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Structure of 2-[N-(2-Hydroxyethyl)]aminomethyl-3-phenylthioindole

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Abstract. $C_{17}H_{18}N_2OS$, $M_r = 298.4$, orthorhombic, *Pbca*, $a = 36.276$ (7), $b = 10.469$ (3), $c = 8.229$ (3) Å, $V = 3125.2$ (5) Å³, $Z = 8$, $D_x = 1.27$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 1.73$ mm⁻¹, $F(000) = 1264$, $T = 293$ K, $R = 0.060$, $wR = 0.063$ for 1709 reflections. The indole moiety is planar with the S atom -0.051 (1) Å out of the plane. The phenyl ring is oriented at an angle of 82.3 (1)^o to the indole moiety. The structure is stabilized by an intra- and two intermolecular hydrogen bonds [N—H \cdots O 2.858 (5) Å and O—H \cdots N 2.766 (6) Å].

Experimental. Colourless rectangular needle-shaped crystals were grown by slow evaporation in ethanol. The preliminary details of the crystal were obtained by oscillation and Weissenberg photographs. A crystal with dimensions $0.1 \times 0.25 \times 0.3$ mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer. Cell dimensions were determined from setting angles of 25 reflections having $15 \leq \theta \leq 22^\circ$. Intensity data were collected by $\omega/2\theta$ -scan technique using Cu $K\alpha$ radiation between 2 and 130° in 2θ ; range in *hkl*: $0 \leq h \leq 42$; $0 \leq k \leq 12$; $0 \leq l \leq 9$. Two standard reflections were monitored after every 200 reflections and showed 2.2% intensity variation for which correction was applied. A total of 2389 reflections were measured of which 1709 reflections with $I \geq 3\sigma(I)$ were used for refinement. The intensities were corrected for background, Lorentz and polarization effects and absorption (maximum/minimum transmission factors 0.987/0.923). The structure was solved by Patterson and difference Fourier synthesis using *SHELXS86* (Sheldrick, 1986), all the H atoms were located from difference Fourier syntheses. Full-

matrix least-squares refinement for 262 parameters was with anisotropic thermal parameters for the non-H atoms and isotropic for the H atoms using the *SHELX76* (Sheldrick, 1976) program. Final $R = 0.060$ and $wR = 0.063$, $w = 2.22/[\sigma^2(F) + gF^2]$, where $g = 0.0013$; the refinement terminated when shift/e.s.d. for non-H atoms was less than 0.002; goodness of fit $S = 1.5$. The maximum and minimum electron densities in the final ΔF map were 0.59 and -0.26 e Å⁻³. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The structural geometry and graphics were obtained using *PARST* (Nardelli, 1983) and *PLUTO* (Motherwell & Clegg, 1978). All calculations were performed on a MicroVAX II computer. The final positional parameters of the non-H atoms with B_{eq} values are given in Table 1.* A perspective view of the molecule with numbering scheme and unit-cell packing are shown in Figs. 1 and 2 respectively. Bond lengths, bond angles, selected torsion angles and the hydrogen-bonding scheme are given in Table 2.

Related literature. The title compound is the key intermediate for the synthesis of tetrahydropyrazino[1,2-*a*]indole. The importance of these

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, torsion angles and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55325 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0069]

Table 1. Fractional coordinates and equivalent isotropic thermal parameters (Å²)

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B _{eq}
S	0.3482 (1)	0.1467 (1)	0.1526 (2)	4.76 (4)
N1	0.4314 (1)	-0.0631 (4)	0.2992 (5)	3.50 (10)
C2	0.4135 (1)	0.0114 (4)	0.1863 (5)	3.33 (11)
C3	0.3800 (1)	0.0453 (4)	0.2482 (6)	3.55 (11)
C4	0.3767 (1)	-0.0096 (4)	0.4060 (6)	3.36 (11)
C5	0.3498 (1)	-0.0067 (5)	0.5275 (7)	4.49 (14)
C6	0.3559 (2)	-0.0749 (6)	0.6678 (8)	5.72 (20)
C7	0.3880 (2)	-0.1432 (6)	0.6933 (7)	5.37 (17)
C8	0.4153 (2)	-0.1462 (5)	0.5766 (7)	4.62 (16)
C9	0.4095 (1)	-0.0774 (4)	0.4330 (6)	3.34 (13)
C10	0.3164 (1)	0.0409 (4)	0.0594 (6)	3.76 (12)
C11	0.2912 (2)	0.0959 (6)	-0.0436 (8)	5.67 (20)
C12	0.2656 (2)	0.0222 (7)	-0.1229 (10)	6.81 (23)
C13	0.2652 (2)	-0.1082 (7)	-0.1036 (9)	5.83 (19)
C14	0.2900 (2)	-0.1628 (6)	0.0012 (8)	5.27 (17)
C15	0.3159 (1)	-0.0907 (5)	0.0814 (7)	4.27 (13)
C16	0.4316 (1)	0.0447 (5)	0.0295 (6)	3.73 (13)
N17	0.4598 (1)	0.1462 (3)	0.0419 (5)	3.66 (10)
C18	0.4445 (1)	0.2711 (5)	0.0882 (7)	3.90 (13)
C19	0.4751 (1)	0.3643 (5)	0.1231 (6)	4.11 (14)
O20	0.4980 (1)	0.3230 (3)	0.2536 (5)	4.24 (8)

Table 2. Bond distances (Å), bond angles (°), selected torsion angles (°) and hydrogen-bonding geometry (Å, °)

S—C3	1.754 (5)	C8—C9	1.400 (7)
S—C10	1.774 (5)	C10—C11	1.373 (8)
N1—C2	1.376 (6)	C10—C15	1.390 (7)
N1—C9	1.366 (6)	C11—C12	1.372 (10)
C2—C3	1.365 (5)	C12—C13	1.374 (10)
C2—C16	1.489 (6)	C13—C14	1.371 (10)
C3—C4	1.425 (7)	C14—C15	1.374 (8)
C4—C5	1.397 (6)	C16—N17	1.478 (6)
C4—C9	1.403 (5)	N17—C18	1.471 (6)
C5—C6	1.375 (9)	C18—C19	1.506 (6)
C6—C7	1.382 (10)	C19—O20	1.425 (6)
C7—C8	1.380 (9)		
C3—S—C10	104.1 (2)	C4—C9—C8	121.4 (4)
C2—N1—C9	109.4 (3)	N1—C9—C8	130.5 (4)
N1—C2—C16	120.6 (3)	N1—C9—C4	108.1 (4)
N1—C2—C3	108.4 (4)	S—C10—C15	124.8 (3)
C3—C2—C16	130.9 (4)	S—C10—C11	116.0 (4)
S—C3—C2	125.1 (4)	C11—C10—C15	119.2 (4)
C2—C3—C4	108.1 (3)	C10—C11—C12	120.5 (6)
S—C3—C4	126.7 (3)	C11—C12—C13	120.7 (7)
C3—C4—C9	106.1 (4)	C12—C13—C14	118.7 (7)
C3—C4—C5	134.6 (4)	C13—C14—C15	121.4 (6)
C5—C4—C9	119.3 (4)	C10—C15—C14	119.4 (5)
C4—C5—C6	118.5 (4)	C2—C16—N17	114.4 (4)
C5—C6—C7	122.1 (6)	C16—N17—C18	113.3 (3)
C6—C7—C8	120.7 (6)	N17—C18—C19	110.3 (3)
C7—C8—C9	117.9 (6)	C18—C19—O20	112.1 (4)
N1—C2—C16—N17	-77.0 (5)	C16—N17—C18—C19	171.4 (4)
C2—C16—N17—C18	-65.5 (5)	N17—C18—C19—O20	-60.7 (5)
D—H...A	D...A	H—A	D—H...A
N17—H17...O20	2.895 (5)	2.34 (5)	112 (4)
N1—H1...O20 ⁱ	2.858 (5)	1.87 (4)	176 (4)
O20—H20...N17 ⁱⁱ	2.766 (6)	2.00 (8)	175 (8)

Symmetry code: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$, (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

compounds is increasing as they are all pharmacologically active compounds (Gadaginath & Siddappa, 1975; Hendi & Basangoudar, 1981). Glioxin, a fungal metabolite, carries pyrazino[1,2-*a*]indole as its backbone (Waring, Eichner, Palni & Mullbacher, 1986). Crystal structures of compounds related to the title compound have been reported

previously by Chandrakantha, Nirmala, Puttaraja & Nethaji (1990), Falkenberg & Carlström (1971), Vijayalakshmi & Srinivasan (1975), Roychowdhury & Basak (1975), Chandrasekhar & Pattabhi (1980), and Everett, Reynolds, Sparks, Pangborn, Strong, Bailey, Dauter, Helliwell & Hollinshead (1989). As expected, there is an intramolecular hydrogen bond between the atoms N17 and O20 of the title compound [N17...O20 2.895 (5) Å]. The atoms N17, C18, C19, O20 and H17 form approximately a closed five-membered ring which assumes a 'twist' conformation.

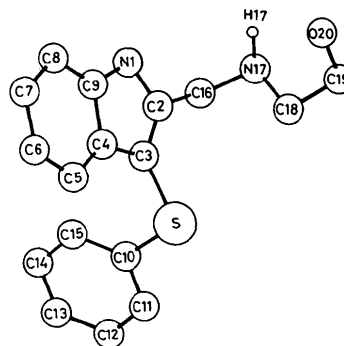


Fig. 1. A perspective view of the molecule with numbering scheme.

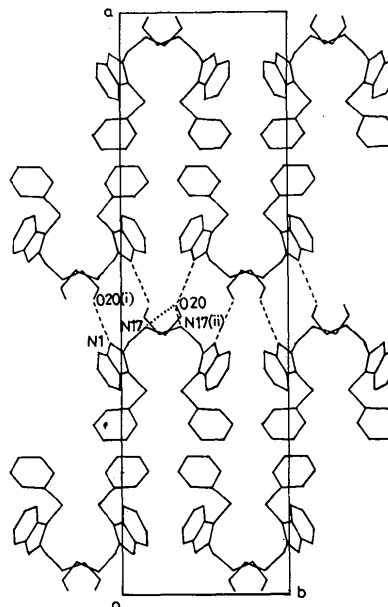


Fig. 2. Unit-cell packing of the molecules down the *c* axis. Intermolecular hydrogen bonding is indicated by dashed lines and intramolecular hydrogen bonding by dotted lines; symmetry code: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

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Structure of 2-(4-Nitrophenyl)-2-trichloromethyloxirane

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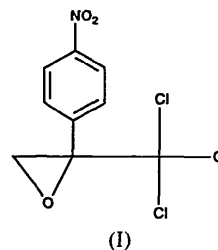
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Abstract. $C_9H_6Cl_3NO_3$, $M_r = 282.51$, monoclinic, $P2_1/n$, $a = 6.104$ (2), $b = 19.538$ (2), $c = 9.286$ (2) Å, $\beta = 93.28$ (2)°, $V = 1105.6$ (7) Å³, $Z = 4$, $D_x = 1.697$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.820$ mm⁻¹, $F(000) = 568$, $T = 292$ K, final $R = 0.068$ for 885 reflections. The title compound is a halogenated *p*-nitroacetophenone synthesized under phase-transfer catalytic (PTC) conditions. The molecules in the crystal lattice are held together by van der Waals forces (m.p. 387 K). Bond lengths and angles are internally consistent and the van der Waals interactions are normal. The nitrophenyl, trichloromethyl and oxirane groups are oriented in a slightly distorted trigonal planar arrangement. The averaged bond angles within the epoxy group and about the trichloro-C(*sp*³) atom are 60.0 (7) and 109.5 (17)°, respectively. Selected mean geometric details are: C—O = 1.428 (4), N—O = 1.208 (3), C—Cl = 1.766 (3) and C—C (ring) = 1.383 (9) Å.

Experimental. Clear colorless irregular shaped crystals of the title compound (I) were prepared

according to a published procedure (Reeves, Creswell, Glass & Scheide, 1985). Crystals suitable for X-ray analysis were obtained after recrystallization from ethanol.



After verifying homogeneity and the biaxial nature of the system by a conoscopic examination, a single crystal of dimensions 0.15 × 0.26 × 0.59 mm was selected for data collection. Data were collected on an Enraf–Nonius CAD-4F diffractometer equipped with a graphite monochromator utilizing Mo $K\alpha$ radiation, take-off angle 5.8°. 25 diffractometer-measured reflections with $4.57 \leq 2\theta \leq 15.59^\circ$ were used to obtain the orientation matrix and initial cell parameters. Final unit-cell parameters were

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