

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	D...A	D—H...A
N18—HBr3...Br3 ⁱ	1.503	3.341 (10)	160.3
O1W—HBr1...Br1	1.036	3.360 (10)	155.3

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

Table 4. Least-squares-planes data

The equations of the planes can be expressed as $PX + QY + RZ = S$ in direct space with $P = 11.7$, $Q = 8.2$, $R = 1.1$ and $S = 6.7$ for plane A and $P = 14.5$, $Q = 1.3$, $R = 1.2$ and $S = 5.0$ for plane B.

Plane	Δ (Å)
Plane A	
C13*	0.0
C14*	0.0
C17*	0.0
C15	-0.890
C16	-0.561
Plane B	
C14*	0.086
C15*	-0.141
C16*	0.141
C17*	-0.086
C13	-0.464

* Atom included in the definition of the least-squares plane.

Data collection, cell refinement and data reduction were performed using CAD-4 EXPRESS (Enraf-Nonius, 1993) and NONIUS93 (Baudoux & Evrard, 1993). Corrections for background, decay, Lorentz and polarization factors, and absorption effects were included in the data reduction. The structure was solved by direct methods using SHELXS86 (Sheldrick, 1990), resulting in reliable positions for all the non-H atoms. The initial model was refined with SHELXL93 (Sheldrick, 1993). One H atom in the water molecule was not found. PLATON93 (Spek, 1990) was used for the generation of the CIF and geometry analysis. The structures retrieved from the Cambridge Structural Database were BIGTEQ, CORXUC, FOYWAR, FUTXEX, FUTXIB, HACDUK, PDZNCB and VUKBEI. The structural statistics display package VISTA-3D (Allen, Kennard & Taylor, 1983) was used for the statistical analysis. Diagrams were prepared using ORTEP (Johnson, 1965) and VISTA-3D.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1122). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CHI 2HU, England.

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4-[(1-Cyclohexenyl)nitromethyl]-2-(3,4-methylenedioxyphenyl)-3-nitro-3,4-dihydro-2H-1-benzopyran

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Abstract

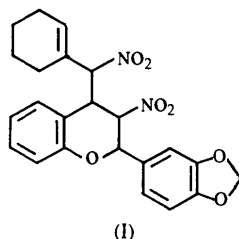
The crystal structure determination of C₂₃H₂₂N₂O₇ shows that the mutual dispositions of the H atoms attached to the four successive chiral centres are *gauche*, *gauche* and *trans*.

Comment

The present study owes its origin to the need for ascertaining the relative stereochemistry in products obtained in the reaction of 1-(1-nitrophenyl)cyclo-

† CDRI Communication No. 4972.

hexene with 3-nitro-2-substituted phenyl-2*H*-1-benzopyrans (Solomonovici & Blumberg, 1965, 1966; Cabrera Escribano, Derri Alcantara & Gomez-Sanchez, 1988). As this reaction is stereoselective, its synthetic utility is increased and in order to provide conclusive evidence for the stereochemistry assigned on the basis of extensive PMR studies (Gupta, 1992), a single-crystal X-ray crystallographic study of the title compound, (I), was undertaken.



The title molecule (Fig. 1) contains two fused-ring systems (*A/B* and *C/D*) and an additional ring (*E*). Rings *B* and *C* are planar [the deviations of the atoms of the least-squares planes are within the range -0.003 (3) to 0.008 (3) Å], while rings *A*, *D* and *E* are puckered. In ring *A*, the deviation of atom C(3) from the least-squares plane through atoms O(1), C(2), C(4), C(5) and C(6) is 0.582 (3) Å and this establishes the sofa conformation of the ring. A value of 0.5 (1)° for torsion angle O(17)—C(14)—C(15)—O(19) in ring *D* indicates that these atoms are planar; atom C(18) deviates slightly [-0.112 (3) Å] from this plane. In ring *E*, atom C(28) deviates by 0.581 (3) Å from the least-squares plane through atoms C(29), C(25), C(26) and C(27), which indicates that the ring has a tendency to adopt a sofa conformation.

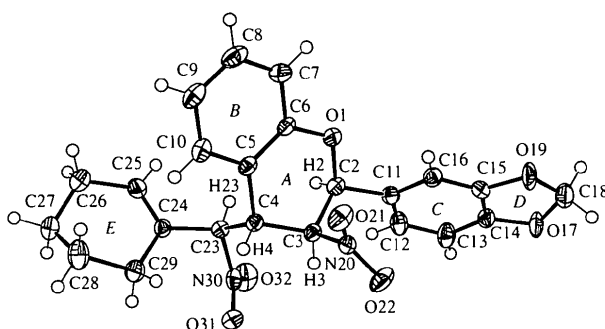


Fig. 1. An ORTEP diagram (Johnson, 1965) showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level for the non-H atoms. Only H atoms attached to chiral centres are numbered.

The torsion angles that define the mutual disposition of the H atoms at the chiral centres are H(2)—C(2)—C(3)—H(3) of 57 , H(3)—C(3)—C(4)—H(4) of 77 and H(4)—C(4)—C(23)—H(23) of 173 °. These values are in agreement with the observed constants for vicinal

coupling of protons H2 and H3 (singlet), H3 and H4 (singlet), and H4 and H23 (8 Hz).

Experimental

The synthesis of the title compound was carried out by reacting 1-(1-nitrophenyl)cyclohexene with 3-nitro-2-[(3,4-methylenedioxy)phenyl]-2*H*-1-benzopyran. Crystals were obtained from a mixture of benzene and ethanol at room temperature by slow evaporation.

Crystal data

$C_{23}H_{22}N_2O_7$
 $M_r = 438.43$
 Monoclinic
 $P2_1/n$
 $a = 10.518$ (2) Å
 $b = 12.932$ (2) Å
 $c = 15.048$ (2) Å
 $\beta = 97.99$ (2)°
 $V = 2026.9$ (6) Å³
 $Z = 4$
 $D_x = 1.437$ Mg m⁻³
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 14 reflections
 $\theta = 10.0$ – 12.5 °
 $\mu = 0.108$ mm⁻¹
 $T = 293$ (2) K
 Block
 $0.20 \times 0.20 \times 0.15$ mm
 Colourless

Data collection

Siemens Stoe four-circle diffractometer
 ω scans
 Absorption correction: none
 7179 measured reflections
 3590 independent reflections
 2794 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.0266$
 $\theta_{max} = 25.08$ °
 $h = -12 \rightarrow 12$
 $k = -15 \rightarrow 15$
 $l = 0 \rightarrow 17$
 3 standard reflections monitored every 97 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R(F) = 0.038$
 $wR(F^2) = 0.095$
 $S = 1.172$
 3590 reflections
 290 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.18$ e Å⁻³
 $\Delta\rho_{min} = -0.18$ e Å⁻³

Extinction correction: *SHELXL93* (Sheldrick, 1993)
 Extinction coefficient: 0.0068 (13)
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

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

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	x	y	z	U_{eq}
O(1)	0.0288 (1)	0.1396 (1)	0.4526 (1)	0.028 (1)
C(2)	0.1344 (2)	0.0786 (1)	0.4330 (1)	0.023 (1)
C(3)	0.2630 (2)	0.1201 (1)	0.4779 (1)	0.020 (1)
C(4)	0.2669 (2)	0.1298 (1)	0.5797 (1)	0.020 (1)
C(5)	0.1436 (2)	0.1774 (1)	0.6014 (1)	0.022 (1)
C(6)	0.0336 (2)	0.1780 (1)	0.5389 (1)	0.023 (1)
C(7)	-0.0791 (2)	0.2224 (1)	0.5585 (1)	0.031 (1)
C(8)	-0.0829 (2)	0.2661 (2)	0.6413 (1)	0.035 (1)

C(9)	0.0253 (2)	0.2670 (1)	0.7045 (1)	0.033 (1)
C(10)	0.1371 (2)	0.2238 (1)	0.6841 (1)	0.028 (1)
C(11)	0.1282 (2)	0.0700 (1)	0.3327 (1)	0.023 (1)
C(12)	0.1805 (2)	-0.0160 (1)	0.2978 (1)	0.030 (1)
C(13)	0.1857 (2)	-0.0268 (2)	0.2065 (1)	0.035 (1)
C(14)	0.1347 (2)	0.0515 (1)	0.1527 (1)	0.026 (1)
C(15)	0.0821 (2)	0.1378 (1)	0.1867 (1)	0.024 (1)
C(16)	0.0776 (2)	0.1506 (1)	0.2764 (1)	0.025 (1)
O(17)	0.1281 (1)	0.0601 (1)	0.0612 (1)	0.039 (1)
C(18)	0.0583 (2)	0.1517 (2)	0.0375 (1)	0.036 (1)
O(19)	0.0387 (1)	0.2046 (1)	0.1178 (1)	0.039 (1)
N(20)	0.2887 (1)	0.2226 (1)	0.4369 (1)	0.023 (1)
O(21)	0.2401 (1)	0.3000 (1)	0.4635 (1)	0.036 (1)
O(22)	0.3549 (1)	0.2226 (1)	0.3769 (1)	0.038 (1)
C(23)	0.2857 (2)	0.0221 (1)	0.6246 (1)	0.022 (1)
C(24)	0.3092 (2)	0.0234 (1)	0.7255 (1)	0.022 (1)
C(25)	0.2192 (2)	-0.0175 (1)	0.7697 (1)	0.030 (1)
C(26)	0.2264 (2)	-0.0166 (2)	0.8688 (1)	0.045 (1)
C(27)	0.3579 (2)	0.0104 (2)	0.9168 (1)	0.036 (1)
C(28)	0.4155 (2)	0.0981 (2)	0.8696 (1)	0.043 (1)
C(29)	0.4279 (2)	0.0712 (2)	0.7734 (1)	0.030 (1)
N(30)	0.3970 (2)	-0.0322 (1)	0.5911 (1)	0.027 (1)
O(31)	0.4992 (1)	0.0138 (1)	0.5967 (1)	0.035 (1)
O(32)	0.3791 (1)	-0.1190 (1)	0.5605 (1)	0.047 (1)

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X-ray Structure Investigations of Potential β -Blockers. II. 6,7-Dimethoxy-1-phenyl-1,2,3,4-tetrahydroisoquinoline

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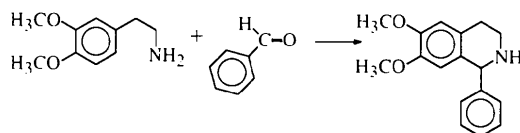
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Abstract

The saturated part of the isoquinoline moiety in the title compound, C₁₇H₁₉NO₂, has a twist conformation and the phenyl ring substituent is in an equatorial position. The absolute configuration of the molecule is *R* and molecules form infinite chains by means of weak hydrogen bonds.

Comment

Tetrahydroisoquinolines represent a class of biologically active phenylethylamines (Brzezińska, 1994). The title compound, (I), seems to be a weak competitive antagonist for β -adrenoreceptors. A series of substituted 1,2,3,4-tetrahydroisoquinolines have been synthesized because of their expected activity. The title compound was obtained by the simple Pictet–Spengler reaction of 2-(3,4-dimethoxyphenyl)ethylamine with benzaldehyde (Sarges, 1974).



(I)

A perspective view of (I) showing the atomic numbering scheme is given in Fig. 1. The saturated part of the isoquinoline moiety has a twist conformation, with a twofold pseudo-axis passing through the midpoints of the C9—C10 and N1—C8 bonds [asymmetry parameter $\Delta_2(\text{N1—C8}) = 0.004(1)$]. The aromatic rings are planar within experimental error. The phenyl ring in position 1 is equatorial and makes an angle of $92.16(8)^\circ$ with the aromatic part of the isoquinoline moiety. The presence of the benzene ring does not cause any significant disturbance within the remainder of the molecule. The skeleton of the molecule, discounting the phenyl ring, displays pseudosymmetry around the twofold axis bi-

The structure was solved by direct methods and the non-H atoms were refined anisotropically using full-matrix least-squares methods. All H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, to which each was bonded for the final cycles of refinement.

Data collection: *DIF4* (Stoe & Cie, 1991a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1991b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Software used to prepare material for publication: *NRCVAX*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: VJ1032). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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