

Structure of 4-Bromo-1-(4-nitrophenyl)-3-phenylpyridazinium-5-olate, $C_{16}H_{10}BrN_3O_3$

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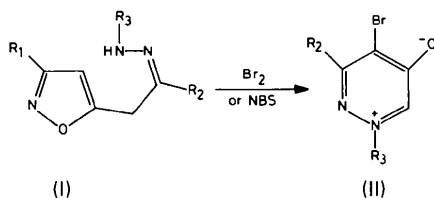
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Abstract. $M_r = 372.2$, monoclinic, $P2_1/n$, $a = 3.950(1)$, $b = 28.137(7)$, $c = 12.841(3)$ Å, $\beta = 94.40(2)^\circ$, $V = 1423.0(7)$ Å³, $Z = 4$, $D_x = 1.74$ Mg m⁻³, $Cu K\alpha$, $\mu = 4.53$ mm⁻¹, $\lambda = 1.5418$ Å, room temperature, $F(000) = 744$, final $R = 0.030$ for 2196 independent reflections having 2θ ($Cu K\alpha$) $< 136^\circ$ and $I > 3\sigma(I)$. The structure consists of a *meta*-substituted heterocyclic ring system with typical zwitterionic resonance forms.

Introduction. Recently Caristi, Gattuso, Ferlazzo & Stagno D'Alcontres (1983) have given evidence for the transformation of isoxazoles (I) into pyridazinium betaines (II) through a novel ring transformation under the influence of Br_2 or *N*-bromosuccinimide (NBS), as follows:



a, $R_1 = Ph$; $R_2 = Ph$; $R_3 = pNO_2-C_6H_4$

b, $R_1 = Ph$; $R_2 = Ph$; $R_3 = Ph$

c, $R_1 = mCl-C_6H_4$; $R_2 = Ph$; $R_3 = pNO_2-C_6H_4$

d, $R_1 = mCl-C_6H_4$; $R_2 = Ph$; $R_3 = Ph$

a, $R_2 = Ph$; $R_3 = pNO_2-C_6H_4$

b, $R_2 = Ph$; $R_3 = Ph$

In view of the fact that few structural data are known for similar betaines, we describe the crystal and molecular structure of (IIa).

Experimental. D_m not measured. Yellow crystal $0.10 \times 0.12 \times 0.65$ mm mounted with needle axis parallel to instrumental φ axis, Nicolet $P\bar{1}$ autodiffractometer, θ - 2θ scans, scan width 1.0° , Ni-filtered $Cu K\alpha$ radiation. Total of 2604 independent reflections with $3^\circ < 2\theta < 136^\circ$; scanning rate $6^\circ (2\theta) \text{ min}^{-1}$ for reflections

with $2\theta < 105^\circ$, and $4^\circ (2\theta) \text{ min}^{-1}$ otherwise. Background counts, for one-quarter the total scan time, taken at 2θ settings 1.0° at either side of the calculated $K\bar{\alpha}$ doublet values. Six standard reflections (monitored every 300 reflections): constant throughout. ψ scans for several intense reflections denoted absence of variable absorption. Structure solved using the heavy-atom Patterson technique. Least-squares refinement with anisotropic thermal parameters for the independent nonhydrogen atoms converged to $R = 0.042$ and $R_w = 0.058$ for 2196 independent reflections having $\sin\theta/\lambda < 0.602$ Å⁻¹ and $I > 3\sigma(I)$ with $\sigma^2(I) = (C_i + k^2B)$, where C_i is the total scan count, k is the ratio of

Table 1. Atomic coordinates and equivalent isotropic thermal parameters (Å²) for nonhydrogen atoms

The numbers in parentheses are the estimated standard deviations in the last significant digit.

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}^\dagger
Br	0.49328 (8)	0.50995 (1)	0.16345 (2)	3.53 (1)
O(11)	-0.4477 (6)	0.2403 (1)	0.6720 (2)	4.76 (6)
O(12)	-0.1197 (8)	0.2746 (1)	0.7901 (2)	5.94 (8)
O(6)	0.3069 (6)	0.5281 (1)	0.3860 (2)	3.99 (5)
N(1)	-0.2422 (6)	0.2707 (1)	0.7005 (2)	3.45 (6)
N(3)	0.2054 (5)	0.3865 (1)	0.3099 (1)	2.63 (5)
N(4)	0.1472 (5)	0.4046 (1)	0.4039 (1)	2.51 (5)
C(1)	0.3367 (6)	0.4663 (1)	0.2599 (2)	2.69 (6)
C(2)	0.2926 (6)	0.4181 (1)	0.2379 (2)	2.51 (6)
C(5)	0.1655 (7)	0.4503 (1)	0.4294 (2)	3.04 (7)
C(6)	0.2742 (7)	0.4854 (1)	0.3590 (2)	3.03 (6)
C(7)	0.3357 (6)	0.3956 (1)	0.1347 (2)	2.54 (6)
C(8)	0.1800 (7)	0.4148 (1)	0.0427 (2)	2.89 (6)
C(9)	0.2083 (7)	0.3917 (1)	-0.0513 (2)	3.24 (7)
C(10)	0.3938 (8)	0.3502 (1)	-0.0553 (2)	3.49 (7)
C(11)	0.5481 (8)	0.3311 (1)	0.0356 (2)	3.36 (7)
C(12)	0.5156 (7)	0.3536 (1)	0.1305 (2)	2.88 (6)
C(13)	0.0542 (6)	0.3696 (1)	0.4796 (2)	2.57 (6)
C(14)	-0.1082 (7)	0.3280 (1)	0.4452 (2)	2.97 (6)
C(15)	-0.2064 (7)	0.2954 (1)	0.5170 (2)	2.96 (6)
C(16)	-0.1356 (7)	0.3050 (1)	0.6226 (2)	2.77 (5)
C(17)	0.0299 (8)	0.3456 (1)	0.6571 (2)	3.17 (7)
C(18)	0.1283 (7)	0.3786 (1)	0.5854 (2)	2.95 (6)

$$\dagger B_{eq} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i a_j a_i^* a_j^*$$

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scan time to background time and B is the total background count. $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1/\sigma_F^2$ where $\sigma_F = \{[\sigma(F_o)]^2 + (p|F_o|)^2\}^{-1/2}$ and $p = 0.01$. H atoms located by means of a $\Delta\rho$ synthesis. Additional counter-weighted full-matrix least-squares refinement of H atoms with isotropic and of nonhydro-

gen atoms with anisotropic thermal parameters (248 variables) finally gave $R = 0.030$ and $R_w = 0.035$ for the observed reflections. Final shifts of atomic parameters all well below the corresponding σ . No extinction effects observed. Final $\Delta\rho$ map showed no peaks above noise level ($0.18 \text{ e } \text{\AA}^{-3}$). Recent tabulations of atomic form factors used together with anomalous-dispersion corrections (*International Tables for X-ray Crystallography*, 1974) to the Br scattering factors. Calculations performed on a 64K Data General Eclipse S-200 computer using a version of the Nicolet *EXTL* interactive crystallographic software package as modified at Crystalytics Company.

Discussion. Final fractional coordinates and bond data are listed in Tables 1 and 2.* Fig. 1 shows a perspective *ORTEP* drawing (Johnson, 1965) of the betaine molecule (IIa) together with the atom-labelling scheme and thermal-vibration ellipsoids of the non-hydrogen atoms.

Least-squares-planes' calculations* show that the C(7-12) and C(13-18) ring systems are planar to within 0.009 (3) \AA . The pyridazine ring, which deviates significantly from planarity [total puckering amplitude 0.042 (3) \AA], shows two pseudo mirrors [through C(2) and the middle of C(1)-C(6)] and has a very flattened boat conformation. On the whole, the molecular skeleton deviates from planarity due to steric interference. Dihedral angles between the heterocycle and the unsubstituted and substituted phenyl groups are 48.0 (1) and 27.2 (1) $^\circ$, respectively, which may be compared to the values observed for a variety of biphenyl derivatives (Fig. 2). At the side of the unsubstituted phenyl ring loss of planarity is related to the interaction of H(12) with the lone-pair electrons of N(3) and to the Br...H(8) contact of 2.86 (3) \AA . As a result, the bond angles in the C(8,7,2,1)Br system are opened up. The H(18)...H(5) and H(14)...N(3) interactions [2.16 (4) and 2.46 (3) \AA , respectively] between the other rings are less severe.

Bond lengths and angles of the phenyl rings are standard [average C-C 1.386 (2) \AA , C-C-C 120.0 (3) $^\circ$]. In accordance with the well known effects of substituents on aromatic-ring geometry, the C(15,16,17) angle of 122.0 (2) $^\circ$ is significantly larger than the ideal value (120°) and more similar to that expected [121.1 (1) $^\circ$] for NO_2 groups (Domenicano, Vaciago & Coulson, 1975; Domenicano, Mazzeo & Vaciago, 1976); also, the C(14,15,16) and C(16,17,18)

* Lists of structure factors, thermal parameters, atomic coordinates and bond data involving H atoms, and a table of least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38690 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (\AA) and angles ($^\circ$) involving nonhydrogen atoms

Br-C(1)	1.883 (3)	C(1)-C(2)	1.394 (4)
N(1)-O(11)	1.216 (3)	C(1)-C(6)	1.420 (4)
N(1)-O(12)	1.219 (3)	C(2)-C(7)	1.489 (3)
C(6)-O(6)	1.254 (3)	C(5)-C(6)	1.428 (4)
N(3)-N(4)	1.347 (3)	C(7)-C(8)	1.398 (3)
N(1)-C(16)	1.473 (3)	C(7)-C(12)	1.383 (4)
N(3)-C(2)	1.345 (3)	C(8)-C(9)	1.382 (4)
N(4)-C(5)	1.327 (3)	C(9)-C(10)	1.381 (4)
N(4)-C(13)	1.451 (3)	C(10)-C(11)	1.383 (4)
		C(11)-C(12)	1.388 (4)
		C(13)-C(14)	1.389 (4)
		C(13)-C(18)	1.391 (3)
		C(14)-C(15)	1.376 (4)
		C(15)-C(16)	1.390 (3)
		C(16)-C(17)	1.373 (4)
		C(17)-C(18)	1.383 (4)
O(11)-N(1)-O(12)	123.6 (3)	N(4)-N(3)-C(2)	115.9 (2)
O(11)-N(1)-C(16)	118.4 (2)	N(3)-N(4)-C(5)	125.3 (2)
O(12)-N(1)-C(16)	118.0 (2)	N(3)-N(4)-C(13)	114.4 (2)
		C(5)-N(4)-C(13)	120.3 (2)
Br-C(1)-C(2)	123.0 (2)	C(8)-C(9)-C(10)	120.5 (3)
Br-C(1)-C(6)	115.5 (2)	C(9)-C(10)-C(11)	119.9 (3)
C(2)-C(1)-C(6)	121.5 (2)	C(10)-C(11)-C(12)	119.9 (3)
N(3)-C(2)-C(1)	122.5 (2)	C(7)-C(12)-C(11)	120.5 (2)
N(3)-C(2)-C(7)	112.6 (2)	N(4)-C(13)-C(14)	119.6 (2)
C(1)-C(2)-C(7)	124.9 (2)	N(4)-C(13)-C(18)	119.0 (2)
N(4)-C(5)-C(6)	121.8 (2)	C(14)-C(13)-C(18)	121.4 (2)
O(6)-C(6)-C(1)	126.1 (3)	C(13)-C(14)-C(15)	119.6 (2)
O(6)-C(6)-C(5)	121.1 (3)	C(14)-C(15)-C(16)	118.6 (2)
C(1)-C(6)-C(5)	112.8 (2)	N(1)-C(16)-C(15)	119.3 (2)
C(2)-C(7)-C(8)	121.1 (2)	N(1)-C(16)-C(17)	118.7 (2)
C(2)-C(7)-C(12)	119.5 (2)	C(15)-C(16)-C(17)	122.0 (2)
C(8)-C(7)-C(12)	119.3 (2)	C(16)-C(17)-C(18)	119.7 (3)
C(7)-C(8)-C(9)	119.9 (2)	C(13)-C(18)-C(17)	118.6 (2)

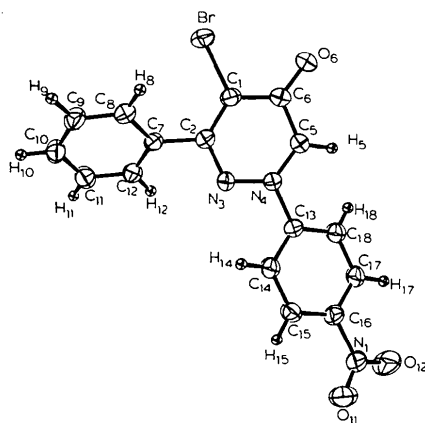


Fig. 1. Perspective drawing of the pyridazinium betaine showing the atom-labelling scheme and 50% probability thermal-vibration ellipsoids; H atoms are represented by small spheres of arbitrary size.

angles [average $119.1(2)^\circ$] are standard [$118.7(1)^\circ$] (Domenicano *et al.*, 1975). The C—N bond length at the nitro group [$1.473(3) \text{ \AA}$] conforms to the extensive compilation of Domenicano *et al.* (1975) (mean value 1.464 \AA). The other bond data [N—O $1.218(2) \text{ \AA}$; O—N—O $123.6(3)^\circ$, C—N—O $118.2(2)^\circ$] are very close to those for most nitroaromatic compounds: *e.g.* 1,3,5-trinitrobenzene (*s*-TNB) (Chang Sun Choi & Abel, 1972) [$1.207(9) \text{ \AA}$, $125.0(7)^\circ$ and $118.0(6)^\circ$], *trans*-azobenzene—(*s*-TNB)₂ and *N*-benzylidene-aniline—(*s*-TNB)₂ with mean values of $1.211(4) \text{ \AA}$, $124.7(3)^\circ$ and $117.6(2)^\circ$, respectively (Bar & Bernstein, 1981). The out-of-plane rotation of the NO₂ group is small [$15.1(3)^\circ$], again as usual in most simple nitroaromatic compounds without steric interference or particular packing requirements (absent in our case in view of the short *a* axis). The mean C—H distance of $0.94(1) \text{ \AA}$ in the benzene rings is also in good accordance with the usual findings [$0.95(2) \text{ \AA}$] (Churchill, 1973). The inter-ring C(2)—C(7) bond length [$1.489(3) \text{ \AA}$] is almost identical with the mean value of $1.490(9) \text{ \AA}$ (range $1.453\text{--}1.522 \text{ \AA}$) determined by X-ray analysis of substituted azobenzene

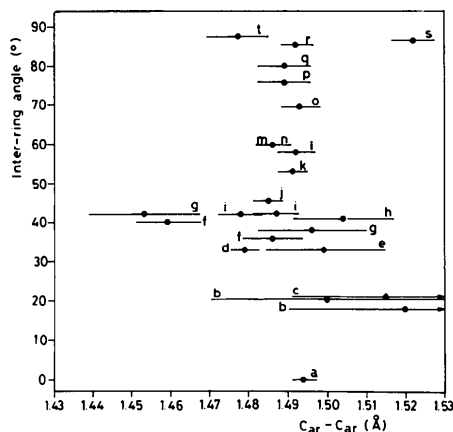
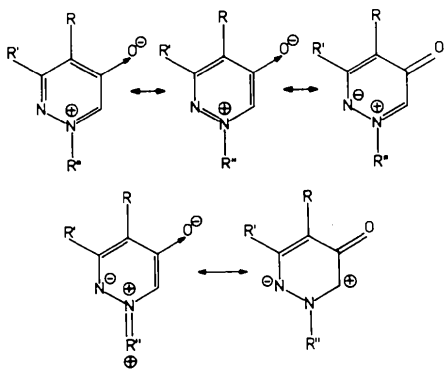


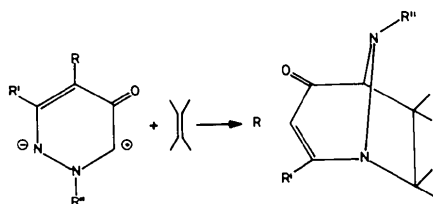
Fig. 2. Inter-ring $C_{ar}\text{--}C_{ar}$ bond length ($\pm 1\sigma$) vs angle of twist in biphenyl derivatives [substituent; reference, AC = *Acta Cryst.*, ACS = *Acta Chem. Scand.*, CSC = *Cryst. Struct. Commun.*, JCSP = *J. Chem. Soc. Perkin Trans. 2*, N = *Nature (London)*]: (a) none, N 1961 **191** 593; (a) none, AC 1976 **B32** 1420; (a) none, AC 1977 **B33** 1586; (b) 4-bromo, AC 1980 **B36** 968; (c) 4,4'-diamino-3,3'-dichloro, AC 1968 **B24** 1633; (d) 4-nitro, JCSP 1973 342; (e) 4,4'-dinitro, AC 1963 **16** 816; (f) 4,4'-dimethyl, AC 1969 **B25** 1741; (g) 4,4'-dibromo, CSC 1976 **5** 839; (h) 4,4'-diamino-3,3'-dimethyl, AC 1968 **B24** 1222; (i) 4,4'-dichloro, AC 1978 **B34** 981; (j) 4-acetyl-2'-nitro, AC 1974 **B30** 1562; (k) 2,3,4,5,6-pentafluoro, AC 1978 **A34** S93; (l) 2,3,5,6-tetrafluoro, AC 1978 **B34** 1644; (m) decafluoro, CSC 1976 **5** 483; (n) 2*H*-nonafluoro, AC 1978 **B34** 863; (o) 2,2'-dichloro, ACS 1974 **A28** 507; (p) 2,2'-dibromooctafluoro, AC 1980 **B36** 1402; (q) 2,2'-dibromo-4,4'-bis(*p*-methoxybenzylidene), AC 1975 **B31** 653; (r) 2-nitrononafluoro, AC 1978 **B34** 2994; (s) decachloro, AC 1975 **B31** 2931; (t) 2,2',4,4',6,6'-hexanitro, AC 1979 **B35** 259.

dyes (Fig. 2). It should be pointed out that Dewar & Schmeising (1968) suggest $1.485(-) \text{ \AA}$ as the length of a single bond between two sp^2 -hybridized C atoms. As shown in Fig. 2 no obvious relation exists between inter-ring bond length and angle of twist. This may be related to the fact that electron delocalization in biphenyl is only very slight (Hamor & Hamor, 1978) and to the influence of the electronegativity of the substituents. In the case of (IIa) there is the further complication that one of the two connected rings is not a phenyl but a pyridazine ring, with a different π delocalization and lack of planarity which certainly exerts some influence on the inter-ring connection.

The heterocyclic ring constitutes the more interesting part of the structure. The C—Br bond length of $1.883(3) \text{ \AA}$ agrees well with other C(sp^2)—Br bonds, as in *trans*-4,4'-dibromoazobenzene [$1.891(6) \text{ \AA}$] (Amit & Hope, 1966). The C=O bond length of $1.254(3) \text{ \AA}$ is typical of zwitterionic forms [$1.26(1) \text{ \AA}$], as, for example, in DL-serine. The C(1)—C(6) and C(5)—C(6) bond distances [$1.420(4)$ and $1.428(4) \text{ \AA}$, respectively] are significantly longer than the typical C(sp^2)—C(sp^2) bond length [$1.395(3) \text{ \AA}$] (*Molecular Structures and Dimensions*, 1972). The latter value is close to the C(1)—C(2) bond length of $1.394(4) \text{ \AA}$. The N(3)—N(4) bond distance is longer than in HN=NH [$1.238(7) \text{ \AA}$] (Trombetti, 1968), in azobenzene structures in the azo form [mean value $1.248(4) \text{ \AA}$] (Kelemen, Kormány & Rihs, 1982) and in the protonated 2,4-diaminoazobenzene cation [$1.313(4) \text{ \AA}$] (Moreiras, Solans, Solans, Miravittles, Germain & Declercq, 1981) but shorter than the N—N single-bond distance of 1.44 \AA (*International Tables for X-ray Crystallography*, 1968). The observed value is close to that measured in dyes in the hydrazone form [$1.359(6) \text{ \AA}$] (Keleman *et al.*, 1982). It is of interest to notice that in 4-oxo-2-phenyl-1,2,3-benzotriazinium-1-olate (Ballard & Norris, 1975) a N—N bond of $1.328(7) \text{ \AA}$ has been observed which is structurally comparable to N(3)—N(4). The measured $\Delta R(\text{CN})$ of 0.106 \AA in (IIa) [exceeding the limiting value of 0.06 \AA for azo-hydrazone forms (Kelemen *et al.*, 1982)] is in accordance with the picture. On the basis of Lofthus's (1959) bond-order—bond-length relationships bond orders of 1.4 v.u. are derived for the NN bond and 1.6, 1.7 and 1.1 v.u. for N(3)—C(2), N(4)—C(5) and N(4)—C(13), respectively. The latter bond distance is longer than in azobenzenes [$1.426(5) \text{ \AA}$] (Kelemen *et al.*, 1982) whereas the former two are typical partial double bonds, as in C_5H_5N [$1.352(5) \text{ \AA}$] and $HCO.NH_2$ [$1.322(3) \text{ \AA}$]. The C—N bond lengths in 4-oxo-2-phenyl-1,2,3-benzotriazinium-1-olate (Ballard & Norris, 1975) corresponding to N(4)—C(13) and N(3)—C(2) measure $1.450(8)$ and $1.352(8) \text{ \AA}$, respectively, as compared to $1.451(3)$ and $1.345(3) \text{ \AA}$ in (IIa). Our observations are in keeping with zwitterionic resonance forms, as follows:



Charge distribution on the ring leads one to predict 1,3-dipolar cycloaddition of double and triple bonds, as follows:



The potential 1,3-dipolar character of betaines, such as (II), is as expected (Katritzky & Dennis, 1979).

The type of layered packing arrangement (along *a* with an offset of 0.33 Å) found in (IIa) is associated with relatively small inter-ring dihedral angles. Apparently, sufficient energy is gained in this type of structure relative to some hypothetical arrangement having less planar molecules to offset the increase in intramolecular energy. Only Br...C(9ⁱ) 3.373 (3), Br...H(9ⁱ) 3.01 (3) and O(6)...H(5ⁱⁱ) 2.31 (3) Å [(i) 1-*x*, 1-*y*, *z*; (ii) \bar{x} , 1-*y*, 1-*z*] are considerably shorter

than the sum of the van der Waals radii (Br 1.95, C 1.70, N 1.50, O 1.40, H 1.20 Å) (Pauling, 1960).

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Structure of 1'-(*p*-Bromophenyl)-3'-ethyl-1',3',4',5'-tetrahydro-1,2-dideoxy-D-glycero-L-gluco-heptofuranosyl[1,2-*d*]imidazole-2'-thione,* C₁₆H₂₁BrN₂O₅S

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Abstract. $M_r = 433.3$, orthorhombic, $P2_12_12_1$, $a = 8.466$ (2), $b = 27.136$ (5), $c = 7.776$ (5) Å, $V = 1786$ (1) Å³, $Z = 4$, $D_m = 1.60$ (1), $D_x = 1.61$ Mg m⁻³,

* IUPAC name: 6-(*p*-bromophenyl)-4-ethyl-3-hydroxy-2-(1,2,3-trihydroxypropyl)-2,3,3a,5,6,6a-hexahydrofuro[1,2-*d*]imidazole-5(4*f*)-thione.

Mo $K\alpha$, $\lambda = 0.7107$ Å, $\mu = 2.42$ mm⁻¹, $F(000) = 888$, 300 K, $R = 0.045$ for 1196 observed independent reflexions. The sugar ring adopts a ⁴T₃ conformation and the dihedral angle in the bicycle is 72.2 (4)°. Intermolecular hydrogen bonds link molecules related by a screw axis to give helical chains parallel to [001].

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