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(2*S**,3*R**,4*S**,5*R**)-3-(*S**-1-Benzyloxyethyl)-4-methyl-4-nitro-5-phenylproline methyl ester

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At 173 K, the five-membered ring of the title compound, $C_{22}H_{26}N_2O_5$, has an envelope conformation. The amine group is involved in both intramolecular and intermolecular hydrogen bonds, the latter linking the molecules into centrosymmetric dimers.

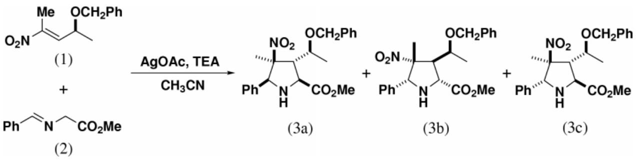
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.142
Data-to-parameter ratio = 17.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (3*c*), is one of three diastereoisomeric products, (3*a*–*c*), obtained from the [3+2]-cycloaddition of the chiral nitroalkene (1) with the silver azomethine ylide derived from imine (2) (Ayerbe *et al.*, 1998). Diastereoisomer (3*a*) could not be isolated; its absolute configuration was assumed by analogy with the compounds obtained in our previous work (Ayerbe *et al.*, 1998). Compound (3*b*) did not give crystals that were suitable for X-ray diffraction. Its relative configuration was determined by NOE experiments. The minor diastereoisomer (3*c*) was successfully crystallized from ethanol and fully characterized.



The puckering parameters (Cremer & Pople, 1975) for the five-membered ring are: q_2 = 0.463 (2) Å and φ_2 = 321.4 (2)°. The latter parameter is close to a value (324°) that is appropriate for an envelope conformation. The envelope flap is formed by C5, which lies 0.699 (3) Å from the plane defined by atoms N1, C2, C3 and C4. This puckering causes significant contraction of the N1—C5—C4 angle (Table 1).

The amine group of the molecule forms long, weak bifurcated hydrogen bonds with the carbonyl-O atom of the ester substituent (Table 2). One interaction is intramolecular and completes a closed five-membered loop with a graph-set motif of $S(5)$ (Bernstein *et al.*, 1995). The second is an intermolecular interaction with a neighbouring molecule, which is related to the original molecule by a centre of inversion. The intermolecular interactions thus link the molecules into hydrogen-bonded dimers which have a graph-set motif of $R_2^2(10)$.

Experimental

The title compound was prepared according to the procedure of Ayerbe *et al.* (1998). The reaction gave a mixture of three diastereoisomers, (3*a*–*c*), in the proportions 59:29:12, which were separated

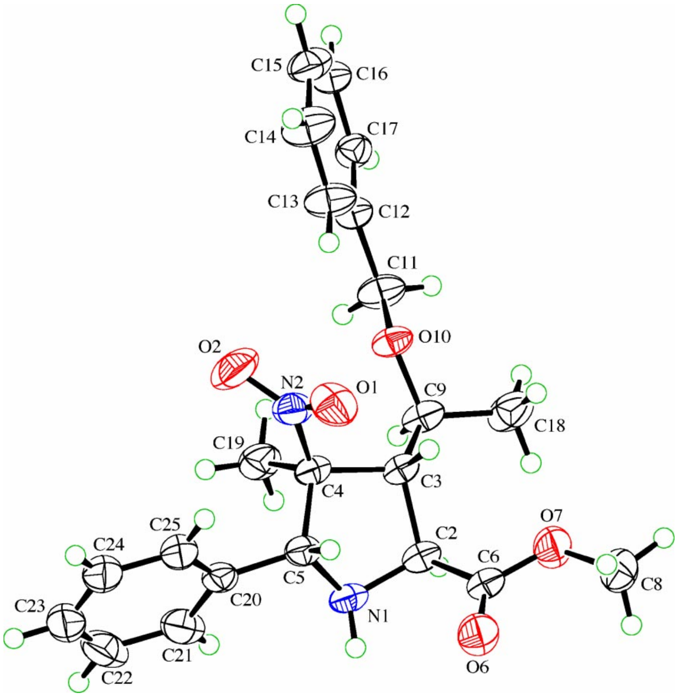


Figure 1
View of the molecule of (3*c*) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

by flash chromatography (ethyl acetate/hexane, 1:10). Suitable crystals were obtained by evaporation of an ethanol solution (m.p. 454–455 K). Spectroscopic analysis: IR (KBr, ν cm^{-1}): 3328, 1722, 1535, 1381; ^{1}H NMR (CDCl$_3$, δ, p.p.m.): 7.31–7.14 (*m*, 10 H), 4.96 (s_b, 1 H), 4.59 (*d*, 1 H, $J = 11.5$ Hz), 4.33 (*d*, 1 H, $J = 11.5$ Hz), 3.81 (*s*, 3 H), 3.71 (*m*, 3 H), 2.52 (*s*, 1 H), 1.30 (*d*, 3 H, $J = 5.3$ Hz), 1.26 (*s*, 3 H); ^{13}C NMR (CDCl$_3$, δ, p.p.m.): 174.8, 137.6, 135.7, 128.6, 128.3, 128.2, 127.6, 127.5, 127.1, 112.3, 94.6, 73.7, 70.6, 70.1, 58.3, 57.0, 52.7, 17.0, 13.7; analysis, calculated for $C_{22}H_{26}N_2O_5$: C 66.30, H 6.59, N 7.03%; found: C 65.99, H 6.56, N 7.08%.

Crystal data

$C_{22}H_{26}N_2O_5$
$M_r = 398.45$
Monoclinic, $P2_1/n$
$a = 9.245$ (2) Å
$b = 8.4082$ (17) Å
$c = 26.6330$ (15) Å
$\beta = 94.802$ (11)°
$V = 2063.1$ (6) Å^3
$Z = 4$
$D_x = 1.283$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 17.0$–$19.5°$
$\mu = 0.09$ mm^{-1}
$T = 173$ (1) K
Prism, colourless
0.50 × 0.32 × 0.22 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω scans
Absorption correction: none
5348 measured reflections
4719 independent reflections
2920 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 10$
$l = -34 \rightarrow 34$
3 standard reflections every 150 reflections intensity decay: none

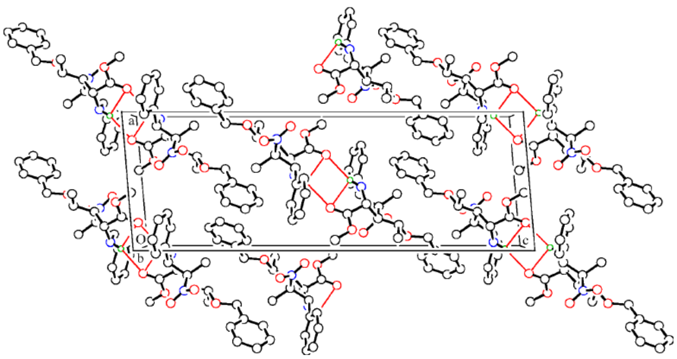


Figure 2
The molecular packing of (3*c*) viewed along the *b* axis. H atoms bonded to C atoms have been omitted for clarity. Red lines indicate the hydrogen bonding interactions.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.142$
$S = 1.06$
4719 reflections
269 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.4415P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.458 (3)	C2—C3	1.563 (2)
N1—C2	1.459 (3)	C3—C4	1.549 (3)
N2—C4	1.513 (3)	C4—C5	1.550 (2)
C5—N1—C2	107.02 (15)	C3—C4—C5	101.94 (14)
N1—C2—C3	105.08 (16)	N1—C5—C4	99.45 (16)
C2—C3—C4	103.10 (15)		
C5—N1—C2—C3	−29.00 (19)	C2—N1—C5—C4	46.76 (17)
N1—C2—C3—C4	−1.20 (18)	C3—C4—C5—N1	−45.43 (17)
C2—C3—C4—C5	28.37 (18)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O6	0.91 (2)	2.46 (2)	2.778 (2)	101.1 (16)
N1—H1$\cdots$O6$^{\rm i}$	0.91 (2)	2.39 (2)	3.278 (2)	164 (2)

Symmetry code: (i) $2 - x, 1 - y, -z$.

All H atoms were initially located in a difference Fourier map. The methyl-H atoms were then constrained to an ideal

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geometry with C—H distances of 0.98 Å and $U_{iso}(H) = 1.5U_{eq}(C)$, but each group was allowed to rotate freely about its C—C bond. The position of the amine-H atom was refined freely along with an isotropic displacement parameter. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.95–1.00 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2001).

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