Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Benzimidazolium hydrogen phenylmalonate

Shunsuke Ueda,[a]* Takeo Fukunaga[a] and Hiroyuki Ishida[b]

[a]Graduate School of Natural Science and Technology, Okayama University, Okayama 700-8530, Japan, and [b]Department of Chemistry, Faculty of Science, Okayama University, Okayama 700-8530, Japan

Correspondence e-mail: gsc16102@cc.okayama-u.ac.jp

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.059
wR factor = 0.214
Data-to-parameter ratio = 17.0

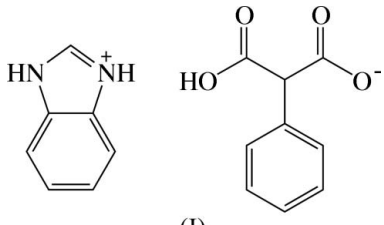
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_7H_7N_2^+\cdot C_9H_7O_4^-$, hydrogen phenylmalonate anions are connected by O—H···O hydrogen bonds to form zigzag chains. The chains are connected to each other by N—H···O hydrogen bonds through benzimidazolium cations, forming a layer.

Received 21 April 2005
Accepted 28 April 2005
Online 21 May 2005

Comment

The title compound, (I), was investigated as part of a structural study on D—H···A hydrogen bonding (D = N, O or C; A = N or O) in carboxylic acid and pyridine systems (Kashino *et al.*, 2001; Ishida *et al.*, 2001, 2002; Fukunaga *et al.*, 2003, 2004). Phenylmalonic acid is an interesting candidate for the selective synthesis and crystallization of optically active substances, because its acidic anion, the hydrogen phenylmalonate ion, is optical active. However, no crystal data of compounds composed of phenylmalonic acid and organic bases are recorded in the Cambridge Structural Database (Version 5.26; Allen, 2002). To our knowledge, this is the first report of a phenylmalonic acid–organic base system.



(I)

In (I), the asymmetric unit is composed of benzimidazolium and hydrogen phenylmalonate ions and an acid–base interaction involving a proton transfer is observed between phenylmalonic acid and benzimidazole (Fig. 1). Anions related by an inversion center are connected by O—H···O hydrogen bonds (O1—H1···O1^{ii} and O3—H3···O3^{iii}; symmetry codes are as in Table 2) and form a zigzag chain running along [110]. In the hydrogen bonds, atoms H1 and H3 are disordered between two positions (Fig. 2). Benzimidazolium cations connect neighboring chains through N1—H1N···O2 and N2—H2N···O4^{i} hydrogen bonds, forming a layer extending parallel to the *ab* plane (Fig. 3). In the layer, a C—H···O hydrogen bond and a C—H···π interaction are observed.

Experimental

Crystals of (I) were grown by slow evaporation at room temperature of a methanol solution of phenylmalonic acid and benzimidazole in a molar ratio of 1:1.

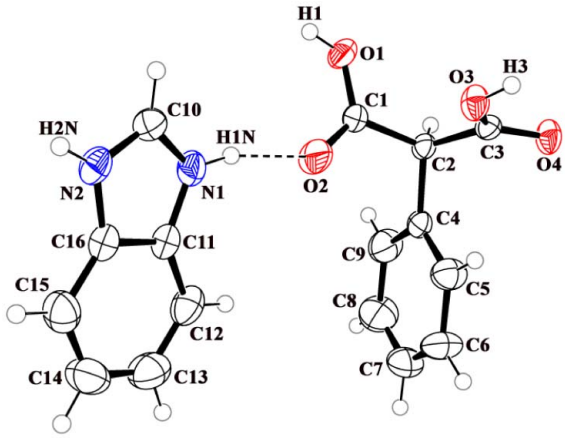


Figure 1
ORTEP-3 (Farrugia, 1997) drawing of the asymmetric unit of (I). Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. The occupancy factors for H1 and H3 are 0.5. The hydrogen bond is indicated by a dashed line.

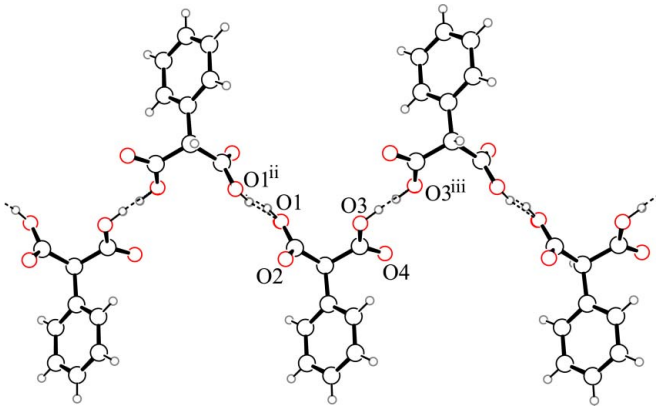


Figure 2
The zigzag chain formed by hydrogen phenylmalonate anions. The H atoms attached to O atoms are disordered between two positions (symmetry codes are as in Table 2). Dashed lines indicate hydrogen bonds.

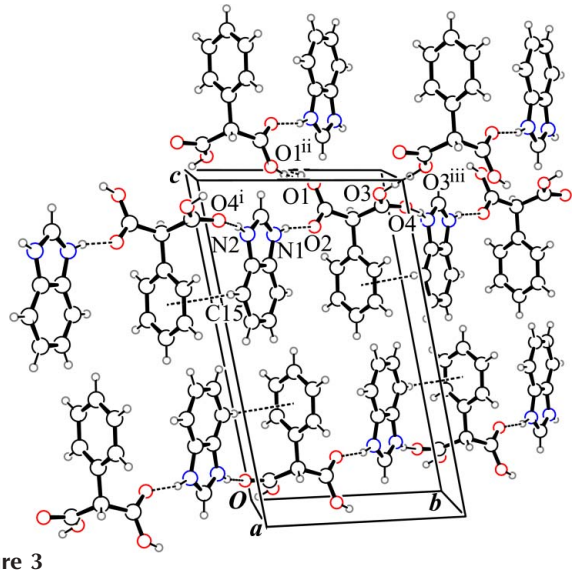


Figure 3
Packing diagram of (I). Dashed lines indicate the N—H···O and O—H···O hydrogen bonds and C—H···π interactions (symmetry codes are as in Table 2).

Crystal data

$C_7H_7N_2^+\cdot C_9H_7O_4^-$
M_r = 298.30
Triclinic, $P\overline{1}$
a = 5.1003 (13) Å
b = 9.244 (3) Å
c = 15.274 (4) Å
α = 102.2 (5)°
β = 90.64 (2)°
γ = 99.87 (2)°
V = 692.6 (14) Å^3
Z = 2
D_x = 1.430 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.5–12.4°
μ = 0.11 mm^{-1}
T = 298 K
Prism, colorless
0.50 × 0.30 × 0.25 mm

Data collection

Rigaku AFC-5R diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.95, T_{max} = 0.97
4300 measured reflections
3657 independent reflections
2059 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 29.0°
$h = -6 \rightarrow 6$
$k = -1 \rightarrow 12$
$l = -20 \rightarrow 20$
3 standard reflections
every 97 reflections
intensity decay: 0.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.214
S = 1.00
3657 reflections
215 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.25P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.280 (4)	N2—C10	1.316 (5)
O2—C1	1.225 (5)	N2—C16	1.383 (4)
O3—C3	1.278 (3)	C1—C2	1.528 (5)
O4—C3	1.237 (3)	C2—C4	1.520 (4)
N1—C10	1.316 (5)	C2—C3	1.533 (6)
N1—C11	1.379 (6)		
C10—N1—C11	108.4 (3)	C4—C2—C3	110.9 (4)
C10—N2—C16	108.1 (4)	C1—C2—C3	110.9 (5)
O2—C1—O1	124.3 (3)	O4—C3—O3	124.6 (3)
O2—C1—C2	120.1 (3)	O4—C3—C2	119.5 (3)
O1—C1—C2	115.6 (4)	O3—C3—C2	115.9 (6)
C4—C2—C1	111.1 (4)	N2—C10—N1	111.0 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2	0.89 (4)	1.78 (4)	2.645 (4)	163 (4)
N2—H2N···O4^{i}	0.87 (4)	1.94 (4)	2.788 (3)	165 (4)
O1—H1···O1^{ii}	0.82 (6)	1.70 (7)	2.504 (3)	167 (8)
O3—H3···O3^{iii}	0.87 (6)	1.63 (6)	2.494 (3)	177 (8)
C10—H10···O3^{iv}	0.93	2.40	2.972 (4)	119
C15—H15···Cg^{i}	0.93	2.92	3.713 (4)	144

Symmetry codes: (i) $1+x, y-1, z$; (ii) $1-x, 1-y, 2-z$; (iii) $2-x, 2-y, 2-z$; (iv) $2-x, 1-y, 2-z$. Note: Cg donates the centroid of the benzene ring of the anion.

H atoms attached to C atoms were treated as riding, with C—H = 0.98 (methine H) or 0.93 Å (aromatic H), and U_{iso}(H) = 1.2U_{eq}(C). H atoms involved in O—H···O and N—H···O hydrogen bonds were refined [N—H = 0.87 (4)–0.89 (4) Å and O—H = 0.82 (6)–0.86 (5) Å]. At the final stage of the least-squares refinement, the occupancy factors of H1 and H3 were fixed at 0.5.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1990); cell refinement: *MSC/AFC*

Diffractometer Control Software; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN for Windows*.

X-ray measurements were made at the X-ray Laboratory of Okayama University. This work was supported by a Grant-in-Aid for Scientific Research (C) (No. 16550014) from the Ministry of Education, Science, Sports and Culture of Japan.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Fukunaga, T., Kashino, S. & Ishida, H. (2003). *Acta Cryst.* E**59**, o420–o422.
Fukunaga, T., Kashino, S. & Ishida, H. (2004). Acat *Cryst.* C**60**, o717–o721.
Ishida, H., Fukunaga, T. & Kashino, S. (2002). *Acta Cryst.* E**58**, o1081–o1082.
Ishida, H., Rahman, B. & Kashino, S. (2001). *Acta Cryst.* C**57**, 876–879.
Kashino, S., Fukunaga, T., Izutsu, H. & Miyashita, S. (2001). *Acta Cryst.* C**57**, 549–552.
Molecular Structure Corporation (1990). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1999). *TEXSAN for Windows*. Version 1.06. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.