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3-(2-Methyl-2-nitropropyl)-1*H*-indole

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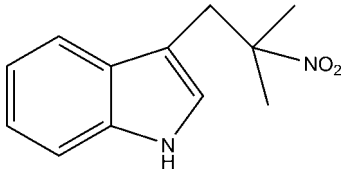
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.057; wR factor = 0.179; data-to-parameter ratio = 14.7.

In the title compound, $C_{12}H_{14}N_2O_2$, the indole ring is essentially planar, with an r.m.s. deviation of 0.0136 Å. In the crystal, pairs of N—H···O hydrogen bonds link the molecules into inversion dimers..

Related literature

The title compound is an intermediate of the β-adrenergic receptor antagonist (β blocker) bucindolol {systematic name: 1-[[2-(3-indolyl)-1,1-dimethylethyl]amino]-3-(2-nitrilearyl-oxy)-2-propanol)}, see: Qiu *et al.*, (2003). For synthetic procedures, see: Kerighbaum *et al.* (1980). For a related structure, see: Léger *et al.* (1984).



Experimental

Crystal data

$C_{12}H_{14}N_2O_2$
M_r = 218.25
Monoclinic, $P2_1/n$
a = 6.1170 (12) Å
b = 10.123 (2) Å
c = 18.868 (4) Å
β = 91.36 (3)°
V = 1168.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.983, T_{max} = 0.992
2354 measured reflections
2141 independent reflections
1089 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.179
S = 1.00
2141 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.86	2.55	3.187 (4)	132

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2541).

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supporting information

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3-(2-Methyl-2-nitropropyl)-1*H*-indole

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S1. Comment

Bucindolol, 1-[[2-(3-indolyl)-1,l-dimethylethyl]-amino]-3-(2-nitrile-aryloxy)-2-propanol, is one of the *β*-adrenergic receptor antagonists (*β* blocker) for the treatment of essential hypertension (Qiu *et al.*, 2003). As a part of our studies on the synthesis of Bucindolol, the title compound (Fig. 1) which is used as the key intermediate, has been synthesized and its crystal structure reported in this article. The crystal structure of the title compound is stabilized by N1—H1A···O2 hydrogen bonds resulting in dimers of molecules lying about inversion centers (Fig. 1 and Tab. 1). The bond distances and angles in the title molecule are in excellent agreement with the corresponding bond distances and angles reported for a related structure (Léger *et al.*, 1984).

S2. Experimental

A mixture of gramine (13.0 g,(0.069 mol), 2-nitropropane (44 g,(0.49 mol), and NaOH pellets (2.9 g, 0.072 mol) was stirred and heated at reflux for 18 h. After the mixture had cooled to 298 K, 10% HOAc (60 ml) was added and stirring was continued for 1 h. The mixture was partitioned between 150 ml each of EtOH and water to afford an organic layer, which was separated, washed three times with water, and dried over $MgS0_4$. Evaporation afforded 16.5 g of dark oil which slowly crystallized on standing at 298 K. Recrystallization of the crude product from EtOH-H_20 (1:1) gave 12.6 g (78%) of the title compound as pure yellow crystals (Kerighbaum *et al.*, 1980). Crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with N—H = 0.86 Å and C—H = 0.93, 0.96 and 0.97 Å, for aryl, methyl and methylene H-atoms, respectively. The U_{iso}(H) were allowed at 1.5U_{eq}(methyl C) or 1.2U_{eq}(non-methyl C/N).

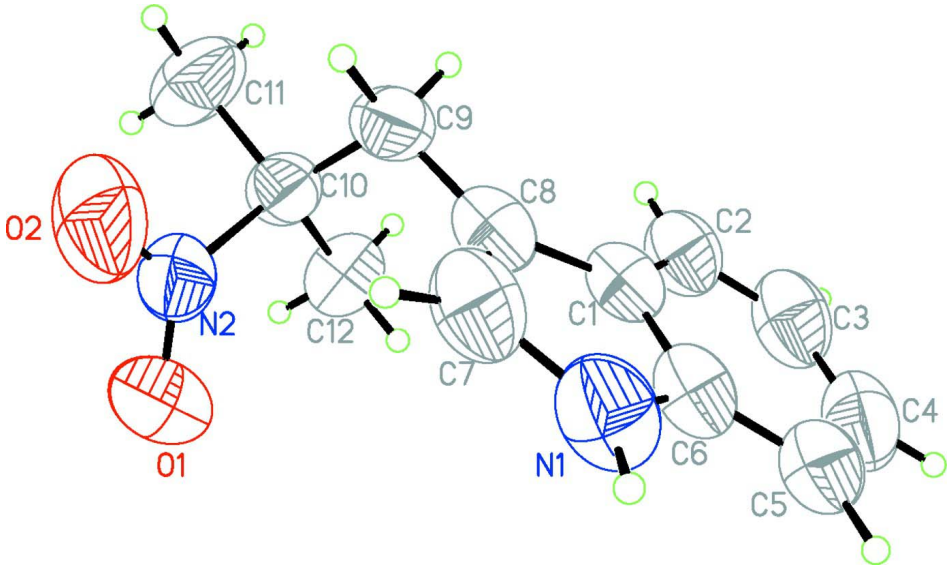

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

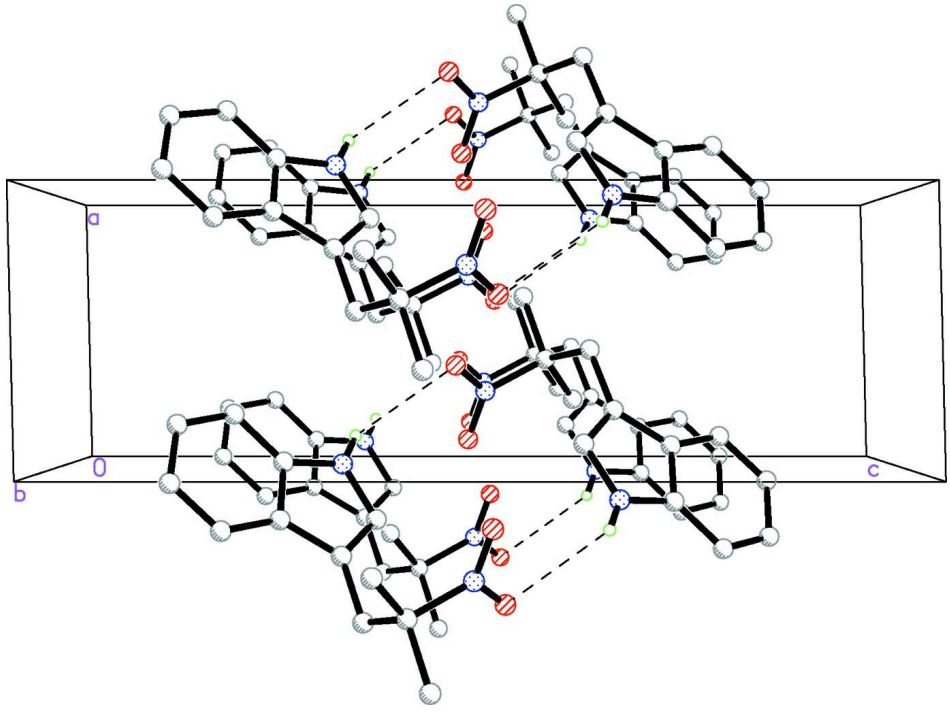

Figure 2

A view of the N—H···O hydrogen bonds (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen- bonding were omitted for clarity.

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Crystal data

$C_{12}H_{14}N_2O_2$
$M_r = 218.25$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.1170$ (12) Å
$b = 10.123$ (2) Å
$c = 18.868$ (4) Å
$\beta = 91.36$ (3)°
$V = 1168.0$ (4) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.241$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, yellow
0.20 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.983$, $T_{max} = 0.992$
2354 measured reflections
2141 independent reflections
1089 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 25.4°$, $\theta_{min} = 2.2°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 12$
$l = -22 \rightarrow 22$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.179$
$S = 1.00$
2141 reflections
146 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.043 (7)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.3793 (4)	0.2257 (3)	1.01215 (14)	0.1125 (10)
C1	0.3885 (5)	0.3891 (3)	0.78945 (14)	0.0565 (8)

N1	0.5544 (5)	0.5489 (3)	0.85178 (14)	0.0795 (9)
H1A	0.6445	0.6098	0.8650	0.095*
O2	0.1178 (5)	0.3613 (3)	1.02354 (13)	0.0997 (9)
C2	0.3631 (5)	0.3039 (3)	0.73199 (16)	0.0680 (9)
H2A	0.2434	0.2474	0.7288	0.082*
N2	0.2086 (5)	0.2746 (3)	0.99150 (14)	0.0704 (8)
C3	0.5157 (7)	0.3042 (4)	0.68028 (18)	0.0818 (11)
H3A	0.4991	0.2472	0.6419	0.098*
C4	0.6966 (7)	0.3888 (4)	0.68414 (19)	0.0860 (11)
H4A	0.7994	0.3858	0.6487	0.103*
C5	0.7254 (6)	0.4763 (4)	0.73929 (19)	0.0784 (10)
H5A	0.8442	0.5335	0.7418	0.094*
C6	0.5676 (5)	0.4748 (3)	0.79116 (16)	0.0651 (8)
C7	0.3738 (6)	0.5096 (3)	0.88767 (17)	0.0742 (10)
H7A	0.3307	0.5450	0.9306	0.089*
C8	0.2668 (5)	0.4125 (3)	0.85229 (14)	0.0593 (8)
C9	0.0639 (5)	0.3434 (3)	0.87513 (15)	0.0681 (9)
H9A	−0.0179	0.3157	0.8330	0.082*
H9B	−0.0259	0.4065	0.9000	0.082*
C10	0.0990 (4)	0.2237 (3)	0.92255 (15)	0.0594 (8)
C11	−0.1206 (5)	0.1680 (4)	0.9453 (2)	0.0956 (13)
H11A	−0.2075	0.2377	0.9646	0.143*
H11B	−0.0964	0.1010	0.9806	0.143*
H11C	−0.1961	0.1303	0.9049	0.143*
C12	0.2438 (5)	0.1190 (3)	0.89062 (18)	0.0751 (10)
H12A	0.3789	0.1583	0.8767	0.113*
H12B	0.1709	0.0811	0.8498	0.113*
H12C	0.2732	0.0511	0.9251	0.113*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0832 (17)	0.165 (3)	0.0884 (18)	0.013 (2)	−0.0211 (15)	−0.0015 (18)
C1	0.0663 (19)	0.0544 (17)	0.0482 (17)	0.0097 (16)	−0.0089 (14)	−0.0007 (14)
N1	0.103 (2)	0.0663 (18)	0.0686 (18)	−0.0168 (17)	−0.0142 (16)	−0.0007 (15)
O2	0.131 (2)	0.104 (2)	0.0646 (15)	0.0075 (17)	0.0064 (14)	−0.0244 (15)
C2	0.085 (2)	0.065 (2)	0.0542 (18)	0.0045 (18)	−0.0038 (16)	−0.0053 (16)
N2	0.0663 (18)	0.087 (2)	0.0576 (16)	−0.0094 (17)	0.0052 (14)	0.0047 (16)
C3	0.115 (3)	0.073 (2)	0.0575 (19)	0.018 (2)	0.005 (2)	−0.0036 (18)
C4	0.099 (3)	0.094 (3)	0.066 (2)	0.024 (3)	0.015 (2)	0.019 (2)
C5	0.080 (2)	0.079 (2)	0.076 (2)	0.001 (2)	−0.007 (2)	0.024 (2)
C6	0.079 (2)	0.0619 (19)	0.0543 (18)	0.0063 (19)	−0.0058 (17)	0.0084 (16)
C7	0.099 (3)	0.069 (2)	0.0540 (19)	0.004 (2)	−0.0044 (19)	−0.0060 (17)
C8	0.072 (2)	0.0553 (18)	0.0500 (17)	0.0110 (17)	−0.0094 (15)	−0.0021 (15)
C9	0.0590 (19)	0.091 (2)	0.0543 (18)	0.0182 (18)	−0.0052 (14)	−0.0103 (17)
C10	0.0523 (17)	0.071 (2)	0.0552 (17)	−0.0026 (16)	−0.0003 (14)	−0.0109 (15)
C11	0.062 (2)	0.117 (3)	0.108 (3)	−0.020 (2)	0.020 (2)	−0.020 (3)
C12	0.075 (2)	0.068 (2)	0.083 (2)	−0.0041 (18)	0.0151 (18)	−0.0052 (18)

Geometric parameters (Å, º)

O1—N2	1.212 (3)	C5—H5A	0.9300
C1—C2	1.391 (4)	C7—C8	1.349 (4)
C1—C6	1.397 (4)	C7—H7A	0.9300
C1—C8	1.435 (4)	C8—C9	1.497 (4)
N1—C7	1.369 (4)	C9—C10	1.519 (4)
N1—C6	1.372 (4)	C9—H9A	0.9700
N1—H1A	0.8600	C9—H9B	0.9700
O2—N2	1.208 (3)	C10—C12	1.516 (4)
C2—C3	1.366 (4)	C10—C11	1.528 (4)
C2—H2A	0.9300	C11—H11A	0.9600
N2—C10	1.538 (4)	C11—H11B	0.9600
C3—C4	1.400 (5)	C11—H11C	0.9600
C3—H3A	0.9300	C12—H12A	0.9600
C4—C5	1.374 (5)	C12—H12B	0.9600
C4—H4A	0.9300	C12—H12C	0.9600
C5—C6	1.391 (5)		
C2—C1—C6	118.4 (3)	C7—C8—C1	105.9 (3)
C2—C1—C8	134.1 (3)	C7—C8—C9	126.4 (3)
C6—C1—C8	107.5 (3)	C1—C8—C9	127.7 (3)
C7—N1—C6	108.5 (3)	C8—C9—C10	115.8 (2)
C7—N1—H1A	125.7	C8—C9—H9A	108.3
C6—N1—H1A	125.7	C10—C9—H9A	108.3
C3—C2—C1	119.3 (3)	C8—C9—H9B	108.3
C3—C2—H2A	120.3	C10—C9—H9B	108.3
C1—C2—H2A	120.3	H9A—C9—H9B	107.4
O2—N2—O1	122.6 (3)	C12—C10—C9	113.6 (2)
O2—N2—C10	118.0 (3)	C12—C10—C11	112.3 (3)
O1—N2—C10	119.3 (3)	C9—C10—C11	110.3 (3)
C2—C3—C4	121.1 (3)	C12—C10—N2	108.9 (2)
C2—C3—H3A	119.4	C9—C10—N2	106.5 (2)
C4—C3—H3A	119.4	C11—C10—N2	104.8 (2)
C5—C4—C3	121.4 (3)	C10—C11—H11A	109.5
C5—C4—H4A	119.3	C10—C11—H11B	109.5
C3—C4—H4A	119.3	H11A—C11—H11B	109.5
C4—C5—C6	116.6 (4)	C10—C11—H11C	109.5
C4—C5—H5A	121.7	H11A—C11—H11C	109.5
C6—C5—H5A	121.7	H11B—C11—H11C	109.5
N1—C6—C5	129.5 (3)	C10—C12—H12A	109.5
N1—C6—C1	107.3 (3)	C10—C12—H12B	109.5
C5—C6—C1	123.2 (3)	H12A—C12—H12B	109.5
C8—C7—N1	110.8 (3)	C10—C12—H12C	109.5
C8—C7—H7A	124.6	H12A—C12—H12C	109.5
N1—C7—H7A	124.6	H12B—C12—H12C	109.5
C6—C1—C2—C3	1.7 (4)	C2—C1—C8—C7	−179.3 (3)

C8—C1—C2—C3	−178.4 (3)	C6—C1—C8—C7	0.6 (3)
C1—C2—C3—C4	−0.1 (5)	C2—C1—C8—C9	1.2 (5)
C2—C3—C4—C5	−1.2 (5)	C6—C1—C8—C9	−178.8 (3)
C3—C4—C5—C6	0.9 (5)	C7—C8—C9—C10	−87.7 (4)
C7—N1—C6—C5	−178.0 (3)	C1—C8—C9—C10	91.6 (3)
C7—N1—C6—C1	0.8 (3)	C8—C9—C10—C12	−56.2 (3)
C4—C5—C6—N1	179.4 (3)	C8—C9—C10—C11	176.7 (3)
C4—C5—C6—C1	0.8 (4)	C8—C9—C10—N2	63.6 (3)
C2—C1—C6—N1	179.1 (3)	O2—N2—C10—C12	178.4 (3)
C8—C1—C6—N1	−0.9 (3)	O1—N2—C10—C12	−2.7 (4)
C2—C1—C6—C5	−2.0 (4)	O2—N2—C10—C9	55.6 (3)
C8—C1—C6—C5	178.0 (3)	O1—N2—C10—C9	−125.5 (3)
C6—N1—C7—C8	−0.4 (4)	O2—N2—C10—C11	−61.3 (4)
N1—C7—C8—C1	−0.1 (3)	O1—N2—C10—C11	117.6 (3)
N1—C7—C8—C9	179.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.86	2.55	3.187 (4)	132

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+2.