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5-Acetoxy-2,3-diphenylisoxazolidine and 5-acetoxy-3-(4-nitrophenyl)-2-phenylisoxazolidine

Shubhasish Mukherjee,[a] Virinder S. Parmar[a] and William Errington[b]

[a]*Department of Chemistry, University of Delhi, Delhi 110 007, India, and* [b]*Department of Chemistry, University of Warwick, Coventry CV4 7AL, England. E-mail: w.errington@warwick.ac.uk*

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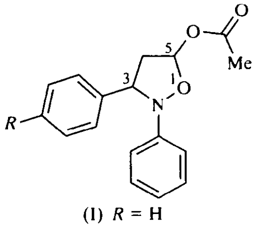
Abstract

The configurations of the isoxazolidine rings in the title compounds, 2,3-diphenylisoxazolidin-5-yl acetate ($C_{17}H_{17}NO_3$), (I), and 3-(4-nitrophenyl)-2-phenylisoxazolidin-5-yl acetate ($C_{17}H_{16}N_2O_5$), (II), are compared. The 2-phenyl group is in an equatorial position, the 5-acetoxy group is axial whilst the C3 substituent occupies a bisectional position. The angle between the phenyl groups in (I) is 80.95 (6)°, but in (II) it is 64.85 (8)°.

Comment

Reactions generally classified as 1,3-dipolar cycloaddition have been extensively employed in the synthesis of a diverse array of heterocyclic compounds (Huisgen, 1968; Hammer & Macaluso, 1964). Cycloadditions are not only ring-forming, but also proceed with a high degree of stereoselectivity. During the course of synthetic studies, a variety of isoxazolidines were prepared in our laboratory by [3+2] cycloadditions of nitrones with olefins (Cum *et al.*, 1968). It was critical to the success of this project that the configuration of the cycloadducts be firmly established. However, analysis of the ^{1}H NMR coupling constants of various isoxazolidines did not result in the unambiguous assignment of configuration because of the limitations of applying the Karplus equation to five-membered ring systems. In order to unambiguously assign the structures of the title compounds, their X-ray crystal structures were determined.



(I) *R* = H
(II) *R* = NO_2

The molecular structures of the title compounds are illustrated in Figs. 1 and 2; in each case the space groups are consistent with the presence of both enantiomers and for ease of comparison the illustrations show molecules with the same chirality at atoms C3(*S*) and C5(*R*). The internal bond angles in the isoxazolidine rings range from 102.7 (2) to 106.9 (2)° in (I) and from 103.3 (2) to 106.1 (2)° in (II); in both cases the smallest angle is N2—C3—C4 and the largest is O1—C5—C4. Corresponding bond lengths are the same within experimental error, except for O1—N2 [1.4809 (18) and 1.453 (3) Å for (I) and (II), respectively] and N2—C3 [1.479 (2) and 1.491 (3) Å, respectively]. Very few comparable isoxazolidine structures are available in the literature; the structures of 2,3-diphenyl-5-fluoromethyl-5-methoxy-4-(*p*-tolylsulfinyl)isoxazolidine (Bravo *et al.*, 1993) and 4-ethoxycarbonyl-5-trifluoromethyl-3-phenylisoxazolidin-5-ol (Bonnet-Delpon *et al.*, 1996) have been reported, but we were unable to reach any useful conclusions from an examination of corresponding bonds and angles.

The ring conformations for the isoxazolidine rings may be defined by the Cremer–Pople puckering parameters (Cremer & Pople, 1975); for compound (I) these are q_2 = 0.376 Å and φ_2 = 20.84° , whilst for (II) they are q_2 = 0.360 Å and φ_2 = 2.97°. These values imply that the puckering in (I) is best described as a half-chair structure twisted on O1—N2, but for (II) the structure approximates an envelope conformation (Spek, 1998).

The most important aspect of these structure determinations, in the context of our chemical studies, is the relative stereochemistry of the substituents in the isoxazolidine ring. In both structures the phenyl group at N2 is in an equatorial position [the angles to the Cremer–Pople plane normals are 65.92 and 60.60° for (I) and (II), respectively], the acetoxy group at C5 is axial (at 18.18 and 12.58°, respectively) whilst the substituent at C3 occupies a bisectional position (at 53.25 and 46.28°, respectively). Finally, the angle between the aromatic planes is 80.95 (6)° in compound (I); in (II) this angle is only 64.85 (8)°, whilst the plane of the nitro group

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is twisted through 7.8 (4)° with respect to its attached phenyl group.

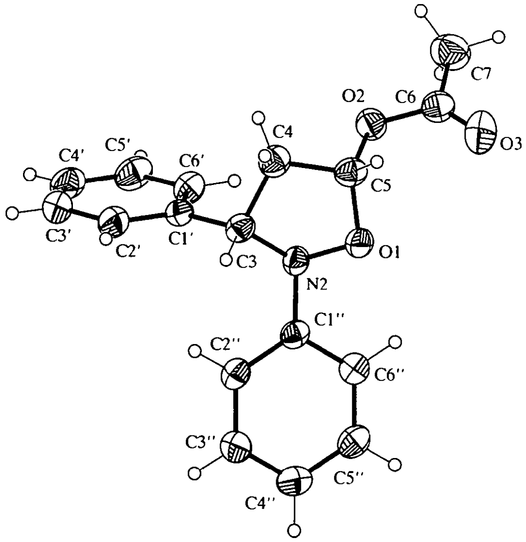


Fig. 1. View of molecule (I) showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms. H atoms are shown as circles of arbitrary radius.

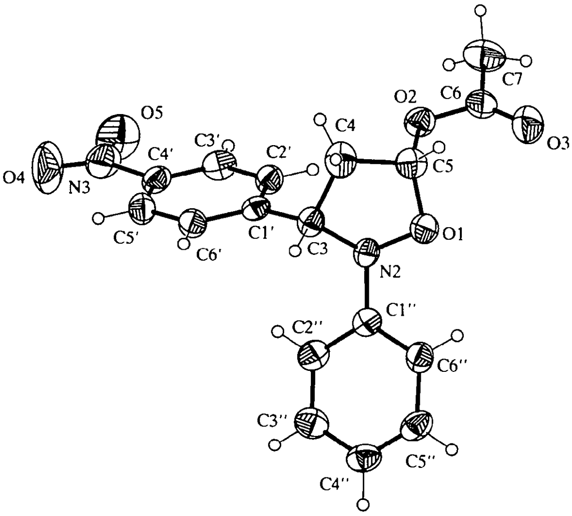
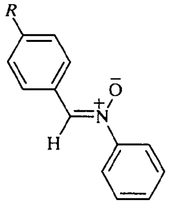


Fig. 2. View of molecule (II) showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms. H atoms are shown as circles of arbitrary radius.

Experimental



(III) $R = H$
(IV) $R = NO_2$

N-α-Diphenylnitrone [(III); 1 g, 5 mmol] and vinyl acetate (0.46 ml, 5 mmol) were dissolved in anhydrous benzene and stirred at 333 K in an oil bath; the reaction was over after 72 h. The benzene was removed under reduced pressure. After flash column chromatography (ethyl acetate–petroleum ether, 1:9) and crystallization from benzene, colourless crystals of (I) were obtained [1.1 g, 76.6% yield; m.p. 377.4 K, literature m.p. 378 K (Cum *et al.*, 1968)].

Compound (II) was obtained *via* a similar reaction involving α-(4-nitrophenyl)-N-phenylnitrone [(IV); 3 g, 12 mmol] and vinyl acetate (1.1 ml, 12 mmol). The reaction was over after 96 h, the reaction mixture was concentrated by removing the excess of benzene. The compound crystallized from benzene as yellow crystals [2.9 g, 71.3% yield; m.p. 405.2 K, literature m.p. 405 K (Cum *et al.*, 1968)].

Compound (I)

Crystal data

$C_{17}H_{17}NO_3$
$M_r = 283.32$
Monoclinic
$P2_1/c$
$a = 11.7637 (13)$ Å
$b = 13.9610 (15)$ Å
$c = 9.6642 (10)$ Å
$\beta = 113.054 (3)°$
$V = 1460.4 (3)$ Å^3
$Z = 4$
$D_x = 1.289$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 3395 reflections
$\theta = 2.38$–$26.00°$
$\mu = 0.089$ mm^{-1}
$T = 180 (2)$ K
Plate
0.28 × 0.22 × 0.08 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.976$, $T_{\max} = 0.993$
7904 measured reflections
2848 independent reflections

1790 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\max} = 26°$
$h = -14 \rightarrow 14$
$k = -10 \rightarrow 17$
$l = -10 \rightarrow 11$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.123$
$S = 0.997$
2848 reflections
191 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °) for (I)*

O1—C5	1.397 (2)	N2—C3	1.479 (2)
O1—N2	1.4809 (18)	C3—C1′	1.515 (3)
O2—C5	1.438 (2)	C3—C4	1.539 (3)
N2—C1″	1.424 (2)	C4—C5	1.513 (3)
C5—O1—N2	104.77 (12)	C5—C4—C3	104.91 (16)
C3—N2—O1	104.29 (14)	O1—C5—C4	106.88 (15)
N2—C3—C4	102.72 (15)		
C5—O1—N2—C3	−42.08 (17)	N2—O1—C5—C4	32.69 (18)
O1—N2—C3—C4	33.01 (17)	C3—C4—C5—O1	−11.7 (2)
N2—C3—C4—C5	−13.67 (19)		

Compound (II)

Crystal data

$C_{17}H_{16}N_2O_5$
$M_r = 328.32$
Monoclinic
Cc
$a = 17.9115\ (13)$ Å
$b = 11.6279\ (13)$ Å
$c = 7.7010\ (7)$ Å
$\beta = 95.842\ (3)^\circ$
$V = 1595.6\ (3)$ Å^3
$Z = 4$
$D_x = 1.367$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 2974 reflections
$\theta = 2.09–28.54^\circ$
$\mu = 0.102$ mm^{-1}
$T = 180\ (2)$ K
Block
0.40 × 0.38 × 0.32 mm
Pale yellow

Data collection

Siemens SMART area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.96$, $T_{\max} = 0.968$
4693 measured reflections
2388 independent reflections

1714 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.055$
$\theta_{\max} = 28.54^\circ$
$h = -12 \rightarrow 23$
$k = -15 \rightarrow 15$
$l = -9 \rightarrow 10$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.098$
$S = 0.912$
2388 reflections
218 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C5	1.397 (3)	N2—C3	1.491 (3)
O1—N2	1.453 (3)	C3—C4	1.541 (4)
O4—N3	1.225 (4)	C4—C5	1.506 (4)
O5—N3	1.233 (4)		
C5—O1—N2	104.9 (2)	N2—C3—C4	103.3 (2)
O1—N2—C3	105.68 (19)	C5—C4—C3	104.5 (2)
O4—N3—O5	124.2 (3)	O1—C5—C4	106.1 (2)
C5—O1—N2—C3	−39.5 (2)	N2—O1—C5—C4	38.0 (2)
O1—N2—C3—C4	24.2 (3)	C3—C4—C5—O1	−22.0 (3)
N2—C3—C4—C5	−1.9 (3)		

The crystal structure of (II) is non-centrosymmetric, but the Flack parameter [1.9 (13); Flack, 1983] was indeterminate and hence the absolute structure could not be determined.

For both compounds, data collection: *SMART* (Siemens, 1994); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structures: *SHELXTL/PC* (Sheldrick, 1994); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

We wish to acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher *et al.*, 1996) for access to the Cambridge Structural Database (Allen & Kennard, 1993).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1330). Services for accessing these data are described at the back of the journal.

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A new polymorph of sulfathiazole

David S. Hughes,[a] Michael B. Hursthouse,[b] Terry Threlfall[c] and Stewart Tavener[c]

[a]*Department of Chemistry, University of Wales Cardiff, PO Box 912, Park Place, Cardiff CF1 3TB, Wales,* [b]*Department of Chemistry, University of Southampton, Highfield, Southampton SO17 1BJ, England, and* [c]*Department of Chemistry, University of York, Heslington, York YO10 5DD, England. E-mail: mbh@soton.ac.uk*

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Abstract

Crystals of sulfathiazole, 4-amino-*N*-thiazol-2-ylidenebenzenesulfonamide, $C_9H_9N_3O_2S_2$, formed from boiling water are shown to be a fifth polymorph. The crystal structure contains two independent molecules, which associate through hydrogen bonds and van der Waals

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