

C5—C6—C1	116.5 (2)	C16—C15—C14	121.3 (2)
C5—C6—C7	120.8 (2)	C15—C16—C17	120.7 (2)
C1—C6—C7	122.7 (2)	C18—C17—C16	118.3 (2)
N20—C7—C14	111.17 (14)	C17—C18—C19	121.2 (2)
N20—C7—C6	106.75 (14)	C14—C19—C18	121.4 (2)
C14—C7—C6	111.50 (15)	C21—N20—C24	106.0 (2)
N20—C7—C8	108.76 (14)	C21—N20—C7	124.7 (2)
C14—C7—C8	107.43 (15)	C24—N20—C7	129.2 (2)
C6—C7—C8	111.24 (15)	N22 C21—N20	112.5 (2)
C9—C8—C13	117.4 (2)	C21—N22—C23	104.2 (2)
C9—C8—C7	122.5 (2)	C24—C23—N22	111.1 (2)
C13—C8—C7	120.2 (2)	C23—C24—N20	106.1 (2)
C8—C9—C10	121.2 (2)		

The space group $P\bar{1}$ was determined from non-systematic absences. Intensity data were corrected for Lorentz and polarization effects. The crystal structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and refined by full-matrix least-squares methods using *SHELXL93* (Sheldrick, 1993) with anisotropic displacement parameters for all non-H atoms. All H atoms were located from difference Fourier maps and were refined isotropically in the final cycles. There were no significant features in the final difference Fourier map. Refinement of the H atoms led to C—H distances in the range 0.88 (3)–1.02 (2) Å. *U* values vary from 0.038 (6) to 0.078 (8) with a mean of 0.057 (8) Å². All geometrical calculations were performed using *GEOM*. All computations were performed using IBM PCs.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *Xtal3.2* (Hall *et al.*, 1992). Program(s) used to solve structure: *SHELXS86*. Program(s) used to refine structure: *SHELXL93*. Molecular graphics: *ORTEX* (McArdle, 1995). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1362). Services for accessing these data are described at the back of the journal.

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(4*S*,5*R*,2'*R*)-3,4-Dimethyl-1-[2'-(1-naphthyl)-2'-phenylacetyl]-5-phenylimidazolidin-2-one

RICARDO BAGGIO,^a GUILLERMO LOTZ,^b SARA PALACIOS^b AND RAÚL A. MARIEZCURRENA^c

^aDepartamento de Física, Comisión Nacional de Energía Atómica, Buenos Aires, Argentina, ^bCEQUIMAP, Facultad de Ciencias Químicas, Universidad Nacional de Córdoba, Córdoba, Argentina, and ^cLaboratorio de Cristalografía y Química del Estado Sólido, Facultad de Química, Universidad de la República, Montevideo, Uruguay. E-mail: baggio@cnea.edu.ar

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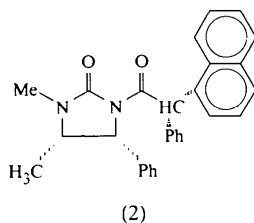
Abstract

The title compound, (4*S*,5*R*)-3,4-dimethyl-1-[(2*R*)-2-(1-naphthyl)-2-phenylacetyl]-5-phenylimidazolidin-2-one, C₂₉H₂₆N₂O₂, has two different aryl groups at the α-carbon which confer a stereogenic character on the site. The geometry of the molecule is stabilized by an intramolecular hydrogen bond linking the acetamide and imidazolidine groups, which gives a planar character to the core.

Comment

Acetamides have been reported as herbicides, especially those with an α-chloro substituent (Couderchet *et al.*, 1986). We have been studying the families of α-aryl- and α,α-diarylacetamides and both have shown interesting growth-inhibitor properties against *Avena sativa* and *Cyperus rotundus* (Palacios *et al.*, 1995). The diaryl family has a stereogenic center when the aryl groups are different. In order to synthesize enantiomerically pure α,α-(1-naphthyl)phenylacetamide, we have recently prepared the imidazolidin-2-one derivative (Lotz *et al.*, 1994). The reaction described (see *Experimental*) provides two epimeric species, viz. (4*S*,5*R*,2'*S*)-, (1), and (4*S*,5*R*,2'*R*)-3,4-dimethyl-1-[2'-(1-naphthyl)-2'-phenyl-

acetyl]-5-phenylimidazolidin-2-one, (2). We report here the crystal and molecular structure of (2).



Bond distances, angles and ring geometries are normal. The naphthyl group presents the usual 'hinge' deformation along the bond joining the fused phenyl rings, with an angle of 4.0 (1)° between normals. The imidazolidine ring displays a clear envelope character, with atoms C3, C7, N1 and N2 being coplanar [maximum deviation from planarity 0.018 (3) Å] and C5 0.395 (3) Å out of the plane.

The central C atom (C5) is chiral. This allows speculation about the possible existence in this family of derivatives of different isomers with an eventual diversity in biological activity; however, to our knowledge, no work has yet been reported on this subject. Three fairly large groups are linked to the tetrahedral C1 atom and the torsion angles (Table 2) show how they are oriented relative to the bonds around C1; they give a minimum distance of 2.08 (4) Å between the H atoms on C22 and C1.

The geometry of the 3,4-dimethyl-5-phenylimidazolidin-2-one group does not differ significantly from that determined by Mulzer *et al.* (1995), except for the orientation of the phenyl group.

The geometry of the molecule is stabilized by an intramolecular hydrogen bond [C1—H1 0.94 (3), H1···O2 2.25 (3) Å and C1—H1···O2 125 (2)°; heavy dashed line in Fig. 1] which clamps any rotation around the acetamide N1—C2 bond, and favors coplanarity of the acetamide and imidazolidine groups. As a bonus, another short contact [O1···H7B 2.49 (2) Å; thin dashed line in Fig. 1] helps the process.

There is one weak intermolecular hydrogen-bond contact [C4—H4 0.99 (3), H4···O1ⁱ 2.52 (4) Å and C4—H4···O1ⁱ 127 (3)°; symmetry code: (i) $x - 1, y, z$], which links molecules by translation along the crystallographic *a* axis. The large naphthalene groups are in two orientations, parallel to the crystallographic *a* axis [roughly (013) and (01 $\bar{3}$)], in an arrangement which suggests that the accommodation of these large planar groups dominates the packing process.

The absolute configuration of (2) was determined from the known absolute structure of the precursor, and the result is in accordance with the most probable conformation derived from calculations performed with the program AM1 (Dewar *et al.*, 1985). Determination of the chirality from the X-ray data was not possible.

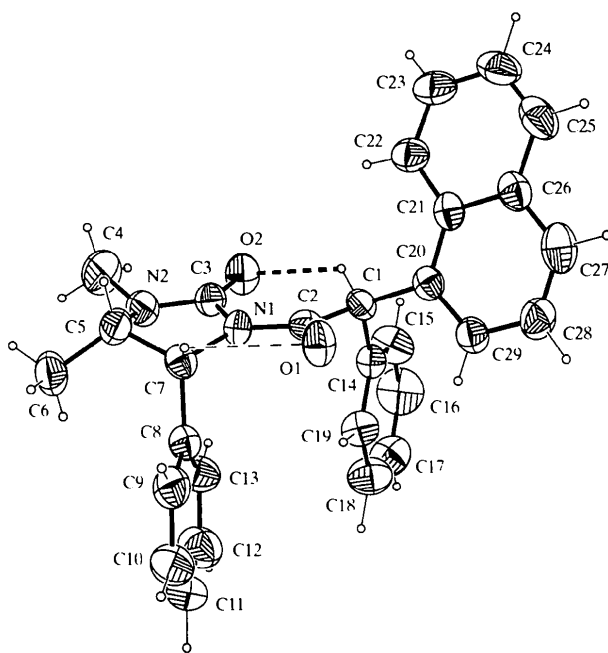


Fig. 1. The molecular diagram of (2) showing the numbering of atoms, as well as a schematic representation of intramolecular contacts. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

To distilled liquid ammonia (150 ml, previously dried under nitrogen with sodium metal) under nitrogen was added lithium metal (1 mmol) and the mixture allowed to react for 1 h until the amide was formed. Then the precursor (4*S*,5*R*)-3,4-dimethyl-5-phenyl-1-(2'-phenylacetyl)imidazolidin-2-one was added. The system was allowed to react under irradiation ($h\nu_{\max} = 350$ nm). After 2 h, the reaction was quenched by adding ammonium nitrate in excess and the ammonia was allowed to evaporate. The residue was dissolved in water, the solution extracted with dichloromethane and the solvent removed under reduced pressure. Finally, the residue was separated by chromatographic methods to yield (1) (m.p. 428 K, yield: 33.6%, crystallized from hexane); ¹H NMR (200 MHz, CDCl₃): δ 0.79 (*d*, 3H, *J* = 8 Hz), 2.77 (*s*, 3H), 3.91 (*dq*, 1H), 5.3 (*s*, 3H), 5.39 (*d*, 1H, *J* = 8 Hz), 6.9–8.2 (*m*, 17H); ¹³C NMR (50 MHz, CDCl₃): 14.9, 28.2, 51.6, 53.6, 59.7, 123.9, 124.9, 125.5, 125.9, 126.3, 127.0, 127.5, 127.8, 128.1, 128.2, 128.6, 129.7, 131.8, 134.0, 134.8, 136.1, 138.4, 155.3, 171.3; MS (*m/e*) (relative intensity): 434 (40.5, *M*⁺), 308 (2.3), 244 (62.8), 217 (55.8), 215 (100), 202 (19.6), 189 (13.6), 58 (11.4); analysis calculated for C₂₉H₂₆N₂O₂: C 80.18, H 5.99, N 6.45%; found: C 80.94, H 6.09, N 6.50%; and (2) (solid, m.p. 459–460 K, yield: 20%, crystallized from ethyl acetate); ¹H NMR (200 MHz, CDCl₃): δ 0.78 (*d*, 3H, *J* = 7.5 Hz), 2.82 (*s*, 3H), 3.92 (*dq*, 1H), 5.29 (*s*, 3H), 5.38 (*d*, 1H, *J* = 10 Hz), 7.0–8.19 (*m*, 17H); ¹³C NMR (50 MHz, CDCl₃): 14.9, 28.2, 51.3, 53.6, 59.7, 123.4, 125.2, 125.4, 126.4, 126.9, 127.2, 127.3, 127.6, 128.0, 128.2, 128.4, 128.7, 129.8, 131.9, 133.8, 135.7, 136.1, 137.8, 155.3, 171.2; MS (*m/e*) (relative intensity): 434 (41.0, *M*⁺), 244 (62.7), 217 (55.4), 215 (100), 202 (19.3), 189 (13.3), 58 (9.6); analysis calculated for C₂₉H₂₆N₂O₂: C

80.18, H 5.99, N 6.45%; found: C 80.94, H 6.09, N 6.50%. Good single crystals of the latter, suitable for X-ray diffraction analysis, were obtained from slow evaporation of a solution of ethyl ether and *n*-pentane.

Crystal data

C₂₉H₂₆N₂O₂
M_r = 434.52
 Orthorhombic
*P*2₁2₁2₁
a = 8.205 (1) Å
b = 10.319 (1) Å
c = 27.168 (3) Å
V = 2300.2 (4) Å³
Z = 4
D_x = 1.255 Mg m⁻³
D_m not measured

Data collection

Rigaku AFC-7S diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 4334 measured reflections
 4038 independent reflections
 2882 reflections with $I > 2\sigma(I)$

Mo K α radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 7.5$ – 12.5°
 $\mu = 0.079$ mm⁻¹
T = 293 (2) K
 Plate
 0.35 × 0.20 × 0.12 mm
 Colorless

*R*_{int} = 0.042
 $\theta_{\max} = 25^\circ$
 $h = -9 \rightarrow 9$
 $k = 0 \rightarrow 12$
 $l = 0 \rightarrow 32$
 2 standard reflections every 98 reflections
 intensity decay: <2%

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.040
wR(*F*²) = 0.087
S = 0.991
 4038 reflections
 402 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 0.20P]$
 where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} = 0.015
 $\Delta\rho_{\max} = 0.11$ e Å⁻³
 $\Delta\rho_{\min} = -0.11$ e Å⁻³
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)
 Absolute structure: Flack (1983)
 Flack parameter = 1.6 (16)

Table 1. Selected torsion angles (°)

C14—C1—C2—N1	-71.5 (3)	C5—C7—C8—C9	-106.0 (3)
N1—C7—C8—C13	-41.4 (3)	C20—C1—C2—N1	163.7 (2)
C3—N1—C2—O1	166.7 (2)	C2—C1—C20—C21	-105.9 (3)
C3—N1—C2—C1	-16.5 (3)	C2—C1—C20—C29	75.6 (3)

The data set was collected with Mo K α radiation up to 50° in 2 θ . The structure was solved by direct methods and completed through successive cycles of ΔF synthesis, where all the H atoms in the structure were finally found. The resulting model was refined on *F*² using the whole data set, with anisotropic displacement factors for non-H atoms and an isotropic model for H atoms. The final ΔF synthesis proved remarkably featureless.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *MSCIAFC Diffractometer Control Software*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *XP* in *SHELXTLIPC* (Sheldrick, 1994). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1223). Services for accessing these data are described at the back of the journal.

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2-(7-Ethylidene-6-methyl-1-oxa-4-thiaspiro-[4.5]dec-7-yl)ethanol

MASOOD PARVEZ,^a VEEJENDRA K. YADAV^b AND DURAISWAMY A. JEYARAJ^b

^aDepartment of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4, and ^bDepartment of Chemistry, Indian Institute of Technology, Kanpur 208 016, India. E-mail: parvez@ucalgary.ca

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Abstract

The crystal of the title compound, C₁₃H₂₂O₂S, has two independent molecules in the asymmetric unit. The molecular dimensions are normal. Molecules are linked into chains along the *b* direction by O—H···O hydrogen bonds, with O···O distances of 2.655 (5) and 2.693 (5) Å.

Comment

In an investigation aimed at assessing the diastereoselectivity caused by an allylically positioned hemithioacetal in the orthoester Claisen rearrangement, we reacted al-