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2-Phenylbenzo-1,2,3-triaziniumbetaine 1-Oxide

BY R. E. BALLARD AND E. K. NORRIS

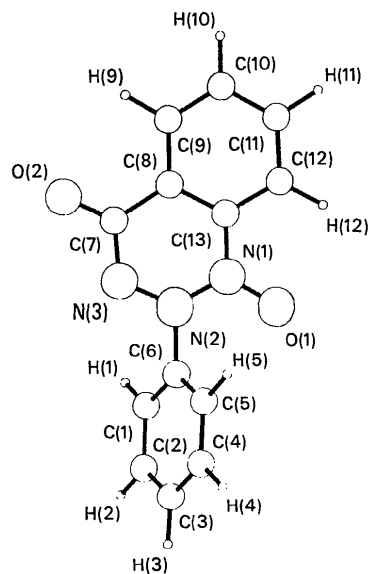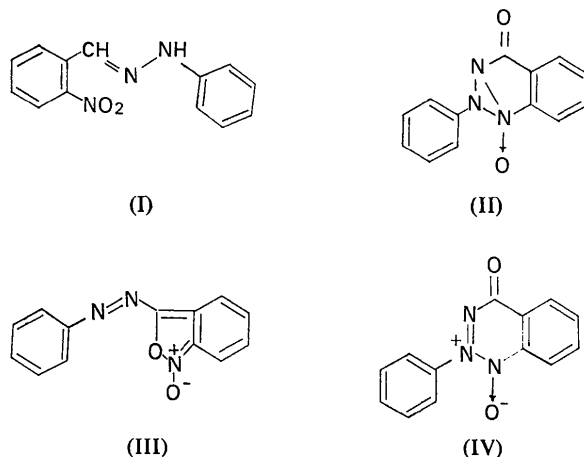*School of Chemical Sciences, University of East Anglia, University Plain, Norwich, Norfolk, England*

Abstract. $C_{13}N_3O_2H_9$, monoclinic, space group $P2_1/n$, $a = 8·13$ (5), $b = 21·02$ (5), $c = 8·13$ (5) Å, $\beta = 128·6$ (2)°, $Z = 4$, $D_m = 1·435$ (10), $D_x = 1·463$ g cm^{-3}. The compound is a 1,2,3-triaziniumbetaine 1-oxide (IV) rather than a phenylazoanthranil N-oxide (III) or an 'isodiazomethane' (II).

Experimental. The compound was prepared by Kobylecki's (1973) method and gave bright-yellow needles m.p. 147–149° on recrystallization from $CHCl_3$. The intensities were recorded on Weissenberg photographs (1401 independent reflexions about three axes) and measured automatically by the Optronics P-100 scanner at the Atlas Computer Laboratory (S.R.C. Microdensitometer Service). No correction was made for absorption.

Finding the best set of starting phases presented the same problems as were experienced with 2,7-dimethyl-5-acetylaminopyrazolo[1,5-*a*]pyrimidine (Ballard, Nor-

(I)

(II)

(III)

(IV)

Introduction. Chattaway described as an 'isodiazomethane' (II) the compound obtained by bromination and dehydrohalogenation of *o*-nitrobenzaldehyde phenylhydrazone (I) (Chattaway & Walker, 1927; Chattaway & Adamson, 1930; Chattaway & Parkes, 1935). Later (Gibson, 1962) it was suggested that the structure was that of a phenylazoanthranil N-oxide (III) and finally (Kerber, 1972) that it was a 1,2,3-triaziniumbetaine 1-oxide (IV). Although the chemical evidence favours (IV) the spectroscopic results were ambiguous and an X-ray study was undertaken.

Fig. 1. The molecule of 2-phenylbenzo-1,2,3-triaziniumbetaine 1-oxide.

ris & Sheldrick, 1975); the same method was used for the solution, the fourth E map in the ranking order being largely correct. The final unweighted R was 0·056 and all atoms were located.* Since the hydrogen atoms did not all refine satisfactorily their final positions

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30746 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Atomic coordinates* ($\times 10^4$)

	x/a	y/b	z/c
C(1)	5676 (6)	895 (1)	4410 (6)
C(2)	6472 (6)	370 (2)	4086 (7)
C(3)	5168 (7)	−10 (2)	2347 (7)
C(4)	3065 (7)	122 (2)	923 (6)
C(5)	2240 (6)	640 (2)	1217 (5)
C(6)	3566 (5)	1011 (1)	2948 (5)
C(7)	3029 (5)	2679 (1)	3505 (4)
C(8)	1291 (4)	2665 (1)	3583 (4)
C(9)	457 (5)	3219 (2)	3722 (5)
C(10)	−1136 (5)	3188 (2)	3857 (5)
C(11)	−1896 (5)	2604 (2)	3899 (5)
C(12)	−1111 (5)	2046 (2)	3768 (5)
C(13)	489 (4)	2088 (1)	3602 (4)
N(1)	1287 (4)	1528 (1)	3414 (4)
N(2)	2786 (4)	1586 (1)	3235 (4)
N(3)	3706 (4)	2119 (1)	3305 (4)
O(1)	636 (4)	993 (1)	3439 (4)
O(2)	3902 (3)	3175 (1)	3642 (4)

Calculated hydrogen positions

	x/a	y/b	z/c
H(1)	6680	1200	5762
H(2)	8127	258	5207
H(3)	5809	−415	2103
H(4)	2051	−181	−433
H(5)	582	750	104
H(9)	1066	3676	3724
H(10)	−1804	3620	3930
H(11)	−3132	2588	4039
H(12)	−1712	1591	3791

Table 3. *Interatomic distances* (Å)

C(1)–C(2)	1·384 (8)	C(8)—C(9)	1·388 (8)
C(1)–C(6)	1·368 (9)	C(8)—C(13)	1·381 (8)
C(2)–C(3)	1·375 (10)	C(9)—C(10)	1·368 (9)
C(3)–C(4)	1·367 (10)	C(10)–C(11)	1·384 (9)
C(4)–C(5)	1·377 (9)	C(11)–C(12)	1·371 (9)
C(5)–C(6)	1·363 (8)	C(12)–C(13)	1·392 (8)
C(6)–N(2)	1·450 (8)	C(13)–N(1)	1·398 (8)
C(7)–C(8)	1·454 (8)	N(1)—N(2)	1·320 (7)
C(7)–N(3)	1·352 (8)	N(1)–O(1)	1·248 (7)
C(7)–O(2)	1·228 (7)	N(2)—N(3)	1·328 (7)

Table 4. *Bond angles* (°)

C(6)—C(1)—C(2)	117·3 (5)	C(10)–C(9)–C(8)	120·1 (5)
C(3)—C(2)—C(1)	120·4 (6)	C(11)–C(10)—C(9)	120·3 (5)
C(4)—C(3)—C(2)	120·5 (5)	C(12)–C(11)–C(10)	121·3 (5)
C(5)—C(4)—C(3)	120·0 (5)	C(13)–C(12)–C(11)	117·5 (5)
C(6)—C(5)—C(4)	118·3 (5)	C(12)–C(13)–C(8)	122·2 (5)
C(5)—C(6)—C(1)	123·4 (5)	N(1)—C(13)–C(8)	118·9 (5)
N(2)—C(6)—C(1)	116·5 (5)	N(1)—C(13)–C(12)	118·9 (5)
N(2)—C(6)—C(5)	119·9 (5)	N(2)—N(1)—C(13)	117·2 (4)
N(3)—C(7)—C(8)	117·9 (4)	O(1)—N(1)—C(13)	121·8 (4)
O(2)—C(7)—C(8)	122·4 (5)	O(1)—N(1)—N(2)	121·0 (4)
O(2)—C(7)—N(3)	119·7 (5)	N(1)—N(2)—C(6)	117·9 (4)
C(9)—C(8)—C(7)	121·6 (5)	N(3)—N(2)—C(6)	114·8 (4)
C(13)–C(8)—C(7)	119·8 (5)	N(3)—N(2)—N(1)	127·4 (4)
C(13)–C(8)–C(9)	118·6 (5)	N(2)—N(3)—C(7)	118·6 (4)

were calculated. Final atomic coordinates are given in Table 1, temperature factors in Table 2, bond lengths in Table 3 and bond angles in Table 4.

Discussion. The identification of C, N and O atoms follows from chemical considerations, bond lengths and temperature factors. Structure (IV) was correct (Fig. 1). The molecules are packed with the sandwich structure common among planar organic molecules with benzenoid rings at van der Waals separations (Fig. 2). This characteristic packing is also observed between the 2-phenyl groups and probably controls the angle of twist observed (59°) between the 2-phenyl plane and the plane of the betaine system.

Table 2. *Anisotropic vibrational amplitudes* ($\text{Å}^2 \times 10^4$)

$$T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{13}hla^*c^* + 2U_{12}hka^*b^*)\right]$$

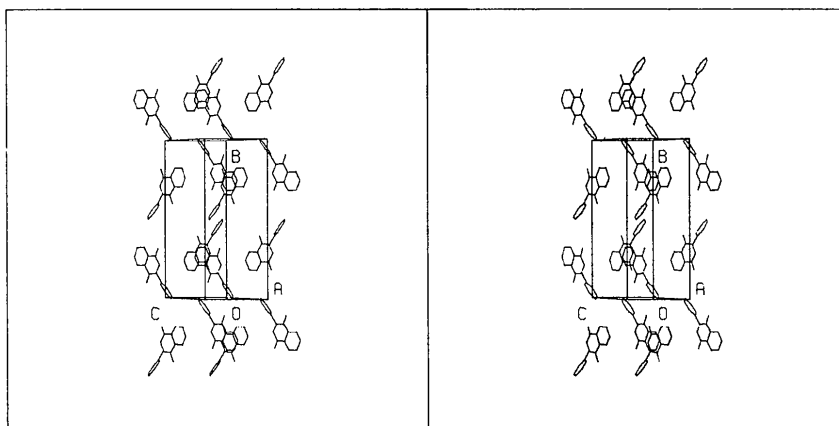|	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	473 (21)	400 (17)	572 (21)	6 (14)	352 (19)	33 (14)
C(2)	574 (23)	428 (19)	888 (28)	77 (19)	528 (23)	107 (17)
C(3)	839 (31)	348 (17)	1016 (32)	−20 (19)	754 (29)	49 (19)
C(4)	765 (30)	405 (19)	739 (25)	−163 (17)	556 (25)	−127 (18)
C(5)	569 (21)	406 (17)	516 (19)	−42 (14)	376 (18)	−44 (15)
C(6)	436 (19)	306 (14)	461 (17)	25 (12)	333 (16)	24 (13)
C(7)	296 (15)	322 (14)	338 (15)	1 (11)	191 (13)	−6 (11)
C(8)	264 (15)	407 (16)	295 (15)	−17 (11)	160 (13)	4 (12)
C(9)	386 (18)	452 (18)	453 (18)	−43 (13)	276 (16)	52 (14)
C(10)	417 (20)	657 (23)	473 (19)	−42 (16)	285 (17)	117 (17)
C(11)	310 (17)	825 (26)	386 (17)	−9 (16)	236 (15)	75 (17)
C(12)	365 (19)	623 (21)	386 (17)	0 (14)	263 (16)	−49 (15)
C(13)	277 (17)	447 (16)	319 (15)	−9 (12)	192 (14)	−2 (12)
N(1)	387 (15)	337 (13)	442 (14)	7 (10)	305 (13)	−47 (10)
N(2)	333 (14)	342 (12)	390 (13)	−9 (10)	257 (12)	−19 (10)
N(3)	349 (14)	304 (12)	443 (14)	12 (10)	290 (13)	−15 (10)
O(1)	720 (19)	450 (14)	896 (20)	−11 (12)	617 (17)	−129 (12)
O(2)	375 (12)	334 (11)	580 (14)	−4 (9)	310 (11)	−44 (9)

Fig. 2. Packing diagram for 2-phenyl-1,2,3-triaziniumbetaine 1-oxide. Note the sandwich-dimer arrangement between 2-phenyl groups from different molecules.

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1'-[4-(4-Fluorophenyl)-4-oxobutyl]-[1,4'-bipiperidine]-4'-carboxamide (Pipamperone)

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Abstract. $C_{21}H_{30}N_3O_2F$, triclinic, $P\bar{1}$, $a = 12\cdot488$ (7), $b = 11\cdot177$ (7), $c = 9\cdot808$ (6) Å; $\alpha = 115\cdot34$ (2), $\beta = 115\cdot65$ (2), $\gamma = 99\cdot23$ (1)°, 25 °C; M.W. 375·2; $Z = 2$.

Introduction. Slow evaporation of a solution in ethanol yielded transparent crystals of this neuroleptic.

The lattice parameters were obtained from the setting angles of ten reflexions measured on a Picker card-controlled diffractometer. The experimental conditions during the measurement of the intensities are given in Table 1.

Table 1. *Experimental conditions*

Source Cu $K\bar{\alpha}$; $\lambda = 1\cdot5418$ Å; $\omega - 2\theta$ scan
$\theta_{max} = 60°$; confidence level: 2·5
Total number of independent reflexions: 2998
Total observed: 2661

The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971) and refined by block-diagonal least-squares calculations with the programs written by Ahmed, Hall, Pippy & Huber (1966). The positions of the hydrogen atoms were determined from a difference map. Only the positional parameters were refined for these atoms. A
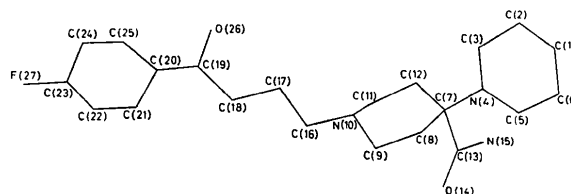

Fig. 1. Conformation of the molecule and numbering.