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Syntheses and crystal structures of three salts of 1-(4-nitrophenyl)piperazine

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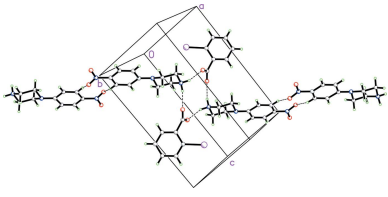
The crystal structures and Hirshfeld surface analyses of three salts of 1-(4-nitrophenyl)piperazine with 2-chlorobenzoic acid, 2-bromobenzoic acid and 2-iodobenzoic acid are reported. The chlorobenzoate salt, $C_{10}H_{14}N_3O_2^+\cdot C_7H_4ClO_2^-$, contains whole-ion-disordered cations and anions, which were modeled with two equivalent conformations with occupancies of 0.745 (10)/0.255 (10) and 0.563 (13)/0.437 (13), respectively. The bromobenzoate and iodobenzoate derivatives are isomorphous and crystallize as hemihydrates, *viz*. $C_{10}H_{14}N_3O_2^+\cdot C_7H_4BrO_2^-\cdot 0.5H_2O$ and $C_{10}H_{14}N_3O_2^+\cdot C_7H_4IO_2^-\cdot 0.5H_2O$, respectively [the water molecule is disordered over two locations with occupancies of 0.276 (3)/0.223 (3) for the iodobenzoate derivative]. In the extended structures, all three salts feature an $R_4^4(12)$ loop of two anions and two cations linked by N—H···O hydrogen bonds.

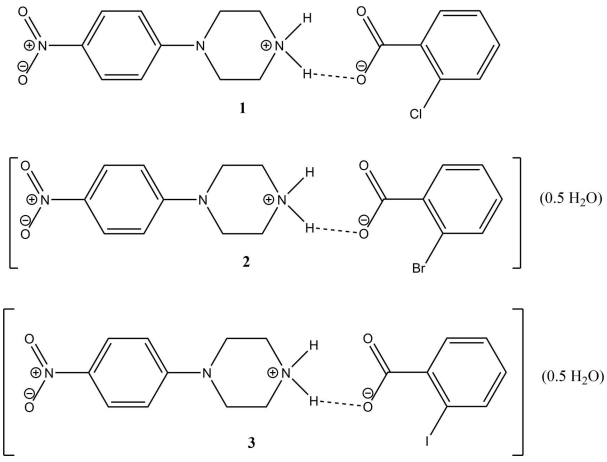
1. Chemical context

Piperazines and substituted piperazines are pharmacophores that can be found in many biologically active compounds across a number of different therapeutic areas (Berkheij, 2005) such as antifungal (Upadhayaya *et al.*, 2004), anti-bacterial, anti-malarial and anti-psychotic agents (Chaudhary *et al.*, 2006). A review on the current pharmacological and toxicological information for piperazine derivatives was described (Elliott, 2011). 4-(4-Nitrophenyl)piperazin-1-ium 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). 4-Nitrophenylpiperazine was the starting material in the synthesis and biological evaluation of piperazine containing hydrazone derivatives (Kaya *et al.*, 2016).

Very recently, we have reported the syntheses, crystal structures and Hirshfeld surface analysis of 4-(4-nitrophenyl)piperazin-1-ium trifluoroacetate (Cambridge Structural Database refcode BEYREG) and 4-(4-nitrophenyl)piperazin-1-ium trichloroacetate (BEYRIK) (Shankara Prasad *et al.*, 2023). As part of our ongoing studies in this area, the present paper reports the crystal structure studies and Hirshfeld surface analysis of three salts of 1-(4-nitrophenyl)piperazine with organic acids *viz*., 4-(4-nitrophenyl)piperazin-1-ium 2-chlorobenzoate, $C_{10}H_{14}N_3O_2^+\cdot C_7H_4ClO_2^-$ (**1**), 4-(4-nitro-



phenyl)piperazin-1-ium 2-bromobenzoate hemihydrate, $C_{10}H_{14}N_3O_2^+\cdot C_7H_4BrO_2^-\cdot 0.5H_2O$ (**2**), and 4-(4-nitrophenyl)-piperazin-1-ium 2-iodobenzoate hemihydrate, $C_{10}H_{14}N_3O_2^+\cdot C_7H_4IO_2^-\cdot 0.5H_2O$ (**3**).



2. Structural commentary

Structure **1** consists of a 4-nitropiperazinium cation linked to a 2-chlorobenzoate anion by two N—H···O hydrogen bonds (Fig. 1, Table 1), which will be discussed in further detail in the *Supramolecular features* section of the paper. Both the cation and the anion exhibit whole-ion disorder, which was modeled with two equivalent conformations with occupancies of 0.745 (10)/0.255 (10) and 0.563 (13)/0.437 (13) respectively. When discussing the conformations of the anion and cation, only the major components will be used. In the chlorobenzoate anion, the carboxylate group is significantly twisted with respect to the 2-chlorophenyl ring with a dihedral angle of 76.7 (4)°, which is likely due to the steric interaction between the *ortho*-chloro substituent and the carboxylate group. Structures **2** and **3** exhibit similar cation conformations, with equivalent dihedral angles of 65.5 (3) and 67.1 (5)°, respectively. Additionally, in all three structures, the 4-nitrophenyl group occupies an equatorial position in its attachment to the piperazinium ring.

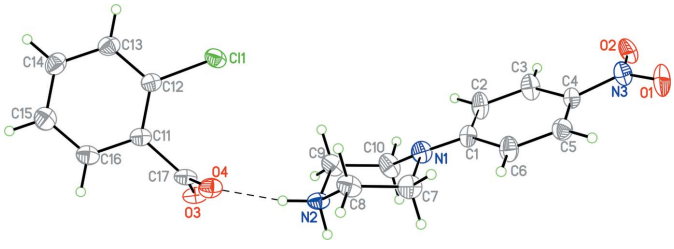


Figure 1
The molecular structure of **1** with the N—H···O hydrogen bond shown as a dashed line. Atomic displacement parameters are at the 30% probability level.

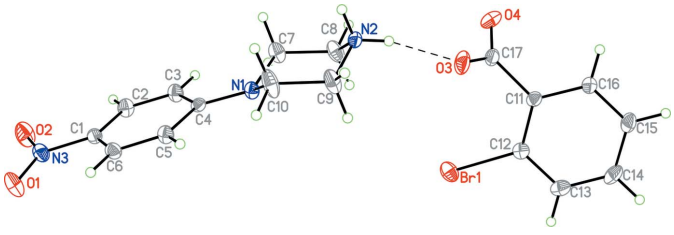


Figure 2
The molecular structure of **2** with the N—H···O hydrogen bond shown as a dashed line. Atomic displacement parameters are at the 30% probability level.

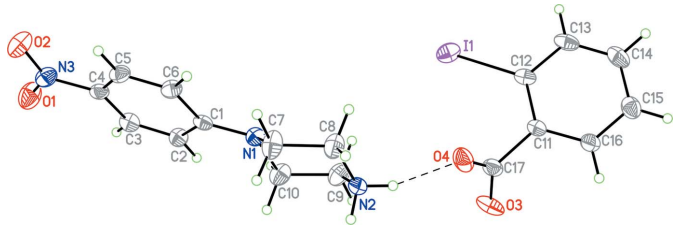


Figure 3
The molecular structure of **3** with the N—H···O hydrogen bond shown as a dashed line. Atomic displacement parameters are at the 30% probability level.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*A*···O2^{i}	0.93	2.13	2.880 (15)	137
N2—H2*B*···O3^{ii}	0.88 (2)	1.86 (2)	2.740 (6)	172 (3)
N2—H2*B*···O3*A*^{ii}	0.88 (2)	1.73 (2)	2.590 (13)	164 (3)
N2—H2*C*···O4	0.89 (2)	1.83 (2)	2.705 (8)	169 (3)
N2—H2*C*···O4*A*	0.89 (2)	1.81 (3)	2.644 (19)	156 (3)
C8—H8*A*···Cl1^{iii}	0.97	2.82	3.629 (5)	142
C8—H8*A*···Cl1^{iv}	0.97	2.95	3.780 (5)	144
C8—H8*A*···Cl1*A*^{iv}	0.97	2.88	3.643 (12)	136
C8—H8*B*···O4*A*^{iv}	0.97	2.58	3.13 (2)	116
C10—H10*A*···O3*A*^{ii}	0.97	2.65	3.285 (19)	123

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O3	0.85 (2)	1.83 (2)	2.655 (3)	164 (2)
N2—H2*B*···O4^{i}	0.88 (2)	1.82 (2)	2.701 (3)	173 (2)
C2—H2···O2^{ii}	0.93	2.50	3.307 (4)	145
C7—H7*B*···Br1^{iii}	0.97	3.11	4.032 (2)	160
C10—H10*B*···O1*W*^{i}	0.97	2.10	3.057 (8)	169

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···I1^{i}	0.93	3.28	4.110 (4)	150
C3—H3···O1^{ii}	0.93	2.53	3.347 (7)	147
C6—H6···I1^{iii}	0.93	3.26	3.940 (4)	132
C7—H7*A*···O1*WA*	0.97	2.14	3.09 (3)	167
C7—H7*A*···O1*WB*	0.97	2.01	2.85 (3)	145
N2—H2*A*···O3^{iv}	0.88 (5)	1.86 (5)	2.717 (5)	164 (5)
N2—H2*B*···O4	0.93 (5)	1.77 (5)	2.666 (6)	160 (5)
O1*WB*—H1*W*3···O3^{iv}	0.83 (2)	1.71 (10)	2.37 (2)	135 (12)

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

Since **2** and **3** are isostructural, only **2** will be discussed in detail. This structure consists of a 4-(4-nitrophenyl)piperazin-1-ium cation linked to a 2-bromobenzoate anion by two N—H···O hydrogen bonds (Figs. 2 and 3, Tables 2 and 3). Both **2** and **3** contain 0.5 water molecules of crystallization [disordered over two locations with occupancies of 0.276 (3)/0.223 (3) for the iodobenzoate derivative]. Additionally, there is a weak C—H···Br interaction accepted by the bromine atom in the 2-bromobezoate anion and a carbon atom in the piperazinium ring, as well as a pair of weak C—H···O interactions between adjacent 4-nitrophenyl rings in the 4-(4-nitrophenyl)piperazin-1-ium cation.

3. Supramolecular features

In the packing of **1**, which contains both a disordered cation and anion as well as disordered water of solvation, the discussion will focus solely on the major component. The cation forms an $R_4^4(12)$ loop involving N—H···O hydrogen bonds with two adjacent anions and an adjacent cation (symmetry codes: $2-x, 1-y, 1-z$; $x, 1+y, z$; $1-x, 1-y, 1-z$; see Fig. 4 for packing and Fig. 5 for fingerprint plots). There is also a π–π interaction between the nitro group in the cation and the phenyl ring of an adjacent cation [symmetry code: $1-x, -y, -z$; $Y\cdots Cg$ distance = 3.488 (18) Å; X—$Y\cdots Cg$: 85.8 (12)°].

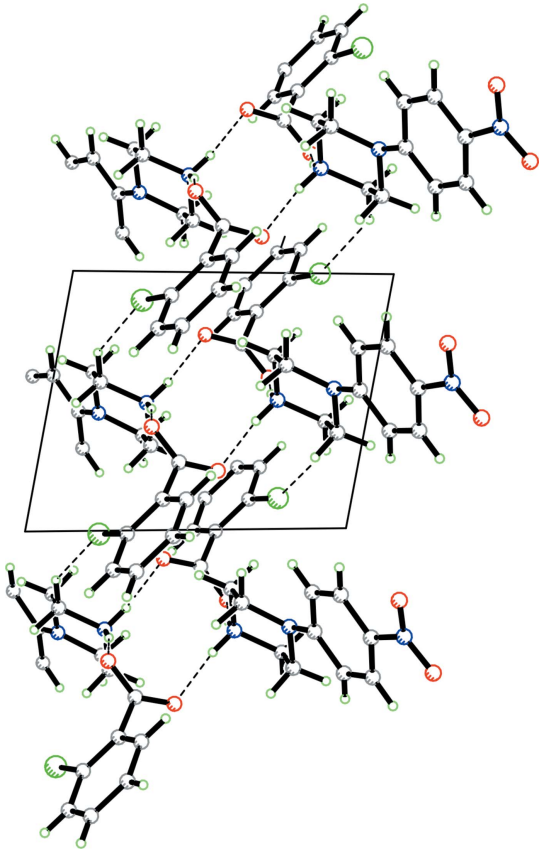


Figure 4
Packing diagram for **1** showing an $R_4^4(12)$ loop of N—H···O hydrogen bonds with two cations and two anions (symmetry codes: $2-x, 1-y, 1-z$; $x, 1+y, z$; $1-x, 1-y, 1-z$). Hydrogen bonds shown as dashed lines.

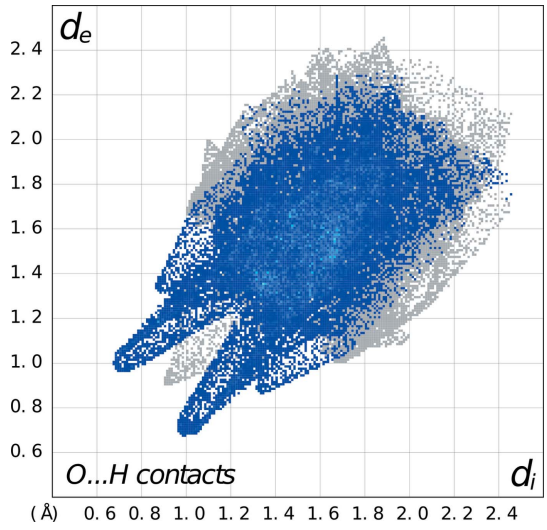


Figure 5
Fingerprint plot for **1** showing the N—H···O hydrogen bonds as prominent spikes.

In the packing of **2**, two cations and two anions form an $R_4^4(12)$ loop (Etter *et al.*, 1990) of N—H···O hydrogen bonds (symmetry code: $1-x, -y, -z$; see Fig. 6 for packing and Fig. 7 for fingerprint plots). Additionally, there are weak C—H···O interactions between adjacent nitrophenyl rings (symmetry code: $-x, 1-y, 1-z$) that form an $R_2^2(10)$ ring (Fig. 6), as well as a weak C—H···Br interaction between the piperazine ring and the bromine atom in an adjacent 2-bromobenzoate anion (symmetry code: $-1+x, y, z$). The phenyl rings in adjacent cations form π–π interactions with a perpendicular distance between centroids of 3.5332 (11) Å (symmetry code: $1-x, 1-y, 1-z$; slippage = 0.737 Å). These are all clearly seen in the fingerprint plot generated by *CrystalExplorer* (Spackman *et al.*, 2021).

In the packing of **3**, a pair of cations and a pair of anions form an $R_4^4(12)$ loop linked by N—H···O hydrogen bonds

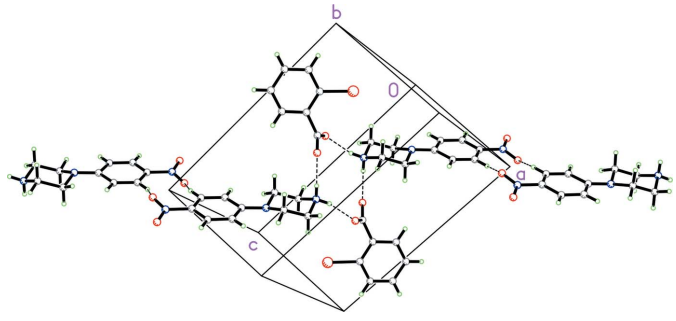


Figure 6
Packing diagram for **2** showing an $R_4^4(12)$ loop arising from N—H···O hydrogen bonds with an adjacent cation and anion (symmetry code: $1-x, -y, -z$) and an $R_2^2(10)$ loop comprised of weak C—H···O interactions between adjacent nitrophenyl rings (symmetry code: $-x, 1-y, 1-z$). Hydrogen bonds and C—H···O interactions shown as dashed lines. The half occupancy water molecule is omitted for clarity.

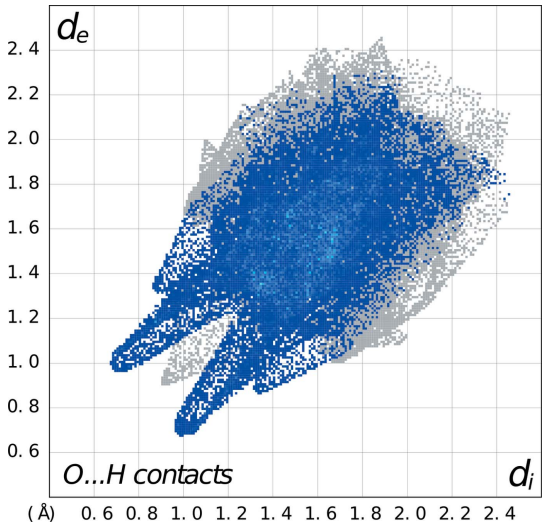


Figure 7
Fingerprint plot for **2** showing the N—H···O hydrogen bonds as prominent spikes.

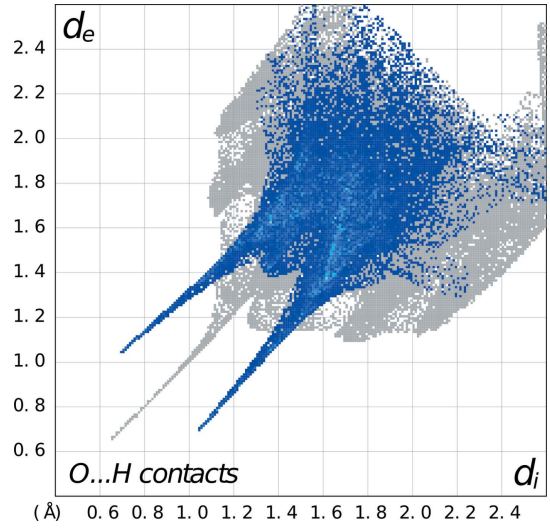


Figure 9
Fingerprint plot for **3** showing the N—H···O hydrogen bonding as prominent spikes.

(symmetry code: $1-x, 1-y, 1-z$; see Fig. 8 for packing and Fig. 9 for fingerprint plots). Additionally, there are weak C—H···O interactions between adjacent nitrophenyl rings (symmetry code: $1-x, 1-y, -z$) that form an $R_2^2(10)$ ring. This structure contains a partially occupied water molecule close to a center of inversion for which the hydrogen atoms were not able to be located (see *Refinement*). This species is likely to be involved in hydrogen bonding with an adjacent oxygen atom in the anion (symmetry code: $1-x, 1-y, 1-z$) and with the piperazine ring in the cation, forming an $R_3^3(10)$ ring. The phenyl rings in adjacent cations form π–π interactions with a perpendicular distance between centroids of 3.586 (4) Å (symmetry code: $-x$, $1-y$, $-z$; slippage = 0.379 Å).

4. Database survey

Related structures containing the 4-(4-nitrophenyl)piperazin-1-ium cation include 4-(4-nitrophenyl)piperazin-1-ium chloride monohydrate (refcode LIJNAU; Lu, 2007) and 4,6-dimethoxy pyrimidin-2-amine-1-(4-nitrophenyl)piperazine (1:1) (LUDMUU; Wang *et al.*, 2014). Very recently, we have reported the crystal structures of six salts of 1-(4-nitrophenyl)piperazine (NEBVOJ, NEBVUP, NEBWAW, NEBWEA, NEBWIE, NEBWOK; Mahesha *et al.*, 2022*a*). The syntheses and crystal structures of 4-(4-nitrophenyl)piperazin-1-ium benzoate monohydrate (BEFGIG) and 4-(4-nitrophenyl)piperazin-1-ium 2-carboxy-4,6-dinitrophenolate (BEFGOM) have been reported (Shankara Prasad *et al.*, 2022). A survey of these published derivatives containing the 4-(4-nitrophenyl)piperazin-1-ium cation shows that the most common conformation adopted by the 4-nitrophenyl substituent with respect to the six-membered piperazinium ring is equatorial (LUDMUU, NEBVOJ, NEBVUP, NEBWAW, NEBWEA, NEBWOK, BEFGIG and BEYRIK) with only three adopting the axial conformation (LUDMUU, BEFGOM, and BEYREG). One published structure contains two 4-nitrophenyl cations, with one adopting an equatorial conformation and the other an axial conformation (Mahesha *et al.*, 2022*a*).

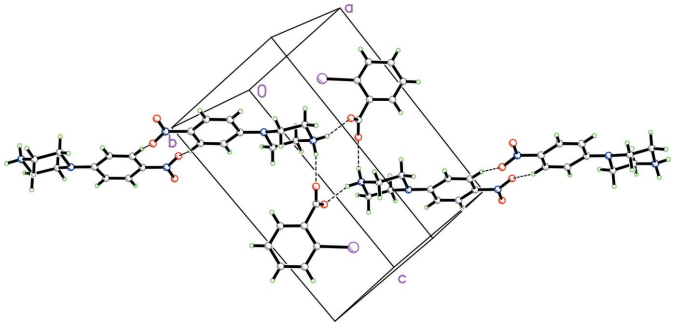


Figure 8
Packing diagram for **3** showing the same features as Fig. 6.

5. Synthesis and crystallization

For the synthesis of salts (**1**)–(**3**), a solution of commercially available (from Sigma-Aldrich) 1-(4-nitrophenyl)piperazine (100 mg, 0.483 mmol) in methanol (10 ml) was mixed with equimolar solutions of the appropriate acids in methanol (10 ml) and ethyl acetate (10 ml), *viz*., 2-chlorobenzoic acid (76 mg, 0.483 mmol) for (**1**), 2-bromobenzoic acid (97 mg, 0.483 mmol) for (**2**), and 2-iodobenzoic acid (120 mg, 0.483 mmol) for (**3**), The resulting solutions were stirred for 15 minutes at room temperature and allowed to stand at the same temperature. X-ray quality crystals were formed on slow evaporation after one week for all compounds, where ethanol:ethylacetate (1:1) was used for crystallization. The melting points are 439–441 K (**1**), 443–445 K (**2**) and 451–453 K (**3**).

Table 4
Experimental details.

	1	**2**	**3**
Crystal data			
Chemical formula	$C_{10}H_{14}N_3O_2^+\cdot C_7H_4ClO_2^-$	$C_{10}H_{14}N_3O_2^+\cdot C_7H_4BrO_2^-\cdot 0.5H_2O$	$C_{10}H_{14}N_3O_2^+\cdot C_7H_4IO_2^-\cdot 0.5H_2O$
M_r	363.79	417.26	928.50
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)	6.6073 (5), 8.2708 (5), 16.984 (1)	7.2570 (5), 9.7772 (6), 14.202 (1)	7.3949 (6), 9.3440 (8), 14.498 (1)
α, β, γ (°)	102.385 (6), 91.745 (6), 99.903 (6)	102.101 (6), 99.534 (6), 110.981 (6)	104.967 (8), 94.707 (7), 107.430 (8)
V (Å^3)	890.84 (10)	887.41 (11)	909.44 (13)
Z	2	2	1
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.24	2.35	1.79
Crystal size (mm)	0.48 × 0.44 × 0.24	0.36 × 0.32 × 0.20	0.50 × 0.44 × 0.24
Data collection			
Diffractometer	Oxford Diffraction Xcalibur CCD	Oxford Diffraction Xcalibur CCD	Oxford Diffraction Xcalibur CCD
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}	0.894, 1.000	0.781, 1.000	0.697, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6637, 3787, 1916	6177, 3856, 2478	6275, 3904, 2443
R_{int}	0.014	0.016	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.653	0.660	0.661
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.065, 0.162, 1.06	0.036, 0.084, 0.94	0.046, 0.117, 1.02
No. of reflections	3787	3856	3904
No. of parameters	357	244	264
No. of restraints	714	5	13
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})	0.15, −0.15	0.51, −0.33	0.61, −0.44

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2009), *SHELXL2018* (Sheldrick, 2015*b*), *SHELXL2018/3* (Sheldrick, 2015*a*) and *OLEX2* (Dolomanov *et al.*, 2009).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. For all structures, the hydrogen atoms were located in difference maps and relocated to idealized locations (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) while the N—H hydrogen atoms were refined isotropically. For **1**, in which both the cation and the anion exhibit whole-ion disorder, two equivalent conformations were modeled with occupancies of 0.745 (10)/ 0.255 (10) and 0.563 (13)/ 0.437 (13) respectively. The water hydrogen atoms were refined isotropically with idealized geometries.

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Syntheses and crystal structures of three salts of 1-(4-nitrophenyl)piperazine

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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: *SHELXL2018* (Sheldrick, 2015b); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015a); molecular graphics: Olex2 1.5 (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 1.5 (Dolomanov *et al.*, 2009).

4-(4-Nitrophenyl)piperazin-1-ium 2-chlorobenzoate (1)

Crystal data

$C_{10}H_{14}N_3O_2^+ \cdot C_7H_4ClO_2^-$
M_r = 363.79
Triclinic, $P\overline{1}$
a = 6.6073 (5) Å
b = 8.2708 (5) Å
c = 16.984 (1) Å
α = 102.385 (6)°
β = 91.745 (6)°
γ = 99.903 (6)°
V = 890.84 (10) Å^3
Z = 2
$F(000)$ = 380
D_x = 1.356 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2365 reflections
θ = 2.6–27.6°
μ = 0.24 mm^{-1}
T = 293 K
Prism, yellow
0.48 × 0.44 × 0.24 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
ω scans
Absorption correction: multi-scan (CrysalisRed; Oxford Diffraction, 2009)
T_{min} = 0.894, T_{max} = 1.000
6637 measured reflections
3787 independent reflections
1916 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 27.7°, θ_{min} = 2.6°
h = −8→8
k = −10→7
l = −20→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.162
S = 1.06
3787 reflections
357 parameters
714 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.3137P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ 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}	Occ. (<1)
Cl1	1.0680 (5)	0.6989 (5)	0.5627 (2)	0.0833 (8)	0.745 (10)
O3	0.7965 (9)	1.0649 (9)	0.6034 (5)	0.0842 (18)	0.745 (10)
O4	0.6067 (11)	0.8100 (10)	0.5952 (5)	0.082 (2)	0.745 (10)
C11	0.9076 (7)	0.8971 (6)	0.6843 (2)	0.0615 (11)	0.745 (10)
C12	1.0545 (7)	0.7974 (6)	0.6616 (3)	0.0650 (12)	0.745 (10)
C13	1.1959 (7)	0.7776 (5)	0.7195 (3)	0.0835 (16)	0.745 (10)
H13	1.294232	0.710907	0.704298	0.100*	0.745 (10)
C14	1.1905 (8)	0.8575 (5)	0.8000 (3)	0.0943 (18)	0.745 (10)
H14	1.285124	0.844228	0.838711	0.113*	0.745 (10)
C15	1.0436 (9)	0.9572 (6)	0.8227 (2)	0.0931 (16)	0.745 (10)
H15	1.039942	1.010634	0.876538	0.112*	0.745 (10)
C16	0.9022 (8)	0.9770 (6)	0.7648 (3)	0.0776 (13)	0.745 (10)
H16	0.803866	1.043720	0.779953	0.093*	0.745 (10)
C17	0.7612 (19)	0.9300 (14)	0.6263 (9)	0.0666 (17)	0.745 (10)
Cl1A	1.0723 (17)	0.6949 (14)	0.5994 (9)	0.100 (3)	0.255 (10)
O3A	0.741 (3)	1.065 (3)	0.6227 (13)	0.082 (4)	0.255 (10)
O4A	0.654 (3)	0.785 (3)	0.5841 (13)	0.073 (4)	0.255 (10)
C11A	0.877 (2)	0.906 (2)	0.7016 (7)	0.066 (2)	0.255 (10)
C12A	1.030 (2)	0.8078 (18)	0.6923 (8)	0.069 (2)	0.255 (10)
C13A	1.1458 (19)	0.7951 (16)	0.7596 (10)	0.083 (2)	0.255 (10)
H13A	1.247604	0.729405	0.753378	0.099*	0.255 (10)
C14A	1.110 (2)	0.8807 (17)	0.8362 (8)	0.092 (3)	0.255 (10)
H14A	1.187433	0.872219	0.881231	0.111*	0.255 (10)
C15A	0.958 (2)	0.9790 (17)	0.8455 (7)	0.090 (3)	0.255 (10)
H15A	0.933424	1.036208	0.896755	0.108*	0.255 (10)
C16A	0.841 (2)	0.9916 (19)	0.7782 (8)	0.081 (2)	0.255 (10)
H16A	0.739583	1.057381	0.784426	0.097*	0.255 (10)
C17A	0.735 (7)	0.904 (5)	0.617 (3)	0.067 (3)	0.255 (10)
O1A	0.437 (2)	0.1420 (17)	−0.0678 (8)	0.143 (4)	0.437 (13)
O2A	0.725 (2)	0.2969 (18)	−0.0749 (8)	0.127 (3)	0.437 (13)
N3A	0.575 (2)	0.2519 (16)	−0.0385 (7)	0.105 (2)	0.437 (13)
C1A	0.548 (2)	0.5327 (19)	0.1991 (6)	0.071 (2)	0.437 (13)
C2A	0.713 (2)	0.5730 (16)	0.1536 (7)	0.085 (3)	0.437 (13)
H2	0.820154	0.661738	0.175667	0.102*	0.437 (13)
C3A	0.7186 (17)	0.4807 (15)	0.0753 (7)	0.089 (3)	0.437 (13)
H3	0.829021	0.507621	0.044865	0.107*	0.437 (13)

C4A	0.5589 (19)	0.3480 (14)	0.0424 (5)	0.086 (2)	0.437 (13)
C5A	0.3939 (18)	0.3077 (15)	0.0878 (7)	0.093 (2)	0.437 (13)
H5	0.287089	0.218996	0.065773	0.112*	0.437 (13)
C6A	0.389 (2)	0.4001 (18)	0.1662 (7)	0.089 (2)	0.437 (13)
H6	0.278218	0.373111	0.196576	0.107*	0.437 (13)
O1	0.5473 (18)	0.1306 (13)	−0.0808 (7)	0.144 (3)	0.563 (13)
O2	0.836 (2)	0.2893 (15)	−0.0755 (6)	0.161 (4)	0.563 (13)
N3	0.675 (2)	0.2455 (14)	−0.0465 (6)	0.109 (3)	0.563 (13)
C1	0.5784 (18)	0.5063 (14)	0.1894 (4)	0.070 (2)	0.563 (13)
C2	0.7641 (16)	0.5504 (13)	0.1562 (5)	0.089 (3)	0.563 (13)
H2A	0.866018	0.635336	0.185813	0.107*	0.563 (13)
C3	0.7975 (15)	0.4676 (12)	0.0787 (5)	0.095 (2)	0.563 (13)
H3A	0.921692	0.497043	0.056517	0.114*	0.563 (13)
C4	0.6452 (15)	0.3406 (11)	0.0345 (4)	0.086 (2)	0.563 (13)
C5	0.4595 (14)	0.2966 (11)	0.0676 (5)	0.087 (2)	0.563 (13)
H5A	0.357591	0.211638	0.038029	0.105*	0.563 (13)
C6	0.4261 (15)	0.3794 (13)	0.1451 (5)	0.0822 (19)	0.563 (13)
H6A	0.301914	0.349929	0.167326	0.099*	0.563 (13)
N1	0.5532 (4)	0.5957 (3)	0.27470 (16)	0.0768 (8)	
N2	0.5064 (4)	0.7717 (3)	0.43581 (17)	0.0675 (7)	
H2B	0.417 (4)	0.834 (3)	0.4252 (17)	0.081*	
H2C	0.530 (5)	0.795 (4)	0.4892 (11)	0.081*	
C7	0.3683 (5)	0.5526 (4)	0.3162 (2)	0.0921 (11)	
H7A	0.313237	0.433276	0.297924	0.110*	
H7B	0.264769	0.614262	0.302794	0.110*	
C8	0.4136 (5)	0.5935 (3)	0.4054 (2)	0.0858 (10)	
H8A	0.286923	0.568239	0.431384	0.103*	
H8B	0.506956	0.523703	0.419185	0.103*	
C9	0.6955 (5)	0.8126 (4)	0.3947 (2)	0.0899 (11)	
H9A	0.796744	0.749561	0.408843	0.108*	
H9B	0.752465	0.931538	0.412976	0.108*	
C10	0.6532 (6)	0.7714 (4)	0.3053 (2)	0.0929 (11)	
H10A	0.565368	0.844595	0.290746	0.111*	
H10B	0.781839	0.792344	0.279945	0.111*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0608 (8)	0.0760 (9)	0.0980 (18)	0.0096 (6)	0.0027 (12)	−0.0109 (13)
O3	0.056 (3)	0.069 (2)	0.139 (4)	0.019 (2)	0.014 (3)	0.041 (3)
O4	0.048 (3)	0.077 (3)	0.115 (4)	0.000 (2)	0.000 (3)	0.018 (2)
C11	0.053 (2)	0.0330 (18)	0.092 (3)	−0.0025 (16)	0.002 (2)	0.0088 (19)
C12	0.058 (2)	0.0400 (19)	0.088 (3)	0.0002 (16)	−0.009 (2)	0.003 (2)
C13	0.087 (3)	0.050 (2)	0.108 (4)	0.015 (2)	−0.016 (3)	0.007 (3)
C14	0.110 (4)	0.069 (3)	0.096 (4)	0.012 (3)	−0.029 (3)	0.009 (3)
C15	0.107 (4)	0.064 (3)	0.097 (3)	0.002 (3)	−0.004 (3)	0.004 (2)
C16	0.077 (3)	0.049 (2)	0.101 (3)	0.007 (2)	0.009 (2)	0.009 (2)
C17	0.047 (4)	0.048 (4)	0.101 (4)	0.007 (3)	0.010 (3)	0.009 (3)

Cl1A	0.071 (3)	0.079 (3)	0.140 (6)	0.017 (2)	0.020 (5)	−0.004 (5)
O3A	0.064 (8)	0.063 (5)	0.128 (9)	0.024 (6)	0.017 (7)	0.030 (6)
O4A	0.048 (8)	0.062 (6)	0.104 (7)	0.005 (6)	0.004 (6)	0.014 (5)
C11A	0.057 (4)	0.038 (4)	0.099 (4)	0.005 (3)	0.001 (4)	0.009 (4)
C12A	0.063 (4)	0.039 (4)	0.098 (4)	0.004 (3)	−0.007 (4)	0.009 (4)
C13A	0.085 (4)	0.054 (4)	0.103 (5)	0.011 (4)	−0.015 (4)	0.010 (4)
C14A	0.096 (5)	0.068 (5)	0.105 (5)	0.009 (5)	−0.010 (5)	0.009 (5)
C15A	0.089 (6)	0.066 (5)	0.106 (5)	0.001 (4)	0.000 (5)	0.011 (5)
C16A	0.080 (5)	0.054 (4)	0.101 (4)	0.001 (4)	0.002 (4)	0.008 (4)
C17A	0.051 (5)	0.047 (5)	0.102 (5)	0.011 (4)	0.006 (4)	0.015 (5)
O1A	0.145 (8)	0.134 (6)	0.107 (6)	−0.007 (6)	−0.031 (6)	−0.035 (5)
O2A	0.135 (7)	0.122 (6)	0.103 (6)	0.019 (6)	0.005 (6)	−0.019 (5)
N3A	0.117 (5)	0.090 (4)	0.089 (4)	0.007 (5)	−0.014 (4)	−0.007 (3)
C1A	0.090 (5)	0.033 (4)	0.080 (4)	0.002 (4)	−0.014 (3)	−0.001 (3)
C2A	0.095 (5)	0.053 (4)	0.091 (4)	−0.005 (4)	−0.014 (4)	−0.001 (3)
C3A	0.094 (5)	0.072 (4)	0.087 (4)	−0.001 (4)	−0.010 (4)	0.004 (3)
C4A	0.104 (5)	0.068 (3)	0.074 (3)	0.001 (4)	−0.018 (4)	−0.001 (3)
C5A	0.112 (5)	0.071 (4)	0.077 (5)	−0.009 (4)	−0.017 (4)	0.000 (4)
C6A	0.107 (5)	0.063 (4)	0.079 (5)	−0.006 (4)	−0.017 (4)	−0.001 (4)
O1	0.146 (7)	0.123 (4)	0.116 (5)	−0.016 (5)	−0.006 (5)	−0.043 (4)
O2	0.144 (7)	0.150 (5)	0.140 (5)	−0.011 (6)	0.036 (5)	−0.051 (4)
N3	0.125 (6)	0.090 (4)	0.088 (4)	−0.008 (5)	−0.003 (4)	−0.008 (3)
C1	0.090 (4)	0.033 (3)	0.082 (3)	0.007 (3)	−0.017 (3)	0.006 (3)
C2	0.099 (5)	0.059 (4)	0.092 (3)	−0.004 (4)	−0.003 (3)	−0.004 (3)
C3	0.103 (5)	0.074 (3)	0.090 (3)	−0.005 (4)	−0.005 (4)	−0.002 (3)
C4	0.105 (5)	0.068 (3)	0.076 (3)	0.004 (4)	−0.012 (3)	0.004 (2)
C5	0.108 (5)	0.068 (3)	0.069 (4)	−0.010 (3)	−0.015 (3)	0.002 (3)
C6	0.101 (4)	0.058 (3)	0.070 (4)	−0.011 (3)	−0.012 (3)	−0.001 (3)
N1	0.0767 (17)	0.0420 (12)	0.099 (2)	−0.0114 (11)	−0.0042 (15)	0.0076 (13)
N2	0.0543 (15)	0.0425 (12)	0.103 (2)	0.0079 (10)	0.0018 (15)	0.0101 (14)
C7	0.079 (2)	0.0508 (17)	0.127 (3)	−0.0174 (16)	−0.006 (2)	0.0043 (19)
C8	0.086 (2)	0.0425 (16)	0.121 (3)	−0.0017 (15)	0.014 (2)	0.0099 (17)
C9	0.061 (2)	0.074 (2)	0.111 (3)	−0.0155 (16)	0.0106 (19)	−0.0102 (19)
C10	0.096 (3)	0.0543 (18)	0.105 (3)	−0.0236 (17)	0.011 (2)	−0.0043 (18)

Geometric parameters (Å, º)

Cl1—C12	1.715 (3)	C3A—C4A	1.3900
Cl1—H8A^{i}	2.953 (5)	C3A—H3	0.9300
O3—C17	1.247 (14)	C4A—C5A	1.3900
O4—C17	1.304 (10)	C5A—C6A	1.3900
C11—C12	1.3900	C5A—H5	0.9300
C11—C16	1.3900	C6A—H6	0.9300
C11—C17	1.458 (15)	O1—N3	1.189 (9)
C12—C13	1.3900	O2—N3	1.216 (10)
C13—C14	1.3900	N3—C4	1.468 (8)
C13—H13	0.9300	C1—C2	1.3900
C14—C15	1.3900	C1—C6	1.3900

C14—H14	0.9300	C1—N1	1.507 (7)
C15—C16	1.3900	C2—C3	1.3900
C15—H15	0.9300	C2—H2A	0.9300
C16—H16	0.9300	C3—C4	1.3900
Cl1A—C12A	1.711 (4)	C3—H3A	0.9300
O3A—C17A	1.31 (4)	C4—C5	1.3900
O4A—C17A	1.06 (4)	C5—C6	1.3900
C11A—C12A	1.3900	C5—H5A	0.9300
C11A—C16A	1.3900	C6—H6A	0.9300
C11A—C17A	1.68 (4)	N1—C7	1.459 (4)
C12A—C13A	1.3900	N1—C10	1.464 (3)
C13A—C14A	1.3900	N2—C8	1.469 (3)
C13A—H13A	0.9300	N2—C9	1.474 (4)
C14A—C15A	1.3900	N2—H2B	0.882 (17)
C14A—H14A	0.9300	N2—H2C	0.889 (17)
C15A—C16A	1.3900	C7—C8	1.488 (5)
C15A—H15A	0.9300	C7—H7A	0.9700
C16A—H16A	0.9300	C7—H7B	0.9700
O1A—N3A	1.182 (10)	C8—H8A	0.9700
O2A—N3A	1.235 (12)	C8—H8B	0.9700
N3A—C4A	1.448 (9)	C9—C10	1.490 (5)
C1A—N1	1.275 (9)	C9—H9A	0.9700
C1A—C2A	1.3900	C9—H9B	0.9700
C1A—C6A	1.3900	C10—H10A	0.9700
C2A—C3A	1.3900	C10—H10B	0.9700
C2A—H2	0.9300		

C12—Cl1—H8A^{i}	92.6 (2)	C4A—C5A—H5	120.0
C12—C11—C16	120.0	C5A—C6A—C1A	120.0
C12—C11—C17	122.9 (6)	C5A—C6A—H6	120.0
C16—C11—C17	117.0 (6)	C1A—C6A—H6	120.0
C11—C12—C13	120.0	O1—N3—O2	122.6 (9)
C11—C12—Cl1	121.34 (18)	O1—N3—C4	120.6 (8)
C13—C12—Cl1	118.66 (18)	O2—N3—C4	116.9 (7)
C14—C13—C12	120.0	C2—C1—C6	120.0
C14—C13—H13	120.0	C2—C1—N1	117.8 (6)
C12—C13—H13	120.0	C6—C1—N1	122.1 (6)
C13—C14—C15	120.0	C1—C2—C3	120.0
C13—C14—H14	120.0	C1—C2—H2A	120.0
C15—C14—H14	120.0	C3—C2—H2A	120.0
C14—C15—C16	120.0	C4—C3—C2	120.0
C14—C15—H15	120.0	C4—C3—H3A	120.0
C16—C15—H15	120.0	C2—C3—H3A	120.0
C15—C16—C11	120.0	C3—C4—C5	120.0
C15—C16—H16	120.0	C3—C4—N3	122.1 (5)
C11—C16—H16	120.0	C5—C4—N3	117.9 (5)
O3—C17—O4	122.2 (12)	C4—C5—C6	120.0
O3—C17—C11	119.3 (7)	C4—C5—H5A	120.0

O4—C17—C11	118.2 (10)	C6—C5—H5A	120.0
C12A—C11A—C16A	120.0	C5—C6—C1	120.0
C12A—C11A—C17A	116.6 (17)	C5—C6—H6A	120.0
C16A—C11A—C17A	123.3 (17)	C1—C6—H6A	120.0
C11A—C12A—C13A	120.0	C1A—N1—C7	117.5 (7)
C11A—C12A—Cl1A	121.3 (4)	C1A—N1—C10	118.6 (7)
C13A—C12A—Cl1A	118.7 (4)	C7—N1—C10	111.7 (3)
C14A—C13A—C12A	120.0	C7—N1—C1	121.9 (5)
C14A—C13A—H13A	120.0	C10—N1—C1	120.3 (5)
C12A—C13A—H13A	120.0	C8—N2—C9	109.7 (2)
C13A—C14A—C15A	120.0	C8—N2—H2B	108 (2)
C13A—C14A—H14A	120.0	C9—N2—H2B	109.3 (19)
C15A—C14A—H14A	120.0	C8—N2—H2C	111 (2)
C16A—C15A—C14A	120.0	C9—N2—H2C	112 (2)
C16A—C15A—H15A	120.0	H2B—N2—H2C	107 (3)
C14A—C15A—H15A	120.0	N1—C7—C8	111.2 (3)
C15A—C16A—C11A	120.0	N1—C7—H7A	109.4
C15A—C16A—H16A	120.0	C8—C7—H7A	109.4
C11A—C16A—H16A	120.0	N1—C7—H7B	109.4
O4A—C17A—O3A	140 (4)	C8—C7—H7B	109.4
O4A—C17A—C11A	118 (4)	H7A—C7—H7B	108.0
O3A—C17A—C11A	101 (2)	N2—C8—C7	111.6 (3)
O1A—N3A—O2A	123.5 (11)	N2—C8—H8A	109.3
O1A—N3A—C4A	118.5 (10)	C7—C8—H8A	109.3
O2A—N3A—C4A	117.8 (10)	N2—C8—H8B	109.3
N1—C1A—C2A	121.6 (9)	C7—C8—H8B	109.3
N1—C1A—C6A	117.4 (9)	H8A—C8—H8B	108.0
C2A—C1A—C6A	120.0	N2—C9—C10	111.3 (3)
C1A—C2A—C3A	120.0	N2—C9—H9A	109.4
C1A—C2A—H2	120.0	C10—C9—H9A	109.4
C3A—C2A—H2	120.0	N2—C9—H9B	109.4
C4A—C3A—C2A	120.0	C10—C9—H9B	109.4
C4A—C3A—H3	120.0	H9A—C9—H9B	108.0
C2A—C3A—H3	120.0	N1—C10—C9	111.9 (3)
C3A—C4A—C5A	120.0	N1—C10—H10A	109.2
C3A—C4A—N3A	118.0 (6)	C9—C10—H10A	109.2
C5A—C4A—N3A	122.0 (6)	N1—C10—H10B	109.2
C6A—C5A—C4A	120.0	C9—C10—H10B	109.2
C6A—C5A—H5	120.0	H10A—C10—H10B	107.9

C16—C11—C12—C13	0.0	O1A—N3A—C4A—C5A	4.2 (16)
C17—C11—C12—C13	177.0 (7)	O2A—N3A—C4A—C5A	179.5 (10)
C16—C11—C12—Cl1	−179.2 (4)	C3A—C4A—C5A—C6A	0.0
C17—C11—C12—Cl1	−2.3 (6)	N3A—C4A—C5A—C6A	177.9 (10)
H8A^{i}—Cl1—C12—C11	−77.7	C4A—C5A—C6A—C1A	0.0
H8A^{i}—Cl1—C12—C13	103.1	N1—C1A—C6A—C5A	−169.0 (13)
C11—C12—C13—C14	0.0	C2A—C1A—C6A—C5A	0.0
Cl1—C12—C13—C14	179.2 (4)	C6—C1—C2—C3	0.0

C12—C13—C14—C15	0.0	N1—C1—C2—C3	177.8 (9)
C13—C14—C15—C16	0.0	C1—C2—C3—C4	0.0
C14—C15—C16—C11	0.0	C2—C3—C4—C5	0.0
C12—C11—C16—C15	0.0	C2—C3—C4—N3	−177.9 (8)
C17—C11—C16—C15	−177.1 (6)	O1—N3—C4—C3	175.2 (11)
C12—C11—C17—O3	−99.0 (12)	O2—N3—C4—C3	−4.2 (14)
C16—C11—C17—O3	78.0 (14)	O1—N3—C4—C5	−2.8 (15)
C12—C11—C17—O4	75.1 (13)	O2—N3—C4—C5	177.8 (10)
C16—C11—C17—O4	−107.8 (11)	C3—C4—C5—C6	0.0
C16A—C11A—C12A—C13A	0.0	N3—C4—C5—C6	178.0 (8)
C17A—C11A—C12A—C13A	−176.2 (19)	C4—C5—C6—C1	0.0
C16A—C11A—C12A—Cl1A	177.7 (12)	C2—C1—C6—C5	0.0
C17A—C11A—C12A—Cl1A	1.5 (19)	N1—C1—C6—C5	−177.7 (9)
C11A—C12A—C13A—C14A	0.0	C2A—C1A—N1—C7	176.8 (6)
Cl1A—C12A—C13A—C14A	−177.7 (12)	C6A—C1A—N1—C7	−14.3 (11)
C12A—C13A—C14A—C15A	0.0	C2A—C1A—N1—C10	37.6 (12)
C13A—C14A—C15A—C16A	0.0	C6A—C1A—N1—C10	−153.6 (5)
C14A—C15A—C16A—C11A	0.0	C2—C1—N1—C7	−176.9 (4)
C12A—C11A—C16A—C15A	0.0	C6—C1—N1—C7	0.9 (9)
C17A—C11A—C16A—C15A	176 (2)	C2—C1—N1—C10	32.7 (8)
C12A—C11A—C17A—O4A	57 (5)	C6—C1—N1—C10	−149.5 (5)
C16A—C11A—C17A—O4A	−119 (4)	C1A—N1—C7—C8	164.1 (9)
C12A—C11A—C17A—O3A	−132 (2)	C10—N1—C7—C8	−54.0 (4)
C16A—C11A—C17A—O3A	52 (3)	C1—N1—C7—C8	153.4 (6)
N1—C1A—C2A—C3A	168.6 (14)	C9—N2—C8—C7	−57.1 (4)
C6A—C1A—C2A—C3A	0.0	N1—C7—C8—N2	56.4 (4)
C1A—C2A—C3A—C4A	0.0	C8—N2—C9—C10	56.2 (4)
C2A—C3A—C4A—C5A	0.0	C1A—N1—C10—C9	−164.9 (9)
C2A—C3A—C4A—N3A	−178.0 (10)	C7—N1—C10—C9	53.6 (4)
O1A—N3A—C4A—C3A	−177.9 (12)	C1—N1—C10—C9	−153.2 (6)
O2A—N3A—C4A—C3A	−2.6 (14)	N2—C9—C10—N1	−55.0 (4)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*A*···O2^{ii}	0.93	2.13	2.880 (15)	137
N2—H2*B*···O3^{iii}	0.88 (2)	1.86 (2)	2.740 (6)	172 (3)
N2—H2*B*···O3*A*^{iii}	0.88 (2)	1.73 (2)	2.590 (13)	164 (3)
N2—H2*C*···O4	0.89 (2)	1.83 (2)	2.705 (8)	169 (3)
N2—H2*C*···O4*A*	0.89 (2)	1.81 (3)	2.644 (19)	156 (3)
C8—H8*A*···Cl1^{iv}	0.97	2.82	3.629 (5)	142
C8—H8*A*···Cl1^{i}	0.97	2.95	3.780 (5)	144
C8—H8*A*···Cl1*A*^{i}	0.97	2.88	3.643 (12)	136
C8—H8*B*···O4*A*^{i}	0.97	2.58	3.13 (2)	116
C10—H10*A*···O3*A*^{iii}	0.97	2.65	3.285 (19)	123

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

4-(4-Nitrophenyl)piperazin-1-ium 2-bromobenzoate hemihydrate (2)

Crystal data

$C_{10}H_{14}N_3O_2^+ \cdot C_7H_4BrO_2^- \cdot 0.5H_2O$
$M_r = 417.26$
Triclinic, $P\overline{1}$
$a = 7.2570$ (5) Å
$b = 9.7772$ (6) Å
$c = 14.202$ (1) Å
$\alpha = 102.101$ (6)°
$\beta = 99.534$ (6)°
$\gamma = 110.981$ (6)°
$V = 887.41$ (11) Å^3
$Z = 2$
$F(000) = 426$
$D_x = 1.562$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2832 reflections
$\theta = 3.0$–$27.9°$
$\mu = 2.35$ mm^{-1}
$T = 293$ K
Prism, brown
0.36 × 0.32 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
ω scans
Absorption correction: multi-scan (CrysalisRed; Oxford Diffraction, 2009)
$T_{min} = 0.781$, $T_{max} = 1.000$
6177 measured reflections
3856 independent reflections
2478 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 28.0°$, $\theta_{min} = 3.0°$
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -11 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.084$
$S = 0.94$
3856 reflections
244 parameters
5 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ 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}	Occ. (<1)
O1	0.4472 (3)	0.9088 (2)	0.57716 (16)	0.0866 (7)	
O2	0.1466 (4)	0.7360 (3)	0.53813 (19)	0.0965 (8)	
N1	0.5490 (3)	0.3809 (2)	0.27923 (14)	0.0452 (5)	
N2	0.6259 (3)	0.1873 (2)	0.12301 (15)	0.0462 (5)	
H2A	0.686 (3)	0.132 (3)	0.1015 (18)	0.055*	
H2B	0.556 (3)	0.198 (3)	0.0700 (14)	0.055*	
N3	0.3218 (4)	0.7818 (3)	0.53126 (16)	0.0593 (6)	
C1	0.3797 (4)	0.6788 (3)	0.46525 (17)	0.0447 (6)	

C2	0.2487 (4)	0.5276 (3)	0.4245 (2)	0.0540 (7)	
H2	0.123215	0.491207	0.439437	0.065*	
C3	0.3015 (4)	0.4304 (3)	0.36200 (19)	0.0518 (6)	
H3	0.209824	0.328530	0.333702	0.062*	
C4	0.4910 (3)	0.4806 (2)	0.33958 (16)	0.0399 (5)	
C5	0.6204 (4)	0.6357 (3)	0.38273 (19)	0.0493 (6)	
H5	0.746350	0.673689	0.368381	0.059*	
C6	0.5675 (4)	0.7326 (3)	0.44508 (18)	0.0505 (6)	
H6	0.657730	0.834750	0.473932	0.061*	
C7	0.3935 (4)	0.2306 (3)	0.2201 (2)	0.0611 (8)	
H7A	0.307731	0.241586	0.164666	0.073*	
H7B	0.307496	0.187484	0.261102	0.073*	
C8	0.4847 (5)	0.1232 (3)	0.1806 (2)	0.0676 (8)	
H8A	0.557431	0.102503	0.235884	0.081*	
H8B	0.376210	0.026977	0.138441	0.081*	
C9	0.7856 (4)	0.3349 (3)	0.1852 (2)	0.0781 (10)	
H9A	0.874756	0.378980	0.145729	0.094*	
H9B	0.867453	0.319121	0.239824	0.094*	
C10	0.6991 (5)	0.4438 (3)	0.2265 (3)	0.0808 (10)	
H10A	0.809491	0.536313	0.271886	0.097*	
H10B	0.635383	0.471694	0.172320	0.097*	
Br1	0.92610 (5)	−0.00832 (3)	0.31967 (2)	0.06679 (14)	
O3	0.8488 (3)	0.0263 (3)	0.09269 (16)	0.0782 (6)	
O4	0.6156 (3)	−0.2125 (3)	0.03302 (17)	0.0860 (7)	
C11	0.9379 (3)	−0.1685 (3)	0.12893 (18)	0.0438 (6)	
C12	1.0135 (4)	−0.1304 (3)	0.23087 (19)	0.0469 (6)	
C13	1.1536 (4)	−0.1827 (3)	0.2715 (2)	0.0609 (7)	
H13	1.201561	−0.157757	0.340436	0.073*	
C14	1.2204 (4)	−0.2701 (3)	0.2107 (3)	0.0671 (8)	
H14	1.316352	−0.303199	0.238116	0.080*	
C15	1.1471 (4)	−0.3102 (3)	0.1086 (2)	0.0630 (8)	
H15	1.193664	−0.369875	0.067067	0.076*	
C16	1.0048 (4)	−0.2615 (3)	0.0683 (2)	0.0545 (7)	
H16	0.952495	−0.291267	−0.000611	0.065*	
C17	0.7895 (5)	−0.1121 (4)	0.0817 (2)	0.0564 (7)	
O1W	0.5508 (9)	−0.5202 (7)	−0.0662 (6)	0.138 (2)	0.5
H1W1	0.435882	−0.430124	−0.060307	0.207*	0.5
H1W2	0.458306	−0.629327	−0.042348	0.207*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0975 (16)	0.0587 (13)	0.0842 (15)	0.0191 (13)	0.0368 (13)	−0.0055 (11)
O2	0.0777 (15)	0.0870 (16)	0.1195 (19)	0.0333 (13)	0.0502 (15)	0.0009 (14)
N1	0.0415 (11)	0.0324 (10)	0.0568 (12)	0.0109 (9)	0.0146 (10)	0.0093 (9)
N2	0.0471 (13)	0.0423 (12)	0.0490 (12)	0.0236 (10)	0.0069 (10)	0.0080 (10)
N3	0.0699 (17)	0.0574 (15)	0.0511 (13)	0.0261 (14)	0.0222 (13)	0.0119 (11)
C1	0.0523 (15)	0.0453 (14)	0.0398 (13)	0.0228 (13)	0.0121 (12)	0.0143 (11)

C2	0.0445 (14)	0.0516 (15)	0.0666 (17)	0.0165 (13)	0.0229 (13)	0.0177 (13)
C3	0.0435 (15)	0.0382 (13)	0.0670 (17)	0.0108 (12)	0.0159 (13)	0.0112 (12)
C4	0.0384 (13)	0.0357 (12)	0.0452 (13)	0.0148 (11)	0.0067 (11)	0.0149 (10)
C5	0.0413 (14)	0.0372 (13)	0.0630 (16)	0.0100 (11)	0.0163 (12)	0.0105 (12)
C6	0.0506 (15)	0.0378 (13)	0.0524 (15)	0.0121 (12)	0.0083 (13)	0.0066 (11)
C7	0.0520 (16)	0.0403 (14)	0.0762 (18)	0.0041 (13)	0.0293 (15)	0.0039 (13)
C8	0.088 (2)	0.0366 (14)	0.0735 (18)	0.0160 (15)	0.0382 (17)	0.0111 (13)
C9	0.0509 (17)	0.0548 (17)	0.101 (2)	0.0095 (14)	0.0262 (17)	−0.0155 (16)
C10	0.075 (2)	0.0370 (15)	0.125 (3)	0.0103 (14)	0.061 (2)	0.0078 (16)
Br1	0.0855 (2)	0.05620 (19)	0.05928 (19)	0.02352 (16)	0.03290 (16)	0.01687 (13)
O3	0.1037 (16)	0.0759 (15)	0.0967 (15)	0.0672 (14)	0.0419 (13)	0.0421 (12)
O4	0.0686 (14)	0.1081 (18)	0.0862 (15)	0.0493 (15)	−0.0021 (13)	0.0322 (14)
C11	0.0435 (13)	0.0383 (13)	0.0501 (14)	0.0172 (11)	0.0089 (12)	0.0154 (11)
C12	0.0479 (14)	0.0379 (13)	0.0538 (15)	0.0126 (12)	0.0135 (12)	0.0194 (11)
C13	0.0569 (17)	0.0535 (16)	0.0625 (17)	0.0141 (15)	0.0009 (15)	0.0236 (14)
C14	0.0530 (17)	0.0570 (18)	0.094 (2)	0.0271 (15)	0.0038 (17)	0.0318 (17)
C15	0.0583 (17)	0.0508 (16)	0.088 (2)	0.0320 (15)	0.0202 (16)	0.0180 (15)
C16	0.0564 (16)	0.0532 (16)	0.0554 (15)	0.0288 (14)	0.0108 (13)	0.0107 (13)
C17	0.067 (2)	0.076 (2)	0.0507 (15)	0.0494 (18)	0.0227 (15)	0.0258 (15)
O1W	0.118 (4)	0.096 (4)	0.197 (7)	0.036 (4)	0.044 (4)	0.046 (4)

Geometric parameters (Å, º)

O1—N3	1.204 (3)	C8—H8A	0.9700
O2—N3	1.216 (3)	C8—H8B	0.9700
N1—C4	1.387 (3)	C9—C10	1.487 (4)
N1—C10	1.452 (3)	C9—H9A	0.9700
N1—C7	1.456 (3)	C9—H9B	0.9700
N2—C9	1.461 (3)	C10—H10A	0.9700
N2—C8	1.465 (3)	C10—H10B	0.9700
N2—H2A	0.846 (16)	Br1—C12	1.896 (2)
N2—H2B	0.881 (16)	O3—C17	1.230 (3)
N3—C1	1.452 (3)	O4—C17	1.254 (3)
C1—C2	1.370 (3)	C11—C12	1.378 (3)
C1—C6	1.376 (3)	C11—C16	1.389 (3)
C2—C3	1.363 (3)	C11—C17	1.502 (4)
C2—H2	0.9300	C12—C13	1.389 (4)
C3—C4	1.400 (3)	C13—C14	1.355 (4)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.399 (3)	C14—C15	1.377 (4)
C5—C6	1.361 (3)	C14—H14	0.9300
C5—H5	0.9300	C15—C16	1.375 (4)
C6—H6	0.9300	C15—H15	0.9300
C7—C8	1.492 (3)	C16—H16	0.9300
C7—H7A	0.9700	O1W—H1W1	1.4134
C7—H7B	0.9700	O1W—H1W2	1.1905
C4—N1—C10	118.22 (18)	C7—C8—H8A	109.4

C4—N1—C7	118.65 (17)	N2—C8—H8B	109.4
C10—N1—C7	112.0 (2)	C7—C8—H8B	109.4
C9—N2—C8	109.6 (2)	H8A—C8—H8B	108.0
C9—N2—H2A	106.7 (16)	N2—C9—C10	112.0 (2)
C8—N2—H2A	114.7 (17)	N2—C9—H9A	109.2
C9—N2—H2B	110.9 (17)	C10—C9—H9A	109.2
C8—N2—H2B	108.5 (16)	N2—C9—H9B	109.2
H2A—N2—H2B	106 (2)	C10—C9—H9B	109.2
O1—N3—O2	122.2 (2)	H9A—C9—H9B	107.9
O1—N3—C1	119.5 (2)	N1—C10—C9	112.8 (2)
O2—N3—C1	118.3 (2)	N1—C10—H10A	109.0
C2—C1—C6	120.1 (2)	C9—C10—H10A	109.0
C2—C1—N3	119.9 (2)	N1—C10—H10B	109.0
C6—C1—N3	120.0 (2)	C9—C10—H10B	109.0
C3—C2—C1	120.2 (2)	H10A—C10—H10B	107.8
C3—C2—H2	119.9	C12—C11—C16	118.0 (2)
C1—C2—H2	119.9	C12—C11—C17	122.6 (2)
C2—C3—C4	121.4 (2)	C16—C11—C17	119.3 (2)
C2—C3—H3	119.3	C11—C12—C13	120.8 (2)
C4—C3—H3	119.3	C11—C12—Br1	121.09 (19)
N1—C4—C5	121.73 (19)	C13—C12—Br1	118.1 (2)
N1—C4—C3	121.6 (2)	C14—C13—C12	120.0 (3)
C5—C4—C3	116.6 (2)	C14—C13—H13	120.0
C6—C5—C4	121.9 (2)	C12—C13—H13	120.0
C6—C5—H5	119.0	C13—C14—C15	120.4 (3)
C4—C5—H5	119.0	C13—C14—H14	119.8
C5—C6—C1	119.7 (2)	C15—C14—H14	119.8
C5—C6—H6	120.2	C16—C15—C14	119.7 (3)
C1—C6—H6	120.2	C16—C15—H15	120.2
N1—C7—C8	112.2 (2)	C14—C15—H15	120.2
N1—C7—H7A	109.2	C15—C16—C11	121.0 (3)
C8—C7—H7A	109.2	C15—C16—H16	119.5
N1—C7—H7B	109.2	C11—C16—H16	119.5
C8—C7—H7B	109.2	O3—C17—O4	126.0 (3)
H7A—C7—H7B	107.9	O3—C17—C11	117.9 (3)
N2—C8—C7	111.3 (2)	O4—C17—C11	116.2 (3)
N2—C8—H8A	109.4	H1W1—O1W—H1W2	105.4

O1—N3—C1—C2	170.2 (3)	N1—C7—C8—N2	−55.5 (3)
O2—N3—C1—C2	−9.8 (4)	C8—N2—C9—C10	−56.2 (3)
O1—N3—C1—C6	−8.7 (4)	C4—N1—C10—C9	165.9 (2)
O2—N3—C1—C6	171.2 (3)	C7—N1—C10—C9	−50.6 (4)
C6—C1—C2—C3	−1.4 (4)	N2—C9—C10—N1	53.6 (4)
N3—C1—C2—C3	179.7 (2)	C16—C11—C12—C13	0.5 (4)
C1—C2—C3—C4	1.4 (4)	C17—C11—C12—C13	−178.8 (2)
C10—N1—C4—C5	−26.0 (3)	C16—C11—C12—Br1	−178.41 (17)
C7—N1—C4—C5	−167.1 (2)	C17—C11—C12—Br1	2.4 (3)
C10—N1—C4—C3	155.3 (3)	C11—C12—C13—C14	1.2 (4)

C7—N1—C4—C3	14.2 (3)	Br1—C12—C13—C14	−179.9 (2)
C2—C3—C4—N1	177.4 (2)	C12—C13—C14—C15	−1.3 (4)
C2—C3—C4—C5	−1.3 (4)	C13—C14—C15—C16	−0.2 (4)
N1—C4—C5—C6	−177.4 (2)	C14—C15—C16—C11	1.9 (4)
C3—C4—C5—C6	1.4 (4)	C12—C11—C16—C15	−2.0 (4)
C4—C5—C6—C1	−1.4 (4)	C17—C11—C16—C15	177.2 (3)
C2—C1—C6—C5	1.4 (4)	C12—C11—C17—O3	65.2 (3)
N3—C1—C6—C5	−179.6 (2)	C16—C11—C17—O3	−114.1 (3)
C4—N1—C7—C8	−165.1 (2)	C12—C11—C17—O4	−115.8 (3)
C10—N1—C7—C8	51.5 (3)	C16—C11—C17—O4	64.9 (3)
C9—N2—C8—C7	57.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O3	0.85 (2)	1.83 (2)	2.655 (3)	164 (2)
N2—H2*B*···O4^{i}	0.88 (2)	1.82 (2)	2.701 (3)	173 (2)
C2—H2···O2^{ii}	0.93	2.50	3.307 (4)	145
C7—H7*B*···Br1^{iii}	0.97	3.11	4.032 (2)	160
C10—H10*B*···O1*W*^{i}	0.97	2.10	3.057 (8)	169

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

4-(4-Nitrophenyl)piperazin-1-ium 2-iodobenzoate hemihydrate (3)

Crystal data

$C_{10}H_{14}N_3O_2^+·C_7H_4IO_2^-·0.5H_2O$
M_r = 928.50
Triclinic, $P\overline{1}$
a = 7.3949 (6) Å
b = 9.3440 (8) Å
c = 14.498 (1) Å
α = 104.967 (8)°
β = 94.707 (7)°
γ = 107.430 (8)°
V = 909.44 (13) Å^3
Z = 1
$F(000)$ = 462
D_x = 1.695 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2991 reflections
θ = 3.0–28.0°
μ = 1.79 mm^{-1}
T = 293 K
Prism, orange
0.50 × 0.44 × 0.24 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
ω scans
Absorption correction: multi-scan (CrysalisRed; Oxford Diffraction, 2009)
T_{min} = 0.697, T_{max} = 1.000
6275 measured reflections
3904 independent reflections
2443 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.0°, θ_{min} = 3.0°
h = −9→9
k = −9→12
l = −19→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.117
S = 1.02
3904 reflections
264 parameters
13 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.5457P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: mixed	$(\Delta/\sigma)_{max} < 0.001$
H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{max} = 0.61$ e Å^{-3}
	$\Delta\rho_{min} = -0.44$ 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}	Occ. (<1)
C1	0.1376 (6)	0.6393 (4)	0.1581 (3)	0.0555 (10)	
C2	−0.0631 (7)	0.5730 (5)	0.1440 (3)	0.0687 (12)	
H2	−0.116914	0.503974	0.178262	0.082*	
C3	−0.1822 (7)	0.6059 (6)	0.0822 (4)	0.0724 (13)	
H3	−0.314731	0.558330	0.073932	0.087*	
C4	−0.1064 (7)	0.7096 (5)	0.0318 (3)	0.0636 (11)	
C5	0.0897 (7)	0.7763 (6)	0.0429 (3)	0.0723 (13)	
H5	0.141500	0.845343	0.008346	0.087*	
C6	0.2093 (7)	0.7419 (5)	0.1044 (3)	0.0667 (12)	
H6	0.341659	0.787923	0.110643	0.080*	
C7	0.4418 (7)	0.7234 (7)	0.2667 (5)	0.0957 (18)	
H7A	0.418923	0.802683	0.317971	0.115*	
H7B	0.499045	0.773784	0.220487	0.115*	
C8	0.5801 (7)	0.6593 (7)	0.3092 (4)	0.0927 (17)	
H8A	0.619508	0.592894	0.257190	0.111*	
H8B	0.693988	0.745578	0.346016	0.111*	
C9	0.3145 (8)	0.4451 (6)	0.3207 (4)	0.0897 (16)	
H9A	0.256781	0.389355	0.364560	0.108*	
H9B	0.340842	0.370385	0.267916	0.108*	
C10	0.1766 (7)	0.5111 (6)	0.2811 (4)	0.0835 (15)	
H10A	0.061412	0.425824	0.244767	0.100*	
H10B	0.139797	0.577245	0.334373	0.100*	
C11	0.8424 (6)	0.2081 (5)	0.3805 (3)	0.0609 (11)	
C12	0.8206 (6)	0.1340 (5)	0.2821 (3)	0.0635 (12)	
C13	0.9344 (9)	0.0414 (6)	0.2499 (4)	0.0850 (16)	
H13	0.920205	−0.009287	0.184112	0.102*	
C14	1.0663 (9)	0.0254 (7)	0.3150 (5)	0.0919 (17)	
H14	1.143373	−0.034148	0.292932	0.110*	
C15	1.0857 (8)	0.0952 (7)	0.4112 (5)	0.0857 (15)	
H15	1.174200	0.082483	0.455149	0.103*	
C16	0.9743 (7)	0.1845 (6)	0.4432 (4)	0.0769 (13)	
H16	0.987579	0.231020	0.509488	0.092*	
C17	0.7295 (7)	0.3142 (7)	0.4194 (3)	0.0746 (13)	
I1	0.61864 (5)	0.15483 (4)	0.18127 (2)	0.08807 (17)	

N1	0.2589 (5)	0.6027 (4)	0.2184 (3)	0.0622 (9)	
N2	0.4954 (6)	0.5683 (5)	0.3724 (3)	0.0686 (10)	
H2A	0.482 (7)	0.635 (6)	0.424 (4)	0.082*	
H2B	0.596 (7)	0.539 (6)	0.396 (4)	0.082*	
N3	−0.2340 (7)	0.7474 (5)	−0.0316 (3)	0.0825 (12)	
O1	−0.4061 (7)	0.6959 (6)	−0.0342 (4)	0.1259 (16)	
O2	−0.1641 (7)	0.8273 (6)	−0.0819 (3)	0.1165 (14)	
O3	0.6139 (6)	0.2673 (6)	0.4715 (3)	0.1206 (15)	
O4	0.7641 (6)	0.4395 (5)	0.4000 (3)	0.0969 (11)	
O1WA	0.426 (5)	0.976 (3)	0.449 (2)	0.154 (7)	0.224 (3)
H1W1	0.396 (13)	1.048 (8)	0.483 (6)	0.231*	0.224 (3)
H1W2	0.470 (14)	1.011 (10)	0.406 (4)	0.231*	0.224 (3)
O1WB	0.507 (4)	0.935 (3)	0.4569 (19)	0.149 (6)	0.276 (3)
H1W3	0.427 (8)	0.892 (15)	0.486 (6)	0.224*	0.276 (3)
H1W4	0.604 (7)	0.916 (12)	0.475 (10)	0.224*	0.276 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.061 (2)	0.043 (2)	0.057 (2)	0.0145 (19)	0.023 (2)	0.0061 (18)
C2	0.068 (3)	0.062 (3)	0.076 (3)	0.014 (2)	0.020 (2)	0.027 (2)
C3	0.065 (3)	0.065 (3)	0.079 (3)	0.012 (2)	0.017 (2)	0.017 (2)
C4	0.078 (3)	0.057 (3)	0.049 (2)	0.021 (2)	0.015 (2)	0.0047 (19)
C5	0.083 (3)	0.071 (3)	0.063 (3)	0.017 (2)	0.026 (2)	0.026 (2)
C6	0.062 (3)	0.067 (3)	0.067 (3)	0.010 (2)	0.018 (2)	0.025 (2)
C7	0.067 (3)	0.082 (3)	0.140 (5)	0.007 (3)	0.005 (3)	0.062 (3)
C8	0.063 (3)	0.124 (4)	0.113 (4)	0.029 (3)	0.024 (3)	0.073 (4)
C9	0.098 (4)	0.081 (3)	0.091 (4)	0.015 (3)	0.013 (3)	0.047 (3)
C10	0.069 (3)	0.090 (4)	0.094 (3)	0.009 (3)	0.018 (3)	0.051 (3)
C11	0.066 (3)	0.056 (2)	0.063 (2)	0.022 (2)	0.024 (2)	0.016 (2)
C12	0.073 (3)	0.042 (2)	0.066 (3)	0.008 (2)	0.028 (2)	0.0089 (19)
C13	0.110 (4)	0.056 (3)	0.085 (3)	0.026 (3)	0.048 (3)	0.004 (2)
C14	0.097 (4)	0.073 (3)	0.125 (5)	0.044 (3)	0.051 (4)	0.035 (3)
C15	0.082 (3)	0.086 (4)	0.110 (4)	0.039 (3)	0.033 (3)	0.046 (3)
C16	0.085 (3)	0.089 (3)	0.072 (3)	0.040 (3)	0.031 (3)	0.031 (3)
C17	0.073 (3)	0.079 (3)	0.064 (3)	0.032 (3)	0.012 (2)	0.000 (2)
I1	0.0918 (3)	0.0799 (2)	0.0696 (2)	0.00239 (18)	0.00710 (17)	0.01674 (16)
N1	0.061 (2)	0.057 (2)	0.071 (2)	0.0143 (17)	0.0198 (18)	0.0276 (17)
N2	0.073 (2)	0.081 (3)	0.065 (2)	0.038 (2)	0.0256 (19)	0.025 (2)
N3	0.092 (3)	0.082 (3)	0.063 (2)	0.019 (2)	0.002 (2)	0.019 (2)
O1	0.091 (3)	0.159 (4)	0.138 (4)	0.035 (3)	0.005 (3)	0.075 (3)
O2	0.116 (3)	0.134 (4)	0.100 (3)	0.022 (3)	0.003 (2)	0.064 (3)
O3	0.119 (3)	0.124 (3)	0.130 (3)	0.058 (3)	0.075 (3)	0.019 (3)
O4	0.106 (3)	0.077 (2)	0.116 (3)	0.051 (2)	0.018 (2)	0.020 (2)
O1WA	0.208 (17)	0.155 (15)	0.160 (13)	0.119 (12)	0.070 (15)	0.070 (12)
O1WB	0.210 (16)	0.145 (13)	0.161 (12)	0.123 (11)	0.079 (14)	0.069 (11)

Geometric parameters (Å, º)

C1—N1	1.377 (5)	C10—H10A	0.9700
C1—C6	1.398 (6)	C10—H10B	0.9700
C1—C2	1.401 (6)	C11—C16	1.384 (7)
C2—C3	1.359 (7)	C11—C12	1.387 (6)
C2—H2	0.9300	C11—C17	1.511 (7)
C3—C4	1.376 (6)	C12—C13	1.403 (7)
C3—H3	0.9300	C12—I1	2.090 (5)
C4—C5	1.373 (6)	C13—C14	1.369 (8)
C4—N3	1.443 (7)	C13—H13	0.9300
C5—C6	1.367 (7)	C14—C15	1.354 (8)
C5—H5	0.9300	C14—H14	0.9300
C6—H6	0.9300	C15—C16	1.365 (7)
C7—N1	1.454 (6)	C15—H15	0.9300
C7—C8	1.496 (7)	C16—H16	0.9300
C7—H7A	0.9700	C17—O4	1.232 (6)
C7—H7B	0.9700	C17—O3	1.243 (6)
C8—N2	1.461 (6)	N2—H2A	0.88 (5)
C8—H8A	0.9700	N2—H2B	0.93 (5)
C8—H8B	0.9700	N3—O1	1.212 (6)
C9—N2	1.465 (7)	N3—O2	1.212 (6)
C9—C10	1.487 (7)	O1WA—H1W1	0.82 (2)
C9—H9A	0.9700	O1WA—H1W2	0.82 (2)
C9—H9B	0.9700	O1WB—H1W3	0.83 (2)
C10—N1	1.453 (5)	O1WB—H1W4	0.82 (2)
N1—C1—C6	121.4 (4)	N1—C10—H10B	109.3
N1—C1—C2	122.6 (4)	C9—C10—H10B	109.3
C6—C1—C2	115.9 (4)	H10A—C10—H10B	107.9
C3—C2—C1	122.4 (4)	C16—C11—C12	118.0 (4)
C3—C2—H2	118.8	C16—C11—C17	120.1 (4)
C1—C2—H2	118.8	C12—C11—C17	121.9 (4)
C2—C3—C4	120.0 (4)	C11—C12—C13	119.3 (5)
C2—C3—H3	120.0	C11—C12—I1	121.3 (3)
C4—C3—H3	120.0	C13—C12—I1	119.4 (4)
C5—C4—C3	119.5 (5)	C14—C13—C12	120.1 (5)
C5—C4—N3	120.9 (4)	C14—C13—H13	119.9
C3—C4—N3	119.6 (4)	C12—C13—H13	119.9
C6—C5—C4	120.5 (4)	C15—C14—C13	120.8 (5)
C6—C5—H5	119.8	C15—C14—H14	119.6
C4—C5—H5	119.8	C13—C14—H14	119.6
C5—C6—C1	121.7 (4)	C14—C15—C16	119.4 (6)
C5—C6—H6	119.2	C14—C15—H15	120.3
C1—C6—H6	119.2	C16—C15—H15	120.3
N1—C7—C8	112.7 (4)	C15—C16—C11	122.3 (5)
N1—C7—H7A	109.1	C15—C16—H16	118.9
C8—C7—H7A	109.1	C11—C16—H16	118.9

N1—C7—H7B	109.1	O4—C17—O3	126.3 (5)
C8—C7—H7B	109.1	O4—C17—C11	118.0 (5)
H7A—C7—H7B	107.8	O3—C17—C11	115.6 (5)
N2—C8—C7	111.8 (4)	C1—N1—C10	118.7 (4)
N2—C8—H8A	109.3	C1—N1—C7	117.8 (3)
C7—C8—H8A	109.3	C10—N1—C7	111.5 (4)
N2—C8—H8B	109.3	C8—N2—C9	110.3 (4)
C7—C8—H8B	109.3	C8—N2—H2A	107 (3)
H8A—C8—H8B	107.9	C9—N2—H2A	114 (3)
N2—C9—C10	111.7 (4)	C8—N2—H2B	104 (3)
N2—C9—H9A	109.3	C9—N2—H2B	119 (3)
C10—C9—H9A	109.3	H2A—N2—H2B	102 (5)
N2—C9—H9B	109.3	O1—N3—O2	122.4 (5)
C10—C9—H9B	109.3	O1—N3—C4	119.3 (5)
H9A—C9—H9B	107.9	O2—N3—C4	118.3 (5)
N1—C10—C9	111.8 (4)	H1W1—O1WA—H1W2	103 (3)
N1—C10—H10A	109.3	H1W3—O1WB—H1W4	103 (3)
C9—C10—H10A	109.3		

N1—C1—C2—C3	−178.0 (4)	C12—C11—C16—C15	1.9 (7)
C6—C1—C2—C3	−0.1 (6)	C17—C11—C16—C15	−177.1 (5)
C1—C2—C3—C4	−1.0 (7)	C16—C11—C17—O4	111.4 (5)
C2—C3—C4—C5	1.5 (7)	C12—C11—C17—O4	−67.5 (6)
C2—C3—C4—N3	−178.2 (4)	C16—C11—C17—O3	−66.2 (6)
C3—C4—C5—C6	−0.8 (7)	C12—C11—C17—O3	114.9 (5)
N3—C4—C5—C6	178.8 (4)	C6—C1—N1—C10	172.5 (4)
C4—C5—C6—C1	−0.3 (7)	C2—C1—N1—C10	−9.7 (6)
N1—C1—C6—C5	178.7 (4)	C6—C1—N1—C7	32.9 (6)
C2—C1—C6—C5	0.8 (6)	C2—C1—N1—C7	−149.3 (5)
N1—C7—C8—N2	−53.1 (7)	C9—C10—N1—C1	164.4 (4)
N2—C9—C10—N1	56.1 (6)	C9—C10—N1—C7	−53.6 (6)
C16—C11—C12—C13	−1.3 (6)	C8—C7—N1—C1	−165.4 (4)
C17—C11—C12—C13	177.7 (4)	C8—C7—N1—C10	52.3 (7)
C16—C11—C12—I1	177.8 (3)	C7—C8—N2—C9	54.3 (7)
C17—C11—C12—I1	−3.3 (6)	C10—C9—N2—C8	−56.0 (6)
C11—C12—C13—C14	−0.4 (7)	C5—C4—N3—O1	−174.4 (5)
I1—C12—C13—C14	−179.5 (4)	C3—C4—N3—O1	5.2 (7)
C12—C13—C14—C15	1.6 (8)	C5—C4—N3—O2	7.1 (7)
C13—C14—C15—C16	−1.0 (8)	C3—C4—N3—O2	−173.3 (5)
C14—C15—C16—C11	−0.7 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···I1^{i}	0.93	3.28	4.110 (4)	150
C3—H3···O1^{ii}	0.93	2.53	3.347 (7)	147
C6—H6···I1^{iii}	0.93	3.26	3.940 (4)	132
C7—H7*A*···O1*WA*	0.97	2.14	3.09 (3)	167

C7—H7*A*···O1*WB*	0.97	2.01	2.85 (3)	145
N2—H2*A*···O3^{iv}	0.88 (5)	1.86 (5)	2.717 (5)	164 (5)
N2—H2*B*···O4	0.93 (5)	1.77 (5)	2.666 (6)	160 (5)
O1*WB*—H1*W*3···O3^{iv}	0.83 (2)	1.71 (10)	2.37 (2)	135 (12)

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