configurations of the latter model. This correspondence suggests the possibility of disorder in the latter type in the room-temperature phase.

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Structure of *p*-(*N*,*N*-Dimethylamino)phenyl 1,2,4-Triazol-1-yl Ketone *p*-Nitrophenylhydrazone

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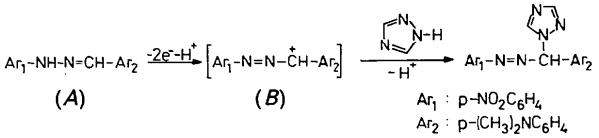
Abstract. $C_{17}H_{17}N_7O_2$, $M_r = 351·37$, triclinic, $P\bar{1}$, $a = 11·361\,(5)$, $b = 10·327\,(3)$, $c = 7·763\,(3)$ Å, $\alpha = 80·47\,(1)$, $\beta = 104·95\,(4)$, $\gamma = 104·89\,(3)°$, $V = 845·6\,(6)$ Å^3, $Z = 2$, $F(000) = 368$, $D_x = 1·380$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $\mu = 0·90$ mm^{-1}, room temperature, $R = 0·058$ for 1281 observed reflections. The best plane of the five-membered triazole ring lies almost perpendicular to the best planes of two benzene rings. The symmetry-related molecules are linked together by two N—H···N hydrogen bonds forming dimers.

Introduction. The title compound was prepared by electrochemical oxidation of *p*-(*N*,*N*-dimethylamino)benzaldehyde *p*-nitrophenylhydrazone (*A*) in the presence of 1,2,4-triazole (Gunić & Tabaković, 1988). It was concluded that the electrochemical oxidation of (*A*) in the presence of heteroaromatic bases occurs through the formation of cation (*B*), which is attacked by base as a nucleophile. This excluded the possibility of annelation of the heteroaromatics through nitrilimine as an intermediate. 1,2,4-Triazole can attack the cation (*B*) with all five positions according to its behaviour towards electrophiles (Potts, 1961). On the basis of high-resolution mass spectral and NMR data the structure of the product isolated was assigned as *N*,*N*-dimethyl-*p*-[(*p*-nitrophenylazo)(1,2,4-triazol-1-yl)methyl]aniline. It is the purpose of the present paper to correct the proposed structure on the basis of crystal structure analysis.



Experimental. The title compound crystallizes as red transparent prisms. A crystal of dimensions 0·32 × 0·68 × 0·84 mm was selected for X-ray analysis and mounted on a Philips PW1100 diffractometer. The refined lattice parameters, $a = 11·361\,(5)$, $b = 10·327\,(3)$, $c = 7·763\,(3)$ Å, $\alpha = 80·47\,(1)$, $\beta = 104·95\,(4)$ and $\gamma = 104·89\,(3)°$, were obtained by a least-squares procedure from the setting angles of 13 reflections with $8 < \theta < 12°$. Intensities were collected with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0·7107$ Å). Scan range $2 < \theta < 30°$, ω scan, h −15 to 15, k −12 to 13, l 0 to 8. A standard reflection was measured every hour and remained constant throughout the measurement. A total of 1583 reflections were collected of which 1568 were unique (R_{int}

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Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses for non*-H *atoms*

$B_{eq} = 4/3\,\mathrm{trace}(B\mathbf{G})$ where **G** is the direct metric tensor.

	x	y	z	B_{eq}
O(1)	−0·2370 (5)	0·1070 (5)	−0·1035 (7)	5·9 (2)
O(2)	−0·3338 (4)	0·2618 (5)	−0·1057 (7)	5·8 (2)
N(1)	0·4106 (4)	0·5475 (5)	0·7333 (7)	3·4 (2)
N(2)	0·5909 (4)	0·6117 (5)	0·6380 (8)	3·6 (2)
N(3)	0·3999 (4)	0·6378 (5)	0·5835 (7)	2·8 (1)
N(4)	0·1884 (4)	0·6174 (5)	0·4205 (7)	3·0 (1)
N(5)	0·1888 (4)	0·4968 (5)	0·3768 (8)	3·3 (2)
N(6)	−0·2423 (5)	0·2124 (6)	−0·0560 (8)	4·6 (2)
N(7)	0·2772 (4)	1·1726 (5)	0·7587 (8)	3·7 (2)
C(1)	0·5275 (6)	0·5361 (6)	0·7594 (9)	3·6 (2)
C(2)	0·5089 (5)	0·6754 (6)	0·5300 (9)	3·2 (2)
C(3)	0·2875 (5)	0·6879 (6)	0·5145 (8)	2·8 (2)
C(4)	0·0817 (5)	0·4261 (6)	0·2718 (8)	2·9 (2)
C(5)	−0·0311 (5)	0·4694 (6)	0·2330 (9)	3·1 (2)
C(6)	−0·1358 (5)	0·3991 (6)	0·1249 (9)	3·2 (2)
C(7)	−0·1316 (6)	0·2857 (7)	0·0592 (9)	3·9 (2)
C(8)	−0·0213 (6)	0·2385 (6)	0·1033 (9)	3·7 (2)
C(9)	0·0841 (5)	0·3087 (6)	0·2094 (9)	3·4 (2)
C(10)	0·2897 (5)	0·8179 (6)	0·5683 (8)	2·9 (2)
C(11)	0·1872 (5)	0·8770 (6)	0·5013 (10)	3·6 (2)
C(12)	0·1834 (5)	0·9926 (6)	0·5619 (10)	3·6 (2)
C(13)	0·2806 (5)	1·0561 (6)	0·6922 (9)	3·1 (2)
C(14)	0·3873 (5)	1·0006 (6)	0·7501 (9)	3·5 (2)
C(15)	0·3895 (5)	0·8851 (6)	0·6888 (10)	3·6 (2)
C(16)	0·3578 (6)	1·2081 (6)	0·9293 (10)	4·1 (2)
C(17)	0·1578 (7)	1·2106 (7)	0·7213 (11)	5·0 (3)

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

O(1)—N(6)	1·227 (8)	N(4)—C(3)	1·289 (7)	C(5)—C(6)	1·374 (8)
O(2)—N(6)	1·221 (5)	N(5)—C(4)	1·381 (7)	C(6)—C(7)	1·369 (9)
N(1)—N(3)	1·365 (7)	N(6)—C(7)	1·452 (8)	C(7)—C(8)	1·401 (5)
N(1)—C(1)	1·324 (4)	N(7)—C(13)	1·399 (8)	C(8)—C(9)	1·371 (8)
N(2)—C(1)	1·354 (8)	N(7)—C(16)	1·445 (9)	C(10)—C(11)	1·395 (6)
N(2)—C(2)	1·316 (7)	N(7)—C(17)	1·453 (5)	C(10)—C(15)	1·380 (8)
N(3)—C(2)	1·349 (4)	C(3)—C(10)	1·463 (9)	C(11)—C(12)	1·368 (9)
N(3)—C(3)	1·442 (5)	C(4)—C(5)	1·408 (5)	C(12)—C(13)	1·394 (9)
N(4)—N(5)	1·344 (7)	C(4)—C(9)	1·387 (9)	C(13)—C(14)	1·410 (5)
				C(14)—C(15)	1·364 (9)

N(3)—N(1)—C(1)	101·8 (8)	N(5)—C(4)—C(9)	119·7 (8)
C(1)—N(2)—C(2)	103·5 (8)	C(5)—C(4)—C(9)	119·7 (9)
N(1)—N(3)—C(2)	110·0 (7)	C(4)—C(5)—C(6)	119·5 (9)
N(1)—N(3)—C(3)	120·5 (7)	C(5)—C(6)—C(7)	120·3 (9)
C(2)—N(3)—C(3)	129·2 (8)	N(6)—C(7)—C(6)	120·1 (9)
N(5)—N(4)—C(3)	120·7 (8)	N(6)—C(7)—C(8)	119·4 (9)
N(4)—N(5)—C(4)	118·8 (8)	C(6)—C(7)—C(8)	120·6 (10)
O(1)—N(6)—O(2)	123·6 (9)	C(7)—C(8)—C(9)	119·5 (10)
O(1)—N(6)—C(7)	118·1 (9)	C(4)—C(9)—C(8)	120·2 (9)
O(2)—N(6)—C(7)	118·2 (9)	C(3)—C(10)—C(11)	120·2 (9)
C(13)—N(7)—C(16)	119·5 (8)	C(3)—C(10)—C(15)	122·4 (9)
C(13)—N(7)—C(17)	117·9 (8)	C(11)—C(10)—C(15)	117·4 (9)
C(16)—N(7)—C(17)	113·8 (9)	C(10)—C(11)—C(12)	120·8 (9)
N(1)—C(1)—N(2)	115·0 (9)	C(11)—C(12)—C(13)	121·9 (9)
N(2)—C(2)—N(3)	109·7 (8)	N(7)—C(13)—C(12)	122·7 (9)
N(3)—C(3)—N(4)	121·4 (8)	N(7)—C(13)—C(14)	120·5 (9)
N(3)—C(3)—C(10)	116·3 (8)	C(12)—C(13)—C(14)	116·7 (9)
N(4)—C(3)—C(10)	122·2 (8)	C(13)—C(14)—C(15)	120·6 (9)
N(5)—C(4)—C(5)	120·6 (8)	C(10)—C(15)—C(14)	122·3 (9)

N(2)—C(2)—N(3)—N(1)	0·8 (7)	C(2)—N(3)—C(3)—N(4)	107·3 (10)
N(3)—N(1)—C(1)—N(2)	−0·1 (6)	C(4)—N(5)—N(4)—C(3)	−178·2 (10)
N(5)—N(4)—C(3)—N(3)	−3·1 (8)	C(6)—C(7)—N(6)—O(1)	−175·8 (11)
C(1)—N(2)—C(2)—N(3)	−0·8 (7)	C(6)—C(7)—N(6)—O(2)	7·0 (10)
C(2)—N(2)—C(1)—N(1)	0·6 (7)	C(9)—C(4)—N(5)—N(4)	173·1 (11)
C(2)—N(3)—N(1)—C(1)	−0·4 (8)	C(11)—C(10)—C(3)—N(3)	177·1 (10)
C(3)—N(3)—N(1)—C(1)	−175·0 (10)	C(16)—N(7)—C(13)—C(12)	159·9 (10)
N(4)—C(3)—N(3)—N(1)	−79·0 (9)	C(17)—N(7)—C(13)—C(12)	14·3 (9)

= 0·04). Lorentz and polarization corrections were applied to the data. The linear absorption coefficient is 0·9 cm^{-1} for Mo $K\alpha$ radiation. No absorption correction was made.

The structure was solved by direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Using 450 reflections (minimum E of 0·96) and the *RANTAN* option, a total of 160 phase sets were produced. A total of 26 non-H atoms were located from an E map (R = 0·44). H atoms were located and added to the structure-factor calculations with isotropic temperature factors $B_{i\mathrm{H}} = B_{i\mathrm{C}} + 1$ Å^2, but their positions were not refined. The structure was refined by full-matrix least squares, where the function minimized was $\sum(\Delta F)^2$ with unit weights for all observed reflections.

Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964). Only the 1281 reflections having $I > 3\sigma(I)$ were used in the refinements. The

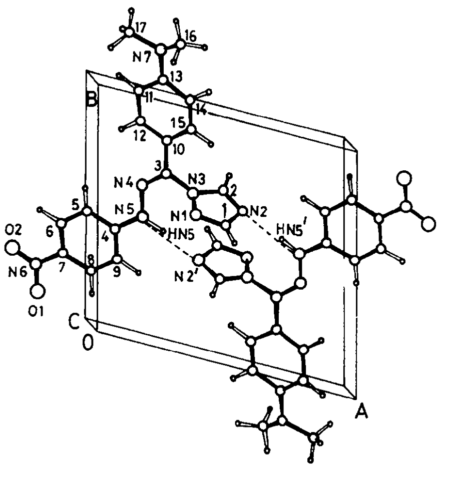

Fig. 1. Symmetry-related molecules linked together by a pair of hydrogen bonds (shown by dashed lines). The numbers are for C atoms unless otherwise indicated.

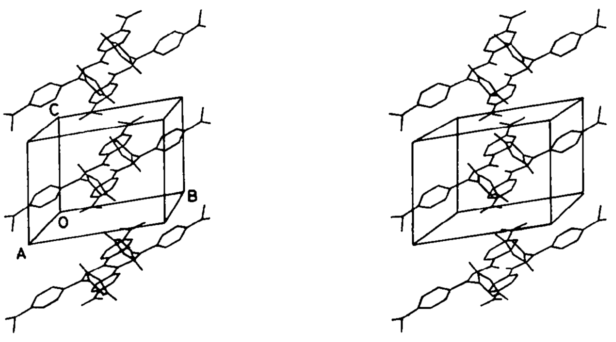

Fig. 2. Stereo packing diagram viewed down **a**.

final cycle of refinement included 235 variable parameters and converged with $R = 0{\cdot}058$. $wR = 0{\cdot}069$; $S = 1{\cdot}07$; the highest peak in the final ΔF map had a height of 0·21 (6) e Å^{-3}, $(\Delta/\sigma)_{\max} = 0{\cdot}282$. All calculations were performed on a PDP-11 minicomputer using *SDP* (Frenz, 1978) and local programs.

Discussion. The final atomic positions and equivalent isotropic temperature factors for non-H atoms are given with their e.s.d.'s in Table 1.* Bond lengths, bond angles and relevant torsion angles are listed in Table 2. A perspective view of the molecule with the atom-labelling system is shown in Fig. 1. Distances and angles in the five-membered triazole ring are in excellent agreement with those reported earlier (Ribár, Stanković, Argay, Kálmán & Koczó, 1987), except the N(3)—N(1) distance which is significantly shorter. The triazole ring is fairly planar. The dihedral angles formed by the least-squares planes of the triazole and the two benzene rings are 84·4 (2) for C(4)—C(9) and 86·7 (2)° for C(10)—C(15). Between the best planes of two benzene rings the dihedral angle is 9·0 (2)°. Torsion angles indicate that C(7), N(6), O(1), O(2) and C(13), N(7), C(16), C(17) lie in the planes of the corresponding benzene rings.

The symmetry-related molecules are linked together by a pair of N···N hydrogen bonds with N(5)···N(2)′ = 3·028 (6), H(N5)···N(2)′ = 2·080 (6) Å and N(5)—H(N5)···N(2)′ = 149·0 (4)°, forming dimers (Fig. 1). Fig. 2 shows the molecular packing in a stereoview down *a* axis.

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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 53483 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4,5-Dimethoxy-2-[3-(1,4-oxazinan-4-yl)-3-oxopropyl]-*N*-(2-pyridyl)-benzenesulfonamide Methanol Solvate

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Abstract. $C_{20}H_{25}N_3O_6S.CH_4O$, $M_r = 467{\cdot}54$, triclinic, $P\bar{1}$, $a = 8{\cdot}429\,(6)$, $b = 11{\cdot}271\,(3)$, $c = 12{\cdot}606\,(3)$ Å, $\alpha = 76{\cdot}302\,(8)$, $\beta = 78{\cdot}320\,(4)$, $\gamma = 78{\cdot}227\,(8)°$, $V = 1124{\cdot}02\,(90)$ Å^3, $Z = 2$, $D_m = 1{\cdot}34$, $D_x = 1{\cdot}38$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 1{\cdot}54$ mm^{-1}, $F(000) = 496$, $T = 293$ K, final $R = 0{\cdot}051$ for 2940 observed reflections. There are two molecules of methanol per unit cell, each with two alternative orientations. C(*A*1) is common to both orientations whereas the methanol O atom occupies sites O(*A*1) and O(*A*2) with equal probability.

Introduction. Derivatives of benzothiazinone have anti-inflammatory activity (Lombardino & Wiseman, 1971) and are known to affect the central nervous system (Sianesi, Redaelli, Magistretti & Massarani, 1973). Sulfonamides, similar in structure to benzothiazinone, have been synthesized (Camoutsis & Catsoulakos, 1976, 1980; Catsoulakos & Camoutsis, 1980) and are being tested for pharmacological properties.

Crystallographic studies of a series of these compounds have been carried out (Hamodrakas &

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