hydrogen bonding between the hydroxyl group and either of the two triazole carbonyl oxygens. In (I), O(25) interacts with O(5′) [$\Delta = 2{\cdot}757\ (7)$ Å] *via* a diagonal translation in the *ac* plane whereas (II) forms an O(25)—H···O(3′) hydrogen bond [$\Delta = 2{\cdot}873\ (6)$ Å] involving the screw axis parallel to **a**.

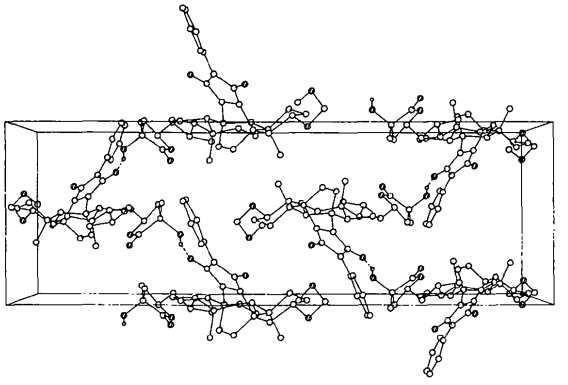

Fig. 3. Packing diagram of (II) viewed along **c** with **b** horizontal and **a** vertical. See Fig. 2 for further details.

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Structure of 1,2-Bis[(2-nitrophenyl)-*NNO*-azoxy]benzene, $C_{18}H_{12}N_6O_6$

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Abstract. $M_r = 408{\cdot}3$, monoclinic, space group $P2_1$, $a = 7{\cdot}618\ (3)$, $b = 11{\cdot}677\ (3)$, $c = 11{\cdot}267\ (4)$ Å, $\beta = 111{\cdot}13\ (2)°$, $Z = 2$, $D_m = 1{\cdot}445$, $D_x = 1{\cdot}450$ Mg m^{-3}. The structure was solved by direct methods and refined to $R = 0{\cdot}069$ for 1214 observed reflections. The conformations around the two azoxy groups are both *trans*. The six-membered rings of the two nitrophenyl groups form dihedral angles of 1·8 and 68·1° with the benzene plane respectively.

Introduction. The title compound is a thermolysis product of 1-[(2-nitrophenyl)imino]pyridine (Tamura, Tsujimoto, Ikeda & Tomita, 1971). The colorless needle-shaped crystal from ethanol had previously been interpreted as tris(2-nitrophenyl)triaziridine by means of chemical and spectroscopic studies (Tamura *et al.*, 1971; Oae, Tsujimoto & Nakanishi, 1973). However, the present X-ray work determined it as 1,2-bis-[(2-nitrophenyl)-*NNO*-azoxy]benzene, which was recently suggested by Leuenberger, Hoesch & Dreiding (1980).

Experimental. Crystal 0·2 × 0·2 × 0·1 mm, Rigaku automated four-circle diffractometer, graphite-monochromated Cu $K\alpha$; cell constants refined with a least-squares procedure for 25 reflections; 1395 independent reflections, ω–2θ scan mode, scan speed of 2° min^{-1} with background counts for 10 s.

Lp corrections but not absorption; 1214 non-zero reflections with $|F_o| > 3\sigma|F_o|$ in the range $\sin\theta/\lambda < 0{\cdot}560$ Å^{-1}; structure solved by the direct method using *MULTAN* (Germain, Main & Woolfson, 1971); all non-H atoms from the E map with the highest combined figure of merit; refinement by block-diagonal least-squares, $w = 1$; H atoms from difference Fourier map at $R = 0{\cdot}100$, and introduced in subsequent refinement without constraints; in final least-squares refinement, the weighting scheme was $w = 0{\cdot}2$ for

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$F_o = 0{\cdot}0$, $w = 1{\cdot}0$ for $0 < F_o \leq 12{\cdot}0$ and $w = [1{\cdot}0 + 0{\cdot}166(F_o - 12{\cdot}0)]^{-1}$ for $F_o > 12{\cdot}0$; final R 0·069, R_w 0·085; scattering factors from *International Tables for X-ray Crystallography* (1974); $F(000) = 420$. All the numerical computations were done on an ACOS 700 computer of the Crystallographic Research Center, Institute for Protein Research, Osaka University, using *UNICS* (1979). Refined coordinates and isotropic temperature factors for all the atoms are given in Table 1.*

* Lists of anisotropic thermal parameters, structure factors and least-squares planes and deviations of atoms from the planes (Table 3) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38046 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates and isotropic temperature factors with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3} \sum_i \sum_j B_{ij}\, \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}/B (Å^2)
C(1)	0·3783 (11)	0·8272 (7)	0·5689 (7)	3·36 (8)
C(2)	0·4981 (11)	0·8042 (7)	0·6880 (7)	3·23 (8)
C(3)	0·5840 (12)	0·8894 (8)	0·7757 (9)	4·13 (9)
C(4)	0·5353 (14)	1·0046 (8)	0·7321 (10)	4·65 (12)
C(5)	0·4147 (13)	1·0273 (7)	0·6139 (9)	4·12 (11)
C(6)	0·3346 (12)	0·9418 (7)	0·5288 (8)	3·76 (9)
N(1)	0·2884 (9)	0·7384 (6)	0·4793 (6)	3·43 (6)
N(2)	0·5575 (9)	0·6843 (5)	0·7272 (6)	3·39 (7)
O(1)	0·2903 (9)	0·7445 (5)	0·3681 (5)	4·17 (7)
O(2)	0·6109 (8)	0·6286 (5)	0·6523 (5)	4·00 (7)
C(11)	0·1292 (10)	0·5645 (7)	0·4383 (7)	3·18 (8)
C(12)	0·0035 (11)	0·5698 (8)	0·3170 (7)	3·53 (7)
C(13)	−0·0713 (13)	0·4680 (9)	0·2452 (9)	4·60 (10)
C(14)	−0·0088 (15)	0·3638 (8)	0·3023 (10)	4·22 (12)
C(15)	0·1130 (15)	0·3586 (8)	0·4223 (10)	4·44 (12)
C(16)	0·1902 (13)	0·4582 (8)	0·4944 (8)	4·36 (10)
N(11)	0·2073 (9)	0·6597 (6)	0·5213 (6)	3·52 (7)
N(12)	−0·0742 (9)	0·6755 (7)	0·2519 (6)	4·41 (7)
O(11)	−0·1004 (10)	0·7532 (6)	0·3178 (6)	6·04 (8)
O(12)	−0·1201 (10)	0·6823 (7)	0·1379 (6)	5·93 (8)
C(21)	0·6097 (11)	0·5362 (7)	0·8674 (7)	3·57 (7)
C(22)	0·7820 (11)	0·4912 (7)	0·8913 (9)	4·15 (10)
C(23)	0·8222 (14)	0·3759 (8)	0·9270 (10)	5·24 (12)
C(24)	0·6832 (16)	0·3084 (8)	0·9384 (10)	5·64 (12)
C(25)	0·5110 (16)	0·3521 (10)	0·9190 (10)	6·01 (12)
C(26)	0·4728 (12)	0·4678 (9)	0·8802 (9)	4·98 (11)
N(21)	0·5563 (9)	0·6550 (6)	0·8337 (6)	3·56 (7)
N(22)	0·9411 (11)	0·5627 (8)	0·8931 (11)	7·13 (19)
O(21)	0·9293 (9)	0·6603 (6)	0·8805 (7)	6·39 (8)
O(22)	1·0991 (15)	0·5209 (10)	0·9495 (20)	13·73 (41)
H(3)	0·681 (11)	0·876 (7)	0·880 (7)	2·2
H(4)	0·611 (16)	1·068 (11)	0·801 (10)	6·9
H(5)	0·382 (14)	1·102 (9)	0·570 (9)	5·3
H(6)	0·242 (10)	0·951 (7)	0·438 (6)	1·7
H(13)	−0·164 (11)	0·477 (8)	0·147 (7)	3·0
H(14)	−0·044 (12)	0·304 (8)	0·229 (8)	3·6
H(15)	0·181 (17)	0·294 (11)	0·479 (11)	6·9
H(16)	0·287 (9)	0·457 (7)	0·582 (6)	1·1
H(23)	0·953 (15)	0·348 (11)	0·952 (9)	5·8
H(24)	0·706 (14)	0·231 (9)	0·980 (9)	5·3
H(25)	0·420 (14)	0·308 (9)	0·924 (9)	5·1
H(26)	0·338 (10)	0·493 (7)	0·860 (7)	2·1

Discussion. A stereoscopic drawing with thermal ellipsoids for atoms of the molecule is shown in Fig. 1 with the numbering scheme.

As can be seen in Table 2, the bond lengths and angles are normal.

Atomic deviations from the least-squares planes are summarized in Table 3.*

Each of the three six-membered rings is planar within the limit of experimental error, while all N atoms except

* See previous footnote.

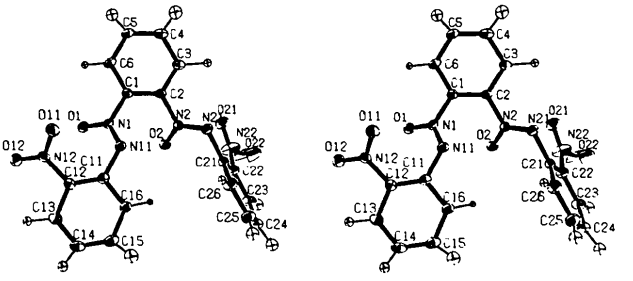

Fig. 1. Thermal-ellipsoid stereodrawing of the molecule with the numbering scheme.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·350 (12)	C(2)–C(3)	1·389 (13)
C(3)–C(4)	1·434 (15)	C(4)–C(5)	1·344 (15)
C(5)–C(6)	1·366 (14)	C(6)–C(1)	1·413 (13)
C(1)–N(1)	1·437 (11)	C(2)–N(2)	1·489 (11)
N(1)–O(1)	1·260 (10)	N(2)–O(2)	1·244 (9)
N(1)–N(11)	1·289 (10)	N(2)–N(21)	1·251 (10)
C(11)–C(12)	1·358 (12)	C(12)–C(13)	1·435 (14)
C(13)–C(14)	1·378 (15)	C(14)–C(15)	1·337 (16)
C(15)–C(16)	1·419 (15)	C(16)–C(11)	1·394 (13)
C(11)–N(11)	1·436 (11)	C(12)–N(12)	1·449 (12)
N(12)–O(11)	1·233 (11)	N(12)–O(12)	1·206 (11)
C(21)–C(22)	1·348 (13)	C(22)–C(23)	1·407 (15)
C(23)–C(24)	1·363 (17)	C(24)–C(25)	1·350 (17)
C(25)–C(26)	1·418 (16)	C(26)–C(21)	1·362 (13)
C(21)–N(21)	1·457 (11)	C(22)–N(22)	1·466 (16)
N(22)–O(21)	1·148 (15)	N(22)–O(22)	1·241 (26)

C(1)–C(2)–C(3)	122·8 (8)	C(1)–C(2)–N(2)	120·4 (7)
C(2)–C(3)–C(4)	115·5 (9)	C(3)–C(2)–N(2)	116·6 (8)
C(3)–C(4)–C(5)	121·6 (10)	C(4)–C(5)–C(6)	121·6 (10)
C(5)–C(6)–C(1)	118·3 (9)	C(6)–C(1)–C(2)	120·2 (8)
C(6)–C(1)–N(1)	117·5 (8)	C(2)–C(1)–N(1)	122·3 (8)
C(1)–N(1)–N(11)	115·3 (7)	C(1)–N(1)–O(1)	119·0 (7)
N(11)–N(1)–O(1)	125·7 (7)	N(1)–N(11)–C(11)	117·0 (7)
N(11)–C(11)–C(12)	126·6 (8)	N(11)–C(11)–C(16)	113·7 (8)
C(12)–C(11)–C(16)	119·7 (8)	C(11)–C(12)–N(12)	124·0 (8)
C(11)–C(12)–C(13)	121·5 (8)	C(13)–C(12)–N(12)	114·4 (8)
C(12)–C(13)–C(14)	117·9 (9)	C(13)–C(14)–C(15)	120·6 (10)
C(14)–C(15)–C(16)	122·3 (10)	C(15)–C(16)–C(11)	117·9 (9)
C(12)–N(12)–O(12)	120·3 (8)	C(12)–N(12)–O(11)	116·6 (8)
O(11)–N(12)–O(12)	123·0 (8)	C(2)–N(2)–O(2)	115·4 (7)
C(2)–N(2)–N(21)	115·8 (7)	O(2)–N(2)–N(21)	128·7 (7)
N(2)–N(21)–C(21)	114·4 (7)	N(21)–C(21)–C(22)	125·7 (8)
C(22)–C(21)–C(26)	118·6 (9)	N(21)–C(21)–C(26)	115·7 (8)
C(21)–C(22)–C(23)	121·7 (9)	C(21)–C(22)–N(22)	121·4 (9)
C(23)–C(22)–N(22)	116·6 (10)	C(22)–C(23)–C(24)	118·9 (11)
C(23)–C(24)–C(25)	120·6 (11)	C(24)–C(25)–C(26)	119·3 (11)
C(25)–C(26)–C(21)	120·8 (10)	C(22)–N(22)–O(21)	122·4 (11)
C(22)–N(22)–O(22)	115·3 (14)	O(21)–N(22)–O(22)	118·2 (15)

Table 4. *Torsion angles* (°) *with e.s.d.'s in parentheses*

Signs are given according to the convention of Klyne & Prelog (1960).

N(1)–C(1)–C(2)–N(2)	6·4 (12)	N(21)–N(2)–C(2)–C(1)	−135·2 (8)
N(11)–N(1)–C(1)–C(2)	49·8 (11)	C(21)–N(21)–N(2)–C(2)	178·2 (7)
C(11)–N(11)–N(1)–C(1)	−175·0 (7)	C(22)–C(21)–N(21)–N(2)	67·3 (11)
C(12)–C(11)–N(11)–N(1)	−54·5 (12)	N(22)–C(22)–C(21)–N(21)	3·9 (15)
N(12)–C(12)–C(11)–N(11)	−0·7 (14)	O(21)–N(22)–C(22)–C(21)	3·8 (18)
O(11)–N(12)–C(12)–C(11)	−34·1 (13)		

N(1) deviate significantly, probably for the relief of intramolecular short contacts. The dihedral angles of the paired planes (I)–(II), (I)–(III) and (II)–(III) are 1·8, 68·1 and 66·4°, respectively. Some torsion angles in the molecule are listed in Table 4.

The conformations around the two azoxy groups are both *trans*. The nitro group bound to ring (II) is considerably twisted out of the six-membered-ring plane, while that bound to ring (III) is not. Rotations about the C(1)–N(1) and C(2)–N(2) bonds are fixed so as to minimize the unusual contacts between the two azoxy groups and also to stagger the nitro groups over the azoxy group moieties.

As shown in Fig. 2, the crystal structure consists of columns of parallel-stacked six-membered rings. The columns are parallel to the crystallographic *a* axis and the stacking distance is 3·8 Å (*a*/2), which is larger than that reported for the overlap interactions of an aromatic ring system. As a whole, the molecules are well packed and are nearly parallel to the $(10\bar{1})$ plane without short intermolecular contacts other than weak van der Waals interactions.

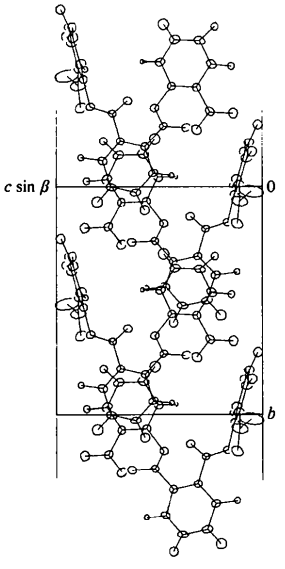

Fig. 2. The *a*-axis projection of the packing of the molecules.

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Structure of 3-Benzyl-7-methyl-3,7-diazabicyclo[3.3.1]nonan-9-one, $C_{15}H_{20}N_2O$

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Abstract. $M_r = 244·4$ (5), monoclinic, space group $P2_1/n$, $a = 9·779$ (2), $b = 16·208$ (3), $c = 8·596$ (2) Å, $\beta = 90·69$ (1)°, $V = 1362$ Å^3, $Z = 4$, $d_m = 1·20$, $d_x = 1·191$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0·5580$ mm, $T = 294$ K. Final $R = 0·050$ for 2177 observed reflections. The bicyclic system adopts a

0108-2701/83/010101-03$01.50