

4-Methylpiperazin-1-ium 2-amino-5-iodobenzoate

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Key indicators

Single-crystal X-ray study
 $T = 294\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
 R factor = 0.032
 wR factor = 0.088
 Data-to-parameter ratio = 16.2

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

In the title salt, $\text{C}_5\text{H}_{13}\text{N}_2^+ \cdot \text{C}_7\text{H}_5\text{INO}_2^-$, the packing of the ions is stabilized by $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds and van der Waals forces.

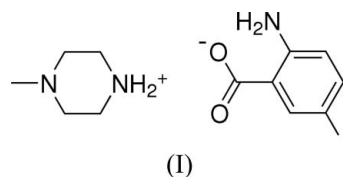
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Comment

Our interest in piperazine derivatives stems from their application in host-guest systems. We have recently reported the crystal structure of 1-methylpiperazine-1,4-dium 4-nitrophthalate(2-) 4-nitrophthalic acid monohydrate (Guo, 2004). The structure of 4-methylpiperazin-1-ium 2-amino-5-iodobenzoate, (I), is reported here.



The structures of the cation and anion are shown in Fig. 1. The cation adopts a normal chair conformation, as reported previously (Guo, 2004), and participates in hydrogen bonds formed between the $\text{N}2-\text{H}2\text{B}$ group and atom $\text{O}1^{\text{ii}}$ of a 2-amino-5-iodobenzoate anion and the $\text{N}2-\text{H}2\text{A}$ group and atom $\text{O}2^{\text{iii}}$ of another anion (see Table 1 for symmetry codes). Within the anion, the amino (NH_2) and carboxy ($\text{C}7/\text{O}1/\text{O}2$) groups are almost coplanar with the central six-membered

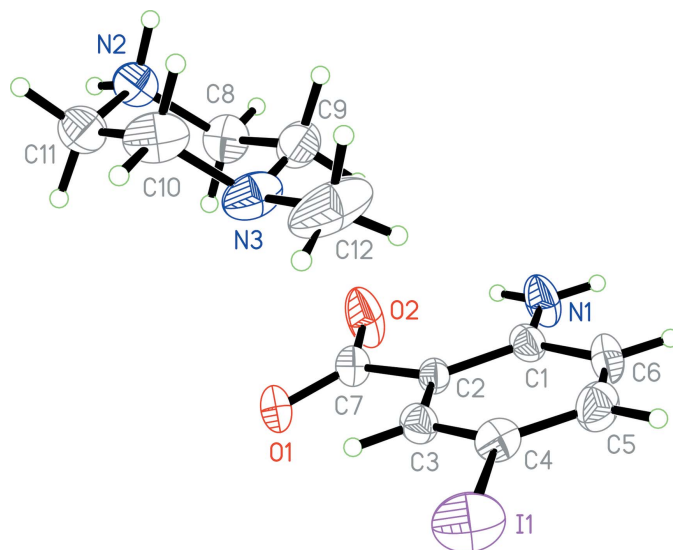


Figure 1

A view of the structure of (I), showing the atom-numbering scheme; displacement ellipsoids for non-H atoms are drawn at the 30% probability level. H atoms are represented by circles of arbitrary size

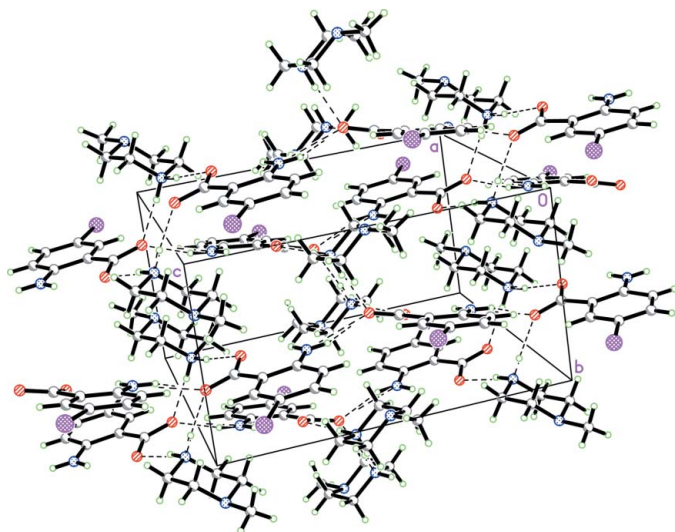


Figure 2
Packing diagram, showing the hydrogen-bond interactions as dashed lines.

ring. Atom H1B is involved in an intramolecular N1—H1B···O2 hydrogen bond, while atom H1A is responsible for the formation of an N1—H1A···O1ⁱ hydrogen bond (see Table 1 for symmetry code), which links another anion. These hydrogen-bond contacts build up a number of different sized rings and further aggregate into a three-dimensional framework. A packing diagram for the structure of (I) is shown in Fig. 2.

Experimental

Single crystals of the title salt were obtained from ethanol solutions (20 ml) of 2-amino-5-iodobenzoic acid (0.7 g) and 1-methylpiperazine (0.5 g) by slow concentration over a period of 2 d at room temperature.

Crystal data

$C_5H_{13}N_2^+ \cdot C_7H_5INO_2^-$
 $M_r = 363.19$
 Monoclinic, $P2_1/c$
 $a = 14.829$ (3) Å
 $b = 7.3472$ (14) Å
 $c = 13.713$ (3) Å
 $\beta = 95.313$ (3)°
 $V = 1487.6$ (5) Å³
 $Z = 4$

$D_x = 1.622$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2209 reflections
 $\theta = 2.8$ – 24.1 °
 $\mu = 2.15$ mm⁻¹
 $T = 294$ (2) K
 Prism, colorless
 $0.26 \times 0.22 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.578$, $T_{\max} = 0.656$
 7380 measured reflections

2655 independent reflections
 1678 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\max} = 25.1$ °
 $h = -17 \rightarrow 17$
 $k = -8 \rightarrow 8$
 $l = -16 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.0888$
 $S = 1.02$
 2655 reflections
 164 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.5464P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.33$ e Å⁻³
 $\Delta\rho_{\min} = -0.65$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1A···O1 ⁱ	0.86	2.06	2.889 (4)	162
N1—H1B···O2	0.86	1.99	2.637 (4)	131
N2—H2B···O1 ⁱⁱ	0.90	1.88	2.780 (4)	174
N2—H2A···O2 ⁱⁱⁱ	0.90	1.79	2.675 (4)	168

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

The H atoms involved in N—H···O hydrogen bonds were found in difference Fourier maps; however, during refinement, N—H distances were fixed at 0.86 or 0.90 Å and the U_{iso} values were set at $1.2U_{\text{eq}}(\text{N})$. H atoms bonded to C atoms were included in the refinement in the riding-model approximation, with C—H = 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the non-methyl C atoms and C—H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl atom C12.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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References

- Bruker (1997). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2000). SHELXTL. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
 Guo, M.-L. (2004). Acta Cryst. C60, o690–o692.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.