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(±)-*threo*-3-Hydroxy-2,3-diphenylpropanoic Acid Dimethylamide

TSONKO KOLEV, HANS PREUT AND PAUL BLECKMANN

*Fachbereich Chemie, Universität Dortmund,
D-44227 Dortmund, Germany*

IVAN JUCHNOVSKI

*Institute of Organic Chemistry, Bulgarian Academy
of Sciences, Sofia 1113, Bulgaria*

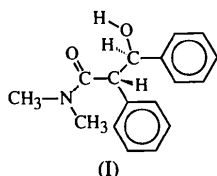
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Abstract

The crystal structure of (±)-*threo*-3-hydroxy-*N,N*-dimethyl-2,3-diphenylpropanamide, $C_{17}H_{19}NO_2$, has been determined. In the crystalline state the hydroxyl group bound to the β -C atom and the phenyl group bound to the α -C atom are *trans* with respect to one another and the two H atoms at these two chiral C atoms have an antiperiplanar conformation. The structure is stabilized by intermolecular hydrogen bonds between carbonyl and hydroxy O atoms. Bond distances are as expected.

Comment

The structural determination of the title compound (I) was undertaken in order to obtain more structural information about substituted diphenylethanes possessing two chiral C atoms. This paper is part of a project investigating *threo* and *erythro* forms, as well as chiral forms, of substituted diphenylethanes. This information is significant because the diphenylethane skeleton is present in some natural products and plays an important role in some stereochemical reactions (Cuvigny, Hullot, Lorcheveque & Normant, 1974; Mladenova, Blagoev & Kurtev, 1974, 1979). We are also interested in the structural and electronic effects of the replacement of the methylene H atoms by OH, COOH, COOR, CONR₂ and aryl groups. The solid-state IR spectra confirm the existence of hydrogen bonding.



The O(hydroxyl)—C(5)—C(4)—C(phenyl) torsion angle is $174.7(2)^\circ$. The dihedral angle between the least-squares planes of the phenyl rings is $67.57(9)^\circ$. The carbonyl and dimethylamido groups are almost

coplanar with O—C—N—C torsion angles of $177.4(3)$ and $-0.9(5)^\circ$. The short intermolecular contact of $2.790(3)$ Å between the carbonyl O(1) atom and the hydroxyl O(2)($1-x, -y, -z$) atom indicates a hydrogen bond. The bond distance between the C(3) and C(4) atoms [$1.526(4)$ Å] is in the normal range, as are the remaining distances and angles.

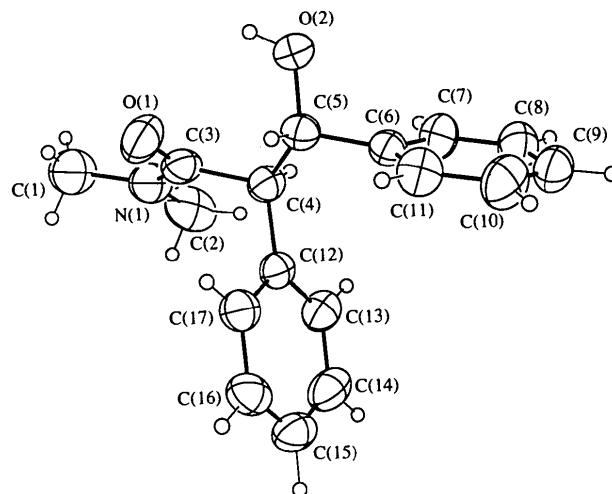


Fig. 1. General view (SHELXTL-Plus; Sheldrick, 1987) of the title molecule showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

Experimental

The title compound was synthesized according to the methods of Cuvigny, Hullot, Lorcheveque & Normant (1974) and Mladenova, Blagoev & Kurtev (1979). Suitable crystals were grown from benzene. The melting point is in the range 449–450 K.

Crystal data

$C_{17}H_{19}NO_2$
 $M_r = 269.34$
 Monoclinic
 $P2_1/n$
 $a = 6.537(4)$ Å
 $b = 15.040(9)$ Å
 $c = 14.572(10)$ Å
 $\beta = 92.92(5)^\circ$
 $V = 1431(2)$ Å³
 $Z = 4$
 $D_x = 1.250$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 6.4-14.7^\circ$
 $\mu = 0.08$ mm⁻¹
 $T = 291$ K
 Column
 $0.56 \times 0.32 \times 0.28$ mm
 Colourless

Data collection

Nicolet R3m/V diffractometer
 $\theta/2\theta$ scans
 Absorption correction: none

$R_{int} = 0.048$
 $\theta_{max} = 25.0^\circ$
 $h = -8 \rightarrow 8$
 $k = -1 \rightarrow 18$
 $l = -18 \rightarrow 18$

5819 measured reflections
2536 independent reflections
1562 observed reflections
[$F > 4\sigma(F)$]

6 standard reflections
frequency: 240 min
intensity decay: none

C(2)—N(1)—C(3)—C(4) —5.2 (4)
C(1)—N(1)—C(3)—C(4) 176.4 (3)
C(2)—N(1)—C(3)—O(1) 177.4 (3)
C(1)—N(1)—C(3)—O(1) -0.9 (5)
O(1)—C(3)—C(4)—C(5) -24.2 (4)
N(1)—C(3)—C(4)—C(5) 158.4 (3)
O(1)—C(3)—C(4)—C(12) 99.0 (3)
N(1)—C(3)—C(4)—C(12) -78.4 (3)
C(3)—C(4)—C(5)—O(2) -64.5 (3)
C(3)—C(4)—C(12)—C(13) 124.7 (3)
C(3)—C(4)—C(12)—C(17) -54.6 (3)
C(3)—C(4)—C(5)—C(6) 176.4 (2)
C(12)—C(4)—C(5)—O(2) 174.7 (2)
C(5)—C(4)—C(12)—C(13) -114.6 (3)
C(5)—C(4)—C(12)—C(17) 66.1 (3)
C(12)—C(4)—C(5)—C(6) 55.6 (3)
C(4)—C(5)—C(6)—C(7) 62.4 (3)
O(2)—C(5)—C(6)—C(7) -58.4 (3)
C(4)—C(5)—C(6)—C(11) -120.9 (3)
O(2)—C(5)—C(6)—C(11) 118.3 (3)

Refinement

Refinement on F

$R = 0.0445$

$wR = 0.0680$

$S = 0.73$

1562 reflections

182 parameters

Only H-atom U 's refined

$w = 1/[\sigma^2(F) + 0.005961F^2]$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.2 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.2 \text{ e } \text{\AA}^{-3}$

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV, Table

2.2B)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
N(1)	0.2638 (4)	0.1523 (1)	-0.1269 (1)	0.0597 (9)
O(1)	0.4903 (3)	0.1103 (1)	-0.0150 (1)	0.0665 (7)
O(2)	0.2106 (3)	-0.0040 (1)	0.0899 (1)	0.0667 (7)
C(1)	0.4169 (6)	0.1472 (2)	-0.1953 (2)	0.0879 (14)
C(2)	0.0598 (5)	0.1764 (2)	-0.1615 (2)	0.0774 (12)
C(3)	0.3156 (4)	0.1334 (2)	-0.0399 (2)	0.0494 (9)
C(4)	0.1544 (4)	0.1446 (2)	0.0314 (2)	0.0436 (8)
C(5)	0.2153 (4)	0.0873 (2)	0.1152 (2)	0.0467 (8)
C(6)	0.0740 (4)	0.0972 (2)	0.1935 (2)	0.0422 (8)
C(7)	-0.1293 (4)	0.0740 (2)	0.1833 (2)	0.0544 (10)
C(8)	-0.2537 (4)	0.0782 (2)	0.2565 (2)	0.0616 (10)
C(9)	-0.1780 (4)	0.1063 (2)	0.3411 (2)	0.0601 (11)
C(10)	0.0228 (5)	0.1293 (2)	0.3517 (2)	0.0674 (12)
C(11)	0.1471 (4)	0.1255 (2)	0.2786 (2)	0.0561 (9)
C(12)	0.1366 (4)	0.2426 (2)	0.0546 (1)	0.0406 (7)
C(13)	-0.0495 (4)	0.2858 (2)	0.0461 (2)	0.0526 (9)
C(14)	-0.0633 (5)	0.3762 (2)	0.0647 (2)	0.0643 (11)
C(15)	0.1058 (5)	0.4229 (2)	0.0926 (2)	0.0609 (10)
C(16)	0.2908 (5)	0.3812 (2)	0.1031 (2)	0.0588 (10)
C(17)	0.3069 (4)	0.2909 (2)	0.0838 (2)	0.0506 (9)

Table 2. Selected geometric parameters (\AA , $^\circ$)

N(1)—C(1)	1.450 (4)	C(7)—C(8)	1.375 (4)
N(1)—C(2)	1.448 (4)	C(8)—C(9)	1.371 (4)
N(1)—C(3)	1.327 (3)	C(9)—C(10)	1.358 (4)
O(1)—C(3)	1.230 (3)	C(10)—C(11)	1.374 (4)
O(2)—C(5)	1.422 (3)	C(12)—C(13)	1.379 (4)
C(3)—C(4)	1.526 (4)	C(12)—C(17)	1.378 (3)
C(4)—C(5)	1.532 (3)	C(13)—C(14)	1.390 (4)
C(4)—C(12)	1.518 (3)	C(14)—C(15)	1.355 (4)
C(5)—C(6)	1.511 (3)	C(15)—C(16)	1.364 (4)
C(6)—C(7)	1.375 (4)	C(16)—C(17)	1.391 (4)
C(6)—C(11)	1.373 (3)		
C(2)—N(1)—C(3)	124.9 (3)	C(7)—C(6)—C(11)	117.8 (3)
C(1)—N(1)—C(3)	119.6 (3)	C(6)—C(7)—C(8)	120.8 (3)
C(1)—N(1)—C(2)	115.4 (3)	C(7)—C(8)—C(9)	120.7 (3)
N(1)—C(3)—O(1)	122.0 (3)	C(8)—C(9)—C(10)	118.9 (3)
O(1)—C(3)—C(4)	119.6 (3)	C(9)—C(10)—C(11)	120.5 (3)
N(1)—C(3)—C(4)	118.3 (3)	C(6)—C(11)—C(10)	121.4 (3)
C(3)—C(4)—C(12)	108.8 (2)	C(4)—C(12)—C(17)	120.7 (3)
C(3)—C(4)—C(5)	108.6 (2)	C(4)—C(12)—C(13)	120.9 (2)
C(5)—C(4)—C(12)	112.8 (2)	C(13)—C(12)—C(17)	118.3 (3)
O(2)—C(5)—C(4)	109.6 (2)	C(12)—C(13)—C(14)	120.5 (3)
C(4)—C(5)—C(6)	113.8 (2)	C(13)—C(14)—C(15)	120.4 (3)
O(2)—C(5)—C(6)	106.6 (2)	C(14)—C(15)—C(16)	120.0 (3)
C(5)—C(6)—C(11)	120.9 (3)	C(15)—C(16)—C(17)	120.1 (3)
C(5)—C(6)—C(7)	121.2 (2)	C(12)—C(17)—C(16)	120.6 (3)

The IR spectrum of the title compound in the solid state (KBr pellet) shows a C=O stretching band at 1621 cm^{-1} , an O—H stretching band at 3415 cm^{-1} and a Raman C=O band at 1620 cm^{-1} . The lattice parameters were determined from a symmetry-constrained least-squares fit. Refinement was based on full-matrix least-squares methods with anisotropic displacement parameters for non-H atoms. The H atoms were placed in calculated positions (C—H 0.96 \AA) and a common isotropic displacement parameter [$0.096 (3) \text{ \AA}^2$] was refined for these atoms.

Data collection: Nicolet *R3m/V* software. Cell refinement: Nicolet *R3m/V* software. Data reduction: Nicolet *R3m/V* software. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus* (Sheldrick, 1987). Software used to prepare material for publication: *PARST* (Nardelli, 1983), *PLATON* (Spek, 1982) and *MISSYM* (Le Page, 1987).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1137). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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