Absorption correction: empirical ψ scans (Siemens, 1994)
T_{min} = 0.853, T_{max} = 0.939
3939 measured reflections
2934 independent reflections
1828 reflections with $I > 2\sigma(I)$
$h = -1 \rightarrow 12$
$k = -1 \rightarrow 9$
$l = -22 \rightarrow 22$
3 standard reflections every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 0.901
2934 reflections
216 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.266 e Å^{-3}
$\Delta\rho_{min}$ = −0.211 e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.019 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected bond lengths (Å)*

S1—O2	1.4214 (15)	S1—C7	1.756 (2)
S1—O3	1.4232 (14)	O1—C5	1.431 (2)
S1—O1	1.584 (2)	N1—C1	1.365 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2	0.96 (2)	2.47 (2)	2.901 (2)	107 (1)
N1—H1N1···O3^{i}	0.81 (2)	2.31 (2)	3.098 (3)	165 (2)
N1—H2N1···O2^{ii}	0.79 (3)	2.52 (3)	3.278 (3)	162 (2)

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

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93 and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1211). Services for accessing these data are described at the back of the journal.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.
Kurita, K. (1974). *Chem. Ind. (London)*, p. 345.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1990). *SHELXTL/PC Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93 *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1994). *XSCANS Users Manual*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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2-(4-Nitroanilino)-2-phenylethanol

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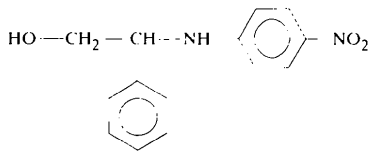
Abstract

In the title compound, $C_{14}H_{14}N_2O_3$, the nitro group is twisted from coplanarity with the benzene ring by 3.8 (3)°. The benzene ring is perpendicular to the phenyl ring. The molecules are packed around the threefold axis to form an infinite channel containing disordered solvent molecules. C—H···O, O—H···O and N—H···O intermolecular hydrogen bonds stabilize the crystal structure.

Comment

The β-aminoalcohol sequence plays an important role in organic as well as in medicinal chemistry (Goodman & Gilman, 1980). Specifically, the β-amino alcohol subunit has been of particular value in the study of acetylcholine metabolism in intact nerve terminal preparations (Rogers *et al.*, 1989). The crystal structure determination of the title compound, (I), one of the above derivatives, was carried out in order to elucidate the molecular conformation.



(I)

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The bond lengths and angles in the structure are normal and agree with reported values (Allen *et al.*, 1987). Both N atoms are in a planar configuration. The benzene and phenyl rings are individually planar and these planes are perpendicular [dihedral angle 90.0 (5)°]. The nitro group is twisted out of the benzene-ring plane by 3.8 (3)°. The molecules are packed around the threefold axis to form a cylindrical void (or channel) with the nitroaniline moieties defining the outer boundary and the phenyl rings pointing towards the threefold axis (Fig. 2). The hydroxy groups lie on the outer surface of the channel. The benzene planes of the inversion-related molecules pack as parallel planes with a short contact of 3.514 (9) Å between the C2 and C6 atoms. The molecules defining the channel are involved in weak C—H···O intermolecular hydrogen bonds and O—H···O hydrogen bonds link molecules of neighbouring columns. The N—H group forms a weak intermolecular N—H···O hydrogen bond with the O2 atom (Table 2).

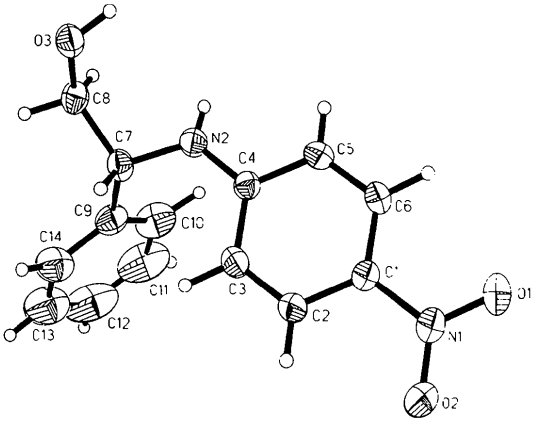

Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

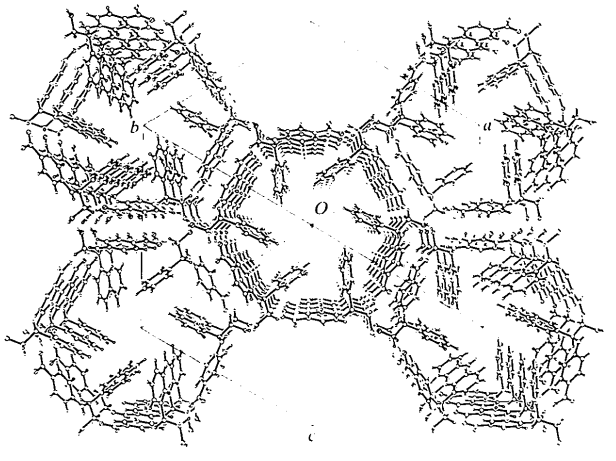

Fig. 2. Packing of the molecules viewed normal to the (111) plane.

Experimental

A mixture of styrene oxide (1 g, 8.32 mmol), *p*-nitroaniline (1.37 g, 9.98 mmol) and alumina (5 g) was refluxed in 30 ml of dry benzene (353 K). After completion of the reaction (followed by TLC), the reaction mixture was filtered and the solvent evaporated to dryness. Chromatographic purification of the residue furnished two products: 2-(4-nitroanilino)-2-phenylethanol as the major product (83%) and 2-hydroxy-*N*-(4-nitrophenyl)phenylethylamine (less than 10%) as the minor product. The structures of both isomers were confirmed by spectral data (Sriraghavan & Ramakrishnan, 1997). Single crystals were grown by slow evaporation of solutions of the compounds in chloroform–methanol (1:1) solvent systems.

Crystal data

$C_{14}H_{14}N_2O_3$
M_r = 258.27
Trigonal
$R\bar{3}$
a = 18.618 (2) Å
α = 118.34 (1)°
V = 2141 (4) Å^3
Z = 6
D_x = 1.202 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 42 reflections
θ = 5.373–12.386°
μ = 0.086 mm^{-1}
T = 293 (2) K
Needle
0.78 × 0.36 × 0.26 mm
Yellow

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4190 measured reflections
3220 independent reflections
1487 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.51°
h = −12 → 23
k = −24 → 3
l = −12 → 24
3 standard reflections every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R(F)$ = 0.045
$wR(F^2)$ = 0.109
S = 1.069
3220 reflections
216 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.123 e Å^{-3}
$\Delta\rho_{min}$ = −0.166 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—N1	1.224 (3)	N1—C1	1.441 (7)
O2—N1	1.231 (5)	N2—C4	1.356 (6)
O3—C8	1.426 (8)	N2—C7	1.443 (4)
O1—N1—O2	121.5 (4)	O2—N1—C1	119.6 (2)
O1—N1—C1	118.9 (4)	C4—N2—C7	124.6 (4)
O1—N1—C1—C2	−176.4 (5)	C4—N2—C7—C9	−75.5 (7)
O2—N1—C1—C2	4.3 (7)	N2—C7—C8—O3	−63.2 (5)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1O3···O3^i	0.88 (3)	1.85 (4)	2.710 (5)	164 (3)
C2—H2···O2	0.92 (5)	2.40 (5)	2.733 (7)	101 (2)
C6—H6···O1^{ii}	0.98 (3)	2.42 (4)	3.346 (5)	159 (5)

C8—H8A···O2^{iii}	1.01 (3)	2.48 (4)	3.481 (4)	172 (5)
C8—H8B···O1^{iv}	0.99 (4)	2.54 (3)	3.490 (6)	160 (3)
N2—H1N2···O2^{iv}	0.74 (5)	2.62 (4)	3.209 (5)	138 (2)

Symmetry codes: (i) $y, z, x-1$; (ii) $-x, -1-y, -1-z$; (iii) $1-y, 1-z, 1-x$; (iv) $-y, -z, -x$.

The title structure was solved by direct methods and refined by full-matrix least squares. 11 H atoms were located from a difference Fourier map and refined isotropically; the remaining three not found in the map were geometrically fixed and allowed to ride on their parent atoms. At this stage, the refinement converged to an R value of 0.054 (wR = 0.184). The s.u.'s of the structural parameters were high and the difference map showed peaks (0.30 to 0.45 e Å^{-3}) of almost equal interval (around 1 to 1.1 Å) on the threefold axis and origin. Refinement based on a disordered solvent model led to unstable refinement with very high displacement parameters. A search for solvent-accessible voids in the crystal using *PLATON* (Spek, 1990) showed a potential solvent volume of 309.9 Å^3 and subsequent application of *SQUEEZE* procedures (van der Sluis & Spek, 1990) showed only one relevant void (or channel) with a solvent-accessible volume of 206 Å^3. The number of electrons found in that channel is 12 and the estimated volume per atom is 143 Å^3. This indicates that the void is only partially occupied and that the original contents had probably disappeared by the time the crystal was used for data collection, without collapsing the structure. Further refinement of the structure with solvent-free reflection data obtained from the above procedure converged to an R value of 0.045 (wR = 0.125) and the accuracy of structural parameters was found to have improved. The final F_o–F_c listing was generated using the *CALC FCF* option in *PLATON*.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93 and *PLATON*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1162). Services for accessing these data are described at the back of the journal.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.
Goodman, L. S. & Gilman, A. (1980). In *The Pharmacological Basis of Therapeutics*. New York: MacMillan.
Rogers, G. A., Parsons, S. M., Anderson, D. C., Nilsson, L. M., Bahr, B. A., Kornreich, W. D., Kaufman, R., Jacobs, R. S. & Kirtman, B. (1989). *J. Med. Chem.* **32**, 1217–1230.
Sheldrick, G. M. (1990). *SHELXTL/PC User Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1994). *XSCANS Users Manual*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sluis, P. van der & Spek, A. L. (1990). *Acta Cryst.* A**46**, 194–201.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Sriraghavan, K. & Ramakrishnan, V. T. (1997). Unpublished work.

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9-(4-Dimethylaminophenyl)-3,4,6,7,9,10-hexahydro-1,8(2*H*,5*H*)-acridinedione

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Abstract

The title compound, $C_{21}H_{24}N_2O_2$, contains an acridine moiety and a dimethylaminophenyl ring system. The side rings adopt half-chair conformations. The acridine chromophore is perpendicular to the substituted phenyl ring.

Comment

Acridines are potent DNA intercalators, with very sensitive and characteristic fluorescent properties which respond to changes in the microenvironment (Lerman, 1961). Acridines are useful for tagging molecules of interest, but their application is currently limited to covalent modification of small oligonucleotides, as no technology currently exists to attach them to larger DNAs and proteins (Selladurai *et al.*, 1990). Acridine dyes reacting with nucleic acids have received increasing interest as mutagens in micro-organisms (Sivaraman *et al.*, 1996), but relatively little attention has been given to acridine-induced mutation in higher plants, except for barley. Apart from the above, acridinediones are used as antibacterial agents for wound therapy (Acheson, 1956) and as antitumour drugs (Hempel *et al.*, 1979). In view of the above interest, we decided to analyse the conformation of the acridine moiety with respect to a dimethylaminophenyl ring system.

The *ZORTEP* (Zsolnai, 1997) plot of the title molecule, (I), with the atomic numbering scheme is shown in Fig. 1. The acridine moiety is not planar: the central