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(±)-3-Methyl-1,1-diphenyl-2-butyl 3-Acetamidocrotonate and its Hydrogenation Product

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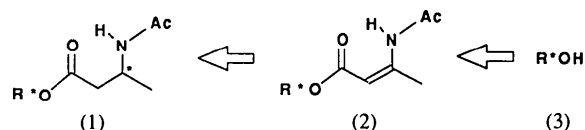
Abstract

Synthesis of (±)-3-methyl-1,1-diphenyl-2-butyl β-acetamidobutanoate, $C_{23}H_{29}NO_3$, (10), with high diastereoisomeric excess was achieved by asymmetric hydrogenation of the stereogenic title compound, $C_{23}H_{27}NO_3$, (9), in which the chirality is present in the ester part. The acetamidocrotonate (9) is planar and delimits a pro-R and a pro-S face. An intramolecular hydrogen bond between the amino group and the carboxylate group is
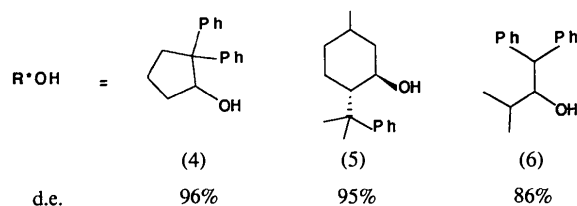
essential to ensure planarity. One of the phenyl rings of the ester hinders one face. The hydrogenation occurs by the opposite face as proved by the crystal structure of its product of hydrogenation, (10).
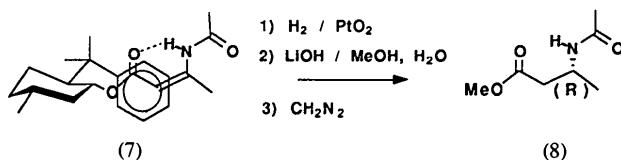
Comment

The asymmetric synthesis of β-acetamidobutyrates (1), which are useful synthons in the preparation of β-lactams, has recently been achieved through the hydrogenation of stereogenic β-acetamidocrotonates (2) (Potin, Dumas & d'Angelo, 1990). With this aim in mind, new simplified chiral auxiliaries (3), bearing only one stereogenic centre, were designed.

The cyclopentanol derivative (4) proved to be a highly potent auxiliary, as efficient as (−)-8-phenylmenthol (5), while the related acyclic compound 1,1-diphenyl-3-methyl-2-butanol, (6), gave, by comparison, a lower but still satisfactory diastereoisomeric excess (d.e.).

The π-facial discrimination obtained in the hydrogenation of the enaminoester (7) derived from (5) may be reasonably rationalized in terms of the π-stacking interaction (d'Angelo & Maddaluno, 1986; Giessner-Prettre, Gresh & Maddaluno, 1992). The sense of the induction of this reaction, determined by chemical correlation to the known R-(+)-3-acetamidobutyrate (8), supports this proposal.

The crystal structure of the acetamidocrotonate (9), derived from the open-chain auxiliary (6), was undertaken to determine its preferred conformation, while that of its product of catalytic hydrogenation, the related β-acetamidobutyrate (10), was undertaken to determine its relative configuration at C3 *versus* C11 and so indicate the preferred π-face of hydrogenation.
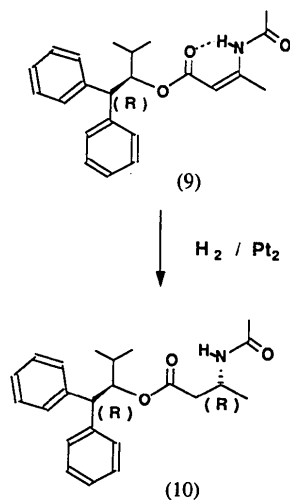
(9)

H_2 / Pt_2

(10)

Fig. 1 shows a view of (9) in which the configuration at C11 has been chosen as R. In this molecule, the acetamidocrotonate group is quite planar. The maximum deviation is less than 0.09 Å (C1 0.007, C2 −0.018, C3 −0.028, C4 −0.054, N5 −0.005, C6 0.019, O7 0.088, C8 −0.035, O9 −0.046, O10 0.072 Å). The intramolecular hydrogen bond between N5 and O9 (N5···O9 2.727, H···O9 1.93 Å, N—H···O 134°), which induces a pseudo six-membered ring (O9—C1—C2—C3—N5—H5), is essential to ensure planarity. This large plane delimits the pro-R and the pro-S faces according to the induced configuration (R or S) at C3 in (10). The crystal structure of (10) (Fig. 1) which exhibits a (C11 R, C3 R) relative configuration confirms that the hydrogenation occurs mainly through the less hindered face. Indeed, as shown in Fig. 2, one of the phenyl rings hinders the pro-S face and hence prevents the hydrogenation being performed by this face.

As shown by the torsion angles, staggered conformations are observed along the single bonds in molecules (9) and (10). The isopropyl group is rotated along the C11—C12 bond in order to minimize the steric con-
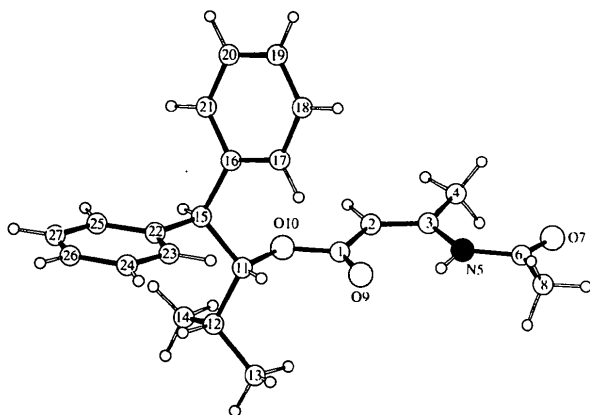

Fig. 1. Perspective view of (9).

straints induced by the phenyl ring; C12—H eclipses the C15—C22 bond. The ester group is planar and the H atom at C11 eclipses the C1—O9 bond in both molecules.

In the crystal of (9), stacking of the crotonate planes of two molecules related by a centre of symmetry, superimposing the atoms C1, C2, C3, N5, C6 and O7 of the first on the atoms O7′, C6′, N5′, C3′, C2′ and C1′ of the second (1−x, −y, −z) with distances of C1···O7′ 3.501 (3), C2···C6′ 3.573 (4) and C3···N5′ 3.613 (3) Å. In the crystal of (10), a hydrogen bond links the H atom at N5 with atom O7 of a neighbouring molecule ($\frac{3}{2}$ − x, y, −$\frac{1}{2}$ + z) [N···O 2.828 (11), H···O 1.94 Å, N—H···O 146°].
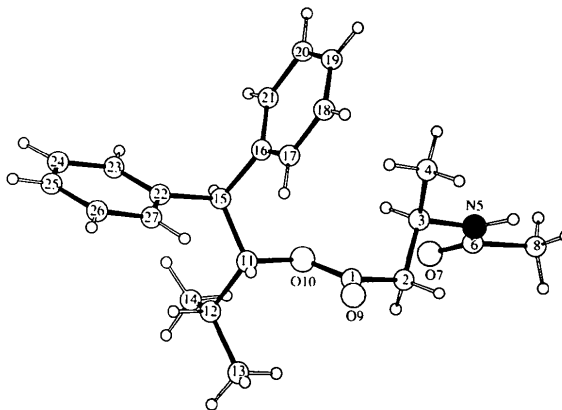

Fig. 2. Perspective view of (10).

Experimental

Compound (9)

Crystal data

$C_{23}H_{27}NO_3$	Cu $K\alpha$ radiation
$M_r = 365.47$	$\lambda = 1.5418$ Å
Monoclinic	Cell parameters from 25
$P2_1/c$	reflections
$a = 11.049$ (4) Å	$\theta = 8.3–21.4°$
$b = 18.659$ (6) Å	$\mu = 0.56$ mm^{-1}
$c = 11.602$ (4) Å	$T = 293$ K
$\beta = 117.08$ (2)°	Prism
$V = 2129.7$ (12) Å^3	$0.40 \times 0.30 \times 0.10$ mm
$Z = 4$	Colourless
$D_x = 1.14$ Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4	$R_{int} = 0.051$
diffractometer	$\theta_{max} = 64.81°$
$\theta/2\theta$ scans	$h = -12 \rightarrow 11$
Absorption correction:	$k = 0 \rightarrow 21$
none	$l = 0 \rightarrow 13$
4367 measured reflections	3 standard reflections
3413 independent reflections	frequency: 120 min
2748 observed reflections	intensity decay: 5%
[$I > 3.0\sigma(I)$]	

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.01$
$R = 0.051$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
$wR = 0.070$	$\Delta\rho_{min} = -0.21$ e Å^{-3}
$S = 0.67$	Extinction correction: none
2742 reflections	Atomic scattering factors
244 parameters	from *International Tables*
H-atom parameters not re-	*for X-ray Crystallogra-*
fined	*phy* (1974, Vol. IV, Table
$w = 1/[\sigma^2(F) + 0.0002F^2]$	2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for compound* (9)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
C1	0.6902 (2)	0.1491 (1)	0.0436 (2)	0.059 (2)
C2	0.6533 (2)	0.0973 (1)	−0.0599 (2)	0.068 (3)
C3	0.6518 (2)	0.0258 (1)	−0.0451 (2)	0.068 (3)
C4	0.6116 (2)	−0.0231 (1)	−0.1593 (3)	0.103 (4)
N5	0.6864 (2)	−0.0029 (1)	0.0760 (2)	0.068 (2)
C6	0.6886 (2)	−0.0736 (1)	0.1114 (3)	0.079 (3)
O7	0.6522 (2)	−0.1222 (1)	0.0347 (2)	0.117 (3)
C8	0.7391 (3)	−0.0845 (1)	0.2535 (3)	0.100 (4)
O9	0.7312 (2)	0.1360 (1)	0.1579 (2)	0.071 (2)
O10	0.6755 (1)	0.2163 (1)	−0.0037 (1)	0.060 (2)
C11	0.7172 (2)	0.2774 (1)	0.0840 (2)	0.055 (2)
C12	0.5918 (2)	0.3168 (1)	0.0724 (2)	0.072 (3)
C13	0.5169 (2)	0.2739 (1)	0.1307 (3)	0.104 (4)
C14	0.4966 (3)	0.3397 (2)	−0.0656 (3)	0.123 (5)
C15	0.8104 (2)	0.3223 (1)	0.0471 (2)	0.056 (2)
C16	0.9344 (2)	0.2816 (1)	0.0558 (2)	0.056 (2)
C17	0.9999 (2)	0.2296 (1)	0.1468 (2)	0.076 (3)
C18	1.1190 (2)	0.1984 (1)	0.1588 (3)	0.084 (3)
C19	1.1745 (2)	0.2185 (1)	0.0804 (3)	0.081 (3)
C20	1.1093 (3)	0.2697 (2)	−0.0122 (3)	0.092 (3)
C21	0.9900 (2)	0.3008 (1)	−0.0253 (2)	0.072 (3)
C22	0.8584 (2)	0.3901 (1)	0.1290 (2)	0.065 (3)
C23	0.8453 (3)	0.4560 (1)	0.0707 (3)	0.106 (4)
C24	0.8943 (4)	0.5172 (1)	0.1443 (5)	0.141 (7)
C25	0.9587 (3)	0.5134 (2)	0.2771 (5)	0.129 (6)
C26	0.9715 (3)	0.4490 (1)	0.3360 (3)	0.101 (4)
C27	0.9219 (2)	0.3877 (1)	0.2620 (3)	0.076 (3)

Table 2. *Geometric parameters* (Å, °) *for compound* (9)

C1—C2	1.449 (3)	C15—C16	1.529 (3)
C1—O9	1.216 (3)	C15—C22	1.524 (3)
C1—O10	1.347 (2)	C16—C17	1.372 (3)
C2—C3	1.346 (3)	C16—C21	1.386 (3)
C3—C4	1.500 (4)	C17—C18	1.387 (4)
C3—N5	1.384 (3)	C18—C19	1.360 (4)
N5—C6	1.378 (3)	C19—C20	1.372 (4)
C6—O7	1.205 (3)	C20—C21	1.384 (4)
C6—C8	1.494 (4)	C22—C23	1.380 (3)
O10—C11	1.456 (2)	C22—C27	1.375 (4)
C11—C12	1.520 (3)	C23—C24	1.380 (4)
C11—C15	1.533 (3)	C24—C25	1.373 (7)
C12—C13	1.514 (4)	C25—C26	1.359 (4)
C12—C14	1.524 (4)	C26—C27	1.385 (4)
C2—C1—O9	126.4 (2)	C11—C15—C22	111.5 (2)
C2—C1—O10	110.3 (2)	C16—C15—C22	108.9 (2)
O9—C1—O10	123.2 (2)	C15—C16—C17	123.1 (2)
C1—C2—C3	124.9 (2)	C15—C16—C21	119.1 (2)
C2—C3—C4	120.4 (2)	C17—C16—C21	117.6 (2)
C2—C3—N5	119.9 (2)	C16—C17—C18	121.2 (2)
C4—C3—N5	119.7 (2)	C17—C18—C19	120.9 (2)
C3—N5—C6	129.2 (2)	C18—C19—C20	118.6 (2)
N5—C6—O7	123.1 (2)	C19—C20—C21	120.8 (3)
N5—C6—C8	114.0 (2)	C16—C21—C20	120.8 (2)
O7—C6—C8	122.9 (2)	C15—C22—C23	120.3 (2)
C1—O10—C11	120.2 (2)	C15—C22—C27	121.9 (2)
O10—C11—C12	109.4 (2)	C23—C22—C27	117.7 (2)
O10—C11—C15	105.8 (1)	C22—C23—C24	120.6 (3)

C12—C11—C15	114.3 (2)	C23—C24—C25	120.6 (4)
C11—C12—C13	111.4 (2)	C24—C25—C26	119.5 (4)
C11—C12—C14	113.2 (2)	C25—C26—C27	119.8 (3)
C13—C12—C14	111.2 (2)	C22—C27—C26	121.7 (2)
C11—C15—C16	113.9 (2)		
O10—C1—C2—C3	−177.2 (3)	O10—C11—C12—C13	70.9 (2)
C1—C2—C3—C4	179.9 (3)	O10—C11—C12—C14	−55.3 (2)
C1—C2—C3—N5	0.4 (2)	O10—C11—C15—C16	−58.2 (2)
C2—C3—N5—C6	178.4 (3)	O10—C11—C15—C22	178.1 (2)
C3—N5—C6—O7	−3.6 (2)	C11—C15—C16—C17	−33.9 (2)
C3—N5—C6—C8	176.8 (3)	C11—C15—C16—C21	150.8 (2)
C2—C1—O10—C11	−175.0 (2)	C11—C15—C22—C23	−129.1 (3)
C1—O10—C11—C12	−108.3 (2)	C11—C15—C22—C27	54.4 (2)
C1—O10—C11—C15	128.2 (2)		

Compound (10)

Crystal data

$C_{23}H_{29}NO_3$	Cu $K\alpha$ radiation
$M_r = 367.49$	$\lambda = 1.5418$ Å
Orthorhombic	Cell parameters from 25
$Pccn$	reflections
$a = 22.41$ (3) Å	$\theta = 6.6$–13.6°
$b = 20.76$ (2) Å	$\mu = 0.56$ mm^{-1}
$c = 9.195$ (6) Å	$T = 293$ K
$V = 4277.8$ (75) Å^3	Prism
$Z = 8$	0.50 × 0.25 × 0.06 mm
$D_x = 1.14$ Mg m^{-3}	Colourless

Data collection

Nonius CAD-4 diffractom-	$\theta_{max} = 65.43°$
eter	$h = 0 \rightarrow 10$
$\theta/2\theta$ scans	$k = 0 \rightarrow 24$
Absorption correction:	$l = 0 \rightarrow 26$
none	3 standard reflections
3651 measured reflections	frequency: 60 min
3651 independent reflections	intensity decay: 10%
1281 observed reflections	
$[I > 3.0\sigma(I)]$	

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.06$
$R = 0.105$	$\Delta\rho_{max} = 0.34$ e Å^{-3}
$wR = 0.145$	$\Delta\rho_{min} = -0.35$ e Å^{-3}
$S = 1.05$	Extinction correction: none
1256 reflections	Atomic scattering factors
244 parameters	from *International Tables*
H-atom parameters not re-	*for X-ray Crystallogra-*
fined	*phy* (1974, Vol. IV, Table
$w = 1/[\sigma^2(F) + 0.0204F^2]$	2.2B)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for compound* (10)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
C1	0.6051 (5)	0.0537 (5)	0.0262 (13)	0.058 (12)
C2	0.6526 (5)	0.1006 (5)	−0.0226 (13)	0.065 (13)
C3	0.7144 (4)	0.0720 (5)	−0.0524 (11)	0.052 (11)
C4	0.7112 (5)	0.0161 (6)	−0.1588 (14)	0.087 (16)
N5	0.7531 (3)	0.1226 (4)	−0.1137 (8)	0.047 (8)
C6	0.7863 (4)	0.1594 (5)	−0.0245 (14)	0.057 (13)
O7	0.7886 (3)	0.1567 (4)	0.1064 (9)	0.077 (10)
C8	0.8249 (4)	0.2085 (5)	−0.1091 (12)	0.058 (12)
O9	0.5589 (3)	0.0473 (4)	−0.0323 (9)	0.083 (10)
O10	0.6229 (2)	0.0226 (3)	0.1475 (7)	0.050 (7)
C11	0.5796 (4)	−0.0203 (4)	0.2182 (10)	0.040 (10)

C12	0.5578 (4)	0.0110 (5)	0.3547 (10)	0.053 (11)
C13	0.5263 (5)	0.0741 (5)	0.3295 (12)	0.074 (14)
C14	0.6067 (5)	0.0180 (6)	0.4705 (12)	0.076 (14)
C15	0.6085 (4)	−0.0865 (4)	0.2326 (9)	0.039 (9)
C16	0.6326 (4)	−0.1133 (4)	0.0899 (11)	0.041 (10)
C17	0.5966 (4)	−0.1162 (5)	−0.0351 (13)	0.052 (11)
C18	0.6184 (5)	−0.1437 (6)	−0.1611 (12)	0.065 (13)
C19	0.6730 (6)	−0.1688 (6)	−0.1677 (14)	0.072 (15)
C20	0.7103 (6)	−0.1684 (6)	−0.0430 (17)	0.084 (17)
C21	0.6886 (5)	−0.1392 (6)	0.0854 (13)	0.070 (14)
C22	0.5692 (4)	−0.1355 (4)	0.3091 (10)	0.042 (10)
C23	0.5914 (4)	−0.1719 (5)	0.4199 (12)	0.054 (12)
C24	0.5560 (6)	−0.2188 (5)	0.4901 (11)	0.067 (14)
C25	0.4985 (6)	−0.2286 (5)	0.4413 (13)	0.067 (14)
C26	0.4760 (5)	−0.1925 (5)	0.3327 (13)	0.064 (14)
C27	0.5105 (5)	−0.1454 (4)	0.2656 (11)	0.054 (12)

Table 4. *Geometric parameters (Å, °) for compound* (10)

C1—C2	1.512 (14)	C15—C16	1.524 (13)
C1—O9	1.173 (13)	C15—C22	1.519 (12)
C1—O10	1.350 (13)	C16—C17	1.406 (14)
C2—C3	1.531 (13)	C16—C21	1.367 (13)
C3—C4	1.518 (16)	C17—C18	1.380 (16)
C3—N5	1.475 (12)	C18—C19	1.332 (17)
N5—C6	1.345 (13)	C19—C20	1.418 (19)
C6—O7	1.207 (15)	C20—C21	1.413 (18)
C6—C8	1.547 (14)	C22—C23	1.362 (13)
O10—C11	1.468 (10)	C22—C27	1.391 (13)
C11—C12	1.495 (13)	C23—C24	1.411 (15)
C11—C15	1.524 (12)	C24—C25	1.380 (19)
C12—C13	1.506 (15)	C25—C26	1.347 (16)
C12—C14	1.535 (15)	C26—C27	1.391 (14)
C2—C1—O9	123.9 (10)	C11—C15—C22	113.5 (7)
C2—C1—O10	110.2 (8)	C16—C15—C22	111.1 (7)
O9—C1—O10	125.8 (10)	C15—C16—C17	121.1 (8)
C1—C2—C3	116.0 (9)	C15—C16—C21	119.7 (8)
C2—C3—C4	111.7 (8)	C17—C16—C21	119.0 (9)
C2—C3—N5	108.8 (8)	C16—C17—C18	120.1 (9)
C4—C3—N5	109.0 (8)	C17—C18—C19	121.7 (11)
C3—N5—C6	119.9 (8)	C18—C19—C20	120.2 (12)
N5—C6—O7	127.3 (10)	C19—C20—C21	118.4 (11)
N5—C6—C8	112.2 (9)	C16—C21—C20	120.7 (10)
O7—C6—C8	120.6 (9)	C15—C22—C23	120.5 (8)
C1—O10—C11	117.5 (7)	C15—C22—C27	120.9 (8)
O10—C11—C12	108.9 (7)	C23—C22—C27	118.6 (9)
O10—C11—C15	107.7 (7)	C22—C23—C24	121.3 (9)
C12—C11—C15	117.2 (7)	C23—C24—C25	118.6 (10)
C11—C12—C13	113.7 (8)	C24—C25—C26	120.6 (11)
C11—C12—C14	113.0 (8)	C25—C26—C27	120.8 (10)
C13—C12—C14	111.0 (8)	C22—C27—C26	120.1 (9)
C11—C15—C16	113.9 (7)		
O10—C1—C2—C3	57.2 (9)	O10—C11—C12—C13	61.3 (8)
C1—C2—C3—C4	53.5 (10)	O10—C11—C12—C14	−66.4 (9)
C1—C2—C3—N5	173.9 (11)	O10—C11—C15—C16	−53.6 (7)
C2—C3—N5—C6	90.7 (10)	O10—C11—C15—C22	178.0 (9)
C3—N5—C6—O7	−0.4 (9)	C11—C15—C16—C17	−51.7 (8)
C3—N5—C6—C8	178.6 (12)	C11—C15—C16—C21	133.1 (11)
C2—C1—O10—C11	174.1 (11)	C11—C15—C22—C23	−130.9 (10)
C1—O10—C11—C12	−106.7 (9)	C11—C15—C22—C27	50.6 (8)
C1—O10—C11—C15	125.3 (9)		

Data collection: Enraf–Nonius CAD-4 software. Data reduction: *NONIUS* (Riche, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *R3M* (Riche, 1983); *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *ACTACIF* (Riche, 1992).

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Un sel de Maprotiline (Ludiomil), Médicament Psychotrope, C$_{20}$H$_{24}$N$^+$.C$_{21}$H$_{22}$NO$_2^-$.0,5H$_2$O

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Abstract

Maprotiline (ludiomil) is a tetracyclic antidepressant drug which crystallized in DMF as the salt [3-(9,10-dihydro-9,10-ethano-9-anthracenyl)propyl]methylammo-