with a maximum scan time of 150 s. Two reflections were used as intensity monitors every 100 min of exposure time. Their intensities changed slowly during the first 20 to 30 h of exposure to X-rays and much more rapidly on further irradiation of the crystal. Only data collected in that first period were accepted. A decrease in intensity tolerated with each crystal did not exceed 4%.

The three sets of data were merged to produce 1757 unique reflections out of which 1346 (77%) were observed above threshold of $2\sigma(I_{\text{net}})$. The net intensities, after scaling, were corrected for Lorentz and polarization effects but not for absorption.

The atomic coordinates were found by direct methods and refined by full-matrix minimization of $\sum w(|F_o| - |F_c|)^2$ using the program system SHELX 76 (9).

The weighting function applied was

$$w = 16.64/(\sigma^2|F_o| + 0.00015|F_o|^2)$$

where $\sigma(|F_o|)$ is derived from counting statistics.

The H atoms were located on a difference map, carried out when R was 0.09. The atomic scattering factors for C, N, B, and F were taken from International tables for X-ray crystallography (10) and those for H from Stewart, Davidson, and Simpson (11). Refinement with anisotropic thermal parameters for non-hydrogen atoms and isotropic for H atoms reduced R to a final value of 0.063 ($R_w = 0.075$) for the 1346 observed reflections. The final difference map showed distribution in the range -0.32 to 0.45 e Å⁻³.

Some calculations were carried out with the NRC crystallographic computer programs by Ahmed, Hall, Pippy, and Huber (12).

Final positional and thermal parameters are presented in Table 1.²

Discussion

The bond lengths and angles of both ions are shown in Fig. 1. The angle C(1)—N(1)—N(2) in the present structure is $179.5(3)^\circ$ and the C—H bond lengths are in the range 0.89 to 1.01(4) Å.

The benzenediazonium ion has an approximate plane of symmetry passing through the N(2), N(1),

TABLE 1. Fractional coordinates with esd's in parentheses ($\times 10^4$ for non-hydrogen atoms and $\times 10^3$ for H) and isotropic temperature factors $\times 10^3$ ($U_{\text{eq}} = 1/3(U_{11} + U_{22} + U_{33})$)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} or <i>U</i> (Å ²)
B	1709(2)	2928(4)	4962(6)	56(2)
F(1)	1871(1)	2425(3)	2729(3)	87(2)
F(2)	1399(1)	1640(3)	6104(4)	102(2)
F(3)	2384(1)	3401(4)	6009(5)	127(2)
F(4)	1188(1)	4107(3)	4822(6)	126(2)
N(1)	2902(1)	4627(3)	442(4)	54(1)
N(2)	2361(1)	5266(4)	517(5)	74(2)
C(1)	3610(1)	3796(3)	323(4)	48(1)
C(2)	4034(2)	4070(4)	-1645(5)	61(2)
C(3)	4728(2)	3271(4)	-1717(6)	68(2)
C(4)	4958(2)	2257(4)	63(7)	67(2)
C(5)	4506(2)	2002(4)	1977(6)	66(2)
C(6)	3897(2)	2278(4)	2138(5)	59(2)
H(2)	386(2)	482(4)	-296(6)	70(9)
H(3)	504(2)	345(4)	-316(6)	75(10)
H(4)	540(2)	173(5)	6(6)	76(10)
H(5)	466(2)	126(4)	315(6)	72(10)
H(6)	374(2)	263(4)	326(5)	52(7)

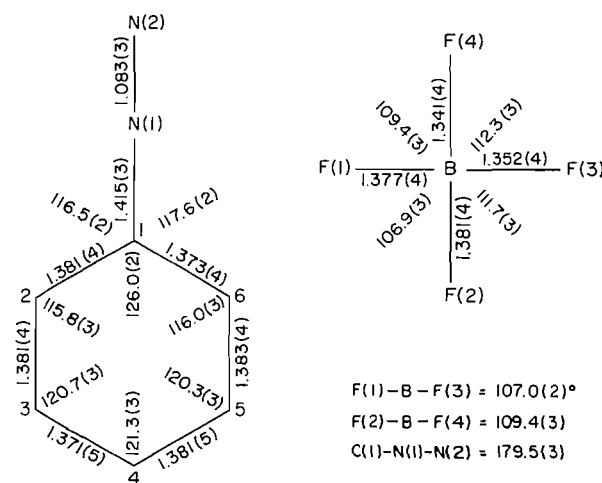


FIG. 1. Bond lengths (Å) and valency angles (deg) in benzenediazonium tetrafluoroborate and their esd's in parentheses.

C(1), and C(4) atoms as is indicated by the similarity of appropriate bonds and angles (see Fig. 1). Table 2 shows the comparison of the averaged geometry of the benzene ring in the present compound and in benzenediazonium chloride (3, 13), where the molecule was situated on a two-fold axis. The bond lengths and angles of the fluoroborate salt agree better with those from the more accurate analysis of benzenediazonium chloride, carried out at -160°C , with the exception of the three averaged bonds of the ring. However, the small differences shown by the bonds of the benzene ring in the tetrafluoroborate salt are not statistically signifi-

²Tables of observed and calculated structure factors and of temperature factors are available, at a nominal charge, from the Depository of Unpublished Data, CISTI, National Research Council of Canada, Ottawa, Ont., Canada K1A 0S2.

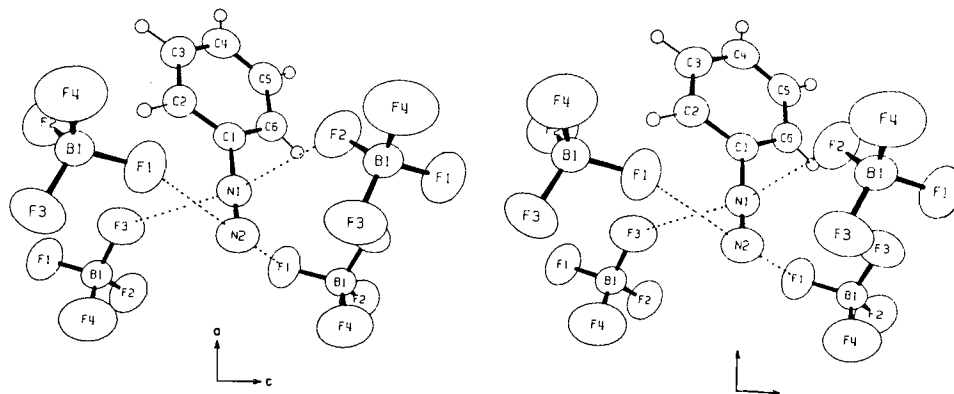


FIG. 2. A stereoscopic view of benzenediazonium cation with surrounding BF_4^- groups. The closest N to F contacts are shown by broken lines.

TABLE 2. Bond lengths and angles in benzenediazonium tetrafluoroborate and in benzenediazonium chloride (esd's in parentheses)

Bond	$\text{C}_6\text{H}_5\text{N}_2\text{BF}_4^*$	Length	
		$\text{C}_6\text{H}_5\text{N}_2\text{Cl}^\dagger$	
		<i>a</i>	<i>b</i>
N(1)—N(2)	1.083(3) Å	1.097(6) Å	1.093(2) Å
N(1)—C(1)	1.415(3)	1.385(9)	1.410(2)
C(1)—C(2)	1.377(4)	1.374(8)	1.396(2)
C(2)—C(3)	1.382(4)	1.383(10)	1.392(2)
C(3)—C(4)	1.376(5)	1.376(8)	1.395(2)
C(1)—C(4)	2.682(4)	2.689(8)	

Bonds	$\text{C}_6\text{H}_5\text{N}_2\text{BF}_4^*$	Angle	
		$\text{C}_6\text{H}_5\text{N}_2\text{Cl}^\dagger$	
		<i>a</i>	<i>b</i>
C(1)—C(2)—C(3)	115.9(3)	117.6(7)	115.9(2)
C(2)—C(3)—C(4)	120.5(3)	119.8(7)	120.6(2)
C(3)—C(4)—C(5)	121.3(3)	121.7(7)	121.1(2)
C(6)—C(1)—C(2)	126.0(2)	124.8(6)	126.1(2)

*The averaged values for the symmetry related pairs.

[†]*a*, ref. 3, data collected at room temperature; *b*, ref. 13, investigation at -160°C .

cant and since they do not appear in the low temperature study of the chloride salt, they may not be real. In both compounds the C(1)···C(4) distance is shorter than in benzene.

The bond lengths and angles reported for benzenediazonium tribromide (14) are also very similar. However, they are not included in Table 2 as, having been derived by two-dimensional methods, they are less accurate.

The endocyclic torsion angles of the benzene ring vary from 0 to $2.1(0.5)^\circ$ and the ring deviates significantly from planarity ($\chi^2 = 26.6$). The C(2) and C(6) atoms are at $0.010(3)$ and $0.008(3)$ Å, respectively, from the plane calculated for the six atoms of the ring, and C(1) with C(4) are on the opposite side with distances of $-0.006(2)$ and

$-0.005(4)$ Å. The N(1) and N(2) atoms were found at $-0.016(2)$ and $-0.023(3)$ Å from the plane.

The packing of BF_4^- ions surrounding the benzenediazonium cation is shown in the stereoscopic drawing by ORTEP (15) in Fig. 2. Each nitrogen atom makes four short contacts < 3.1 Å with F of each of the four anions. These distances are listed in order of increasing values:

Atoms	Distance (Å)	Atoms	Distance (Å)
N(1)···F(2)	2.829(3)*	N(2)···F(1)	2.841*
N(1)···F(3)	2.837(4)*	N(2)···F(1)	2.941(4)*
N(1)···F(1)	2.912(3)	N(2)···F(3)	3.005(4)
N(1)···F(1)	2.996(3)	N(2)···F(2)	3.056(3)

The two shortest contacts of each group, marked by asterisks, are depicted in Fig. 2 by broken lines. They include the three contacts which are slightly shorter than the sum of van der Waals radii (2.85 Å).

The close approaches of F to both N atoms indicate that the electron density of the N(1)—N(2) triple bond may be displaced towards the N(1) atom resulting in sharing of the positive charge between the two nitrogen atoms. Similar observations were made concerning the structures of benzenediazonium chloride and benzenediazonium tribromide. It seems therefore that in addition to the main resonance form 1, shown in "Introduction", the form 3 makes some contribution to the structure of the cation. There is no evidence of 2.

In benzenediazonium chloride and tribromide derivatives the shortest of the halogen to nitrogen distances are those to the outer nitrogen N(2). In the present structure those to N(1) are the shortest. Since the small differences between N(1)···F and N(2)···F closest contacts depend not only on the charge distribution but also on the geometrical arrangement, it is not possible to derive on their basis which nitrogen is likely to have a greater

positive charge. It is interesting, however, that the N(1)···F closest approaches agree with the results of the CNDO/2 calculations carried out by Rømming (16) for the diazonium ion and for benzenediazonium surrounded by four negative fluorine ions, as the positive charges on N(1) turned out to be slightly higher (+0.31 and +0.33) than on N(2) (+0.18 and +0.25).

The N···F distances can be correlated with the differences in the B—F bond lengths. Since F(2) makes the closest approach to N of 2.829 Å and F(1) has four close contacts, B—F(2) and B—F(1) are expected to be the longest. They are 1.381(4) and 1.377(4) Å respectively, vs. 1.352(4) Å for B—F(3). F(4)···N(2) is appreciably longer (3.384(4) Å) and accordingly B—F(4) is the shortest of the four bonds (1.341(4) Å).

The closest F···H approaches are F(2)···H(2) = 2.38(3) Å and F(2)···H(3) = 2.42(3) Å. They are slightly shorter than the sum of van der Waals radii of 2.55 Å and they indicate that very weak C—H···F hydrogen bonds may be present. C(2)—H(2) and C(3)—H(3) are 1.01(3) and 1.00(4) Å, respectively, C(2)—H(2)···F(2) = 174.1(2.7)° and C(3)—H(3)···F(2) = 133.6(2.6)°. Since the redox potential of benzenediazonium tetrafluoroborate is normal and virtually identical to that of the perchlorate salt, it must be assumed that the bond lengths of the benzene ring are unaffected by the weak H···F interactions. Even the much stronger H(2)···Cl

interactions in benzenediazonium chloride do not seem to have any such effect.

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