This work was supported by a Grant-in-Aid for Scientific Research from the Ministry of Education, Science and Culture, Japan (No. 04640495).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete bond distances and angles, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71024 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1030]

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Acta Cryst. (1993). C**49**, 1401–1402

Structure of 2-Ethyl-2-nitroindan-1,3-dione

J. Gabriel Garcia,* Joel D. Enas and Edcon Chang

Lawrence Berkeley Laboratory, UC-Berkeley, Berkeley, CA 94720, USA

Frank R. Fronczek

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803-1804, USA

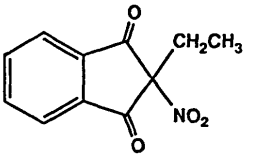
(*Received* 5 *August* 1992; *accepted* 26 *January* 1993)

Abstract

The indan-1,3-dione system is slightly non-planar, with the tetrahedral C atom lying 0.149 (2) Å out of the best plane of the other eight C and two O atoms. The terminal C atom of the ethyl group is directed *anti* to the nitro group and *gauche* to the carbonyl C atoms, forming C—C—C—C torsion angles of 53.3 (2) and −64.3 (2)°. The nitro group is nearly coplanar with the ethyl substituent, forming an O—N—C—C torsion angle of 175.9 (2)°. The C=O distances are 1.194 (2) and 1.204 (2) Å.

Comment

The crystal structures of a number of substituted indan-1,3-diones have been studied over the last 20 years on account of the well known anticoagulant activity of the parent compound in vitamin K dependent biosynthesis (Ernster, Lind & Rase, 1972; Bravic, Gaultier & Hauw, 1974; Csöregh & Eckstein, 1979). The crystal structure determination of the title compound (I), which was unexpectedly prepared by reacting 2-ethyl-1-inden-3-one-1-yl acetate with a



(I)

nitric acid–sulfuric acid 1:1 mixture (Garcia & Enas, 1992), affording colorless crystals when crystallized from slow cooling and evaporation of ethanol, is part of a program of structure analysis of some new derivatives of indan-1,3-dione. Structural data for 2-nitroindan-1,3-dione dihydrate (Selenius & Lundgren, 1980) and 2-(2-nitrobenzylidene)indan-1,3-dione (Varghese, Srinivasan, Ramadas & Padmanabhan, 1986) are in agreement with those of the title compound.

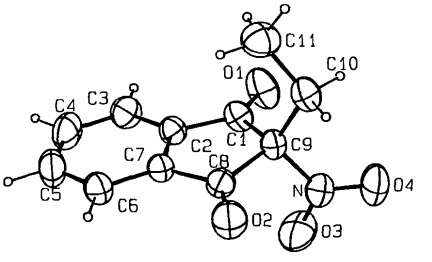


Fig. 1. *ORTEP* drawing (Johnson, 1965) of the molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radii.

Experimental

Crystal data

$C_{11}H_9NO_4$
M_r = 219.2
Monoclinic
D_x = 1.386 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54184 Å

$P2_1/c$
a = 12.9862 (5) Å
b = 6.4062 (5) Å
c = 14.0857 (12) Å
β = 116.275 (6)°
V = 1050.7 (3) Å^3
Z = 4

Cell parameters from 25 reflections
θ = 25–30°
μ = 0.86 mm^{-1}
T = 298 K
Lath fragment
0.47 × 0.32 × 0.22 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical
T_{min} = 0.94, T_{max} = 1.00
2445 measured reflections
2152 independent reflections
1685 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.014
θ_{max} = 75°
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 8$
$l = -17 \rightarrow 15$
3 standard reflections
frequency: 167 min
intensity variation: 2.1%

Refinement

Refinement on F
Final R = 0.041
wR = 0.054
S = 2.565
1685 reflections
182 parameters
All H-atom parameters refined
$w = 4F^2[\sigma^2(I) + (0.02F^2)^2]^{-1}$
$(\Delta/\sigma)_{max}$ = 0.03

$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: $(1 + gI)^{-1}$
Extinction coefficient: 8.6 (4)×10^6
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = \frac{8\pi^2}{3}\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
O1	0.6261 (1)	0.6607 (2)	0.4472 (1)	6.12 (3)
O2	0.8413 (1)	0.0411 (2)	0.56066 (9)	0.565 (3)
O3	0.8233 (1)	0.4696 (3)	0.6762 (1)	7.41 (4)
O4	0.6731 (1)	0.3341 (4)	0.6724 (1)	9.66 (6)
N	0.7328 (1)	0.3784 (3)	0.6310 (1)	5.05 (4)
C1	0.6908 (1)	0.5212 (3)	0.4563 (1)	3.87 (3)
C2	0.7741 (1)	0.5042 (3)	0.4118 (1)	3.63 (3)
C3	0.7933 (2)	0.6440 (3)	0.3462 (1)	4.88 (4)
C4	0.8762 (2)	0.5912 (4)	0.3142 (1)	5.78 (5)
C5	0.9374 (1)	0.4071 (4)	0.3460 (1)	5.52 (5)
C6	0.9192 (1)	0.2695 (3)	0.4113 (1)	4.46 (4)
C7	0.8360 (1)	0.3201 (3)	0.4442 (1)	3.56 (3)
C8	0.8009 (1)	0.2010 (3)	0.5142 (1)	3.76 (3)
C9	0.6981 (1)	0.3162 (3)	0.5176 (1)	3.62 (3)
C10	0.5881 (1)	0.1902 (3)	0.4721 (1)	4.55 (4)
C11	0.5439 (2)	0.1452 (3)	0.3559 (2)	5.79 (5)

Table 2. *Geometric parameters* (Å, °)

O1—C1	1.194 (2)	C3—C4	1.380 (3)
O2—C8	1.204 (2)	C4—C5	1.381 (3)
O3—N	1.211 (2)	C5—C6	1.369 (3)
O4—N	1.194 (3)	C6—C7	1.390 (3)
N—C9	1.509 (2)	C7—C8	1.469 (3)
C1—C2	1.475 (3)	C8—C9	1.545 (2)
C1—C9	1.551 (2)	C9—C10	1.514 (2)
C2—C3	1.387 (3)	C10—C11	1.504 (3)
C2—C7	1.386 (2)		
O3—N—O4	123.2 (2)	C2—C1—C9	107.3 (1)
C7—C8—C9	107.5 (1)	N—C9—C10	111.8 (2)
C1—C9—C8	103.2 (1)	C9—C10—C11	113.3 (2)

The crystal was sealed in a capillary to prevent sublimation. The *MolEN* (Fair, 1990) package was used for computations.

We thank DOE for support of this research through grant No. DE-AC03-76SF00098.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71037 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1038]

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Acta Cryst. (1993). C**49**, 1402–1404

Structure of 1-Phenylsemicarbazide

NOBUO OKABE

Department of Pharmaceutical Sciences, Kinki University, Kowakae 3-4-1, Higashi Osaka, Osaka 577, Japan

(*Received* 21 *April* 1992; *accepted* 5 *January* 1993)

Abstract

The semicarbazide moiety of the title compound is fairly planar (torsion angle −0.8°). The phenyl ring is nearly perpendicular to the plane of the semicarbazide group and intermolecular hydrogen bonds are formed between the N and O atoms of the semicarbazide groups.

Comment

1-Phenylsemicarbazide, also known as cryogenin, has anti-inflammatory activity (Kaplan, Wolke &