Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-4-(*tert*-Butylamino)-4-oxo-2-butenoic acid

Kazuyuki Takahashi, Taka-aki Okamura, Hitoshi Yamamoto and Norikazu Ueyama*

Department of Macromolecular Science, Graduate School of Science, Osaka University, Toyonaka, Osaka 560-0043, Japan

Correspondence e-mail: ueyama@chem.sci.osaka-u.ac.jp

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.134
Data-to-parameter ratio = 19.6

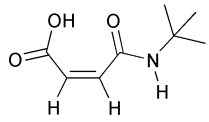
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 February 2004
Accepted 20 February 2004
Online 28 February 2004

The title compound, $C_8H_{13}NO_3$, is an amidated maleic acid derivative. There are intramolecular O—H···O and intermolecular step-like N—H···O hydrogen bonds, forming zigzag chains along the *c* axis.

Comment

On deprotonation, a carboxylic acid having a neighboring amide group may form an intramolecular N—H···O hydrogen bond between the carboxylate O atom and the neighboring amide NH group. Previously, we have synthesized various aliphatic carboxylic acids having the neighboring amide group and reported the crystal structures (Takahashi *et al.*, 2003, 2004). In these carboxylic acids, however, only intermolecular hydrogen bonds are observed. In the present study, we synthesized a novel amidated carboxylic acid derivative, (I), from maleic anhydride and *tert*-butylamine.



(I)

In (I), there is an O3—H2···O1═C4 intramolecular hydrogen bond (Fig. 1, Tables 1 and 2). This intramolecular O—H···O═C hydrogen bond is also observed in a chloroform[D_1] solution using ^{1}H NMR and NOESY spectroscopy (10 m*M*). There is also an intermolecular N1—H1···O2^{i}═C1^{i} [symmetry code: (i) $x, -\frac{1}{2}-y, \frac{1}{2}+z$] hydrogen bond, forming zigzag chains along the *c* axis (Fig. 2). This zigzag-chain hydrogen-bonded network results from the bulky *tert*-butyl group and is very rare in comparison with similar half-amidated maleic acid derivatives, as discussed in previous reports (*e.g.* Allen & Kennard, 1973; Prasad *et al.*, 2002; Lynch & McClenaghan, 2002). There is a hydrophobic layer containing *tert*-butyl groups sandwiched between layers of the hydrogen-bonded chains.

Experimental

To maleic anhydride (10 g, 0.10 mol), *tert*-butylamine (10 ml, 0.095 mol) was added dropwise over an ice–water bath, without direct sunlight, and the mixed solution was stirred at room temperature. After 12 h, 3.5% aqueous HCl solution was added, yielding a precipitate, which was collected by filtration and washed with water. The product was recrystallized from ethyl acetate to give colorless crystals of (I) suitable for X-ray analysis (yield 71%; m.p. 431–434 K). ^{1}H NMR ([D_6]DMSO at 303 K): δ 15.1 (1H, *br*), 8.73 (1H, *s*), 6.31 [1H, ^3J(H,H) = 12.9 Hz, *d*], 6.10 [1H, ^3J(H,H) = 12.9 Hz, *d*], 1.23 (9H, *s*). Elemental analysis calculated for $C_8H_{13}NO_3$: C 56.13, H 7.65, N 8.18%; found: C 55.98, H 7.65, N 8.20%.

Crystal data

$C_8H_{13}NO_3$
M_r = 171.19
Monoclinic, $P2_1/c$
a = 9.283 (4) Å
b = 8.416 (3) Å
c = 12.680 (5) Å
β = 105.42 (3)°
V = 955.0 (7) Å^3
Z = 4
D_x = 1.191 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 14.3–15.0°
μ = 0.09 mm^{-1}
T = 296 K
Prism, colorless
0.50 × 0.20 × 0.20 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.900, T_{max} = 0.962
2325 measured reflections
2195 independent reflections
994 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.5°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 10$
$l = -16 \rightarrow 15$
3 standard reflections
every 150 reflections
intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.134
S = 0.98
2195 reflections
112 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 0.0332P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.248 (2)	N1—C4	1.319 (2)
O2—C1	1.216 (3)	N1—C5	1.486 (3)
O3—C1	1.305 (3)	C2—C3	1.329 (3)
C3—C2—C1	131.6 (2)	C2—C3—C4	128.58 (19)
O2—C1—C2—C3	−167.3 (2)	C1—C2—C3—C4	−2.5 (4)
O3—C1—C2—C3	13.6 (4)	C5—N1—C4—C3	−178.40 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.05	2.897 (3)	168
O3—H2···O1	1.01 (3)	1.46 (3)	2.458 (2)	172 (2)

Symmetry code: (i) $x, -\frac{1}{2} - y, \frac{1}{2} + z$.

Atom H2 of the carboxylic group was located in a difference map and refined freely. Other H atoms were positioned geometrically and were treated as riding on their parent atoms, with methyl and methylene C—H distances and an amide N—H distance of 0.96 Å, 0.93 Å and 0.86 Å, respectively. Rotating-group refinement was used for the methyl groups and carboxylic acid group. U_{iso}(H) values were set to 1.2U_{eq}(carrier atom).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991); cell refinement: *MSC/AFC Diffractometer Control Software*; 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*.

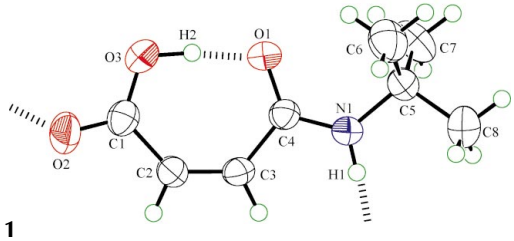


Figure 1
The molecular structure of (I), with the atom-labelling scheme and with displacement ellipsoids drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

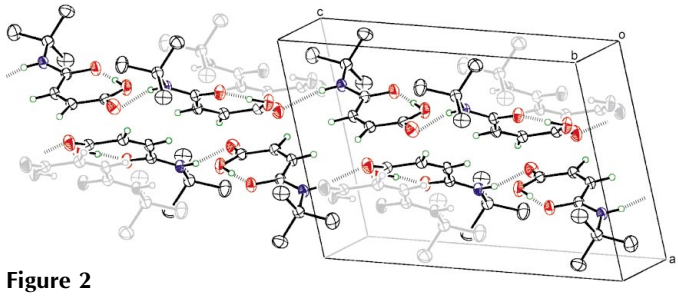


Figure 2
A packing diagram of (I). Dashed lines indicate hydrogen bonds, and the grey molecules are affiliated with other hydrogen-bonded networks.

Support of this work by a Research Fellowship of the 21st Century Center Of Excellence program 'Integrated EcoChemistry' for Young Scientists (K. Takahashi, 2002–2004) and a Grant-in-Aid for Scientific Research on Priority Area (No. 12304040) from the Ministry of Education, Science, Sports and Culture, Japan, is gratefully acknowledged.

References

Allen, F. H. & Kennard, O. (1973). *Cryst. Struct. Commun.* **2**, 145–148.
Altomare, A., Burla, M. C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A. & Polidori, G. (1994). *J. Appl. Cryst.* **27**, 435–436.
Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* B**58**, 389–397.
Lynch, D. E. & McClenaghan, I. (2002). *Acta Cryst.* E**58**, o678–o679.
Molecular Structure Corporation (1991). *MSC/AFC Diffractometer Control Software*. MSC, 9009 New Trails Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (2000). *TEXSAN*. Version 1.11. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Prasad, S. M., Sinha, R. B. P., Mandal, D. K. & Rani, A. (2002). *Acta Cryst.* E**58**, o1296–o1297.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Takahashi, K., Okamura, T., Yamamoto, H. & Ueyama, N. (2003). *Acta Cryst.* E**59**, o1953–o1955.
Takahashi, K., Okamura, T., Yamamoto, H. & Ueyama, N. (2004). *Acta Cryst.* E**60**, o19–o21.