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1-Benzoyl-2(*S*)-*tert*-butyl-4-methoxy-6(*S*)-carbomethoxy-1,2,5,6-tetrahydro-1,3-pyrimidine: a useful precursor of *α*-alkylated aspartic acids

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Key indicators

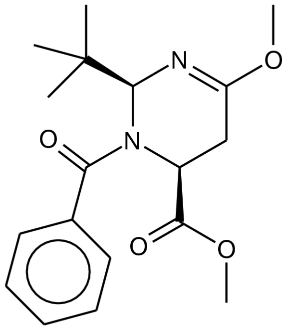
Single-crystal X-ray study
T = 120 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.088
Data-to-parameter ratio = 12.5

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

In the title compound, $C_{18}H_{24}N_2O_4$, the *tert*-butyl and carbomethoxy substituents on the central ring are *syn* with respect to each other. The conformation of the central ring is such that the C atom carrying the carbomethoxy group lies 0.609 (2) Å out of the mean plane of the other five atoms.

Comment

α-Amino acids are receiving increasing attention in view of their interesting chemical and biological properties, both in free forms and as constituents of peptides (Spatola, 1983). The title compound, (I), was prepared to be used as a precursor of *α*-alkylated aspartic acid derivatives. Similar derivatives of *α*-alkylated aspartic acid precursors had been synthesized before (Juaristi *et al.*, 1998). The crystal structure of (I) was determined to prove its successful synthesis.



(I)

The *tert*-butyl group at C1 and the carbomethoxy group at C4 are *syn* with respect to each other. The conformation of the central diazine ring is such that C4 lies 0.609 (2) Å out of the mean plane of the other five atoms, which are coplanar to within a maximum deviation of −0.043 (2) Å (for C1).

Experimental

To a solution of 1-benzoyl-2(*S*)-*tert*-butyl-(6*S*)-carboxyperhydropyrimidin-4-one (Juaristi *et al.*, 1996) (2 g, 6 mmol) was added silver oxide (2.85 g, 12.3 mmol) in 100 ml of dry tetrahydrofuran under argon. The resulting mixture was stirred at ambient temperature for 30 min. Methyl iodide (1.11 ml, 18 mmol) was added, and stirring was continued for 72 h. The reaction mixture was filtered over Celite (eluting with CH_2Cl_2) and concentrated at reduced pressure. The product was purified by flash chromatography, at first eluting with (hexanes/ethyl acetate, 9:1) and gradually increasing the concentration of ethyl acetate (hexanes/ethyl acetate, 4:1) and finally eluting with ethyl acetate and acetic acid (ethyl acetate/acetic acid, 4:1) to give 1.47 g (74.2% yield) as a crystalline solid. Crystals were grown by evaporation from hexanes/ethyl acetate, (9:1).

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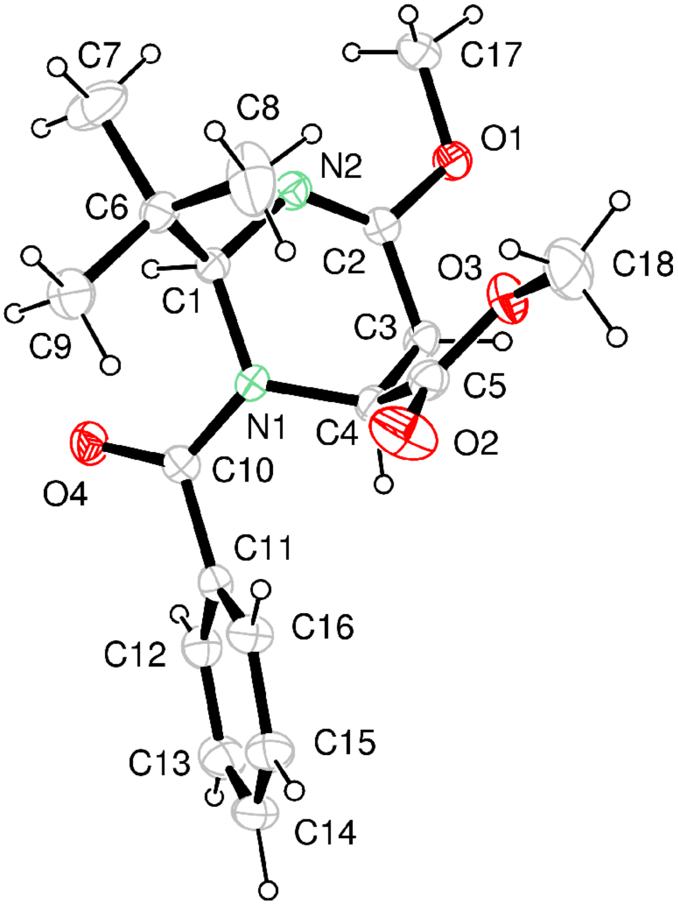

Figure 1
View of the title compound showing atom-numbering scheme with displacement ellipsoids drawn at the 50% probability level.

Crystal data

$C_{18}H_{24}N_2O_4$
$M_r = 332.39$
Monoclinic, $P2_1$
$a = 10.283\ (3)$ Å
$b = 8.124\ (2)$ Å
$c = 11.757\ (4)$ Å
$\beta = 113.673\ (11)^\circ$
$V = 899.5\ (5)$ Å^3
$Z = 2$

$D_x = 1.227$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2391 reflections
$\theta = 2.5$–30.0°
$\mu = 0.09$ mm^{-1}
$T = 120$ K
Fragment, colorless
0.45 × 0.25 × 0.10 mm

Data collection

KappaCCD diffractometer with Oxford Cryostream
ω scans with κ offsets
Absorption correction: none
8506 measured reflections
2780 independent reflections

2368 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\rm max} = 30.0^\circ$
$h = -14 \rightarrow 14$
$k = -9 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.088$
$S = 1.03$
2780 reflections
223 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 0.1725P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.19$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.035 (4)

Table 1
Selected geometric parameters (Å, °).

N1—C10	1.375 (2)	N2—C2	1.262 (2)
N1—C4	1.471 (2)	N2—C1	1.453 (2)
N1—C1	1.497 (2)		
C2—N2—C1—N1	7.0 (2)	N2—C2—C3—C4	−26.2 (3)
C4—N1—C1—N2	20.5 (2)	C1—N1—C4—C3	−48.13 (19)
C1—N2—C2—C3	−3.0 (3)	C2—C3—C4—N1	48.18 (19)
C17—O1—C2—N2	−4.5 (2)		

The absolute configuration could not be determined, but was assumed to correspond to the known configuration of the starting materials. H atoms were placed in calculated positions with C—H bond distances in the range 0.95–1.00 Å and $U_{\rm iso} = 1.2U_{\rm eq}$ of the attached atom (1.5 for methyl groups), and thereafter treated as riding. A torsional parameter was refined for each methyl group. Friedel pairs were averaged.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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