O(3)—C(3)—C(4)—O(4)	−88.0 (2)	−79.8 (5)
C(5)—C(4)—O(4)—C(1)	−144.5 (2)	−143.0 (5)
O(4)—C(4)—C(5)—O(5)	−63.1 (3)	47.3 (7)
O(4)—C(4)—C(5)—C(6)	175.1 (2)	−75.5 (6)
C(3)—C(4)—C(5)—O(5)	−179.7 (2)	−70.3 (7)
C(3)—C(4)—C(5)—C(6)	58.5 (3)	166.9 (5)
O(5)—C(5)—C(6)—O(6)	78.2 (2)	−178.3 (5)
O(5)—C(5)—C(6)—C(7)	−41.2 (3)	54.1 (7)
C(4)—C(5)—C(6)—O(6)	−159.8 (2)	−52.4 (7)
C(4)—C(5)—C(6)—C(7)	80.8 (3)	179.9 (4)
O(6)—C(6)—C(7)—O(7)	54.1 (3)	−69.0 (7)
C(5)—C(6)—C(7)—O(7)	171.7 (2)	58.4 (6)

Table 3. *Hydrogen-bonding geometry* (Å, °) *for compounds* (I) *and* (II)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
(I)			
O(2)—H(O2)···O(6^{i})	2.635 (3)	1.83 (4)	174 (4)
O(3)—H(O3)···O(2^{ii})	2.755 (3)	1.99 (3)	161 (3)
O(5)—H(O5)···O(7^{iii})	2.683 (3)	1.91 (4)	158 (4)
O(6)—H(O6)···O(3^{iv})	2.710 (3)	1.92 (3)	175 (3)
O(7)—H(O7)···O(1^{v})	2.913 (3)	2.20 (3)	168 (4)
(II)			
O(3)—H(O3)···O(1^{iii})	2.750 (6)	2.09 (6)	158 (6)
O(5)—H(O5)···O(3)	2.718 (6)	2.17 (7)	124 (6)

Symmetry codes: (i) $x, 1+y, z$; (ii) $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$; (iii) $x-1, y, z$; (iv) $\frac{1}{2}+x, \frac{1}{2}-y, z$; (v) $x+1, y-1, z$.

Except for the two CH_3 groups in (II), the H atoms were located in difference Fourier maps and refined isotropically. Note that C(21) of one of the CH_3 groups has a large temperature parameter. There is a significant difference between the *R* values for the two possible enantiomorphic structures of (II). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular geometry calculations: *PLATON* (Spek, 1990). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71663 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1093]

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2,6-Dimesyl-D-*manno*-hexono- (III), 2,6-Dimesyl-D-*allo*-hexono- (IV) and 2,6-Dimesyl-D-*gulo*-hexono-1,4-lactone (V)

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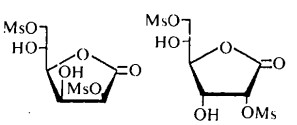
Abstract

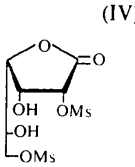
The geometries of the lactone rings in the three structures are similar. Differences between $C_8H_{14}O_{10}S_2$ (III), $C_8H_{14}O_{10}S_2$ (IV) and $C_8H_{14}O_{10}S_2$ (V) arise in the conformation of the side chain and in the crystal packing of the structures.

Comment

The present structure analyses are part of an investigation of tosylated and mesylated 1,4-lactones (Søtofte, 1994). The di-*O*-mesylation of D-*manno*-, D-*allo*- and D-*gulo*-1,4-lactones gave the 2,6-di-*O*-mesylates (III), (IV) and (V) in 42, 16 and 27% yields, respectively. The yields of the di-*O*-mesylates are lower than those of the di-*O*-tosylates, indicating lower selectivity. All three compounds were prepared by literature methods (Lundt & Madsen, 1992).



(III) (IV)



(V)

Compound (III) was recrystallized at room temperature from acetonitrile, while compounds (IV) and (V) were recrystallized from ethyl acetate. Unfortunately, the structure determined for (III) is rather poor, as no larger and more suitable crystals could be obtained by recrystallization. The bond lengths and angles that are listed in Table 2 agree well with those observed in related structures. The labelling of the atoms is shown in Fig. 1. The lactone rings are nonplanar.

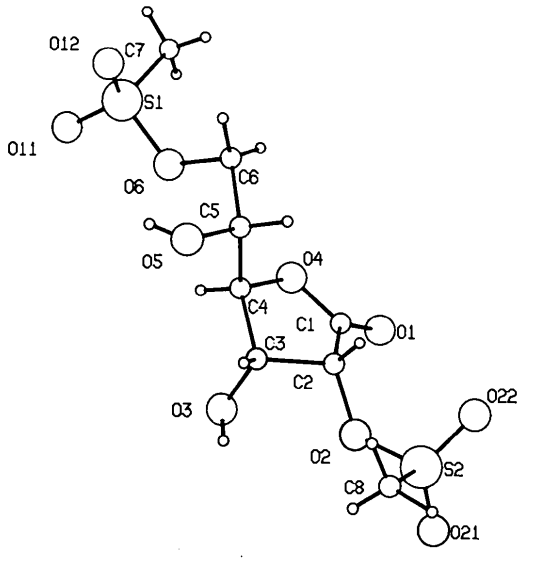


Fig. 1. View of molecule (IV) with atomic labelling.

The pseudorotation parameters, P, τ_m (Rao, Westof & Sundaralingam, 1981), for the three compounds are P(III) = 192 (1), P(IV) = 180.4 (3), P(V) = 187.7 (4)° and τ_m(III) = 44 (1), τ_m(IV) = 32.7 (2), τ_m(V) = 40.3 (2)°. Thus the conformation of (IV) is ${}^2_3T$, while (III) are (V) exhibit a conformation between ${}^2_3T$ and ${}_3E$. The corresponding puckering parameters, φ_2, q_2 (Cremer & pople, 1975), are φ_2(III) = 103 (2), φ_2(IV) = 91.6 (5), φ_2(V) = 98.4 (5)° and q_2(III) = 0.43 (2), q_2(IV) = 0.321 (3), q_2(V) = 0.399 (4). These values are in agreement with those found in other 1,4-lactones (Søtofte, 1994).

Selected torsion angles are listed in Table 2. Differences between the three structures appear in the conformation of the side chain [C(5)–O(6)] and in the crystal packing, which to some extent is influenced by hydrogen bonding. These are described in Table 3 and shown in Figs. 2, 3 and 4. In (IV), hydrogen bonding occurs between molecules related by a twofold screw axis and in (V), between molecules related by translational symmetry along the a axis. Unfortunately, in (III), the compound with the largest yield, the H atoms could not be located. A comparison between short intra- and intermolecular contacts in (III) with those in (IV) and (V) may indicate where hydrogen bonds might be. These possible contacts are O(3)···O(21)($2-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$) and O(5)···O(11) ($\frac{3}{2}-x$, $1-y$, $\frac{1}{2}+z$), and are 2.89 (2) and 2.88 (2) Å, respectively. No indication that intramolecular hydrogen bonds could be present was found.

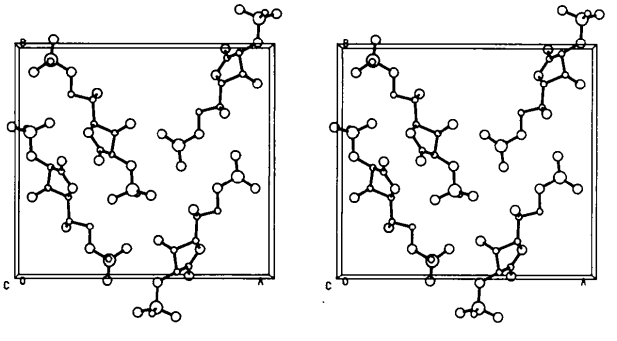


Fig. 2. Stereoscopic view of 2,6-dimesyl-D-*manno*-hexono-1,4-lactone (III); hydrogen bonds are drawn as thin lines.

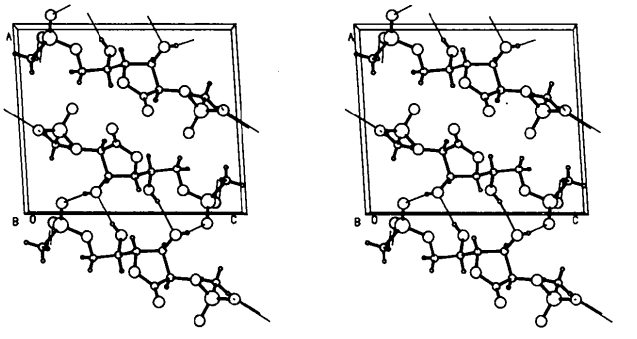


Fig. 3. Stereoscopic view of 2,6-dimesyl-D-*allo*-hexono-1,4-lactone (IV); hydrogen bonds are drawn as thin lines.

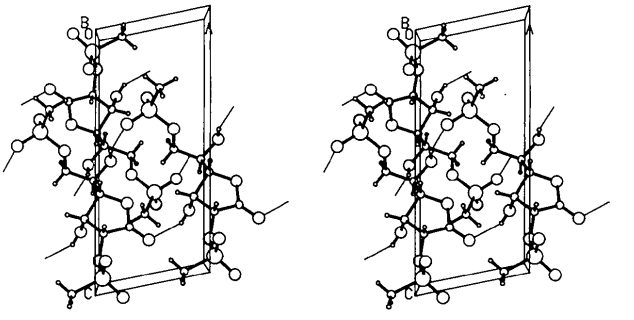


Fig. 4. Stereoscopic view of 2,6-dimesyl-D-*gulo*-hexono-1,4-lactone (V); hydrogen bonds are drawn as thin lines.

Experimental

Compound (III)

Crystal data

$C_8H_{14}O_{10}S_2$
M_r = 334.31
Orthorhombic
$P2_12_12_1$
a = 16.548 (7) Å
b = 14.854 (8) Å
c = 5.377 (9) Å
V = 1322 (2) Å^3
Z = 4
D_x = 1.680 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 23 reflections
θ = 9-34°
μ = 4.08 mm^{-1}
T = 120 K
Needle
0.23 × 0.04 × 0.02 mm
Colourless

Data collection

Enraf–Nonius CAD-4F diffractometer
ω–2θ scans
Absorption correction: none
2350 measured reflections
1178 independent reflections
751 observed reflections [$I \geq 2.0\sigma(I)$]

$R_{int} = 0.072$
$\theta_{max} = 60°$
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 20$
$l = -10 \rightarrow 10$
2 standard reflections
frequency: 168 min
intensity variation: none

Refinement

Refinement on F
$R = 0.081$
$wR = 0.106$
$S = 1.23$
741 reflections
181 parameters

$w = 1/[\sigma^2(F) + 0.015|F|^2]$
$(\Delta/\sigma)_{max} = 0.033$
$\Delta\rho_{max} = 0.62$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (IV)

Crystal data

$C_8H_{14}O_{10}S_2$
$M_r = 334.31$
Monoclinic
$P2_1$
$a = 10.277$ (2) Å
$b = 5.260$ (2) Å
$c = 12.070$ (3) Å
$\beta = 93.36$ (2)°
$V = 651.3$ (3) Å^3
$Z = 2$
$D_x = 1.705$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–$16°$
$\mu = 0.44$ mm^{-1}
$T = 120$ K
Needle
0.32 × 0.06 × 0.05 mm
Colourless

Data collection

Enraf–Nonius CAD-4F diffractometer
ω scans
Absorption correction: none
4097 measured reflections
3796 independent reflections
2829 observed reflections [$I \geq 3.0\sigma(I)$]

$R_{int} = 0.022$
$\theta_{max} = 30°$
$h = 0 \rightarrow 14$
$k = -7 \rightarrow 7$
$l = -17 \rightarrow 17$
2 standard reflections
frequency: 240 min
intensity variation: none

Refinement

Refinement on F
$R = 0.040$
$wR = 0.041$
$S = 1.21$
2815 reflections
236 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F) + 0.0004|F|^2]$
$(\Delta/\sigma)_{max} = 0.027$
$\Delta\rho_{max} = 0.73$ e Å^{-3}
$\Delta\rho_{min} = -0.72$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (V)

Crystal data

$C_8H_{14}O_{10}S_2$
$M_r = 334.31$
Monoclinic
$P2_1$
$a = 5.711$ (3) Å
$b = 8.941$ (3) Å
$c = 12.990$ (5) Å
$\beta = 101.71$ (3)°
$V = 649.5$ (5) Å^3
$Z = 2$
$D_x = 1.709$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–$11°$
$\mu = 0.44$ mm^{-1}
$T = 120$ K
Plate
0.35 × 0.12 × 0.01 mm
Colourless

Data collection

Enraf–Nonius CAD-4F diffractometer
ω scans
Absorption correction: none
4694 measured reflections
3767 independent reflections
2536 observed reflections [$I \geq 3.0\sigma(I)$]

$R_{int} = 0.019$
$\theta_{max} = 30°$
$h = 0 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 18$
2 standard reflections
frequency: 240 min
intensity variation: none

Refinement

Refinement on F
$R = 0.043$
$wR = 0.043$
$S = 1.19$
2511 reflections
236 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F) + 0.0004|F|^2]$
$(\Delta/\sigma)_{max} = 0.31$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for compounds* (III), (IV) *and* (V)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
(III)				
S(1)	0.6361 (3)	0.5677 (3)	0.1607 (9)	0.049 (2)
S(2)	0.9534 (3)	1.1252 (3)	0.3245 (10)	0.053 (2)
O(1)	0.8244 (8)	0.9980 (8)	−0.081 (2)	0.055 (4)
O(2)	0.9484 (7)	1.0200 (8)	0.296 (3)	0.059 (5)
O(3)	0.9455 (7)	0.8402 (8)	0.303 (3)	0.073 (6)
O(4)	0.7834 (7)	0.8798 (7)	0.144 (2)	0.052 (4)
O(5)	0.8130 (8)	0.7057 (8)	0.604 (3)	0.065 (5)
O(6)	0.7152 (6)	0.6173 (7)	0.261 (3)	0.053 (4)
O(11)	0.6450 (7)	0.4770 (7)	0.232 (3)	0.059 (4)
O(12)	0.5688 (7)	0.6178 (9)	0.255 (3)	0.073 (5)
O(21)	1.0242 (7)	1.1483 (8)	0.198 (3)	0.069 (5)
O(22)	0.8762 (7)	1.1651 (8)	0.263 (2)	0.062 (5)
C(1)	0.8276 (10)	0.9539 (11)	0.110 (3)	0.044 (6)
C(2)	0.8732 (10)	0.9723 (11)	0.342 (3)	0.045 (6)
C(3)	0.8856 (10)	0.8797 (12)	0.446 (4)	0.051 (7)
C(4)	0.8024 (11)	0.8407 (11)	0.396 (4)	0.052 (7)
C(5)	0.7972 (11)	0.7356 (12)	0.359 (4)	0.058 (7)
C(6)	0.7144 (10)	0.7138 (11)	0.272 (4)	0.059 (7)
C(7)	0.6385 (12)	0.5790 (16)	−0.163 (4)	0.078 (8)
C(8)	0.9705 (12)	1.1422 (13)	0.644 (4)	0.066 (8)

(IV)				
S(1)	0.06164 (8)	0.08510	0.84382 (6)	0.0161 (2)
S(2)	0.44793 (8)	−0.0301 (2)	0.16958 (6)	0.0135 (2)
O(1)	0.4541 (2)	0.4304 (5)	0.4171 (2)	0.0190 (7)
O(2)	0.3472 (2)	0.1144 (5)	0.2429 (2)	0.0142 (6)
O(3)	0.1113 (2)	0.1587 (5)	0.3232 (2)	0.0212 (7)
O(4)	0.3036 (2)	0.2599 (4)	0.5207 (2)	0.0129 (6)
O(5)	0.1208 (2)	−0.3263 (5)	0.5704 (2)	0.0192 (7)
O(6)	0.1212 (2)	0.0943 (5)	0.7272 (2)	0.0183 (6)
O(11)	−0.0561 (2)	0.2312 (5)	0.8304 (2)	0.0237 (8)
O(12)	0.0526 (3)	−0.1730 (5)	0.8798 (2)	0.0261 (8)
O(21)	0.4475 (2)	0.1203 (5)	0.0709 (2)	0.0207 (7)
O(22)	0.5677 (2)	−0.0656 (6)	0.2334 (2)	0.0277 (8)
C(1)	0.3757 (3)	0.2679 (7)	0.4323 (2)	0.0145 (8)
C(2)	0.3380 (3)	0.0424 (6)	0.3576 (2)	0.0125 (8)
C(3)	0.1972 (3)	−0.0084 (7)	0.3849 (2)	0.0138 (8)
C(4)	0.1991 (3)	0.0703 (7)	0.5065 (2)	0.0130 (7)
C(5)	0.2232 (3)	−0.1474 (7)	0.5880 (3)	0.0148 (9)
C(6)	0.2379 (3)	−0.0567 (7)	0.7065 (3)	0.0172 (9)
C(7)	0.1747 (4)	0.2485 (9)	0.9316 (3)	0.0237 (10)
C(8)	0.3723 (5)	−0.3206 (8)	0.1420 (4)	0.0284 (13)
(V)				
S(1)	0.4716 (2)	0.43860	0.33247 (7)	0.0162 (3)
S(2)	1.0573 (2)	0.7560 (2)	0.94959 (7)	0.0209 (3)
O(1)	1.4718 (5)	0.5084 (3)	0.8424 (2)	0.0200 (8)
O(2)	1.0222 (5)	0.6031 (3)	0.8833 (2)	0.0205 (8)
O(3)	0.8407 (5)	0.3662 (3)	0.7596 (2)	0.0221 (9)
O(4)	1.2707 (4)	0.4480 (3)	0.6819 (2)	0.0157 (7)
O(5)	1.1160 (5)	0.2747 (3)	0.5023 (2)	0.0203 (8)
O(6)	0.6942 (5)	0.4367 (3)	0.4262 (2)	0.0222 (7)
O(11)	0.4535 (6)	0.2967 (3)	0.2808 (2)	0.0260 (9)
O(12)	0.2674 (5)	0.4888 (3)	0.3705 (2)	0.0290 (9)
O(21)	1.2388 (6)	0.7281 (4)	1.0394 (2)	0.0368 (13)
O(22)	1.0875 (6)	0.8753 (4)	0.8818 (2)	0.0336 (10)
C(1)	1.2925 (7)	0.5165 (4)	0.7754 (3)	0.0164 (11)
C(2)	1.0613 (7)	0.5976 (4)	0.7791 (3)	0.0170 (10)
C(3)	0.8805 (7)	0.4992 (4)	0.7086 (3)	0.0149 (9)
C(4)	1.0223 (6)	0.4602 (4)	0.6234 (3)	0.0138 (9)
C(5)	0.9537 (7)	0.3135 (4)	0.5674 (3)	0.0146 (10)
C(6)	0.6989 (7)	0.3183 (5)	0.5056 (3)	0.0164 (10)
C(7)	0.5638 (8)	0.5769 (5)	0.2555 (3)	0.0219 (11)
C(8)	0.7813 (8)	0.7668 (7)	0.9861 (4)	0.0274 (14)

Table 2. *Selected geometric parameters* (Å, °) *for compounds* (III), (IV) *and* (V)

	(III)	(IV)	(V)
S(1)—O(6)	1.60 (1)	1.569 (2)	1.571 (3)
S(1)—O(11)	1.41 (1)	1.435 (2)	1.429 (3)
S(1)—O(12)	1.43 (1)	1.430 (3)	1.428 (3)
S(1)—C(7)	1.75 (2)	1.751 (4)	1.738 (4)
S(2)—O(2)	1.57 (1)	1.594 (2)	1.607 (3)
S(2)—O(21)	1.40 (1)	1.430 (2)	1.417 (3)
S(2)—O(22)	1.45 (1)	1.426 (2)	1.416 (4)
S(2)—C(8)	1.76 (2)	1.738 (5)	1.739 (5)
O(1)—C(1)	1.22 (2)	1.196 (4)	1.204 (5)
O(2)—C(2)	1.45 (2)	1.444 (3)	1.416 (5)
O(3)—C(3)	1.38 (2)	1.425 (4)	1.402 (5)
O(4)—C(1)	1.33 (2)	1.334 (3)	1.343 (5)
O(4)—C(4)	1.51 (2)	1.468 (4)	1.471 (4)
O(5)—C(5)	1.41 (3)	1.418 (4)	1.419 (5)
O(6)—C(6)	1.44 (2)	1.472 (4)	1.475 (5)
C(1)—C(2)	1.48 (2)	1.526 (4)	1.516 (6)
C(2)—C(3)	1.50 (2)	1.526 (4)	1.515 (5)
C(3)—C(4)	1.52 (2)	1.524 (4)	1.538 (5)
C(4)—C(5)	1.58 (2)	1.520 (5)	1.512 (5)
C(5)—C(6)	1.48 (3)	1.507 (5)	1.513 (6)
O(6)—S(1)—O(11)	105.3 (7)	104.7 (1)	108.9 (2)
O(6)—S(1)—O(12)	106.2 (7)	109.7 (2)	108.9 (2)
O(6)—S(1)—C(7)	105.9 (9)	104.4 (2)	99.3 (2)
O(11)—S(1)—O(12)	118.8 (8)	118.2 (2)	117.1 (2)
O(11)—S(1)—C(7)	111.1 (10)	109.3 (2)	111.3 (2)
O(12)—S(1)—C(7)	108.7 (10)	109.6 (2)	109.8 (2)
O(2)—S(2)—O(21)	103.9 (7)	103.2 (1)	106.4 (2)
O(2)—S(2)—O(22)	109.8 (7)	109.3 (1)	109.0 (2)
O(2)—S(2)—C(8)	104.3 (9)	103.2 (2)	99.9 (2)
O(21)—S(2)—O(22)	121.9 (8)	118.9 (2)	118.9 (2)
O(21)—S(2)—C(8)	107.8 (9)	110.3 (2)	109.7 (2)
O(22)—S(2)—C(8)	107.8 (8)	110.5 (2)	111.1 (3)
S(2)—O(2)—C(2)	121 (1)	119.2 (2)	121.0 (2)
C(1)—O(4)—C(4)	109 (1)	111.4 (2)	109.6 (3)
S(1)—O(6)—C(6)	118 (1)	120.0 (2)	116.5 (2)
O(1)—C(1)—O(4)	122 (2)	123.7 (3)	122.4 (4)
O(1)—C(1)—C(2)	129 (2)	127.9 (3)	128.6 (4)
O(4)—C(1)—C(2)	108 (1)	108.3 (3)	109.0 (3)
O(2)—C(2)—C(1)	112 (1)	109.5 (2)	110.7 (3)
O(2)—C(2)—C(3)	113 (1)	111.7 (2)	112.2 (3)
C(1)—C(2)—C(3)	102 (1)	102.7 (2)	101.2 (3)
O(3)—C(3)—C(2)	106 (2)	110.2 (2)	111.3 (3)
O(3)—C(3)—C(4)	113 (2)	108.0 (3)	107.9 (3)
C(2)—C(3)—C(4)	99 (1)	101.6 (2)	100.0 (3)
O(4)—C(4)—C(3)	102 (1)	105.3 (2)	103.8 (3)
O(4)—C(4)—C(5)	105 (2)	110.2 (2)	108.2 (3)
C(3)—C(4)—C(5)	117 (2)	114.3 (3)	114.8 (3)
O(5)—C(5)—C(4)	101 (2)	108.1 (3)	111.2 (3)
O(5)—C(5)—C(6)	113 (2)	112.6 (3)	111.4 (3)
C(4)—C(5)—C(6)	108 (1)	112.3 (3)	111.0 (3)
O(6)—C(6)—C(5)	103 (1)	107.1 (3)	105.7 (3)
O(1)—C(1)—O(4)—C(4)	179 (2)	168.8 (3)	171.9 (3)
O(1)—C(1)—C(2)—O(2)	−32 (2)	−33.2 (4)	−29.8 (5)
O(4)—C(1)—C(2)—O(2)	152 (1)	145.8 (2)	149.4 (3)
C(1)—C(2)—O(2)—S(2)	97 (2)	117.9 (2)	106.4 (3)
O(2)—C(2)—C(3)—O(3)	−46 (2)	−34.7 (3)	−43.0 (4)
O(3)—C(3)—C(4)—O(4)	−73 (2)	−89.2 (3)	−80.8 (3)
C(5)—C(4)—O(4)—C(1)	−145 (1)	112.7 (3)	−140.7 (3)
O(4)—C(4)—C(5)—O(5)	−178 (1)	−179.4 (2)	−55.5 (4)
O(4)—C(4)—C(5)—C(6)	−59 (2)	55.7 (3)	179.9 (4)
C(3)—C(4)—C(5)—O(5)	70 (2)	−61.0 (3)	−170.9 (3)
C(3)—C(4)—C(5)—C(6)	−171 (2)	174.1 (3)	64.5 (4)
O(5)—C(5)—C(6)—O(6)	−66 (2)	−67.7 (4)	−62.1 (4)
C(4)—C(5)—C(6)—O(6)	−176 (2)	54.6 (3)	62.4 (4)
C(5)—C(6)—O(6)—S(1)	−177 (1)	144.3 (2)	164.5 (2)

Table 3. *Hydrogen-bonding geometry* (Å, °) *for compounds* (IV) *and* (V)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
(IV)			
O(3)—H(O3)···O(11^{i})	2.949 (4)	2.19 (4)	159 (4)
O(5)—H(O5)···O(3^{i})	2.776 (3)	2.02 (3)	152 (5)
(V)			
O(3)—H(O3)···O(1^{ii})	2.852 (4)	2.16 (4)	147 (4)
O(5)—H(O5)···O(12^{iii})	2.819 (4)	2.08 (4)	157 (5)

Symmetry codes: (i) $-x, y-\frac{1}{2}, 1-z$; (ii) $x-1, y, z$; (iii) $1+x, y, z$.

For (IV) and (V) all H atoms were located from difference Fourier maps and refined isotropically. The H atoms in (III) were not located. There is a significant difference between the *R* values for the two enantiomorphic structures of (IV) and (V). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular geometry calculations: *PLATON* (Spek, 1990). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978).

The author thanks Flemming Hansen and Sine Larsen for collecting the X-ray data of (III).

Lists of structure factors, anisotropic displacement parameters, complete geometry and H-atom coordinates for (IV) and (V) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71666 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1094]

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

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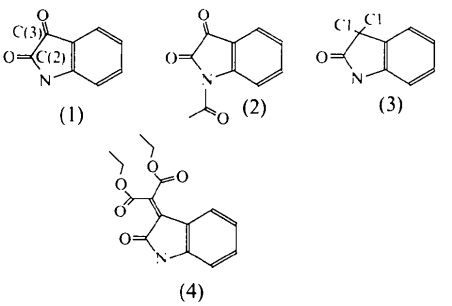
Abstract

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) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$].

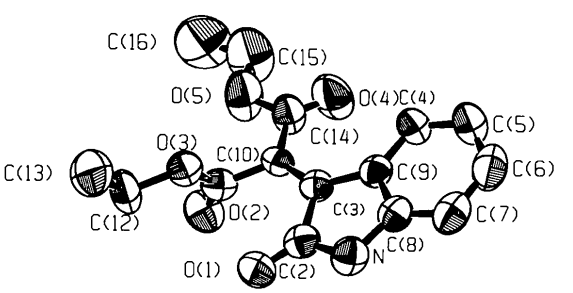


Fig. 1. The molecular structure of $C_{15}H_{15}NO_5$ showing the atom labelling. 50% displacement ellipsoids are shown for non-H atoms.

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