trans-3-Benzoyl-2-*tert*-butyl-4-isobutyl-1,3-oxazolidin-5-one

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Abstract

The oxazolidinone ring adopts a shallow envelope conformation with the *tert*-butyl and isobutyl groups occupying *trans* positions with respect to the ring. The angles at the N atom sum to 356.2°, indicating a very small degree of pyramidalization at this atom. This is consistent with electron delocalization between the N atom and the carbonyl centre [N—C=O = 1.374(3) Å].

Comment

The structure determination of the title compound was undertaken to establish whether an isobutyl group would be formed *cis* or *trans* to the *tert*-butyl group as part of a study of diastereoselectivities of free-radical reactions. Details of the synthetic work will be published elsewhere (Beckwith, Chai & Tozer, 1991).

The *tert*-butyl and isobutyl groups are *trans*, as expected from NMR evidence. The oxazolidinone ring is in an envelope conformation with C5 the out-of-plane atom. The angle between the least-squares plane through atoms O1, C2, C3 and N4 and the plane of N4, C5 and O1 is 10.9(2)°. Intra-annular torsion angles [starting with C5—O1—C2—C3 and proceeding around the ring] are 9.5, −2.3, −5.4, 10.7 and −12.3(3)°. A search of the Jan-
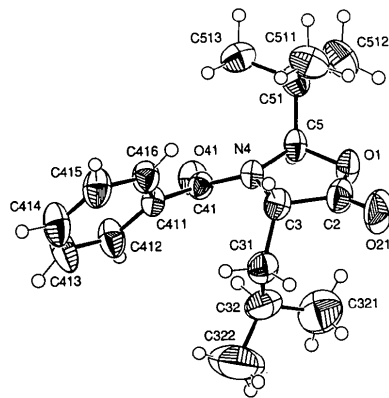
uary 1990 version of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) revealed three structure determinations with 1,3-oxazolidin-5-one fragments (Seebach, Boes, Naef & Schweizer, 1983; Karady, Amato & Weinstock, 1984; Weber, Aeschimann, Maetzke & Seebach, 1986). Bond lengths and angles in these compounds are comparable with those in the present study; the magnitudes of their respective maximum intra-annular torsion angles are 10.2, 1.9 and 10.2°. Diagrams and most calculations were performed with the *Xtal*3.0 package (Hall & Stewart, 1990), as was the generation of the Crystallo-

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å²)

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

	x	*y*	*z*	U_{eq}
O1	0.4154 (4)	0.5699 (1)	0.3026 (1)	0.060 (1)
C2	0.5630 (5)	0.5087 (2)	0.3246 (1)	0.060 (2)
C3	0.5350 (5)	0.4920 (2)	0.3997 (1)	0.048 (1)
N4	0.3570 (3)	0.5558 (1)	0.4167 (1)	0.039 (1)
C5	0.3000 (5)	0.6122 (2)	0.3581 (1)	0.045 (1)
O21	0.6958 (5)	0.4738 (2)	0.2874 (1)	0.090 (2)
C31	0.4869 (6)	0.3929 (2)	0.4143 (2)	0.059 (2)
C32	0.2552 (7)	0.3558 (2)	0.3953 (2)	0.073 (2)
C321	0.209 (1)	0.3542 (4)	0.3211 (3)	0.111 (4)
C322	0.230 (1)	0.2626 (3)	0.4264 (3)	0.149 (5)
C41	0.2034 (4)	0.5476 (2)	0.4682 (1)	0.041 (1)
O41	0.0124 (3)	0.5776 (1)	0.4624 (1)	0.054 (1)
C411	0.2826 (4)	0.5093 (2)	0.5334 (1)	0.043 (1)
C412	0.1315 (5)	0.4613 (2)	0.5725 (1)	0.064 (2)
C413	0.1914 (7)	0.4324 (3)	0.6366 (2)	0.086 (2)
C414	0.3987 (6)	0.4522 (3)	0.6620 (1)	0.078 (2)
C415	0.5494 (6)	0.4997 (2)	0.6241 (1)	0.065 (2)
C416	0.4920 (5)	0.5279 (2)	0.5594 (1)	0.053 (2)
C51	0.3745 (5)	0.7097 (2)	0.3634 (1)	0.052 (2)
C511	0.6299 (6)	0.7166 (3)	0.3669 (2)	0.072 (2)
C512	0.2881 (8)	0.7581 (3)	0.3000 (2)	0.079 (2)
C513	0.2757 (7)	0.7522 (2)	0.4267 (2)	0.068 (2)

Table 2. *Geometric parameters* (Å, °)

O1—C2	1.342 (4)	C41—O41	1.229 (3)
O1—C5	1.439 (3)	C41—C411	1.485 (3)
C2—C3	1.512 (4)	C411—C412	1.386 (4)
C2—O21	1.199 (4)	C411—C416	1.378 (4)
C3—N4	1.465 (3)	C412—C413	1.383 (4)
C3—C31	1.537 (4)	C413—C414	1.366 (5)
N4—C5	1.472 (3)	C414—C415	1.368 (5)
N4—C41	1.374 (3)	C415—C416	1.387 (4)
C5—C51	1.528 (4)	C51—C511	1.527 (5)
C31—C32	1.535 (5)	C51—C512	1.533 (5)
C32—C321	1.490 (7)	C51—C513	1.520 (5)
C32—C322	1.531 (6)		
C2—O1—C5	111.6 (2)	N4—C41—O41	121.0 (2)
O1—C2—C3	110.9 (2)	N4—C41—C411	117.6 (2)
O1—C2—O21	122.2 (3)	O41—C41—C411	121.1 (2)
C3—C2—O21	127.0 (3)	C41—C411—C412	118.4 (2)
C2—C3—N4	101.3 (2)	C41—C411—C416	122.2 (2)
C2—C3—C31	111.3 (2)	C412—C411—C416	119.0 (2)
N4—C3—C31	116.7 (2)	C411—C412—C413	120.3 (3)
C3—N4—C5	111.2 (2)	C412—C413—C414	120.1 (3)
C3—N4—C41	126.4 (2)	C413—C414—C415	120.4 (3)
C5—N4—C41	118.6 (2)	C414—C415—C416	120.0 (3)
O1—C5—N4	103.6 (2)	C411—C416—C415	120.4 (3)
O1—C5—C51	109.4 (2)	C5—C51—C511	110.9 (3)
N4—C5—C51	115.3 (2)	C5—C51—C512	107.3 (3)
C3—C31—C32	118.1 (3)	C5—C51—C513	110.0 (2)
C31—C32—C321	114.5 (4)	C511—C51—C512	109.9 (3)
C31—C32—C322	108.6 (4)	C511—C51—C513	108.7 (3)
C321—C32—C322	111.2 (4)	C512—C51—C513	110.0 (3)

Fig. 1. View of $C_{18}H_{25}NO_3$ showing the labelling of the non-H atoms. Thermal ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radius.

graphic Information File used for the submission of this paper.

Experimental

Crystal data

$C_{18}H_{25}NO_3$	$V = 1759.0\,(3)$ Å^3
$M_r = 303.40$	$Z = 4$
Orthorhombic	$D_x = 1.146$ Mg m^{-3}
$P2_12_12_1$	Cu $K\alpha$
$a = 5.959\,(1)$ Å	$\lambda = 1.5418$ Å
$b = 14.956\,(1)$ Å	$\mu = 0.59$ mm^{-1}
$c = 19.737\,(3)$ Å	$T = 293$ K
Cell parameters from 25 reflections	Prism
$\theta = 25-31°$	0.32 × 0.27 × 0.10 mm
	Colourless

Data collection

Philips PW1100/20 diffractometer	$R_{int} = 0$
$\theta/2\theta$	$\theta_{max} = 61.97°$
Absorption correction:	$h = 0 \rightarrow 6$
SHELX76 Gaussian	$k = -17 \rightarrow 0$
$T_{min} = 0.933$, $T_{max} = 0.824$	$l = 0 \rightarrow 22$
1592 measured reflections	3 standard reflections
1408 observed reflections	frequency: 120 min
$[F > 6.0\sigma(F)]$	intensity variation: 0%

Refinement

Refinement on F	$\Delta\rho_{max} = 0.131$ e Å^{-3}
Final $R = 0.034$	$\Delta\rho_{min} = -0.108$ e Å^{-3}
$wR = 0.042$	Extinction correction:
$S = 1.515$	Zachariasen Gaussian
1408 reflections	coefficient = 3.5(4) × 10^3
272 parameters	Source of atomic scattering
H-atom coordinates refined except for H332B	factors: International Tables for X-ray Crystallography (1974, Vol. IV, Table 2.2B)
$w = 1/[\sigma^2(F) + 0.0004F^2]$	
$(\Delta/\sigma)_{max} = 0.164$	

Data collection: Philips PW1100/20 software 1976. Cell refinement: Xtal3.0 LATCON (Hall & Stewart, 1990). Data reduction: PWREDU (McLaughlin, 1983), Xtal3.0 ADDREF SORTRF (Hall & Stewart, 1990). Program used to solve structure: SHELXS86 (Sheldrick, 1985). Programs used to refine structure: SHELX76 (Sheldrick, 1976), Xtal3.0 CRYLSQ (Hall & Stewart, 1990). Software used to prepare material for publication: Xtal3.0 BONDLA CIFIO (Hall & Stewart, 1990).

The θ-scan width was $(1.0+0.14\tan\theta)°$ with a θ-scan rate of 1.2° min^{-1} and background counts for 5 s on each side of every scan. The absolute configuration has been assigned to agree with the known chirality at C3 arising from its precursor L-leucine. Refinement was by full-matrix least-squares methods.

References

Allen, F. H., Kennard, O. & Taylor, R. (1983). Acc. Chem. Res. 16, 146–153.
Beckwith, A. L. J., Chai, C. L. L. & Tozer, M. J. (1991). In preparation.
Hall, S. R. & Stewart, J. M. (1990). Editors. Xtal3.0 Reference Manual. Univs. of Western Australia, Australia, and Maryland, USA.
Karady, S., Amato, J. S. & Weinstock, L. M. (1984). Tetrahedron Lett. 25, 4337–4340.
McLaughlin, G. M. (1983). PWREDU. Program for data reduction for Philips PW1100/20 diffractometer. Australian National Univ., Canberra, Australia.
Seebach, D., Boes, M., Naef, W. B. & Schweizer, W. B. (1983). J. Am. Chem. Soc. 105, 5390–5398.
Sheldrick, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.
Sheldrick, G. M. (1985). SHELXS86. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
Weber, T., Aeschimann, R., Maetzke, T. & Seebach, D. (1986). Helv. Chim. Acta, 69, 1365–1377.