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4-Bromoanilinium hydrogen phthalate

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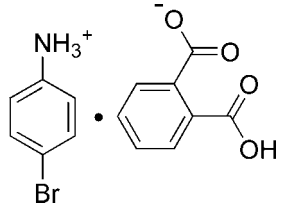
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.011 Å; R factor = 0.054; wR factor = 0.127; data-to-parameter ratio = 12.7.

In the anion of the title compound, $C_6H_7BrN^+\cdot C_8H_5O_4^-$, the dihedral angles formed by the benzene ring and the mean planes of the –COOH and –COO^- groups are 20.6 (3) and 83.2 (3)°, respectively. In the crystal, intermolecular N—H···O and O—H···O hydrogen bonds connect the cations and anions, forming a two-dimensional network parallel to (001).

Related literature

For applications of phthalimides and *N*-substituted phthalimides, see: Lima *et al.* (2002). For the crystal structures of 4-chloroanilinium, 2-hydroxyanilinium and 3-hydroxyanilinium hydrogen phthalates, see: Jagan & Sivakumar (2009).



Experimental

Crystal data

$C_6H_7BrN^+\cdot C_8H_5O_4^-$
M_r = 338.16
Monoclinic, $C2$
a = 13.0890 (14) Å
b = 7.6670 (7) Å
c = 14.6900 (14) Å
β = 106.671 (1)°
V = 1412.2 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.92 mm^{-1}
T = 298 K
0.41 × 0.37 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.380, T_{max} = 0.621
3555 measured reflections
2364 independent reflections
1659 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.127
S = 0.94
2364 reflections
186 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}
Absolute structure: Flack (1983), 1027 Friedel pairs
Flack parameter: 0.012 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3^{i}	0.80 (6)	1.76 (6)	2.518 (6)	158 (7)
N1—H1C···O4	0.89	2.37	2.962 (7)	125
N1—H1C···O1^{ii}	0.89	2.13	2.916 (7)	147
N1—H1B···O4^{iii}	0.89	1.96	2.804 (7)	159
N1—H1A···O3^{iv}	0.89	1.96	2.828 (6)	164

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

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: LH5245).

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supporting information

Acta Cryst. (2011). E**67**, o1430 [doi:10.1107/S1600536811017727]

4-Bromoanilinium hydrogen phthalate

Zu Pei Liang

S1. Comment

Phthalimides and N-substituted phthalimides are animportant class of compounds because of their interesting biological activities (Lima *et al.*, 2002). 4-Bromoanilinium hydrogen phthalate is an intermediate in the preparation of N-substituted phthalimides. The crystal structures of 4-chloroanilinium, 2-hydroxyanilinium and 3-hydroxyanilinium hydrogen phthalates have already been reported (Jagan & Sivakumar, 2009). In this paper, the structure of the title compound is reported. The asymmetric unit of the title compound (I) contains one 4-bromoanilinium cation and one hydrogen phthalate anion (Fig. 1). The dihedral angles formed by the benzene ring and the mean planes of the —COOH and —COO^- groups are 20.6 (3) and 83.2 (3) °, respectively. In the crystal, intermolecular N—H···O and O—H···O hydrogen bonds connect cations and anions to form a two-dimensional network parallel to (001) (Fig. 2).

S2. Experimental

A mixture of phthalic anhydride (1.52 g, 0.01 mol) and 4-bromoaniline (1.72 g, 0.01 mol) in 20 ml ethanol(95%) solution was refluxed for 0.5 h. The solution was kept at room temperature for 7 d. Natural evaporation gave colourless single crystals of the title compound, suitable for X-ray analysis.

S3. Refinement

H atoms bonded to C and N were initially located in difference maps and then refined in a riding-model approximation with C—H = 0.93 Å and N—H = 0.89 Å and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(N). The H atom bonded to O was refined independently with an isotropic displacment parameter.

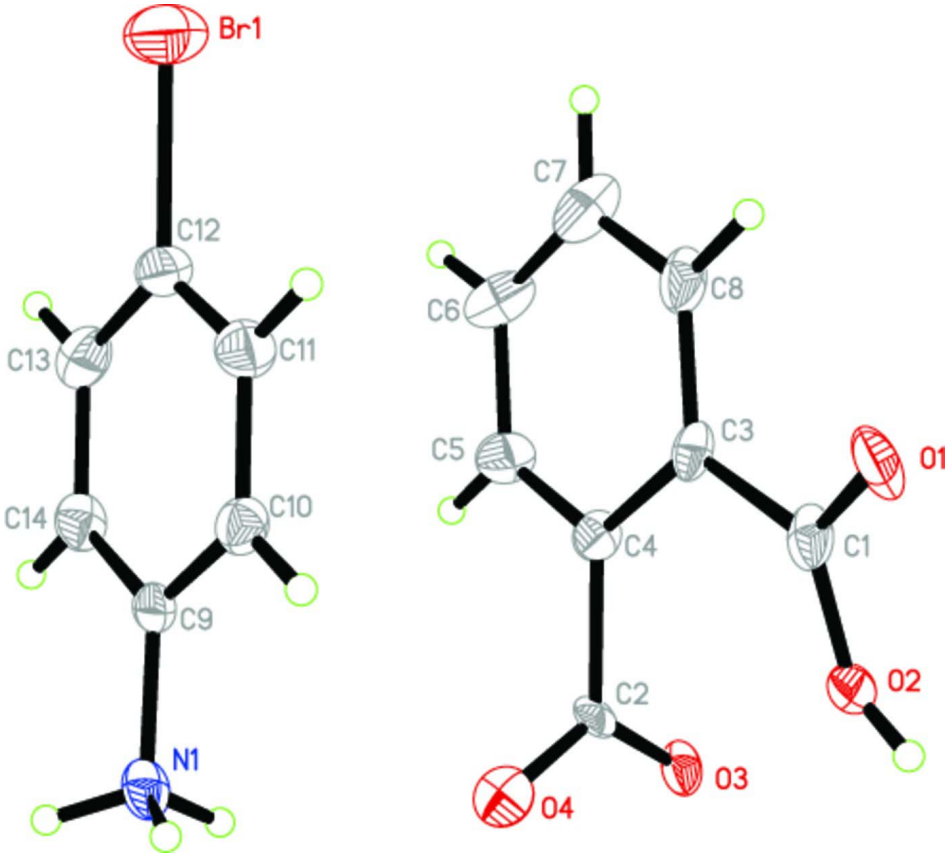

Figure 1

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

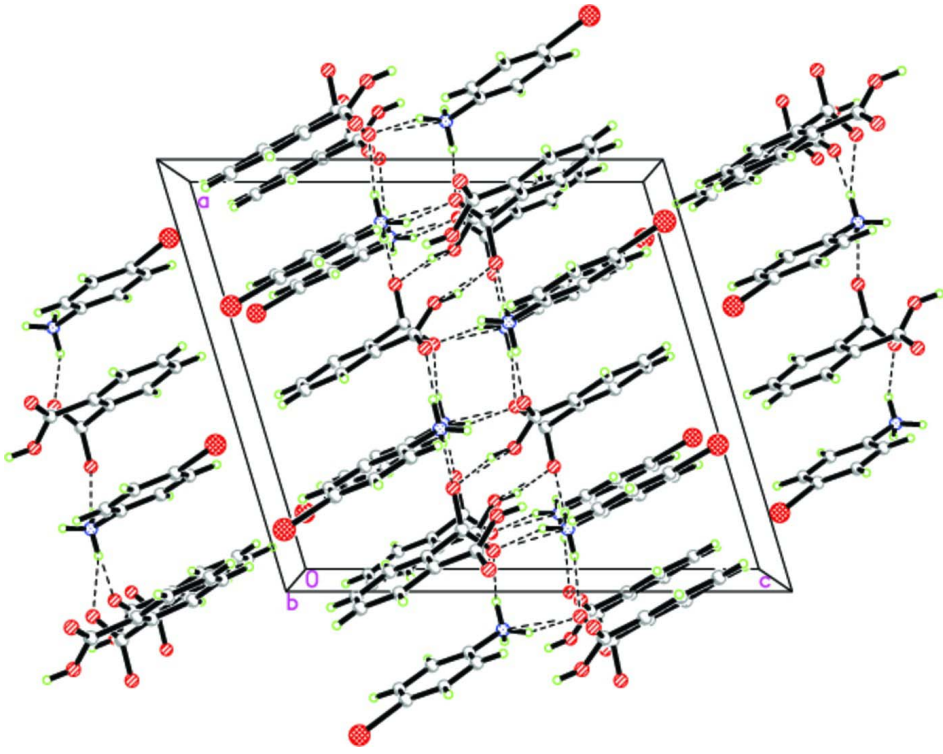

Figure 2

Part of the crystal structure of the title compound with hydrogen bonds shown as dashed lines.

4-Bromoanilinium 2-carboxybenzoate

Crystal data

$C_6H_7BrN^+·C_8H_5O_4^-$
M_r = 338.16
Monoclinic, *C*2
Hall symbol: C 2y
a = 13.0890 (14) Å
b = 7.6670 (7) Å
c = 14.6900 (14) Å
β = 106.671 (1)°
V = 1412.2 (2) Å^3
Z = 4
F(000) = 680
D_x = 1.590 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1173 reflections
θ = 2.9–20.8°
μ = 2.92 mm^{-1}
T = 298 K
Block, colorless
0.41 × 0.37 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.380, T_{max} = 0.621
3555 measured reflections
2364 independent reflections
1659 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 25.0°, θ_{min} = 2.9°
h = −13→15
k = −8→9
l = −17→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.127$
$S = 0.94$
2364 reflections
186 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.82$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ e Å^{-3}
Absolute structure: Flack (1983), 1027 Friedel pairs
Absolute structure parameter: 0.012 (16)

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}
Br1	0.16160 (6)	0.43398 (15)	0.05674 (5)	0.0677 (3)
N1	0.3761 (4)	−0.0249 (6)	0.4004 (3)	0.0329 (13)
H1A	0.3407	−0.1253	0.3952	0.049*
H1B	0.3731	0.0303	0.4528	0.049*
H1C	0.4438	−0.0462	0.4035	0.049*
O1	0.5651 (4)	0.7623 (6)	0.4163 (4)	0.0553 (16)
O2	0.6767 (3)	0.5378 (6)	0.4617 (4)	0.0377 (12)
H2	0.699 (4)	0.569 (9)	0.516 (4)	0.03 (2)*
O3	0.7303 (3)	0.1975 (5)	0.3875 (3)	0.0324 (10)
O4	0.5788 (4)	0.1691 (7)	0.4309 (4)	0.0385 (12)
C1	0.5990 (5)	0.6178 (9)	0.4009 (6)	0.0345 (18)
C2	0.6314 (5)	0.2287 (7)	0.3804 (5)	0.0255 (15)
C3	0.5581 (5)	0.5282 (9)	0.3075 (6)	0.0307 (17)
C4	0.5732 (5)	0.3475 (9)	0.2962 (5)	0.0297 (17)
C5	0.5343 (5)	0.2691 (11)	0.2082 (5)	0.044 (2)
H5	0.5446	0.1504	0.2013	0.053*
C6	0.4791 (6)	0.3685 (13)	0.1293 (6)	0.056 (3)
H6	0.4520	0.3151	0.0704	0.067*
C7	0.4648 (6)	0.5461 (13)	0.1385 (7)	0.056 (2)
H7	0.4297	0.6127	0.0859	0.067*
C8	0.5031 (5)	0.6231 (10)	0.2269 (6)	0.043 (2)
H8	0.4920	0.7418	0.2329	0.052*
C9	0.3276 (4)	0.0844 (8)	0.3171 (4)	0.0305 (15)

C10	0.3175 (5)	0.2651 (8)	0.3301 (5)	0.0342 (16)
H10	0.3433	0.3149	0.3901	0.041*
C11	0.2688 (4)	0.3668 (9)	0.2525 (5)	0.0403 (17)
H11	0.2614	0.4862	0.2598	0.048*
C12	0.2314 (5)	0.2920 (9)	0.1649 (5)	0.0378 (17)
C13	0.2417 (5)	0.1129 (9)	0.1509 (5)	0.0425 (17)
H13	0.2166	0.0638	0.0908	0.051*
C14	0.2908 (4)	0.0107 (8)	0.2296 (5)	0.0356 (16)
H14	0.2983	−0.1086	0.2223	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0717 (5)	0.0667 (6)	0.0567 (5)	0.0124 (6)	0.0056 (3)	0.0159 (6)
N1	0.033 (2)	0.020 (3)	0.047 (3)	−0.002 (2)	0.013 (2)	−0.003 (2)
O1	0.052 (3)	0.027 (3)	0.082 (4)	0.015 (2)	0.011 (3)	−0.012 (3)
O2	0.035 (2)	0.024 (2)	0.045 (3)	0.0035 (19)	−0.002 (2)	−0.009 (2)
O3	0.025 (2)	0.021 (2)	0.050 (3)	0.0016 (17)	0.0098 (19)	0.001 (2)
O4	0.033 (2)	0.040 (3)	0.043 (3)	−0.007 (2)	0.013 (2)	0.004 (2)
C1	0.019 (3)	0.030 (4)	0.055 (5)	0.002 (3)	0.011 (3)	0.005 (3)
C2	0.029 (4)	0.011 (3)	0.036 (4)	−0.002 (2)	0.008 (3)	−0.007 (2)
C3	0.016 (3)	0.036 (4)	0.040 (4)	0.004 (3)	0.007 (3)	0.007 (3)
C4	0.032 (4)	0.022 (4)	0.034 (5)	−0.004 (3)	0.006 (3)	0.007 (3)
C5	0.043 (4)	0.043 (5)	0.044 (5)	−0.007 (3)	0.009 (4)	−0.004 (4)
C6	0.043 (4)	0.074 (7)	0.044 (5)	−0.013 (4)	0.001 (4)	−0.002 (4)
C7	0.039 (4)	0.066 (7)	0.054 (6)	−0.008 (4)	−0.001 (4)	0.017 (5)
C8	0.033 (4)	0.027 (4)	0.063 (6)	0.003 (3)	0.003 (4)	0.014 (4)
C9	0.021 (3)	0.035 (4)	0.037 (4)	−0.003 (3)	0.010 (3)	0.008 (3)
C10	0.032 (3)	0.030 (4)	0.038 (4)	−0.002 (3)	0.005 (3)	−0.004 (3)
C11	0.036 (3)	0.034 (4)	0.050 (5)	0.005 (3)	0.012 (3)	0.005 (3)
C12	0.033 (3)	0.041 (5)	0.038 (4)	0.003 (3)	0.007 (3)	0.008 (3)
C13	0.036 (4)	0.052 (5)	0.038 (4)	−0.004 (3)	0.008 (3)	−0.007 (3)
C14	0.037 (3)	0.024 (4)	0.045 (4)	0.000 (3)	0.012 (3)	−0.006 (3)

Geometric parameters (Å, º)

Br1—C12	1.927 (6)	C5—H5	0.9300
N1—C9	1.468 (8)	C6—C7	1.386 (12)
N1—H1A	0.8900	C6—H6	0.9300
N1—H1B	0.8900	C7—C8	1.382 (11)
N1—H1C	0.8900	C7—H7	0.9300
O1—C1	1.238 (8)	C8—H8	0.9300
O2—C1	1.299 (8)	C9—C14	1.359 (8)
O2—H2	0.80 (6)	C9—C10	1.410 (9)
O3—C2	1.292 (7)	C10—C11	1.377 (9)
O4—C2	1.236 (8)	C10—H10	0.9300
C1—C3	1.490 (11)	C11—C12	1.365 (9)
C2—C4	1.548 (9)	C11—H11	0.9300

C3—C8	1.400 (10)	C12—C13	1.400 (10)
C3—C4	1.415 (8)	C13—C14	1.392 (9)
C4—C5	1.385 (11)	C13—H13	0.9300
C5—C6	1.402 (12)	C14—H14	0.9300
C9—N1—H1A	109.5	C8—C7—C6	119.2 (9)
C9—N1—H1B	109.5	C8—C7—H7	120.4
H1A—N1—H1B	109.5	C6—C7—H7	120.4
C9—N1—H1C	109.5	C7—C8—C3	122.2 (8)
H1A—N1—H1C	109.5	C7—C8—H8	118.9
H1B—N1—H1C	109.5	C3—C8—H8	118.9
C1—O2—H2	122 (5)	C14—C9—C10	121.0 (6)
O1—C1—O2	123.2 (7)	C14—C9—N1	120.1 (6)
O1—C1—C3	121.9 (7)	C10—C9—N1	118.9 (6)
O2—C1—C3	114.8 (6)	C11—C10—C9	118.9 (6)
O4—C2—O3	127.0 (6)	C11—C10—H10	120.5
O4—C2—C4	117.8 (6)	C9—C10—H10	120.5
O3—C2—C4	115.2 (5)	C12—C11—C10	119.9 (6)
C8—C3—C4	117.7 (8)	C12—C11—H11	120.1
C8—C3—C1	120.1 (7)	C10—C11—H11	120.1
C4—C3—C1	122.2 (7)	C11—C12—C13	121.8 (6)
C5—C4—C3	120.5 (8)	C11—C12—Br1	119.8 (5)
C5—C4—C2	117.1 (6)	C13—C12—Br1	118.4 (5)
C3—C4—C2	122.4 (7)	C14—C13—C12	118.1 (6)
C4—C5—C6	120.0 (8)	C14—C13—H13	121.0
C4—C5—H5	120.0	C12—C13—H13	121.0
C6—C5—H5	120.0	C9—C14—C13	120.4 (6)
C7—C6—C5	120.3 (9)	C9—C14—H14	119.8
C7—C6—H6	119.8	C13—C14—H14	119.8
C5—C6—H6	119.8		
O1—C1—C3—C8	18.0 (10)	C5—C6—C7—C8	−1.6 (13)
O2—C1—C3—C8	−157.9 (6)	C6—C7—C8—C3	1.3 (12)
O1—C1—C3—C4	−162.5 (7)	C4—C3—C8—C7	−0.3 (10)
O2—C1—C3—C4	21.7 (9)	C1—C3—C8—C7	179.3 (7)
C8—C3—C4—C5	−0.2 (10)	C14—C9—C10—C11	0.4 (9)
C1—C3—C4—C5	−179.8 (6)	N1—C9—C10—C11	−177.8 (5)
C8—C3—C4—C2	−179.1 (5)	C9—C10—C11—C12	0.0 (9)
C1—C3—C4—C2	1.3 (10)	C10—C11—C12—C13	−0.5 (10)
O4—C2—C4—C5	−95.0 (7)	C10—C11—C12—Br1	179.2 (4)
O3—C2—C4—C5	82.4 (7)	C11—C12—C13—C14	0.7 (9)
O4—C2—C4—C3	83.9 (8)	Br1—C12—C13—C14	−179.0 (4)
O3—C2—C4—C3	−98.7 (7)	C10—C9—C14—C13	−0.2 (8)
C3—C4—C5—C6	−0.1 (11)	N1—C9—C14—C13	178.0 (5)
C2—C4—C5—C6	178.9 (6)	C12—C13—C14—C9	−0.3 (9)
C4—C5—C6—C7	1.0 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3^{i}	0.80 (6)	1.76 (6)	2.518 (6)	158 (7)
N1—H1*C*···O4	0.89	2.37	2.962 (7)	125
N1—H1*C*···O1^{ii}	0.89	2.13	2.916 (7)	147
N1—H1*B*···O4^{iii}	0.89	1.96	2.804 (7)	159
N1—H1*A*···O3^{iv}	0.89	1.96	2.828 (6)	164

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+1; (ii) *x*, *y*−1, *z*; (iii) −*x*+1, *y*, −*z*+1; (iv) *x*−1/2, *y*−1/2, *z*.