Molecule 1, cyclopropyl ring *B*

Equation of plane: $5.477\,(10)x - 3.285\,(36)y + 10.022\,(12)z = 6.077\,(24)$

Interplanar dihedral angles in molecule 1 (°)

Diimidazoquinazoline ring–oxadiazole ring	7.08 (7)
Diimidazoquinazoline ring–cyclopropyl ring	82.64 (7)
Oxadiazole ring–cyclopropyl ring	85.82 (7)

Molecule 2, diimidazoquinazoline group

Equation of plane: $6.833\,(1)x + 0.446\,(7)y + 7.203\,(2)z = 6.311\,(2)$

Deviation of atoms from the plane (Å):
(* Denotes an atom included in the calculation of the least-squares plane)

Diimidazoquinazoline group (rings *C′*, *D′*, *E′* and *F′*)			
C6′*	−0.043 (1)	C15′*	0.033 (1)
N7′*	0.001 (1)	C14′*	0.029 (1)
C8′*	0.051 (1)	C13′*	−0.015 (1)
N9′*	0.036 (1)	C12′*	−0.056 (1)
C5′*	−0.021 (1)	C11′*	−0.018 (1)
N4′*	−0.044 (1)	C10′*	0.026 (1)
C16′*	0.021 (1)		
N1′	0.036 (2)	C2′	−0.045 (2)
C3′	−0.036 (2)		
Oxadiazole ring *A′*			
N1*A′*	−0.242 (2)	N4*A′*	−0.263 (2)
O2*A′*	−0.101 (2)	C5*A′*	−0.332 (2)
C3*A′*	−0.130 (2)		
Cyclopropyl ring *B′*			
C1*B′*	−0.506 (3)	C3*B′*	−1.386 (3)
C2*B′*	0.076 (3)		

R.m.s. deviation of fitted atoms from plane = 0.034 Å

Molecule 2, oxadiazole ring *A′*

Equation of plane: $7.175\,(3)x + 2.605\,(18)y + 6.404\,(7)z = 7.031\,(6)$

Deviation of atoms from the plane (Å):
(* Denotes an atom included in the calculation of the least-squares plane)

Oxadiazole ring *A′*			
N1*A′**	0.000 (1)	N4*A′**	0.002 (1)
O2*A′**	0.002 (1)	C5*A′**	−0.001 (1)
C3*A′**	−0.002 (1)		
Cyclopropyl ring *B′*			
C1*B*	−0.017 (3)	C3*B*	−0.816 (4)
C2*B*	0.658 (4)		

R.m.s. deviation of fitted atoms from plane = 0.002 Å

Molecule 2, cyclopropyl ring *B′*

Equation of plane: $4.745\,(11)x - 4.318\,(36)y + 10.659\,(10)z = 9.760\,(13)$

Interplanar dihedral angles in molecule 2 (°)

Diimidazoquinazoline ring–oxadiazole ring	6.24 (7)
Diimidazoquinazoline ring–cyclopropyl ring	85.63 (6)
Oxadiazole ring–cyclopropyl ring	89.49 (7)

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1992). Molecular graphics: *SNOOPI* (Davies, 1982).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: LI1112). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Polymorph (II) of Methyl 3-(4-Ethoxy-α-hydroxyiminobenzyl)-1,2,2-trimethylcyclopentanecarboxylate

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Abstract

The title compound, $C_{19}H_{27}NO_4$, is an intermediate product in the synthesis of oxazepinic and piperidinic compounds. Two crystalline forms of the title compound have been obtained but only one [form (II)] gave suitable crystals for X-ray structure analysis. The five-membered ring has an envelope conformation with C(2) 0.60 (5) Å out of the plane defined by the other four atoms. It has been established that the oxime group has a *syn* conformation and the two enantiomers form a dimer around the centre of symmetry by means of hydrogen bonds between their oxime groups.

Comment

The starting material for the synthesis of these oximes is camphoric anhydride (Chevallet *et al.*, 1988). Recrystallization of methyl 3-(4-ethoxy-α-hydroxyiminobenzyl)-1,2,2-trimethylcyclopentane-

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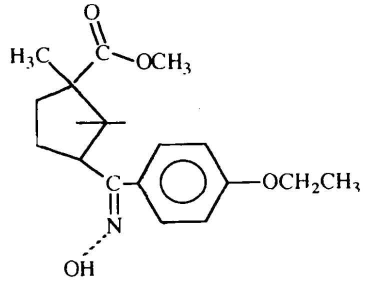
carboxylate (II) gives two polymorphic forms with different melting points: 401 K for polymorph (I) and 398 K for polymorph (II) (Nurit, Pauvert, Doumbia, Chevallet & Térol, 1995). The crystal structure determination was carried out to compare the conformations of the two polymorphs and to determine the configuration (*syn* or *anti*) of the oxime functional group. Only polymorph (II) afforded suitable crystals for data collection and so we present its crystallographic structure here.



(II)

The bond distances of the cyclopentane ring in the title compound show the same differences as displayed by those of the cyclopentane ring in each of the two polymorphs of (1*R*,3*S*)-1,2,2-trimethyl-3-(4-methylthiobenzoyl)cyclopentanecarboxylic acid (Rambaud *et al.*, 1992, 1993). It is probable that these small differences, which are slightly more important for the C(2)—C(3), C(3)—C(4) and C(4)—C(5) bonds, result from the presence of the oxime group in the α position in place of the carbonyl group, leading to shorter interatomic bonds because of π-electron delocalization.

The five-membered ring has an envelope conformation with atom C(2) out of the plane defined by the other four atoms C(1), C(5), C(4) and C(3), giving a *cis* conformation (Rambaud *et al.*, 1993). The dihedral angle C(3)—C(31)—N—O(2) of 180 (0.5)° shows that the oxime is in a *syn* configuration.

The length of the O—H bond is about 1.2 Å, greater than the values usually found for a hydroxyl group. This can be explained by the presence of two hydrogen bonds between the oxime groups. The oxime groups are thus involved in hydrogen bonding leading to the formation of a dimer *via* a ring of six atoms (Fig. 1). The two bonds are 2.753 (5) Å in length with an N···H—O angle of 141.3 (5)°; the two molecules forming the dimer are enantiomers [(x, y, z) and ($-1-x$, $1-y$, $-z$)] related by the centre of symmetry. The packing in the unit cell is the result of the hydrogen bonds forming the dimer and short van der Waals contacts.

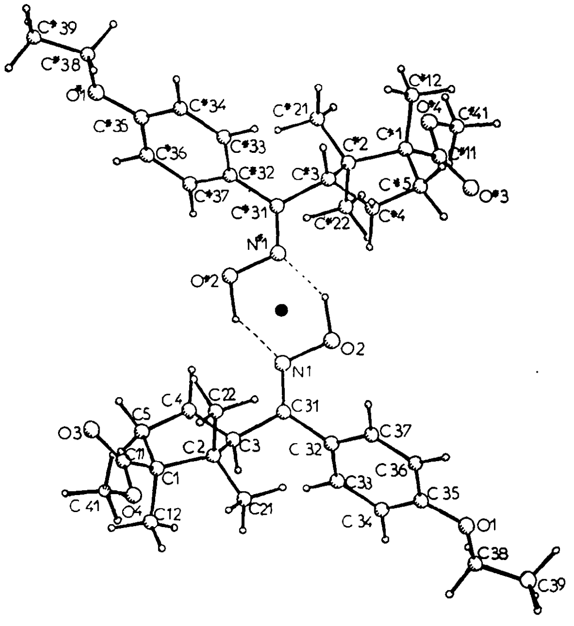


Fig. 1. View (*PLUTO*; Motherwell & Clegg, 1978) of the two enantiomers with respect to the centre of symmetry.

Experimental

Crystal data

$C_{19}H_{27}NO_4$
$M_r = 333.428$
Monoclinic
$C2/c$
$a = 24.179$ (2) Å
$b = 11.203$ (1) Å
$c = 17.286$ (2) Å
$\beta = 125.62$ (1)°
$V = 3806$ (1) Å^3
$Z = 8$
$D_x = 1.16$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 18 reflections
$\theta = 15$–25°
$\mu = 0.578$ mm^{-1}
$T = 293$ K
Prism
0.43 × 0.3 × 0.3 mm
Colourless

Data collection

Huber four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3432 measured reflections
3432 independent reflections
2850 observed reflections [$I \geq 2.5\sigma(I)$]

$\theta_{\max} = 67.5°$
$h = -28 \rightarrow 23$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 20$
3 standard reflections monitored every 50 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.058$
$wR = 0.088$
$S = 1.53$
2850 reflections
228 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.005315F^2]$

$(\Delta/\sigma)_{\max} = 0.1$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: none
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)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
C(1)	−0.7484 (1)	0.4269 (2)	−0.3402 (1)	4.7 (1)
C(11)	−0.7706 (1)	0.5256 (2)	−0.4123 (2)	5.6 (1)
C(12)	−0.8001 (1)	0.3248 (3)	−0.3881 (2)	6.8 (2)
C(2)	−0.6730 (1)	0.3861 (2)	−0.2962 (1)	4.8 (1)
C(21)	−0.6679 (2)	0.2891 (4)	−0.3538 (2)	9.8 (2)
C(22)	−0.6304 (2)	0.4922 (4)	−0.2858 (3)	9.2 (2)
C(3)	−0.6501 (1)	0.3388 (2)	−0.1977 (2)	4.3 (1)
C(31)	−0.5742 (1)	0.3175 (2)	−0.1278 (2)	4.2 (1)
C(32)	−0.5454 (1)	0.2011 (2)	−0.1293 (1)	4.0 (1)
C(33)	−0.4878 (1)	0.1941 (2)	−0.1294 (2)	4.8 (1)
C(34)	−0.4622 (1)	0.0851 (2)	−0.1304 (2)	5.3 (1)
C(35)	−0.4915 (1)	−0.0197 (2)	−0.1288 (1)	4.6 (1)
C(36)	−0.5487 (1)	−0.0145 (2)	−0.1283 (1)	5.0 (1)
C(37)	−0.5750 (1)	0.0953 (2)	−0.1286 (1)	4.8 (1)
C(38)	−0.4872 (1)	−0.2329 (2)	−0.1218 (2)	6.3 (2)
C(39)	−0.4400 (2)	−0.3292 (3)	−0.1101 (2)	7.6 (2)
C(4)	−0.6823 (1)	0.4236 (3)	−0.1655 (1)	6.9 (1)
C(41)	−0.7948 (2)	0.5745 (4)	−0.5622 (2)	8.5 (2)
C(5)	−0.7460 (1)	0.4711 (3)	−0.2542 (2)	7.0 (1)
N	−0.5392 (1)	0.4027 (2)	−0.0707 (1)	4.7 (1)
O(1)	−0.4607 (1)	−0.1223 (1)	−0.1269 (1)	6.0 (1)
O(2)	−0.4687 (1)	0.3777 (1)	−0.0067 (1)	5.8 (1)
O(3)	−0.7808 (1)	0.6272 (2)	−0.4026 (2)	9.4 (1)
O(4)	−0.7777 (1)	0.4869 (2)	−0.4909 (1)	6.2 (1)

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

C(34)—C(35)	1.380 (3)	C(1)—C(5)	1.537 (3)
C(36)—C(35)	1.388 (3)	C(2)—C(1)	1.580 (2)
O(1)—C(35)	1.359 (3)	C(12)—C(1)	1.533 (3)
C(33)—C(34)	1.374 (3)	C(11)—C(1)	1.512 (3)
C(32)—C(33)	1.396 (3)	C(21)—C(2)	1.525 (3)
C(37)—C(32)	1.388 (3)	C(22)—C(2)	1.513 (4)
C(31)—C(32)	1.484 (3)	C(39)—C(38)	1.496 (3)
C(36)—C(37)	1.384 (3)	O(1)—C(38)	1.421 (3)
C(3)—C(31)	1.518 (2)	O(3)—C(11)	1.197 (3)
N—C(31)	1.274 (2)	O(4)—C(11)	1.338 (3)
C(4)—C(3)	1.522 (3)	O(4)—C(41)	1.435 (3)
C(2)—C(3)	1.547 (3)	O(2)—N	1.417 (2)
C(5)—C(4)	1.502 (3)		
C(36)—C(35)—C(34)	119.3 (2)	C(12)—C(1)—C(5)	108.8 (2)
O(1)—C(35)—C(34)	116.1 (2)	C(12)—C(1)—C(2)	113.2 (2)
O(1)—C(35)—C(36)	124.7 (2)	C(11)—C(1)—C(5)	110.9 (2)
C(33)—C(34)—C(35)	121.1 (2)	C(11)—C(1)—C(2)	111.4 (2)
C(32)—C(33)—C(34)	120.5 (2)	C(11)—C(1)—C(12)	108.3 (2)
C(37)—C(32)—C(33)	118.1 (2)	C(1)—C(2)—C(3)	101.6 (1)
C(31)—C(32)—C(33)	121.8 (2)	C(21)—C(2)—C(3)	111.2 (2)
C(31)—C(32)—C(37)	120.1 (2)	C(21)—C(2)—C(1)	113.6 (2)
C(36)—C(37)—C(32)	121.4 (2)	C(22)—C(2)—C(3)	110.9 (2)
C(37)—C(36)—C(35)	119.7 (2)	C(22)—C(2)—C(1)	110.2 (2)
C(3)—C(31)—C(32)	119.1 (2)	C(22)—C(2)—C(21)	109.2 (3)
N—C(31)—C(32)	124.3 (2)	O(1)—C(38)—C(39)	107.5 (2)
N—C(31)—C(3)	116.6 (2)	O(3)—C(11)—C(1)	125.6 (2)
C(4)—C(3)—C(31)	116.5 (2)	O(4)—C(11)—C(1)	111.9 (2)
C(2)—C(3)—C(31)	114.9 (2)	O(4)—C(11)—O(3)	122.5 (3)
C(2)—C(3)—C(4)	104.9 (2)	O(2)—N—C(31)	114.2 (2)
C(5)—C(4)—C(3)	106.7 (2)	C(38)—O(1)—C(35)	118.5 (2)
C(1)—C(5)—C(4)	108.1 (2)	C(41)—O(4)—C(11)	116.9 (2)
C(2)—C(1)—C(5)	104.2 (2)		

Program used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Refinement was by full-matrix least-squares methods (*SHELX*76; Sheldrick, 1976). Program used for calculation of dihedral angles: *XANADU* (Roberts & Sheldrick, 1975).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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6′-Diethylamino-2′-nitrospiro[isobenzofuran-1(3*H*),9′-[9*H*]xanthen]-3-one

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Abstract

The title molecule, $C_{24}H_{20}N_2O_5$, is composed of two parts, namely, a butterfly-like xanthene moiety and an almost planar isobenzofuran fragment. The isobenzofuran plane is almost perpendicular to the two xanthene planes. The C—O bond length in the five-membered lactone ring is 1.487 (5) Å, which is longer than the usual lactone C—O single-bond length.

Comment

Since Meyer & Hoffmeyer (1892) first synthesized fluoran, several derivatives have been prepared and

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