

Institute of Structural Chemistry, University of Parma, for a fellowship.

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Structure of (2*S*,4*R*)-*cis*-4-Methyl-2-phthalimido- γ -butyrolactone

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Abstract. C₁₃H₁₁NO₄, $M_r = 245.23$, orthorhombic, $P2_12_12_1$, $a = 23.251$ (8), $b = 8.453$ (5), $c = 5.813$ (4) Å, $V = 1143$ (1) Å³, $Z = 4$, $D_x = 1.426$ g cm⁻³, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 8.541$ cm⁻¹, $F(000) = 512$, room temperature, $R = 0.050$ for 2012 unique observed reflections [$I > 3\sigma(I)$]. The lactone ring has an envelope conformation with C3 deviating by 0.505 (2) Å from the least-squares plane formed by the remaining four ring atoms. The absolute configuration of two chiral C atoms was deduced as (2*S*, 4*R*). The phthalimido and methyl groups are in pseudo-equatorial positions, *cis* to each other.

Introduction. The structure of (2*S*,4*R*)-*cis*-4-methyl-2-phthalimido- γ -butyrolactone has been established as part of our investigation of the stereochemistry of different 2,4-disubstituted γ -butyrolactones (Matijašić, Bocelli, Ugozzoli & Sgarabotto, 1988).

Experimental. Colorless prismatic crystal recrystallized from benzene/ether solution, 0.24 × 0.45 ×

0.18 mm approximately. Data measured on a Siemens AED diffractometer, Ni-filtered Cu $K\alpha$ radiation, cell parameters from least-squares fit of 30 reflections accurately measured on the diffractometer in the range $38 < \theta < 43^\circ$, one check reflection measured every 50 counts without significant variation, profile analysis with Lehmann & Larsen (1974) procedure modified by Belletti, Ugozzoli, Cantoni & Pasquinelli (1979), $3 < \theta < 70^\circ$, Lp correction, absorption correction with Walker & Stuart (1983) procedure using *ABSORB* (Ugozzoli, 1986), maximum and minimum values of the absorption coefficients in the polar angles φ and μ are 1.19 and 0.84 respectively, $-28 < h < 28$, $0 < k < 10$, $0 < l < 7$, 2571 reflections collected, 2332 observed at $3\sigma(I)$ level, 2012 unique reflections ($R_{\text{int}} = 0.023$), direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), full-matrix least-squares refinement with anisotropic thermal parameters for non-H atoms, H atoms located from difference Fourier map and refined

Table 1. Atomic fractional coordinates ($\times 10^4$) and U_{eq} ($\times 10^4 \text{Å}^2$)
$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
C1	-5429 (1)	309 (2)	-4450 (4)	382 (6)
C2	-5551 (1)	-1461 (3)	-4612 (4)	367 (6)
C3	-5005 (1)	-2099 (3)	-5701 (4)	408 (6)
C4	-4550 (1)	-968 (3)	-4808 (5)	400 (6)
O1	-4861 (1)	544 (2)	-4518 (3)	431 (5)
O2	-5769 (1)	1365 (2)	-4270 (4)	543 (6)
C5	-4046 (1)	-703 (4)	-6376 (6)	576 (10)
C6	-6251 (1)	-1081 (3)	-7853 (4)	368 (6)
C7	-6851 (1)	-1607 (2)	-8270 (4)	372 (6)
C8	-7226 (1)	-1227 (3)	-10033 (5)	462 (8)
C9	-7772 (1)	-1896 (3)	-9946 (6)	537 (8)
C10	-7931 (1)	-2900 (3)	-8181 (6)	564 (9)
C11	-7552 (1)	-3285 (3)	-6430 (6)	498 (8)
C12	-7011 (1)	-2611 (3)	-6516 (4)	384 (6)
C13	-6520 (1)	-2740 (2)	-4877 (4)	372 (6)
N1	-6088 (1)	-1770 (2)	-5773 (3)	366 (5)
O3	-5943 (1)	-243 (2)	-9018 (3)	524 (6)
O4	-6481 (1)	-3495 (2)	-3131 (4)	534 (6)

isotropically, 207 parameters refined, final $R = 0.0505$, $wR = 0.0545$. For completeness, the opposite enantiomorph was also refined and convergence was attained at $R = 0.0503$ and $wR = 0.0543$ with $w = 1.7341/[\sigma^2(F) + 0.001775F^2]$, $\sum w(F)^2$ minimized. The ratio of wR factors (1.004) is greater than the value of 1.002 required for the Hamilton (1965) R -factor ratio at a probability level of 99.5% and the enantiomorph with the lower R factor corresponds to the (2*S*, 4*R*) configuration. $(\Delta/\sigma)_{max}$ in the final cycle < 0.08 , the final difference map was essentially featureless showing the highest peak at 0.39 e Å^{-3} , scattering factors of *SHELX76* (Sheldrick, 1976), anomalous-dispersion factors taken from *International Tables for X-ray Crystallography* (1974), all calculations performed on a Gould SEL 32/77 computer.

Discussion. The refined positional and equivalent isotropic thermal parameters for all non-H atoms are given in Table 1.* The absolute configuration with atomic numbering scheme used is shown in Fig. 1. Bond distances, bond angles and selected torsion angles are listed in Table 2.

The structure of the title compound consists of phthalimido and methyl groups bonded to the α - and γ -C atoms of a butyrolactone moiety. Considering only the overall conformation, the molecule may be described in terms of two ring systems, whose mean plane subtends a dihedral angle of $73.98 (8)^\circ$.

Butyrolactone rings usually assume an envelope conformation (Harlow & Simonsen, 1976; Bocelli &

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51924 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (Å), bond angles ($^\circ$) and selected torsion angles ($^\circ$)

C1—C2	1.526 (3)	C6—O3	1.214 (3)
C1—O1	1.336 (3)	C7—C8	1.383 (4)
C1—O2	1.197 (3)	C7—C12	1.378 (3)
C2—C3	1.518 (3)	C8—C9	1.391 (3)
C2—N1	1.443 (3)	C9—C10	1.382 (4)
C3—C4	1.517 (4)	C10—C11	1.385 (4)
C4—O1	1.478 (3)	C11—C12	1.382 (3)
C4—C5	1.501 (4)	C12—C13	1.491 (3)
C6—C7	1.484 (3)	C13—N1	1.397 (3)
C6—N1	1.395 (3)	C13—O4	1.202 (3)
O1—C1—O2	123.0 (2)	C6—C7—C8	130.1 (2)
C2—C1—O2	127.9 (2)	C8—C7—C12	121.4 (2)
C2—C1—O1	109.1 (1)	C7—C8—C9	117.0 (2)
C1—C2—N1	111.5 (2)	C8—C9—C10	121.3 (2)
C1—C2—C3	102.6 (2)	C9—C10—C11	121.3 (2)
C3—C2—N1	117.7 (2)	C10—C11—C12	117.1 (2)
C2—C3—C4	102.5 (2)	C7—C12—C11	121.8 (2)
C3—C4—C5	115.5 (2)	C11—C12—C13	130.1 (2)
C3—C4—O1	104.1 (2)	C7—C12—C13	108.1 (2)
O1—C4—C5	108.8 (2)	C12—C13—O4	129.5 (2)
C1—O1—C4	111.0 (2)	C12—C13—N1	105.6 (2)
N1—C6—O3	124.6 (2)	N1—C13—O4	124.9 (2)
C7—C6—O3	129.7 (2)	C6—N1—C13	111.9 (2)
C7—C6—O1	105.8 (2)	C2—N1—C13	123.6 (2)
C6—C7—C12	108.5 (2)	C2—N1—C6	124.4 (2)
O2—C1—O1—C4	-178.5 (2)	C1—C2—C3—C4	31.1 (2)
C2—C1—O1—C4	2.0 (3)	C3—C2—N1—C6	-70.1 (3)
O1—C1—C2—N1	-148.3 (2)	C3—C2—N1—C13	113.2 (3)
O1—C1—C2—C3	-21.4 (3)	N1—C2—C3—C4	153.9 (2)
O2—C1—C2—N1	32.2 (4)	C2—C3—C4—O1	-30.4 (2)
O2—C1—C2—C3	159.1 (3)	C2—C3—C4—C5	-149.6 (2)
C1—C2—N1—C6	48.1 (3)	C3—C4—O1—C1	18.3 (3)
C1—C2—N1—C13	-128.6 (2)	C5—C4—O1—C1	142.0 (2)

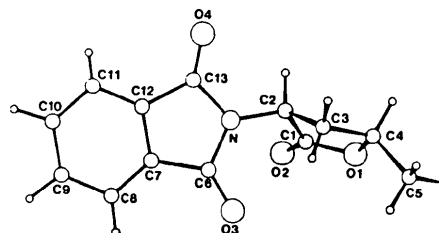


Fig. 1. A perspective view of the molecule with the atomic numbering scheme.

Grenier-Loustalot, 1981, 1982; Secco & Trotter, 1983), in which the only atom deviating significantly from the plane of the remaining four atoms is the β -carbon. The planarity of the remaining atoms in the ring stems from the carbonyl group and the partial sp^2 character of the hetero O atom. Our results are consistent with an envelope conformation with the β -C atom, C3, out-of-plane by $0.505 (2) \text{ Å}$. In Cremer & Pople (1975) notation the puckering parameters calculated for the lactone ring are: the amplitude of puckering, Q , $0.322 (2) \text{ Å}$ and the phase angle, φ , -75.7° .

Bond lengths and angles (Table 2) in the lactone ring are generally close to normal values reported for the other γ -butyrolactones except for those involving the O atom of the ring. The values of the O—C(sp^2) and O—C(sp^3) bond distances are $1.336 (3)$ and

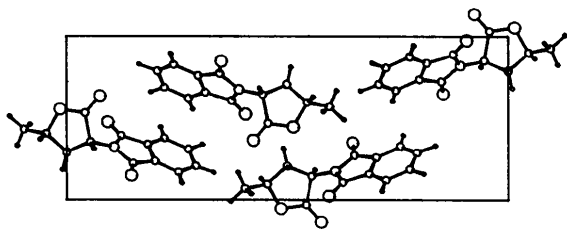


Fig. 2. A projection of the structure viewed along *c* (*a* is horizontal).

1.478 (3) Å respectively. The angles at C1 exhibit the usual deviations from 120°.

Both substituents on the lactone ring are in pseudo-equatorial positions *cis* to each other. There are two optically active C atoms in the molecule and the absolute configurations at C2 and C4 were determined as *S* and *R*, respectively.

The six- and five-membered rings of the phthalimido group are planar within 0.018 (3) and 0.003 (3) Å. The dihedral angle between these two rings is 1.82 (8)°. The parameters of the phthalimido group are consistent with those in other phthalimido derivatives (Matijašić *et al.*, 1988; Iwasaki & Masuko, 1986; Bats & Teuber, 1985; Loehlin, 1985; Voliotis, Arrieta & Germain, 1984).

The packing of the molecule in the solid state, illustrated in Fig. 2, is due only to intermolecular van der Waals forces.

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Structure of Diethyl 3,6-Dihydroxy-11,11-dimethoxypentacyclo[6.5.0.0^{4,12}.0^{5,10}.0^{9,13}]-tridecane-2,6-diene-2,7-dicarboxylate

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Abstract. C₂₁H₂₆O₈, *M_r* = 406.43, orthorhombic, *Pcab* (non-standard setting of *Pbca*), *a* = 8.181 (1), *b* = 18.852 (3), *c* = 25.413 (3) Å, *V* = 3918.1 (8) Å³, *Z* = 8, *D_x* = 1.38 Mg m⁻³, λ(Cu *Kα*) = 1.54178 Å, μ = 0.84 mm⁻¹, *F*(000) = 1728, *T* = 295 K, final *R* = 0.049, *wR* = 0.050 for 2105 observed reflections. Strain is

evidenced in the molecule by the fact that two of the three *sp*³—*sp*³ cross-cage carbon bonds are quite long [1.588 (4) and 1.596 (4) Å] and by the acute internal cage angle of 95.0 (2)° at the C atom which is common to both five-membered rings. Further strain is added to the molecule by the presence of the