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Butane-1,4-diaminium 2-(methoxy-carbonyl)benzoate dihydrate

Jian Li

Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China
Correspondence e-mail: ljwfu@163.com

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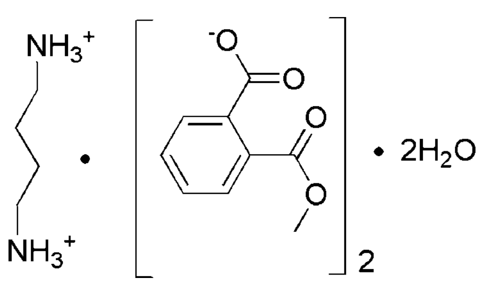
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.123; data-to-parameter ratio = 13.9.

In the title compound, $C_4H_{14}N_2^{+}\cdot 2C_9H_7O_4^{-}\cdot 2H_2O$, the butane-1,4-diaminium cation lies on an inversion center. In the crystal, intermolecular N—H···O and O—H···O hydrogen bonds link the components into layers parallel to (100). Addtional stabilization within these layers is provided by weak intermolecular C—H···O hydrogen bonds.

Related literature

For the appications of phthalimides and *N*-substituted phthalimides, see: Lima *et al.* (2002). For a related structure, see: Liang (2008).



Experimental

Crystal data

$C_4H_{14}N_2^{2+}\cdot 2C_9H_7O_4^{-}\cdot 2H_2O$
M_r = 484.50
Monoclinic, $P2_1/c$
a = 14.0344 (15) Å
b = 8.6746 (9) Å
c = 10.2304 (11) Å
β = 95.620 (1)°
V = 1239.5 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 K
0.50 × 0.48 × 0.47 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.950, T_{max} = 0.953
6001 measured reflections
2178 independent reflections
1601 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.123
S = 1.07
2178 reflections
157 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4^{i}	0.89	1.95	2.815 (2)	164
N1—H1B···O3	0.89	2.00	2.823 (2)	154
N1—H1C···O5	0.89	1.99	2.876 (2)	172
O5—H5C···O3^{ii}	0.85	2.03	2.873 (2)	172
O5—H5D···O4^{iii}	0.85	1.96	2.808 (2)	172
C11—H11A···O2^{i}	0.97	2.46	3.346 (2)	151

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x, y-1, z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5202).

References

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Lima, L. M., Castro, P., Machado, A. L., Frage, C. A. M., Lugniur, C., Moraes, V. L. G. & Barreiro, E. (2002). *J. Bioorg. Med. Chem.* **10**, 3067–3073.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2011). E**67**, o587 [**doi:10.1107/S1600536811003618**]

Butane-1,4-diaminium 2-(methoxycarbonyl)benzoate dihydrate

J. Li

Comment

Phthalimides and N-substituted phthalimides are animportant class of compounds because of their interesting biological activities (Lima *et al.*, 2002). 2-(Methoxycarbonyl)benzoic acid is an intermediate in the preparation of N-substituted phthalimides. In this paper, the structure of the title compound is reported. The asymmetric unit of the title compound (I) contains one half a butane-1,4-diaminium cation, a 2-(methoxycarbonyl)benzoate anion and a solvent water molecule (Fig. 1). The bond lengths and angles agree with those in ethane-1,2-diaminium 2-(methoxycarbonyl)-3,4,5,6-tetrabromobenzoate methanol solvate (Liang, 2008). In the crystal, intermolecular N—H···O and O—H···O hydrogen bonds link the components of the structure into two-dimensional layers parallel to (100) (Fig. 2 and Table 1). Addtional stabilization within these layers is provided by weak intermolecular C—H···O hydrogen bonds.

Experimental

A mixture of phthalic anhydride (1.52 g, 0.01 mol) and methanol (15 ml) was refluxed for 0.5 h. 1,4-Butanediamine (0.44 g, 0.005 mol) was added to the above solution and mixed for 10 min at room temperature. The solution was kept at room temperature for 5 d. Natural evaporation gave colourless single crystals of the title compound, suitable for X-ray analysis.

Refinement

H atoms were initially located in difference maps and then refined in a riding-model approximation with C—H = 0.93–0.97 Å, N—H = 0.89 Å, O—H = 0.82Å and U_{iso}(H) = 1.2U_{eq}(C, O) or 1.5U_{eq}(N, methyl C).

Figures

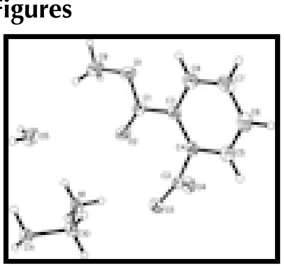

Fig. 1. The asymmetric unit of (I), drawn with 30% probability ellipsoids.

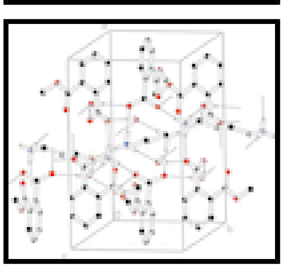

Fig. 2. Part of the crystal structure of the title compound with hydrogen bonds shown as dashed lines. Only H atoms involved in hydrogen bonds are shown.

Butane-1,4-diaminium 2-(methoxycarbonyl)benzoate dihydrate

Crystal data

$C_4H_{14}N_2^{2+}\cdot 2C_9H_7O_4^{-}\cdot 2H_2O$	$F(000) = 516$
$M_r = 484.50$	$D_x = 1.298$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2198 reflections
$a = 14.0344$ (15) Å	$\theta = 2.8$–$27.5°$
$b = 8.6746$ (9) Å	$\mu = 0.10$ mm^{-1}
$c = 10.2304$ (11) Å	$T = 298$ K
$\beta = 95.620$ (1)°	Block, colorless
$V = 1239.5$ (2) Å^3	0.50 × 0.48 × 0.47 mm
$Z = 2$	

Data collection

Bruker SMART CCD diffractometer	2178 independent reflections
Radiation source: fine-focus sealed tube	1601 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.037$
φ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.8°$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$h = -12 \rightarrow 16$
$T_{min} = 0.950$, $T_{max} = 0.953$	$k = -10 \rightarrow 10$
6001 measured reflections	$l = -10 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.123$	$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 0.314P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
2178 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
157 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.118 (8)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.44862 (11)	0.25834 (17)	0.79462 (15)	0.0369 (4)
H1A	0.5061	0.2597	0.7647	0.055*
H1B	0.4252	0.3537	0.7944	0.055*
H1C	0.4094	0.1986	0.7432	0.055*
O1	0.13265 (10)	0.39089 (19)	0.54038 (16)	0.0622 (5)
O2	0.28346 (10)	0.43510 (19)	0.61583 (16)	0.0596 (5)
O3	0.37929 (10)	0.54277 (15)	0.88018 (14)	0.0458 (4)
O4	0.35667 (9)	0.74614 (15)	0.74950 (14)	0.0479 (4)
O5	0.33244 (12)	0.04093 (17)	0.63792 (15)	0.0628 (5)
H5C	0.3453	0.0256	0.5595	0.075*
H5D	0.3382	−0.0443	0.6790	0.075*
C1	0.19982 (14)	0.4525 (2)	0.62583 (19)	0.0376 (5)
C2	0.32751 (13)	0.6373 (2)	0.81441 (18)	0.0342 (4)
C3	0.15919 (12)	0.5400 (2)	0.73169 (18)	0.0351 (5)
C4	0.22064 (13)	0.6245 (2)	0.82091 (18)	0.0344 (5)
C5	0.18168 (15)	0.7007 (2)	0.9231 (2)	0.0490 (6)
H5	0.2216	0.7570	0.9833	0.059*
C6	0.08535 (16)	0.6941 (3)	0.9366 (2)	0.0587 (6)
H6	0.0608	0.7457	1.0056	0.070*
C7	0.02511 (16)	0.6117 (3)	0.8485 (2)	0.0565 (6)
H7	−0.0401	0.6075	0.8579	0.068*
C8	0.06155 (14)	0.5354 (2)	0.7462 (2)	0.0469 (5)
H8	0.0206	0.4804	0.6863	0.056*
C9	0.16701 (18)	0.2992 (4)	0.4371 (3)	0.0783 (9)
H9A	0.2048	0.3625	0.3851	0.117*
H9B	0.1135	0.2580	0.3825	0.117*
H9C	0.2055	0.2161	0.4751	0.117*
C10	0.45726 (13)	0.1967 (2)	0.93045 (17)	0.0337 (4)
H10A	0.3955	0.2014	0.9654	0.040*
H10B	0.5020	0.2592	0.9860	0.040*
C11	0.49175 (14)	0.0324 (2)	0.93138 (17)	0.0353 (5)
H11A	0.5510	0.0274	0.8901	0.042*
H11B	0.4447	−0.0306	0.8801	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0428 (9)	0.0320 (8)	0.0361 (9)	0.0064 (7)	0.0048 (7)	0.0075 (7)
O1	0.0412 (8)	0.0820 (12)	0.0614 (11)	−0.0001 (8)	−0.0048 (7)	−0.0318 (9)
O2	0.0391 (9)	0.0752 (11)	0.0644 (11)	−0.0021 (7)	0.0052 (7)	−0.0322 (9)
O3	0.0441 (8)	0.0439 (8)	0.0481 (9)	0.0115 (6)	−0.0026 (6)	0.0025 (7)
O4	0.0462 (9)	0.0374 (8)	0.0612 (10)	−0.0023 (6)	0.0106 (7)	0.0063 (7)
O5	0.0936 (13)	0.0428 (8)	0.0488 (10)	−0.0037 (8)	−0.0094 (8)	0.0011 (7)
C1	0.0365 (11)	0.0357 (10)	0.0398 (11)	−0.0011 (8)	0.0001 (8)	−0.0013 (8)
C2	0.0391 (10)	0.0279 (9)	0.0351 (10)	0.0004 (8)	0.0008 (8)	−0.0071 (8)
C3	0.0352 (10)	0.0325 (10)	0.0374 (11)	0.0026 (8)	0.0023 (8)	0.0043 (8)
C4	0.0382 (10)	0.0275 (9)	0.0372 (11)	0.0038 (8)	0.0024 (8)	0.0037 (8)
C5	0.0479 (13)	0.0505 (12)	0.0486 (13)	0.0046 (10)	0.0041 (10)	−0.0111 (10)
C6	0.0530 (14)	0.0701 (15)	0.0548 (15)	0.0104 (12)	0.0146 (11)	−0.0149 (12)
C7	0.0386 (12)	0.0694 (15)	0.0634 (15)	0.0079 (11)	0.0144 (10)	−0.0010 (13)
C8	0.0378 (11)	0.0493 (12)	0.0530 (14)	−0.0004 (9)	0.0014 (9)	0.0017 (10)
C9	0.0604 (16)	0.101 (2)	0.0702 (19)	0.0054 (14)	−0.0111 (13)	−0.0481 (16)
C10	0.0425 (11)	0.0302 (9)	0.0286 (10)	0.0033 (8)	0.0044 (8)	0.0020 (8)
C11	0.0457 (11)	0.0307 (9)	0.0295 (10)	0.0041 (8)	0.0035 (8)	0.0007 (8)

Geometric parameters (Å, °)

N1—C10	1.483 (2)	C5—C6	1.373 (3)
N1—H1A	0.8900	C5—H5	0.9300
N1—H1B	0.8900	C6—C7	1.374 (3)
N1—H1C	0.8900	C6—H6	0.9300
O1—C1	1.333 (2)	C7—C8	1.378 (3)
O1—C9	1.443 (3)	C7—H7	0.9300
O2—C1	1.198 (2)	C8—H8	0.9300
O3—C2	1.248 (2)	C9—H9A	0.9600
O4—C2	1.246 (2)	C9—H9B	0.9600
O5—H5C	0.8500	C9—H9C	0.9600
O5—H5D	0.8500	C10—C11	1.505 (2)
C1—C3	1.481 (3)	C10—H10A	0.9700
C2—C4	1.512 (3)	C10—H10B	0.9700
C3—C8	1.393 (3)	C11—C11^{i}	1.509 (3)
C3—C4	1.400 (3)	C11—H11A	0.9700
C4—C5	1.393 (3)	C11—H11B	0.9700
C10—N1—H1A	109.5	C7—C6—H6	119.9
C10—N1—H1B	109.5	C6—C7—C8	119.8 (2)
H1A—N1—H1B	109.5	C6—C7—H7	120.1
C10—N1—H1C	109.5	C8—C7—H7	120.1
H1A—N1—H1C	109.5	C7—C8—C3	120.6 (2)
H1B—N1—H1C	109.5	C7—C8—H8	119.7
C1—O1—C9	115.83 (17)	C3—C8—H8	119.7
H5C—O5—H5D	108.2	O1—C9—H9A	109.5

O2—C1—O1	121.97 (18)	O1—C9—H9B	109.5
O2—C1—C3	125.28 (17)	H9A—C9—H9B	109.5
O1—C1—C3	112.75 (16)	O1—C9—H9C	109.5
O4—C2—O3	125.51 (18)	H9A—C9—H9C	109.5
O4—C2—C4	117.28 (16)	H9B—C9—H9C	109.5
O3—C2—C4	117.10 (16)	N1—C10—C11	110.12 (14)
C8—C3—C4	119.66 (18)	N1—C10—H10A	109.6
C8—C3—C1	121.09 (17)	C11—C10—H10A	109.6
C4—C3—C1	119.22 (16)	N1—C10—H10B	109.6
C5—C4—C3	118.40 (17)	C11—C10—H10B	109.6
C5—C4—C2	117.51 (17)	H10A—C10—H10B	108.2
C3—C4—C2	124.08 (16)	C10—C11—C11^{i}	112.22 (19)
C6—C5—C4	121.2 (2)	C10—C11—H11A	109.2
C6—C5—H5	119.4	C11^{i}—C11—H11A	109.2
C4—C5—H5	119.4	C10—C11—H11B	109.2
C5—C6—C7	120.3 (2)	C11^{i}—C11—H11B	109.2
C5—C6—H6	119.9	H11A—C11—H11B	107.9

C9—O1—C1—O2	1.4 (3)	O3—C2—C4—C5	86.2 (2)
C9—O1—C1—C3	−177.8 (2)	O4—C2—C4—C3	90.4 (2)
O2—C1—C3—C8	−170.6 (2)	O3—C2—C4—C3	−93.2 (2)
O1—C1—C3—C8	8.6 (3)	C3—C4—C5—C6	−0.2 (3)
O2—C1—C3—C4	7.4 (3)	C2—C4—C5—C6	−179.6 (2)
O1—C1—C3—C4	−173.44 (17)	C4—C5—C6—C7	−0.1 (4)
C8—C3—C4—C5	0.7 (3)	C5—C6—C7—C8	0.0 (4)
C1—C3—C4—C5	−177.31 (17)	C6—C7—C8—C3	0.5 (3)
C8—C3—C4—C2	−179.96 (17)	C4—C3—C8—C7	−0.8 (3)
C1—C3—C4—C2	2.1 (3)	C1—C3—C8—C7	177.12 (19)
O4—C2—C4—C5	−90.2 (2)	N1—C10—C11—C11^{i}	176.08 (19)

Symmetry codes: (i) $-x+1, -y, -z+2$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O4^{ii}	0.89	1.95	2.815 (2)	164
N1—H1B···O3	0.89	2.00	2.823 (2)	154
N1—H1C···O5	0.89	1.99	2.876 (2)	172
O5—H5C···O3^{iii}	0.85	2.03	2.873 (2)	172
O5—H5D···O4^{iv}	0.85	1.96	2.808 (2)	172
C11—H11A···O2^{ii}	0.97	2.46	3.346 (2)	151

Symmetry codes: (ii) $-x+1, y-1/2, -z+3/2$; (iii) $x, -y+1/2, z-1/2$; (iv) $x, y-1, z$.

Fig. 1

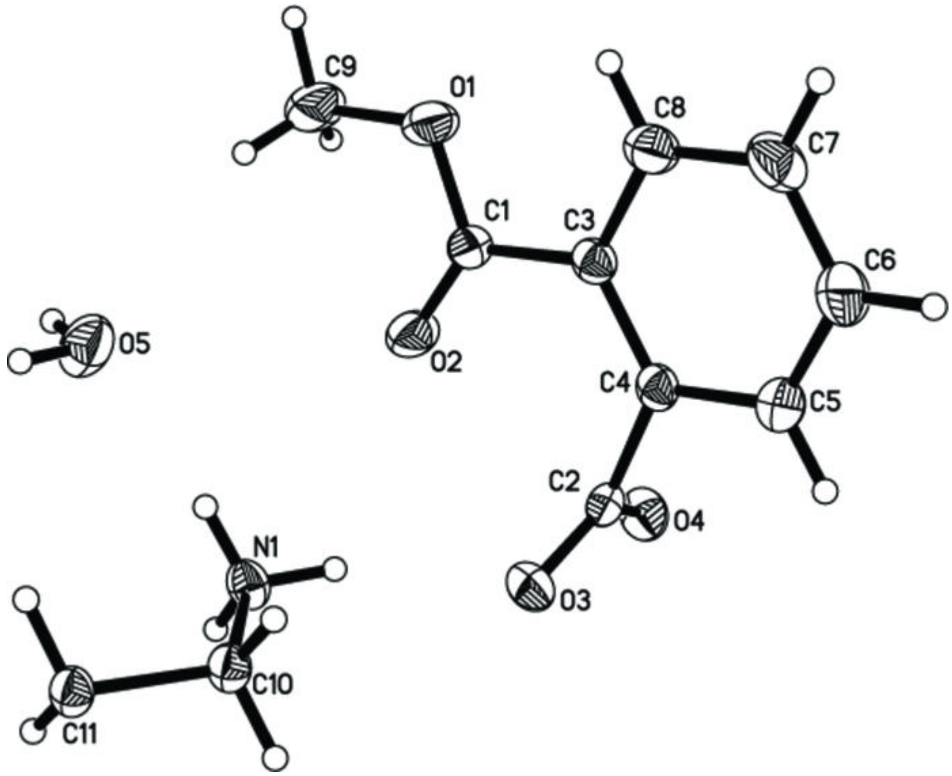

Fig. 2