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## Key indicators

Single-crystal X-ray study  
 $T = 293\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.010\text{ \AA}$   
 $R$  factor = 0.056  
 $wR$  factor = 0.161  
Data-to-parameter ratio = 7.4For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## Piperazinediium 3-nitrophthalate dihydrate

The title salt,  $\text{C}_4\text{H}_{12}\text{N}_2^{2+} \cdot \text{C}_8\text{H}_3\text{NO}_6^{2-} \cdot 2\text{H}_2\text{O}$ , crystallizes in the orthorhombic space group  $Pna2_1$ . All O atoms of the 3-nitrophthalate anion are involved in hydrogen bonding with the piperazine dication and the water molecules of crystallization.

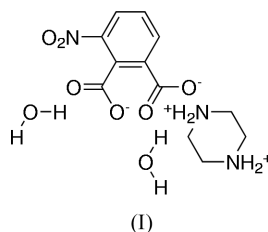
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## Comment

The present work is part of a structural study of piperazine and its derivatives (Guo, 2004*a,b,c*). The author has recently reported the crystal structures of *N*-[4-(4-methylpiperazin-1-ylsulfonyl)phenyl]acetamide monohydrate (Guo, 2004*a*) 2-(4-methylpiperazin-4-ium-1-ylcarbonyl)benzoate monohydrate (Guo, 2004*b*) and 4-ammonio-1-methylpiperazin-1-ium oxalate dihydrate (Guo, 2004*c*). The structure of the salt piperazinediium 3-nitrophthalate dihydrate, (I), is reported here.



A view of the hydrogen bonding of (I) and its numbering scheme are illustrated in Fig. 1. The piperazinediium cation adopts a normal chair conformation, as reported previously

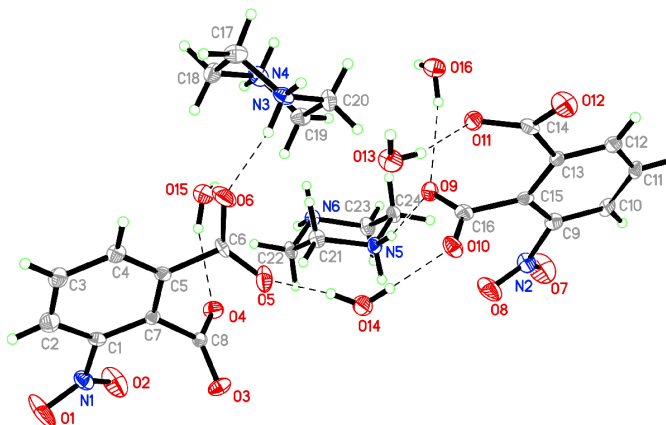
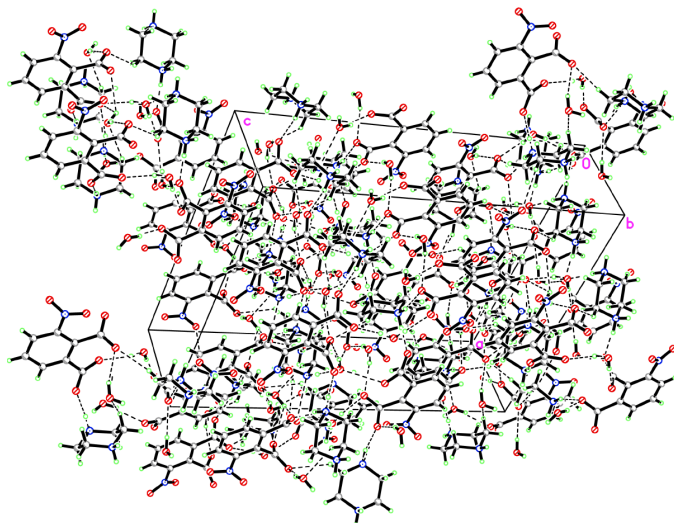


Figure 1

The structure of the title salt, (I), with the atom-numbering scheme, showing 30% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii and hydrogen bonds are indicated by dashed lines.



**Figure 2**  
Packing diagram of (I), showing the hydrogen-bond interactions (dashed lines)

(Kuppayee *et al.*, 1999). The bond distances and angles in the cation, the anion and water molecules are normal, within experimental error. Selected geometric parameters are shown in Table 1.

In the crystal structure, a dense hydrogen-bond network is built up (Fig. 2 and Table 2).

## Experimental

Crystals of the title salt were obtained from a 1:1 aqueous solution of piperazine and 3-nitrophthalic acid by slow concentration over a period of 3 d.

### Crystal data

$C_4H_{12}N_2^{2+} \cdot C_8H_3NO_6^{2-} \cdot 2H_2O$   
 $M_r = 333.30$   
 Orthorhombic,  $Pna2_1$   
 $a = 18.436$  (8) Å  
 $b = 6.551$  (3) Å  
 $c = 24.274$  (10) Å  
 $V = 2932$  (2) Å<sup>3</sup>  
 $Z = 8$   
 $D_x = 1.510$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 993 reflections  
 $\theta = 2.4$ – $26.0^\circ$   
 $\mu = 0.13$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Prism, colorless  
 $0.20 \times 0.16 \times 0.14$  mm

### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.954$ ,  $T_{\max} = 0.982$   
 15732 measured reflections

3053 independent reflections  
 2278 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.062$   
 $\theta_{\max} = 26.4^\circ$   
 $h = -22 \rightarrow 18$   
 $k = -6 \rightarrow 8$   
 $l = -30 \rightarrow 27$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.161$   
 $S = 1.04$   
 3053 reflections  
 415 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0991P)^2 + 0.5343P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.004$   
 $\Delta\rho_{\max} = 0.48$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.25$  e Å<sup>-3</sup>

**Table 1**  
Selected geometric parameters (Å, °).

O3—C8	1.246 (6)	O9—C16	1.258 (7)
O4—C8	1.237 (6)	O10—C16	1.252 (7)
O5—C6	1.216 (9)	O11—C14	1.278 (9)
O6—C6	1.248 (7)	O12—C14	1.240 (7)
O5—C6—O6	126.9 (6)	O12—C14—O11	123.9 (7)
O4—C8—O3	126.9 (6)	O10—C16—O9	126.0 (7)
C4—C5—C6—O5	149.7 (6)	C15—C13—C14—O12	157.2 (5)
C7—C5—C6—O5	-28.0 (7)	C12—C13—C14—O12	-24.5 (7)
C4—C5—C6—O6	-25.6 (7)	C15—C13—C14—O11	-26.0 (7)
C7—C5—C6—O6	156.6 (5)	C12—C13—C14—O11	152.3 (5)
C6—C5—C7—C8	-1.2 (7)	C14—C13—C15—C16	1.0 (7)
C1—C7—C8—O4	103.8 (6)	C13—C15—C16—O10	-79.0 (7)
C5—C7—C8—O4	-77.1 (7)	C9—C15—C16—O10	103.0 (6)
C1—C7—C8—O3	-76.2 (6)	C13—C15—C16—O9	104.4 (6)
C5—C7—C8—O3	102.9 (6)	C9—C15—C16—O9	-73.6 (7)

**Table 2**  
Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N3—H3A $\cdots$ O3 <sup>i</sup>	0.90	1.92	2.765 (6)	156
N3—H3B $\cdots$ O6	0.90	1.77	2.648 (6)	166
N4—H4A $\cdots$ O5 <sup>ii</sup>	0.90	1.92	2.799 (6)	164
N4—H4B $\cdots$ O13 <sup>iii</sup>	0.90	1.83	2.730 (6)	175
N5—H5A $\cdots$ O12 <sup>iii</sup>	0.90	1.79	2.664 (6)	165
N5—H5B $\cdots$ O9	0.90	1.89	2.760 (6)	161
N6—H6B $\cdots$ O11 <sup>iv</sup>	0.90	1.92	2.793 (6)	164
N6—H6A $\cdots$ O14 <sup>ii</sup>	0.90	1.81	2.708 (6)	172
O13—H13A $\cdots$ O4 <sup>i</sup>	0.85	1.89	2.729 (6)	167
O13—H13B $\cdots$ O11	0.85	1.96	2.792 (6)	166
O14—H14A $\cdots$ O5	0.85	1.89	2.714 (6)	164
O14—H14B $\cdots$ O10	0.85	1.86	2.707 (6)	173
O15—H15A $\cdots$ O4	0.85	2.04	2.858 (6)	161
O15—H15B $\cdots$ O3 <sup>ii</sup>	0.87	2.47	3.036 (6)	124
O15—H15B $\cdots$ O5 <sup>ii</sup>	0.87	2.53	2.997 (7)	115
O16—H16A $\cdots$ O9	0.85	2.18	3.007 (6)	166
O16—H16B $\cdots$ O10 <sup>ii</sup>	0.85	2.03	2.861 (6)	167

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

The value of the absolute structure parameter (Flack, 1983) of the title salt is meaningless because Mo radiation was used and no atoms heavier than O are present. Therefore, in the absence of anomalous dispersion effects, 1678 Friedel-pair reflections were merged before the final refinement. The H atoms of the water molecule were found in difference Fourier maps. However, during refinement, they were fixed at O—H distances of 0.85–0.87 Å and their  $U_{\text{iso}}$  values were set at 1.2  $U_{\text{eq}}(\text{O})$ . The H atoms of N—H and C—H groups were treated as riding, with N—H = 0.90 Å and C—H = 0.93–0.97 Å, and  $U_{\text{iso}} = 1.2 U_{\text{eq}}(\text{N or C})$ .

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*.

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