Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[(Dimethylamino)(phenyl)methyl]benzoic acid

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.058
Data-to-parameter ratio = 8.4

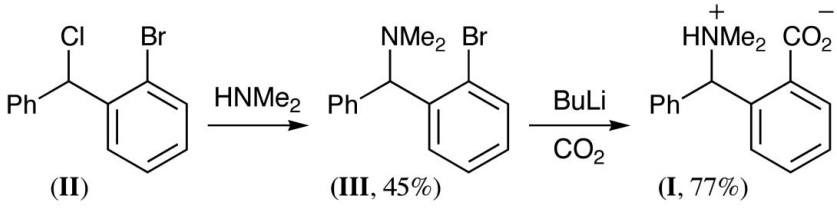
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound {systematic name: [(2-carboxylatophenyl)(phenyl)methyl]-*N*,*N*-dimethylammonium}, $C_{16}H_{17}NO_2$, crystallizes as a hydrogen-bonded zwitterion.

Received 22 June 2005
Accepted 4 July 2005
Online 13 July 2005

Comment

The title compound, (I), was prepared as described and shown in the scheme. This compound crystallizes as the zwitterion [(2-carboxylatophenyl)(phenyl)methyl]-*N*,*N*-dimethylammonium (Fig. 1). There are infinite chains of hydrogen-bonded molecules, with alternating stereochemistry at C1, running parallel to the crystallographic *c* axis (Fig. 2). The molecules are connected by hydrogen bonds between N1H and O1 of a neighbouring molecule [N1$\cdots$O1^{i} = 2.670 (3) Å; symmetry code: (i) $x, -y+1, z-\frac{1}{2}$]. There also appears to be intramolecular C1—H11$\cdots$O1 hydrogen bonding (Desiraju, 2005), as shown by the C$\cdots$O distance of 2.848 (3) Å. This interaction is strengthened by the increased CH acidity due to the adjacent positively-charged N atom.



Experimental

Dimethylamine (4.7 ml of a 40 wt% solution in water, 35 mmol) and 1-bromo-2-[chloro(phenyl)methyl]benzene, (II) (Katsura *et al.*, 1997) (500 mg, 1.78 mmol), in dimethyl sulfoxide (3.8 ml) were heated at reflux for 24 h. The product was purified by column chromatography and recrystallisation from dichloromethane/light petroleum to give [(2-bromophenyl)(phenyl)methyl]-*N*,*N*-dimethylamine, (III), as a white crystalline solid (234 mg, 45%, m.p. 333 K). Butyllithium (0.86 ml of 1.6 *M* hexane solution, 1.38 mmol) was added dropwise to a solution of (III) (200 mg, 0.69 mmol) in anhydrous tetrahydrofuran (4 ml) at 195 K and stirred for 2 h. The reaction mixture was warmed to room temperature whilst dry carbon dioxide was bubbled through the solution for a further 2 h. Water (5 ml) and acetic acid (0.13 ml, 2.27 mmol) were added until a pH of 7 was achieved. The product was purified by column chromatography (2:25 methanol–dichloromethane) to yield (I) as colourless crystals (136 mg, 77%). Crystals suitable for single-crystal X-ray diffraction analysis were obtained by slow evaporation of a solution in propan-2-ol.

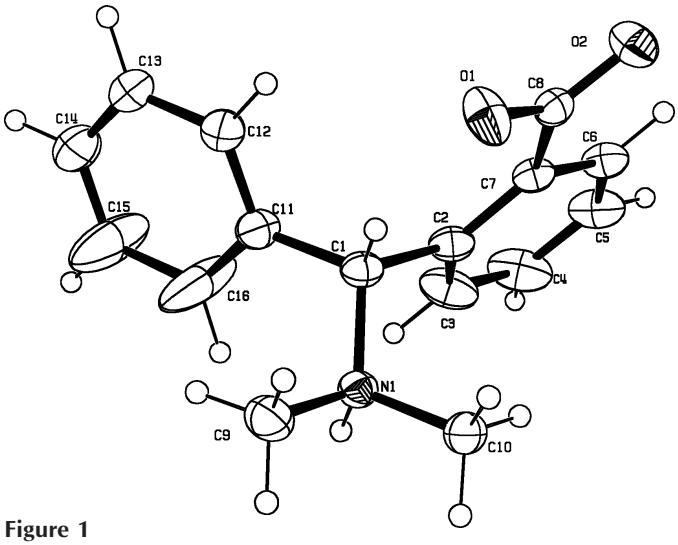


Figure 1
The zwitterionic form of compound (I), showing 40% probability displacement ellipsoids and H atoms of fixed radii.

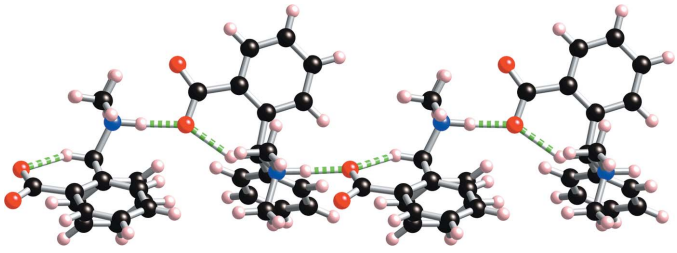


Figure 2
View of one of the infinite hydrogen-bonded chains of (I) parallel to the c axis. The H···O interactions are shown as green and white lines (CrystalMaker Software Limited, 2002).

Crystal data

$C_{16}H_{17}NO_2$
M_r = 255.32
Monoclinic, $C2/c$
a = 24.5562 (10) Å
b = 9.2464 (4) Å
c = 11.9764 (5) Å
β = 91.559 (2)°
V = 2718.3 (2) Å^3
Z = 8
D_x = 1.248 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3144 reflections
θ = 5–27°
μ = 0.08 mm^{-1}
T = 150 K
Block, colourless
0.32 × 0.18 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan *SCALEPACK* (Otwinowski & Minor, 1997)
T_{min} = 0.97, T_{max} = 0.99
13569 measured reflections
3079 independent reflections
1487 reflections with $I > 3\sigma(I)$
R_{int} = 0.075
θ_{max} = 27.5°
$h = -31 \rightarrow 31$
$k = -11 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.058
S = 1.14
1487 reflections
176 parameters
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme: see below
$(\Delta/\sigma)_{max}$ = 0.010
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.515 (3)	C1—C11	1.516 (4)
N1—C9	1.485 (4)	C2—C7	1.404 (4)
N1—C10	1.487 (3)	C7—C8	1.531 (3)
N1—H1	1.04 (3)	C8—O1	1.270 (3)
C1—C2	1.523 (4)	C8—O2	1.232 (3)
C1—N1—C9	110.3 (2)	N1—C1—C11	111.7 (2)
C1—N1—C10	111.8 (2)	C2—C1—C11	112.6 (2)
C9—N1—C10	109.0 (2)	C1—C2—C7	123.4 (2)
C1—N1—H1	112.9 (18)	C2—C7—C8	126.6 (2)
C9—N1—H1	106.7 (19)	C7—C8—O1	118.7 (2)
C10—N1—H1	106.0 (18)	C7—C8—O2	117.6 (2)
N1—C1—C2	110.8 (2)	O1—C8—O2	123.7 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	1.04 (3)	1.64 (3)	2.670 (3)	176 (3)
C1—H11···O1	1.00	2.02	2.848 (3)	139

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

A Chebychev polynomial (Carruthers & Watkin, 1979; Prince, 1982) was used in the weighting scheme, [weight] = $1.0/[A_0T_0(x) + A_1T_1(x) + \cdots + A_{n-1}T_{n-1}(x)]$, where A_i are the Chebychev coefficients 0.491, 0.269 and 0.192, and $x = F/F_{max}$; robust weighting (Prince, 1982) W = [weight] $[1 - (\delta F/6\sigma F)^2]^2$. The N-bound H atom was located in a difference Fourier map and its coordinates and isotropic displacement parameter subsequently refined. Other H atoms were positioned geometrically, with C—H = 1.00 Å and U_{iso}(H) = $1.2U_{eq}$(C).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Issue 12; Betteridge *et al.*, 2003); molecular graphics: *CrystalMaker* (CrystalMaker Software Limited, 2002); software used to prepare material for publication: *CRYSTALS*.

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supporting information

Acta Cryst. (2005). E**61**, o2502–o2503 [https://doi.org/10.1107/S1600536805021215]

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$V = 2718.3\ (2)$ Å^3
$Z = 8$
$F(000) = 1088$
$D_x = 1.248$ Mg m^{-3}
Melting point: 446 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3144 reflections
$\theta = 5–27°$
$\mu = 0.08$ mm^{-1}
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Block, colourless
0.32 × 0.18 × 0.14 mm

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$\theta_{max} = 27.5°$, $\theta_{min} = 5.5°$
$h = -31\rightarrow31$
$k = -11\rightarrow12$
$l = -15\rightarrow15$

Refinement

Refinement on F
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.058$
$S = 1.14$
1487 reflections
176 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = $1.0/[A_0*T_0(x) + A_1*T_1(x) \cdots + A_{n-1}]*T_{n-1}(x)]$ where A_i are the Chebychev coefficients listed below and x = F /Fmax Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta$F$/6*sigma$F$)^2]^2 A_i are: 0.491 0.269 0.192
$(\Delta/\sigma)_{max} = 0.010$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Experimental. The material was prepared as described above and recrystallized from propan-2-ol.
Additional spectroscopic data before recrystallization:
^{1}H NMR: (400.1 MHz, $CDCl_3$) 2.54(6*H*, s), 4.82 (1*H*, s), 7.32–7.42 (6*H*, m) 7.54 (2*H*, d, J 7.0), 8.35 (1*H*, d, J 9.2)
^{13}C NMR: (500.3 MHz, $CDCl_3$) 42.1, 77.2, 128.9, 129.0, 129.2, 129.5, 130.5, 131.5, 134.6, 134.9, 135.7, 136.5, 171.2
HR—MS (ESI^-) Found 254.1179 $(M—H)^-$, calc 254.1181

Refinement. The large refined displacement parameters for the C atoms C15 and C16 and the variations in the C—C bond lengths of the phenyl group containing these atoms suggest there to be some disorder of this group. Attempts to model this did not lead to any improvement is the agreement with the X-ray data and were abandoned.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.10685 (8)	0.6128 (2)	0.37916 (18)	0.0311
C1	0.13544 (10)	0.4876 (3)	0.43757 (19)	0.0320
C2	0.10653 (11)	0.3455 (3)	0.4112 (2)	0.0340
C3	0.10272 (15)	0.3000 (3)	0.2991 (2)	0.0531
C4	0.07689 (17)	0.1727 (4)	0.2691 (2)	0.0608
C5	0.05430 (14)	0.0855 (3)	0.3492 (3)	0.0509
C6	0.05869 (11)	0.1276 (3)	0.4598 (2)	0.0377
C7	0.08438 (10)	0.2561 (3)	0.4931 (2)	0.0298
C8	0.08572 (10)	0.2831 (3)	0.6193 (2)	0.0309
O1	0.10760 (9)	0.3985 (2)	0.65640 (14)	0.0478
O2	0.06552 (8)	0.1902 (2)	0.67923 (15)	0.0430
C9	0.13230 (13)	0.7520 (3)	0.4138 (3)	0.0469
C10	0.04786 (11)	0.6172 (3)	0.4040 (2)	0.0383
C11	0.19555 (11)	0.4829 (3)	0.4119 (2)	0.0391
C12	0.23187 (12)	0.4437 (4)	0.4950 (3)	0.0546
C13	0.28734 (13)	0.4357 (4)	0.4771 (3)	0.0599
C14	0.30757 (13)	0.4678 (4)	0.3769 (3)	0.0563
C15	0.27275 (16)	0.5083 (8)	0.2933 (4)	0.1261
C16	0.21662 (15)	0.5157 (8)	0.3102 (3)	0.1192
H1	0.1090 (13)	0.608 (4)	0.293 (3)	0.064 (10)*
H11	0.1334	0.5033	0.5200	0.0383*
H31	0.1191	0.3613	0.2399	0.0637*
H41	0.0745	0.1434	0.1887	0.0726*
H51	0.0352	−0.0062	0.3276	0.0606*
H61	0.0429	0.0639	0.5181	0.0450*
H91	0.1131	0.8336	0.3747	0.0560*
H92	0.1294	0.7641	0.4964	0.0560*
H93	0.1716	0.7519	0.3936	0.0560*
H101	0.0304	0.7010	0.3641	0.0461*
H102	0.0434	0.6284	0.4863	0.0461*
H103	0.0301	0.5253	0.3782	0.0461*
H121	0.2181	0.4202	0.5707	0.0654*
H131	0.3127	0.4057	0.5396	0.0715*
H141	0.3476	0.4617	0.3645	0.0676*
H151	0.2873	0.5334	0.2185	0.1516*

H161	0.1915	0.5452	0.2471	0.1432*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0302 (11)	0.0345 (12)	0.0288 (11)	0.0031 (9)	0.0027 (9)	0.0035 (9)
C1	0.0357 (14)	0.0352 (14)	0.0249 (12)	0.0041 (11)	−0.0008 (10)	0.0018 (10)
C2	0.0411 (16)	0.0339 (14)	0.0268 (13)	0.0082 (12)	−0.0033 (11)	−0.0021 (11)
C3	0.087 (2)	0.0444 (18)	0.0274 (14)	0.0092 (17)	0.0003 (15)	0.0004 (13)
C4	0.107 (3)	0.0470 (19)	0.0276 (16)	0.0114 (19)	−0.0118 (16)	−0.0107 (14)
C5	0.073 (2)	0.0323 (15)	0.0464 (17)	0.0060 (15)	−0.0199 (16)	−0.0091 (14)
C6	0.0441 (16)	0.0322 (14)	0.0362 (14)	0.0067 (12)	−0.0083 (12)	−0.0032 (12)
C7	0.0327 (13)	0.0302 (13)	0.0263 (12)	0.0062 (11)	−0.0035 (10)	−0.0037 (10)
C8	0.0267 (13)	0.0339 (14)	0.0319 (13)	0.0039 (11)	0.0000 (10)	−0.0004 (11)
O1	0.0654 (13)	0.0521 (12)	0.0262 (9)	−0.0242 (11)	0.0067 (9)	−0.0066 (9)
O2	0.0562 (13)	0.0393 (11)	0.0336 (10)	−0.0056 (9)	0.0027 (9)	0.0027 (9)
C9	0.0537 (19)	0.0382 (16)	0.0481 (18)	−0.0067 (14)	−0.0090 (14)	0.0059 (13)
C10	0.0343 (15)	0.0446 (16)	0.0363 (14)	0.0062 (12)	0.0076 (11)	0.0058 (12)
C11	0.0371 (15)	0.0432 (16)	0.0370 (14)	0.0051 (13)	−0.0005 (12)	0.0021 (12)
C12	0.0422 (18)	0.071 (2)	0.0503 (18)	−0.0075 (16)	−0.0055 (14)	0.0239 (16)
C13	0.0373 (17)	0.074 (2)	0.068 (2)	−0.0047 (16)	−0.0121 (15)	0.0243 (19)
C14	0.0371 (17)	0.064 (2)	0.068 (2)	0.0096 (16)	0.0014 (15)	0.0069 (18)
C15	0.047 (2)	0.269 (7)	0.063 (2)	0.054 (3)	0.0180 (18)	0.050 (4)
C16	0.043 (2)	0.265 (7)	0.050 (2)	0.053 (3)	0.0092 (16)	0.048 (3)

Geometric parameters (Å, º)

N1—C1	1.515 (3)	C8—O2	1.232 (3)
N1—C9	1.485 (4)	C9—H91	1.000
N1—C10	1.487 (3)	C9—H92	1.000
N1—H1	1.04 (3)	C9—H93	1.000
C1—C2	1.523 (4)	C10—H101	1.000
C1—C11	1.516 (4)	C10—H102	1.000
C1—H11	1.000	C10—H103	1.000
C2—C3	1.408 (4)	C11—C12	1.367 (4)
C2—C7	1.404 (4)	C11—C16	1.371 (5)
C3—C4	1.380 (5)	C12—C13	1.387 (5)
C3—H31	1.000	C12—H121	1.000
C4—C5	1.381 (5)	C13—C14	1.344 (5)
C4—H41	1.000	C13—H131	1.000
C5—C6	1.382 (4)	C14—C15	1.352 (5)
C5—H51	1.000	C14—H141	1.000
C6—C7	1.399 (4)	C15—C16	1.400 (5)
C6—H61	1.000	C15—H151	1.000
C7—C8	1.531 (3)	C16—H161	1.000
C8—O1	1.270 (3)		
C1—N1—C9	110.3 (2)	O1—C8—O2	123.7 (2)

C1—N1—C10	111.8 (2)	N1—C9—H91	109.466
C9—N1—C10	109.0 (2)	N1—C9—H92	109.467
C1—N1—H1	112.9 (18)	H91—C9—H92	109.476
C9—N1—H1	106.7 (19)	N1—C9—H93	109.466
C10—N1—H1	106.0 (18)	H91—C9—H93	109.475
N1—C1—C2	110.8 (2)	H92—C9—H93	109.476
N1—C1—C11	111.7 (2)	N1—C10—H101	109.467
C2—C1—C11	112.6 (2)	N1—C10—H102	109.467
N1—C1—H11	108.078	H101—C10—H102	109.475
C2—C1—H11	107.138	N1—C10—H103	109.467
C11—C1—H11	106.176	H101—C10—H103	109.476
C1—C2—C3	118.3 (2)	H102—C10—H103	109.476
C1—C2—C7	123.4 (2)	C1—C11—C12	118.7 (2)
C3—C2—C7	118.3 (3)	C1—C11—C16	124.5 (2)
C2—C3—C4	121.4 (3)	C12—C11—C16	116.8 (3)
C2—C3—H31	119.311	C11—C12—C13	121.7 (3)
C4—C3—H31	119.312	C11—C12—H121	119.141
C3—C4—C5	120.5 (3)	C13—C12—H121	119.143
C3—C4—H41	119.727	C12—C13—C14	121.0 (3)
C5—C4—H41	119.727	C12—C13—H131	119.500
C4—C5—C6	118.6 (3)	C14—C13—H131	119.501
C4—C5—H51	120.696	C13—C14—C15	118.7 (3)
C6—C5—H51	120.695	C13—C14—H141	120.636
C5—C6—C7	122.4 (3)	C15—C14—H141	120.636
C5—C6—H61	118.805	C14—C15—C16	120.8 (4)
C7—C6—H61	118.805	C14—C15—H151	119.615
C2—C7—C6	118.7 (2)	C16—C15—H151	119.613
C2—C7—C8	126.6 (2)	C11—C16—C15	121.0 (3)
C6—C7—C8	114.7 (2)	C11—C16—H161	119.509
C7—C8—O1	118.7 (2)	C15—C16—H161	119.513
C7—C8—O2	117.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	1.04 (3)	1.64 (3)	2.670 (3)	176 (3)
C1—H11···O1	1.00	2.02	2.848 (3)	139

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