Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Yvette L. Dann, Andrew R. Cowley and Harry L. Anderson*

University of Oxford, Department of Chemistry, Chemistry Research Laboratory, 12 Mansfield Road, Oxford OX1 3TA, England

Correspondence e-mail: harry.anderson@chemistry.ox.ac.uk

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

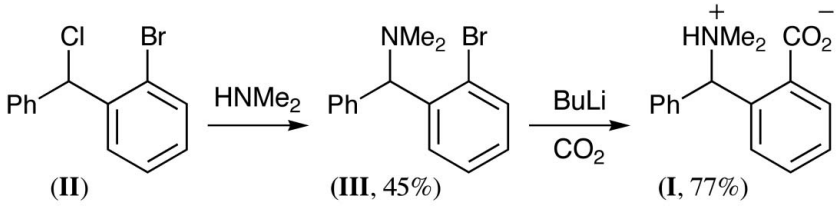
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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.

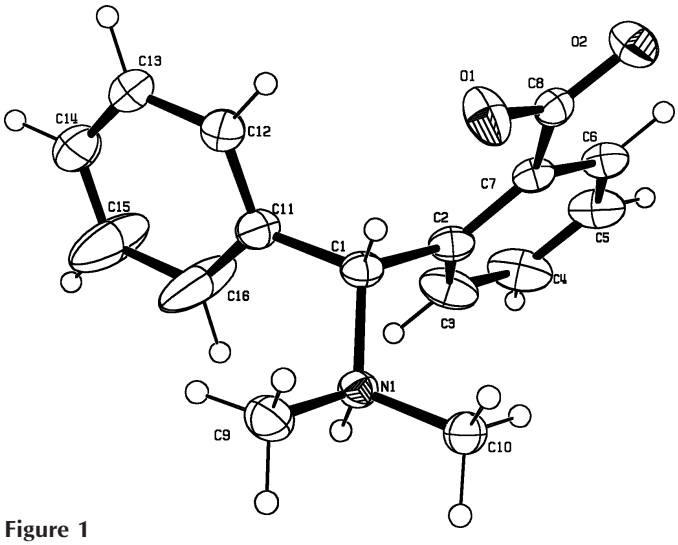


Figure 1
The zwitterionic form of compound (I), showing 40% probability displacement ellipsoids and H atoms of fixed radii.

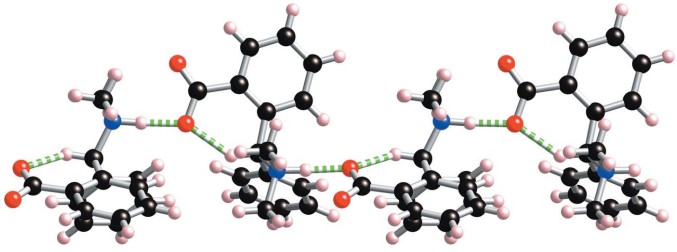


Figure 2
View of one of the infinite hydrogen-bonded chains of (I) parallel to the c axis. The H···O interactions are shown as green and white lines (CrystalMaker Software Limited, 2002).

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*.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Betteridge, P. W., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Carruthers, J. R. & Watkin, D. J. (1979). *Acta Cryst.* A**35**, 698–699.
CrystalMaker Software Limited (2002). *CrystalMaker*. Version 5.0. CrystalMaker Software Limited, Begbroke Science Park, Building 5, Sandy Lane, Yarnton, Oxfordshire, OX5 1PF, England.
Desiraju, G. R. (2005). *Chem. Commun.* 2995–3001.
Katsura, Y., Zhang, X. Y., Homma, K., Rice, K. C., Calderon, S. N., Rothman, R. B., Yamamura, H. I., Davis, P., Flippen-Anderson, J. L., Xu, H., Becketts, K., Foltz, E. J. & Porreca, F. J. (1997). *J. Med. Chem.* **40**, 2936–2947.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307-326. New York: Academic Press.
Prince, E. (1982). *Mathematical Techniques in Crystallography and Material Science*. New York: Springer-Verlag.