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Diethyl (2,3-Dihydro-2-oxo-3-indolylidene)propanedioate

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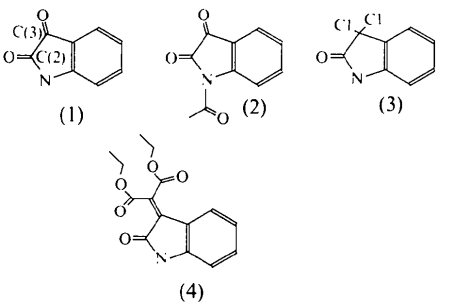
(*Received* 21 *May* 1993; *accepted* 15 *October* 1993)

Abstract

The 3*H*-indole-2(1*H*)-one moiety of $C_{15}H_{15}NO_5$ is essentially planar, the C(2)—C(3) distance being 1.510 (7) Å. The molecules are linked through hydrogen bonds forming isolated dimers.

Comment

The study of the structural features of isatin (1), (Palenik, Koziol, Katritsky & Fan, 1990) and some of its derivatives such as (2) (Zukerman-Schpector, Castellano, Pinto, Da Silva & Barcellos, 1992) and (3) (Zukerman-Schpector, Pinto, Da Silva & Barcellos, 1993) led to the observation that the C(2)—C(3) bond length is, in these cases, significantly longer than the values of 1.48 and 1.50 Å expected for C_{sp^2}—C_{sp^2} and C_{sp^2}—C_{sp^3} single bonds, respectively. In the present structure (4), the C(2)—C(3) distance of 1.510 (7) Å is within the expected range for a C_{sp^2}—C_{sp^2} bond, showing that the diethylcarboxy-methylene group bonded to C(3) does not affect the C(2)—C(3) bond length, as do the carbonyl O atoms in (1) and (2), and the Cl atoms in (3).



(1) (2) (3)

(4)

The 3*H*-indole-2-one moiety is essentially planar: $\sigma_{av} = 0.014$ Å $[\sigma_{av} = (\sum_i d_i^2/N - 3)^{1/2}]$. The main interaction determining the packing of the molecules in the crystal is a hydrogen bond: N—H(N) 1.036 (4), N···O(2^i) 2.878 (6), O(2^i)—H(N) 1.880 (4) Å, N—H(N)···O(2^i) 160.9 (3)° [symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$].

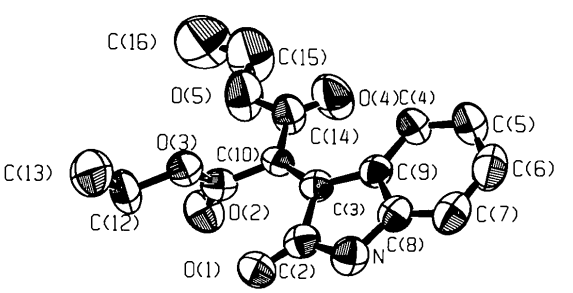


Fig. 1. The molecular structure of $C_{15}H_{15}NO_5$ showing the atom labelling. 50% displacement ellipsoids are shown for non-H atoms.

Experimental

Crystal data

$C_{15}H_{15}NO_5$
$M_r = 289.29$
Monoclinic
$P2_1/c$
$a = 8.674$ (1) Å
$b = 13.293$ (1) Å
$c = 12.670$ (3) Å
$\beta = 92.55$ (2)°
$V = 1459.3$ (6) Å^3
$Z = 4$
$D_x = 1.32$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–21°
$\mu = 0.093$ mm^{-1}
$T = 292$ K
Irregular
0.38 × 0.10 mm
Red
Crystal source: from ethanol

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
T_{min} = 0.64, T_{max} = 1.44
2147 measured reflections
2052 independent reflections
1136 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.021
θ_{max} = 25°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 15$
2 standard reflections
frequency: 30 min
intensity variation: ±1.1%

Refinement

R = 0.060
wR = 0.067
S = 2.06
1136 reflections
192 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(|F_o|) + 0005|F_o|^2]$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Atomic scattering factors from *SHELX*76 (Sheldrick, 1976)

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

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
N	0.9897 (5)	−0.1691 (3)	0.3651 (4)	5.5 (2)
O(1)	1.0093 (4)	−0.0953 (3)	0.2030 (3)	5.9 (2)
O(2)	0.9191 (5)	0.1227 (3)	0.1327 (3)	6.9 (2)
O(3)	0.7406 (4)	0.0022 (3)	0.1071 (3)	5.6 (1)
O(4)	0.6906 (6)	0.1612 (4)	0.4119 (4)	9.9 (2)
O(5)	0.6467 (6)	0.1944 (3)	0.2454 (3)	10.0 (2)
C(2)	0.9580 (6)	−0.0984 (4)	0.2909 (5)	4.7 (2)
C(3)	0.8522 (6)	−0.0224 (3)	0.3388 (4)	4.3 (2)
C(4)	0.7464 (7)	−0.0247 (4)	0.5306 (4)	5.8 (2)
C(5)	0.7482 (8)	−0.0803 (6)	0.6230 (5)	7.2 (3)
C(6)	0.8374 (8)	−0.1685 (5)	0.6319 (5)	7.0 (3)
C(7)	0.9194 (7)	−0.2026 (4)	0.5495 (5)	6.2 (2)
C(8)	0.9150 (6)	−0.1478 (4)	0.4586 (4)	4.6 (2)
C(9)	0.8317 (6)	−0.0572 (4)	0.4464 (4)	4.5 (2)
C(10)	0.7956 (6)	0.0547 (4)	0.2804 (4)	4.2 (2)
C(11)	0.8291 (7)	0.0637 (4)	0.1665 (4)	4.8 (2)
C(12)	0.7697 (8)	0.0033 (6)	−0.0048 (5)	8.0 (3)
C(13)	0.6702 (9)	−0.0786 (6)	−0.0561 (5)	9.5 (3)
C(14)	0.7046 (8)	0.1390 (4)	0.3210 (5)	6.2 (2)
C(15)	0.568 (1)	0.2870 (7)	0.2750 (8)	13.3 (5)
C(16)	0.515 (1)	0.3331 (8)	0.1908 (9)	15.1 (6)

Table 2. *Selected geometric parameters* (Å, °)

N—C(2)	1.349 (7)	N—C(8)	1.404 (7)
O(1)—C(2)	1.218 (7)	O(2)—C(11)	1.199 (7)
O(3)—C(11)	1.331 (7)	O(3)—C(12)	1.451 (7)
O(4)—C(14)	1.200 (8)	O(5)—C(14)	1.292 (8)
O(5)—C(15)	1.46 (1)	C(2)—C(3)	1.510 (7)
C(3)—C(9)	1.458 (7)	C(3)—C(10)	1.344 (7)
C(4)—C(5)	1.384 (8)	C(4)—C(9)	1.394 (8)
C(5)—C(6)	1.41 (1)	C(6)—C(7)	1.366 (9)
C(7)—C(8)	1.362 (8)	C(8)—C(9)	1.409 (7)
C(10)—C(11)	1.489 (7)	C(10)—C(14)	1.476 (8)
C(12)—C(13)	1.52 (1)	C(15)—C(16)	1.30 (1)
C(2)—N—C(8)	111.1 (4)	C(11)—O(3)—C(12)	115.2 (4)
C(14)—O(5)—C(15)	117.4 (6)	N—C(2)—O(1)	126.1 (5)
N—C(2)—C(3)	107.1 (4)	O(1)—C(2)—C(3)	126.7 (5)
C(2)—C(3)—C(9)	105.4 (4)	C(2)—C(3)—C(10)	120.2 (4)
C(9)—C(3)—C(10)	134.4 (5)	C(5)—C(4)—C(9)	119.7 (5)
C(4)—C(5)—C(6)	119.9 (6)	C(5)—C(6)—C(7)	121.2 (6)
C(6)—C(7)—C(8)	118.2 (6)	N—C(8)—C(7)	127.5 (5)
N—C(8)—C(9)	109.5 (4)	C(7)—C(8)—C(9)	123.0 (5)
C(3)—C(9)—C(4)	135.1 (5)	C(3)—C(9)—C(8)	106.9 (4)
C(4)—C(9)—C(8)	117.9 (5)	C(3)—C(10)—C(11)	120.8 (4)
C(3)—C(10)—C(14)	125.1 (5)	C(11)—C(10)—C(14)	114.0 (4)
O(2)—C(11)—O(3)	124.6 (5)	O(2)—C(11)—C(10)	123.8 (5)
O(3)—C(11)—C(10)	111.5 (4)	O(3)—C(12)—C(13)	106.9 (5)
O(4)—C(14)—O(5)	121.2 (6)	O(4)—C(14)—C(10)	126.8 (6)
O(5)—C(14)—C(10)	111.7 (5)	O(5)—C(15)—C(16)	109.8 (9)

Data were corrected for Lorentz, polarization and absorption effects. The structure was solved by direct methods. Refinement was by full-matrix least squares. H atoms were included as fixed contributors; those of the 3*H*-indole-2(1*H*)-one moiety were found in difference synthesis and the remaining H atoms were placed in calculated positions. Two overall isotropic temperature factors were refined, one for each group of H atoms. Programs used were: *SHELXS*86 (Sheldrick, 1985), *SHELX*-76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

This work has received partial support from CNPq, FAPESP, CAPES and FINEP.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71756 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1067]

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Acta Cryst. (1994). C**50**, 946–948

1,16-Hexadecanediol, $C_{16}H_{34}O_2$

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(*Received* 6 *May* 1993; *accepted* 25 *October* 1993)

Abstract

The crystal structure of the title compound was determined by X-ray diffraction. The skeleton of the molecule is all *trans* and the molecules form a layer