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Structure of *N,N*-Bis(*p*-nitrophenylsulfonyl)phenethylamine,* $C_{20}H_{17}N_3O_8S_2$

BY V. A. CURTIS

Department of Chemistry, Northeastern Illinois University, Chicago, Illinois 60625, *USA*

AND S. F. PAVKOVIC

Department of Chemistry, Loyola University of Chicago, Chicago, Illinois 60626, *USA*

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Abstract. $M_r = 491{\cdot}5$, monoclinic, $P2_1/c$, $a = 9{\cdot}2355$ (6), $b = 10{\cdot}5196$ (7), $c = 22{\cdot}5386$ (16) Å, $\beta = 97{\cdot}62$ (1)°, $V = 2170{\cdot}4$ (3) Å^3, $Z = 4$, $D_m = 1{\cdot}49$ (2), $D_x = 1{\cdot}505$ (1) Mg m^{-3}, $\lambda(\text{Cu } K\bar{\alpha}) = 1{\cdot}5418$ Å, $\mu(\text{Cu } K\bar{\alpha}) = 25{\cdot}7$ cm^{-1}, $F(000) = 1016$, $T = 294$–296 K, final $R = 0{\cdot}048$ for 2772 reflections. The average S–N bond length is 0·29 (5) Å longer than in dibenzenesulfonamide.

Introduction. The title compound is prepared by the reaction of one equivalent of phenethylamine with two equivalents of *p*-nitrobenzenesulfonyl chloride in a two-step process (DeChristopher, Adamek, Klein, Lyon & Baumgarten, 1975). It is one in a series of compounds being studied for their reactivity in a solid-state and stereoselective pyrolytic *cis*-elimination reaction. This compound is of particular interest because it is inactive in this regard (Curtis, Knutson & Baumgarten, 1981). The structure determination was undertaken to identify molecular parameters leading to an understanding of its inactivity.

Experimental. Large square-tabular crystals from acetone, D_m *via* flotation in C_6H_5Cl/CCl_4, cut fragment of 0·33 × 0·48 × 0·51 mm, automated Picker diffractometer, lattice parameters from least-squares refinement of $\pm 2\theta$ pairs of 15 reflections with $34 < \theta < 39°$, ratio of maximum to minimum empirical absorption correction of 1·30 (φ-scan technique), intensities *via* θ:2θ method to a maximum $(\sin\theta)/\lambda$ of 0·56 Å^{-1} for h, k, $\pm l$ reflections, intensity variations less than 2% for three standards monitored every 50 reflections; 3675 reflections measured, 3223 unique, 951 unobserved [$I < 2{\cdot}0\sigma(I)$]. Structure solved by direct methods (*MULTAN*80), refinement based on magnitudes of F (*SHELX*76), all H atoms located in a difference Fourier electron density map and included at idealized positions, refinement of 33 anisotropic non-H atoms and 2 isotropic H-group temperature factors (ring and non-ring H); $R = 0{\cdot}048$, $wR = 0{\cdot}065$ and $S = 1{\cdot}15$; $w = 1/[\sigma^2(F) + (0{\cdot}0065)F^2]$, ratio of maximum least-squares shift to error in final refinement cycle of 0·013 for C(18) y coordinate, maximum and minimum heights in final difference Fourier electron density map of 0·15 and −0·18e$^-$, non-H atom scattering factors from Cromer & Mann (1968), H-atom scattering factors from Stewart, Davidson & Simpson (1965), f' and f'' values for S from Cromer & Liberman (1970). The computer programs used were *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *SHELX*76 (Sheldrick, 1976), and *ORTEP*II (Johnson, 1971).

Discussion. The atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1, and Table 2 contains the intramolecular bond distances and angles and their e.s.d.'s.† A perspective view showing the molecule and the labeling scheme appears in Fig. 1, and Fig. 2 is a stereoview (Johnson, 1971) of the contents of the unit cell.

A mechanism proposed to account for the pyrolysis reaction involves the central amine N(3) and adjoining atoms. At this site, N(3) is 0·232 (1) Å above the plane of the S(1), S(2), C(13) atoms; the most strained torsion angle including N(3) is 22·8 (8)° for the O(5)–S(2)–N(3)–C(13) chain of atoms; and the O(5)–C(13) contact distance spanning N(3) is 2·798 (8) Å.

The structures of dibenzenesulfonamide and its sodium salt have been reported (Cotton & Stokely, 1970). The title compound more resembles dibenzenesulfonamide in overall conformation although the S–N bond distances are longer by an average of 0·029 (5) Å.

* Also known as *N*-2-phenylethyl-*N,N*-di(*p*-nitrobenzene)sulfonimide.

† Tables of anisotropic thermal parameters, hydrogen-atom coordinates and isotropic thermal parameters, and lists of structure factors (containing h, k, l, $10F_o$ and $10F_c$) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38509 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Final atomic coordinates* ($\times 10^4$, $\times 10^5$ *for* S) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$, $\times 10^4$ *for* S)

$U_{eq} = \frac{1}{3}\sum U_{ii}$.

	x	*y*	*z*	U_{eq}
S(1)	32599 (8)	66951 (8)	9017 (3)	408 (9)
S(2)	60771 (9)	67547 (9)	16296 (4)	468 (10)
O(1)	4120 (3)	6128 (3)	493 (1)	53 (3)
O(2)	2181 (3)	7618 (2)	700 (1)	55 (3)
O(3)	621 (4)	1547 (3)	2054 (2)	80 (4)
O(4)	−397 (4)	2982 (4)	2528 (2)	95 (4)
O(5)	6719 (3)	7512 (3)	2116 (1)	69 (3)
O(6)	5938 (3)	5415 (3)	1705 (1)	63 (3)
O(7)	10367 (3)	6704 (3)	−484 (1)	71 (3)
O(8)	9895 (4)	8702 (4)	−478 (2)	89 (4)
N(1)	397 (4)	2656 (4)	2169 (2)	64 (4)
N(2)	9767 (3)	7634 (4)	−302 (1)	56 (3)
N(3)	4417 (3)	7402 (3)	1426 (1)	42 (3)
C(1)	2401 (3)	5483 (3)	1268 (1)	40 (3)
C(2)	2974 (4)	4258 (3)	1291 (2)	46 (3)
C(3)	2310 (4)	3323 (3)	1586 (2)	46 (3)
C(4)	1100 (4)	3644 (3)	1855 (2)	46 (3)
C(5)	518 (4)	4847 (4)	1831 (2)	52 (4)
C(6)	1175 (4)	5782 (3)	1531 (2)	48 (3)
C(7)	7071 (3)	7025 (3)	1021 (2)	44 (3)
C(8)	7447 (4)	6007 (4)	690 (2)	51 (4)
C(9)	8333 (4)	6224 (4)	259 (2)	50 (4)
C(10)	8798 (3)	7423 (3)	165 (2)	47 (3)
C(11)	8438 (4)	8452 (4)	490 (2)	56 (4)
C(12)	7552 (4)	8243 (4)	929 (2)	55 (4)
C(13)	3773 (4)	8178 (4)	1889 (2)	52 (4)
C(14)	3270 (4)	9487 (4)	1685 (2)	58 (4)
C(15)	4388 (4)	10292 (4)	1445 (2)	57 (4)
C(16)	5591 (5)	10707 (5)	1821 (2)	82 (5)
C(17)	6600 (7)	11487 (6)	1581 (3)	108 (8)
C(18)	6453 (8)	11806 (5)	983 (4)	118 (9)
C(19)	5270 (7)	11356 (6)	623 (3)	107 (8)
C(20)	4251 (6)	10604 (5)	849 (2)	78 (5)

Table 2. *Interatomic distances* (Å) *and angles* (°)

S(1)–O(1)	1·424 (2)	C(2)–C(3)	1·375 (5)
S(1)–O(2)	1·422 (3)	C(3)–C(4)	1·382 (5)
S(1)–N(3)	1·660 (3)	C(4)–C(5)	1·373 (5)
S(1)–C(1)	1·764 (3)	C(5)–C(6)	1·379 (5)
S(2)–O(5)	1·419 (3)	C(7)–C(8)	1·377 (5)
S(2)–O(6)	1·427 (3)	C(7)–C(12)	1·380 (5)
S(2)–N(3)	1·685 (3)	C(8)–C(9)	1·368 (5)
S(2)–C(7)	1·771 (3)	C(9)–C(10)	1·359 (5)
O(3)–N(1)	1·219 (5)	C(10)–C(11)	1·372 (5)
O(4)–N(1)	1·213 (5)	C(11)–C(12)	1·383 (5)
O(7)–N(2)	1·221 (4)	C(13)–C(14)	1·507 (6)
O(8)–N(2)	1·203 (5)	C(14)–C(15)	1·491 (5)
N(1)–C(4)	1·458 (5)	C(15)–C(16)	1·376 (6)
N(2)–C(10)	1·487 (5)	C(15)–C(20)	1·371 (6)
N(3)–C(13)	1·509 (4)	C(16)–C(17)	1·402 (7)
C(1)–C(2)	1·392 (5)	C(17)–C(18)	1·377 (11)
C(1)–C(6)	1·382 (5)	C(18)–C(19)	1·357 (10)
		C(19)–C(20)	1·379 (7)
O(1)–S(1)–O(2)	120·4 (2)	C(2)–C(3)–C(4)	118·4 (3)
O(1)–S(1)–N(3)	106·6 (1)	N(1)–C(4)–C(3)	118·5 (3)
O(2)–S(1)–N(3)	106·2 (2)	N(1)–C(4)–C(5)	118·6 (3)
O(1)–S(1)–C(1)	108·9 (2)	C(3)–C(4)–C(5)	122·9 (3)
O(2)–S(1)–C(1)	107·7 (2)	C(4)–C(5)–C(6)	118·7 (3)
N(3)–S(1)–C(1)	106·2 (1)	C(1)–C(6)–C(5)	119·2 (3)
O(5)–S(2)–O(6)	119·8 (2)	S(2)–C(7)–C(8)	119·3 (3)
O(5)–S(2)–N(3)	104·8 (2)	S(2)–C(7)–C(12)	118·4 (3)
O(6)–S(2)–N(3)	109·8 (2)	C(8)–C(7)–C(12)	122·1 (3)
O(5)–S(2)–C(7)	107·5 (2)	C(7)–C(8)–C(9)	118·2 (3)
O(6)–S(2)–C(7)	108·3 (2)	C(8)–C(9)–C(10)	119·8 (3)
N(3)–S(2)–C(7)	105·7 (2)	N(2)–C(10)–C(9)	118·8 (3)
O(3)–N(1)–O(4)	123·2 (4)	N(2)–C(10)–C(11)	118·2 (3)
O(3)–N(1)–C(4)	118·7 (4)	C(9)–C(10)–C(11)	123·0 (3)
O(4)–N(1)–C(4)	118·1 (4)	C(10)–C(11)–C(12)	117·7 (4)
O(7)–N(2)–O(8)	124·7 (3)	C(7)–C(12)–C(11)	119·2 (4)
O(7)–N(2)–C(10)	117·4 (3)	N(3)–C(13)–C(14)	114·6 (3)
O(8)–N(2)–C(10)	117·9 (4)	C(13)–C(14)–C(15)	115·4 (3)
S(1)–N(3)–S(2)	119·3 (2)	C(14)–C(15)–C(16)	119·9 (4)
S(1)–N(3)–C(13)	117·3 (2)	C(14)–C(15)–C(20)	120·8 (4)
S(2)–N(3)–C(13)	117·2 (2)	C(16)–C(15)–C(20)	119·3 (4)
S(1)–C(1)–C(2)	119·6 (2)	C(15)–C(16)–C(17)	118·2 (5)
S(1)–C(1)–C(6)	118·8 (3)	C(16)–C(17)–C(18)	122·3 (6)
C(2)–C(1)–C(6)	121·6 (3)	C(17)–C(18)–C(19)	117·9 (5)
C(1)–C(2)–C(3)	119·2 (3)	C(18)–C(19)–C(20)	120·9 (6)
		C(15)–C(20)–C(19)	121·4 (5)

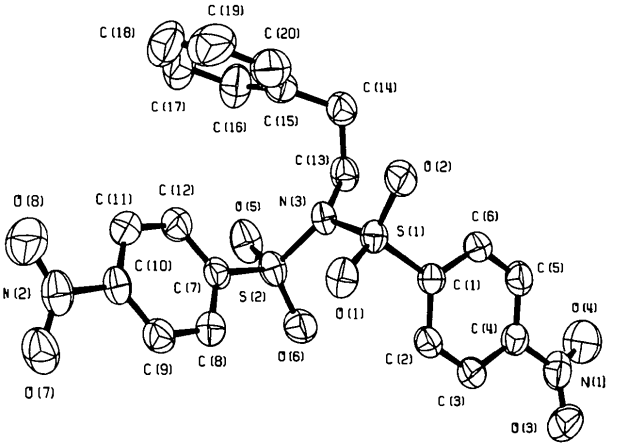

Fig. 1. A perspective view of $C_{20}H_{17}N_3O_8S_2$ showing the crystallographic labeling scheme. Thermal ellipsoids are depicted at the 50% probability level.

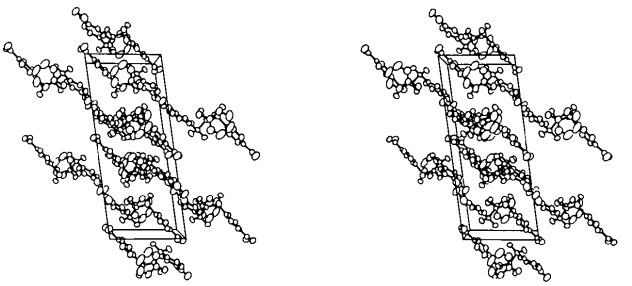

Fig. 2. A stereoview of the contents of a unit cell of $C_{20}H_{17}N_3O_8S_2$. The view shows the *a* axis as horizontal and the *c* axis as approximately vertical.

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