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Crystal structures of six 4-(4-nitrophenyl)piperazin-1-ium salts

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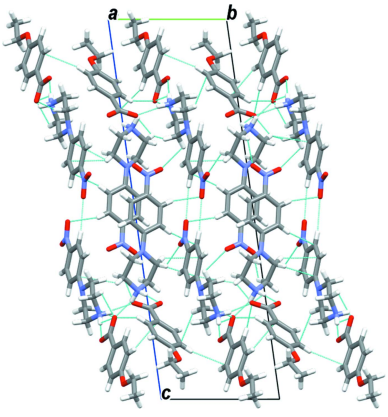
Six piperazinium salts, namely 4-(4-nitrophenyl)piperazin-1-ium 4-bromobenzoate dihydrate, $C_{10}H_{14}N_3O_2^+\cdot C_7H_4BrO_2^-\cdot 2H_2O$, (I), 4-(4-nitrophenyl)piperazin-1-ium 4-iodobenzoate dihydrate, $C_{10}H_{14}N_3O_2^+\cdot C_7H_4IO_2^-\cdot 2H_2O$, (II), 4-(4-nitrophenyl)piperazin-1-ium 4-hydroxybenzoate monohydrate, $C_{10}H_{14}N_3O_2^+\cdot C_7H_5O_3^-\cdot H_2O$, (III), 4-(4-nitrophenyl)piperazin-1-ium 4-methylbenzoate monohydrate, $C_{10}H_{14}N_3O_2^+\cdot C_8H_7O_2^-\cdot H_2O$, (IV), 4-(4-nitrophenyl)piperazin-1-ium 4-methoxybenzoate hemihydrate, $2C_{10}H_{14}N_3O_2^+\cdot 2C_8H_7O_3^-\cdot H_2O$, (V), and 4-(4-nitrophenyl)piperazin-1-ium 4-ethoxybenzoate, $2C_{10}H_{14}N_3O_2^+\cdot 2C_9H_9O_3^-$, (VI), have been synthesized and their crystal structures solved by single-crystal X-ray diffraction, revealing that all of them crystallize in the triclinic space group $P\overline{1}$ except for (V), which crystallizes in the monoclinic space group $P2_1/c$ and has a disordered nitro group. Compounds (I) and (II) are isostructural. The crystal packing of (I)–(V) is constructed from organic chains formed by a combination of hydrogen bonds of type N—H···O and/or O—H···O and other weak interactions of type C—H···O and/or C—H···π, forming sheets, whereas (VI) shows a cationic and anionic-based layer structure.

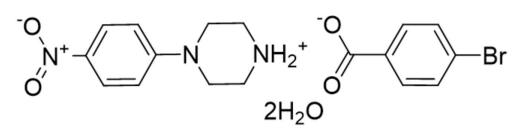
1. Chemical context

Piperazines and substituted piperazines are important pharmacophores that can be found in many biologically active compounds used to treat a number of different diseases (Berkheij, 2005) as antifungal (Upadhayaya *et al.*, 2004), antibacterial, anti-malarial and anti-psychotic agents (Choudhary *et al.*, 2006). A valuable insight into advances on the antimicrobial activity of piperazine derivatives was given by Kharb *et al.* (2012). Piperazines are among the most important building blocks in current drug discovery and are found in biologically active compounds across a number of different therapeutic areas (Brockunier *et al.*, 2004; Bogatcheva *et al.*, 2006). Pharmacological and toxicological information for piperazine derivatives is reviewed by Elliott (2011).

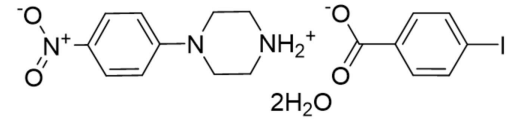
4-Nitrophenylpiperazinium chloride monohydrate has been used as an intermediate in the synthesis of anticancer drugs, transcriptase inhibitors and antifungal reagents and is also an important reagent for potassium channel openers, which show considerable biomolecular current-voltage rectification characteristics (Lu, 2007). The inclusion behaviours of 4-sulfonatocalix[n]arenes (SCX*n*) (*n* = 4, 6, 8) with 1-(4-nitrophenyl)piperazine (NPP) were investigated by UV spectroscopy and fluorescence spectroscopy at different pH values (Zhang *et al.*, 2014). The design, synthesis and biological profiling of arylpiperazine-based scaffolds for the manage-



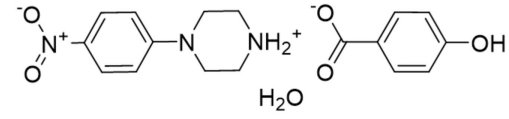
ment of androgen-sensitive prostatic disorders was reported by Gupta *et al.* (2016). 4-Nitrophenylpiperazine was the starting material in the synthesis and biological evaluation of novel piperazine-containing hydrazone derivatives (Kaya *et al.*, 2016). The crystal structure of 4-nitrophenyl piperazinium chloride monohydrate was reported by Lu (2007) and that of 4,6-dimethoxypyrimidin-2-amine-1-(4-nitrophenyl)piperazine (1:1) by Wang *et al.* (2014) while Ayeni *et al.* (2019) described the synthesis and crystal structure of a Schiff base, 5-methyl-2-{[4-(4-nitrophenyl)piperazin-1-yl]methyl}phenol is published. NMR-based investigations of acyl-functionalized piperazines concerning their conformational behaviour in solution has been studied and the crystal structures of 1-(4-fluorobenzoyl)-4-(4-nitrophenyl)piperazine, 1-(4-bromobenzoyl)-4-(4-nitrophenyl)piperazine and 1-(3-bromobenzoyl)-4-(4-nitrophenyl)piperazine have been reported (Wodtke *et al.*, 2018). We have recently reported the crystal structures of some salts of 4-methoxyphenylpiperazine (Kiran Kumar *et al.*, 2019) and also 2-methoxyphenylpiperazine (Harish Chinthal *et al.*, 2020), as well as some salts of piperazine derivatives (Archana *et al.*, 2021).



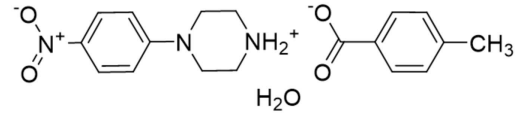
(I)



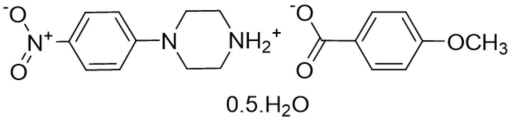
(II)



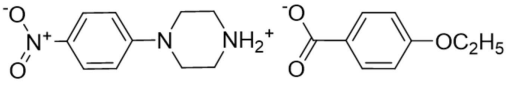
(III)



(IV)



(V)



(VI)

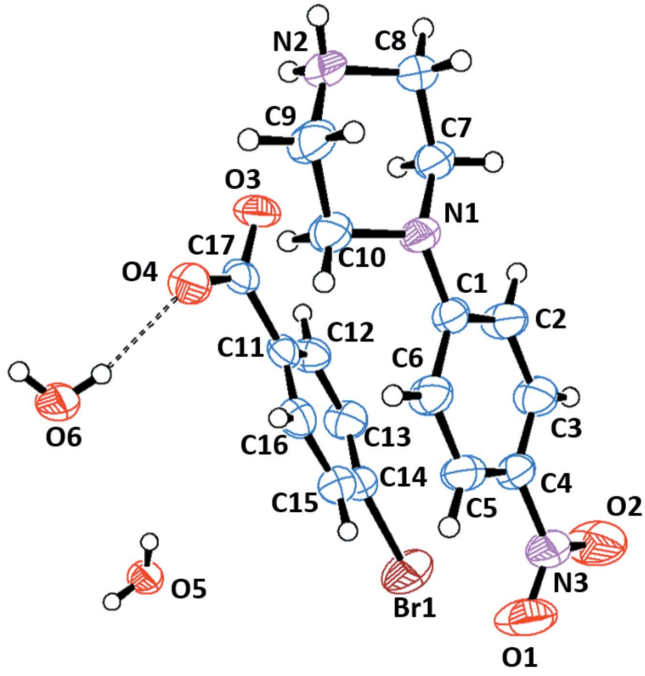


Figure 1
The independent components of compound (I) showing the atom-labelling scheme and the hydrogen bonds, drawn as dashed lines. Displacement ellipsoids are drawn at the 50% probability level.

In view of the importance of piperazines in general and the use of 4-nitrophenylpiperazine in particular, the present paper reports the crystal structures of some salts of 4-nitrophenylpiperazine with organic acids. The crystal structures of 4-(4-nitrophenyl)piperazin-1-ium 4-bromobenzoate dihydrate (I), 4-(4-nitrophenyl)piperazin-1-ium 4-iodobenzoate dihydrate (II), 4-(4-nitrophenyl)piperazin-1-ium 4-hydroxybenzoate monohydrate (III), 4-(4-nitrophenyl)piperazin-1-ium

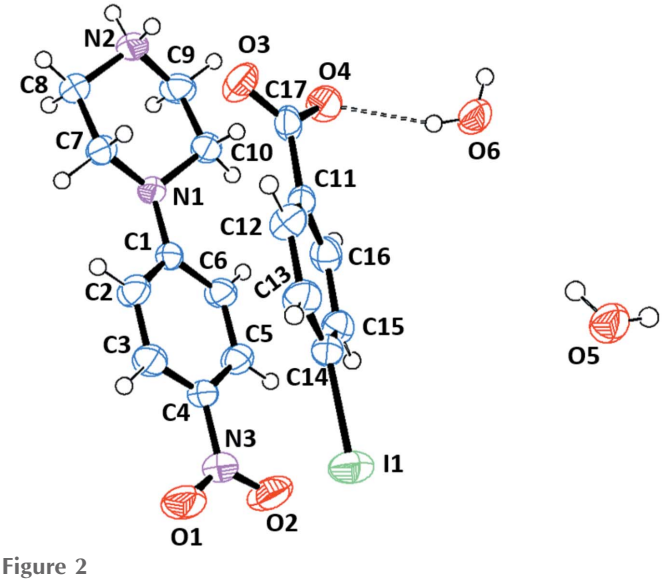


Figure 2
The independent components of compound (II) showing the atom-labelling scheme and the hydrogen bonds, drawn as dashed lines. Displacement ellipsoids are drawn at the 50% probability level.

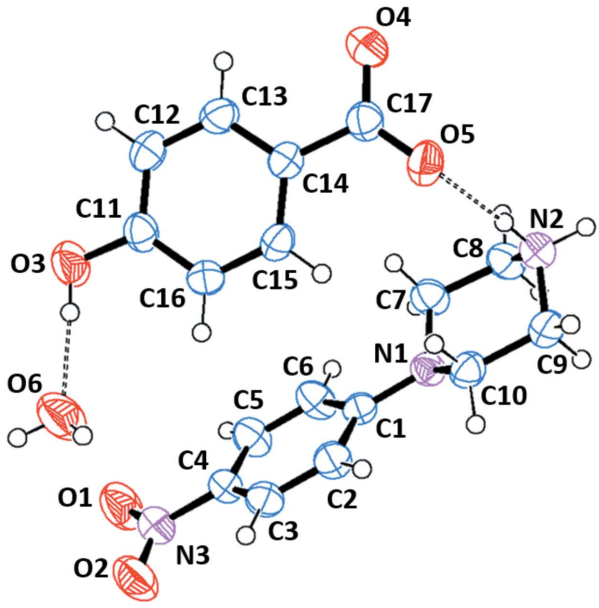


Figure 3
The independent components of compound (III) showing the atom-labelling scheme and the hydrogen bonds, drawn as dashed lines. Displacement ellipsoids are drawn at the 50% probability level.

4-methylbenzoate monohydrate (IV), 4-(4-nitrophenyl)piperazin-1-ium 4-methoxybenzoate hemihydrate (V) and 4-(4-nitrophenyl)piperazin-1-ium 4-ethoxybenzoate (VI) are reported herein.

2. Structural commentary

The asymmetric units of the title salts are shown in Figs. 1–6. They include 1:1 dihydrated salts [(I), (II)], 1:1 monohydrated salts [(III), (IV)], 2:2 monohydrated salt (V) and solvent-free

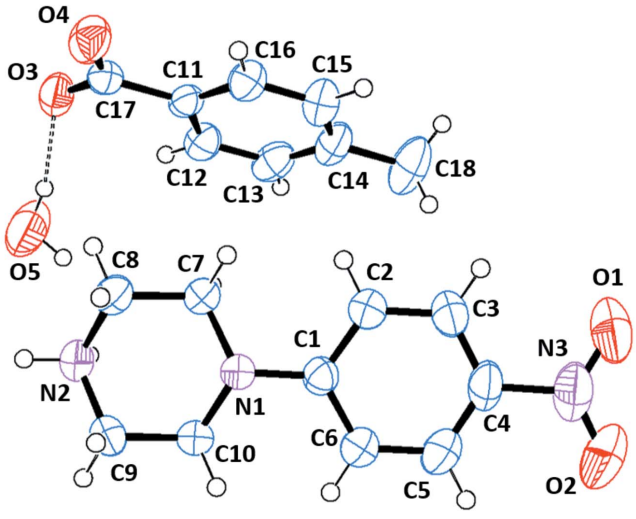


Figure 4
The independent components of compound (IV) showing the atom-labelling scheme and the hydrogen bonds, drawn as dashed lines. Displacement ellipsoids are drawn at the 50% probability level.

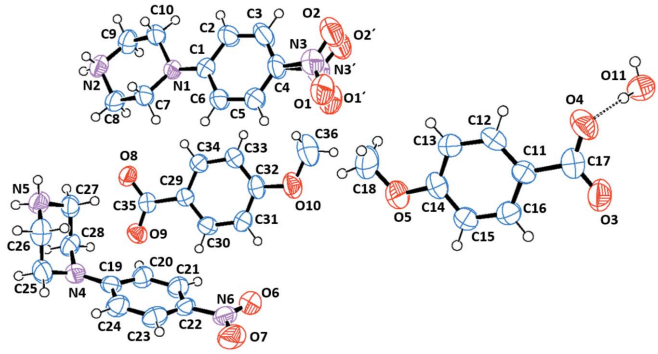


Figure 5
The independent components of compound (V) showing the atom-labelling scheme and the hydrogen bonds, drawn as dashed lines. Displacement ellipsoids are drawn at the 50% probability level.

2:2 salt (VI). Compounds (I) and (II) are isostructural. In all salts, the cation is common and consists of a protonated chair-shaped piperazine ring (N1/N2/C7–C10), which makes dihedral angles of 10.91 (1), 12.13 (1), 14.82 (6), 3.11 (8), 5.73 (1) and 13.08 (9)°, respectively, for compounds (I)–(VI) with the nitrobenzene moiety (N3/O1/O2/C1–C6) and exhibits a maximum deviation from its mean plane at atom N2 of −0.253 (2), 0.254 (2), 0.288 (2), 0.278 (2), 0.241 (3) and 0.303 (3) Å in (I)–(VI), respectively. The piperazine rings of the additional cations (N4/N5/C25–C28) in compounds (V) and (VI) have the same conformation, making dihedral angles of 64.53 (1) and 21.70 (1)°, respectively, with the nitrobenzene moieties (N6/O6/O7/C19–C25). Within the cations, the benzene rings are almost planar, with maximum deviations from mean plane ranging from −0.016 (3) Å at atom C20 for (VI) to 0.003 (2) Å at atom C4 for (III). The *p*-nitro substituent groups deviate significantly from planes of the benzene rings in all compounds except the (C1–C6) ring of (VI). The anions of the title salts are formed from a benzoate anion with different *p*-substituents for each compound that deviate significantly from planarity, with maximum deviations of 0.045 (1) Å at Br1 for (I), 0.063 (1) Å at I1 for (II), −0.021 (2) Å at hydroxyl atom O3 for (III), −0.010 (1) Å at methyl atom C18 for (IV), −0.033 (1) and 0.034 (1) Å at methoxy atoms O5 and O10 for (V) and −0.025 (2) and −0.013 (2) Å at ethoxy atoms O5 and O10 for (VI).

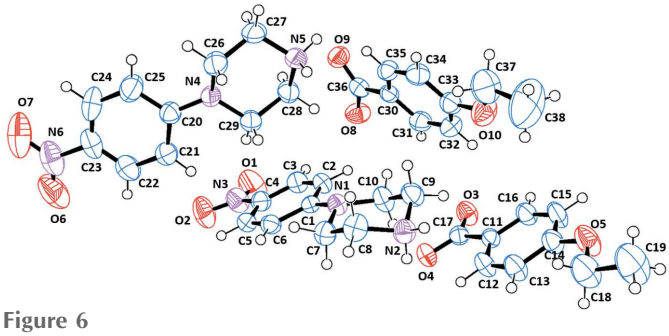


Figure 6
The independent components of compound (VI) showing the atom-labelling scheme Displacement ellipsoids are drawn at the 50% probability level.

Table 1
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H21···O5^{i}	0.85 (2)	1.99 (2)	2.810 (3)	162 (3)
N2—H22···O6^{ii}	0.83 (2)	1.91 (2)	2.707 (3)	160 (3)
C3—H3···O1^{iii}	0.93	2.59	3.260 (4)	130
C13—H13···O4^{iv}	0.93	2.57	3.483 (3)	166
C15—H15···O2^{v}	0.93	2.47	3.269 (4)	144
O5—H1*W*···O3^{vi}	0.80 (2)	1.97 (2)	2.759 (2)	169 (3)
O5—H2*W*···O3^{i}	0.80 (2)	2.00 (2)	2.772 (2)	161 (3)
O6—H4*W*···O4	0.82 (2)	2.03 (2)	2.832 (3)	166 (3)
O6—H3*W*···O4^{vii}	0.78 (2)	1.99 (2)	2.760 (3)	169 (3)

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x, y+1, z$; (iii) $-x+2, -y+2, -z+1$; (iv) $x+1, y, z$; (v) $-x+1, -y+1, -z+1$; (vi) $x, y-1, z$; (vii) $-x, -y+1, -z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H21···O5^{i}	0.86 (2)	1.99 (2)	2.825 (4)	164 (4)
N2—H22···O6^{ii}	0.85 (2)	1.88 (2)	2.702 (3)	163 (4)
C3—H3···O1^{iii}	0.93	2.59	3.275 (4)	131
C13—H13···O4^{iv}	0.93	2.62	3.526 (4)	166
C15—H15···O2^{v}	0.93	2.49	3.311 (4)	147
O5—H1*W*···O3^{vi}	0.81 (2)	1.96 (2)	2.756 (3)	170 (4)
O5—H2*W*···O3^{i}	0.81 (2)	1.96 (2)	2.753 (3)	166 (4)
O6—H4*W*···O4	0.80 (2)	2.08 (2)	2.836 (3)	160 (4)
O6—H3*W*···O4^{vii}	0.80 (2)	1.95 (2)	2.728 (3)	165 (4)

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x, y+1, z$; (iii) $-x+2, -y+2, -z+1$; (iv) $x+1, y, z$; (v) $-x+1, -y+1, -z+1$; (vi) $x, y-1, z$; (vii) $-x, -y+1, -z$.

3. Supramolecular features

In the crystal structures of the two isomorphous salts (I) and (II), the ions are arranged in chains perpendicular to the *a*-axis direction. The water molecules play an essential role in holding the chains together, forming complex sheets in the *bc* plane (Figs. 7 and 8, Tables 1 and 2). The cations and anions in

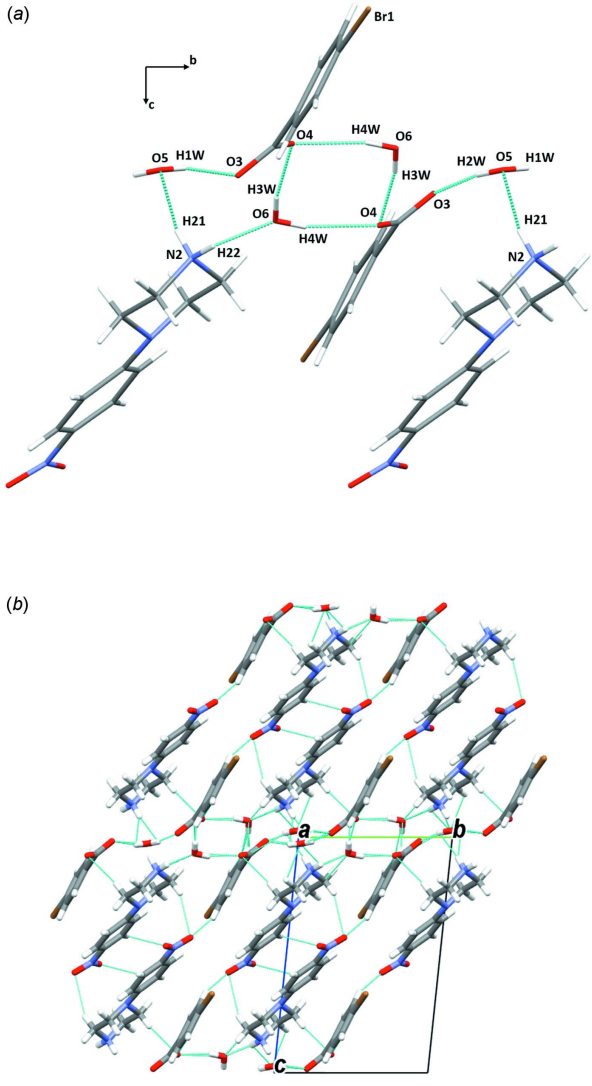


Figure 7
(*a*) A general view of the main intermolecular interactions (N—H···O and O—H···O) and (*b*) the molecular packing of (I) with hydrogen bonds shown as dashed lines.

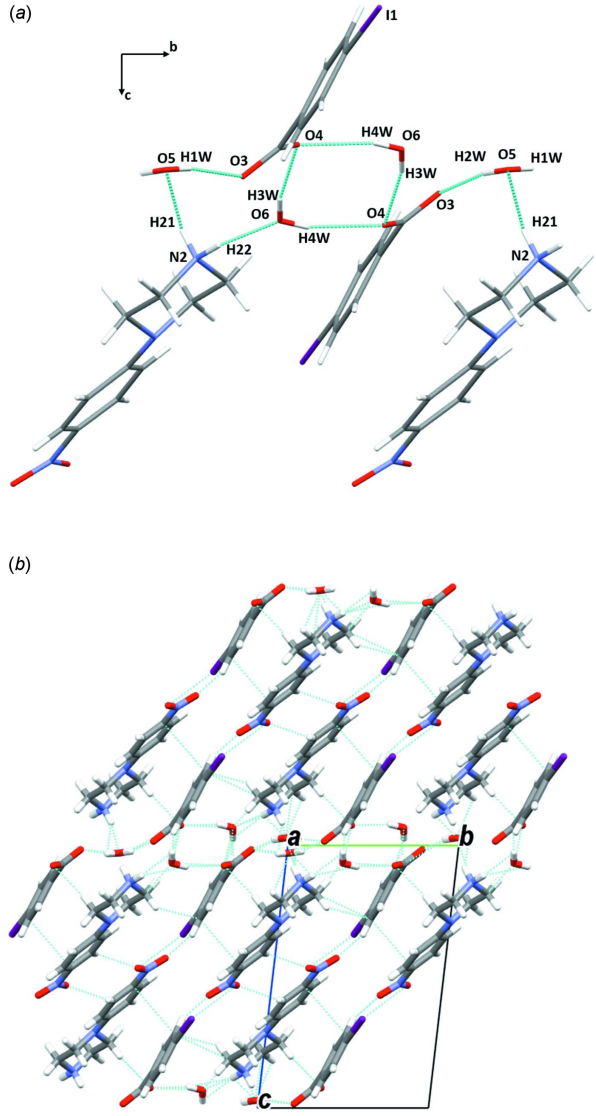


Figure 8
(*a*) A general view of the main intermolecular interactions (N—H···O and O—H···O) in (II) and (*b*) the molecular packing of (II) with hydrogen bonds shown as dashed lines.

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H21···O5	0.89 (2)	1.93 (2)	2.819 (2)	177 (3)
N2—H22···O4^{i}	0.94 (2)	1.65 (2)	2.583 (2)	177 (3)
O3—H17···O6	0.85 (2)	1.82 (2)	2.669 (2)	177 (3)
O6—H1*W*···O5^{ii}	0.83 (2)	1.95 (2)	2.768 (2)	169 (3)
O6—H2*W*···O1^{iii}	0.83 (2)	2.11 (2)	2.944 (2)	178 (3)

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

(III) are linked through strong O—H···O and N—H···O hydrogen bonds, forming chains along the [011] direction (Fig. 9*a*, Table 3). These chains are further linked *via* the water molecules and C9—H9*A*···O3 interactions, generating a three-dimensional supramolecular architecture along the *a* axis (Fig. 9*b*). The structure of (IV) is constructed from double

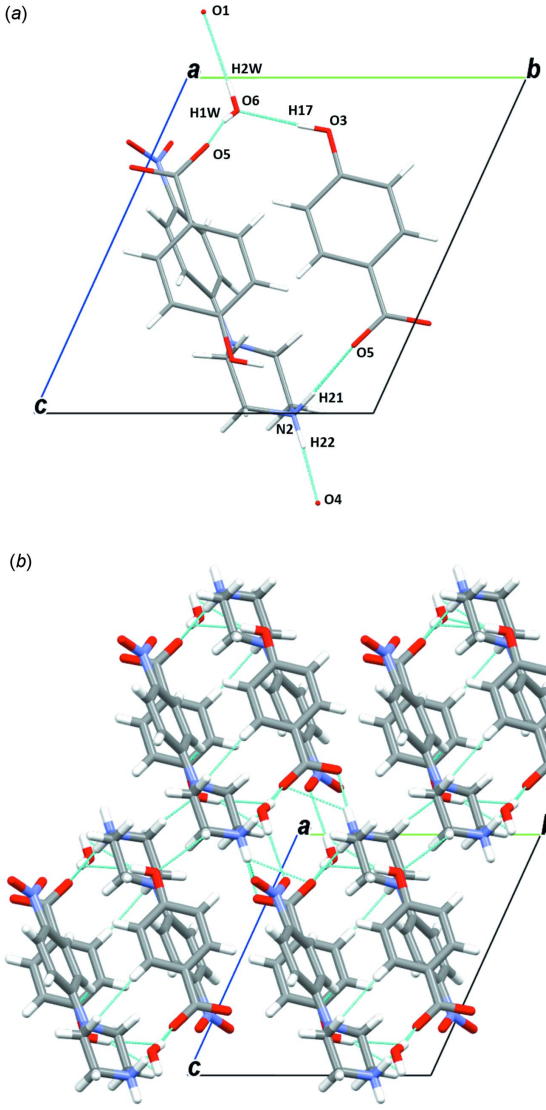


Figure 9
(*a*) A general view of the main intermolecular interactions (N—H···O and O—H···O) in (III) and (*b*) the molecular packing of (III) with hydrogen bonds shown as dashed lines.

Table 4
Hydrogen-bond geometry (Å, °) for (IV).

*Cg*3 is the centroids of the C11–C16 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H21···O4^{i}	0.89 (2)	1.93 (2)	2.811 (3)	167 (4)
N2—H22···O3^{ii}	0.91 (2)	1.81 (2)	2.717 (3)	177 (4)
C3—H3···O1^{iii}	0.93	2.54	3.427 (4)	161
C9—H9*A*···O5^{iv}	0.97	2.31	3.113 (3)	140
O5—H1*W*···O4^{i}	0.84 (2)	1.92 (2)	2.756 (3)	171 (4)
O5—H2*W*···O3	0.85 (2)	1.94 (2)	2.772 (3)	164 (4)
C6—H6···*Cg*3^{v}	0.93	2.93	3.590 (3)	129

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

chains running along the [101] direction. Each chain is formed by linking the molecules through a combination of N—H···O, O—H···O and C—H···O interactions (Fig. 10*a*, Table 4); the resulting double chains are symmetrically related by an inversion center and are connected *via* N2—H21···O4 and C7—H7*A*···O4 interactions. These hydrated double chains are weakly linked into sheets lying in the *bc* plane by C—H···π (arene) interactions (Fig. 10*b*). The supramolecular assembly of compound (V), which has a disordered nitro group, is built up of N2—H22*N*···O11, O11—H11*O*···O4 and N5—H51···O9 hydrogen bonds linking the ions into organic chains running parallel to the [010] direction (Fig. 11*a*, Table 5). The chains are further connected cooperatively through other interactions of type N—H···O, generating a multilayer network along the *b*-axis direction (Fig. 11*b*). In compound (VI), a set of N—H···O, C—H···O and C—H···π interactions (Fig. 12*a*, Table 6) link the molecules into cationic and anionic layers running parallel to the *b*-axis direction and join these layer motifs, generating the complete molecular structure along the *a* axis (Fig. 12*b*).

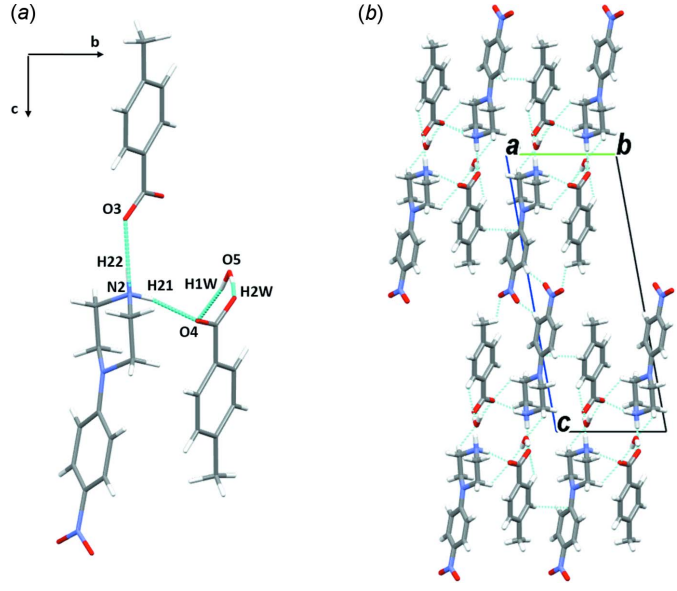


Figure 10
(*a*) A general view of the main intermolecular interactions (N—H···O and O—H···O) in (IV) and (*b*) the molecular packing of (IV) with hydrogen bonds shown as dashed lines.

Table 5
Hydrogen-bond geometry (Å, °) for (V).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7B···O7^{i}	0.97	2.54	3.451 (5)	157
C9—H9B···O4^{ii}	0.97	2.31	3.270 (5)	169
C20—H20···O9	0.93	2.53	3.461 (5)	174
C25—H25A···O2a^{iii}	0.97	2.5	3.206 (10)	130
C25—H25A···O2′b^{iii}	0.97	2.49	3.212 (11)	131
C27—H27A···O7^{i}	0.97	2.58	3.548 (5)	175
C28—H28B···O9	0.97	2.55	3.489 (5)	164
C36—H36C···O1′b^{ii}	0.96	2.49	3.395 (14)	158
N2—H21N···O8^{iv}	0.88 (2)	1.83 (2)	2.697 (4)	166 (4)
N2—H21N···O9^{iv}	0.88 (2)	2.57 (3)	3.196 (4)	129 (3)
N2—H22N···O11^{v}	0.88 (2)	1.89 (2)	2.758 (5)	169 (4)
N5—H51N···O9^{vi}	0.87 (2)	1.93 (2)	2.778 (5)	164 (4)
N5—H52N···O3^{vii}	0.91 (2)	1.82 (2)	2.724 (5)	171 (4)
O11—H11O···O4	0.84 (2)	1.83 (2)	2.663 (4)	176 (4)
O11—H12O···O8^{v}	0.84 (2)	1.92 (2)	2.754 (4)	173 (4)

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

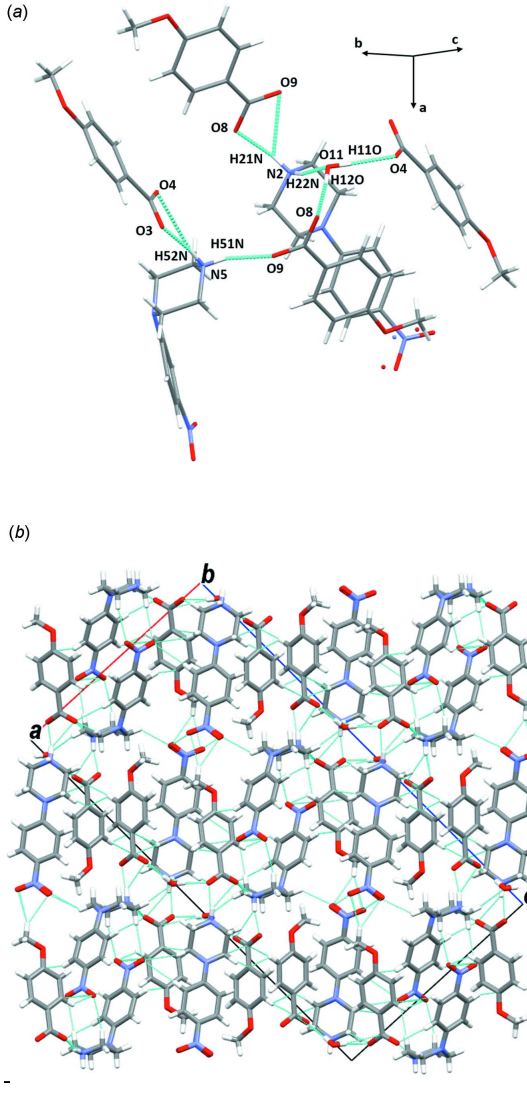


Figure 11
(*a*) A general view of the main intermolecular interactions (N—H···O and O—H···O) in (V) and (*b*) the molecular packing of (V) with hydrogen bonds shown as dashed lines.

Table 6
Hydrogen-bond geometry (Å, °) for (VI).

*Cg*2 and *Cg*6 are the centroids of the C1–C6 and C30–C35 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H31N···O3^{i}	0.94 (2)	1.68 (2)	2.613 (3)	172 (5)
N2—H31N···O4^{i}	0.94 (2)	2.51 (4)	3.157 (3)	127 (4)
N2—H32N···O9^{ii}	0.90 (2)	1.96 (2)	2.843 (3)	171 (5)
N5—H61N···O8^{i}	0.91 (2)	1.78 (2)	2.686 (3)	175 (5)
N5—H61N···O9^{i}	0.91 (2)	2.59 (4)	3.174 (3)	122 (4)
N5—H62N···O4^{iii}	0.90 (2)	1.83 (2)	2.708 (3)	165 (5)
C22—H22···O2^{iv}	0.93	2.6	3.502 (5)	165
C27—H27B···O9^{i}	0.97	2.59	3.215 (3)	123
C28—H28B···O7^{v}	0.97	2.65	3.410 (4)	135
C29—H29B···O1^{i}	0.97	2.53	3.249 (4)	131
C35—H35···O4^{iii}	0.93	2.52	3.263 (3)	137
C10—H10A···Cg6	0.97	2.82	3.746 (3)	159
C29—H29A···Cg2	0.97	2.76	3.556 (3)	139

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

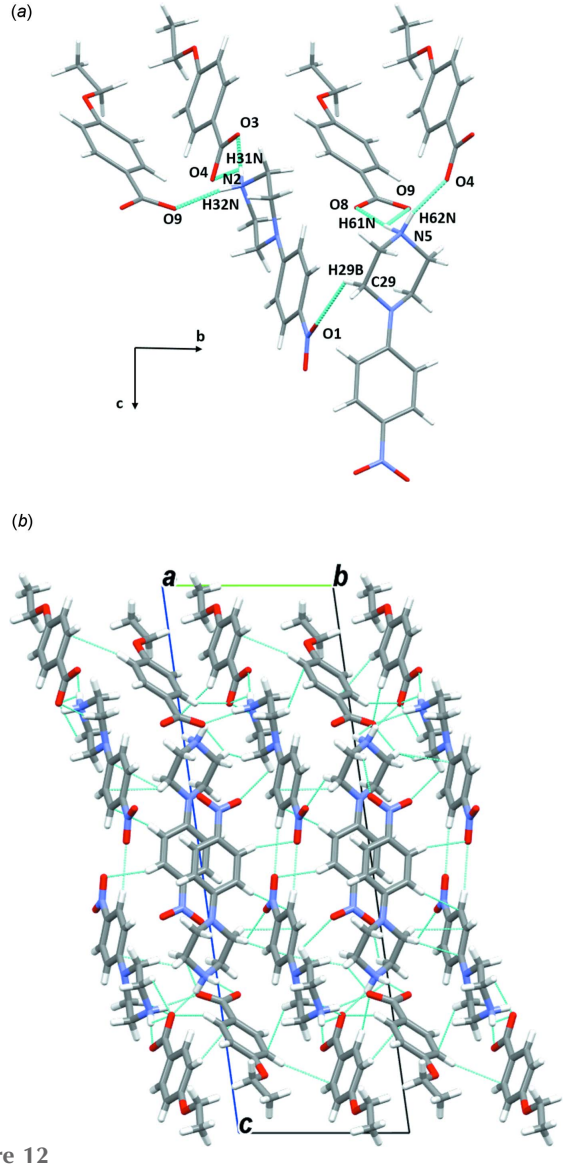


Figure 12
(*a*) A general view of the main intermolecular interactions (N—H···O, O—H···O and C—H···O) in (VI) and (*b*) the molecular packing of (VI) with hydrogen bonds shown as dashed lines.

Table 7
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{10}H_{14}N_3O_2^+\cdot C_7H_4BrO_2^-\cdot 2H_2O$	$C_{10}H_{14}N_3O_2^+\cdot C_7H_4IO_2^-\cdot 2H_2O$	$C_{10}H_{14}N_3O_2^+\cdot C_7H_5O_3^-\cdot H_2O$
M_r	444.28	491.28	363.37
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	293	293	293
a, b, c (Å)	7.738 (1), 9.320 (1), 13.949 (2)	7.7652 (4), 9.2852 (5), 13.930 (1)	9.636 (1), 10.301 (1), 10.867 (1)
α, β, γ (°)	94.46 (1), 95.04 (1), 104.71 (2)	94.985 (5), 95.331 (5), 104.875 (6)	103.90 (1), 108.32 (1), 112.96 (1)
V (Å^3)	964.0 (2)	960.09 (10)	857.80 (17)
Z	2	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.17	1.71	0.11
Crystal size (mm)	0.48 × 0.44 × 0.24	0.48 × 0.48 × 0.2	0.50 × 0.32 × 0.24
Data collection			
Diffractometer	Oxford Diffraction Xcalibur	Oxford Diffraction Xcalibur	Oxford Diffraction Xcalibur
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min}, T_{max}	0.367, 0.422	0.458, 0.711	0.959, 0.974
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6123, 3536, 2520	6331, 3518, 2952	5342, 3140, 2342
R_{int}	0.019	0.017	0.013
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602	0.602	0.602
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.104, 1.04	0.029, 0.069, 1.03	0.043, 0.106, 1.05
No. of reflections	3528	3513	3135
No. of parameters	262	262	251
No. of restraints	6	6	5
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.50, −0.51	0.54, −0.66	0.19, −0.19

	(IV)	(V)	(VI)
Crystal data			
Chemical formula	$C_{10}H_{14}N_3O_2^+\cdot C_8H_7O_2^-\cdot H_2O$	$2C_{10}H_{14}N_3O_2^+\cdot 2C_8H_7O_3^-\cdot H_2O$	$C_{10}H_{14}N_3O_2^+\cdot C_9H_9O_3^-$
M_r	361.39	736.77	373.4
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$
Temperature (K)	293	293	293
a, b, c (Å)	6.1136 (5), 7.6965 (7), 19.708 (2)	15.808 (1), 7.5198 (7), 31.020 (2)	7.874 (1), 9.263 (1), 27.996 (3)
α, β, γ (°)	79.577 (8), 87.162 (8), 86.699 (8)	90, 92.561 (7), 90	81.030 (6), 85.675 (6), 68.229 (5)
V (Å^3)	909.79 (15)	3683.8 (5)	1872.8 (4)
Z	2	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.10	0.1	0.10
Crystal size (mm)	0.48 × 0.26 × 0.02	0.5 × 0.36 × 0.36	0.44 × 0.32 × 0.08
Data collection			
Diffractometer	Oxford Diffraction Xcalibur	Oxford Diffraction Xcalibur	Oxford Diffraction Xcalibur
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min}, T_{max}	0.970, 0.998	0.958, 0.965	0.963, 0.992
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5980, 3347, 1911	15326, 6718, 2602	13344, 6868, 3803
R_{int}	0.019	0.066	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602	0.602	0.602
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.053, 0.138, 1.01	0.074, 0.169, 1.00	0.061, 0.137, 1.05
No. of reflections	3343	6715	6858
No. of parameters	248	507	501
No. of restraints	4	45	16
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.16	0.27, −0.18	0.23, −0.22

Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2009), *CrysAlis RED* (Oxford Diffraction, 2009), *SHELXT* (Sheldrick, 2015*a*), *Mercury* (Macrae *et al.*, 2020), *SHELXL2014* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

4. Database survey

A search of the Cambridge Structural Database (Version 2020.3, last update February 2022; Groom *et al.*, 2016) for the phenyl piperazinium cation and *para* substituent benzoate anion involved in the reported six salts gave the following hits, 4-(4-methoxyphenyl)piperazin-1-ium 4-fluorobenzoate monohydrate, 4-(4-methoxyphenyl)piperazin-1-ium 4-chlorobenzoate monohydrate and 4-(4-methoxyphenyl)piperazin-1-ium 4-bromobenzoate monohydrate (FOVPOY, FOVPUE and FOVQAL; Kiran Kumar *et al.*, 2019) and 4-(4-methoxyphenyl)piperazin-1-ium 4-iodobenzoate monohydrate (KUJPUD; Kiran Kumar *et al.*, 2020). They exhibit a methoxy group as a substituent in the phenyl piperazinium cation rather than a nitro group as in the title compounds (I)–(VI) and they also crystallize as monohydrates similar to compounds (III)–(V). Although the title compounds (I) and (II) have halogen-based anions and chain-based structures, they are not isostructural with the above compounds, the crystal structures of which are based on differently sized chains of rings formed *via* a combination of hydrogen bonds of type N—H···O and O—H···O and other weak interactions of type C—H···O and C—H···π to form sheets. In 4-(4-methoxyphenyl)piperazin-1-ium 4-aminobenzoate monohydrate (IHIMEU; Kiran Kumar *et al.*, 2020) the presence of an amino substituent on the anion, which acts as both a donor and an acceptor of hydrogen bonds, makes the supramolecular assembly of this compound more complex than for the compounds reported herein.

5. Synthesis and crystallization

Synthesis:

For the synthesis of salts (I)–(VI), a solution of commercially available (from Sigma–Aldrich) 4-nitrophenylpiperazine (100 mg, 0.483 mol) in methanol (10 ml) was mixed with equimolar solutions of the appropriate acids in methanol (10 ml) and ethyl acetate (10 ml), *viz*.4-bromobenzoic acid (97 mg, 0.483 mol) for (I), 4-iodobenzoic acid (120 mg, 0.483 mol) for (II), 4-hydroxybenzoic acid (67 mg, 0.483 mol) for (III), 4-methylbenzoic acid (66 mg, 0.483 mol) for (IV), 4-methoxybenzoic acid (73 mg, 0.483 mol) for (V) and 4-ethoxybenzoicacid (80 mg, 0.483 mol) for (VI). The corresponding solutions were stirred for 15 minutes at room temperature and allowed to stand at the same temperature. The products obtained were subjected to crystallization.

Crystallization: Crystallization was carried out using the slow evaporation technique. X-ray quality crystals were formed on slow evaporation in a week for all compounds, where ethanol:ethylacetate (1:1) was used for crystallization. The corresponding melting points were 430–432 K (I), 453–455 K (II), 446–448 K (III), 398–400 K (IV), 413–415 K (V) and 408–410 K (VI).

6. Refinement

Crystal data, data collection and refinement details are summarized in Table 7. C-bound H atoms were positioned with idealized geometry and refined using a riding model with C—H = 0.93 Å (aromatic), 0.96 Å (methyl) or 0.97 Å (methylene). The H atoms on the N atom were located in a difference map and later restrained to N—H = 0.86 (2) Å. All H atoms were refined with isotropic displacement parameters set at 1.2 U_{eq} (C-aromatic, C-methylene, N) or 1.5 U_{eq} (C-methyl) times those of the parent atom. For the disordered nitro group in (V), the component atoms were restrained to have the same U^{ij} components and the occupancy ratio is 0.519 (6):0.481 (6).

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References

Archana, S. D., Kumar, H. K., Yathirajan, H. S., Foro, S., Abdelbaky, M. S. M. & Garcia-Granda, S. (2021). *Acta Cryst.* E**77**, 1135–1139.
O. Ayeni, A., M. Watkins, G. & C. Hosten, E. (2019). *Bull. Chem. Soc. Eth.* **33**, 341.
Berkheij, M., van der Sluis, L., Sewing, C., den Boer, D. J., Terpstra, J. W., Hiemstra, H., Iwema Bakker, W. I., van den Hoogenband, A. & van Maarseveen, J. H. (2005). *Tetrahedron Lett.* **46**, 2369–2371.
Bogatcheva, E., Hanrahan, C., Nikonenko, B., Samala, R., Chen, P., Gearhart, J., Barbosa, F., Einck, L., Nacy, C. A. & Protopopova, M. (2006). *J. Med. Chem.* **49**, 3045–3048.
Brockunier, L. L., He, J., Colwell, L. F. Jr, Habulihaz, B., He, H., Leiting, B., Lyons, K. A., Marsilio, F., Patel, R. A., Teffera, Y., Wu, J. K., Thornberry, N. A., Weber, A. E. & Parmee, E. R. (2004). *Bioorg. Med. Chem. Lett.* **14**, 4763–4766.
Chaudhary, P., Kumar, R., Verma, K., Singh, D., Yadav, V., Chhillar, A. K., Sharma, G. L. & Chandra, R. (2006). *Bioorg. Med. Chem.* **14**, 1819–1826.
Elliott, S. (2011). *Drug Test. Anal.* **3**, 430–438.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Gupta, S., Pandey, D., Mandalapu, D., Bala, V., Sharma, V., Shukla, M., Yadav, S. K., Singh, N., Jaiswal, S., Maikhuri, J. P., Lal, J., Siddiqi, M. I., Gupta, G. & Sharma, V. L. (2016). *Med. Chem. Commun.* **7**, 2111–2121.
Harish Chinthal, C., Kavitha, C. N., Yathirajan, H. S., Foro, S., Rathore, R. S. & Glidewell, C. (2020). *Acta Cryst.* E**76**, 1779–1793.
Kaya, B., Ozkay, Y., Temel, H. E. & &Kaplancikli, Z. A. (2016). *J. Chem. Art. ID*, pp. 5878410 http://dx.doi.org/10.1155/2016/5878410.
Kharb, R., Bansal, K. & Sharma, A. K. (2012). *Der Pharma Chem.* **4**, 2470–2488.
Kiran Kumar, H., Yathirajan, H. S., Foro, S. & Glidewell, C. (2019). *Acta Cryst.* E**75**, 1494–1506.
Kiran Kumar, H., Yathirajan, H. S., Harish Chinthal, C., Foro, S. & Glidewell, C. (2020). *Acta Cryst.* E**75**, 488–495.
Lu, Y.-X. (2007). *Acta Cryst.* E**63**, o3611.
Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.
Upadhayaya, P. S., Sinha, N., Jain, S., Kishore, N., Chandra, R. & Arora, S. K. (2004). *Bioorg. Med. Chem.* **12**, 2225–2238.
Wang, X.-Y., Wang, M.-Z., Guo, F.-J., Sun, J. & Qian, S.-S. (2014). *Z. Kristallogr. New Cryst. Struct.* **229**, 97–98.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Wodtke, R., Steinberg, J., Köckerling, M., Löser, R. & Mamat, C. (2018). *RSC Adv.* **8**, 40921–40933.
Zhang, Y., Chao, J., Zhao, S., Xu, P., Wang, H., Guo, Z. & Liu, D. (2014). *Spectrochim. Acta Part A*, **132**, 44–51.

supporting information

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Crystal structures of six 4-(4-nitrophenyl)piperazin-1-ium salts

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Computing details

For all structures, data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020). Software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010) for (I), (II), (III), (V), (VI); *SHELXL2014* (Sheldrick, 2015b),*PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010) for (IV).

4-(4-Nitrophenyl)piperazin-1-ium 4-bromobenzoate dihydrate (I)

Crystal data

$C_{10}H_{14}N_3O_2^+ \cdot C_7H_4BrO_2^- \cdot 2H_2O$
$M_r = 444.28$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.738$ (1) Å
$b = 9.320$ (1) Å
$c = 13.949$ (2) Å
$\alpha = 94.46$ (1)°
$\beta = 95.04$ (1)°
$\gamma = 104.71$ (2)°
$V = 964.0$ (2) Å^3
$Z = 2$
$F(000) = 456$
$D_x = 1.531$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6123 reflections
$\theta = 3.0$–25.3°
$\mu = 2.17$ mm^{-1}
$T = 293$ K
Prism, yellow
0.48 × 0.44 × 0.24 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)
$T_{min} = 0.367$, $T_{max} = 0.422$
6123 measured reflections
3536 independent reflections
2520 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 25.3°$, $\theta_{min} = 3.0°$
$h = -9 \rightarrow 8$
$k = -6 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.104$
$S = 1.04$
3528 reflections
262 parameters
6 restraints
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: structure-invariant direct methods
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.9273 (3)	0.7891 (3)	0.53814 (19)	0.0873 (8)
O2	0.6916 (3)	0.6357 (3)	0.57409 (17)	0.0767 (6)
N1	0.3360 (2)	1.0060 (2)	0.29246 (14)	0.0392 (5)
N2	0.1306 (3)	1.1025 (2)	0.13987 (16)	0.0433 (5)
N3	0.7641 (3)	0.7395 (3)	0.53118 (17)	0.0537 (6)
C1	0.4412 (3)	0.9374 (2)	0.34950 (16)	0.0353 (5)
C2	0.6297 (3)	0.9791 (3)	0.35426 (19)	0.0462 (6)
H2	0.685335	1.050743	0.316027	0.055*
C3	0.7335 (3)	0.9160 (3)	0.4144 (2)	0.0479 (6)
H3	0.858174	0.947226	0.418128	0.057*
C4	0.6524 (3)	0.8067 (3)	0.46921 (17)	0.0401 (6)
C5	0.4678 (3)	0.7621 (3)	0.46618 (18)	0.0460 (6)
H5	0.414057	0.688671	0.503722	0.055*
C6	0.3636 (3)	0.8264 (3)	0.40761 (18)	0.0430 (6)
H6	0.239197	0.796118	0.406198	0.052*
C7	0.4258 (3)	1.1047 (3)	0.2239 (2)	0.0479 (6)
H7A	0.454966	1.044787	0.17075	0.058*
H7B	0.537417	1.169773	0.256358	0.058*
C8	0.3088 (3)	1.1978 (3)	0.1846 (2)	0.0493 (7)
H8A	0.29158	1.266385	0.236558	0.059*
H8B	0.368224	1.256083	0.136469	0.059*
C9	0.0406 (3)	1.0114 (3)	0.2121 (2)	0.0495 (6)
H9A	−0.074749	0.948618	0.182599	0.059*
H9B	0.01919	1.076165	0.265103	0.059*
C10	0.1561 (3)	0.9148 (3)	0.2506 (2)	0.0462 (6)
H10A	0.097528	0.858526	0.29974	0.055*
H10B	0.168666	0.844357	0.1984	0.055*
Br1	0.91659 (5)	0.45794 (4)	0.33416 (3)	0.08360 (18)
O3	0.4483 (2)	0.78822 (19)	0.01696 (15)	0.0543 (5)
O4	0.2197 (2)	0.63974 (18)	0.07787 (14)	0.0505 (5)
C11	0.5153 (3)	0.6356 (2)	0.13620 (18)	0.0350 (5)
C12	0.6851 (3)	0.6376 (3)	0.10986 (19)	0.0431 (6)
H12	0.71881	0.674975	0.052451	0.052*
C13	0.8046 (3)	0.5846 (3)	0.1680 (2)	0.0499 (7)
H13	0.916747	0.583844	0.149142	0.06*

C14	0.7551 (3)	0.5332 (3)	0.2538 (2)	0.0466 (6)
C15	0.5891 (3)	0.5325 (3)	0.28271 (19)	0.0459 (6)
H15	0.55828	0.499101	0.341588	0.055*
C16	0.4690 (3)	0.5821 (2)	0.22315 (19)	0.0424 (6)
H16	0.35572	0.579557	0.241532	0.051*
C17	0.3848 (3)	0.6919 (2)	0.07250 (19)	0.0391 (6)
O5	0.7579 (2)	0.01738 (19)	0.02770 (14)	0.0468 (4)
O6	0.0305 (3)	0.3352 (2)	0.06917 (16)	0.0581 (5)
H21	0.143 (4)	1.051 (3)	0.0892 (17)	0.07*
H22	0.075 (4)	1.160 (3)	0.118 (2)	0.07*
H1W	0.674 (3)	−0.053 (3)	0.031 (2)	0.07*
H2W	0.720 (4)	0.088 (3)	0.018 (2)	0.07*
H4W	0.095 (4)	0.421 (2)	0.080 (2)	0.07*
H3W	−0.041 (4)	0.331 (3)	0.0257 (18)	0.07*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0490 (13)	0.0892 (16)	0.122 (2)	0.0142 (11)	−0.0189 (13)	0.0497 (15)
O2	0.0701 (14)	0.0858 (15)	0.0809 (16)	0.0235 (12)	−0.0004 (12)	0.0500 (13)
N1	0.0347 (10)	0.0406 (11)	0.0417 (11)	0.0091 (8)	−0.0042 (9)	0.0124 (9)
N2	0.0459 (12)	0.0431 (12)	0.0435 (13)	0.0189 (10)	−0.0065 (10)	0.0088 (10)
N3	0.0542 (15)	0.0524 (13)	0.0551 (14)	0.0173 (11)	−0.0090 (12)	0.0161 (11)
C1	0.0396 (13)	0.0344 (12)	0.0327 (13)	0.0132 (10)	−0.0010 (10)	0.0026 (10)
C2	0.0378 (14)	0.0518 (15)	0.0515 (16)	0.0111 (11)	0.0047 (12)	0.0229 (12)
C3	0.0361 (13)	0.0503 (15)	0.0583 (17)	0.0125 (11)	−0.0001 (12)	0.0145 (13)
C4	0.0441 (14)	0.0411 (13)	0.0363 (13)	0.0156 (11)	−0.0037 (11)	0.0060 (11)
C5	0.0489 (15)	0.0463 (14)	0.0429 (15)	0.0100 (11)	0.0016 (12)	0.0177 (11)
C6	0.0339 (12)	0.0498 (14)	0.0455 (15)	0.0093 (11)	0.0025 (11)	0.0145 (12)
C7	0.0416 (14)	0.0450 (14)	0.0524 (16)	0.0036 (11)	−0.0082 (12)	0.0166 (12)
C8	0.0558 (16)	0.0385 (13)	0.0499 (16)	0.0096 (12)	−0.0116 (13)	0.0117 (11)
C9	0.0392 (14)	0.0603 (16)	0.0517 (16)	0.0187 (12)	−0.0040 (12)	0.0138 (13)
C10	0.0354 (13)	0.0487 (14)	0.0530 (16)	0.0077 (11)	−0.0030 (12)	0.0159 (12)
Br1	0.0734 (3)	0.0942 (3)	0.0856 (3)	0.0319 (2)	−0.02375 (19)	0.0272 (2)
O3	0.0418 (10)	0.0434 (10)	0.0808 (13)	0.0119 (8)	0.0005 (9)	0.0298 (9)
O4	0.0291 (9)	0.0497 (10)	0.0727 (13)	0.0090 (7)	0.0003 (8)	0.0169 (9)
C11	0.0330 (12)	0.0242 (11)	0.0468 (14)	0.0078 (9)	−0.0007 (11)	0.0021 (10)
C12	0.0403 (14)	0.0432 (13)	0.0504 (16)	0.0160 (11)	0.0087 (12)	0.0125 (11)
C13	0.0345 (13)	0.0548 (15)	0.0659 (19)	0.0194 (11)	0.0057 (13)	0.0145 (14)
C14	0.0443 (15)	0.0381 (13)	0.0548 (17)	0.0096 (11)	−0.0109 (13)	0.0102 (12)
C15	0.0500 (16)	0.0433 (14)	0.0403 (15)	0.0053 (11)	−0.0007 (12)	0.0082 (11)
C16	0.0376 (13)	0.0378 (13)	0.0499 (16)	0.0068 (10)	0.0051 (12)	0.0034 (11)
C17	0.0375 (13)	0.0266 (11)	0.0533 (16)	0.0108 (10)	−0.0010 (11)	0.0032 (11)
O5	0.0370 (10)	0.0413 (10)	0.0619 (12)	0.0103 (7)	−0.0023 (9)	0.0133 (9)
O6	0.0491 (12)	0.0429 (10)	0.0816 (15)	0.0141 (8)	−0.0142 (10)	0.0195 (10)

Geometric parameters (Å, º)

O1—N3	1.222 (3)	C8—H8B	0.97
O2—N3	1.217 (3)	C9—C10	1.516 (3)
N1—C1	1.391 (3)	C9—H9A	0.97
N1—C7	1.474 (3)	C9—H9B	0.97
N1—C10	1.477 (3)	C10—H10A	0.97
N2—C9	1.476 (4)	C10—H10B	0.97
N2—C8	1.490 (3)	Br1—C14	1.906 (2)
N2—H21	0.850 (17)	O3—C17	1.264 (3)
N2—H22	0.833 (18)	O4—C17	1.257 (3)
N3—C4	1.454 (3)	C11—C16	1.388 (3)
C1—C2	1.405 (3)	C11—C12	1.391 (3)
C1—C6	1.410 (3)	C11—C17	1.506 (3)
C2—C3	1.376 (3)	C12—C13	1.385 (3)
C2—H2	0.93	C12—H12	0.93
C3—C4	1.377 (4)	C13—C14	1.374 (4)
C3—H3	0.93	C13—H13	0.93
C4—C5	1.378 (4)	C14—C15	1.378 (4)
C5—C6	1.372 (3)	C15—C16	1.382 (3)
C5—H5	0.93	C15—H15	0.93
C6—H6	0.93	C16—H16	0.93
C7—C8	1.502 (3)	O5—H1W	0.802 (17)
C7—H7A	0.97	O5—H2W	0.802 (17)
C7—H7B	0.97	O6—H4W	0.823 (17)
C8—H8A	0.97	O6—H3W	0.778 (18)
C1—N1—C7	117.46 (19)	N2—C8—H8B	109.4
C1—N1—C10	117.30 (18)	C7—C8—H8B	109.4
C7—N1—C10	112.08 (19)	H8A—C8—H8B	108
C9—N2—C8	109.8 (2)	N2—C9—C10	110.3 (2)
C9—N2—H21	113 (2)	N2—C9—H9A	109.6
C8—N2—H21	110 (2)	C10—C9—H9A	109.6
C9—N2—H22	115 (2)	N2—C9—H9B	109.6
C8—N2—H22	106 (2)	C10—C9—H9B	109.6
H21—N2—H22	102 (3)	H9A—C9—H9B	108.1
O2—N3—O1	122.4 (2)	N1—C10—C9	111.3 (2)
O2—N3—C4	118.8 (2)	N1—C10—H10A	109.4
O1—N3—C4	118.8 (2)	C9—C10—H10A	109.4
N1—C1—C2	121.5 (2)	N1—C10—H10B	109.4
N1—C1—C6	121.4 (2)	C9—C10—H10B	109.4
C2—C1—C6	117.1 (2)	H10A—C10—H10B	108
C3—C2—C1	121.2 (2)	C16—C11—C12	118.6 (2)
C3—C2—H2	119.4	C16—C11—C17	120.5 (2)
C1—C2—H2	119.4	C12—C11—C17	120.9 (2)
C2—C3—C4	119.9 (2)	C13—C12—C11	120.9 (2)
C2—C3—H3	120	C13—C12—H12	119.5
C4—C3—H3	120	C11—C12—H12	119.5

C3—C4—C5	120.6 (2)	C14—C13—C12	119.0 (2)
C3—C4—N3	119.2 (2)	C14—C13—H13	120.5
C5—C4—N3	120.2 (2)	C12—C13—H13	120.5
C6—C5—C4	119.8 (2)	C13—C14—C15	121.4 (2)
C6—C5—H5	120.1	C13—C14—Br1	119.7 (2)
C4—C5—H5	120.1	C15—C14—Br1	118.9 (2)
C5—C6—C1	121.4 (2)	C14—C15—C16	119.2 (2)
C5—C6—H6	119.3	C14—C15—H15	120.4
C1—C6—H6	119.3	C16—C15—H15	120.4
N1—C7—C8	111.5 (2)	C15—C16—C11	120.9 (2)
N1—C7—H7A	109.3	C15—C16—H16	119.6
C8—C7—H7A	109.3	C11—C16—H16	119.6
N1—C7—H7B	109.3	O4—C17—O3	124.3 (2)
C8—C7—H7B	109.3	O4—C17—C11	117.8 (2)
H7A—C7—H7B	108	O3—C17—C11	117.9 (2)
N2—C8—C7	111.17 (19)	H1W—O5—H2W	108 (3)
N2—C8—H8A	109.4	H4W—O6—H3W	109 (3)
C7—C8—H8A	109.4		

C7—N1—C1—C2	10.7 (3)	C9—N2—C8—C7	58.1 (3)
C10—N1—C1—C2	148.9 (2)	N1—C7—C8—N2	−55.1 (3)
C7—N1—C1—C6	−171.5 (2)	C8—N2—C9—C10	−58.5 (3)
C10—N1—C1—C6	−33.3 (3)	C1—N1—C10—C9	165.8 (2)
N1—C1—C2—C3	176.8 (2)	C7—N1—C10—C9	−53.9 (3)
C6—C1—C2—C3	−1.1 (4)	N2—C9—C10—N1	56.8 (3)
C1—C2—C3—C4	2.0 (4)	C16—C11—C12—C13	1.3 (3)
C2—C3—C4—C5	−1.6 (4)	C17—C11—C12—C13	−179.4 (2)
C2—C3—C4—N3	179.0 (2)	C11—C12—C13—C14	−1.7 (4)
O2—N3—C4—C3	−174.9 (3)	C12—C13—C14—C15	0.4 (4)
O1—N3—C4—C3	5.0 (4)	C12—C13—C14—Br1	179.36 (19)
O2—N3—C4—C5	5.7 (4)	C13—C14—C15—C16	1.2 (4)
O1—N3—C4—C5	−174.4 (3)	Br1—C14—C15—C16	−177.75 (18)
C3—C4—C5—C6	0.5 (4)	C14—C15—C16—C11	−1.6 (3)
N3—C4—C5—C6	179.9 (2)	C12—C11—C16—C15	0.4 (3)
C4—C5—C6—C1	0.3 (4)	C17—C11—C16—C15	−178.9 (2)
N1—C1—C6—C5	−177.9 (2)	C16—C11—C17—O4	−26.9 (3)
C2—C1—C6—C5	0.0 (4)	C12—C11—C17—O4	153.8 (2)
C1—N1—C7—C8	−166.7 (2)	C16—C11—C17—O3	153.3 (2)
C10—N1—C7—C8	53.0 (3)	C12—C11—C17—O3	−26.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H21···O5^{i}	0.85 (2)	1.99 (2)	2.810 (3)	162 (3)
N2—H22···O6^{ii}	0.83 (2)	1.91 (2)	2.707 (3)	160 (3)
C3—H3···O1^{iii}	0.93	2.59	3.260 (4)	130
C13—H13···O4^{iv}	0.93	2.57	3.483 (3)	166
C15—H15···O2^{v}	0.93	2.47	3.269 (4)	144

O5—H1*W*···O3^{vi}	0.80 (2)	1.97 (2)	2.759 (2)	169 (3)
O5—H2*W*···O3^{i}	0.80 (2)	2.00 (2)	2.772 (2)	161 (3)
O6—H4*W*···O4	0.82 (2)	2.03 (2)	2.832 (3)	166 (3)
O6—H3*W*···O4^{vii}	0.78 (2)	1.99 (2)	2.760 (3)	169 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*, *y*+1, *z*; (iii) −*x*+2, −*y*+2, −*z*+1; (iv) *x*+1, *y*, *z*; (v) −*x*+1, −*y*+1, −*z*+1; (vi) *x*, *y*−1, *z*; (vii) −*x*, −*y*+1, −*z*.

4-(4-Nitrophenyl)piperazin-1-ium 4-iodobenzoate dihydrate (II)

Crystal data

$C_{10}H_{14}N_3O_2^+·C_7H_4IO_2^-·2H_2O$
M_r = 491.28
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.7652 (4) Å
b = 9.2852 (5) Å
c = 13.930 (1) Å
α = 94.985 (5)°
β = 95.331 (5)°
γ = 104.875 (6)°
V = 960.09 (10) Å^3
Z = 2
F(000) = 492
D_x = 1.699 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6331 reflections
θ = 2.6–25.4°
μ = 1.71 mm^{-1}
T = 293 K
Prism, brown
0.48 × 0.48 × 0.2 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)
T_{min} = 0.458, T_{max} = 0.711
6331 measured reflections
3518 independent reflections
2952 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 25.4°, θ_{min} = 2.6°
h = −8→9
k = −11→9
l = −14→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.069
S = 1.03
3513 reflections
262 parameters
6 restraints
0 constraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 0.5892P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.9211 (3)	0.7945 (3)	0.5327 (2)	0.0765 (8)
O2	0.6869 (3)	0.6415 (3)	0.5706 (2)	0.0702 (7)
N1	0.3324 (3)	1.0055 (2)	0.29012 (17)	0.0344 (5)

N2	0.1277 (3)	1.1016 (3)	0.13972 (19)	0.0404 (6)
N3	0.7583 (4)	0.7448 (3)	0.52680 (19)	0.0471 (6)
C1	0.4369 (3)	0.9384 (3)	0.34660 (19)	0.0311 (6)
C2	0.6239 (4)	0.9794 (3)	0.3495 (2)	0.0421 (7)
H2	0.678241	1.049839	0.310458	0.051*
C3	0.7273 (4)	0.9175 (3)	0.4087 (2)	0.0434 (7)
H3	0.851637	0.947138	0.411003	0.052*
C4	0.6475 (4)	0.8107 (3)	0.4653 (2)	0.0348 (6)
C5	0.4638 (4)	0.7665 (3)	0.4644 (2)	0.0407 (7)
H5	0.411197	0.694461	0.502907	0.049*
C6	0.3609 (4)	0.8297 (3)	0.4063 (2)	0.0390 (7)
H6	0.236893	0.800552	0.405831	0.047*
C7	0.4203 (4)	1.1048 (3)	0.2222 (2)	0.0412 (7)
H7A	0.448427	1.044706	0.168453	0.049*
H7B	0.532139	1.170403	0.255077	0.049*
C8	0.3038 (4)	1.1984 (3)	0.1836 (2)	0.0439 (7)
H8A	0.287569	1.267775	0.236	0.053*
H8B	0.361796	1.256375	0.135285	0.053*
C9	0.0381 (4)	1.0095 (4)	0.2115 (2)	0.0456 (7)
H9A	−0.07676	0.945606	0.181239	0.055*
H9B	0.016429	1.074515	0.264773	0.055*
C10	0.1531 (4)	0.9144 (3)	0.2497 (2)	0.0413 (7)
H10A	0.096206	0.859686	0.299686	0.05*
H10B	0.163241	0.841846	0.19758	0.05*
I1	0.92211 (3)	0.44728 (3)	0.33815 (2)	0.05798 (10)
O3	0.4459 (3)	0.7894 (2)	0.01595 (18)	0.0503 (6)
O4	0.2183 (3)	0.6411 (2)	0.07685 (16)	0.0463 (5)
C11	0.5106 (3)	0.6368 (3)	0.1340 (2)	0.0321 (6)
C12	0.6798 (4)	0.6390 (3)	0.1072 (2)	0.0387 (7)
H12	0.71341	0.676998	0.050132	0.046*
C13	0.7975 (4)	0.5851 (3)	0.1648 (2)	0.0411 (7)
H13	0.909115	0.584052	0.146196	0.049*
C14	0.7470 (4)	0.5326 (3)	0.2509 (2)	0.0376 (7)
C15	0.5813 (4)	0.5331 (3)	0.2798 (2)	0.0398 (7)
H15	0.550434	0.499714	0.338523	0.048*
C16	0.4631 (4)	0.5834 (3)	0.2208 (2)	0.0378 (7)
H16	0.350345	0.581695	0.238875	0.045*
C17	0.3825 (4)	0.6931 (3)	0.0705 (2)	0.0356 (6)
O5	0.7577 (3)	0.0174 (2)	0.02906 (17)	0.0446 (5)
O6	0.0304 (3)	0.3341 (3)	0.06807 (19)	0.0529 (6)
H21	0.144 (5)	1.051 (4)	0.0882 (19)	0.063*
H22	0.076 (4)	1.162 (3)	0.116 (3)	0.063*
H1W	0.671 (4)	−0.051 (3)	0.032 (3)	0.063*
H2W	0.711 (5)	0.084 (3)	0.022 (3)	0.063*
H4W	0.100 (4)	0.414 (3)	0.080 (3)	0.063*
H3W	−0.052 (4)	0.326 (4)	0.027 (2)	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0437 (15)	0.0752 (18)	0.108 (2)	0.0122 (13)	−0.0192 (14)	0.0400 (16)
O2	0.0633 (16)	0.0754 (18)	0.0786 (18)	0.0200 (13)	0.0033 (13)	0.0469 (15)
N1	0.0300 (12)	0.0321 (13)	0.0391 (13)	0.0054 (10)	−0.0037 (10)	0.0090 (10)
N2	0.0420 (14)	0.0388 (15)	0.0413 (15)	0.0159 (11)	−0.0066 (12)	0.0072 (11)
N3	0.0496 (17)	0.0450 (16)	0.0461 (15)	0.0151 (13)	−0.0076 (13)	0.0084 (13)
C1	0.0328 (14)	0.0280 (14)	0.0316 (14)	0.0093 (11)	−0.0008 (11)	0.0009 (11)
C2	0.0340 (15)	0.0433 (17)	0.0496 (18)	0.0067 (13)	0.0047 (13)	0.0196 (14)
C3	0.0279 (15)	0.0464 (18)	0.0548 (19)	0.0086 (13)	−0.0010 (13)	0.0115 (15)
C4	0.0369 (15)	0.0344 (15)	0.0332 (15)	0.0118 (12)	−0.0029 (12)	0.0049 (12)
C5	0.0422 (17)	0.0417 (17)	0.0383 (16)	0.0087 (13)	0.0047 (13)	0.0139 (13)
C6	0.0292 (14)	0.0443 (17)	0.0430 (17)	0.0073 (12)	0.0016 (12)	0.0118 (13)
C7	0.0353 (15)	0.0389 (17)	0.0459 (17)	0.0034 (13)	−0.0040 (13)	0.0148 (14)
C8	0.0483 (18)	0.0350 (16)	0.0447 (17)	0.0078 (14)	−0.0075 (14)	0.0095 (13)
C9	0.0354 (16)	0.055 (2)	0.0479 (18)	0.0160 (14)	−0.0023 (14)	0.0103 (15)
C10	0.0297 (15)	0.0401 (17)	0.0524 (18)	0.0071 (13)	−0.0035 (13)	0.0110 (14)
I1	0.05063 (15)	0.06217 (17)	0.06139 (16)	0.01852 (11)	−0.01269 (10)	0.01870 (11)
O3	0.0370 (11)	0.0400 (12)	0.0758 (16)	0.0088 (9)	0.0016 (11)	0.0276 (11)
O4	0.0283 (11)	0.0460 (12)	0.0629 (14)	0.0073 (9)	−0.0016 (10)	0.0134 (10)
C11	0.0290 (14)	0.0241 (14)	0.0415 (16)	0.0059 (11)	−0.0004 (12)	0.0023 (12)
C12	0.0376 (16)	0.0394 (16)	0.0431 (17)	0.0138 (13)	0.0088 (13)	0.0119 (13)
C13	0.0315 (15)	0.0436 (17)	0.0516 (19)	0.0157 (13)	0.0044 (13)	0.0082 (14)
C14	0.0336 (15)	0.0306 (15)	0.0458 (17)	0.0078 (12)	−0.0064 (13)	0.0047 (13)
C15	0.0421 (17)	0.0365 (16)	0.0373 (16)	0.0043 (13)	0.0025 (13)	0.0068 (13)
C16	0.0288 (14)	0.0356 (16)	0.0471 (17)	0.0062 (12)	0.0048 (13)	0.0018 (13)
C17	0.0328 (15)	0.0241 (14)	0.0475 (17)	0.0073 (12)	−0.0011 (13)	−0.0012 (12)
O5	0.0345 (11)	0.0374 (13)	0.0610 (14)	0.0094 (9)	−0.0027 (10)	0.0111 (11)
O6	0.0442 (13)	0.0383 (12)	0.0735 (17)	0.0097 (10)	−0.0123 (11)	0.0164 (12)

Geometric parameters (Å, º)

O1—N3	1.222 (3)	C8—H8B	0.97
O2—N3	1.221 (3)	C9—C10	1.503 (4)
N1—C1	1.376 (3)	C9—H9A	0.97
N1—C10	1.462 (3)	C9—H9B	0.97
N1—C7	1.469 (4)	C10—H10A	0.97
N2—C8	1.473 (4)	C10—H10B	0.97
N2—C9	1.479 (4)	I1—C14	2.090 (3)
N2—H21	0.859 (18)	O3—C17	1.257 (3)
N2—H22	0.850 (18)	O4—C17	1.257 (3)
N3—C4	1.440 (4)	C11—C16	1.391 (4)
C1—C2	1.400 (4)	C11—C12	1.395 (4)
C1—C6	1.410 (4)	C11—C17	1.493 (4)
C2—C3	1.361 (4)	C12—C13	1.377 (4)
C2—H2	0.93	C12—H12	0.93
C3—C4	1.378 (4)	C13—C14	1.386 (4)

C3—H3	0.93	C13—H13	0.93
C4—C5	1.378 (4)	C14—C15	1.385 (4)
C5—C6	1.357 (4)	C15—C16	1.372 (4)
C5—H5	0.93	C15—H15	0.93
C6—H6	0.93	C16—H16	0.93
C7—C8	1.502 (4)	O5—H1W	0.805 (18)
C7—H7A	0.97	O5—H2W	0.805 (18)
C7—H7B	0.97	O6—H4W	0.795 (18)
C8—H8A	0.97	O6—H3W	0.800 (18)

C1—N1—C10	117.2 (2)	N2—C8—H8B	109.6
C1—N1—C7	117.8 (2)	C7—C8—H8B	109.6
C10—N1—C7	112.8 (2)	H8A—C8—H8B	108.1
C8—N2—C9	110.6 (2)	N2—C9—C10	110.3 (2)
C8—N2—H21	108 (3)	N2—C9—H9A	109.6
C9—N2—H21	115 (3)	C10—C9—H9A	109.6
C8—N2—H22	104 (3)	N2—C9—H9B	109.6
C9—N2—H22	118 (3)	C10—C9—H9B	109.6
H21—N2—H22	101 (3)	H9A—C9—H9B	108.1
O2—N3—O1	122.5 (3)	N1—C10—C9	111.5 (2)
O2—N3—C4	119.1 (3)	N1—C10—H10A	109.3
O1—N3—C4	118.4 (3)	C9—C10—H10A	109.3
N1—C1—C2	121.1 (2)	N1—C10—H10B	109.3
N1—C1—C6	121.5 (2)	C9—C10—H10B	109.3
C2—C1—C6	117.4 (2)	H10A—C10—H10B	108
C3—C2—C1	120.9 (3)	C16—C11—C12	119.5 (3)
C3—C2—H2	119.6	C16—C11—C17	120.4 (2)
C1—C2—H2	119.6	C12—C11—C17	120.1 (2)
C2—C3—C4	119.9 (3)	C13—C12—C11	120.4 (3)
C2—C3—H3	120.1	C13—C12—H12	119.8
C4—C3—H3	120.1	C11—C12—H12	119.8
C5—C4—C3	121.2 (3)	C12—C13—C14	119.0 (3)
C5—C4—N3	119.5 (3)	C12—C13—H13	120.5
C3—C4—N3	119.3 (3)	C14—C13—H13	120.5
C6—C5—C4	118.9 (3)	C15—C14—C13	121.4 (3)
C6—C5—H5	120.5	C15—C14—I1	119.1 (2)
C4—C5—H5	120.5	C13—C14—I1	119.6 (2)
C5—C6—C1	121.8 (3)	C16—C15—C14	119.2 (3)
C5—C6—H6	119.1	C16—C15—H15	120.4
C1—C6—H6	119.1	C14—C15—H15	120.4
N1—C7—C8	111.9 (2)	C15—C16—C11	120.5 (3)
N1—C7—H7A	109.2	C15—C16—H16	119.8
C8—C7—H7A	109.2	C11—C16—H16	119.8
N1—C7—H7B	109.2	O4—C17—O3	125.0 (3)
C8—C7—H7B	109.2	O4—C17—C11	116.9 (2)
H7A—C7—H7B	107.9	O3—C17—C11	118.1 (2)
N2—C8—C7	110.2 (2)	H1W—O5—H2W	101 (4)
N2—C8—H8A	109.6	H4W—O6—H3W	117 (4)

C7—C8—H8A	109.6		
C10—N1—C1—C2	148.6 (3)	C9—N2—C8—C7	58.1 (3)
C7—N1—C1—C2	8.9 (4)	N1—C7—C8—N2	−54.7 (3)
C10—N1—C1—C6	−33.7 (4)	C8—N2—C9—C10	−58.5 (3)
C7—N1—C1—C6	−173.4 (3)	C1—N1—C10—C9	165.8 (3)
N1—C1—C2—C3	177.1 (3)	C7—N1—C10—C9	−52.6 (3)
C6—C1—C2—C3	−0.8 (4)	N2—C9—C10—N1	55.3 (4)
C1—C2—C3—C4	1.3 (5)	C16—C11—C12—C13	1.6 (4)
C2—C3—C4—C5	−1.0 (5)	C17—C11—C12—C13	−179.0 (3)
C2—C3—C4—N3	179.3 (3)	C11—C12—C13—C14	−1.7 (4)
O2—N3—C4—C5	6.3 (4)	C12—C13—C14—C15	0.1 (4)
O1—N3—C4—C5	−173.9 (3)	C12—C13—C14—I1	179.0 (2)
O2—N3—C4—C3	−173.9 (3)	C13—C14—C15—C16	1.6 (4)
O1—N3—C4—C3	5.8 (4)	I1—C14—C15—C16	−177.4 (2)
C3—C4—C5—C6	0.1 (5)	C14—C15—C16—C11	−1.6 (4)
N3—C4—C5—C6	179.8 (3)	C12—C11—C16—C15	0.0 (4)
C4—C5—C6—C1	0.4 (5)	C17—C11—C16—C15	−179.3 (3)
N1—C1—C6—C5	−178.0 (3)	C16—C11—C17—O4	−26.0 (4)
C2—C1—C6—C5	−0.1 (4)	C12—C11—C17—O4	154.6 (3)
C1—N1—C7—C8	−166.2 (2)	C16—C11—C17—O3	153.1 (3)
C10—N1—C7—C8	52.5 (3)	C12—C11—C17—O3	−26.2 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H21···O5^{i}	0.86 (2)	1.99 (2)	2.825 (4)	164 (4)
N2—H22···O6^{ii}	0.85 (2)	1.88 (2)	2.702 (3)	163 (4)
C3—H3···O1^{iii}	0.93	2.59	3.275 (4)	131
C13—H13···O4^{iv}	0.93	2.62	3.526 (4)	166
C15—H15···O2^{v}	0.93	2.49	3.311 (4)	147
O5—H1*W*···O3^{vi}	0.81 (2)	1.96 (2)	2.756 (3)	170 (4)
O5—H2*W*···O3^{i}	0.81 (2)	1.96 (2)	2.753 (3)	166 (4)
O6—H4*W*···O4	0.80 (2)	2.08 (2)	2.836 (3)	160 (4)
O6—H3*W*···O4^{vii}	0.80 (2)	1.95 (2)	2.728 (3)	165 (4)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*, *y*+1, *z*; (iii) −*x*+2, −*y*+2, −*z*+1; (iv) *x*+1, *y*, *z*; (v) −*x*+1, −*y*+1, −*z*+1; (vi) *x*, *y*−1, *z*; (vii) −*x*, −*y*+1, −*z*.

4-(4-Nitrophenyl)piperazin-1-ium 4-hydroxybenzoate monohydrate (III)

Crystal data

$C_{10}H_{14}N_3O_2^+·C_7H_5O_3^-·H_2O$
M_r = 363.37
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 9.636 (1) Å
b = 10.301 (1) Å
c = 10.867 (1) Å
α = 103.90 (1)°
β = 108.32 (1)°
γ = 112.96 (1)°
V = 857.80 (17) Å^3
Z = 2
F(000) = 384
D_x = 1.407 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5342 reflections
θ = 2.6–25.3°
μ = 0.11 mm^{-1}

T = 293 K
Rod, yellow

0.50 × 0.32 × 0.24 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)
T_{min} = 0.959, T_{max} = 0.974
5342 measured reflections

3140 independent reflections
2342 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 25.3°, θ_{min} = 2.6°
h = −11→11
k = −12→11
l = −13→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.106
S = 1.05
3135 reflections
251 parameters
5 restraints
0 constraints
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.3231P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: SHELXL2018/3 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.032 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.3138 (2)	0.37000 (19)	0.65529 (18)	0.0346 (4)
C2	0.1431 (2)	0.2527 (2)	0.57638 (19)	0.0418 (4)
H2	0.068235	0.250847	0.613904	0.05*
C3	0.0843 (2)	0.1407 (2)	0.4450 (2)	0.0434 (5)
H3	−0.029409	0.063818	0.39416	0.052*
C4	0.1942 (2)	0.1428 (2)	0.38907 (19)	0.0411 (4)
C5	0.3624 (2)	0.2553 (2)	0.4636 (2)	0.0483 (5)
H5	0.436252	0.255393	0.42532	0.058*
C6	0.4208 (2)	0.3670 (2)	0.5942 (2)	0.0472 (5)
H6	0.53484	0.443048	0.64379	0.057*
C7	0.5230 (2)	0.6338 (2)	0.8284 (2)	0.0453 (5)
H7A	0.489341	0.67826	0.763471	0.054*
H7B	0.611237	0.616548	0.817407	0.054*
C8	0.5933 (2)	0.7472 (2)	0.9784 (2)	0.0493 (5)
H8A	0.643572	0.71093	1.044494	0.059*
H8B	0.681545	0.846719	0.99504	0.059*
C9	0.3302 (3)	0.6143 (2)	0.9836 (2)	0.0457 (5)
H9A	0.242868	0.625614	1.002896	0.055*

H9B	0.381743	0.579404	1.050389	0.055*
C10	0.2514 (2)	0.4953 (2)	0.83370 (19)	0.0403 (4)
H10A	0.176601	0.394834	0.82667	0.048*
H10B	0.183115	0.521491	0.76885	0.048*
C11	0.1749 (2)	0.5843 (2)	0.29147 (19)	0.0418 (5)
C12	0.2469 (3)	0.7413 (2)	0.3258 (2)	0.0495 (5)
H12	0.262285	0.778209	0.258209	0.059*
C13	0.2959 (2)	0.8431 (2)	0.4597 (2)	0.0438 (5)
H13	0.344454	0.94863	0.481898	0.053*
C14	0.2738 (2)	0.7906 (2)	0.56226 (18)	0.0364 (4)
C15	0.2004 (2)	0.6329 (2)	0.52544 (19)	0.0395 (4)
H15	0.18337	0.595519	0.592378	0.047*
C16	0.1520 (2)	0.5299 (2)	0.39188 (19)	0.0410 (4)
H16	0.104259	0.424435	0.369591	0.049*
C17	0.3293 (2)	0.9024 (2)	0.7080 (2)	0.0440 (5)
N1	0.37669 (18)	0.48418 (16)	0.78933 (15)	0.0372 (4)
N2	0.4599 (2)	0.76559 (19)	1.00415 (18)	0.0479 (4)
N3	0.1322 (2)	0.02537 (19)	0.25010 (18)	0.0518 (4)
O1	0.2282 (2)	0.04014 (19)	0.19537 (17)	0.0759 (5)
O2	−0.0112 (2)	−0.08579 (18)	0.19233 (16)	0.0715 (5)
O3	0.1311 (2)	0.48813 (18)	0.15852 (15)	0.0637 (4)
O4	0.3985 (2)	1.04290 (17)	0.73256 (16)	0.0744 (5)
O5	0.30323 (18)	0.85085 (16)	0.79750 (14)	0.0547 (4)
O6	−0.0679 (3)	0.1917 (2)	0.09903 (19)	0.0809 (6)
H21	0.412 (3)	0.796 (3)	0.941 (2)	0.097*
H22	0.513 (3)	0.838 (3)	1.099 (2)	0.097*
H17	0.070 (3)	0.394 (2)	0.143 (3)	0.097*
H1W	−0.140 (3)	0.168 (3)	0.129 (3)	0.097*
H2W	−0.112 (3)	0.128 (3)	0.015 (2)	0.097*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0348 (10)	0.0343 (9)	0.0377 (10)	0.0187 (8)	0.0167 (8)	0.0173 (8)
C2	0.0357 (10)	0.0427 (10)	0.0444 (11)	0.0159 (8)	0.0211 (8)	0.0168 (9)
C3	0.0356 (10)	0.0380 (10)	0.0439 (11)	0.0113 (8)	0.0148 (8)	0.0145 (9)
C4	0.0463 (11)	0.0347 (9)	0.0373 (10)	0.0191 (8)	0.0175 (8)	0.0119 (8)
C5	0.0429 (11)	0.0481 (11)	0.0505 (12)	0.0215 (9)	0.0255 (9)	0.0120 (10)
C6	0.0315 (10)	0.0440 (11)	0.0514 (12)	0.0139 (8)	0.0177 (9)	0.0081 (9)
C7	0.0379 (10)	0.0401 (10)	0.0487 (11)	0.0133 (8)	0.0209 (9)	0.0137 (9)
C8	0.0459 (11)	0.0395 (11)	0.0468 (11)	0.0154 (9)	0.0160 (9)	0.0117 (9)
C9	0.0590 (12)	0.0451 (11)	0.0434 (11)	0.0277 (10)	0.0295 (10)	0.0231 (9)
C10	0.0429 (10)	0.0421 (10)	0.0420 (10)	0.0213 (9)	0.0239 (9)	0.0211 (9)
C11	0.0398 (10)	0.0443 (11)	0.0361 (10)	0.0170 (9)	0.0194 (8)	0.0133 (9)
C12	0.0595 (13)	0.0525 (12)	0.0441 (11)	0.0248 (10)	0.0315 (10)	0.0269 (10)
C13	0.0494 (11)	0.0392 (10)	0.0482 (11)	0.0207 (9)	0.0274 (9)	0.0224 (9)
C14	0.0357 (9)	0.0405 (10)	0.0381 (10)	0.0210 (8)	0.0190 (8)	0.0183 (8)
C15	0.0410 (10)	0.0440 (10)	0.0378 (10)	0.0201 (8)	0.0208 (8)	0.0223 (9)

C16	0.0409 (10)	0.0356 (10)	0.0423 (11)	0.0151 (8)	0.0194 (8)	0.0167 (8)
C17	0.0482 (11)	0.0462 (12)	0.0409 (11)	0.0255 (9)	0.0218 (9)	0.0180 (9)
N1	0.0349 (8)	0.0348 (8)	0.0385 (8)	0.0157 (7)	0.0170 (7)	0.0130 (7)
N2	0.0616 (11)	0.0410 (9)	0.0425 (10)	0.0259 (8)	0.0257 (9)	0.0168 (8)
N3	0.0575 (11)	0.0430 (10)	0.0443 (10)	0.0214 (9)	0.0209 (9)	0.0124 (8)
O1	0.0806 (12)	0.0662 (11)	0.0599 (10)	0.0220 (9)	0.0434 (9)	0.0052 (8)
O2	0.0600 (10)	0.0523 (9)	0.0564 (10)	0.0087 (8)	0.0163 (8)	0.0010 (8)
O3	0.0759 (11)	0.0545 (9)	0.0430 (8)	0.0171 (8)	0.0337 (8)	0.0121 (7)
O4	0.1147 (14)	0.0416 (9)	0.0554 (10)	0.0278 (9)	0.0450 (10)	0.0145 (7)
O5	0.0721 (10)	0.0620 (9)	0.0425 (8)	0.0375 (8)	0.0324 (7)	0.0259 (7)
O6	0.0936 (14)	0.0547 (10)	0.0652 (11)	0.0142 (10)	0.0484 (10)	0.0041 (8)

Geometric parameters (Å, º)

C1—N1	1.392 (2)	C10—H10A	0.97
C1—C6	1.397 (2)	C10—H10B	0.97
C1—C2	1.402 (2)	C11—O3	1.360 (2)
C2—C3	1.371 (3)	C11—C16	1.379 (3)
C2—H2	0.93	C11—C12	1.382 (3)
C3—C4	1.373 (3)	C12—C13	1.374 (3)
C3—H3	0.93	C12—H12	0.93
C4—C5	1.373 (3)	C13—C14	1.389 (2)
C4—N3	1.446 (2)	C13—H13	0.93
C5—C6	1.365 (3)	C14—C15	1.383 (2)
C5—H5	0.93	C14—C17	1.494 (3)
C6—H6	0.93	C15—C16	1.378 (2)
C7—N1	1.468 (2)	C15—H15	0.93
C7—C8	1.501 (3)	C16—H16	0.93
C7—H7A	0.97	C17—O4	1.250 (2)
C7—H7B	0.97	C17—O5	1.260 (2)
C8—N2	1.470 (3)	N2—H21	0.893 (17)
C8—H8A	0.97	N2—H22	0.935 (17)
C8—H8B	0.97	N3—O2	1.221 (2)
C9—N2	1.476 (2)	N3—O1	1.230 (2)
C9—C10	1.507 (3)	O3—H17	0.850 (17)
C9—H9A	0.97	O6—H1W	0.832 (17)
C9—H9B	0.97	O6—H2W	0.834 (17)
C10—N1	1.467 (2)		

N1—C1—C6	120.65 (15)	N1—C10—H10B	108.9
N1—C1—C2	122.40 (15)	C9—C10—H10B	108.9
C6—C1—C2	116.95 (16)	H10A—C10—H10B	107.8
C3—C2—C1	121.37 (17)	O3—C11—C16	122.11 (17)
C3—C2—H2	119.3	O3—C11—C12	118.10 (17)
C1—C2—H2	119.3	C16—C11—C12	119.78 (17)
C2—C3—C4	119.60 (17)	C13—C12—C11	120.14 (18)
C2—C3—H3	120.2	C13—C12—H12	119.9
C4—C3—H3	120.2	C11—C12—H12	119.9

C3—C4—C5	120.68 (17)	C12—C13—C14	120.94 (17)
C3—C4—N3	119.73 (17)	C12—C13—H13	119.5
C5—C4—N3	119.59 (17)	C14—C13—H13	119.5
C6—C5—C4	119.67 (17)	C15—C14—C13	118.02 (16)
C6—C5—H5	120.2	C15—C14—C17	121.42 (16)
C4—C5—H5	120.2	C13—C14—C17	120.55 (16)
C5—C6—C1	121.72 (17)	C16—C15—C14	121.51 (17)
C5—C6—H6	119.1	C16—C15—H15	119.2
C1—C6—H6	119.1	C14—C15—H15	119.2
N1—C7—C8	113.07 (16)	C15—C16—C11	119.60 (17)
N1—C7—H7A	109	C15—C16—H16	120.2
C8—C7—H7A	109	C11—C16—H16	120.2
N1—C7—H7B	109	O4—C17—O5	124.14 (18)
C8—C7—H7B	109	O4—C17—C14	116.82 (17)
H7A—C7—H7B	107.8	O5—C17—C14	119.04 (17)
N2—C8—C7	110.93 (16)	C1—N1—C10	116.66 (14)
N2—C8—H8A	109.5	C1—N1—C7	115.79 (14)
C7—C8—H8A	109.5	C10—N1—C7	114.73 (14)
N2—C8—H8B	109.5	C8—N2—C9	108.67 (15)
C7—C8—H8B	109.5	C8—N2—H21	109.1 (18)
H8A—C8—H8B	108	C9—N2—H21	108.9 (18)
N2—C9—C10	110.96 (15)	C8—N2—H22	106.2 (17)
N2—C9—H9A	109.4	C9—N2—H22	110.8 (17)
C10—C9—H9A	109.4	H21—N2—H22	113 (2)
N2—C9—H9B	109.4	O2—N3—O1	122.24 (17)
C10—C9—H9B	109.4	O2—N3—C4	119.43 (17)
H9A—C9—H9B	108	O1—N3—C4	118.32 (17)
N1—C10—C9	113.15 (15)	C11—O3—H17	110.2 (19)
N1—C10—H10A	108.9	H1W—O6—H2W	108 (3)
C9—C10—H10A	108.9		

N1—C1—C2—C3	−179.46 (17)	O3—C11—C16—C15	179.34 (17)
C6—C1—C2—C3	−0.3 (3)	C12—C11—C16—C15	0.4 (3)
C1—C2—C3—C4	0.0 (3)	C15—C14—C17—O4	178.09 (18)
C2—C3—C4—C5	0.4 (3)	C13—C14—C17—O4	−1.4 (3)
C2—C3—C4—N3	−179.58 (17)	C15—C14—C17—O5	−2.4 (3)
C3—C4—C5—C6	−0.5 (3)	C13—C14—C17—O5	178.03 (18)
N3—C4—C5—C6	179.41 (18)	C6—C1—N1—C10	169.31 (17)
C4—C5—C6—C1	0.3 (3)	C2—C1—N1—C10	−11.5 (2)
N1—C1—C6—C5	179.30 (18)	C6—C1—N1—C7	29.6 (2)
C2—C1—C6—C5	0.1 (3)	C2—C1—N1—C7	−151.19 (18)
N1—C7—C8—N2	−53.2 (2)	C9—C10—N1—C1	176.12 (15)
N2—C9—C10—N1	52.1 (2)	C9—C10—N1—C7	−43.8 (2)
O3—C11—C12—C13	−178.90 (18)	C8—C7—N1—C1	−175.25 (16)
C16—C11—C12—C13	0.1 (3)	C8—C7—N1—C10	44.3 (2)
C11—C12—C13—C14	−0.2 (3)	C7—C8—N2—C9	61.2 (2)
C12—C13—C14—C15	−0.3 (3)	C10—C9—N2—C8	−60.7 (2)
C12—C13—C14—C17	179.26 (18)	C3—C4—N3—O2	−9.7 (3)

C13—C14—C15—C16	0.8 (3)	C5—C4—N3—O2	170.3 (2)
C17—C14—C15—C16	−178.76 (17)	C3—C4—N3—O1	171.61 (19)
C14—C15—C16—C11	−0.8 (3)	C5—C4—N3—O1	−8.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H21···O5	0.89 (2)	1.93 (2)	2.819 (2)	177 (3)
N2—H22···O4^{i}	0.94 (2)	1.65 (2)	2.583 (2)	177 (3)
O3—H17···O6	0.85 (2)	1.82 (2)	2.669 (2)	177 (3)
O6—H1*W*···O5^{ii}	0.83 (2)	1.95 (2)	2.768 (2)	169 (3)
O6—H2*W*···O1^{iii}	0.83 (2)	2.11 (2)	2.944 (2)	178 (3)

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+2; (ii) −*x*, −*y*+1, −*z*+1; (iii) −*x*, −*y*, −*z*.

4-(4-Nitrophenyl)piperazin-1-ium 4-methylbenzoate monohydrate (IV)

Crystal data

$C_{10}H_{14}N_3O_2^+·C_8H_7O_2^-·H_2O$
M_r = 361.39
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.1136 (5) Å
b = 7.6965 (7) Å
c = 19.708 (2) Å
α = 79.577 (8)°
β = 87.162 (8)°
γ = 86.699 (8)°
V = 909.79 (15) Å^3
Z = 2
F(000) = 384
D_x = 1.319 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5980 reflections
θ = 3.1–25.4°
μ = 0.10 mm^{-1}
T = 293 K
Plate, yellow
0.48 × 0.26 × 0.02 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)
T_{min} = 0.970, T_{max} = 0.998
5980 measured reflections
3347 independent reflections
1911 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.4°, θ_{min} = 3.1°
h = −5→7
k = −8→9
l = −23→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.138
S = 1.01
3343 reflections
248 parameters
4 restraints
0 constraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.2441P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.7597 (5)	−0.1408 (4)	0.52918 (12)	0.1114 (9)
O2	1.0599 (5)	−0.2808 (4)	0.50875 (13)	0.1312 (11)
N1	0.8270 (3)	0.0591 (2)	0.20408 (9)	0.0426 (5)
N2	0.8376 (3)	0.2441 (3)	0.06365 (10)	0.0492 (5)
N3	0.9029 (6)	−0.1858 (4)	0.48992 (13)	0.0795 (8)
C1	0.8503 (4)	0.0072 (3)	0.27523 (12)	0.0421 (6)
C2	0.6848 (5)	0.0416 (4)	0.32264 (13)	0.0620 (8)
H2	0.559385	0.107832	0.306884	0.074*
C3	0.7020 (5)	−0.0200 (4)	0.39241 (13)	0.0660 (8)
H3	0.58958	0.005008	0.423273	0.079*
C4	0.8844 (5)	−0.1175 (4)	0.41582 (13)	0.0589 (7)
C5	1.0511 (5)	−0.1516 (4)	0.37142 (15)	0.0698 (8)
H5	1.175889	−0.217226	0.388096	0.084*
C6	1.0361 (4)	−0.0894 (4)	0.30170 (13)	0.0606 (8)
H6	1.152207	−0.112351	0.271761	0.073*
C7	0.6682 (4)	0.2049 (3)	0.18122 (12)	0.0490 (6)
H7A	0.721093	0.313511	0.191295	0.059*
H7B	0.530333	0.182601	0.207033	0.059*
C8	0.6287 (4)	0.2287 (4)	0.10502 (12)	0.0546 (7)
H8A	0.553149	0.128385	0.095886	0.066*
H8B	0.535474	0.334375	0.091454	0.066*
C9	0.9774 (4)	0.0810 (3)	0.08466 (12)	0.0525 (7)
H9A	1.113597	0.087916	0.057129	0.063*
H9B	0.902753	−0.020822	0.076595	0.063*
C10	1.0256 (4)	0.0595 (3)	0.15971 (12)	0.0499 (6)
H10A	1.110342	−0.050791	0.173238	0.06*
H10B	1.11409	0.155146	0.166426	0.06*
O3	0.2656 (3)	0.7140 (3)	0.07107 (9)	0.0598 (5)
O4	0.0053 (3)	0.5296 (3)	0.11199 (9)	0.0620 (5)
C11	0.2728 (4)	0.5764 (3)	0.18855 (12)	0.0399 (6)
C12	0.4646 (4)	0.6537 (3)	0.19936 (13)	0.0484 (6)
H12	0.533917	0.726876	0.162881	0.058*
C13	0.5535 (4)	0.6230 (3)	0.26365 (14)	0.0575 (7)
H13	0.68258	0.675862	0.269499	0.069*
C14	0.4567 (5)	0.5162 (3)	0.31955 (14)	0.0562 (7)
C15	0.2645 (4)	0.4395 (4)	0.30874 (14)	0.0584 (7)
H15	0.195058	0.366932	0.345395	0.07*
C16	0.1748 (4)	0.4689 (3)	0.24468 (13)	0.0496 (6)
H16	0.045866	0.41565	0.238897	0.06*

C17	0.1749 (4)	0.6080 (3)	0.11893 (13)	0.0437 (6)
C18	0.5550 (6)	0.4855 (4)	0.38973 (16)	0.0863 (10)
H18A	0.442848	0.502929	0.424033	0.129*
H18B	0.667715	0.567332	0.389742	0.129*
H18C	0.617002	0.366684	0.400046	0.129*
O5	0.7006 (3)	0.7136 (5)	0.02422 (13)	0.1211 (12)
H21	0.903 (6)	0.338 (4)	0.073 (2)	0.145*
H22	0.807 (6)	0.256 (5)	0.0183 (11)	0.145*
H1W	0.792 (5)	0.667 (5)	0.0534 (17)	0.145*
H2W	0.576 (4)	0.720 (6)	0.045 (2)	0.145*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.145 (2)	0.132 (2)	0.0477 (14)	0.0055 (18)	0.0100 (15)	−0.0001 (14)
O2	0.153 (2)	0.155 (3)	0.0702 (17)	0.044 (2)	−0.0390 (17)	0.0148 (17)
N1	0.0427 (11)	0.0478 (12)	0.0358 (11)	0.0059 (9)	−0.0018 (9)	−0.0064 (9)
N2	0.0540 (13)	0.0562 (14)	0.0357 (11)	0.0002 (11)	−0.0042 (10)	−0.0041 (10)
N3	0.111 (2)	0.078 (2)	0.0469 (17)	−0.0053 (17)	−0.0113 (16)	−0.0005 (14)
C1	0.0489 (14)	0.0408 (14)	0.0377 (14)	0.0000 (11)	−0.0047 (11)	−0.0100 (11)
C2	0.0675 (17)	0.0685 (19)	0.0442 (16)	0.0202 (15)	0.0004 (13)	−0.0028 (14)
C3	0.083 (2)	0.072 (2)	0.0394 (16)	0.0130 (17)	0.0050 (14)	−0.0057 (14)
C4	0.085 (2)	0.0556 (17)	0.0352 (15)	0.0002 (16)	−0.0113 (14)	−0.0044 (13)
C5	0.0719 (19)	0.083 (2)	0.0515 (19)	0.0176 (17)	−0.0203 (15)	−0.0060 (16)
C6	0.0564 (16)	0.078 (2)	0.0448 (16)	0.0167 (15)	−0.0066 (13)	−0.0092 (14)
C7	0.0428 (14)	0.0616 (17)	0.0401 (14)	0.0080 (12)	−0.0034 (11)	−0.0050 (12)
C8	0.0441 (14)	0.0706 (18)	0.0468 (16)	0.0043 (13)	−0.0071 (12)	−0.0052 (13)
C9	0.0597 (16)	0.0579 (17)	0.0385 (15)	0.0061 (13)	0.0028 (12)	−0.0086 (12)
C10	0.0499 (15)	0.0558 (16)	0.0417 (15)	0.0138 (12)	−0.0008 (12)	−0.0080 (12)
O3	0.0574 (11)	0.0779 (13)	0.0404 (10)	−0.0028 (10)	−0.0074 (8)	0.0006 (9)
O4	0.0577 (11)	0.0758 (13)	0.0557 (12)	−0.0102 (10)	−0.0167 (9)	−0.0143 (10)
C11	0.0404 (13)	0.0387 (13)	0.0413 (14)	0.0068 (11)	−0.0074 (11)	−0.0103 (11)
C12	0.0513 (15)	0.0449 (15)	0.0490 (16)	−0.0006 (12)	−0.0065 (12)	−0.0072 (12)
C13	0.0547 (16)	0.0533 (17)	0.0679 (19)	0.0004 (13)	−0.0224 (14)	−0.0158 (15)
C14	0.0707 (18)	0.0497 (16)	0.0498 (17)	0.0066 (14)	−0.0233 (14)	−0.0103 (13)
C15	0.0706 (18)	0.0570 (17)	0.0453 (16)	−0.0042 (14)	−0.0099 (13)	−0.0007 (13)
C16	0.0488 (15)	0.0518 (16)	0.0485 (16)	−0.0039 (13)	−0.0089 (12)	−0.0074 (13)
C17	0.0427 (14)	0.0473 (15)	0.0420 (15)	0.0071 (12)	−0.0042 (12)	−0.0121 (12)
C18	0.114 (3)	0.079 (2)	0.068 (2)	−0.003 (2)	−0.0467 (19)	−0.0066 (17)
O5	0.0602 (14)	0.210 (3)	0.0697 (16)	−0.0101 (18)	−0.0093 (12)	0.0407 (18)

Geometric parameters (Å, º)

O1—N3	1.216 (3)	C9—C10	1.500 (3)
O2—N3	1.204 (3)	C9—H9A	0.97
N1—C1	1.399 (3)	C9—H9B	0.97
N1—C10	1.460 (3)	C10—H10A	0.97
N1—C7	1.463 (3)	C10—H10B	0.97

N2—C8	1.480 (3)	O3—O3	0.000 (5)
N2—C9	1.483 (3)	O3—C17	1.259 (3)
N2—H21	0.892 (19)	O4—C17	1.254 (3)
N2—H22	0.908 (19)	C11—C12	1.387 (3)
N3—C4	1.468 (3)	C11—C16	1.389 (3)
C1—C2	1.390 (3)	C11—C17	1.498 (3)
C1—C6	1.391 (3)	C12—C13	1.379 (3)
C2—C3	1.378 (4)	C12—H12	0.93
C2—H2	0.93	C13—C14	1.380 (4)
C3—C4	1.360 (4)	C13—H13	0.93
C3—H3	0.93	C14—C15	1.387 (4)
C4—C5	1.356 (4)	C14—C18	1.509 (4)
C5—C6	1.377 (4)	C15—C16	1.377 (3)
C5—H5	0.93	C15—H15	0.93
C6—H6	0.93	C16—H16	0.93
C7—C8	1.509 (3)	C18—H18A	0.96
C7—H7A	0.97	C18—H18B	0.96
C7—H7B	0.97	C18—H18C	0.96
C8—H8A	0.97	O5—H1W	0.843 (19)
C8—H8B	0.97	O5—H2W	0.854 (19)

C1—N1—C10	117.39 (18)	N2—C9—H9A	109.6
C1—N1—C7	117.37 (18)	C10—C9—H9A	109.6
C10—N1—C7	113.94 (18)	N2—C9—H9B	109.6
C8—N2—C9	108.7 (2)	C10—C9—H9B	109.6
C8—N2—H21	107 (3)	H9A—C9—H9B	108.2
C9—N2—H21	110 (3)	N1—C10—C9	112.7 (2)
C8—N2—H22	109 (3)	N1—C10—H10A	109.1
C9—N2—H22	110 (3)	C9—C10—H10A	109.1
H21—N2—H22	112 (4)	N1—C10—H10B	109.1
O2—N3—O1	123.5 (3)	C9—C10—H10B	109.1
O2—N3—C4	118.5 (3)	H10A—C10—H10B	107.8
O1—N3—C4	117.9 (3)	O3—O3—C17	0 (10)
C2—C1—C6	116.8 (2)	C12—C11—C16	117.5 (2)
C2—C1—N1	121.7 (2)	C12—C11—C17	121.2 (2)
C6—C1—N1	121.4 (2)	C16—C11—C17	121.2 (2)
C3—C2—C1	121.6 (3)	C13—C12—C11	120.7 (2)
C3—C2—H2	119.2	C13—C12—H12	119.7
C1—C2—H2	119.2	C11—C12—H12	119.7
C4—C3—C2	119.6 (3)	C12—C13—C14	122.0 (2)
C4—C3—H3	120.2	C12—C13—H13	119
C2—C3—H3	120.2	C14—C13—H13	119
C5—C4—C3	120.7 (2)	C13—C14—C15	117.3 (2)
C5—C4—N3	119.3 (3)	C13—C14—C18	121.3 (3)
C3—C4—N3	120.0 (3)	C15—C14—C18	121.4 (3)
C4—C5—C6	120.1 (3)	C16—C15—C14	121.2 (3)
C4—C5—H5	119.9	C16—C15—H15	119.4
C6—C5—H5	119.9	C14—C15—H15	119.4

C5—C6—C1	121.2 (2)	C15—C16—C11	121.3 (2)
C5—C6—H6	119.4	C15—C16—H16	119.3
C1—C6—H6	119.4	C11—C16—H16	119.3
N1—C7—C8	112.6 (2)	O4—C17—O3	123.8 (2)
N1—C7—H7A	109.1	O4—C17—O3	123.8 (2)
C8—C7—H7A	109.1	O3—C17—O3	0.0 (2)
N1—C7—H7B	109.1	O4—C17—C11	118.1 (2)
C8—C7—H7B	109.1	O3—C17—C11	118.0 (2)
H7A—C7—H7B	107.8	O3—C17—C11	118.0 (2)
N2—C8—C7	111.1 (2)	C14—C18—H18A	109.5
N2—C8—H8A	109.4	C14—C18—H18B	109.5
C7—C8—H8A	109.4	H18A—C18—H18B	109.5
N2—C8—H8B	109.4	C14—C18—H18C	109.5
C7—C8—H8B	109.4	H18A—C18—H18C	109.5
H8A—C8—H8B	108	H18B—C18—H18C	109.5
N2—C9—C10	110.1 (2)	H1W—O5—H2W	108 (4)

C10—N1—C1—C2	164.4 (2)	C8—N2—C9—C10	−60.7 (3)
C7—N1—C1—C2	22.9 (3)	C1—N1—C10—C9	168.3 (2)
C10—N1—C1—C6	−18.9 (3)	C7—N1—C10—C9	−48.9 (3)
C7—N1—C1—C6	−160.4 (2)	N2—C9—C10—N1	55.7 (3)
C6—C1—C2—C3	−1.2 (4)	C16—C11—C12—C13	0.3 (3)
N1—C1—C2—C3	175.6 (3)	C17—C11—C12—C13	−179.7 (2)
C1—C2—C3—C4	−0.3 (5)	C11—C12—C13—C14	−0.3 (4)
C2—C3—C4—C5	1.3 (5)	C12—C13—C14—C15	0.1 (4)
C2—C3—C4—N3	−179.1 (3)	C12—C13—C14—C18	−179.4 (3)
O2—N3—C4—C5	−5.0 (4)	C13—C14—C15—C16	0.1 (4)
O1—N3—C4—C5	173.8 (3)	C18—C14—C15—C16	179.6 (3)
O2—N3—C4—C3	175.5 (3)	C14—C15—C16—C11	−0.1 (4)
O1—N3—C4—C3	−5.7 (4)	C12—C11—C16—C15	−0.1 (4)
C3—C4—C5—C6	−0.8 (5)	C17—C11—C16—C15	179.9 (2)
N3—C4—C5—C6	179.6 (3)	O3—O3—C17—O4	0.00 (14)
C4—C5—C6—C1	−0.8 (5)	O3—O3—C17—C11	0.0 (2)
C2—C1—C6—C5	1.8 (4)	C12—C11—C17—O4	177.7 (2)
N1—C1—C6—C5	−175.0 (3)	C16—C11—C17—O4	−2.4 (3)
C1—N1—C7—C8	−170.1 (2)	C12—C11—C17—O3	−3.5 (3)
C10—N1—C7—C8	47.0 (3)	C16—C11—C17—O3	176.4 (2)
C9—N2—C8—C7	59.5 (3)	C12—C11—C17—O3	−3.5 (3)
N1—C7—C8—N2	−52.7 (3)	C16—C11—C17—O3	176.4 (2)

Hydrogen-bond geometry (Å, º)

*Cg*3 is the centroids of the C11–C16 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H21···O4^{i}	0.89 (2)	1.93 (2)	2.811 (3)	167 (4)
N2—H22···O3^{ii}	0.91 (2)	1.81 (2)	2.717 (3)	177 (4)
C3—H3···O1^{iii}	0.93	2.54	3.427 (4)	161
C9—H9*A*···O5^{iv}	0.97	2.31	3.113 (3)	140

O5—H1*W*···O4^{i}	0.84 (2)	1.92 (2)	2.756 (3)	171 (4)
O5—H2*W*···O3	0.85 (2)	1.94 (2)	2.772 (3)	164 (4)
C6—H6···*Cg*3^{v}	0.93	2.93	3.590 (3)	129

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*; (iii) −*x*+1, −*y*, −*z*+1; (iv) −*x*+2, −*y*+1, −*z*; (v) *x*+1, *y*−1, *z*.

4-(4-Nitrophenyl)piperazin-1-ium 4-methoxybenzoate hemihydrate (V)

Crystal data

$2C_{10}H_{14}N_3O_2^+·2C_8H_7O_3^-·H_2O$
M_r = 736.77
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 15.808 (1) Å
b = 7.5198 (7) Å
c = 31.020 (2) Å
β = 92.561 (7)°
V = 3683.8 (5) Å^3
Z = 4
$F(000)$ = 1560
D_x = 1.328 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2899 reflections
θ = 2.6–25.3°
μ = 0.1 mm^{-1}
T = 293 K
Prism, orange
0.5 × 0.36 × 0.36 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)
T_{min} = 0.958, T_{max} = 0.965
15326 measured reflections
6718 independent reflections
2602 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 25.3°, θ_{min} = 2.6°
h = −19→18
k = −9→8
l = −37→33

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.169
S = 1.00
6715 reflections
507 parameters
45 restraints
0 constraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.9198P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: SHELXL2018/3 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0029 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.2115 (2)	0.2769 (5)	0.15386 (13)	0.0456 (11)	
C2	0.2042 (3)	0.2439 (6)	0.19789 (14)	0.0687 (14)	
H2	0.151236	0.21805	0.208209	0.082*	

C3	0.2734 (3)	0.2487 (7)	0.22634 (14)	0.0860 (16)
H3	0.26712	0.22698	0.255532	0.103*
C4	0.3512 (3)	0.2855 (7)	0.21153 (15)	0.0702 (14)
C5	0.3613 (3)	0.3157 (6)	0.16906 (15)	0.0722 (14)
H5	0.414916	0.339042	0.159279	0.087*
C6	0.2925 (3)	0.3119 (6)	0.14031 (13)	0.0598 (12)
H6	0.300335	0.333225	0.111231	0.072*
C7	0.1555 (2)	0.2450 (6)	0.07998 (12)	0.0599 (12)
H7A	0.166523	0.119002	0.076636	0.072*
H7B	0.205192	0.309359	0.07138	0.072*
C8	0.0813 (3)	0.2965 (6)	0.05086 (13)	0.0690 (13)
H8A	0.075136	0.42483	0.050937	0.083*
H8B	0.09152	0.259489	0.021595	0.083*
C9	−0.0117 (3)	0.2610 (6)	0.10976 (14)	0.0669 (13)
H9A	−0.062504	0.202151	0.118906	0.08*
H9B	−0.020149	0.38837	0.112112	0.08*
C10	0.0624 (2)	0.2059 (6)	0.13852 (12)	0.0631 (13)
H10A	0.052793	0.242915	0.16785	0.076*
H10B	0.066876	0.077208	0.138331	0.076*
C11	0.9721 (3)	0.2971 (6)	0.30361 (13)	0.0510 (11)
C12	0.8980 (3)	0.2023 (6)	0.30443 (12)	0.0563 (12)
H12	0.888658	0.13067	0.328178	0.068*
C13	0.8365 (3)	0.2095 (6)	0.27110 (14)	0.0632 (13)
H13	0.78756	0.14142	0.272281	0.076*
C14	0.8487 (3)	0.3182 (6)	0.23643 (14)	0.0628 (12)
C15	0.9229 (3)	0.4129 (6)	0.23467 (13)	0.0649 (13)
H15	0.93208	0.484494	0.210883	0.078*
C16	0.9839 (3)	0.4028 (6)	0.26775 (14)	0.0626 (13)
H16	1.033592	0.46798	0.26601	0.075*
C17	1.0355 (3)	0.2887 (7)	0.34129 (16)	0.0608 (13)
C18	0.7146 (3)	0.2457 (8)	0.20218 (17)	0.118 (2)
H18C	0.681472	0.273815	0.176387	0.178*
H18B	0.725759	0.120242	0.20308	0.178*
H18A	0.684002	0.279268	0.226948	0.178*
C19	0.3816 (3)	0.5044 (6)	−0.09170 (13)	0.0503 (11)
C20	0.3778 (3)	0.6432 (6)	−0.06180 (13)	0.0582 (12)
H20	0.326082	0.697741	−0.057457	0.07*
C21	0.4478 (3)	0.6998 (6)	−0.03904 (13)	0.0603 (12)
H21	0.443054	0.790435	−0.018877	0.072*
C22	0.5251 (2)	0.6260 (6)	−0.04523 (13)	0.0499 (11)
C23	0.5330 (3)	0.4942 (6)	−0.07542 (14)	0.0610 (12)
H23	0.58581	0.445412	−0.080183	0.073*
C24	0.4629 (3)	0.4354 (6)	−0.09836 (13)	0.0623 (13)
H24	0.468943	0.347143	−0.119	0.075*
C25	0.3099 (3)	0.2720 (7)	−0.13618 (14)	0.0722 (14)
H25A	0.36547	0.248859	−0.147048	0.087*
H25B	0.269401	0.27516	−0.160582	0.087*
C26	0.2867 (3)	0.1245 (6)	−0.10589 (14)	0.0694 (13)

H26A	0.284623	0.011999	−0.121195	0.083*	
H26B	0.329107	0.115475	−0.082393	0.083*	
C27	0.1998 (2)	0.3414 (6)	−0.06853 (12)	0.0579 (12)	
H27A	0.237409	0.345068	−0.042968	0.07*	
H27B	0.142682	0.365292	−0.059877	0.07*	
C28	0.2258 (2)	0.4799 (6)	−0.10009 (13)	0.0583 (12)	
H28A	0.186099	0.480675	−0.124849	0.07*	
H28B	0.224623	0.596261	−0.086644	0.07*	
C29	0.2315 (2)	0.8165 (5)	0.03520 (12)	0.0442 (10)	
C30	0.3063 (2)	0.9094 (5)	0.03221 (13)	0.0517 (11)	
H30	0.319956	0.957242	0.005761	0.062*	
C31	0.3609 (2)	0.9330 (6)	0.06743 (15)	0.0597 (12)	
H31	0.411473	0.994162	0.064463	0.072*	
C32	0.3412 (3)	0.8662 (6)	0.10717 (15)	0.0570 (12)	
C33	0.2661 (3)	0.7752 (6)	0.11108 (13)	0.0590 (12)	
H33	0.251489	0.731519	0.137782	0.071*	
C34	0.2130 (2)	0.7495 (5)	0.07521 (13)	0.0524 (11)	
H34	0.163238	0.685277	0.077981	0.063*	
C35	0.1734 (3)	0.7879 (6)	−0.00372 (14)	0.0459 (11)	
C36	0.3801 (3)	0.8360 (7)	0.18157 (17)	0.1098 (19)	
H36C	0.42611	0.863024	0.201777	0.165*	
H36B	0.329805	0.895356	0.190161	0.165*	
H36A	0.370656	0.709953	0.181018	0.165*	
N1	0.14171 (19)	0.2823 (4)	0.12508 (10)	0.0475 (9)	
N2	0.0026 (2)	0.2144 (5)	0.06465 (13)	0.0581 (10)	
N3	0.4265 (6)	0.2464 (13)	0.2410 (3)	0.075 (2)	0.519 (6)
N3′	0.4213 (6)	0.3345 (15)	0.2420 (3)	0.075 (2)	0.481 (6)
N4	0.3106 (2)	0.4437 (5)	−0.11425 (10)	0.0583 (10)	
N5	0.2034 (2)	0.1639 (5)	−0.08869 (12)	0.0612 (10)	
N6	0.5983 (2)	0.6796 (6)	−0.01867 (13)	0.0650 (11)	
O1	0.4960 (7)	0.2571 (14)	0.2266 (4)	0.099 (2)	0.519 (6)
O1′	0.4904 (8)	0.3633 (14)	0.2283 (4)	0.099 (2)	0.481 (6)
O2	0.4177 (6)	0.2116 (13)	0.2788 (3)	0.097 (2)	0.519 (6)
O2′	0.4086 (6)	0.3408 (15)	0.2797 (3)	0.097 (2)	0.481 (6)
O3	1.1003 (2)	0.3818 (4)	0.33942 (9)	0.0826 (10)	
O4	1.0196 (2)	0.1889 (4)	0.37194 (10)	0.0789 (10)	
O5	0.7922 (2)	0.3398 (5)	0.20234 (9)	0.0895 (11)	
O6	0.58811 (19)	0.7838 (5)	0.01114 (11)	0.0827 (11)	
O7	0.66853 (19)	0.6203 (5)	−0.02668 (10)	0.0881 (11)	
O8	0.10807 (17)	0.6951 (4)	0.00037 (8)	0.0590 (8)	
O9	0.19207 (16)	0.8596 (4)	−0.03853 (9)	0.0593 (8)	
O10	0.40056 (18)	0.8950 (4)	0.13979 (10)	0.0790 (10)	
O11	1.00751 (19)	0.3517 (4)	0.44766 (10)	0.0649 (9)	
H21N	−0.0388 (19)	0.252 (5)	0.0468 (11)	0.078*	
H22N	0.007 (3)	0.099 (3)	0.0609 (12)	0.078*	
H51N	0.190 (2)	0.077 (4)	−0.0717 (11)	0.078*	
H52N	0.164 (2)	0.153 (6)	−0.1110 (9)	0.078*	
H11O	1.010 (3)	0.296 (5)	0.4244 (9)	0.078*	

H12O	0.969 (2)	0.305 (5)	0.4618 (12)	0.078*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.043 (3)	0.040 (3)	0.054 (3)	0.002 (2)	0.000 (2)	−0.002 (2)
C2	0.056 (3)	0.100 (4)	0.051 (3)	−0.008 (3)	0.007 (3)	0.001 (3)
C3	0.073 (3)	0.145 (5)	0.039 (3)	−0.011 (4)	−0.005 (3)	0.004 (3)
C4	0.052 (3)	0.108 (4)	0.050 (3)	−0.009 (3)	−0.013 (2)	0.001 (3)
C5	0.052 (3)	0.107 (4)	0.057 (3)	−0.017 (3)	−0.001 (3)	0.005 (3)
C6	0.049 (3)	0.082 (4)	0.048 (3)	−0.005 (3)	−0.006 (2)	0.009 (2)
C7	0.050 (3)	0.082 (3)	0.047 (3)	−0.009 (3)	−0.002 (2)	0.002 (2)
C8	0.064 (3)	0.077 (3)	0.064 (3)	−0.023 (3)	−0.018 (2)	0.017 (3)
C9	0.052 (3)	0.076 (4)	0.073 (4)	−0.003 (3)	−0.003 (2)	−0.015 (3)
C10	0.052 (3)	0.089 (4)	0.048 (3)	−0.013 (3)	0.000 (2)	−0.003 (2)
C11	0.055 (3)	0.049 (3)	0.049 (3)	0.004 (3)	0.004 (2)	0.002 (2)
C12	0.071 (3)	0.059 (3)	0.039 (3)	0.007 (3)	0.006 (2)	0.008 (2)
C13	0.058 (3)	0.074 (4)	0.058 (3)	−0.010 (3)	0.000 (2)	0.010 (3)
C14	0.064 (3)	0.071 (4)	0.051 (3)	0.007 (3)	−0.012 (3)	0.004 (3)
C15	0.068 (3)	0.070 (4)	0.056 (3)	−0.009 (3)	−0.004 (3)	0.018 (2)
C16	0.058 (3)	0.064 (3)	0.065 (3)	−0.009 (3)	0.000 (3)	0.009 (3)
C17	0.063 (3)	0.057 (4)	0.062 (3)	0.014 (3)	−0.010 (3)	−0.011 (3)
C18	0.103 (5)	0.148 (6)	0.100 (4)	−0.040 (4)	−0.047 (3)	0.023 (4)
C19	0.044 (3)	0.065 (3)	0.042 (3)	−0.002 (3)	0.007 (2)	0.009 (2)
C20	0.038 (3)	0.071 (3)	0.066 (3)	0.003 (3)	0.002 (2)	−0.004 (3)
C21	0.046 (3)	0.071 (3)	0.064 (3)	−0.002 (3)	0.008 (2)	−0.009 (2)
C22	0.035 (3)	0.064 (3)	0.051 (3)	−0.007 (2)	0.002 (2)	0.009 (2)
C23	0.038 (3)	0.068 (3)	0.077 (3)	0.004 (3)	0.012 (2)	0.005 (3)
C24	0.050 (3)	0.067 (3)	0.071 (3)	−0.003 (3)	0.015 (3)	−0.011 (2)
C25	0.063 (3)	0.100 (4)	0.054 (3)	−0.009 (3)	0.006 (2)	−0.023 (3)
C26	0.069 (3)	0.068 (4)	0.070 (3)	0.003 (3)	−0.008 (3)	−0.015 (3)
C27	0.049 (3)	0.070 (3)	0.054 (3)	−0.004 (3)	−0.003 (2)	−0.012 (3)
C28	0.042 (3)	0.063 (3)	0.069 (3)	−0.006 (2)	−0.008 (2)	0.001 (3)
C29	0.043 (2)	0.045 (3)	0.045 (3)	0.005 (2)	0.004 (2)	−0.003 (2)
C30	0.044 (3)	0.063 (3)	0.049 (3)	0.006 (2)	0.004 (2)	0.007 (2)
C31	0.044 (3)	0.071 (3)	0.064 (3)	−0.005 (2)	−0.005 (2)	−0.001 (3)
C32	0.053 (3)	0.064 (3)	0.053 (3)	0.012 (3)	−0.013 (3)	−0.008 (3)
C33	0.062 (3)	0.067 (3)	0.048 (3)	−0.001 (3)	−0.005 (2)	0.005 (2)
C34	0.052 (3)	0.055 (3)	0.050 (3)	−0.004 (2)	−0.002 (2)	0.006 (2)
C35	0.048 (3)	0.040 (3)	0.050 (3)	0.009 (2)	−0.005 (2)	−0.002 (2)
C36	0.117 (5)	0.125 (5)	0.083 (4)	0.008 (4)	−0.035 (3)	−0.004 (4)
N1	0.041 (2)	0.055 (2)	0.046 (2)	−0.0042 (18)	−0.0010 (17)	−0.0018 (17)
N2	0.055 (3)	0.047 (2)	0.070 (3)	−0.005 (2)	−0.0165 (19)	0.004 (2)
N3	0.072 (3)	0.081 (4)	0.070 (3)	−0.002 (3)	−0.004 (2)	−0.001 (3)
N3′	0.072 (3)	0.081 (4)	0.070 (3)	−0.002 (3)	−0.004 (2)	−0.001 (3)
N4	0.051 (2)	0.072 (3)	0.052 (2)	−0.005 (2)	0.0048 (19)	−0.002 (2)
N5	0.059 (3)	0.063 (3)	0.061 (3)	−0.012 (2)	−0.009 (2)	0.010 (2)
N6	0.048 (3)	0.084 (3)	0.064 (3)	−0.012 (3)	0.003 (2)	0.021 (2)

O1	0.075 (3)	0.127 (7)	0.092 (3)	−0.011 (5)	−0.020 (2)	−0.001 (6)
O1′	0.075 (3)	0.127 (7)	0.092 (3)	−0.011 (5)	−0.020 (2)	−0.001 (6)
O2	0.097 (3)	0.131 (6)	0.062 (3)	−0.003 (5)	−0.015 (2)	0.001 (5)
O2′	0.097 (3)	0.131 (6)	0.062 (3)	−0.003 (5)	−0.015 (2)	0.001 (5)
O3	0.073 (2)	0.088 (3)	0.084 (2)	−0.010 (2)	−0.0291 (18)	0.0096 (18)
O4	0.106 (3)	0.069 (2)	0.060 (2)	0.003 (2)	−0.0170 (18)	0.0134 (18)
O5	0.082 (2)	0.115 (3)	0.070 (2)	−0.013 (2)	−0.0256 (19)	0.0253 (19)
O6	0.072 (2)	0.110 (3)	0.066 (2)	−0.023 (2)	0.0016 (18)	−0.002 (2)
O7	0.046 (2)	0.128 (3)	0.090 (2)	−0.001 (2)	0.0000 (18)	0.023 (2)
O8	0.0548 (18)	0.061 (2)	0.060 (2)	−0.0126 (17)	−0.0085 (14)	0.0027 (15)
O9	0.0639 (19)	0.066 (2)	0.0473 (19)	0.0027 (16)	−0.0024 (15)	0.0080 (15)
O10	0.072 (2)	0.102 (3)	0.061 (2)	0.0032 (19)	−0.0220 (18)	−0.0004 (19)
O11	0.063 (2)	0.064 (2)	0.069 (2)	0.0012 (18)	0.0089 (17)	−0.0012 (17)

Geometric parameters (Å, º)

C1—N1	1.388 (4)	C21—H21	0.93
C1—C6	1.391 (5)	C22—C23	1.373 (5)
C1—C2	1.398 (5)	C22—N6	1.448 (5)
C2—C3	1.375 (6)	C23—C24	1.363 (5)
C2—H2	0.93	C23—H23	0.93
C3—C4	1.359 (6)	C24—H24	0.93
C3—H3	0.93	C25—N4	1.459 (5)
C4—C5	1.354 (5)	C25—C26	1.509 (6)
C4—N3′	1.471 (10)	C25—H25A	0.97
C4—N3	1.499 (9)	C25—H25B	0.97
C5—C6	1.375 (5)	C26—N5	1.474 (5)
C5—H5	0.93	C26—H26A	0.97
C6—H6	0.93	C26—H26B	0.97
C7—N1	1.453 (4)	C27—N5	1.476 (5)
C7—C8	1.499 (5)	C27—C28	1.499 (5)
C7—H7A	0.97	C27—H27A	0.97
C7—H7B	0.97	C27—H27B	0.97
C8—N2	1.469 (5)	C28—N4	1.455 (4)
C8—H8A	0.97	C28—H28A	0.97
C8—H8B	0.97	C28—H28B	0.97
C9—N2	1.470 (5)	C29—C30	1.379 (5)
C9—C10	1.499 (5)	C29—C34	1.383 (5)
C9—H9A	0.97	C29—C35	1.499 (5)
C9—H9B	0.97	C30—C31	1.374 (5)
C10—N1	1.457 (4)	C30—H30	0.93
C10—H10A	0.97	C31—C32	1.379 (5)
C10—H10B	0.97	C31—H31	0.93
C11—C12	1.372 (5)	C32—O10	1.366 (4)
C11—C16	1.387 (5)	C32—C33	1.380 (5)
C11—C17	1.506 (6)	C33—C34	1.377 (5)
C12—C13	1.388 (5)	C33—H33	0.93
C12—H12	0.93	C34—H34	0.93

C13—C14	1.371 (5)	C35—O9	1.254 (4)
C13—H13	0.93	C35—O8	1.258 (4)
C14—O5	1.363 (5)	C36—O10	1.421 (5)
C14—C15	1.375 (5)	C36—H36C	0.96
C15—C16	1.378 (5)	C36—H36B	0.96
C15—H15	0.93	C36—H36A	0.96
C16—H16	0.93	N2—H21N	0.884 (18)
C17—O3	1.245 (5)	N2—H22N	0.882 (18)
C17—O4	1.246 (5)	N3—O1	1.208 (9)
C18—O5	1.416 (5)	N3—O2	1.213 (9)
C18—H18C	0.96	N3′—O2′	1.196 (9)
C18—H18B	0.96	N3′—O1′	1.210 (10)
C18—H18A	0.96	N5—H51N	0.872 (18)
C19—N4	1.374 (4)	N5—H52N	0.910 (18)
C19—C20	1.399 (5)	N6—O6	1.228 (4)
C19—C24	1.410 (5)	N6—O7	1.232 (4)
C20—C21	1.354 (5)	O11—H11O	0.836 (18)
C20—H20	0.93	O11—H12O	0.838 (18)
C21—C22	1.363 (5)		

N1—C1—C6	121.1 (4)	C22—C23—H23	120.2
N1—C1—C2	122.4 (4)	C23—C24—C19	121.9 (4)
C6—C1—C2	116.5 (4)	C23—C24—H24	119
C3—C2—C1	121.6 (4)	C19—C24—H24	119
C3—C2—H2	119.2	N4—C25—C26	110.9 (3)
C1—C2—H2	119.2	N4—C25—H25A	109.5
C4—C3—C2	119.6 (4)	C26—C25—H25A	109.5
C4—C3—H3	120.2	N4—C25—H25B	109.5
C2—C3—H3	120.2	C26—C25—H25B	109.5
C5—C4—C3	120.7 (4)	H25A—C25—H25B	108
C5—C4—N3′	117.8 (6)	N5—C26—C25	108.9 (4)
C3—C4—N3′	120.0 (6)	N5—C26—H26A	109.9
C5—C4—N3	120.2 (6)	C25—C26—H26A	109.9
C3—C4—N3	117.3 (6)	N5—C26—H26B	109.9
C4—C5—C6	120.2 (4)	C25—C26—H26B	109.9
C4—C5—H5	119.9	H26A—C26—H26B	108.3
C6—C5—H5	119.9	N5—C27—C28	109.5 (3)
C5—C6—C1	121.3 (4)	N5—C27—H27A	109.8
C5—C6—H6	119.3	C28—C27—H27A	109.8
C1—C6—H6	119.3	N5—C27—H27B	109.8
N1—C7—C8	112.5 (3)	C28—C27—H27B	109.8
N1—C7—H7A	109.1	H27A—C27—H27B	108.2
C8—C7—H7A	109.1	N4—C28—C27	110.5 (3)
N1—C7—H7B	109.1	N4—C28—H28A	109.5
C8—C7—H7B	109.1	C27—C28—H28A	109.5
H7A—C7—H7B	107.8	N4—C28—H28B	109.5
N2—C8—C7	111.5 (3)	C27—C28—H28B	109.5
N2—C8—H8A	109.3	H28A—C28—H28B	108.1

C7—C8—H8A	109.3	C30—C29—C34	117.5 (4)
N2—C8—H8B	109.3	C30—C29—C35	120.9 (4)
C7—C8—H8B	109.3	C34—C29—C35	121.6 (4)
H8A—C8—H8B	108	C31—C30—C29	121.5 (4)
N2—C9—C10	110.5 (3)	C31—C30—H30	119.3
N2—C9—H9A	109.5	C29—C30—H30	119.3
C10—C9—H9A	109.5	C30—C31—C32	120.3 (4)
N2—C9—H9B	109.5	C30—C31—H31	119.8
C10—C9—H9B	109.5	C32—C31—H31	119.8
H9A—C9—H9B	108.1	O10—C32—C31	115.4 (4)
N1—C10—C9	112.3 (3)	O10—C32—C33	125.3 (4)
N1—C10—H10A	109.1	C31—C32—C33	119.2 (4)
C9—C10—H10A	109.1	C34—C33—C32	119.6 (4)
N1—C10—H10B	109.1	C34—C33—H33	120.2
C9—C10—H10B	109.1	C32—C33—H33	120.2
H10A—C10—H10B	107.9	C33—C34—C29	121.8 (4)
C12—C11—C16	117.2 (4)	C33—C34—H34	119.1
C12—C11—C17	120.2 (4)	C29—C34—H34	119.1
C16—C11—C17	122.5 (4)	O9—C35—O8	123.4 (4)
C11—C12—C13	122.4 (4)	O9—C35—C29	118.2 (4)
C11—C12—H12	118.8	O8—C35—C29	118.3 (4)
C13—C12—H12	118.8	O10—C36—H36C	109.5
C14—C13—C12	119.3 (4)	O10—C36—H36B	109.5
C14—C13—H13	120.4	H36C—C36—H36B	109.5
C12—C13—H13	120.4	O10—C36—H36A	109.5
O5—C14—C13	124.7 (4)	H36C—C36—H36A	109.5
O5—C14—C15	116.0 (4)	H36B—C36—H36A	109.5
C13—C14—C15	119.3 (4)	C1—N1—C7	117.7 (3)
C14—C15—C16	120.7 (4)	C1—N1—C10	118.2 (3)
C14—C15—H15	119.6	C7—N1—C10	111.6 (3)
C16—C15—H15	119.6	C8—N2—C9	110.2 (3)
C15—C16—C11	121.0 (4)	C8—N2—H21N	107 (3)
C15—C16—H16	119.5	C9—N2—H21N	112 (3)
C11—C16—H16	119.5	C8—N2—H22N	108 (3)
O3—C17—O4	124.6 (4)	C9—N2—H22N	112 (3)
O3—C17—C11	117.5 (5)	H21N—N2—H22N	107 (4)
O4—C17—C11	117.9 (5)	O1—N3—O2	121.2 (11)
O5—C18—H18C	109.5	O1—N3—C4	118.2 (10)
O5—C18—H18B	109.5	O2—N3—C4	120.6 (9)
H18C—C18—H18B	109.5	O2′—N3′—O1′	122.0 (11)
O5—C18—H18A	109.5	O2′—N3′—C4	119.0 (9)
H18C—C18—H18A	109.5	O1′—N3′—C4	119.0 (10)
H18B—C18—H18A	109.5	C19—N4—C28	121.8 (3)
N4—C19—C20	121.8 (4)	C19—N4—C25	121.4 (4)
N4—C19—C24	122.2 (4)	C28—N4—C25	108.6 (3)
C20—C19—C24	116.0 (4)	C26—N5—C27	112.8 (3)
C21—C20—C19	121.5 (4)	C26—N5—H51N	108 (3)
C21—C20—H20	119.3	C27—N5—H51N	114 (3)

C19—C20—H20	119.3	C26—N5—H52N	107 (3)
C20—C21—C22	121.0 (4)	C27—N5—H52N	112 (3)
C20—C21—H21	119.5	H51N—N5—H52N	103 (4)
C22—C21—H21	119.5	O6—N6—O7	122.1 (4)
C21—C22—C23	120.0 (4)	O6—N6—C22	118.5 (4)
C21—C22—N6	120.4 (4)	O7—N6—C22	119.3 (4)
C23—C22—N6	119.6 (4)	C14—O5—C18	118.7 (4)
C24—C23—C22	119.6 (4)	C32—O10—C36	116.7 (4)
C24—C23—H23	120.2	H11O—O11—H12O	108 (5)

N1—C1—C2—C3	−176.3 (4)	O10—C32—C33—C34	−177.5 (4)
C6—C1—C2—C3	1.0 (6)	C31—C32—C33—C34	1.3 (6)
C1—C2—C3—C4	−0.3 (8)	C32—C33—C34—C29	−1.7 (6)
C2—C3—C4—C5	−0.7 (8)	C30—C29—C34—C33	0.7 (6)
C2—C3—C4—N3′	164.9 (6)	C35—C29—C34—C33	179.9 (4)
C2—C3—C4—N3	−165.5 (6)	C30—C29—C35—O9	−4.2 (5)
C3—C4—C5—C6	0.9 (8)	C34—C29—C35—O9	176.6 (3)
N3′—C4—C5—C6	−165.0 (6)	C30—C29—C35—O8	176.7 (3)
N3—C4—C5—C6	165.4 (6)	C34—C29—C35—O8	−2.5 (5)
C4—C5—C6—C1	−0.2 (7)	C6—C1—N1—C7	27.3 (5)
N1—C1—C6—C5	176.6 (4)	C2—C1—N1—C7	−155.5 (4)
C2—C1—C6—C5	−0.7 (6)	C6—C1—N1—C10	166.2 (4)
N1—C7—C8—N2	−54.0 (5)	C2—C1—N1—C10	−16.6 (6)
N2—C9—C10—N1	56.1 (5)	C8—C7—N1—C1	−166.4 (3)
C16—C11—C12—C13	0.2 (6)	C8—C7—N1—C10	52.2 (4)
C17—C11—C12—C13	178.1 (4)	C9—C10—N1—C1	165.3 (3)
C11—C12—C13—C14	−1.6 (7)	C9—C10—N1—C7	−53.4 (5)
C12—C13—C14—O5	−178.3 (4)	C7—C8—N2—C9	55.9 (5)
C12—C13—C14—C15	2.3 (7)	C10—C9—N2—C8	−56.8 (4)
O5—C14—C15—C16	179.0 (4)	C5—C4—N3—O1	5.9 (11)
C13—C14—C15—C16	−1.6 (7)	C3—C4—N3—O1	170.9 (8)
C14—C15—C16—C11	0.1 (7)	C5—C4—N3—O2	−176.8 (7)
C12—C11—C16—C15	0.6 (6)	C3—C4—N3—O2	−11.8 (11)
C17—C11—C16—C15	−177.3 (4)	C5—C4—N3′—O2′	166.2 (8)
C12—C11—C17—O3	−177.7 (4)	C3—C4—N3′—O2′	0.2 (12)
C16—C11—C17—O3	0.1 (6)	C5—C4—N3′—O1′	−15.5 (12)
C12—C11—C17—O4	2.6 (6)	C3—C4—N3′—O1′	178.5 (9)
C16—C11—C17—O4	−179.5 (4)	C20—C19—N4—C28	−18.5 (6)
N4—C19—C20—C21	178.6 (4)	C24—C19—N4—C28	163.8 (4)
C24—C19—C20—C21	−3.6 (6)	C20—C19—N4—C25	−163.3 (4)
C19—C20—C21—C22	1.6 (6)	C24—C19—N4—C25	19.0 (6)
C20—C21—C22—C23	1.1 (6)	C27—C28—N4—C19	−86.8 (5)
C20—C21—C22—N6	−175.8 (4)	C27—C28—N4—C25	61.9 (4)
C21—C22—C23—C24	−1.4 (6)	C26—C25—N4—C19	86.9 (4)
N6—C22—C23—C24	175.5 (4)	C26—C25—N4—C28	−61.9 (4)
C22—C23—C24—C19	−0.8 (6)	C25—C26—N5—C27	−54.1 (5)
N4—C19—C24—C23	−179.0 (4)	C28—C27—N5—C26	54.7 (4)
C20—C19—C24—C23	3.2 (6)	C21—C22—N6—O6	4.5 (6)

N4—C25—C26—N5	57.6 (5)	C23—C22—N6—O6	−172.4 (4)
N5—C27—C28—N4	−58.0 (4)	C21—C22—N6—O7	−175.1 (4)
C34—C29—C30—C31	0.8 (6)	C23—C22—N6—O7	8.0 (6)
C35—C29—C30—C31	−178.5 (4)	C13—C14—O5—C18	0.5 (7)
C29—C30—C31—C32	−1.2 (6)	C15—C14—O5—C18	179.9 (4)
C30—C31—C32—O10	179.1 (4)	C31—C32—O10—C36	176.8 (4)
C30—C31—C32—C33	0.2 (6)	C33—C32—O10—C36	−4.4 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*B*···O7^{i}	0.97	2.54	3.451 (5)	157
C9—H9*B*···O4^{ii}	0.97	2.31	3.270 (5)	169
C20—H20···O9	0.93	2.53	3.461 (5)	174
C25—H25*A*···O2a^{iii}	0.97	2.5	3.206 (10)	130
C25—H25*A*···O2′b^{iii}	0.97	2.49	3.212 (11)	131
C27—H27*A*···O7^{i}	0.97	2.58	3.548 (5)	175
C28—H28*B*···O9	0.97	2.55	3.489 (5)	164
C36—H36*C*···O1′b^{ii}	0.96	2.49	3.395 (14)	158
N2—H21*N*···O8^{iv}	0.88 (2)	1.83 (2)	2.697 (4)	166 (4)
N2—H21*N*···O9^{iv}	0.88 (2)	2.57 (3)	3.196 (4)	129 (3)
N2—H22*N*···O11^{v}	0.88 (2)	1.89 (2)	2.758 (5)	169 (4)
N5—H51*N*···O9^{vi}	0.87 (2)	1.93 (2)	2.778 (5)	164 (4)
N5—H52*N*···O3^{vii}	0.91 (2)	1.82 (2)	2.724 (5)	171 (4)
O11—H11*O*···O4	0.84 (2)	1.83 (2)	2.663 (4)	176 (4)
O11—H12*O*···O8^{v}	0.84 (2)	1.92 (2)	2.754 (4)	173 (4)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) *x*, −*y*+1/2, *z*−1/2; (iv) −*x*, −*y*+1, −*z*; (v) −*x*+1, *y*−1/2, −*z*+1/2; (vi) *x*, *y*−1, *z*; (vii) *x*−1, −*y*+1/2, *z*−1/2.

4-(4-Nitrophenyl)piperazin-1-ium 4-ethoxybenzoate (VI)

Crystal data

$C_{10}H_{14}N_3O_2^+·C_9H_9O_3^-$
M_r = 373.4
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.874 (1) Å
b = 9.263 (1) Å
c = 27.996 (3) Å
α = 81.030 (6)°
β = 85.675 (6)°
γ = 68.229 (5)°
V = 1872.8 (4) Å^3
Z = 4
F(000) = 792
D_x = 1.324 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4134 reflections
θ = 2.4–28.0°
μ = 0.10 mm^{-1}
T = 293 K
Plate, yellow
0.44 × 0.32 × 0.08 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)
T_{min} = 0.963, T_{max} = 0.992
13344 measured reflections
6868 independent reflections
3803 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

θ_{max} = 25.4°, θ_{min} = 2.4°
h = −9→5
k = −11→10
l = −33→33

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.137
S = 1.05
6858 reflections
501 parameters
16 restraints
0 constraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.7484P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.3466 (3)	0.6545 (4)	0.40786 (9)	0.1126 (10)
O2	−0.1630 (3)	0.5811 (3)	0.46683 (9)	0.0901 (8)
N3	−0.1931 (4)	0.6107 (3)	0.42384 (10)	0.0667 (7)
N1	0.3914 (3)	0.5404 (2)	0.29014 (7)	0.0412 (5)
N2	0.6838 (3)	0.4199 (3)	0.22275 (9)	0.0486 (6)
C1	0.2485 (3)	0.5547 (3)	0.32330 (9)	0.0364 (6)
C2	0.0659 (4)	0.6276 (3)	0.30870 (10)	0.0452 (7)
H2	0.041054	0.663561	0.276117	0.054*
C3	−0.0759 (4)	0.6467 (3)	0.34138 (10)	0.0487 (7)
H3	−0.195824	0.69631	0.330993	0.058*
C4	−0.0417 (4)	0.5927 (3)	0.38966 (10)	0.0470 (7)
C5	0.1352 (4)	0.5197 (3)	0.40558 (10)	0.0460 (7)
H5	0.157333	0.483265	0.438246	0.055*
C6	0.2787 (4)	0.5009 (3)	0.37309 (10)	0.0430 (7)
H6	0.397837	0.451799	0.384052	0.052*
C7	0.5772 (3)	0.4477 (3)	0.30663 (10)	0.0473 (7)
H7A	0.588757	0.338899	0.315234	0.057*
H7B	0.597413	0.484321	0.33554	0.057*
C8	0.7216 (4)	0.4565 (4)	0.26954 (10)	0.0539 (8)
H8A	0.725864	0.561056	0.265102	0.065*
H8B	0.839991	0.382613	0.280859	0.065*
C9	0.5080 (4)	0.5395 (3)	0.20545 (10)	0.0528 (8)
H9A	0.482935	0.520499	0.174084	0.063*
H9B	0.516375	0.642512	0.201592	0.063*
C10	0.3546 (4)	0.5366 (4)	0.23985 (9)	0.0527 (8)
H10A	0.245506	0.626087	0.229683	0.063*
H10B	0.329888	0.442345	0.238274	0.063*

O3	−0.0728 (3)	0.4331 (2)	0.15512 (7)	0.0578 (5)
O4	0.1149 (2)	0.2978 (2)	0.21489 (7)	0.0532 (5)
O5	0.6778 (3)	0.3001 (3)	0.03965 (7)	0.0734 (7)
C11	0.2434 (4)	0.3357 (3)	0.13647 (9)	0.0400 (6)
C12	0.4193 (4)	0.2483 (3)	0.15096 (10)	0.0518 (8)
H12	0.438717	0.198421	0.182667	0.062*
C13	0.5679 (4)	0.2321 (4)	0.11995 (10)	0.0572 (8)
H13	0.685415	0.171644	0.130663	0.069*
C14	0.5409 (4)	0.3059 (3)	0.07314 (10)	0.0524 (8)
C15	0.3656 (4)	0.3930 (4)	0.05764 (10)	0.0609 (9)
H15	0.346406	0.442258	0.02587	0.073*
C16	0.2192 (4)	0.4072 (3)	0.08898 (10)	0.0520 (8)
H16	0.101521	0.466057	0.078056	0.062*
C17	0.0851 (4)	0.3564 (3)	0.17148 (10)	0.0424 (7)
C18	0.8615 (4)	0.2090 (4)	0.05351 (12)	0.0820 (11)
H18A	0.874909	0.100553	0.064059	0.098*
H18B	0.894362	0.249403	0.079923	0.098*
C19	0.9824 (5)	0.2199 (5)	0.01002 (14)	0.1126 (16)
H19A	1.106148	0.152009	0.017216	0.169*
H19B	0.976652	0.32615	0.001769	0.169*
H19C	0.941525	0.188565	−0.016723	0.169*
O6	0.1516 (4)	0.7336 (4)	0.60499 (10)	0.1157 (11)
O7	0.1799 (4)	0.9508 (4)	0.61439 (10)	0.1162 (11)
N6	0.1845 (4)	0.8525 (5)	0.58929 (11)	0.0830 (10)
N4	0.2839 (3)	0.9838 (3)	0.38867 (8)	0.0490 (6)
N5	0.2937 (3)	1.0699 (3)	0.28629 (8)	0.0462 (6)
C20	0.2721 (4)	0.9472 (3)	0.43812 (10)	0.0459 (7)
C21	0.2534 (4)	0.8084 (4)	0.45928 (11)	0.0653 (9)
H21	0.259467	0.733784	0.439839	0.078*
C22	0.2260 (5)	0.7782 (4)	0.50814 (12)	0.0747 (10)
H22	0.211482	0.684754	0.521174	0.09*
C23	0.2199 (4)	0.8824 (5)	0.53759 (11)	0.0636 (9)
C24	0.2430 (5)	1.0184 (5)	0.51872 (13)	0.0783 (11)
H24	0.240692	1.089852	0.538938	0.094*
C25	0.2700 (5)	1.0504 (4)	0.46951 (12)	0.0711 (10)
H25	0.287119	1.143254	0.457048	0.085*
C26	0.3867 (4)	1.0795 (3)	0.36697 (10)	0.0563 (8)
H26A	0.51213	1.012589	0.360971	0.068*
H26B	0.3881	1.149872	0.389107	0.068*
C27	0.3030 (4)	1.1732 (3)	0.32039 (10)	0.0539 (8)
H27A	0.180752	1.245608	0.326783	0.065*
H27B	0.375328	1.234293	0.305927	0.065*
C28	0.1907 (4)	0.9701 (3)	0.30890 (10)	0.0498 (7)
H28A	0.193308	0.897196	0.28723	0.06*
H28B	0.064102	1.035801	0.314055	0.06*
C29	0.2714 (4)	0.8803 (3)	0.35626 (10)	0.0529 (8)
H29A	0.196135	0.822751	0.371369	0.064*
H29B	0.392544	0.804857	0.35057	0.064*

O8	−0.3777 (3)	0.8954 (2)	0.25237 (8)	0.0629 (6)
O9	−0.3341 (3)	1.1185 (2)	0.25354 (7)	0.0569 (5)
O10	0.2958 (3)	0.8011 (3)	0.10557 (8)	0.0771 (7)
C30	−0.1331 (3)	0.9365 (3)	0.20503 (9)	0.0408 (6)
C31	−0.1058 (4)	0.8152 (3)	0.17834 (10)	0.0508 (7)
H31	−0.185001	0.760423	0.183038	0.061*
C32	0.0357 (4)	0.7741 (3)	0.14506 (11)	0.0555 (8)
H32	0.049421	0.694181	0.127006	0.067*
C33	0.1575 (4)	0.8517 (3)	0.13846 (10)	0.0511 (7)
C34	0.1341 (4)	0.9716 (3)	0.16508 (10)	0.0512 (7)
H34	0.215362	1.024354	0.161223	0.061*
C35	−0.0111 (4)	1.0121 (3)	0.19741 (10)	0.0463 (7)
H35	−0.027019	1.09415	0.214785	0.056*
C36	−0.2919 (4)	0.9871 (4)	0.23973 (10)	0.0478 (7)
C37	0.4280 (5)	0.8751 (5)	0.09807 (14)	0.0885 (12)
H37A	0.483742	0.868761	0.128435	0.106*
H37B	0.369923	0.984835	0.085011	0.106*
C38	0.5698 (6)	0.7907 (7)	0.06316 (18)	0.153 (2)
H38A	0.667443	0.830217	0.059976	0.229*
H38B	0.515701	0.807254	0.032204	0.229*
H38C	0.617246	0.680406	0.074893	0.229*
H31N	0.778 (5)	0.425 (6)	0.2007 (14)	0.183*
H32N	0.677 (7)	0.324 (3)	0.2290 (18)	0.183*
H61N	0.407 (4)	1.009 (5)	0.2766 (18)	0.183*
H62N	0.236 (6)	1.131 (5)	0.2599 (12)	0.183*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0514 (15)	0.173 (3)	0.0801 (19)	−0.0138 (17)	0.0103 (14)	0.0040 (18)
O2	0.0855 (17)	0.129 (2)	0.0445 (15)	−0.0320 (16)	0.0107 (13)	−0.0023 (14)
N3	0.0550 (18)	0.076 (2)	0.0573 (19)	−0.0143 (15)	0.0078 (15)	−0.0036 (15)
N1	0.0399 (13)	0.0448 (14)	0.0380 (13)	−0.0148 (11)	−0.0030 (10)	−0.0033 (11)
N2	0.0450 (14)	0.0536 (16)	0.0466 (15)	−0.0175 (13)	0.0038 (11)	−0.0089 (13)
C1	0.0440 (16)	0.0302 (15)	0.0369 (16)	−0.0153 (13)	−0.0019 (13)	−0.0053 (12)
C2	0.0486 (17)	0.0463 (17)	0.0378 (16)	−0.0152 (14)	−0.0064 (14)	−0.0001 (13)
C3	0.0392 (16)	0.0525 (19)	0.0513 (19)	−0.0132 (14)	−0.0007 (14)	−0.0071 (15)
C4	0.0488 (18)	0.0463 (18)	0.0441 (18)	−0.0167 (15)	0.0066 (14)	−0.0065 (14)
C5	0.0584 (19)	0.0406 (17)	0.0386 (16)	−0.0186 (15)	−0.0016 (14)	−0.0020 (13)
C6	0.0433 (16)	0.0381 (16)	0.0457 (17)	−0.0127 (13)	−0.0066 (14)	−0.0027 (13)
C7	0.0407 (16)	0.0543 (18)	0.0470 (18)	−0.0162 (14)	−0.0043 (13)	−0.0088 (14)
C8	0.0440 (17)	0.066 (2)	0.057 (2)	−0.0243 (15)	0.0013 (15)	−0.0150 (16)
C9	0.0544 (18)	0.0547 (19)	0.0451 (18)	−0.0186 (16)	0.0029 (14)	0.0005 (14)
C10	0.0452 (17)	0.068 (2)	0.0392 (17)	−0.0168 (15)	−0.0010 (13)	−0.0005 (15)
O3	0.0458 (12)	0.0732 (15)	0.0477 (12)	−0.0181 (11)	0.0023 (10)	0.0009 (11)
O4	0.0585 (12)	0.0634 (13)	0.0375 (12)	−0.0259 (11)	0.0003 (9)	0.0022 (10)
O5	0.0505 (13)	0.0959 (17)	0.0504 (13)	−0.0107 (12)	0.0086 (11)	0.0129 (12)
C11	0.0480 (17)	0.0392 (16)	0.0352 (15)	−0.0199 (14)	0.0001 (13)	−0.0026 (13)

C12	0.0524 (18)	0.064 (2)	0.0362 (16)	−0.0234 (16)	−0.0022 (14)	0.0074 (14)
C13	0.0453 (17)	0.072 (2)	0.0455 (19)	−0.0155 (16)	−0.0029 (15)	0.0053 (16)
C14	0.0489 (18)	0.060 (2)	0.0424 (18)	−0.0173 (16)	0.0060 (15)	−0.0001 (15)
C15	0.056 (2)	0.075 (2)	0.0327 (17)	−0.0089 (17)	0.0009 (15)	0.0090 (15)
C16	0.0458 (17)	0.0560 (19)	0.0431 (18)	−0.0091 (15)	−0.0006 (14)	0.0016 (15)
C17	0.0498 (18)	0.0416 (17)	0.0418 (18)	−0.0237 (15)	0.0000 (14)	−0.0054 (14)
C18	0.050 (2)	0.106 (3)	0.069 (2)	−0.014 (2)	0.0095 (17)	0.007 (2)
C19	0.058 (2)	0.154 (4)	0.088 (3)	−0.011 (2)	0.022 (2)	0.011 (3)
O6	0.125 (3)	0.136 (3)	0.0580 (18)	−0.028 (2)	0.0049 (16)	0.0186 (18)
O7	0.099 (2)	0.208 (3)	0.0619 (18)	−0.070 (2)	0.0107 (15)	−0.049 (2)
N6	0.0584 (19)	0.129 (3)	0.049 (2)	−0.022 (2)	0.0005 (15)	−0.010 (2)
N4	0.0744 (17)	0.0449 (14)	0.0381 (14)	−0.0343 (13)	−0.0005 (12)	−0.0046 (11)
N5	0.0553 (15)	0.0425 (15)	0.0435 (14)	−0.0231 (12)	0.0049 (12)	−0.0033 (12)
C20	0.0459 (16)	0.0463 (18)	0.0443 (18)	−0.0147 (14)	0.0001 (13)	−0.0083 (14)
C21	0.099 (3)	0.054 (2)	0.0440 (19)	−0.0301 (19)	0.0055 (17)	−0.0063 (16)
C22	0.101 (3)	0.070 (2)	0.047 (2)	−0.031 (2)	0.0048 (19)	0.0032 (18)
C23	0.0510 (19)	0.091 (3)	0.0396 (19)	−0.0177 (19)	−0.0027 (15)	−0.0030 (19)
C24	0.087 (3)	0.110 (3)	0.051 (2)	−0.041 (2)	0.0054 (19)	−0.038 (2)
C25	0.099 (3)	0.074 (2)	0.059 (2)	−0.049 (2)	0.0046 (19)	−0.0196 (19)
C26	0.071 (2)	0.058 (2)	0.0524 (19)	−0.0381 (17)	0.0000 (16)	−0.0085 (16)
C27	0.072 (2)	0.0427 (17)	0.0533 (19)	−0.0303 (16)	0.0104 (16)	−0.0060 (15)
C28	0.0607 (19)	0.0526 (18)	0.0439 (18)	−0.0310 (16)	0.0008 (14)	−0.0043 (14)
C29	0.077 (2)	0.0475 (18)	0.0436 (18)	−0.0344 (16)	−0.0019 (15)	−0.0032 (14)
O8	0.0597 (13)	0.0531 (13)	0.0781 (15)	−0.0273 (11)	0.0142 (11)	−0.0062 (11)
O9	0.0627 (13)	0.0524 (13)	0.0573 (13)	−0.0226 (11)	0.0088 (10)	−0.0129 (11)
O10	0.0744 (15)	0.0862 (17)	0.0732 (16)	−0.0304 (14)	0.0256 (13)	−0.0281 (13)
C30	0.0447 (16)	0.0366 (16)	0.0382 (16)	−0.0133 (13)	−0.0061 (13)	0.0021 (13)
C31	0.0524 (18)	0.0469 (18)	0.0559 (19)	−0.0229 (15)	−0.0043 (15)	−0.0020 (15)
C32	0.064 (2)	0.0469 (18)	0.056 (2)	−0.0192 (16)	−0.0013 (16)	−0.0139 (15)
C33	0.0507 (18)	0.0488 (18)	0.0463 (18)	−0.0113 (15)	0.0020 (14)	−0.0032 (15)
C34	0.0482 (17)	0.0498 (19)	0.058 (2)	−0.0232 (15)	0.0023 (15)	−0.0036 (16)
C35	0.0490 (17)	0.0437 (17)	0.0490 (18)	−0.0186 (14)	−0.0048 (14)	−0.0077 (14)
C36	0.0480 (18)	0.0481 (19)	0.0442 (18)	−0.0162 (15)	−0.0073 (14)	0.0022 (15)
C37	0.075 (3)	0.110 (3)	0.082 (3)	−0.040 (2)	0.025 (2)	−0.014 (2)
C38	0.122 (4)	0.205 (6)	0.147 (5)	−0.072 (4)	0.083 (4)	−0.079 (4)

Geometric parameters (Å, º)

O1—N3	1.220 (3)	O6—N6	1.231 (4)
O2—N3	1.213 (3)	O7—N6	1.223 (4)
N3—C4	1.447 (3)	N6—C23	1.456 (4)
N1—C1	1.384 (3)	N4—C20	1.378 (3)
N1—C7	1.462 (3)	N4—C26	1.449 (3)
N1—C10	1.467 (3)	N4—C29	1.452 (3)
N2—C9	1.475 (3)	N5—C27	1.476 (4)
N2—C8	1.481 (4)	N5—C28	1.486 (3)
N2—H31N	0.937 (19)	N5—H61N	0.910 (19)
N2—H32N	0.895 (19)	N5—H62N	0.896 (19)

C1—C2	1.405 (3)	C20—C21	1.384 (4)
C1—C6	1.412 (3)	C20—C25	1.391 (4)
C2—C3	1.366 (4)	C21—C22	1.370 (4)
C2—H2	0.93	C21—H21	0.93
C3—C4	1.376 (4)	C22—C23	1.349 (4)
C3—H3	0.93	C22—H22	0.93
C4—C5	1.377 (4)	C23—C24	1.359 (5)
C5—C6	1.372 (3)	C24—C25	1.381 (4)
C5—H5	0.93	C24—H24	0.93
C6—H6	0.93	C25—H25	0.93
C7—C8	1.498 (3)	C26—C27	1.497 (4)
C7—H7A	0.97	C26—H26A	0.97
C7—H7B	0.97	C26—H26B	0.97
C8—H8A	0.97	C27—H27A	0.97
C8—H8B	0.97	C27—H27B	0.97
C9—C10	1.493 (3)	C28—C29	1.498 (4)
C9—H9A	0.97	C28—H28A	0.97
C9—H9B	0.97	C28—H28B	0.97
C10—H10A	0.97	C29—H29A	0.97
C10—H10B	0.97	C29—H29B	0.97
O3—C17	1.261 (3)	O8—C36	1.264 (3)
O4—C17	1.253 (3)	O9—C36	1.252 (3)
O5—C14	1.365 (3)	O10—C33	1.363 (3)
O5—C18	1.426 (3)	O10—C37	1.431 (4)
C11—C12	1.375 (4)	C30—C35	1.372 (3)
C11—C16	1.383 (3)	C30—C31	1.387 (4)
C11—C17	1.499 (4)	C30—C36	1.500 (4)
C12—C13	1.379 (4)	C31—C32	1.376 (4)
C12—H12	0.93	C31—H31	0.93
C13—C14	1.373 (4)	C32—C33	1.383 (4)
C13—H13	0.93	C32—H32	0.93
C14—C15	1.378 (4)	C33—C34	1.381 (4)
C15—C16	1.374 (4)	C34—C35	1.378 (4)
C15—H15	0.93	C34—H34	0.93
C16—H16	0.93	C35—H35	0.93
C18—C19	1.502 (4)	C37—C38	1.496 (5)
C18—H18A	0.97	C37—H37A	0.97
C18—H18B	0.97	C37—H37B	0.97
C19—H19A	0.96	C38—H38A	0.96
C19—H19B	0.96	C38—H38B	0.96
C19—H19C	0.96	C38—H38C	0.96

O2—N3—O1	122.7 (3)	O7—N6—O6	123.5 (4)
O2—N3—C4	119.3 (3)	O7—N6—C23	118.2 (4)
O1—N3—C4	118.0 (3)	O6—N6—C23	118.2 (4)
C1—N1—C7	118.1 (2)	C20—N4—C26	121.3 (2)
C1—N1—C10	117.0 (2)	C20—N4—C29	121.3 (2)
C7—N1—C10	116.3 (2)	C26—N4—C29	111.9 (2)

C9—N2—C8	107.9 (2)	C27—N5—C28	110.0 (2)
C9—N2—H31N	110 (3)	C27—N5—H61N	112 (3)
C8—N2—H31N	108 (3)	C28—N5—H61N	110 (3)
C9—N2—H32N	111 (3)	C27—N5—H62N	108 (3)
C8—N2—H32N	106 (3)	C28—N5—H62N	110 (3)
H31N—N2—H32N	113 (4)	H61N—N5—H62N	107 (4)
N1—C1—C2	120.9 (2)	N4—C20—C21	121.8 (3)
N1—C1—C6	122.0 (2)	N4—C20—C25	122.0 (3)
C2—C1—C6	117.1 (2)	C21—C20—C25	116.2 (3)
C3—C2—C1	121.3 (3)	C22—C21—C20	121.6 (3)
C3—C2—H2	119.3	C22—C21—H21	119.2
C1—C2—H2	119.3	C20—C21—H21	119.2
C2—C3—C4	120.1 (3)	C23—C22—C21	120.9 (3)
C2—C3—H3	120	C23—C22—H22	119.6
C4—C3—H3	120	C21—C22—H22	119.6
C3—C4—C5	120.6 (3)	C22—C23—C24	119.7 (3)
C3—C4—N3	119.6 (3)	C22—C23—N6	120.8 (4)
C5—C4—N3	119.8 (3)	C24—C23—N6	119.5 (4)
C6—C5—C4	119.8 (3)	C23—C24—C25	119.9 (3)
C6—C5—H5	120.1	C23—C24—H24	120
C4—C5—H5	120.1	C25—C24—H24	120
C5—C6—C1	121.2 (3)	C24—C25—C20	121.6 (3)
C5—C6—H6	119.4	C24—C25—H25	119.2
C1—C6—H6	119.4	C20—C25—H25	119.2
N1—C7—C8	113.3 (2)	N4—C26—C27	110.5 (2)
N1—C7—H7A	108.9	N4—C26—H26A	109.5
C8—C7—H7A	108.9	C27—C26—H26A	109.5
N1—C7—H7B	108.9	N4—C26—H26B	109.5
C8—C7—H7B	108.9	C27—C26—H26B	109.5
H7A—C7—H7B	107.7	H26A—C26—H26B	108.1
N2—C8—C7	110.9 (2)	N5—C27—C26	111.0 (2)
N2—C8—H8A	109.5	N5—C27—H27A	109.4
C7—C8—H8A	109.5	C26—C27—H27A	109.4
N2—C8—H8B	109.5	N5—C27—H27B	109.4
C7—C8—H8B	109.5	C26—C27—H27B	109.4
H8A—C8—H8B	108	H27A—C27—H27B	108
N2—C9—C10	111.5 (2)	N5—C28—C29	111.1 (2)
N2—C9—H9A	109.3	N5—C28—H28A	109.4
C10—C9—H9A	109.3	C29—C28—H28A	109.4
N2—C9—H9B	109.3	N5—C28—H28B	109.4
C10—C9—H9B	109.3	C29—C28—H28B	109.4
H9A—C9—H9B	108	H28A—C28—H28B	108
N1—C10—C9	113.7 (2)	N4—C29—C28	111.5 (2)
N1—C10—H10A	108.8	N4—C29—H29A	109.3
C9—C10—H10A	108.8	C28—C29—H29A	109.3
N1—C10—H10B	108.8	N4—C29—H29B	109.3
C9—C10—H10B	108.8	C28—C29—H29B	109.3
H10A—C10—H10B	107.7	H29A—C29—H29B	108

C14—O5—C18	118.3 (2)	C33—O10—C37	118.5 (3)
C12—C11—C16	117.5 (2)	C35—C30—C31	117.1 (3)
C12—C11—C17	120.9 (2)	C35—C30—C36	120.9 (3)
C16—C11—C17	121.6 (3)	C31—C30—C36	122.0 (2)
C11—C12—C13	122.1 (3)	C32—C31—C30	121.5 (3)
C11—C12—H12	119	C32—C31—H31	119.2
C13—C12—H12	119	C30—C31—H31	119.2
C14—C13—C12	119.5 (3)	C31—C32—C33	120.0 (3)
C14—C13—H13	120.3	C31—C32—H32	120
C12—C13—H13	120.3	C33—C32—H32	120
O5—C14—C13	124.4 (3)	O10—C33—C34	124.5 (3)
O5—C14—C15	116.1 (3)	O10—C33—C32	116.1 (3)
C13—C14—C15	119.5 (3)	C34—C33—C32	119.4 (3)
C16—C15—C14	120.2 (3)	C35—C34—C33	119.2 (3)
C16—C15—H15	119.9	C35—C34—H34	120.4
C14—C15—H15	119.9	C33—C34—H34	120.4
C15—C16—C11	121.2 (3)	C30—C35—C34	122.7 (3)
C15—C16—H16	119.4	C30—C35—H35	118.6
C11—C16—H16	119.4	C34—C35—H35	118.6
O4—C17—O3	123.5 (2)	O9—C36—O8	124.0 (3)
O4—C17—C11	119.3 (3)	O9—C36—C30	118.3 (3)
O3—C17—C11	117.3 (2)	O8—C36—C30	117.7 (3)
O5—C18—C19	107.3 (3)	O10—C37—C38	107.4 (3)
O5—C18—H18A	110.3	O10—C37—H37A	110.2
C19—C18—H18A	110.3	C38—C37—H37A	110.2
O5—C18—H18B	110.3	O10—C37—H37B	110.2
C19—C18—H18B	110.3	C38—C37—H37B	110.2
H18A—C18—H18B	108.5	H37A—C37—H37B	108.5
C18—C19—H19A	109.5	C37—C38—H38A	109.5
C18—C19—H19B	109.5	C37—C38—H38B	109.5
H19A—C19—H19B	109.5	H38A—C38—H38B	109.5
C18—C19—H19C	109.5	C37—C38—H38C	109.5
H19A—C19—H19C	109.5	H38A—C38—H38C	109.5
H19B—C19—H19C	109.5	H38B—C38—H38C	109.5

C7—N1—C1—C2	−173.1 (2)	C26—N4—C20—C21	−149.7 (3)
C10—N1—C1—C2	−26.5 (3)	C29—N4—C20—C21	2.0 (4)
C7—N1—C1—C6	8.5 (3)	C26—N4—C20—C25	33.3 (4)
C10—N1—C1—C6	155.0 (2)	C29—N4—C20—C25	−175.1 (3)
N1—C1—C2—C3	−177.9 (2)	N4—C20—C21—C22	−174.3 (3)
C6—C1—C2—C3	0.6 (4)	C25—C20—C21—C22	2.8 (5)
C1—C2—C3—C4	−0.7 (4)	C20—C21—C22—C23	−1.3 (5)
C2—C3—C4—C5	0.3 (4)	C21—C22—C23—C24	−0.7 (5)
C2—C3—C4—N3	−178.7 (3)	C21—C22—C23—N6	177.8 (3)
O2—N3—C4—C3	−170.8 (3)	O7—N6—C23—C22	−179.8 (3)
O1—N3—C4—C3	9.6 (4)	O6—N6—C23—C22	−3.4 (5)
O2—N3—C4—C5	10.2 (4)	O7—N6—C23—C24	−1.3 (5)
O1—N3—C4—C5	−169.4 (3)	O6—N6—C23—C24	175.1 (3)

C3—C4—C5—C6	0.2 (4)	C22—C23—C24—C25	0.9 (5)
N3—C4—C5—C6	179.2 (3)	N6—C23—C24—C25	−177.6 (3)
C4—C5—C6—C1	−0.2 (4)	C23—C24—C25—C20	0.8 (5)
N1—C1—C6—C5	178.3 (2)	N4—C20—C25—C24	174.6 (3)
C2—C1—C6—C5	−0.2 (4)	C21—C20—C25—C24	−2.6 (5)
C1—N1—C7—C8	−173.0 (2)	C20—N4—C26—C27	−148.9 (3)
C10—N1—C7—C8	40.3 (3)	C29—N4—C26—C27	57.0 (3)
C9—N2—C8—C7	62.2 (3)	C28—N5—C27—C26	56.4 (3)
N1—C7—C8—N2	−51.8 (3)	N4—C26—C27—N5	−57.5 (3)
C8—N2—C9—C10	−61.5 (3)	C27—N5—C28—C29	−54.8 (3)
C1—N1—C10—C9	173.4 (2)	C20—N4—C29—C28	150.1 (2)
C7—N1—C10—C9	−39.5 (3)	C26—N4—C29—C28	−55.8 (3)
N2—C9—C10—N1	50.3 (3)	N5—C28—C29—N4	54.6 (3)
C16—C11—C12—C13	0.5 (4)	C35—C30—C31—C32	−1.0 (4)
C17—C11—C12—C13	−177.6 (3)	C36—C30—C31—C32	177.1 (3)
C11—C12—C13—C14	0.4 (5)	C30—C31—C32—C33	1.6 (4)
C18—O5—C14—C13	1.3 (5)	C37—O10—C33—C34	0.7 (4)
C18—O5—C14—C15	−178.7 (3)	C37—O10—C33—C32	−178.7 (3)
C12—C13—C14—O5	178.9 (3)	C31—C32—C33—O10	178.6 (3)
C12—C13—C14—C15	−1.0 (5)	C31—C32—C33—C34	−0.8 (4)
O5—C14—C15—C16	−179.2 (3)	O10—C33—C34—C35	−179.8 (3)
C13—C14—C15—C16	0.7 (5)	C32—C33—C34—C35	−0.5 (4)
C14—C15—C16—C11	0.2 (5)	C31—C30—C35—C34	−0.3 (4)
C12—C11—C16—C15	−0.8 (4)	C36—C30—C35—C34	−178.5 (3)
C17—C11—C16—C15	177.3 (3)	C33—C34—C35—C30	1.1 (4)
C12—C11—C17—O4	2.7 (4)	C35—C30—C36—O9	15.2 (4)
C16—C11—C17—O4	−175.3 (2)	C31—C30—C36—O9	−162.9 (3)
C12—C11—C17—O3	−177.3 (3)	C35—C30—C36—O8	−166.4 (2)
C16—C11—C17—O3	4.6 (4)	C31—C30—C36—O8	15.5 (4)
C14—O5—C18—C19	178.7 (3)	C33—O10—C37—C38	176.2 (3)

Hydrogen-bond geometry (Å, º)

*Cg*2 and *Cg*6 are the centroids of the C1–C6 and C30–C35 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H31*N*···O3^{i}	0.94 (2)	1.68 (2)	2.613 (3)	172 (5)
N2—H31*N*···O4^{i}	0.94 (2)	2.51 (4)	3.157 (3)	127 (4)
N2—H32*N*···O9^{ii}	0.90 (2)	1.96 (2)	2.843 (3)	171 (5)
N5—H61*N*···O8^{i}	0.91 (2)	1.78 (2)	2.686 (3)	175 (5)
N5—H61*N*···O9^{i}	0.91 (2)	2.59 (4)	3.174 (3)	122 (4)
N5—H62*N*···O4^{iii}	0.90 (2)	1.83 (2)	2.708 (3)	165 (5)
C22—H22···O2^{iv}	0.93	2.6	3.502 (5)	165
C27—H27*B*···O9^{i}	0.97	2.59	3.215 (3)	123
C28—H28*B*···O7^{v}	0.97	2.65	3.410 (4)	135
C29—H29*B*···O1^{i}	0.97	2.53	3.249 (4)	131
C35—H35···O4^{iii}	0.93	2.52	3.263 (3)	137

C10—H10*A*···*Cg*6	0.97	2.82	3.746 (3)	159
C29—H29*A*···*Cg*2	0.97	2.76	3.556 (3)	139

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*+1, *y*−1, *z*; (iii) *x*, *y*+1, *z*; (iv) −*x*, −*y*+1, −*z*+1; (v) −*x*, −*y*+2, −*z*+1.