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2-(4-Methylpiperazin-4-ium-1-ylcarbonyl)-benzoate monohydrate

Ming-Lin Guo

College of Materials and Chemical Engineering, Tianjin Polytechnic University, Tianjin 300160, People's republic of China

Correspondence e-mail: guomlin@public.tpt.tj.cn

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Key indicators

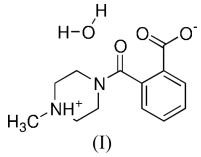
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.110
Data-to-parameter ratio = 15.8

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

The title compound, $C_{13}H_{16}N_2O_3\cdot H_2O$, is an intramolecular salt and combines a water molecule via an O—H···O intermolecular hydrogen bond. In the crystal structure, the packing of the molecules is stabilized by O—H···O and N—H···O hydrogen bonds, and van der Waals forces.

Comment

1-Methylpiperazine reacts with phthalic anhydride to form 2-(4-methylpiperazinylcarbonyl)benzolic acid, which is both a carboxylic acid and a basic amine (Guo, 2004).



(I)

The molecular structure of the title compound, (I), is shown in Fig. 1. The bond distances and angles are normal, within experimental error.

In the crystal structure, symmetry-related molecules are linked by hydrogen bonds (Table 1 and Fig. 2).

The crystal structure of the title compound, (I), owes its formation to a hydrogen bond between atom O1 and atom H4*C* of the water molecule and an intermolecular hydrogen bond between atom O3 and atom H4*D* of the water molecule. Two phthalyl moieties are linked together by these hydrogen bonds; an 18-membered ring is formed. In addition, two molecules are associated into a 20-membered ring via intermolecular hydrogen bonds between atom O2 of the benzoate anion (C13) and a H atom of the 4-methylpiperazin-4-ium (N2) cation. The two rings appear alternately and form a molecular ribbon, which builds up a two-dimensional molecular layer. In the crystal structure, the supramolecular layers of the title compound are stabilized by van der Waals forces.

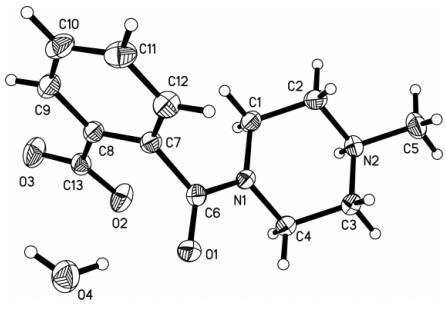


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

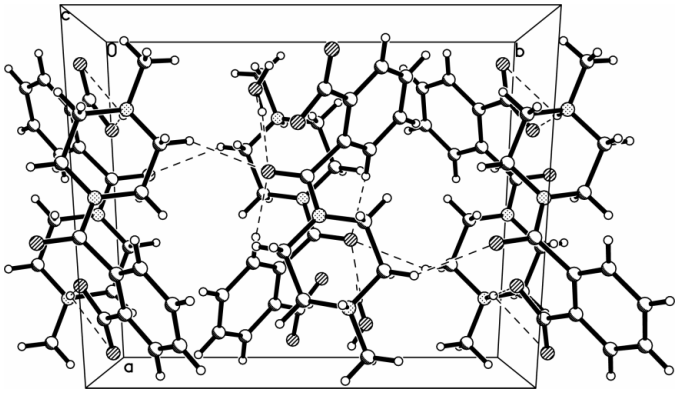


Figure 2
Packing diagram, showing the hydrogen-bonding interactions as dashed lines.

Experimental

The title compound was prepared by the reaction of phthalic anhydride (2.9 g) with 1-methylpiperazine (1.9 g) under microwave irradiation for 4 min. The resulting product was dispersed in ethanol (20 ml), after which 1.8 g of solid product was separated by filtration. Pure 2-(4-methylpiperazin-4-ium-1-carbonyl)benzoate (1.5 g) was heated and dissolved in water (10 ml). Single crystals of (I) were obtained after 3 d at room temperature.

Crystal data

$C_{13}H_{16}N_2O_3\cdot H_2O$
$M_r = 266.29$
Monoclinic, $P2_1/c$
$a = 11.494$ (6) Å
$b = 12.754$ (5) Å
$c = 10.252$ (4) Å
$\beta = 116.478$ (6)°
$V = 1345.3$ (10) Å^3
$Z = 4$
$D_x = 1.315$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 734 reflections
$\theta = 2.5$–24.9°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.34 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.965$, $T_{max} = 0.985$
7655 measured reflections
2757 independent reflections
1844 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 26.4°$
$h = -9 \rightarrow 14$
$k = -15 \rightarrow 15$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.110$
$S = 1.01$
2757 reflections
174 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.1704P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.023 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4D···O3^{i}	0.85	1.95	2.782 (2)	165
O4—H4C···O1	0.85	2.07	2.9139 (19)	171
N2—H2···O3^{ii}	0.91	2.57	3.149 (2)	123
N2—H2···O2^{ii}	0.91	1.73	2.633 (2)	173

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

The water H atoms were found in difference Fourier maps; however, during refinement, they were fixed at O—H distances of 0.85 Å and their U_{iso} values were set at $1.2U_{eq}$(O). The H atoms of the NH and CH groups were treated as riding, with N—H = 0.91 Å and C—H = 0.93–0.97 Å, and U_{iso}(H) = $1.2U_{eq}$(N, C). For the H atoms attached to atom C5, U_{iso}(H) = $1.5U_{eq}$(C5).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

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