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5-(*tert*-Butylamino)-5-oxopentanoic acid

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Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.088
Data-to-parameter ratio = 10.5

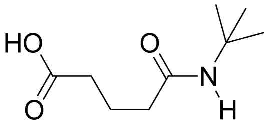
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title novel carboxylic acid derivative, $C_9H_{17}NO_3$, containing the *tert*-butylcarbamoyl group, has been synthesized by the reaction of glutaric anhydride and *tert*-butylamine. In the crystal structure, this compound forms intermolecular hydrogen bonds between the amide and carboxyl groups. However, it does not form a hydrogen-bonded dimer involving two carboxylic acid groups.

Comment

A neighbouring amide, NH, lowers the pK_a value of a carboxylic acid due to formation of an N—H···O hydrogen bond between the amide group and the anionic carboxylate O atom. This hydrogen bond prevents protonation of the carboxylate anion (Onoda *et al.*, 2003, 2004). To date, these effects have been discussed mainly with respect to acylaminobenzoic acid derivatives.

In the present study, we have synthesized a novel amidated carboxylic acid derivative, 5-(*tert*-butylamino)-5-oxopentanoic acid, (I), from glutaric anhydride and *tert*-butylamine. In the crystal structure, the alkyl backbone of (I) displays an extended conformation (Fig. 1). There is no hydrogen-bonded dimer involving the formation of intermolecular O—H···O═C hydrogen bonds between two carboxylic acid groups, as discussed in an early survey (Jeffrey & Sax, 1963). This situation is rare for carboxylic acid structures.



(I)

Generally, amide groups, as in nylon, interact with each other, but in (I) there are no such interactions. There are, however, intermolecular interactions between the carboxylic acid group and the amide group, *viz.* N1—H1···O2^{ii}═C1^{ii} [N1···O2^{ii} 2.968 (2) Å] and O3—H2···O1^{i}═C5^{i} [O3···O1^{i} 2.573 (2) Å] (Fig. 2; symmetry codes as in Table 2). Such interactions have often been reported (*e.g.* Chauhan *et al.*, 1979; Kennard *et al.*, 1982).

Experimental

Glutaric anhydride (10.0 g, 88 mmol) in tetrahydrofuran (50 ml) was added to *tert*-butylamine (10.0 ml, 69 mmol) and the solution was stirred overnight at room temperature. After concentration of the solution, the residue was extracted with ethyl acetate. The organic layer [ethyl acetate containg (I) and an impurity] was washed with 3.5% HCl aqueous solution (30 ml), dried over anhydrous $MgSO_4$

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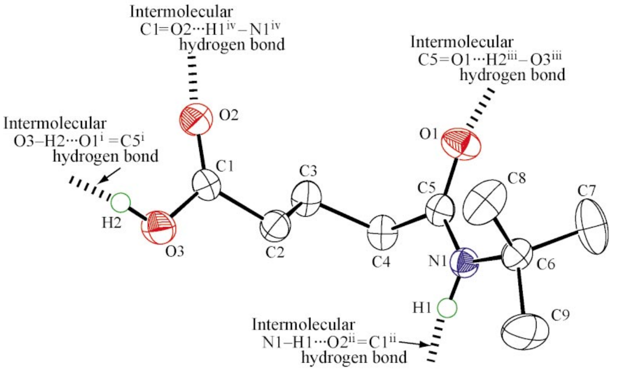

Figure 1
The structure of the title carboxylic acid, (I), with the atom-labelling scheme and with displacement ellipsoids drawn at the 50% probability level. The intermolecular hydrogen bonding is shown by dashed lines [symmetry codes: (i) and (ii) as in Table 2; (iii) $1 - x, y + \frac{1}{2}, 1 - z$; (iv) $x + 1, y, z$].

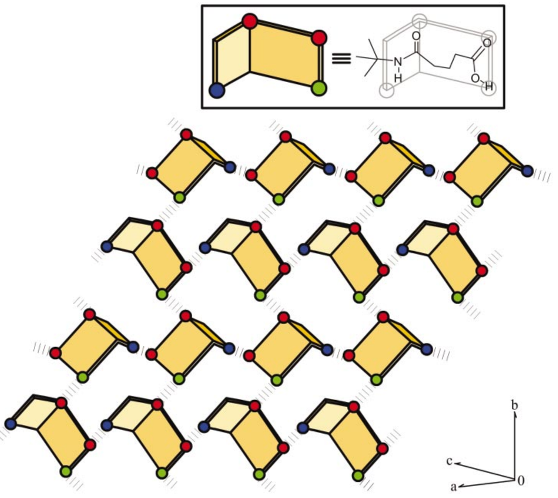

Figure 2
A schematic packing diagram for (I).

and concentrated, to give a white powder. This was recrystallized from ethyl acetate to give colourless block-shaped crystal of (I) (yield 43%). Spectroscopic analysis: ^{1}H NMR (d$_6$-DMSO at 303 K, δ, p.p.m.): 1.22 (9H, *s*), 1.66 (2H, *m*), 2.02 (2H, *t*), 2.17 (2H, *t*), 7.33 (1H, *s*), 12.0 (1H, *s*); analysis calculated for $C_9H_{17}NO_3$: C 57.73, H 9.15, N 7.48%; found: C 57.66, H 9.08, N 7.51%.

Crystal data

$C_9H_{17}NO_3$
$M_r = 187.24$
Monoclinic, $P2_1$
$a = 6.066$ (3) Å
$b = 11.076$ (6) Å
$c = 7.797$ (3) Å
$\beta = 91.56$ (4)°
$V = 523.7$ (4) Å^3
$Z = 2$

$D_x = 1.187$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5001 reflections
$\theta = 2.6$–27.5°
$\mu = 0.09$ mm^{-1}
$T = 200$ K
Block, colourless
0.50 × 0.50 × 0.20 mm

Data collection

Rigaku RAXIS-RAPID imaging-plate diffractometer
ω scans
Absorption correction: none
5164 measured reflections
1246 independent reflections

1128 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 27.5°$
$h = -7 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -10 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.088$
$S = 1.03$
1246 reflections
119 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.247 (2)	N1—C5	1.323 (2)
O2—C1	1.205 (2)	N1—C6	1.484 (2)
O3—C1	1.312 (2)		
C5—N1—C6	127.37 (15)	O1—C5—C4	120.41 (16)
O2—C1—O3	123.13 (16)	N1—C5—C4	116.76 (16)
O2—C1—C2	124.01 (16)	N1—C6—C8	109.92 (16)
O3—C1—C2	112.85 (15)	N1—C6—C7	110.59 (16)
O1—C5—N1	122.78 (17)	N1—C6—C9	105.66 (16)
O2—C1—C2—C3	−35.7 (3)	C3—C4—C5—N1	110.05 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H2···O1^{i}	0.84	1.75	2.573 (2)	165
N1—H1···O2^{ii}	0.88	2.11	2.968 (2)	163

Symmetry codes: (i) $1 - x, y - \frac{1}{2}, 1 - z$; (ii) $x - 1, y, z$.

H atoms were positioned geometrically and treated as riding on their parent atoms, with methyl C—H, methylene C—H, carboxylic O—H and amide N—H distances of 0.98, 0.99, 0.84 and 0.88 Å, respectively. $U_{\rm iso}$(H) values were constrained to be $1.2U_{\rm eq}$ of the carrier atom. Rotating-group refinement was used for the methyl groups and carboxylic acid groups. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *PROCESS-AUTO* (Rigaku Corporation, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2000); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN* and *Mercury* (Bruno *et al.*, 2002); software used to prepare material for publication: *TEXSAN* and *Mercury*.

Support of this work by a Research Fellowship of the 21st century Centre Of Excellence programme 'Integrated EcoChemistry' for Young Scientists (KT, 2002–3) and a Grant-in-Aid for Scientific Research on Priority Areas (No. 12304040) from the Ministry of Education, Science, Sports and Culture, Japan, is gratefully acknowledged.

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