Triclinic
$P\bar{1}$
$a = 9.3876\,(1)$ Å
$b = 12.4039\,(2)$ Å
$c = 12.7550\,(3)$ Å
$\alpha = 104.807\,(1)^\circ$
$\beta = 99.431\,(1)^\circ$
$\gamma = 110.927\,(1)^\circ$
$V = 1286.54\,(4)$ Å^3
$Z = 2$
$D_x = 1.289$ Mg m^{-3}
D_m not measured

Cell parameters from 5053 reflections
$\theta = 1.72\text{–}27.05^\circ$
$\mu = 0.282$ mm^{-1}
$T = 293\,(2)$ K
Parallelepiped
0.32 × 0.22 × 0.10 mm
Yellow

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.915$, $T_{\max} = 0.972$
9032 measured reflections

5102 independent reflections
3148 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.054$
$\theta_{\max} = 27.05^\circ$
$h = -11 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = 0 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.176$
$S = 1.074$
5102 reflections
314 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.426P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.376$ e Å^{-3}
$\Delta\rho_{\min} = -0.273$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.008 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C10	1.213 (4)	N2—C11	1.401 (4)
O2—C17	1.213 (4)	N3—C17	1.357 (4)
N1—C7	1.333 (4)	N3—C16	1.416 (4)
N1—C8	1.341 (4)	N4—C22	1.331 (4)
N2—C10	1.350 (4)	N4—C18	1.337 (4)
C11—N2—C10—C8	177.3 (3)	C17—N3—C16—C15	51.7 (5)
N1—C8—C10—N2	−1.8 (5)	C16—N3—C17—C18	−178.6 (3)
C10—N2—C11—C12	6.0 (6)	N3—C17—C18—N4	−1.6 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12A···O1	0.93	2.276	2.880 (4)	122
C25—H25A···O1$^{\rm i}$	0.96	2.501	3.449 (5)	170

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

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 60 s covered 0.3° in ω. The crystal-to-detector distance was 3 cm and the detector swing angle was −20°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible. The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were geometrically fixed and allowed to ride on their attached atoms.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1233). Services for accessing these data are described at the back of the journal.

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1-Isobutyl-2,6,6-trimethyl-4-oxo-4,5,6,7-tetrahydro-3-indoleacetic acid

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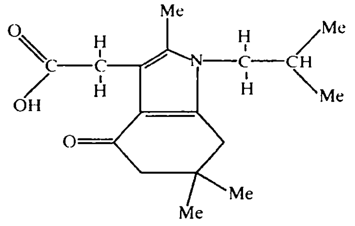
Abstract

The title compound ($C_{17}H_{25}NO_3$) is a perhydroindoleacetic acid which exhibits hypoglycemic activity. The cyclohexyl ring adopts a sofa conformation. The molecule contains an intramolecular O—H···O hydrogen bond.

Comment

Non-insulin-dependent diabetes mellitus (NIDDM) represents a singular endocrinal disorder which is assuming alarming global proportions (Davis & Granner, 1996; Colca & Tannis, 1992). We had reported earlier that a series of 4-oxo-4,5,6,7-tetrahydro-3-indoleacetic acids displayed antidiabetic properties (Nagarajan *et al.*, 1988). One of the most potent compounds in this series was GO 9001, 1-isobutyl-2,6,6-trimethyl-4-oxo-4,5,6,7-tetrahydro-3-indoleacetic acid (Nagarajan *et al.*, 1989). Its structure (I) is reported here.



(I)

The five-membered ring and part of the cyclohexyl ring (C17, C6, C5, C12 and O1) form a delocalized system with the bonds having partial double-bond character. The five-membered ring is in the envelope conformation and C6 deviates significantly (4σ) from the plane through C5, C7, C8 and N1. It is clear from the torsion angles that the cyclohexyl ring is in the sofa conformation and the best plane through the atoms in the ring makes angles of 8.44 (7) and 57.90 (2)° with the five-membered ring and carboxyl group, respectively. The carboxyl group attached to the five-membered ring is planar and is in a perpendicular conformation [118.04 (7)°] with respect to the five-membered ring. The isobutyl group of the five-membered ring is in *g* + *tg* conformation. There is an intramolecular hydrogen bond between O2—H···O1 [O2···O1 = 2.641 (2), H···O1 = 1.71 (2) Å, O2—H···O1 = 173 (2)°] stabilizing the conformation of the molecule.

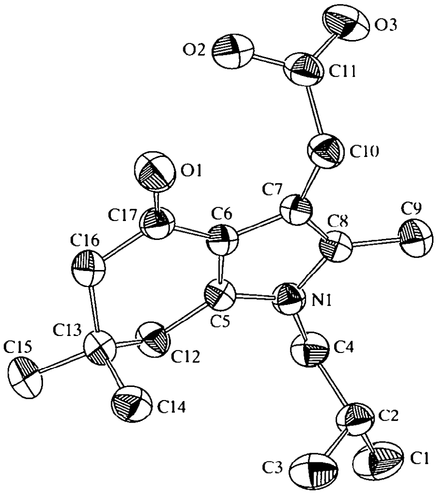


Fig. 1. *ZORTEP* (Zsolnai, 1994) drawing of the title compound with 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title compound was synthesized from dimedone in three steps: addition to maleic acid, treatment of the adduct with acetic anhydride and reaction of the resultant triketo acid with isobutylamine in acetic acid. Single crystals were obtained when the compound was dissolved in benzene and slowly evaporated. The compound melts at 427–429 K.

Crystal data

$C_{17}H_{25}NO_3$
M_r = 291.38
Monoclinic
$P2_1/c$
a = 9.233 (5) Å
b = 12.952 (3) Å
c = 13.943 (5) Å
β = 105.93 (1)°
V = 1603.3 (11) Å^3
Z = 4
D_x = 1.207 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.54180 Å
Cell parameters from 23 reflections
θ = 11–30°
μ = 0.656 mm^{-1}
T = 293 (2) K
Rectangular
0.45 × 0.2 × 0.2 mm
Pale white

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2351 measured reflections
2101 independent reflections
1812 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.019
$\theta_{\rm max}$ = 71.86°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 15$
$l = -17 \rightarrow 16$
3 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.108
S = 1.102
2101 reflections
200 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.3299P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max}$ = 0.009
$\Delta\rho_{\rm max}$ = 0.174 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.120 e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0188 (10)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å)*

C5—N1	1.361 (2)	C6—C7	1.436 (2)
C5—C6	1.382 (3)	C7—C8	1.365 (2)
C5—C12	1.489 (3)	C8—N1	1.398 (3)
C6—C17	1.435 (2)	C17—O1	1.238 (3)

The H atom attached to O2 has been refined isotropically. The other H atoms have been treated as riding.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *SDP* (Frenz, 1978). Data reduction: *CAD*-4

Software. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL93* and *PARST* (Nardelli, 1983).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1085). Services for accessing these data are described at the back of the journal.

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1,3-Bis(3-nitrophenyl)triazene

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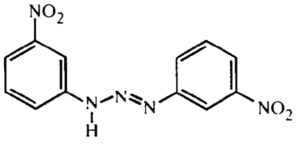
Abstract

The title molecule, $C_{12}H_9N_5O_4$, has *trans* geometry about the azo linkage. The dihedral angle between the two phenyl rings is 6.2 (2)°. The whole molecule is almost planar and the maximum deviation from the mean plane is 0.166 (3) Å. The resonance effect in the triazene group which might explain the antitumour activity of some triazene derivatives is discussed. In the crystal structure, while the twofold screw-related molecules are packed in a simple herringbone pattern, the inversion-centre-related molecules form hydrogen-bonded dimers which are held together through $\pi\cdots\pi$ interactions along the [010] direction.

Comment

It has been reported that 1,3-diphenyltriazene shows antitumour activity comparable to that of 3,3-dimethyl-1-aryltriazenes. 1,3-Diaryltriazenes can act as pro-drugs, or 'carriers' of diazonium compounds, since they undergo hydrolysis to form aryl diazonium compounds (Nifontov *et al.*, 1988, 1994). Surprisingly few X-ray structural studies of these compounds have been reported. As part of the X-ray structural studies of substituted triazene compounds, we have synthesized the title compound, (I), and confirmed its structure by X-ray analysis.



(I)

Within the limits of accuracy, the bond lengths and angles of the two nitrophenyl rings are in agreement with each other, but the whole molecule is asymmetric. N3═N4 [1.261 (2) Å] is longer than expected for a double bond, N2—N3 [1.326 (2) Å] is shorter than expected for a single bond, and C5—N2 and C11—N4 are both short for single C_{ar}—N bonds; there is clear evidence of a resonance effect in this part of the molecule. In view of the carcinogenic activity of some azobenzene derivatives (Kurosaki *et al.*, 1976) closely related to their conjugation structure (Arcos & Arcos, 1962), we think that the resonance effect in the triazene group in triazene derivatives might be related to their antitumour activity. While the nitro groups O3—N5—O4 and O1—N1—O2 are twisted by 3.3 (2) and 6.7 (2)° from their benzene planes, the dihedral angle between the two phenyl rings is 6.2 (2)°, and the torsion angles C5—N2—N3—N4 and N2—N3—N4—C11 are 179.4 (1) and 179.6 (1)°, respectively, showing the near coplanarity of the whole molecule. The shortest distance between the intermolecular mean planes is 3.27 (3) Å.

In the crystal structure, the twofold screw-related molecules are packed in a simple herringbone pattern; interactions between them include the close contact O3$\cdots$N5 (Table 2). The molecules related by inversion centres are linked together to form dimers by O4$\cdots$H—