C6—C5—C14	109.7 (2)	C3′—C4′—C5′	117.9 (3)
C10—C5—C14	114.6 (2)	O2—C5′—C4′	119.4 (3)
C5—C6—C7	111.4 (2)	O2—C5′—C6′	118.1 (2)
C6—C7—C8	113.3 (2)	C4′—C5′—C6′	122.3 (2)
C7—C8—C9	114.2 (2)	C1′—C6′—C5′	120.9 (2)
C7—C8—C13	108.1 (2)	O1—C7′—O3	122.5 (3)
C9—C8—C13	113.8 (2)	O1—C7′—C8′	112.4 (2)
C8—C9—C10	109.4 (2)	O3—C7′—C8′	125.1 (3)
C8—C9—C11	108.0 (2)	O2—C9′—O4	121.4 (4)
C8—C9—C12	111.4 (2)	O2—C9′—C10′	112.5 (3)
C10—C9—C11	109.8 (2)	O4—C9′—C10′	126.0 (4)
C10—C9—C12	112.9 (2)		
C7′—O1—C2′—C1′	−96.6 (3)	C13—C8—C9—C12	−44.1 (3)
C2′—O1—C7′—O3	4.9 (4)	C2—C3—C4—C5	3.4 (4)
C9′—O2—C5′—C4′	95.1 (3)	C8—C9—C11—C1′	63.3 (3)
C5′—O2—C9′—O4	−8.1 (6)	C9—C11—C1′—C2′	−96.2 (3)
C4—C5—C10—C9	−174.7 (2)	C6′—C1′—C2′—O1	−178.3 (2)
C14—C5—C10—C9	66.9 (3)	C3′—C4′—C5′—O2	175.5 (2)

The equipment of the CIMCF of the University 'Federico II' of Naples was used for collection of the crystallographic data. H atoms were placed on the basis of geometrical considerations and ΔF map suggestions in the case of the hydroxy and methyl groups. All H atoms were included with B_{iso} values fixed at the values of B_{eq} of their respective parent atoms. The structure was solved using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). All calculations were performed using Enraf–Nonius *SDP* software (B. A. Frenz & Associates Inc., 1985) on a MicroVAX 3100 computer.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and the atomic parameters of the restrained refinement have been deposited with the IUCr (Reference: NA1165). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Configuration and Conformation of (3*S*,3a*R*,4*R*,7*S*,7a*S*)-4-Methyl-7-(2-propyl)-2-oxo-2,3,3a,4,5,6,7,7a-octahydro-3-benzofurancarboxylic Acid

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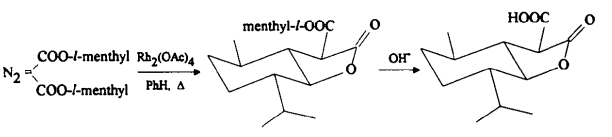
Abstract

The crystal structure determination of the title compound, $C_{13}H_{20}O_4$, together with the knowledge of the configuration of the starting menthyl reagent, define both its absolute configuration and that of the menthyl ester from which it is obtained by hydrolysis. The structure of the title compound in the crystal is compared with that obtained for the isolated molecule by molecular-mechanics energy minimization. The largest discrepancies are observed for the carboxyl group which, in the crystal, is involved in hydrogen bonding with the same group of an adjacent molecule packed about a twofold axis of the $P2_12_12$ space group. The conformation of the molecule is discussed.

Comment

The catalytic decomposition of di(1*R*,3*S*,4*S*)-(−)-menthyldiazomalonate, (1), in the presence of rhodium-(II) acetate dimer gave exclusive formation of the bicyclic γ-lactone (2) by intramolecular carbenic attack on a methylenic C—H bond of the menthyl ring, according to the scheme below.



(1) (2) (3)

The crystal structure analysis of the acid (3), obtained by hydrolysis of the ester (2), was carried out to establish the configurations at the chiral centres of these

products, this knowledge being necessary to interpret the regio- and diastereospecific process of the intramolecular cyclization that produces them.

Fig. 1 shows the chiralities, *S*(C3), *R*(C4), *R*(C5), *S*(C8), *S*(C9), of the five centres and the *transoid* configuration at the junction of the cyclohexane and tetrahydropyran rings. The values of the total puckering amplitudes (Cremer & Pople, 1975) of these rings are 0.590 (3) and 0.409 (2) Å with chair and envelope conformations, respectively, the latter with a local pseudo mirror through C4. The least-squares planes through these rings form a dihedral angle of 3.0 (1)°, with the C3—C4 bond (+)synclinal to C9—O1 [C3—C4—C9—O1 = 40.6 (2)°], and C5—C4 (−)synclinal to C9—C8 [C5—C4—C9—C8 = −66.1 (3)°]. C2—O2 is (−)synclinal to C3—C10 [O2—C2—C3—C10 = −32.2 (4)°].

Table 2 compares relevant geometric descriptors of the molecule in the crystal with those calculated from the optimized geometries obtained for the isolated molecule by the *PCMODEL-MMX* (Serena Software, 1989) and *HYPERCHEM-MM+* (Autodesk, Inc., 1992) force fields, starting from the experimental coordinates and using the program default parameters. The largest deviations are observed for distances and angles of the carboxyl group: this is to be expected since the calculated values relate to an isolated molecule and therefore take no account of the effects of the intermolecular hydrogen bonding in which the group is involved in the crystal. A possible geometry for this fairly strong hydrogen bond [O3···O3^{i} = 2.658 (3) Å; symmetry code: (i) $-x, -y, z$] is depicted in Fig. 2.

Non-bonded energy profiles (deposited), calculated with the *ROTENER* program (Nardelli, 1988) for rotation of the carboxyl group about the C3—C10 bond and of the isopropyl group about C8—C11 in the isolated molecule show the deepest minimum shifted by 22.5 and −4°, respectively [positive rotations are counterclockwise], with respect to the orientation found for these substituents in the crystal. This finding is in agreement with the differences between the observed and calculated torsion angles about the C3—C10 and C8—C11 bonds (Table 2). The differences between the observed and calculated torsion angles about C3—C10 are much larger (*ca* 5 times) than those about C8—C11; the former are governed by the fairly strong O—H···O intermolecular hydrogen bond, the latter by weak intramolecular van der Waals forces.

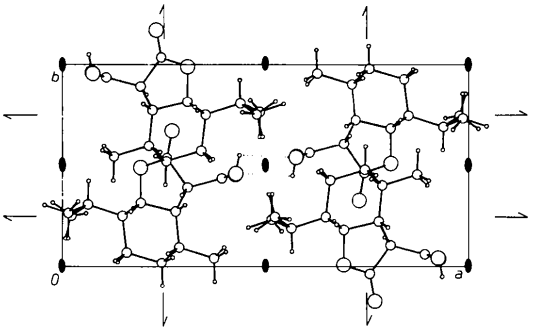


Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) projection of the contents of the unit cell, view down the *z* axis, showing the molecules packed by hydrogen bonding about the twofold axis.

The 'thermal'-motion analysis of the molecule in the crystal, carried out in terms of the Schomaker & Trueblood (1968) **TLS** rigid-body approximation using the *THMV* program (Trueblood, 1984), shows that there are 'internal motions' (or static disorder) of some relevance. Indeed, the agreement between the observed and calculated atomic displacement parameters is not particularly good, the value of the overall residual disagreement index R_{wU} being 0.125, though it improves markedly to 0.073 if the internal motions of O1, O2, O3, O4, C12, C13 and C14 (see Fig. 1) are considered, according to Dunitz & White (1973).

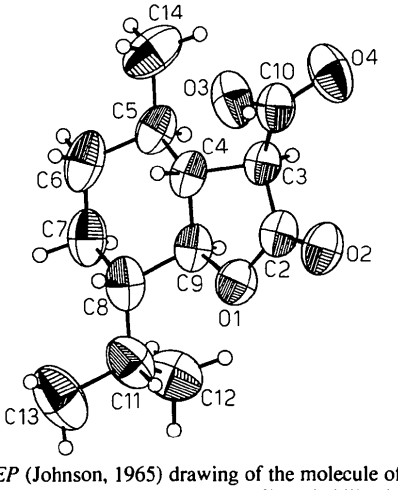


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule of compound (3). Ellipsoids are drawn at the 50% probability level.

Experimental

Preparation of (2). A mixture of diazomalonate (1 mmol) [prepared according to Saba (1994)] and rhodium(II) acetate dimer catalyst (0.05 mmol) was refluxed in benzene (9 ml) under argon until the disappearance of the IR absorption of the diazo function (15 min). The mixture was filtered on a short column of neutral Al_2O_3; removal of the solvent and column chromatography on SiO_2 afforded the bicyclic lactone (2) (82% yield) as a white powder, m.p. 338–339 K; $[\alpha]_D^{25}$ = −73° (*c* 0.32 $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ: 4.78 (1H, *t*, *J* = 11.1 Hz), 4.77 (1H, *t*, *J* = 11.1 Hz), 3.70 (1H, *t*, *J* = 10.8 Hz), 3.26 (1H, *d*, *J* = 12.6 Hz), 2.23 (1H, *dq*, *J* = 10.8 and 12.6 Hz), 2.14–1.86 (3H, *m*), 1.84–1.65 (5H, *m*), 1.64–1.38 (4H, *m*), 1.34–0.82 (20H, *m*), 0.77 p.p.m. (3H, *d*, *J* = 6.3 Hz). IR ($CHCl_3$): 2962, 2871, 1775, 1724, 1453, 1369, 1297, 1241, 1170, 991 cm^{-1}.

Preparation of (3). Hydrolysis of (2) gave colourless crystals, m.p. 409–410 K; $[\alpha]_D^{25}$ = −46° (*c* 0.29 $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ: 3.76 (1H, *t*, *J* = 10.8 Hz), 3.34 (1H, *t*, *J* = 12.9 Hz),

2.24 (1H, *dq*, *J* = 10.8 and 12.9 Hz), 1.93 (1H, *m*), 1.79–1.59 (4H, *m*), 1.16 (2H, *m*), 0.96 (3H, *d*, *J* = 6.6 Hz), 0.95 (3H, *d*, *J* = 6.9 Hz), 0.90 p.p.m. (3H, *d*, *J* = 6.9 Hz). ^{13}C NMR ($CDCl_3$) δ: 173.7, 171.7, 84.8, 52.7, 46.5, 35.1, 34.2, 28.5, 24.8, 19.7, 19.1, 17.8 p.p.m. IR ($CHCl_3$): 2959, 2929, 2840, 2142, 1780, 1753, 1717, 1170, 1148, 1128, 1106, 996 cm^{-1}.

Crystal data

$C_{13}H_{20}O_4$
M_r = 240.30
Orthorhombic
$P2_12_12$
a = 17.067 (7) Å
b = 8.425 (3) Å
c = 9.597 (4) Å
V = 1379.9 (9) Å^3
Z = 4
D_x = 1.157 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 30 reflections
θ = 15–28°
μ = 0.695 mm^{-1}
T = 293 (2) K
Small needle
0.28 × 0.21 × 0.12 mm
Colourless

Data collection

Siemens AED diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2965 measured reflections
2615 independent reflections
1219 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0242
θ_{max} = 70.42°
$h = -20 \rightarrow 20$
$k = -10 \rightarrow 10$
$l = -1 \rightarrow 11$
1 standard reflection monitored every 50 reflections
intensity decay: within statistical fluctuation

Refinement

Refinement on F^2
$R(F)$ = 0.0380
$wR(F^2)$ = 0.1298
S = 0.817
2610 reflections
165 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.027
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.017 (1)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1992, Vol. C, 4.2.6.8, 6.1.1.4)

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

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

	x	y	z	U_{eq}
O1	−0.3079 (1)	0.0102 (2)	−0.2688 (2)	0.0866 (7)
O2	−0.2305 (1)	−0.1722 (2)	−0.3658 (2)	0.1005 (7)
O3	−0.0746 (1)	0.0456 (3)	−0.2544 (2)	0.1109 (8)
O4	−0.0739 (1)	0.0401 (3)	−0.4830 (2)	0.1246 (10)
C2	−0.2431 (2)	−0.0353 (3)	−0.3377 (3)	0.0804 (10)
C3	−0.1924 (1)	0.1077 (3)	−0.3710 (3)	0.0743 (8)
C4	−0.2214 (1)	0.2260 (3)	−0.2623 (3)	0.0766 (9)
C5	−0.2122 (2)	0.4036 (3)	−0.2803 (3)	0.0882 (11)
C6	−0.2553 (2)	0.4802 (3)	−0.1581 (3)	0.1097 (14)
C7	−0.3408 (2)	0.4284 (3)	−0.1455 (3)	0.1056 (14)
C8	−0.3516 (2)	0.2475 (3)	−0.1388 (3)	0.0924 (11)
C9	−0.3075 (2)	0.1845 (3)	−0.2607 (3)	0.0776 (10)
C10	−0.1078 (2)	0.0630 (3)	−0.3699 (3)	0.0843 (11)
C11	−0.4367 (2)	0.1925 (4)	−0.1297 (3)	0.1132 (14)
C12	−0.4854 (2)	0.2344 (5)	−0.2590 (4)	0.1285 (15)
C13	−0.4770 (2)	0.2472 (5)	0.0022 (3)	0.160 (2)
C14	−0.1265 (2)	0.4561 (4)	−0.2925 (4)	0.136 (2)

Table 2. *Comparison of experimental and calculated bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *in the molecule of compound* (3)

MMX = force field of *PCMODEL*; *MM+* = force field of *HYPERCHEM*, Δ = obs.−calc. Maximum differences between observed and calculated values: distances, $|\Delta|_{max}$ *MMX* = 0.085 Å, *MM+* = 0.077 Å; bond angles, $|\Delta|_{max}$ *MMX* = 7.0°, *MM+* = 7.3°; torsion angles, $|\Delta|_{max}$ *MMX* = 26.2°, *MM+* = 27.2°.

	X-ray obs.	*MMX* calc.	*MMX* Δ	*MM+* calc.	*MM+* Δ
O1—C2	1.345 (3)	1.350	−0.005	1.347	−0.002
O1—C9	1.470 (3)	1.409	0.061	1.406	0.064
O2—C2	1.204 (3)	1.208	−0.004	1.207	−0.003
O3—C10	1.254 (3)	1.339	−0.085	1.331	−0.077
O4—C10	1.245 (3)	1.209	0.036	1.207	0.038
C2—C3	1.517 (4)	1.520	−0.003	1.526	−0.009
C3—C4	1.525 (3)	1.535	−0.010	1.528	−0.003
C3—C10	1.493 (4)	1.511	−0.018	1.517	−0.024
C4—C5	1.515 (4)	1.531	−0.016	1.532	−0.017
C4—C9	1.511 (4)	1.531	−0.020	1.530	−0.019
C5—C6	1.528 (4)	1.545	−0.017	1.545	−0.017
C5—C14	1.533 (4)	1.535	−0.002	1.535	−0.002
C6—C7	1.528 (5)	1.543	−0.015	1.543	−0.015
C7—C8	1.537 (4)	1.546	−0.009	1.545	−0.008
C8—C9	1.489 (4)	1.533	−0.044	1.534	−0.045
C8—C11	1.527 (5)	1.547	−0.020	1.547	−0.020
C11—C12	1.534 (5)	1.538	−0.004	1.538	−0.004
C11—C13	1.513 (5)	1.540	−0.027	1.540	−0.027
C2—O1—C9	107.9 (2)	113.0	−5.1	109.7	−1.8
O1—C2—O2	122.0 (2)	125.7	−3.7	124.0	−2.0
O2—C2—C3	127.7 (3)	127.8	−0.1	126.4	1.3
O1—C2—C3	110.2 (2)	106.4	3.8	109.6	0.6
C2—C3—C4	101.0 (2)	102.1	−1.1	100.4	0.6
C2—C3—C10	110.5 (2)	111.6	−1.1	114.1	−3.6
C4—C3—C10	118.3 (2)	115.6	2.7	115.2	3.1
C3—C4—C5	122.3 (2)	121.2	1.1	121.6	0.7
C3—C4—C9	99.8 (2)	99.6	0.2	99.6	0.2
C5—C4—C9	109.3 (2)	109.0	0.3	109.1	0.2
C4—C5—C6	106.2 (2)	107.0	−0.8	107.0	−0.8
C4—C5—C14	113.1 (2)	112.0	1.1	112.2	0.9
C6—C5—C14	113.3 (3)	111.2	2.1	111.1	2.2
C5—C6—C7	113.6 (3)	113.4	0.2	113.4	0.2
C6—C7—C8	113.6 (2)	113.2	0.4	113.2	0.4
C7—C8—C9	105.1 (2)	105.8	−0.7	106.0	−0.9
C7—C8—C11	114.6 (2)	114.9	−0.3	114.9	−0.3
C9—C8—C11	114.7 (2)	113.3	1.4	113.2	1.5
O1—C9—C4	103.6 (2)	101.8	1.8	104.9	−1.3
O1—C9—C8	113.3 (2)	116.0	−2.7	115.1	−1.8
C4—C9—C8	114.7 (2)	111.0	3.7	110.6	4.1
O3—C10—O4	122.8 (3)	122.5	0.3	122.5	0.3
O3—C10—C3	118.3 (2)	111.6	6.7	111.2	7.1
O4—C10—C3	118.9 (2)	125.9	−7.0	126.2	−7.3
C8—C11—C12	113.5 (3)	114.7	−1.2	114.6	−1.1
C8—C11—C13	112.8 (3)	111.8	1.0	111.8	1.0
C12—C11—C13	111.1 (3)	109.7	1.4	109.6	1.5
O2—C2—C3—C10	−32.2 (4)	−35.3	3.1	−32.9	0.7
C2—C3—C10—O3	−76.9 (3)	−52.2	−24.7	−50.7	−26.2
C2—C3—C10—O4	101.0 (3)	127.2	−26.2	128.2	−27.2
C3—C4—C5—C6	174.4 (2)	175.0	−0.6	175.4	−1.0
C3—C4—C5—C14	−60.6 (3)	−62.8	2.2	−62.5	1.9
C3—C4—C9—C8	164.5 (2)	163.9	0.6	163.3	1.2
C5—C4—C9—O1	169.9 (2)	167.7	2.2	166.9	3.0
C6—C7—C8—C11	−178.6 (3)	−179.5	0.9	−179.7	1.1
C7—C8—C9—O1	177.4 (2)	177.3	0.1	−179.5	−3.1
C7—C8—C11—C12	64.1 (3)	60.1	4.0	59.0	5.1
C7—C8—C11—C13	−63.5 (3)	−65.5	2.0	−66.6	3.1
C9—C8—C11—C12	−57.6 (3)	−61.7	4.1	−63.1	5.5
C9—C8—C11—C13	174.8 (3)	172.7	2.1	171.4	3.4

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O3^i	0.83	2.39	2.658 (3)	100

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

The integrated intensities were obtained by a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) peak-profile analysis procedure, and corrected for Lorentz and polarization effects, but not for absorption.

The structure was solved by direct methods and refined on F^2 by anisotropic full-matrix least squares. The H atoms were placed in calculated positions, riding on the attached atoms; four isotropic U(H) parameters, constrained to be equal for chemically equivalent H atoms, were refined, as were the orientation and C—H distance for each of the three methyl groups. H3O was found in a $\Delta\rho$ synthesis out of the plane of the parent CO_2 group and was not subsequently refined. An independent refinement on F with *SHELX*76 (Sheldrick, 1976) using 1223 observed [$I > 2\sigma(I)$] reflections and 194 parameters gave results essentially equal to those reported here.

The anomalous scattering effects did not give unequivocally the absolute configuration of the molecule, the value of the Flack (1983) index being $x = 0.4$ (3). The configuration was assigned on the basis of the known chiralities of the menthyl C atoms in (1).

The calculations were carried out on the ENCORE91 and GOULD-POWERNODE 6040 computers of the Centro di Studio per la Strutturistica Diffrattometrica del CNR (Parma), and on a COMPAQ-486*c* portable computer.

Data collection: local programs. Cell refinement: *LQPARM* (Nardelli & Mangia, 1984). Data reduction: local programs. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP* (Johnson, 1965); *PLUTO* (Motherwell & Clegg, 1976). Geometrical calculations and preparation of the material for publication: *PARST* (Nardelli, 1983); *PARSTCIF* (Nardelli, 1991).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and difference-energy profiles have been deposited with the IUCr (Reference: MU1174). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5a-*p*-Methylphenyl-5a,5b,6,7,8,9,9a,10-octahydro-5*H*-isoindolo[2,1-*a*]benzimidazol-10-one

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Abstract

The title compound, $C_{21}H_{22}N_2O$, was prepared by the reaction of *cis*-2-(4-methylbenzoyl)cyclohexanecarboxylic acid and *o*-phenylenediamine. A mixture of two isomeric compounds was isolated and separated by column chromatography. The compounds differ in the cyclohexane–pyrrolidone annelation, which is *cis* in the title compound. The mutual arrangement of the aryl group and the annelation H atoms is also *cis*.

Comment

For the synthesis of potential anorectic compounds a great number of saturated or partly saturated isoindolone derivatives have been prepared (Stájer, Csende, Bernáth, Sohár & Szúnyog, 1994; Stájer, Csende, Bernáth &