Syntheses and crystal structures of four 4-(4-methoxyphenyl)piperazin-1-ium salts: trifluoroacetate, 2,3,4,5,6-pentafluorobenzoate, 4-iodobenzoate, and a polymorph with 4-methylbenzoate

Vinaya,[a] Yeriyur B. Basavaraju,[a]* Hemmige S. Yathirajan[a]* and Sean Parkin[b]

[a]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysuru-570 006, India, and [b]Department of Chemistry, University of Kentucky, Lexington, KY, 40506-0055, USA. *Correspondence e-mail: ybb2706@gmail.com, yathirajan@hotmail.com

Syntheses and X-ray crystal structures of four 4-(4-methoxyphenyl)piperazin-1-ium (MeOPP) salts, with 2,2,2-trifluoroacetate, $C_{11}H_{17}N_2O^+ \cdot C_2F_3O_2^-$ (**I**), 2,3,4,5,6-pentafluorobenzoate, $C_{11}H_{17}N_2O^+ \cdot C_7F_5O_2^- \cdot H_2O$ (**II**), 4-iodobenzoate $C_{11}H_{17}N_2O^+ \cdot C_7H_4IO_2^- \cdot H_2O$ (**III**), and 4-methylbenzoate, $C_{11}H_{17}N_2O^+ \cdot C_8H_7O_2^- \cdot H_2O$ (**IV**) anions are presented. The salts form directly from equimolar quantities of N-(4-methoxyphenyl)piperazine and the corresponding organic acid in methanol and crystallize from 1:1 methanol/ethyl acetate. Salt **I** is anhydrous whereas **II**, **III**, and **IV** are all monohydrates. In all cases, the MeOPP cation conformation is determined by the torsion about the N—C bond between the piperazinium and 4-methoxybenzene rings. Crystal packing in each structure is largely dictated by N—H···O and (in **II**, **III**, and **IV**) O—H···O hydrogen bonds, although each also features weak C—H···O-type hydrogen bonds. Salt **II** also has π–π-stacking interactions between cation and anion arene rings, and **III** exhibits I···I close contacts.

1. Chemical context

In recent years, N-(4-methoxyphenyl)piperazine (MeOPP) has emerged as an addition to the range of designer recreational drugs. As such, considerable effort has been invested in the development of methods for the detection both of MeOPP itself and of its metabolites; N-(4-hydroxyphenyl)piperazine and 4-hydroxyaniline (Arbo et al., 2012) in human fluids (Staack & Maurer, 2003; Staack et al., 2004). MeOPP imparts euphoric stimulant properties, its actions on human physiology being similar to those of amphetamines (Staack & Maurer, 2005; Wohlfarth et al., 2010), but it has a significantly lower potential for abuse (Nagai et al., 2007). However, no therapeutic applications of MeOPP have been reported to date. In view of the reported psychoactive properties of MeOPP, coupled with the broad range of biological activities exhibited by piperazine derivatives in general (Asif, 2015; Brito et al., 2019), we recently initiated a programme of study centred on N-(4-methoxyphenyl)piperazine derivatives. Thus far, we have reported the synthesis and structures of a series of 1-aroyl-4-(4-methoxyphenyl) piperazines (Kiran Kumar et al., 2019a). We have also reported a series of 4-methoxyphenyl piperazin-1-ium salts formed with simple organic acids (Kiran Kumar et al., 2019b), and also reported crystal structures of the free-base compound N-(4-methoxyphenyl)piperazine (MeOPP) and three of its salts (Kiran Kumar et al., 2020a). More
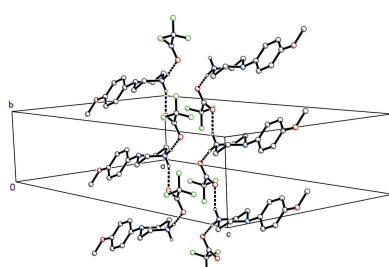
recently, we reported the crystal structures of 4-(4-methoxyphenyl)piperazin-1-ium 4-methylbenzoate monohydrate and bis-[4-(4-methoxyphenyl)piperazin-1-ium] benzene-1,2-dicarboxylate (Shankara Prasad *et al.*, 2022).


I

II

III

IV
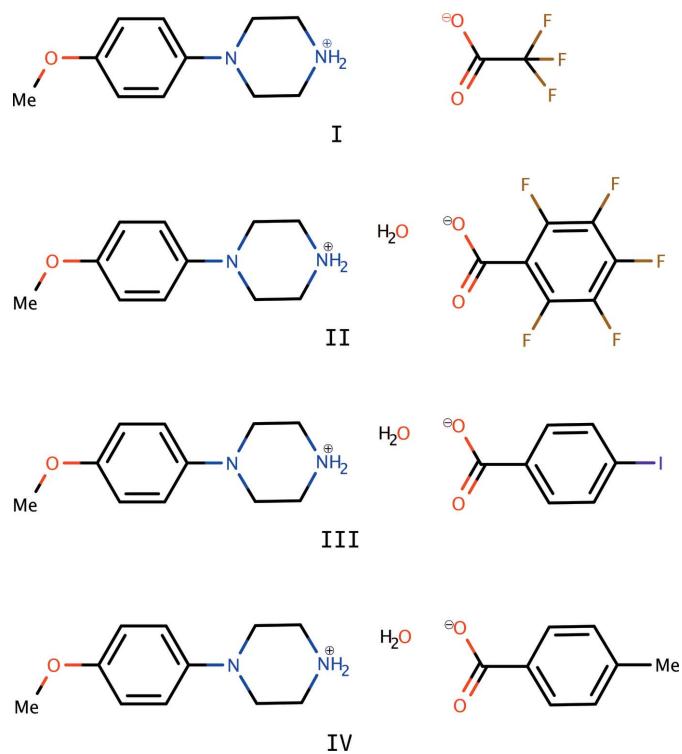
In view of the pharmacological importance of piperazines and the use of *N*-(4-methoxyphenyl)piperazine in particular, this paper presents the syntheses and crystal structures
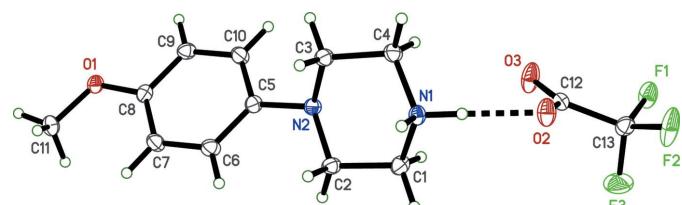
Figure 1
An ellipsoid plot (50% probability) of **I**. The dashed line denotes an N—H···O hydrogen bond. Hydrogen atoms are drawn as arbitrary circles.
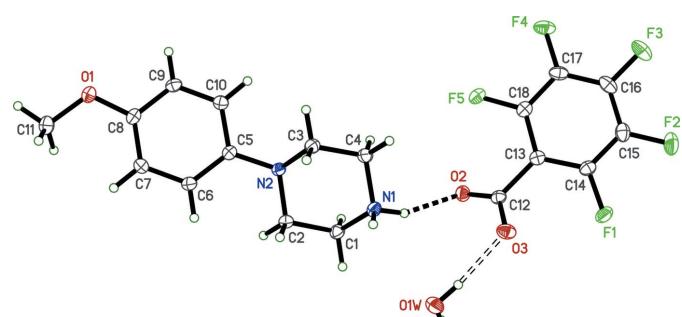
Figure 2
An ellipsoid plot (50% probability) of **II**. The solid dashed line denotes an N—H···O hydrogen bond, while the open dashed line shows an O—H···O hydrogen bond. Hydrogen atoms are drawn as arbitrary circles.
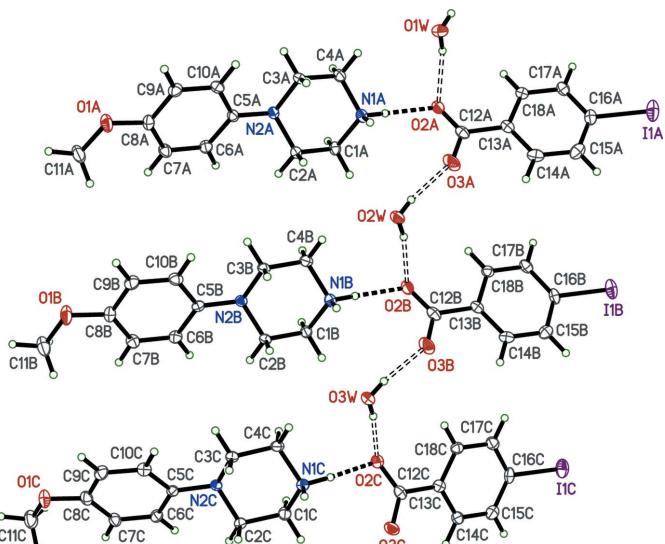
Figure 3
An ellipsoid plot (50% probability) of **III**. The solid dashed lines denote N—H···O hydrogen bonds, while the open dashed lines represent O—H···O hydrogen bonds. Hydrogen atoms are drawn as arbitrary circles.

of *N*-(4-methoxyphenyl)piperazin-1-ium trifluoroacetate, $C_{11}H_{17}N_2O^+ \cdot C_2F_3O_2^-$ (**I**), *N*-(4-methoxyphenyl)piperazin-1-ium 2,3,4,5,6-pentafluorobenzoate monohydrate, $C_{11}H_{17}N_2O^+ \cdot C_7F_5O_2^- \cdot H_2O$ (**II**), *N*-(4-methoxyphenyl)piperazin-1-ium 4-iodobenzoate monohydrate, $C_{11}H_{17}N_2O^+ \cdot C_7H_4IO_2^- \cdot H_2O$ (**III**) and a polymorph of *N*-(4-methoxyphenyl)piperazin-1-ium 4-methylbenzoate monohydrate, $C_{11}H_{17}N_2O^+ \cdot C_8H_7O_2^- \cdot H_2O$ (**IV**).

2. Structural commentary

Three of the four salts (see Figs. 1–4) crystallized as monohydrates; only **I** is anhydrous. Structure **III**, which is a much higher quality, low-temperature re-investigation of CSD entry KUJPUD [Kiran Kumar *et al.*, 2020*b*; CSD = Cambridge Structural Database (Groom *et al.*, 2016)] contains three copies of the cation, anion, and water within its asymmetric unit (*i.e.* Z′ = 3), all others have Z′ = 1. Structure **IV** is a polymorph of CSD entry XEMCIF (Shankara Prasad *et al.*, 2022). The asymmetric units were chosen so as to make the N—H···O hydrogen-bond geometry between the cation and anion as similar as possible, *i.e.* with the equatorial H atom of the NH_2^+ group as donor (see section 3: *Supramolecular*
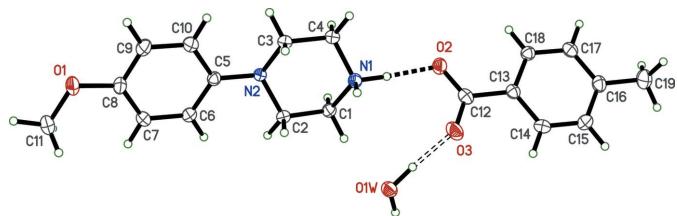
Figure 4
An ellipsoid plot (50% probability) of **IV**. The solid dashed line denotes an N—H···O hydrogen bond, while the open dashed line shows an O—H···O hydrogen bond. Hydrogen atoms are drawn as arbitrary circles.

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

| D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$A\cdots$O2	0.937 (16)	1.777 (16)	2.7084 (13)	172.1 (15)
N1—H1$B\cdots$O3^{i}	0.901 (16)	1.982 (16)	2.8329 (14)	156.7 (13)
C3—H3$B\cdots$O1^{ii}	0.99	2.55	3.2230 (14)	125
C4—H4$B\cdots$F1^{iii}	0.99	2.62	3.3162 (14)	127
C4—H4$A\cdots$O2^{iv}	0.99	2.51	3.1709 (14)	124
C11—H11$A\cdots$F3^{v}	0.98	2.59	3.2796 (15)	128

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$A\cdots$O2	0.912 (16)	1.886 (16)	2.7464 (12)	156.5 (14)
N1—H1$B\cdots$O3^{i}	0.882 (15)	1.909 (15)	2.7692 (12)	164.9 (13)
C1—H1$C\cdots$F5^{ii}	0.99	2.62	3.2261 (12)	119
C3—H3$B\cdots$O1W^{iii}	0.99	2.65	3.5235 (14)	148
C4—H4$A\cdots$O2^{iv}	0.99	2.57	3.4812 (13)	153
C10—H10$\cdots$O1W^{iii}	0.95	2.41	3.3581 (14)	177
C11—H11$A\cdots$F3^{v}	0.98	2.55	3.3961 (14)	144
C11—H11$B\cdots$F2^{vi}	0.98	2.51	3.2436 (15)	131
O1W—H1$W\cdots$O3	0.854 (19)	1.978 (19)	2.8222 (12)	170.0 (17)
O1W—H2$W\cdots$O2^{ii}	0.899 (18)	1.936 (19)	2.8319 (12)	173.7 (16)

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

features). The overall conformations of the cations in **I**–**IV** are determined, in large part, by the twist of the N2—C5 bonds that connect the 4-methoxyphenyl and piperazinium groups (Figs. 1–4). These twists, quantified for example by the dihedral angle between the mean planes of the benzene ring (C5–C10) and the four carbon atoms (C1–C4) of the piperazinium rings, are 40.63 (5)° (**I**), 36.05 (4)° (**II**), 25.28 (13), 26.59 (12), and 24.82 (11)° (for **III**a,b,c, respectively), and 7.57 (8)° (**IV**), showing moderate variability across the four structures (Fig. 5). The geometry of the N2 atoms in each cation is non-planar; the sums of bond angles about N2 ranging from 337.46 (16)° in **I** to 342.4 (3)° for N2C in **III**c. In each structure, the 4-methoxy groups are close to coplanar with their attached benzene rings, the largest deviation out of plane being only 0.188 (4) Å for C11C in **III**c, which corresponds to a C9C—C8C—O1C—C11C torsion angle of 172.3 (2)°.

The conformation of the trifluoroacetate anion in **I**, is largely unremarkable, having a dihedral angle between the plane of the carboxylate group and the plane formed by atoms C12, C13, and F3 of 89.29 (12)°. In the substituted benzoate anions of **II**, **III**, and **IV**, the dihedral angles between the carboxylate groups and the benzene rings are 43.28 (5)° (**II**), 3.8 (2)°, 7.46 (19)°, and 23.6 (2)° (**III**a,b,c, respectively) and 8.60 (11)° (**IV**).

3. Supramolecular features

Strong hydrogen bonds are the dominant intermolecular interactions in each of the four salts. All other hydrogen-bond-type interactions are weak. Salt **II** has weak π–π interactions and **III** has I$\cdots$I contacts, as described below.

The hydrogen bonding in **I** is the simplest of the four salts. There are only two N—H$\cdots$O hydrogen bonds, the shortest being N1—H1$NA\cdots$O2, at $d_{D\cdots A}$ = 2.7084 (13) Å. In addition, N1—H1$NB\cdots$O3^{i} (symmetry code as per Table 1) at $d_{D\cdots A}$ = 2.8329 (14) Å, connects cations and anions into chains that extend parallel to its crystallographic b axis, with inversion-related chains running anti-parallel (Fig. 6). Other than a few C—H$\cdots$O and C—H$\cdots$F close contacts (also listed in Table 1), there are no other significant inter-species interactions.

In **II**, the chosen asymmetric unit includes N1—H1$NA\cdots$O2 as the shortest hydrogen bond, at 2.7464 (12) Å. The cation also hydrogen bonds to an inversion-related anion *via* N1—H1$NB\cdots$O3^{i} (symmetry operator as per Table 2), $d_{D\cdots A}$ = 2.7692 (12) Å. The water molecule acts as donor in two strong O1W—H1$W\cdots$O3 (same asymmetric unit) and O1W—H2$W1\cdots$O2^{ii} (2$_1$ screw-related, as per Table 2) hydrogen bonds, and as acceptor in two weak C—H$\cdots$O$_{water}$ interactions, as listed in Table 2. There are a few much weaker C—H$\cdots$F close contacts (Table 2). The hydrogen bonding in **II** is augmented by π–π stacking of the cation benzene ring with 2$_1$ screw ($-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$) and inversion-related ($1 - x, 1 - y, 1 - z$) anion pentafluorobenzene rings.

Figure 5
An overlay of six 4-MeOPP cations (least-squares fit of piperazinium ring atoms), showing the variability of MeOPP conformation across structures **I**–**IV**.

Overlay legend:
- I
- II
- IIIa
- IIIb
- IIIc
- IV
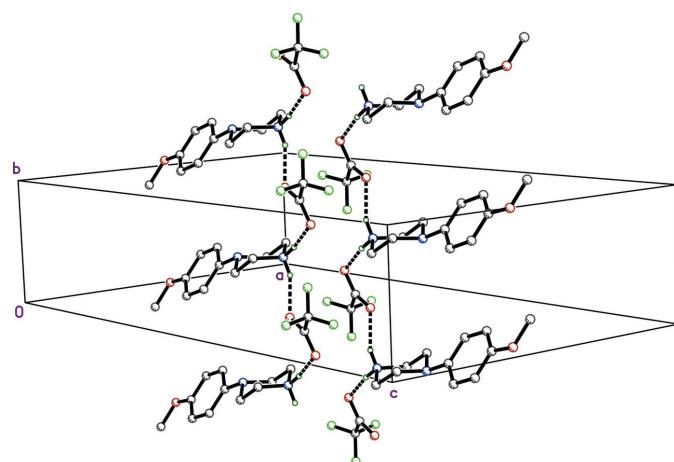
Figure 6
A partial packing plot of **I** showing hydrogen-bonded chains (solid dashed lines) parallel to the b-axis. Hydrogen atoms not involved in hydrogen bonds are omitted.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W1···O3C^i	0.77 (2)	1.93 (2)	2.696 (3)	173 (3)
O1W—H2W1···O2A	0.77 (2)	1.96 (2)	2.700 (3)	162 (3)
O2W—H1W2···O3A	0.77 (1)	1.86 (2)	2.626 (2)	170 (3)
O2W—H2W2···O2B	0.77 (2)	1.97 (2)	2.733 (3)	168 (3)
O3W—H1W3···O3B	0.77 (2)	1.87 (2)	2.636 (2)	172 (3)
O3W—H2W3···O2C	0.78 (1)	1.94 (2)	2.706 (3)	172 (3)
N1A—H1AB···O3W^{ii}	0.94 (3)	1.82 (3)	2.754 (3)	172 (3)
N1A—H1AA···O2A	0.90 (3)	1.89 (3)	2.776 (3)	168 (3)
C1A—H1AC···O2W	0.99	2.57	3.362 (3)	136
C2A—H2AB···O3B^{ii}	0.99	2.51	3.498 (3)	175
C4A—H4AA···O2C^{iii}	0.99	2.47	3.441 (3)	166
C7A—H7A···I1B^{iii}	0.95	3.32	4.212 (2)	156
C11A—H11A···O1B^{iv}	0.98	2.51	3.225 (4)	130
N1B—H1BA···O2B	0.91 (3)	1.87 (3)	2.780 (3)	174 (3)
N1B—H1BB···O2W^{ii}	0.92 (3)	1.85 (3)	2.768 (3)	176 (2)
C1B—H1BC···O3W	0.99	2.44	3.229 (3)	136
C2B—H2BB···O3A^{ii}	0.99	2.63	3.619 (3)	174
C4B—H4BA···O2B^{iii}	0.99	2.51	3.491 (3)	170
C4B—H4BB···O2W	0.99	2.52	3.260 (3)	131
C9B—H9B···I1B^{ii}	0.95	3.25	4.020 (3)	139
N1C—H1CA···O2C	0.96 (3)	1.78 (3)	2.732 (3)	172 (3)
N1C—H1CA···O3C	0.96 (3)	2.64 (3)	3.297 (3)	126 (2)
N1C—H1CB···O1W^{ii}	0.83 (3)	1.96 (3)	2.781 (3)	172 (3)
C1C—H1CC···O1W^v	0.99	2.39	3.299 (3)	152
C4C—H4CA···O2A^{iii}	0.99	2.45	3.438 (3)	174
C9C—H9C···I1A^{ii}	0.95	3.29	4.013 (3)	135

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2	1.028 (17)	1.714 (17)	2.7391 (15)	174.1 (15)
N1—H1B···O1W^i	0.939 (16)	1.873 (17)	2.7977 (16)	167.8 (14)
C1—H1C···O1W	0.99	2.46	3.2617 (17)	138
C2—H2B···O3^i	0.99	2.52	3.5088 (17)	174
C4—H4A···O2^{ii}	0.99	2.55	3.5294 (17)	169
C4—H4B···O1W^{iii}	0.99	2.54	3.3184 (17)	135
O1W—H1W···O3	0.97 (2)	1.67 (2)	2.6418 (14)	176.5 (18)
O1W—H2W···O2^{iv}	0.94 (2)	1.83 (2)	2.7626 (15)	170.5 (17)

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

offset, leading to centroid–centroid distances of 3.567 (2) Å. The net result is a complicated double-layered structure that extends parallel to the *bc* plane, a slice through which is shown in Fig. 7.

The asymmetric unit of **III** contains three crystallographically inequivalent groups of cation, anion, and water molecules (Fig. 3). Within each group, the species are hydrogen bonded in a similar pattern to the asymmetric unit of **II**, and the cation–anion pairs are linked by the water mol-

However, the fluorinated rings are 6.03 (3)° out of co-planarity with the MeOPP arene ring and the stacking is
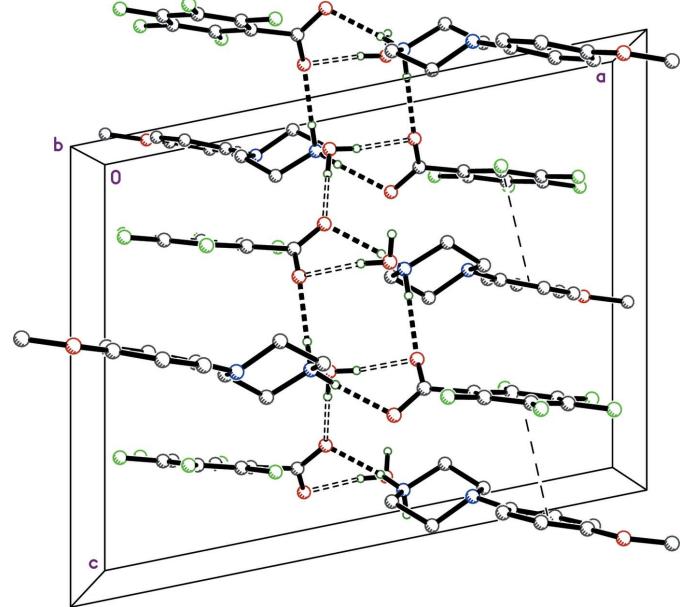
Figure 7
A partial packing plot of **II** viewed down the *b*-axis showing a slice through the N—H···O (solid dashed lines) and O—H···O (open dashed lines) hydrogen-bonded double layers. Thin dashed lines indicate π–π stacking of cation and anion arene rings. Hydrogen atoms not participating in hydrogen bonds are omitted.

Figure 8
A partial packing plot of **III** viewed approximately down the *b*-axis showing a slice through the N—H···O (solid dashed lines) and O—H···O (open dashed lines) hydrogen-bonded double layers, which stack along the *b*-axis direction *via* I···I close contacts (dotted lines). Hydrogen atoms not involved in hydrogen bonds are omitted.
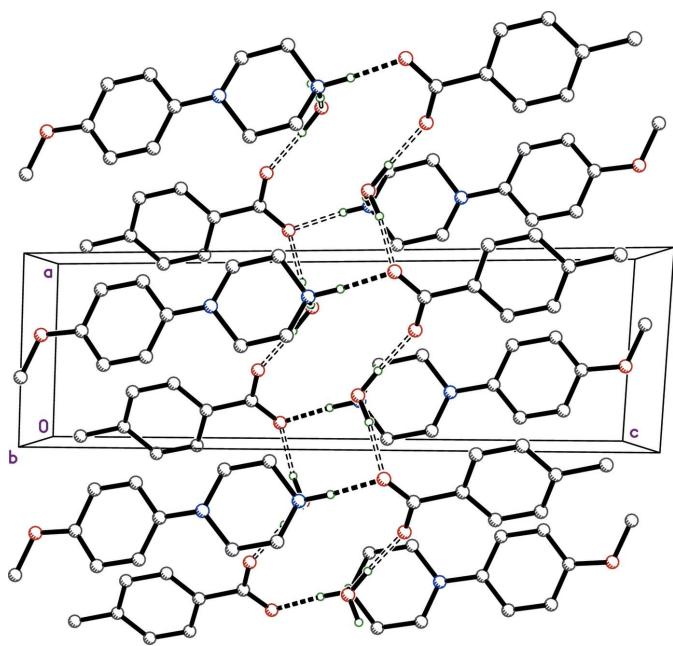
Figure 9
A packing plot of **IV** viewed down the *b*-axis, showing N—H···O (solid dashes) and O—H···O (open dashes) hydrogen bonds, which form bi-layered tapes in the *ac* plane that extend parallel to *a*. Hydrogen atoms not involved in hydrogen bonds are omitted.

ecules. The asymmetric units are linked by O—H···O$_{water}$ hydrogen bonds to glide-related ($x, \frac{1}{2} - y, \frac{1}{2} + z$ and $x, -\frac{1}{2} - y, \frac{1}{2} + z$) equivalents parallel to *c* (Figs. 3 and 8). These connections lead to double layers parallel to the *ac* plane. Details of the hydrogen-bonding interactions are given in Table 3. The double-layers are themselves connected by I···I close contacts to an inversion-related asymmetric unit [$d_{I···I}$ = 3.8586 (4) Å for I1*B*···I1*B*^{inv} and 4.0444 (4) Å for I1*A*···I1*C*^{inv} and I1*C*···I1*A*^{inv} (inv = $-x, -y, 1 - z$)], also depicted in Fig. 8.

In the structure of **IV**, a strong N1—H1*NA*···O2 [$d_{D···A}$ = 2.7391 (15) Å] hydrogen bond links the cation and anion. In combination with hydrogen bonds N1—H1*NB*···O1*W*^{i}, O1*W*—H1*W*···O3, and O1*W*—H2*W*···O2^{iv} (symmetry codes as per Table 4) involving water molecules, bi-layered tapes in the *ac* plane extend along the *a*-axis direction (Fig. 9). There are no other noteworthy interactions other than a few C—H···O contacts, which are also listed in Table 4.

4. Database survey

A search of the Cambridge Structural Database (CSD, v5.43 plus updates to November 2022; Groom *et al.*, 2016) for a search fragment consisting of 1-phenylpiperazine (without substituents) gave 1871 hits. With 'any non-H atom' substituted at the 4-position of the benzene ring, but all other carbons bearing hydrogen, a search found 225 matches, 46 of which had the $R_2H_2^+$ piperazinium cation. This search fragment, but with a methoxy group added at the 4-position of the benzene ring, returned 20 structures. The parent molecule, 4-MeOPP, is present as CSD entry IHILOD (Kiran Kumar *et*

al., 2020*a*). CSD code EGUROO (Zia-ur-Rehman *et al.*, 2009) is the chloride salt of the 4-MeOPP cation and OMUXIG (Gharbi *et al.*, 2021) is a 4-MeOPP salt with Co(NCS)$_4^{2-}$ as its anion. The rest are all salts with a variety of organic anions: FOVPEO, FOVPOY, FOVPUE, FOVQAL, FOVQEP, FOVQIT, FOVQOZ, FOVQUF, FOVRAM, FOVREQ, FOVRIU, and FOVROA having been published by Kiran Kumar *et al.* (2019*a*). Structures IHILUJ, IHIMAQ, & IHIMEU (along with IHILOD) were also published by Kiran Kumar *et al.* (2020*a*), and XEMCIF and XEMCOL by Shankara Prasad *et al.* (2022). Entry KUJPUD, present as a *CSD Communication* (Kiran Kumar *et al.*, 2020*b*), is a poor-quality room-temperature structure of the 4-iodobenzoate, **III**. Similar related structures include the 1-aroyl-4-(4-methoxyphenyl) piperazines VONFOW, VONGAJ, VONGEN, VONGIR, VONGOX, & VONGUD (Kiran Kumar *et al.*, 2019*b*).

5. Synthesis and crystallization

All reagents were obtained commercially and were used as received. For the synthesis of the salts, equimolar quantities (0.52 mmol of each component) of *N*-(4-methoxyphenyl)piperazine (100 mg) (from Sigma-Aldrich) and either trifluoroacetic acid (60 mg, **I**), pentafluorobenzoic acid (110 mg, **II**), 4-iodobenzoic acid (129 mg, **III**), or 4-methylbenzoic acid (71 mg, **IV**) were separately dissolved in methanol (10 ml). The two solutions were mixed and stirred briefly at 333 K and then set aside to crystallize, giving the solid products **I** to **IV** after a few days. The products were collected by filtration and then dried in air (**I**: yield 80%, m.p. 390–392 K; **II**: yield 75%, m.p. 375–377 K; **III**: yield 85%, m.p. 426–428 K; **IV**: yield 70%, m.p. 406–408 K). Crystals of compounds **I** to **IV** suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in methanol:ethyl acetate (initial composition 1:1, *v/v*).

6. Refinement

Crystal data, data collection, and refinement statistics are given in Table 5. All hydrogen atoms were located in difference-Fourier maps. Those bound to nitrogen or oxygen were refined freely, while carbon-bound hydrogens were included in the refinement using riding models with constrained distances set to 0.95 Å (Csp^2H), 0.99 Å (R_2CH_2), and 0.98 Å (RCH_3) using U_{iso}(H) values constrained to $1.2U_{eq}$ or $1.5U_{eq}$ (methyl only) of the attached carbon atom.

Acknowledgements

Table 5
Experimental details.

	I	II	III	IV
Crystal data				
Chemical formula	$C_{11}H_{17}N_2O^+ \cdot C_2F_3O_2^-$	$C_{11}H_{17}N_2O^+ \cdot C_7F_5O_2^- \cdot H_2O$	$C_{11}H_{17}N_2O^+ \cdot C_7H_4IO_2^- \cdot H_2O$	$C_{11}H_{17}N_2O^+ \cdot C_8H_7O_2^- \cdot H_2O$
M_r	306.28	422.35	458.28	346.42
Crystal system, space group	Orthorhombic, $Pbca$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$
Temperature (K)	90	90	90	90
a, b, c (Å)	16.2967 (9), 6.0887 (2), 28.4139 (15)	16.9733 (7), 8.3512 (4), 13.2980 (4)	20.4117 (18), 7.4255 (6), 36.796 (3)	6.1481 (13), 7.3467 (12), 19.980 (4)
α, β, γ (°)	90, 90, 90	90, 101.494 (1), 90	90, 92.970 (3), 90	80.190 (6), 86.089 (5), 82.843 (6)
V (Å^3)	2819.4 (2)	1847.16 (13)	5569.6 (8)	881.3 (3)
Z	8	4	12	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.13	0.14	1.75	0.09
Crystal size (mm)	$0.24 \times 0.18 \times 0.11$	$0.30 \times 0.29 \times 0.14$	$0.30 \times 0.13 \times 0.03$	$0.29 \times 0.21 \times 0.02$
Data collection				
Diffractometer	Bruker D8 Venture dual source	Bruker D8 Venture dual source	Bruker D8 Venture dual source	Bruker D8 Venture dual source
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.847, 0.958	0.919, 0.971	0.726, 0.862	0.877, 0.959
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	26032, 3243, 2802	30724, 4238, 3793	72181, 12876, 10003	23917, 4059, 3137
R_{int}	0.038	0.031	0.056	0.041
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.651	0.650	0.653	0.652
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.087, 1.05	0.031, 0.083, 1.03	0.033, 0.069, 1.03	0.038, 0.080, 1.07
No. of reflections	3243	4238	12876	4059
No. of parameters	200	280	729	245
No. of restraints	0	0	12	0
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	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.39, −0.22	0.34, −0.19	0.84, −0.81	0.19, −0.18

Computer programs: *APEX3* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*), *XP* in *SHELXTL* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2020), *SHELX* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

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Syntheses and crystal structures of four 4-(4-methoxyphenyl)piperazin-1-ium salts: trifluoroacetate, 2,3,4,5,6-pentafluorobenzoate, 4-iodobenzoate, and a polymorph with 4-methylbenzoate

Vinaya, Yeriyur B. Basavaraju, Hemmige S. Yathirajan and Sean Parkin

Computing details

For all structures, data collection: *APEX3* (Bruker, 2016); cell refinement: *APEX3* (Bruker, 2016); data reduction: *APEX3* (Bruker, 2016); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2019/2* (Sheldrick, 2015b); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *SHELX* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

4-(4-Methoxyphenyl)piperazin-1-ium 2,2,2-trifluoroacetate (I)

Crystal data

$C_{11}H_{17}N_2O^+ \cdot C_2F_3O_2^-$	$D_x = 1.443$ Mg m^{-3}
$M_r = 306.28$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, $Pbca$	Cell parameters from 9961 reflections
$a = 16.2967$ (9) Å	$\theta = 2.5$–27.5°
$b = 6.0887$ (2) Å	$\mu = 0.13$ mm^{-1}
$c = 28.4139$ (15) Å	$T = 90$ K
$V = 2819.4$ (2) Å^3	Solvent-rounded block, colourless
$Z = 8$	$0.24 \times 0.18 \times 0.11$ mm
$F(000) = 1280$	

Data collection

Bruker D8 Venture dual source diffractometer	26032 measured reflections
Radiation source: microsource	3243 independent reflections
Detector resolution: 7.41 pixels mm^{-1}	2802 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.038$
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)	$\theta_{max} = 27.6°$, $\theta_{min} = 1.9°$
	$h = -20 \rightarrow 21$
	$k = -7 \rightarrow 7$
$T_{min} = 0.847$, $T_{max} = 0.958$	$l = -36 \rightarrow 37$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.034$	
$wR(F^2) = 0.087$	
$S = 1.05$	Hydrogen site location: mixed
3243 reflections	H atoms treated by a mixture of independent and constrained refinement
200 parameters	
0 restraints	

$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 1.3982P]$
　where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Extinction correction: SHELXL-2019/2
　(Sheldrick 2015b),
　$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0023 (7)

Special details

Experimental. The crystal was mounted using polyisobutene oil on the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder. It was placed directly into the cold gas stream of a liquid-nitrogen based cryostat (Hope, 1994; Parkin & Hope, 1998).
Diffraction data were collected with the crystal at 90K, which is standard practice in this laboratory for the majority of flash-cooled crystals.

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.

Refinement. Refinement progress was checked using *Platon* (Spek, 2020) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.64348 (5)	0.88138 (14)	0.84741 (3)	0.01761 (19)
N1	0.57880 (6)	0.57351 (17)	0.56220 (3)	0.0147 (2)
H1A	0.5746 (10)	0.487 (3)	0.5350 (6)	0.029 (4)*
H1B	0.5680 (9)	0.713 (3)	0.5535 (5)	0.023 (4)*
N2	0.61770 (6)	0.60018 (16)	0.66037 (3)	0.0155 (2)
C1	0.66484 (7)	0.5639 (2)	0.57944 (4)	0.0179 (2)
H1C	0.702091	0.626714	0.555436	0.022*
H1D	0.680835	0.408985	0.584747	0.022*
C2	0.67297 (7)	0.6921 (2)	0.62512 (4)	0.0172 (2)
H2A	0.730261	0.684243	0.636575	0.021*
H2B	0.659190	0.848350	0.619580	0.021*
C3	0.53255 (7)	0.6192 (2)	0.64390 (4)	0.0179 (2)
H3A	0.519051	0.775947	0.638751	0.022*
H3B	0.494967	0.561212	0.668281	0.022*
C4	0.52025 (7)	0.4935 (2)	0.59861 (4)	0.0181 (2)
H4A	0.529255	0.334880	0.604256	0.022*
H4B	0.463242	0.513595	0.587369	0.022*
C5	0.62749 (7)	0.68174 (19)	0.70730 (4)	0.0155 (2)
C6	0.67365 (7)	0.8657 (2)	0.71851 (4)	0.0186 (3)
H6	0.700715	0.944071	0.694126	0.022*
C7	0.68114 (7)	0.9382 (2)	0.76507 (4)	0.0186 (2)
H7	0.713564	1.063514	0.772171	0.022*
C8	0.64107 (7)	0.82630 (19)	0.80068 (4)	0.0154 (2)
C9	0.59454 (7)	0.6405 (2)	0.78982 (4)	0.0176 (2)
H9	0.567039	0.563175	0.814201	0.021*
C10	0.58821 (7)	0.5683 (2)	0.74383 (4)	0.0182 (2)
H10	0.556949	0.440604	0.736930	0.022*
C11	0.68848 (8)	1.0742 (2)	0.85963 (4)	0.0216 (3)

H11A	0.683518	1.100895	0.893519	0.032*
H11B	0.666423	1.200190	0.842301	0.032*
H11C	0.746404	1.053795	0.851455	0.032*
F1	0.62003 (5)	−0.25022 (11)	0.46795 (3)	0.02609 (19)
F2	0.60056 (6)	0.00973 (14)	0.41859 (3)	0.0388 (2)
F3	0.71197 (5)	−0.00256 (16)	0.45876 (4)	0.0485 (3)
O2	0.57475 (6)	0.29613 (14)	0.48772 (3)	0.0226 (2)
O3	0.59231 (7)	0.02571 (15)	0.54005 (3)	0.0290 (2)
C12	0.59453 (7)	0.10857 (19)	0.50037 (4)	0.0151 (2)
C13	0.63112 (8)	−0.0358 (2)	0.46090 (4)	0.0198 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0188 (4)	0.0216 (4)	0.0124 (4)	−0.0011 (3)	−0.0001 (3)	−0.0024 (3)
N1	0.0180 (5)	0.0138 (5)	0.0124 (4)	0.0007 (4)	−0.0012 (4)	0.0000 (4)
N2	0.0148 (5)	0.0202 (5)	0.0114 (4)	−0.0027 (4)	−0.0008 (3)	0.0002 (4)
C1	0.0156 (5)	0.0232 (6)	0.0150 (5)	0.0022 (5)	−0.0001 (4)	0.0000 (4)
C2	0.0146 (5)	0.0231 (6)	0.0138 (5)	−0.0026 (4)	0.0002 (4)	0.0011 (4)
C3	0.0145 (5)	0.0252 (6)	0.0141 (5)	−0.0023 (4)	−0.0008 (4)	−0.0013 (4)
C4	0.0178 (5)	0.0228 (6)	0.0137 (5)	−0.0044 (5)	−0.0009 (4)	−0.0002 (4)
C5	0.0151 (5)	0.0184 (5)	0.0128 (5)	0.0001 (4)	−0.0015 (4)	0.0003 (4)
C6	0.0195 (6)	0.0218 (6)	0.0147 (5)	−0.0048 (5)	0.0004 (4)	0.0024 (4)
C7	0.0199 (6)	0.0188 (6)	0.0169 (5)	−0.0050 (5)	−0.0019 (4)	−0.0008 (4)
C8	0.0145 (5)	0.0186 (6)	0.0130 (5)	0.0028 (4)	−0.0016 (4)	−0.0009 (4)
C9	0.0169 (5)	0.0209 (6)	0.0150 (5)	−0.0020 (5)	0.0007 (4)	0.0027 (4)
C10	0.0191 (6)	0.0185 (6)	0.0170 (5)	−0.0044 (5)	−0.0012 (4)	0.0002 (4)
C11	0.0234 (6)	0.0235 (6)	0.0181 (5)	−0.0025 (5)	−0.0022 (5)	−0.0047 (5)
F1	0.0437 (5)	0.0128 (4)	0.0218 (4)	0.0006 (3)	0.0057 (3)	−0.0020 (3)
F2	0.0776 (7)	0.0254 (4)	0.0134 (4)	0.0096 (4)	0.0024 (4)	−0.0011 (3)
F3	0.0268 (5)	0.0461 (6)	0.0725 (7)	−0.0087 (4)	0.0245 (4)	−0.0251 (5)
O2	0.0341 (5)	0.0161 (4)	0.0174 (4)	0.0053 (4)	−0.0032 (4)	−0.0013 (3)
O3	0.0552 (7)	0.0175 (4)	0.0144 (4)	0.0027 (4)	0.0036 (4)	0.0009 (3)
C12	0.0155 (5)	0.0144 (5)	0.0154 (5)	−0.0020 (4)	−0.0001 (4)	−0.0018 (4)
C13	0.0247 (6)	0.0155 (6)	0.0192 (6)	−0.0020 (5)	0.0054 (5)	0.0002 (4)

Geometric parameters (Å, º)

O1—C8	1.3701 (13)	C5—C6	1.3862 (16)
O1—C11	1.4269 (15)	C5—C10	1.4015 (16)
N1—C1	1.4864 (15)	C6—C7	1.3998 (16)
N1—C4	1.4893 (14)	C6—H6	0.9500
N1—H1A	0.937 (16)	C7—C8	1.3835 (16)
N1—H1B	0.901 (16)	C7—H7	0.9500
N2—C5	1.4318 (14)	C8—C9	1.3964 (17)
N2—C2	1.4587 (14)	C9—C10	1.3824 (16)
N2—C3	1.4692 (14)	C9—H9	0.9500
C1—C2	1.5205 (16)	C10—H10	0.9500

C1—H1C	0.9900	C11—H11A	0.9800
C1—H1D	0.9900	C11—H11B	0.9800
C2—H2A	0.9900	C11—H11C	0.9800
C2—H2B	0.9900	F1—C13	1.3333 (14)
C3—C4	1.5106 (15)	F2—C13	1.3306 (14)
C3—H3A	0.9900	F3—C13	1.3343 (15)
C3—H3B	0.9900	O2—C12	1.2399 (14)
C4—H4A	0.9900	O3—C12	1.2356 (14)
C4—H4B	0.9900	C12—C13	1.5445 (16)
C8—O1—C11	116.87 (9)	C6—C5—C10	118.42 (10)
C1—N1—C4	111.25 (9)	C6—C5—N2	123.69 (10)
C1—N1—H1A	108.6 (10)	C10—C5—N2	117.9 (1)
C4—N1—H1A	110 (1)	C5—C6—C7	121.28 (11)
C1—N1—H1B	108.1 (9)	C5—C6—H6	119.4
C4—N1—H1B	111.9 (9)	C7—C6—H6	119.4
H1A—N1—H1B	106.9 (13)	C8—C7—C6	119.65 (11)
C5—N2—C2	115.95 (9)	C8—C7—H7	120.2
C5—N2—C3	112.00 (9)	C6—C7—H7	120.2
C2—N2—C3	109.51 (9)	O1—C8—C7	125.08 (11)
N1—C1—C2	110.07 (9)	O1—C8—C9	115.34 (10)
N1—C1—H1C	109.6	C7—C8—C9	119.57 (10)
C2—C1—H1C	109.6	C10—C9—C8	120.46 (11)
N1—C1—H1D	109.6	C10—C9—H9	119.8
C2—C1—H1D	109.6	C8—C9—H9	119.8
H1C—C1—H1D	108.2	C9—C10—C5	120.61 (11)
N2—C2—C1	109.59 (9)	C9—C10—H10	119.7
N2—C2—H2A	109.8	C5—C10—H10	119.7
C1—C2—H2A	109.8	O1—C11—H11A	109.5
N2—C2—H2B	109.8	O1—C11—H11B	109.5
C1—C2—H2B	109.8	H11A—C11—H11B	109.5
H2A—C2—H2B	108.2	O1—C11—H11C	109.5
N2—C3—C4	110.90 (9)	H11A—C11—H11C	109.5
N2—C3—H3A	109.5	H11B—C11—H11C	109.5
C4—C3—H3A	109.5	O3—C12—O2	129.27 (11)
N2—C3—H3B	109.5	O3—C12—C13	116.19 (10)
C4—C3—H3B	109.5	O2—C12—C13	114.45 (10)
H3A—C3—H3B	108.0	F2—C13—F1	106.79 (10)
N1—C4—C3	109.93 (9)	F2—C13—F3	107.26 (11)
N1—C4—H4A	109.7	F1—C13—F3	106.81 (11)
C3—C4—H4A	109.7	F2—C13—C12	113.14 (10)
N1—C4—H4B	109.7	F1—C13—C12	113.32 (9)
C3—C4—H4B	109.7	F3—C13—C12	109.16 (10)
H4A—C4—H4B	108.2		
C4—N1—C1—C2	−55.67 (12)	C11—O1—C8—C7	2.48 (16)
C5—N2—C2—C1	170.74 (9)	C11—O1—C8—C9	−178.21 (10)
C3—N2—C2—C1	−61.35 (12)	C6—C7—C8—O1	−179.82 (11)

N1—C1—C2—N2	58.98 (12)	C6—C7—C8—C9	0.89 (18)
C5—N2—C3—C4	−168.97 (9)	O1—C8—C9—C10	−179.47 (10)
C2—N2—C3—C4	60.94 (12)	C7—C8—C9—C10	−0.12 (18)
C1—N1—C4—C3	54.44 (12)	C8—C9—C10—C5	−0.80 (18)
N2—C3—C4—N1	−56.90 (12)	C6—C5—C10—C9	0.91 (18)
C2—N2—C5—C6	12.65 (16)	N2—C5—C10—C9	−179.22 (11)
C3—N2—C5—C6	−114.02 (12)	O3—C12—C13—F2	151.49 (11)
C2—N2—C5—C10	−167.21 (10)	O2—C12—C13—F2	−31.49 (15)
C3—N2—C5—C10	66.12 (13)	O3—C12—C13—F1	29.73 (16)
C10—C5—C6—C7	−0.13 (18)	O2—C12—C13—F1	−153.24 (11)
N2—C5—C6—C7	−179.99 (11)	O3—C12—C13—F3	−89.16 (13)
C5—C6—C7—C8	−0.78 (18)	O2—C12—C13—F3	87.86 (13)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯O2	0.937 (16)	1.777 (16)	2.7084 (13)	172.1 (15)
N1—H1B⋯O3[i]	0.901 (16)	1.982 (16)	2.8329 (14)	156.7 (13)
C3—H3B⋯O1[ii]	0.99	2.55	3.2230 (14)	125
C4—H4B⋯F1[iii]	0.99	2.62	3.3162 (14)	127
C4—H4B⋯O2[iv]	0.99	2.51	3.1709 (14)	124
C11—H11A⋯F3[v]	0.98	2.59	3.2796 (15)	128

Symmetry codes: (i) x, y+1, z; (ii) −x+1, y−1/2, −z+3/2; (iii) −x+1, −y, −z+1; (iv) −x+1, −y+1, −z+1; (v) −x+3/2, −y+1, z+1/2.

4-(4-Methoxyphenyl)piperazin-1-ium 2,3,4,5,6-pentafluorobenzoate monohydrate (II)

Crystal data
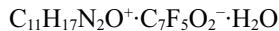
$C_{11}H_{17}N_2O^+·C_7F_5O_2^-·H_2O$	$F(000) = 872$
$M_r = 422.35$	$D_x = 1.519$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 16.9733$ (7) Å	Cell parameters from 9821 reflections
$b = 8.3512$ (4) Å	$\theta = 2.9$–27.5°
$c = 13.2980$ (4) Å	$\mu = 0.14$ mm^{-1}
$\beta = 101.494$ (1)°	$T = 90$ K
$V = 1847.16$ (13) Å^3	Colourless block, colourless
$Z = 4$	$0.30 \times 0.29 \times 0.14$ mm

Data collection

Bruker D8 Venture dual source diffractometer	30724 measured reflections
Radiation source: microsource	4238 independent reflections
Detector resolution: 7.41 pixels mm^{-1}	3793 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.031$
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)	$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
	$h = -22 \rightarrow 22$
	$k = -10 \rightarrow 10$
$T_{min} = 0.919$, $T_{max} = 0.971$	$l = -16 \rightarrow 17$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent
$R[F^2 > 2\sigma(F^2)] = 0.031$	and constrained refinement
$wR(F^2) = 0.083$	$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.7353P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
4238 reflections	$(\Delta/\sigma)_{max} = 0.001$
280 parameters	$\Delta\rho_{max} = 0.34$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.18$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL-2019/2 (Sheldrick 2015b),
Secondary atom site location: difference Fourier map	$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.0073 (14)

Special details

Experimental. The crystal was mounted using polyisobutene oil on the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder. It was placed directly into the cold gas stream of a liquid-nitrogen based cryostat (Hope, 1994; Parkin & Hope, 1998).
Diffraction data were collected with the crystal at 90K, which is standard practice in this laboratory for the majority of flash-cooled crystals.

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.

Refinement. Refinement progress was checked using *Platon* (Spek, 2020) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

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

	x	y	z	$U_{iso}*/U_{eq}$
N1	0.58113 (6)	0.83766 (11)	0.41244 (7)	0.01666 (19)
H1A	0.5398 (10)	0.8859 (19)	0.3686 (12)	0.031 (4)*
H1B	0.5880 (8)	0.8844 (17)	0.4730 (11)	0.022 (3)*
N2	0.70053 (5)	0.59211 (11)	0.44294 (7)	0.01513 (19)
C1	0.65689 (6)	0.85334 (13)	0.37269 (8)	0.0179 (2)
H1C	0.672233	0.967644	0.371987	0.021*
H1D	0.648215	0.812662	0.301405	0.021*
C2	0.72406 (6)	0.75953 (13)	0.43956 (8)	0.0167 (2)
H2A	0.773654	0.767857	0.411334	0.020*
H2B	0.735304	0.804590	0.509829	0.020*
C3	0.62849 (6)	0.57710 (13)	0.48786 (8)	0.0169 (2)
H3A	0.639553	0.621541	0.558294	0.020*
H3B	0.614304	0.462636	0.492007	0.020*
C4	0.55894 (6)	0.66628 (13)	0.42271 (8)	0.0180 (2)
H4A	0.545082	0.616403	0.353911	0.022*
H4B	0.511236	0.659600	0.454907	0.022*
C5	0.76277 (6)	0.48023 (13)	0.48036 (8)	0.0148 (2)
C6	0.84193 (6)	0.52426 (13)	0.51938 (8)	0.0177 (2)
H6	0.855236	0.634658	0.527225	0.021*
C7	0.90243 (6)	0.40977 (14)	0.54733 (8)	0.0188 (2)
H7	0.956319	0.442453	0.572825	0.023*

C8	0.88343 (6)	0.24844 (13)	0.53767 (8)	0.0172 (2)
C9	0.80383 (6)	0.20298 (13)	0.50119 (8)	0.0167 (2)
H9	0.790398	0.092463	0.495695	0.020*
C10	0.74434 (6)	0.31594 (13)	0.47297 (8)	0.0161 (2)
H10	0.690445	0.282547	0.448333	0.019*
O1	0.93660 (5)	0.1247 (1)	0.56258 (7)	0.02279 (19)
C11	1.01917 (7)	0.16511 (16)	0.59700 (11)	0.0303 (3)
H11A	1.051593	0.067127	0.605706	0.045*
H11B	1.025929	0.221583	0.662767	0.045*
H11C	1.036751	0.234384	0.546209	0.045*
F1	0.24103 (4)	1.14509 (8)	0.27860 (5)	0.02317 (16)
O2	0.43941 (5)	0.89873 (9)	0.27760 (6)	0.01884 (17)
O3	0.39764 (5)	1.07117 (10)	0.38371 (6)	0.02156 (18)
C12	0.38699 (6)	0.95869 (13)	0.32036 (8)	0.0156 (2)
C13	0.30287 (6)	0.88933 (13)	0.29215 (8)	0.0154 (2)
C14	0.23480 (7)	0.98503 (13)	0.27223 (8)	0.0177 (2)
C15	0.15862 (7)	0.92190 (15)	0.24210 (8)	0.0209 (2)
C16	0.14848 (7)	0.75875 (15)	0.23275 (8)	0.0216 (2)
C17	0.21451 (7)	0.65934 (14)	0.25387 (8)	0.0197 (2)
C18	0.29016 (6)	0.72513 (13)	0.28256 (8)	0.0167 (2)
F2	0.09439 (4)	1.01803 (10)	0.22123 (6)	0.02973 (18)
F3	0.07470 (4)	0.69754 (10)	0.20350 (6)	0.03065 (18)
F4	0.20518 (4)	0.50048 (8)	0.24420 (6)	0.02718 (17)
F5	0.35283 (4)	0.62368 (8)	0.30265 (5)	0.02060 (15)
O1W	0.55228 (5)	1.20201 (11)	0.39344 (7)	0.02363 (19)
H1W	0.5037 (11)	1.169 (2)	0.3840 (13)	0.044 (5)*
H2W	0.5556 (10)	1.258 (2)	0.3366 (14)	0.041 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0179 (4)	0.0149 (5)	0.0171 (4)	0.0024 (3)	0.0031 (3)	−0.0009 (4)
N2	0.0149 (4)	0.0126 (4)	0.0183 (4)	0.0000 (3)	0.0044 (3)	0.0013 (3)
C1	0.0185 (5)	0.0161 (5)	0.0196 (5)	0.0011 (4)	0.0054 (4)	0.0026 (4)
C2	0.0167 (5)	0.0139 (5)	0.0196 (5)	−0.0015 (4)	0.0037 (4)	0.0011 (4)
C3	0.0175 (5)	0.0156 (5)	0.0191 (5)	0.0003 (4)	0.0068 (4)	0.0010 (4)
C4	0.0171 (5)	0.0154 (5)	0.0220 (5)	−0.0008 (4)	0.0053 (4)	−0.0015 (4)
C5	0.0170 (5)	0.0152 (5)	0.0129 (4)	0.0009 (4)	0.0044 (4)	0.0010 (4)
C6	0.0181 (5)	0.0142 (5)	0.0208 (5)	−0.0013 (4)	0.0042 (4)	0.0009 (4)
C7	0.0155 (5)	0.0187 (5)	0.0219 (5)	−0.0014 (4)	0.0026 (4)	0.0009 (4)
C8	0.0192 (5)	0.0167 (5)	0.0162 (5)	0.0035 (4)	0.0048 (4)	0.0018 (4)
C9	0.0219 (5)	0.0133 (5)	0.0148 (5)	−0.0012 (4)	0.0038 (4)	−0.0007 (4)
C10	0.0175 (5)	0.0165 (5)	0.0142 (5)	−0.0013 (4)	0.0032 (4)	−0.0009 (4)
O1	0.0184 (4)	0.0174 (4)	0.0313 (4)	0.0037 (3)	0.0018 (3)	0.0020 (3)
C11	0.0179 (6)	0.0245 (6)	0.0469 (8)	0.0051 (5)	0.0026 (5)	0.0027 (6)
F1	0.0273 (4)	0.0147 (3)	0.0285 (4)	0.0039 (3)	0.0082 (3)	0.0034 (3)
O2	0.0170 (4)	0.0195 (4)	0.0199 (4)	0.0015 (3)	0.0034 (3)	−0.0017 (3)
O3	0.0245 (4)	0.0205 (4)	0.0205 (4)	−0.0054 (3)	0.0062 (3)	−0.0059 (3)

C12	0.0187 (5)	0.0140 (5)	0.0139 (5)	−0.0003 (4)	0.0025 (4)	0.0026 (4)
C13	0.0187 (5)	0.0161 (5)	0.0117 (4)	−0.0004 (4)	0.0037 (4)	0.0007 (4)
C14	0.0228 (5)	0.0154 (5)	0.0156 (5)	0.0004 (4)	0.0057 (4)	0.0020 (4)
C15	0.0180 (5)	0.0270 (6)	0.0179 (5)	0.0041 (4)	0.0040 (4)	0.0034 (4)
C16	0.0183 (5)	0.0306 (6)	0.0160 (5)	−0.0073 (5)	0.0035 (4)	−0.0019 (4)
C17	0.0264 (6)	0.0177 (5)	0.0161 (5)	−0.0063 (4)	0.0070 (4)	−0.0030 (4)
C18	0.0209 (5)	0.0173 (5)	0.0126 (5)	0.0016 (4)	0.0048 (4)	0.0004 (4)
F2	0.0194 (3)	0.0367 (4)	0.0325 (4)	0.0090 (3)	0.0037 (3)	0.0066 (3)
F3	0.0199 (3)	0.0418 (5)	0.0297 (4)	−0.0113 (3)	0.0036 (3)	−0.0063 (3)
F4	0.0343 (4)	0.0184 (4)	0.0309 (4)	−0.0098 (3)	0.0116 (3)	−0.0072 (3)
F5	0.0246 (3)	0.0146 (3)	0.0230 (3)	0.0033 (3)	0.0056 (3)	0.0009 (2)
O1W	0.0209 (4)	0.0256 (5)	0.0233 (4)	−0.0050 (3)	0.0019 (3)	0.0035 (3)

Geometric parameters (Å, º)

N1—C1	1.4903 (14)	C8—C9	1.3941 (15)
N1—C4	1.4932 (14)	C9—C10	1.3782 (15)
N1—H1A	0.912 (16)	C9—H9	0.9500
N1—H1B	0.882 (15)	C10—H10	0.9500
N2—C5	1.4237 (13)	O1—C11	1.4254 (14)
N2—C2	1.4573 (13)	C11—H11A	0.9800
N2—C3	1.4693 (13)	C11—H11B	0.9800
C1—C2	1.5167 (15)	C11—H11C	0.9800
C1—H1C	0.9900	F1—C14	1.3422 (13)
C1—H1D	0.9900	O2—C12	1.2519 (13)
C2—H2A	0.9900	O3—C12	1.2506 (13)
C2—H2B	0.9900	C12—C13	1.5169 (15)
C3—C4	1.5137 (15)	C13—C14	1.3863 (15)
C3—H3A	0.9900	C13—C18	1.3900 (15)
C3—H3B	0.9900	C14—C15	1.3798 (16)
C4—H4A	0.9900	C15—F2	1.3377 (13)
C4—H4B	0.9900	C15—C16	1.3758 (17)
C5—C6	1.3900 (15)	C16—F3	1.3366 (13)
C5—C10	1.4064 (15)	C16—C17	1.3780 (17)
C6—C7	1.3981 (15)	C17—F4	1.3392 (13)
C6—H6	0.9500	C17—C18	1.3788 (15)
C7—C8	1.3853 (16)	C18—F5	1.3444 (12)
C7—H7	0.9500	O1W—H1W	0.854 (19)
C8—O1	1.3686 (13)	O1W—H2W	0.899 (18)
C1—N1—C4	111.58 (8)	C8—C7—H7	120.2
C1—N1—H1A	110.1 (10)	C6—C7—H7	120.2
C4—N1—H1A	107.8 (10)	O1—C8—C7	125.57 (10)
C1—N1—H1B	108.6 (9)	O1—C8—C9	115.16 (10)
C4—N1—H1B	109.3 (9)	C7—C8—C9	119.25 (10)
H1A—N1—H1B	109.5 (13)	C10—C9—C8	121.0 (1)
C5—N2—C2	116.85 (8)	C10—C9—H9	119.5
C5—N2—C3	114.93 (8)	C8—C9—H9	119.5

C2—N2—C3	110.47 (8)	C9—C10—C5	120.54 (10)
N1—C1—C2	110.35 (9)	C9—C10—H10	119.7
N1—C1—H1C	109.6	C5—C10—H10	119.7
C2—C1—H1C	109.6	C8—O1—C11	117.24 (9)
N1—C1—H1D	109.6	O1—C11—H11A	109.5
C2—C1—H1D	109.6	O1—C11—H11B	109.5
H1C—C1—H1D	108.1	H11A—C11—H11B	109.5
N2—C2—C1	109.57 (9)	O1—C11—H11C	109.5
N2—C2—H2A	109.8	H11A—C11—H11C	109.5
C1—C2—H2A	109.8	H11B—C11—H11C	109.5
N2—C2—H2B	109.8	O3—C12—O2	125.87 (10)
C1—C2—H2B	109.8	O3—C12—C13	117.13 (9)
H2A—C2—H2B	108.2	O2—C12—C13	117.00 (9)
N2—C3—C4	110.04 (8)	C14—C13—C18	116.3 (1)
N2—C3—H3A	109.7	C14—C13—C12	122.31 (10)
C4—C3—H3A	109.7	C18—C13—C12	121.37 (10)
N2—C3—H3B	109.7	F1—C14—C15	117.15 (10)
C4—C3—H3B	109.7	F1—C14—C13	120.66 (10)
H3A—C3—H3B	108.2	C15—C14—C13	122.16 (10)
N1—C4—C3	109.99 (9)	F2—C15—C16	119.56 (10)
N1—C4—H4A	109.7	F2—C15—C14	120.58 (11)
C3—C4—H4A	109.7	C16—C15—C14	119.86 (11)
N1—C4—H4B	109.7	F3—C16—C15	119.88 (11)
C3—C4—H4B	109.7	F3—C16—C17	120.37 (11)
H4A—C4—H4B	108.2	C15—C16—C17	119.74 (10)
C6—C5—C10	117.98 (10)	F4—C17—C16	120.1 (1)
C6—C5—N2	123.48 (10)	F4—C17—C18	120.47 (10)
C10—C5—N2	118.47 (9)	C16—C17—C18	119.41 (11)
C5—C6—C7	121.5 (1)	F5—C18—C17	117.4 (1)
C5—C6—H6	119.2	F5—C18—C13	120.09 (10)
C7—C6—H6	119.2	C17—C18—C13	122.51 (10)
C8—C7—C6	119.69 (10)	H1W—O1W—H2W	105.2 (15)
C4—N1—C1—C2	−54.78 (11)	O3—C12—C13—C18	−137.95 (11)
C5—N2—C2—C1	164.82 (8)	O2—C12—C13—C18	42.46 (14)
C3—N2—C2—C1	−61.29 (11)	C18—C13—C14—F1	−179.42 (9)
N1—C1—C2—N2	57.64 (11)	C12—C13—C14—F1	−0.87 (15)
C5—N2—C3—C4	−163.83 (9)	C18—C13—C14—C15	−1.53 (15)
C2—N2—C3—C4	61.33 (11)	C12—C13—C14—C15	177.02 (10)
C1—N1—C4—C3	54.36 (11)	F1—C14—C15—F2	−0.50 (15)
N2—C3—C4—N1	−56.94 (11)	C13—C14—C15—F2	−178.46 (9)
C2—N2—C5—C6	4.50 (14)	F1—C14—C15—C16	179.1 (1)
C3—N2—C5—C6	−127.38 (11)	C13—C14—C15—C16	1.14 (16)
C2—N2—C5—C10	−172.24 (9)	F2—C15—C16—F3	−0.35 (16)
C3—N2—C5—C10	55.87 (12)	C14—C15—C16—F3	−179.95 (9)
C10—C5—C6—C7	2.19 (15)	F2—C15—C16—C17	179.80 (9)
N2—C5—C6—C7	−174.57 (10)	C14—C15—C16—C17	0.20 (17)
C5—C6—C7—C8	−1.00 (16)	F3—C16—C17—F4	0.66 (16)

C6—C7—C8—O1	−179.7 (1)	C15—C16—C17—F4	−179.49 (10)
C6—C7—C8—C9	−0.72 (16)	F3—C16—C17—C18	179.11 (9)
O1—C8—C9—C10	−179.72 (9)	C15—C16—C17—C18	−1.04 (16)
C7—C8—C9—C10	1.19 (16)	F4—C17—C18—F5	−1.69 (15)
C8—C9—C10—C5	0.04 (16)	C16—C17—C18—F5	179.86 (9)
C6—C5—C10—C9	−1.71 (15)	F4—C17—C18—C13	179.05 (10)
N2—C5—C10—C9	175.22 (9)	C16—C17—C18—C13	0.60 (16)
C7—C8—O1—C11	−3.02 (16)	C14—C13—C18—F5	−178.58 (9)
C9—C8—O1—C11	177.96 (10)	C12—C13—C18—F5	2.86 (15)
O3—C12—C13—C14	43.57 (14)	C14—C13—C18—C17	0.66 (15)
O2—C12—C13—C14	−136.02 (11)	C12—C13—C18—C17	−177.91 (9)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*A*···O2	0.912 (16)	1.886 (16)	2.7464 (12)	156.5 (14)
N1—H1*B*···O3^i	0.882 (15)	1.909 (15)	2.7692 (12)	164.9 (13)
C1—H1*C*···F5^{ii}	0.99	2.62	3.2261 (12)	119
C3—H3*B*···O1*W*^{iii}	0.99	2.65	3.5235 (14)	148
C4—H4*A*···O2^{iv}	0.99	2.57	3.4812 (13)	153
C10—H10···O1*W*^{iii}	0.95	2.41	3.3581 (14)	177
C11—H11*A*···F3^v	0.98	2.55	3.3961 (15)	144
C11—H11*B*···F2^{vi}	0.98	2.51	3.2436 (15)	131
O1*W*—H1*W*···O3	0.854 (19)	1.978 (19)	2.8222 (12)	170.0 (17)
O1*W*—H2*W*···O2^{ii}	0.899 (18)	1.936 (19)	2.8319 (12)	173.7 (16)

Symmetry codes: (i) −x+1, −y+2, −z+1; (ii) −x+1, y+1/2, −z+1/2; (iii) x, y−1, z; (iv) −x+1, y−1/2, −z+1/2; (v) x+1, −y+1/2, z+1/2; (vi) x+1, −y+3/2, z+1/2.

4-(4-Methoxyphenyl)piperazin-1-ium 4-iodobenzoate monohydrate (III)

Crystal data
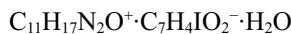
$C_{11}H_{17}N_2O^+ \cdot C_7H_4IO_2^- \cdot H_2O$
$M_r = 458.28$
Monoclinic, $P2_1/c$
$a = 20.4117$ (18) Å
$b = 7.4255$ (6) Å
$c = 36.796$ (3) Å
$β = 92.970$ (3)°
$V = 5569.6$ (8) Å³
$Z = 12$

$F(000) = 2760$
$D_x = 1.640$ Mg m⁻³
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9052 reflections
$\theta = 2.3–27.5°$
$\mu = 1.75$ mm⁻¹
$T = 90$ K
Plate, colourless
$0.30 \times 0.13 \times 0.03$ mm

Data collection

Bruker D8 Venture dual source
  diffractometer
Radiation source: microsource
Detector resolution: 7.41 pixels mm⁻¹
φ and ω scans
Absorption correction: multi-scan
  (*SADABS*; Krause *et al.*, 2015)
$T_{min} = 0.726$, $T_{max} = 0.862$

72181 measured reflections
12876 independent reflections
10003 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 27.7°$, $\theta_{min} = 2.0°$
$h = -26 \rightarrow 26$
$k = -9 \rightarrow 6$
$l = -47 \rightarrow 47$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent
$R[F^2 > 2\sigma(F^2)] = 0.033$	and constrained refinement
$wR(F^2) = 0.069$	$w = 1/[\sigma^2(F_o^2) + (0.0216P)^2 + 1.7628P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
12876 reflections	$(\Delta/\sigma)_{max} = 0.002$
729 parameters	$\Delta\rho_{max} = 0.84$ e Å^{-3}
12 restraints	$\Delta\rho_{min} = -0.80$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL-2019/2 (Sheldrick 2015b),
Secondary atom site location: difference Fourier map	$Fc^* = kFc[1 + 0.001 \times Fc^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.00017 (3)

Special details

Experimental. The crystal was mounted using polyisobutene oil on the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder. It was placed directly into the cold gas stream of a liquid-nitrogen based cryostat (Hope, 1994; Parkin & Hope, 1998).
Diffraction data were collected with the crystal at 90K, which is standard practice in this laboratory for the majority of flash-cooled crystals.

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.

Refinement. Refinement progress was checked using *Platon* (Spek, 2020) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

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

	x	y	z	U_{iso}*/U_{eq}
O1W	0.43969 (10)	0.2205 (3)	0.70712 (5)	0.0221 (4)
H1W1	0.4236 (15)	0.181 (4)	0.7238 (6)	0.043 (10)*
H2W1	0.4260 (15)	0.166 (4)	0.6906 (6)	0.043 (10)*
O2W	0.46521 (10)	0.3419 (3)	0.54225 (5)	0.0203 (4)
H1W2	0.4460 (12)	0.310 (4)	0.5585 (5)	0.024 (8)*
H2W2	0.4469 (14)	0.300 (4)	0.5253 (5)	0.046 (10)*
O3W	0.47153 (9)	0.4637 (3)	0.38524 (5)	0.0189 (4)
H1W3	0.4493 (13)	0.439 (4)	0.4008 (6)	0.039 (9)*
H2W3	0.4552 (13)	0.417 (4)	0.3683 (5)	0.031 (9)*
N1A	0.53728 (11)	0.1701 (3)	0.62582 (6)	0.0169 (5)
H1AA	0.4979 (16)	0.123 (4)	0.6303 (8)	0.039 (9)*
H1AB	0.5305 (15)	0.293 (4)	0.6212 (8)	0.036 (9)*
N2A	0.67722 (10)	0.1578 (3)	0.61970 (5)	0.0153 (5)
C1A	0.56652 (12)	0.0802 (3)	0.59424 (7)	0.0166 (6)
H1AC	0.536526	0.091693	0.572342	0.020*
H1AD	0.573059	−0.049571	0.599461	0.020*
C2A	0.63171 (12)	0.1678 (3)	0.58741 (6)	0.0155 (5)
H2AA	0.651615	0.106930	0.566729	0.019*
H2AB	0.624385	0.295573	0.580691	0.019*
C3A	0.64855 (13)	0.2351 (4)	0.65186 (6)	0.0173 (6)
H3A1	0.643210	0.366608	0.648468	0.021*

H3A2	0.678752	0.215151	0.673412	0.021*
C4A	0.58240 (13)	0.1516 (4)	0.65859 (7)	0.0190 (6)
H4AA	0.588228	0.022477	0.664660	0.023*
H4AB	0.563121	0.211922	0.679520	0.023*
C5A	0.74157 (13)	0.2193 (3)	0.61344 (6)	0.0150 (5)
C6A	0.77415 (13)	0.1533 (3)	0.58374 (7)	0.0175 (6)
H6A	0.752173	0.070253	0.567695	0.021*
C7A	0.83727 (13)	0.2051 (3)	0.57702 (7)	0.0186 (6)
H7A	0.857901	0.158525	0.556458	0.022*
C8A	0.87077 (13)	0.3250 (3)	0.60019 (7)	0.0172 (6)
C9A	0.83902 (13)	0.3916 (4)	0.62985 (7)	0.0187 (6)
H9A	0.861272	0.473954	0.645926	0.022*
C10A	0.77587 (13)	0.3404 (3)	0.63637 (7)	0.0181 (6)
H10A	0.755274	0.388262	0.656829	0.022*
O1A	0.93324 (9)	0.3865 (3)	0.59612 (5)	0.0246 (5)
C11A	0.96791 (14)	0.3085 (4)	0.56715 (7)	0.0277 (7)
H11A	1.012352	0.358505	0.567375	0.042*
H11B	0.944723	0.335895	0.543819	0.042*
H11C	0.970366	0.177628	0.570434	0.042*
I1A	0.07054 (2)	0.22414 (3)	0.65985 (2)	0.03241 (6)
O2A	0.41019 (9)	0.0741 (2)	0.64110 (4)	0.0182 (4)
O3A	0.38729 (10)	0.2563 (3)	0.59416 (5)	0.0327 (5)
C12A	0.37115 (13)	0.1708 (3)	0.62136 (7)	0.0177 (6)
C13A	0.30062 (13)	0.1839 (3)	0.63110 (6)	0.0155 (6)
C14A	0.25598 (14)	0.2798 (3)	0.60871 (7)	0.0202 (6)
H14A	0.270779	0.337633	0.587618	0.024*
C15A	0.19067 (14)	0.2927 (4)	0.61649 (7)	0.0235 (6)
H15A	0.160641	0.357675	0.600863	0.028*
C16A	0.16966 (13)	0.2086 (3)	0.64770 (7)	0.0189 (6)
C17A	0.21308 (13)	0.1131 (3)	0.67042 (7)	0.0185 (6)
H17A	0.198222	0.056099	0.691601	0.022*
C18A	0.27842 (13)	0.1008 (3)	0.66215 (6)	0.0150 (5)
H18A	0.308316	0.035272	0.677757	0.018*
N1B	0.54528 (11)	0.2872 (3)	0.46279 (6)	0.0157 (5)
H1BA	0.5047 (15)	0.247 (4)	0.4678 (8)	0.032 (9)*
H1BB	0.5413 (13)	0.410 (4)	0.4599 (7)	0.019 (7)*
N2B	0.68341 (10)	0.2390 (3)	0.45357 (5)	0.0145 (5)
C1B	0.57009 (12)	0.1948 (3)	0.43022 (6)	0.0156 (6)
H1BC	0.539379	0.215359	0.408906	0.019*
H1BD	0.572919	0.063524	0.434700	0.019*
C2B	0.63691 (12)	0.2675 (3)	0.42252 (6)	0.0151 (5)
H2BA	0.653183	0.206538	0.400822	0.018*
H2BB	0.633515	0.397882	0.417175	0.018*
C3B	0.66000 (12)	0.3265 (3)	0.48621 (6)	0.0161 (6)
H3B1	0.658596	0.458500	0.482408	0.019*
H3B2	0.691065	0.301479	0.507155	0.019*
C4B	0.59236 (13)	0.2603 (4)	0.49477 (6)	0.0173 (6)
H4BA	0.594522	0.130750	0.501113	0.021*

H4BB	0.576808	0.326780	0.515985	0.021*
C5B	0.75017 (12)	0.2699 (3)	0.44638 (6)	0.0134 (5)
C6B	0.77627 (13)	0.1969 (3)	0.41512 (7)	0.0166 (6)
H6B	0.747807	0.135704	0.398002	0.020*
C7B	0.84192 (13)	0.2109 (3)	0.40838 (7)	0.0181 (6)
H7B	0.857875	0.160425	0.386830	0.022*
C8B	0.88469 (13)	0.2985 (3)	0.43297 (7)	0.0185 (6)
C9B	0.85954 (13)	0.3744 (4)	0.46404 (7)	0.0208 (6)
H9B	0.888096	0.435936	0.481045	0.025*
C10B	0.79355 (13)	0.3610 (3)	0.47032 (7)	0.0182 (6)
H10B	0.777431	0.415177	0.491472	0.022*
O1B	0.95052 (9)	0.3195 (3)	0.42936 (5)	0.0280 (5)
C11B	0.97811 (14)	0.2421 (4)	0.39805 (8)	0.0303 (7)
H11D	1.025183	0.268615	0.398486	0.045*
H11E	0.956558	0.293296	0.375985	0.045*
H11F	0.971528	0.111410	0.398221	0.045*
I1B	0.07478 (2)	0.15453 (3)	0.49437 (2)	0.02872 (6)
O2B	0.41837 (9)	0.1894 (2)	0.47849 (5)	0.0198 (4)
O3B	0.38573 (10)	0.3743 (3)	0.43324 (5)	0.0298 (5)
C12B	0.37472 (13)	0.2739 (3)	0.45938 (7)	0.0178 (6)
C13B	0.30402 (13)	0.2499 (3)	0.46834 (6)	0.0145 (5)
C14B	0.25389 (13)	0.3216 (3)	0.44580 (7)	0.0179 (6)
H14B	0.264596	0.389536	0.425054	0.022*
C15B	0.18860 (14)	0.2958 (4)	0.45298 (7)	0.0201 (6)
H15B	0.154697	0.343937	0.437190	0.024*
C16B	0.17363 (13)	0.1985 (3)	0.48365 (7)	0.0186 (6)
C17B	0.22246 (13)	0.1289 (3)	0.50692 (7)	0.0186 (6)
H17B	0.211464	0.064335	0.528047	0.022*
C18B	0.28755 (13)	0.1538 (3)	0.49930 (6)	0.0163 (6)
H18B	0.321254	0.105354	0.515187	0.020*
N1C	0.54732 (11)	0.4096 (3)	0.30188 (6)	0.0151 (5)
H1CA	0.5039 (15)	0.382 (4)	0.3092 (8)	0.036 (9)*
H1CB	0.5487 (13)	0.519 (4)	0.2976 (7)	0.012 (7)*
N2C	0.67789 (10)	0.2903 (3)	0.28952 (5)	0.0140 (5)
C1C	0.56203 (13)	0.3023 (4)	0.26914 (7)	0.0183 (6)
H1CC	0.531549	0.337052	0.248527	0.022*
H1CD	0.555525	0.172679	0.274144	0.022*
C2C	0.63187 (12)	0.3341 (4)	0.25908 (6)	0.0168 (6)
H2CA	0.641494	0.258599	0.237828	0.020*
H2CB	0.637343	0.461852	0.252166	0.020*
C3C	0.66487 (13)	0.3992 (4)	0.32146 (6)	0.0164 (6)
H3C1	0.671354	0.528155	0.315826	0.020*
H3C2	0.696146	0.366146	0.341838	0.020*
C4C	0.59550 (12)	0.3697 (4)	0.33264 (6)	0.0164 (6)
H4CA	0.590283	0.243214	0.340487	0.020*
H4CB	0.586798	0.448762	0.353511	0.020*
C5C	0.74472 (12)	0.2758 (3)	0.28062 (6)	0.0141 (5)
C6C	0.76069 (13)	0.1706 (3)	0.25095 (7)	0.0177 (6)

H6C	0.726496	0.118494	0.235930	0.021*
C7C	0.82550 (13)	0.1404 (4)	0.24290 (7)	0.0186 (6)
H7C	0.835037	0.070293	0.222230	0.022*
C8C	0.87656 (13)	0.2121 (4)	0.26487 (7)	0.0196 (6)
C9C	0.86090 (13)	0.3184 (4)	0.29422 (7)	0.0204 (6)
H9C	0.895187	0.369408	0.309346	0.025*
C10C	0.79633 (13)	0.3517 (3)	0.30189 (7)	0.0177 (6)
H10C	0.786977	0.427006	0.321839	0.021*
O1C	0.94180 (9)	0.1861 (3)	0.26005 (5)	0.0250 (4)
C11C	0.95839 (14)	0.0599 (4)	0.23304 (8)	0.0314 (7)
H11G	1.006183	0.046967	0.233207	0.047*
H11H	0.941860	0.102414	0.209073	0.047*
H11I	0.938558	−0.056985	0.238193	0.047*
I1C	0.09042 (2)	0.06781 (3)	0.31910 (2)	0.03020 (6)
O2C	0.42707 (9)	0.3016 (2)	0.32290 (5)	0.0202 (4)
O3C	0.39400 (9)	0.4141 (2)	0.26896 (5)	0.0235 (4)
C12C	0.38327 (13)	0.3315 (3)	0.29758 (7)	0.0167 (6)
C13C	0.31525 (13)	0.2627 (3)	0.30286 (7)	0.0155 (6)
C14C	0.27202 (13)	0.2325 (4)	0.27301 (7)	0.0192 (6)
H14C	0.286224	0.254050	0.249216	0.023*
C15C	0.20884 (14)	0.1719 (4)	0.27722 (7)	0.0212 (6)
H15C	0.180102	0.148483	0.256620	0.025*
C16C	0.18836 (13)	0.1459 (3)	0.31218 (7)	0.0198 (6)
C17C	0.23004 (13)	0.1737 (3)	0.34227 (7)	0.0204 (6)
H17C	0.215262	0.154375	0.366023	0.024*
C18C	0.29359 (13)	0.2299 (3)	0.33759 (7)	0.0172 (6)
H18C	0.322853	0.246385	0.358253	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1W	0.0265 (12)	0.0229 (11)	0.0167 (10)	−0.0040 (9)	−0.0009 (9)	−0.0004 (9)
O2W	0.0214 (11)	0.0235 (11)	0.0165 (10)	−0.0031 (9)	0.0052 (9)	0.0010 (9)
O3W	0.0203 (11)	0.0206 (11)	0.0162 (10)	−0.0003 (9)	0.0046 (9)	−0.0003 (9)
N1A	0.0159 (13)	0.0171 (12)	0.0181 (11)	−0.0011 (10)	0.0039 (9)	0.0005 (10)
N2A	0.0131 (11)	0.0181 (11)	0.0149 (10)	−0.0004 (9)	0.0023 (9)	−0.0008 (9)
C1A	0.0151 (14)	0.0154 (13)	0.0195 (13)	0.0004 (11)	0.0023 (11)	−0.0022 (11)
C2A	0.0143 (14)	0.0174 (13)	0.0148 (12)	−0.0001 (11)	0.0021 (10)	−0.0003 (11)
C3A	0.0176 (14)	0.0226 (14)	0.0118 (12)	0.0011 (12)	0.0027 (10)	−0.0008 (11)
C4A	0.0172 (14)	0.0234 (15)	0.0169 (13)	0.0007 (12)	0.0045 (11)	0.0036 (11)
C5A	0.0144 (14)	0.0156 (13)	0.0151 (12)	0.0021 (11)	0.002 (1)	0.002 (1)
C6A	0.0169 (14)	0.0176 (14)	0.0178 (13)	−0.0018 (11)	0.0005 (11)	−0.0041 (11)
C7A	0.0190 (15)	0.0186 (14)	0.0184 (13)	−0.0003 (12)	0.0037 (11)	−0.0044 (11)
C8A	0.0135 (14)	0.0154 (13)	0.0227 (13)	0.0011 (11)	0.0023 (11)	0.0019 (11)
C9A	0.0188 (15)	0.0187 (14)	0.0184 (13)	−0.0014 (12)	−0.0022 (11)	−0.0046 (11)
C10A	0.0192 (15)	0.0209 (14)	0.0144 (12)	0.0023 (12)	0.0023 (11)	−0.0048 (11)
O1A	0.0158 (10)	0.0288 (11)	0.0298 (11)	−0.0064 (9)	0.0071 (8)	−0.0099 (9)
C11A	0.0202 (16)	0.0354 (18)	0.0285 (15)	−0.0036 (14)	0.0106 (13)	−0.0057 (13)

I1A	0.01674 (11)	0.02892 (11)	0.05171 (13)	0.00099 (9)	0.00330 (9)	−0.01156 (10)
O2A	0.0175 (10)	0.0203 (10)	0.0172 (9)	−0.0023 (8)	0.0037 (8)	−0.0022 (8)
O3A	0.0343 (13)	0.0381 (13)	0.0272 (11)	−0.0018 (10)	0.0153 (10)	0.0115 (9)
C12A	0.0219 (15)	0.0157 (13)	0.0161 (13)	−0.0048 (12)	0.0049 (11)	−0.0050 (11)
C13A	0.0187 (14)	0.0117 (13)	0.0163 (12)	−0.0049 (11)	0.0036 (11)	−0.0033 (10)
C14A	0.0309 (17)	0.0133 (13)	0.0165 (13)	−0.0043 (12)	0.0016 (12)	0.0018 (11)
C15A	0.0240 (16)	0.0169 (14)	0.0289 (15)	0.0013 (12)	−0.0063 (13)	−0.0009 (12)
C16A	0.0144 (14)	0.0157 (13)	0.0266 (14)	−0.0033 (11)	0.0008 (11)	−0.0078 (11)
C17A	0.0194 (15)	0.0163 (13)	0.0202 (13)	−0.0021 (12)	0.0054 (11)	0.0001 (11)
C18A	0.0183 (14)	0.0118 (12)	0.0150 (12)	−0.0017 (11)	0.0014 (10)	−0.0003 (10)
N1B	0.0149 (12)	0.0150 (12)	0.0173 (11)	−0.0009 (10)	0.0026 (9)	−0.0006 (9)
N2B	0.0153 (12)	0.0160 (11)	0.0122 (10)	−0.0009 (9)	0.0012 (9)	−0.0021 (9)
C1B	0.0146 (14)	0.0169 (13)	0.0156 (12)	−0.0003 (11)	0.0018 (10)	−0.0011 (10)
C2B	0.0160 (14)	0.0181 (13)	0.0112 (12)	0.0007 (11)	0.0017 (10)	−0.0018 (10)
C3B	0.0172 (14)	0.0200 (14)	0.0113 (12)	−0.0013 (11)	0.0015 (10)	−0.002 (1)
C4B	0.0186 (14)	0.0197 (14)	0.0137 (12)	−0.0005 (12)	0.0018 (11)	0.0003 (11)
C5B	0.0164 (14)	0.0098 (12)	0.0137 (12)	0.0006 (11)	−0.0013 (10)	0.0015 (10)
C6B	0.0187 (14)	0.0145 (13)	0.0163 (13)	−0.0023 (11)	−0.0005 (11)	−0.0029 (10)
C7B	0.0201 (15)	0.0158 (13)	0.0186 (13)	−0.0015 (12)	0.0026 (11)	−0.0044 (11)
C8B	0.0141 (14)	0.0169 (14)	0.0247 (14)	0.0006 (11)	0.0020 (11)	0.0000 (11)
C9B	0.0206 (15)	0.0222 (15)	0.0193 (13)	−0.0040 (12)	−0.0032 (11)	−0.0065 (11)
C10B	0.0218 (15)	0.0182 (14)	0.0147 (12)	0.0024 (12)	0.0019 (11)	−0.0032 (11)
O1B	0.0137 (10)	0.0354 (12)	0.0351 (11)	−0.0036 (9)	0.0042 (9)	−0.0124 (9)
C11B	0.0209 (16)	0.0357 (18)	0.0352 (17)	−0.0038 (14)	0.0105 (13)	−0.0099 (14)
I1B	0.01675 (10)	0.02811 (11)	0.04205 (12)	−0.00272 (8)	0.00875 (8)	−0.00998 (9)
O2B	0.0172 (10)	0.0225 (10)	0.0201 (9)	−0.0013 (8)	0.0046 (8)	−0.0032 (8)
O3B	0.0288 (12)	0.0302 (12)	0.0318 (11)	−0.0017 (10)	0.0138 (9)	0.0118 (9)
C12B	0.0227 (15)	0.0143 (13)	0.0168 (13)	−0.0043 (12)	0.0061 (11)	−0.0069 (11)
C13B	0.0195 (14)	0.0099 (12)	0.0142 (12)	−0.0023 (11)	0.0042 (11)	−0.0035 (10)
C14B	0.0244 (15)	0.0155 (13)	0.0139 (12)	−0.0051 (12)	0.0003 (11)	−0.0001 (10)
C15B	0.0212 (15)	0.0175 (14)	0.0212 (14)	−0.0001 (12)	−0.0036 (11)	−0.0016 (11)
C16B	0.0151 (14)	0.0159 (13)	0.0254 (14)	−0.0006 (11)	0.0063 (11)	−0.0054 (11)
C17B	0.0226 (15)	0.0139 (13)	0.0200 (13)	−0.0004 (12)	0.0078 (11)	0.0017 (11)
C18B	0.0193 (14)	0.0153 (13)	0.0143 (12)	0.0017 (11)	0.0020 (11)	0.0010 (11)
N1C	0.0162 (13)	0.0145 (12)	0.0147 (11)	−0.0014 (10)	0.0001 (9)	0.0002 (9)
N2C	0.0143 (11)	0.0166 (11)	0.0111 (10)	0.0006 (9)	0.0010 (9)	−0.0015 (8)
C1C	0.0172 (14)	0.0209 (14)	0.0169 (13)	0.0001 (12)	0.0005 (11)	−0.0040 (11)
C2C	0.0158 (14)	0.0212 (14)	0.0132 (12)	0.0010 (11)	−0.0005 (10)	−0.0002 (11)
C3C	0.0177 (14)	0.0198 (14)	0.0116 (12)	0.0000 (11)	0.0003 (10)	−0.0012 (10)
C4C	0.0195 (14)	0.0181 (14)	0.0116 (12)	0.0016 (12)	0.0002 (10)	0.0009 (10)
C5C	0.0149 (14)	0.0127 (12)	0.0146 (12)	−0.0019 (11)	0.0001 (10)	0.0027 (10)
C6C	0.0163 (14)	0.0201 (14)	0.0166 (13)	−0.0025 (12)	−0.0015 (11)	−0.0024 (11)
C7C	0.0173 (14)	0.0208 (14)	0.0182 (13)	0.0001 (12)	0.0038 (11)	−0.0027 (11)
C8C	0.0181 (15)	0.0165 (13)	0.0245 (14)	−0.0009 (12)	0.0045 (12)	0.0057 (11)
C9C	0.0190 (15)	0.0197 (14)	0.0222 (14)	−0.0047 (12)	−0.0031 (11)	−0.0014 (11)
C10C	0.0213 (15)	0.0135 (13)	0.0183 (13)	−0.0012 (12)	0.0006 (11)	−0.0013 (11)
O1C	0.0143 (10)	0.0282 (11)	0.0330 (11)	−0.0004 (9)	0.0054 (8)	−0.0037 (9)
C11C	0.0221 (17)	0.0376 (18)	0.0352 (17)	0.0049 (14)	0.0083 (13)	−0.0043 (15)

I1C	0.01743 (10)	0.02945 (11)	0.04390 (12)	−0.00327 (8)	0.00347 (8)	−0.00136 (9)
O2C	0.0165 (10)	0.0249 (11)	0.0194 (9)	−0.0022 (8)	0.0017 (8)	0.0013 (8)
O3C	0.0266 (11)	0.0239 (10)	0.0208 (10)	0.0009 (9)	0.0075 (8)	0.0070 (8)
C12C	0.0204 (15)	0.0119 (13)	0.0184 (13)	0.0016 (11)	0.0061 (11)	−0.0043 (11)
C13C	0.0203 (15)	0.0090 (12)	0.0175 (13)	0.0026 (11)	0.0026 (11)	−0.0012 (10)
C14C	0.0201 (15)	0.0206 (14)	0.0172 (13)	0.0042 (12)	0.0030 (11)	−0.0018 (11)
C15C	0.0216 (15)	0.0200 (14)	0.0215 (14)	0.0033 (12)	−0.0030 (12)	−0.0060 (12)
C16C	0.0134 (14)	0.0149 (13)	0.0311 (15)	0.0027 (11)	0.0031 (12)	−0.0010 (12)
C17C	0.0224 (15)	0.0179 (14)	0.0213 (13)	0.0026 (12)	0.0050 (12)	0.0033 (11)
C18C	0.0188 (15)	0.0160 (13)	0.0168 (13)	0.0009 (11)	0.0001 (11)	0.0013 (11)

Geometric parameters (Å, º)

O1W—H1W1	0.769 (15)	C6B—H6B	0.9500
O1W—H2W1	0.768 (15)	C7B—C8B	1.386 (4)
O2W—H1W2	0.770 (14)	C7B—H7B	0.9500
O2W—H2W2	0.774 (15)	C8B—O1B	1.366 (3)
O3W—H1W3	0.771 (15)	C8B—C9B	1.396 (3)
O3W—H2W3	0.775 (14)	C9B—C10B	1.382 (4)
N1A—C4A	1.486 (3)	C9B—H9B	0.9500
N1A—C1A	1.491 (3)	C10B—H10B	0.9500
N1A—H1AA	0.90 (3)	O1B—C11B	1.429 (3)
N1A—H1AB	0.94 (3)	C11B—H11D	0.9800
N2A—C5A	1.421 (3)	C11B—H11E	0.9800
N2A—C3A	1.464 (3)	C11B—H11F	0.9800
N2A—C2A	1.472 (3)	I1B—C16B	2.101 (3)
C1A—C2A	1.514 (3)	O2B—C12B	1.272 (3)
C1A—H1AC	0.9900	O3B—C12B	1.246 (3)
C1A—H1AD	0.9900	C12B—C13B	1.507 (3)
C2A—H2AA	0.9900	C13B—C14B	1.389 (4)
C2A—H2AB	0.9900	C13B—C18B	1.400 (3)
C3A—C4A	1.518 (3)	C14B—C15B	1.385 (4)
C3A—H3A1	0.9900	C14B—H14B	0.9500
C3A—H3A2	0.9900	C15B—C16B	1.388 (4)
C4A—H4AA	0.9900	C15B—H15B	0.9500
C4A—H4AB	0.9900	C16B—C17B	1.380 (4)
C5A—C10A	1.396 (4)	C17B—C18B	1.384 (3)
C5A—C6A	1.397 (3)	C17B—H17B	0.9500
C6A—C7A	1.379 (3)	C18B—H18B	0.9500
C6A—H6A	0.9500	N1C—C1C	1.488 (3)
C7A—C8A	1.388 (4)	N1C—C4C	1.490 (3)
C7A—H7A	0.9500	N1C—H1CA	0.96 (3)
C8A—O1A	1.370 (3)	N1C—H1CB	0.83 (3)
C8A—C9A	1.389 (3)	N2C—C5C	1.424 (3)
C9A—C10A	1.377 (4)	N2C—C2C	1.461 (3)
C9A—H9A	0.9500	N2C—C3C	1.462 (3)
C10A—H10A	0.9500	C1C—C2C	1.510 (3)
O1A—C11A	1.432 (3)	C1C—H1CC	0.9900

C11A—H11A	0.9800	C1C—H1CD	0.9900
C11A—H11B	0.9800	C2C—H2CA	0.9900
C11A—H11C	0.9800	C2C—H2CB	0.9900
I1A—C16A	2.098 (3)	C3C—C4C	1.511 (3)
O2A—C12A	1.272 (3)	C3C—H3C1	0.9900
O3A—C12A	1.244 (3)	C3C—H3C2	0.9900
C12A—C13A	1.505 (3)	C4C—H4CA	0.9900
C13A—C14A	1.392 (4)	C4C—H4CB	0.9900
C13A—C18A	1.394 (3)	C5C—C6C	1.395 (3)
C14A—C15A	1.381 (4)	C5C—C10C	1.398 (4)
C14A—H14A	0.9500	C6C—C7C	1.389 (3)
C15A—C16A	1.394 (4)	C6C—H6C	0.9500
C15A—H15A	0.9500	C7C—C8C	1.392 (4)
C16A—C17A	1.383 (4)	C7C—H7C	0.9500
C17A—C18A	1.386 (3)	C8C—O1C	1.366 (3)
C17A—H17A	0.9500	C8C—C9C	1.388 (4)
C18A—H18A	0.9500	C9C—C10C	1.384 (4)
N1B—C1B	1.492 (3)	C9C—H9C	0.9500
N1B—C4B	1.494 (3)	C10C—H10C	0.9500
N1B—H1BA	0.91 (3)	O1C—C11C	1.420 (3)
N1B—H1BB	0.92 (3)	C11C—H11G	0.9800
N2B—C5B	1.420 (3)	C11C—H11H	0.9800
N2B—C2B	1.462 (3)	C11C—H11I	0.9800
N2B—C3B	1.467 (3)	I1C—C16C	2.110 (3)
C1B—C2B	1.507 (3)	O2C—C12C	1.277 (3)
C1B—H1BC	0.9900	O3C—C12C	1.248 (3)
C1B—H1BD	0.9900	C12C—C13C	1.501 (4)
C2B—H2BA	0.9900	C13C—C14C	1.391 (4)
C2B—H2BB	0.9900	C13C—C18C	1.395 (3)
C3B—C4B	1.514 (3)	C14C—C15C	1.382 (4)
C3B—H3B1	0.9900	C14C—H14C	0.9500
C3B—H3B2	0.9900	C15C—C16C	1.387 (3)
C4B—H4BA	0.9900	C15C—H15C	0.9500
C4B—H4BB	0.9900	C16C—C17C	1.376 (4)
C5B—C10B	1.392 (4)	C17C—C18C	1.382 (4)
C5B—C6B	1.402 (3)	C17C—H17C	0.9500
C6B—C7B	1.379 (3)	C18C—H18C	0.9500
H1W1—O1W—H2W1	106 (2)	C5B—C6B—H6B	118.9
H1W2—O2W—H2W2	105 (2)	C6B—C7B—C8B	120.2 (2)
H1W3—O3W—H2W3	104 (2)	C6B—C7B—H7B	119.9
C4A—N1A—C1A	109.6 (2)	C8B—C7B—H7B	119.9
C4A—N1A—H1AA	110 (2)	O1B—C8B—C7B	125.4 (2)
C1A—N1A—H1AA	111 (2)	O1B—C8B—C9B	116.0 (2)
C4A—N1A—H1AB	108.4 (19)	C7B—C8B—C9B	118.6 (2)
C1A—N1A—H1AB	110.9 (18)	C10B—C9B—C8B	120.7 (2)
H1AA—N1A—H1AB	107 (3)	C10B—C9B—H9B	119.6
C5A—N2A—C3A	114.6 (2)	C8B—C9B—H9B	119.6

C5A—N2A—C2A	113.78 (19)	C9B—C10B—C5B	121.5 (2)
C3A—N2A—C2A	111.8 (2)	C9B—C10B—H10B	119.2
N1A—C1A—C2A	109.1 (2)	C5B—C10B—H10B	119.2
N1A—C1A—H1AC	109.9	C8B—O1B—C11B	117.6 (2)
C2A—C1A—H1AC	109.9	O1B—C11B—H11D	109.5
N1A—C1A—H1AD	109.9	O1B—C11B—H11E	109.5
C2A—C1A—H1AD	109.9	H11D—C11B—H11E	109.5
H1AC—C1A—H1AD	108.3	O1B—C11B—H11F	109.5
N2A—C2A—C1A	111.6 (2)	H11D—C11B—H11F	109.5
N2A—C2A—H2AA	109.3	H11E—C11B—H11F	109.5
C1A—C2A—H2AA	109.3	O3B—C12B—O2B	124.9 (2)
N2A—C2A—H2AB	109.3	O3B—C12B—C13B	116.9 (2)
C1A—C2A—H2AB	109.3	O2B—C12B—C13B	118.1 (2)
H2AA—C2A—H2AB	108.0	C14B—C13B—C18B	118.7 (2)
N2A—C3A—C4A	111.6 (2)	C14B—C13B—C12B	120.5 (2)
N2A—C3A—H3A1	109.3	C18B—C13B—C12B	120.8 (2)
C4A—C3A—H3A1	109.3	C15B—C14B—C13B	121.3 (2)
N2A—C3A—H3A2	109.3	C15B—C14B—H14B	119.4
C4A—C3A—H3A2	109.3	C13B—C14B—H14B	119.4
H3A1—C3A—H3A2	108.0	C14B—C15B—C16B	118.8 (3)
N1A—C4A—C3A	110.4 (2)	C14B—C15B—H15B	120.6
N1A—C4A—H4AA	109.6	C16B—C15B—H15B	120.6
C3A—C4A—H4AA	109.6	C17B—C16B—C15B	121.1 (2)
N1A—C4A—H4AB	109.6	C17B—C16B—I1B	119.67 (18)
C3A—C4A—H4AB	109.6	C15B—C16B—I1B	119.2 (2)
H4AA—C4A—H4AB	108.1	C16B—C17B—C18B	119.6 (2)
C10A—C5A—C6A	117.0 (2)	C16B—C17B—H17B	120.2
C10A—C5A—N2A	123.2 (2)	C18B—C17B—H17B	120.2
C6A—C5A—N2A	119.7 (2)	C17B—C18B—C13B	120.4 (3)
C7A—C6A—C5A	122.0 (2)	C17B—C18B—H18B	119.8
C7A—C6A—H6A	119.0	C13B—C18B—H18B	119.8
C5A—C6A—H6A	119.0	C1C—N1C—C4C	110.8 (2)
C6A—C7A—C8A	120.3 (2)	C1C—N1C—H1CA	110.1 (18)
C6A—C7A—H7A	119.9	C4C—N1C—H1CA	109.0 (18)
C8A—C7A—H7A	119.9	C1C—N1C—H1CB	111.1 (17)
O1A—C8A—C7A	125.3 (2)	C4C—N1C—H1CB	108.2 (19)
O1A—C8A—C9A	116.2 (2)	H1CA—N1C—H1CB	108 (3)
C7A—C8A—C9A	118.4 (2)	C5C—N2C—C2C	115.22 (19)
C10A—C9A—C8A	121.2 (2)	C5C—N2C—C3C	116.3 (2)
C10A—C9A—H9A	119.4	C2C—N2C—C3C	110.9 (2)
C8A—C9A—H9A	119.4	N1C—C1C—C2C	110.3 (2)
C9A—C10A—C5A	121.1 (2)	N1C—C1C—H1CC	109.6
C9A—C10A—H10A	119.4	C2C—C1C—H1CC	109.6
C5A—C10A—H10A	119.4	N1C—C1C—H1CD	109.6
C8A—O1A—C11A	116.4 (2)	C2C—C1C—H1CD	109.6
O1A—C11A—H11A	109.5	H1CC—C1C—H1CD	108.1
O1A—C11A—H11B	109.5	N2C—C2C—C1C	110.8 (2)
H11A—C11A—H11B	109.5	N2C—C2C—H2CA	109.5

O1A—C11A—H11C	109.5	C1C—C2C—H2CA	109.5
H11A—C11A—H11C	109.5	N2C—C2C—H2CB	109.5
H11B—C11A—H11C	109.5	C1C—C2C—H2CB	109.5
O3A—C12A—O2A	124.3 (2)	H2CA—C2C—H2CB	108.1
O3A—C12A—C13A	117.1 (2)	N2C—C3C—C4C	110.5 (2)
O2A—C12A—C13A	118.6 (2)	N2C—C3C—H3C1	109.5
C14A—C13A—C18A	118.7 (2)	C4C—C3C—H3C1	109.5
C14A—C13A—C12A	119.8 (2)	N2C—C3C—H3C2	109.5
C18A—C13A—C12A	121.5 (2)	C4C—C3C—H3C2	109.5
C15A—C14A—C13A	121.5 (2)	H3C1—C3C—H3C2	108.1
C15A—C14A—H14A	119.3	N1C—C4C—C3C	110.8 (2)
C13A—C14A—H14A	119.3	N1C—C4C—H4CA	109.5
C14A—C15A—C16A	118.7 (3)	C3C—C4C—H4CA	109.5
C14A—C15A—H15A	120.6	N1C—C4C—H4CB	109.5
C16A—C15A—H15A	120.6	C3C—C4C—H4CB	109.5
C17A—C16A—C15A	120.9 (2)	H4CA—C4C—H4CB	108.1
C17A—C16A—I1A	119.64 (19)	C6C—C5C—C10C	117.7 (2)
C15A—C16A—I1A	119.5 (2)	C6C—C5C—N2C	119.2 (2)
C16A—C17A—C18A	119.7 (2)	C10C—C5C—N2C	123.0 (2)
C16A—C17A—H17A	120.2	C7C—C6C—C5C	121.4 (2)
C18A—C17A—H17A	120.2	C7C—C6C—H6C	119.3
C17A—C18A—C13A	120.5 (2)	C5C—C6C—H6C	119.3
C17A—C18A—H18A	119.7	C6C—C7C—C8C	120.5 (2)
C13A—C18A—H18A	119.7	C6C—C7C—H7C	119.8
C1B—N1B—C4B	109.9 (2)	C8C—C7C—H7C	119.8
C1B—N1B—H1BA	111.2 (18)	O1C—C8C—C9C	116.5 (2)
C4B—N1B—H1BA	110.7 (18)	O1C—C8C—C7C	125.2 (2)
C1B—N1B—H1BB	113.3 (16)	C9C—C8C—C7C	118.3 (2)
C4B—N1B—H1BB	105.8 (17)	C10C—C9C—C8C	121.3 (3)
H1BA—N1B—H1BB	106 (2)	C10C—C9C—H9C	119.3
C5B—N2B—C2B	114.91 (19)	C8C—C9C—H9C	119.3
C5B—N2B—C3B	115.9 (2)	C9C—C10C—C5C	120.8 (2)
C2B—N2B—C3B	110.5 (2)	C9C—C10C—H10C	119.6
N1B—C1B—C2B	109.5 (2)	C5C—C10C—H10C	119.6
N1B—C1B—H1BC	109.8	C8C—O1C—C11C	117.0 (2)
C2B—C1B—H1BC	109.8	O1C—C11C—H11G	109.5
N1B—C1B—H1BD	109.8	O1C—C11C—H11H	109.5
C2B—C1B—H1BD	109.8	H11G—C11C—H11H	109.5
H1BC—C1B—H1BD	108.2	O1C—C11C—H11I	109.5
N2B—C2B—C1B	111.0 (2)	H11G—C11C—H11I	109.5
N2B—C2B—H2BA	109.4	H11H—C11C—H11I	109.5
C1B—C2B—H2BA	109.4	O3C—C12C—O2C	123.7 (2)
N2B—C2B—H2BB	109.4	O3C—C12C—C13C	118.7 (2)
C1B—C2B—H2BB	109.4	O2C—C12C—C13C	117.6 (2)
H2BA—C2B—H2BB	108.0	C14C—C13C—C18C	118.5 (2)
N2B—C3B—C4B	111.4 (2)	C14C—C13C—C12C	120.4 (2)
N2B—C3B—H3B1	109.3	C18C—C13C—C12C	121.1 (2)
C4B—C3B—H3B1	109.3	C15C—C14C—C13C	121.4 (2)

N2B—C3B—H3B2	109.3	C15C—C14C—H14C	119.3
C4B—C3B—H3B2	109.3	C13C—C14C—H14C	119.3
H3B1—C3B—H3B2	108.0	C14C—C15C—C16C	118.5 (3)
N1B—C4B—C3B	110.4 (2)	C14C—C15C—H15C	120.8
N1B—C4B—H4BA	109.6	C16C—C15C—H15C	120.8
C3B—C4B—H4BA	109.6	C17C—C16C—C15C	121.5 (3)
N1B—C4B—H4BB	109.6	C17C—C16C—I1C	119.61 (19)
C3B—C4B—H4BB	109.6	C15C—C16C—I1C	118.9 (2)
H4BA—C4B—H4BB	108.1	C16C—C17C—C18C	119.3 (2)
C10B—C5B—C6B	116.8 (2)	C16C—C17C—H17C	120.3
C10B—C5B—N2B	123.1 (2)	C18C—C17C—H17C	120.3
C6B—C5B—N2B	120.0 (2)	C17C—C18C—C13C	120.7 (3)
C7B—C6B—C5B	122.2 (2)	C17C—C18C—H18C	119.6
C7B—C6B—H6B	118.9	C13C—C18C—H18C	119.6
C4A—N1A—C1A—C2A	−59.7 (3)	C8B—C9B—C10B—C5B	0.9 (4)
C5A—N2A—C2A—C1A	173.1 (2)	C6B—C5B—C10B—C9B	−1.7 (4)
C3A—N2A—C2A—C1A	−55.1 (3)	N2B—C5B—C10B—C9B	174.0 (2)
N1A—C1A—C2A—N2A	58.0 (3)	C7B—C8B—O1B—C11B	−1.3 (4)
C5A—N2A—C3A—C4A	−175.2 (2)	C9B—C8B—O1B—C11B	179.4 (2)
C2A—N2A—C3A—C4A	53.5 (3)	O3B—C12B—C13B—C14B	6.7 (4)
C1A—N1A—C4A—C3A	58.9 (3)	O2B—C12B—C13B—C14B	−172.3 (2)
N2A—C3A—C4A—N1A	−55.7 (3)	O3B—C12B—C13B—C18B	−173.8 (2)
C3A—N2A—C5A—C10A	1.4 (3)	O2B—C12B—C13B—C18B	7.1 (3)
C2A—N2A—C5A—C10A	131.8 (3)	C18B—C13B—C14B—C15B	−1.6 (4)
C3A—N2A—C5A—C6A	179.3 (2)	C12B—C13B—C14B—C15B	177.9 (2)
C2A—N2A—C5A—C6A	−50.3 (3)	C13B—C14B—C15B—C16B	0.9 (4)
C10A—C5A—C6A—C7A	−0.3 (4)	C14B—C15B—C16B—C17B	0.4 (4)
N2A—C5A—C6A—C7A	−178.3 (2)	C14B—C15B—C16B—I1B	−178.92 (18)
C5A—C6A—C7A—C8A	0.6 (4)	C15B—C16B—C17B—C18B	−1.1 (4)
C6A—C7A—C8A—O1A	−179.6 (2)	I1B—C16B—C17B—C18B	178.25 (19)
C6A—C7A—C8A—C9A	−0.5 (4)	C16B—C17B—C18B—C13B	0.4 (4)
O1A—C8A—C9A—C10A	179.3 (2)	C14B—C13B—C18B—C17B	0.9 (4)
C7A—C8A—C9A—C10A	0.1 (4)	C12B—C13B—C18B—C17B	−178.6 (2)
C8A—C9A—C10A—C5A	0.2 (4)	C4C—N1C—C1C—C2C	−55.2 (3)
C6A—C5A—C10A—C9A	−0.1 (4)	C5C—N2C—C2C—C1C	166.3 (2)
N2A—C5A—C10A—C9A	177.8 (2)	C3C—N2C—C2C—C1C	−59.0 (3)
C7A—C8A—O1A—C11A	−5.2 (4)	N1C—C1C—C2C—N2C	57.1 (3)
C9A—C8A—O1A—C11A	175.6 (2)	C5C—N2C—C3C—C4C	−167.4 (2)
O3A—C12A—C13A—C14A	3.8 (4)	C2C—N2C—C3C—C4C	58.4 (3)
O2A—C12A—C13A—C14A	−176.1 (2)	C1C—N1C—C4C—C3C	55.1 (3)
O3A—C12A—C13A—C18A	−176.6 (2)	N2C—C3C—C4C—N1C	−56.4 (3)
O2A—C12A—C13A—C18A	3.5 (4)	C2C—N2C—C5C—C6C	−49.1 (3)
C18A—C13A—C14A—C15A	−0.5 (4)	C3C—N2C—C5C—C6C	178.6 (2)
C12A—C13A—C14A—C15A	179.1 (2)	C2C—N2C—C5C—C10C	135.7 (2)
C13A—C14A—C15A—C16A	0.6 (4)	C3C—N2C—C5C—C10C	3.5 (3)
C14A—C15A—C16A—C17A	−0.4 (4)	C10C—C5C—C6C—C7C	0.6 (4)
C14A—C15A—C16A—I1A	−179.42 (19)	N2C—C5C—C6C—C7C	−174.8 (2)

C15A—C16A—C17A—C18A	0.1 (4)	C5C—C6C—C7C—C8C	1.3 (4)
I1A—C16A—C17A—C18A	179.17 (19)	C6C—C7C—C8C—O1C	177.9 (2)
C16A—C17A—C18A—C13A	−0.1 (4)	C6C—C7C—C8C—C9C	−1.9 (4)
C14A—C13A—C18A—C17A	0.3 (4)	O1C—C8C—C9C—C10C	−179.2 (2)
C12A—C13A—C18A—C17A	−179.3 (2)	C7C—C8C—C9C—C10C	0.6 (4)
C4B—N1B—C1B—C2B	−58.0 (3)	C8C—C9C—C10C—C5C	1.3 (4)
C5B—N2B—C2B—C1B	168.0 (2)	C6C—C5C—C10C—C9C	−1.9 (4)
C3B—N2B—C2B—C1B	−58.5 (3)	N2C—C5C—C10C—C9C	173.3 (2)
N1B—C1B—C2B—N2B	59.4 (3)	C9C—C8C—O1C—C11C	172.3 (2)
C5B—N2B—C3B—C4B	−170.5 (2)	C7C—C8C—O1C—C11C	−7.6 (4)
C2B—N2B—C3B—C4B	56.6 (3)	O3C—C12C—C13C—C14C	22.7 (4)
C1B—N1B—C4B—C3B	56.4 (3)	O2C—C12C—C13C—C14C	−157.3 (2)
N2B—C3B—C4B—N1B	−55.9 (3)	O3C—C12C—C13C—C18C	−156.5 (2)
C2B—N2B—C5B—C10B	137.5 (2)	O2C—C12C—C13C—C18C	23.6 (3)
C3B—N2B—C5B—C10B	6.6 (3)	C18C—C13C—C14C—C15C	0.2 (4)
C2B—N2B—C5B—C6B	−46.9 (3)	C12C—C13C—C14C—C15C	−179.0 (2)
C3B—N2B—C5B—C6B	−177.8 (2)	C13C—C14C—C15C—C16C	1.8 (4)
C10B—C5B—C6B—C7B	1.1 (4)	C14C—C15C—C16C—C17C	−2.1 (4)
N2B—C5B—C6B—C7B	−174.8 (2)	C14C—C15C—C16C—I1C	176.51 (19)
C5B—C6B—C7B—C8B	0.4 (4)	C15C—C16C—C17C—C18C	0.5 (4)
C6B—C7B—C8B—O1B	179.5 (2)	I1C—C16C—C17C—C18C	−178.12 (19)
C6B—C7B—C8B—C9B	−1.3 (4)	C16C—C17C—C18C—C13C	1.5 (4)
O1B—C8B—C9B—C10B	180.0 (2)	C14C—C13C—C18C—C17C	−1.8 (4)
C7B—C8B—C9B—C10B	0.6 (4)	C12C—C13C—C18C—C17C	177.3 (2)

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1W—H1W1⋯O3C[i]	0.77 (2)	1.93 (2)	2.696 (3)	173 (3)
O1W—H2W1⋯O2A	0.77 (2)	1.96 (2)	2.700 (3)	162 (3)
O2W—H1W2⋯O3A	0.77 (1)	1.86 (2)	2.626 (2)	170 (3)
O2W—H2W2⋯O2B	0.77 (2)	1.97 (2)	2.733 (3)	168 (3)
O3W—H1W3⋯O3B	0.77 (2)	1.87 (2)	2.636 (2)	172 (3)
O3W—H2W3⋯O2C	0.78 (1)	1.94 (2)	2.706 (3)	172 (3)
N1A—H1AB⋯O3W[ii]	0.94 (3)	1.82 (3)	2.754 (3)	172 (3)
N1A—H1AA⋯O2A	0.90 (3)	1.89 (3)	2.776 (3)	168 (3)
C1A—H1AC⋯O2W	0.99	2.57	3.362 (3)	136
C2A—H2AB⋯O3B[ii]	0.99	2.51	3.498 (3)	175
C4A—H4AA⋯O2C[iii]	0.99	2.47	3.441 (3)	166
C7A—H7A⋯I1B[iii]	0.95	3.32	4.212 (2)	156
C11A—H11A⋯O1B[iv]	0.98	2.51	3.225 (4)	130
N1B—H1BA⋯O2B	0.91 (3)	1.87 (3)	2.780 (3)	174 (3)
N1B—H1BB⋯O2W[ii]	0.92 (3)	1.85 (3)	2.768 (3)	176 (2)
C1B—H1BC⋯O3W	0.99	2.44	3.229 (3)	136
C2B—H2BB⋯O3A[ii]	0.99	2.63	3.619 (3)	174
C4B—H4BA⋯O2B[iii]	0.99	2.51	3.491 (3)	170
C4B—H4BB⋯O2W	0.99	2.52	3.260 (3)	131
C9B—H9B⋯I1B[ii]	0.95	3.25	4.020 (3)	139

N1C—H1CA···O2C	0.96 (3)	1.78 (3)	2.732 (3)	172 (3)
N1C—H1CA···O3C	0.96 (3)	2.64 (3)	3.297 (3)	126 (2)
N1C—H1CB···O1W[xi]	0.83 (3)	1.96 (3)	2.781 (3)	172 (3)
C1C—H1CC···O1W[v]	0.99	2.39	3.299 (3)	152
C4C—H4CA···O2A[iii]	0.99	2.45	3.438 (3)	174
C9C—H9C···I1A[ii]	0.95	3.29	4.013 (3)	135

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

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

Crystal data
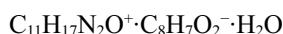
$C_{11}H_{17}N_2O^+ \cdot C_8H_7O_2^- \cdot H_2O$
$M_r = 346.42$
Triclinic, $P\bar{1}$
$a = 6.1481 (13)$ Å
$b = 7.3467 (12)$ Å
$c = 19.980 (4)$ Å
$\alpha = 80.190 (6)°$
$\beta = 86.089 (5)°$
$\gamma = 82.843 (6)°$
$V = 881.3 (3)$ Å^3

$Z = 2$
$F(000) = 372$
$D_x = 1.305$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9904 reflections
$\theta = 2.8–27.5°$
$\mu = 0.09$ mm^{-1}
$T = 90$ K
Plate, colourless
$0.29 \times 0.21 \times 0.02$ mm

Data collection

Bruker D8 Venture dual source
   diffractometer
Radiation source: microsource
Detector resolution: 7.41 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan
   (*SADABS*; Krause *et al.*, 2015)
$T_{min} = 0.877$, $T_{max} = 0.959$

23917 measured reflections
4059 independent reflections
3137 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.1°$
$h = -7 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.080$
$S = 1.07$
4059 reflections
245 parameters
0 restraints
Primary atom site location: structure-invariant
   direct methods
Secondary atom site location: difference Fourier
   map

Hydrogen site location: mixed
H atoms treated by a mixture of independent
   and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0124P)^2 + 0.3732P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: SHELXL-2019/2
   (Sheldrick 2015b),
   Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Extinction coefficient: 0.0038 (8)

Special details

Experimental. The crystal was mounted using polyisobutene oil on the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder. It was placed directly into the cold gas stream of a liquid-nitrogen based cryostat (Hope, 1994; Parkin & Hope, 1998).
Diffraction data were collected with the crystal at 90K, which is standard practice in this laboratory for the majority of flash-cooled crystals.

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.

Refinement. Refinement progress was checked using *Platon* (Spek, 2020) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

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

	x	*y*	*z*	$U_{iso}*/U_{eq}$
N1	0.23887 (18)	0.74787 (16)	0.54355 (5)	0.0192 (2)
H1A	0.203 (3)	0.727 (2)	0.4961 (9)	0.042 (5)*
H1B	0.271 (3)	0.871 (2)	0.5385 (8)	0.033 (4)*
N2	0.28444 (17)	0.65390 (15)	0.68884 (5)	0.0179 (2)
C1	0.4273 (2)	0.61887 (18)	0.57271 (6)	0.0202 (3)
H1C	0.558241	0.630796	0.541287	0.024*
H1D	0.392248	0.489067	0.578218	0.024*
C2	0.4760 (2)	0.66315 (19)	0.64091 (6)	0.0196 (3)
H2A	0.598899	0.573865	0.660243	0.024*
H2B	0.522549	0.789461	0.634542	0.024*
C3	0.0887 (2)	0.76711 (19)	0.66009 (6)	0.0211 (3)
H3A	0.108509	0.900258	0.656176	0.025*
H3B	−0.039758	0.743193	0.691596	0.025*
C4	0.0427 (2)	0.7273 (2)	0.59089 (6)	0.0220 (3)
H4A	0.002660	0.599190	0.595399	0.026*
H4B	−0.082879	0.814471	0.572187	0.026*
C5	0.3246 (2)	0.68052 (18)	0.75566 (6)	0.0183 (3)
C6	0.5285 (2)	0.62481 (19)	0.78350 (6)	0.0227 (3)
H6	0.644111	0.569862	0.756838	0.027*
C7	0.5680 (2)	0.6472 (2)	0.84923 (7)	0.0254 (3)
H7	0.709269	0.608607	0.866686	0.030*
C8	0.4021 (2)	0.7252 (2)	0.88893 (6)	0.0240 (3)
C9	0.1971 (2)	0.7774 (2)	0.86282 (7)	0.0274 (3)
H9	0.080563	0.827557	0.890402	0.033*
C10	0.1588 (2)	0.7577 (2)	0.79726 (7)	0.0252 (3)
H10	0.017294	0.797357	0.780137	0.030*
O1	0.42146 (17)	0.75852 (16)	0.95411 (5)	0.0337 (3)
C11	0.6322 (3)	0.7136 (3)	0.98204 (7)	0.0392 (4)
H11A	0.623438	0.740327	1.028619	0.059*
H11B	0.737047	0.788298	0.954254	0.059*
H11C	0.681120	0.581268	0.982463	0.059*
O2	0.12234 (15)	0.71150 (13)	0.41700 (4)	0.0241 (2)
O3	0.39970 (17)	0.87663 (14)	0.37969 (5)	0.0321 (3)
C12	0.2413 (2)	0.79566 (18)	0.36975 (7)	0.0206 (3)
C13	0.1882 (2)	0.79539 (18)	0.29744 (6)	0.0190 (3)
C14	0.3302 (2)	0.86250 (19)	0.24411 (7)	0.0229 (3)
H14	0.457991	0.912589	0.253423	0.027*
C15	0.2855 (2)	0.8564 (2)	0.17733 (7)	0.0259 (3)

H15	0.384517	0.901179	0.141471	0.031*
C16	0.0986 (2)	0.78599 (19)	0.16202 (7)	0.0241 (3)
C17	−0.0448 (2)	0.72276 (19)	0.21558 (7)	0.0226 (3)
H17	−0.174944	0.676288	0.206177	0.027*
C18	−0.0004 (2)	0.72670 (18)	0.28226 (6)	0.0201 (3)
H18	−0.099671	0.682046	0.318066	0.024*
C19	0.0474 (3)	0.7789 (2)	0.08986 (7)	0.0353 (4)
H19A	−0.003498	0.658960	0.087676	0.053*
H19B	0.179989	0.793648	0.060251	0.053*
H19C	−0.067539	0.879581	0.074736	0.053*
O1W	0.72107 (18)	0.87492 (14)	0.46115 (5)	0.0241 (2)
H1W	0.600 (3)	0.874 (3)	0.4328 (10)	0.062 (6)*
H2W	0.850 (3)	0.813 (3)	0.4429 (10)	0.060 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0202 (6)	0.0197 (6)	0.0178 (6)	−0.0037 (5)	−0.0013 (4)	−0.0026 (4)
N2	0.0149 (5)	0.0234 (6)	0.0157 (5)	−0.0021 (4)	−0.0007 (4)	−0.0038 (4)
C1	0.0192 (7)	0.0225 (7)	0.0187 (6)	−0.0006 (5)	−0.0003 (5)	−0.0045 (5)
C2	0.0162 (6)	0.0244 (7)	0.0184 (6)	−0.0033 (5)	0.0006 (5)	−0.0034 (5)
C3	0.0156 (6)	0.0276 (7)	0.0199 (7)	−0.0012 (6)	−0.0008 (5)	−0.0037 (5)
C4	0.0175 (7)	0.0277 (7)	0.0208 (7)	−0.0037 (6)	−0.0009 (5)	−0.0026 (5)
C5	0.0198 (7)	0.0187 (7)	0.0166 (6)	−0.0047 (5)	0.0001 (5)	−0.0020 (5)
C6	0.0188 (7)	0.0298 (8)	0.0197 (7)	−0.0016 (6)	0.0009 (5)	−0.0059 (5)
C7	0.0191 (7)	0.0358 (8)	0.0214 (7)	−0.0026 (6)	−0.0041 (5)	−0.0043 (6)
C8	0.0269 (8)	0.0303 (8)	0.0160 (6)	−0.0056 (6)	−0.0009 (5)	−0.0059 (5)
C9	0.0250 (7)	0.0362 (9)	0.0210 (7)	0.0008 (6)	0.0024 (6)	−0.0092 (6)
C10	0.0197 (7)	0.0334 (8)	0.0224 (7)	−0.0002 (6)	−0.0020 (5)	−0.0064 (6)
O1	0.0288 (6)	0.0545 (7)	0.0199 (5)	−0.0010 (5)	−0.0044 (4)	−0.0138 (5)
C11	0.0319 (9)	0.0664 (12)	0.0227 (8)	−0.0079 (8)	−0.0061 (6)	−0.0135 (7)
O2	0.0261 (5)	0.0270 (5)	0.0194 (5)	−0.0023 (4)	−0.0018 (4)	−0.0044 (4)
O3	0.0324 (6)	0.0365 (6)	0.0310 (6)	−0.0122 (5)	−0.0115 (5)	−0.0059 (4)
C12	0.0207 (7)	0.0171 (7)	0.0243 (7)	0.0025 (5)	−0.0051 (5)	−0.0061 (5)
C13	0.0187 (6)	0.0185 (7)	0.0198 (6)	0.0008 (5)	−0.0025 (5)	−0.0045 (5)
C14	0.0176 (7)	0.0236 (7)	0.0283 (7)	−0.0037 (6)	−0.0015 (5)	−0.0059 (6)
C15	0.0241 (7)	0.0307 (8)	0.0218 (7)	−0.0031 (6)	0.0029 (5)	−0.0032 (6)
C16	0.0266 (7)	0.0261 (7)	0.0195 (7)	0.0009 (6)	−0.0033 (5)	−0.0055 (5)
C17	0.0216 (7)	0.0234 (7)	0.0244 (7)	−0.0034 (6)	−0.0048 (5)	−0.0061 (5)
C18	0.0194 (7)	0.0195 (7)	0.0210 (7)	−0.0020 (5)	−0.0006 (5)	−0.0027 (5)
C19	0.0397 (9)	0.045 (1)	0.0223 (7)	−0.0043 (8)	−0.0038 (7)	−0.0080 (7)
O1W	0.0229 (5)	0.0260 (5)	0.0245 (5)	−0.0045 (4)	−0.0043 (4)	−0.0045 (4)

Geometric parameters (Å, º)

N1—C1	1.4864 (17)	C9—H9	0.9500
N1—C4	1.4879 (17)	C10—H10	0.9500
N1—H1A	1.028 (17)	O1—C11	1.4242 (18)

N1—H1B	0.939 (16)	C11—H11A	0.9800
N2—C5	1.4239 (16)	C11—H11B	0.9800
N2—C3	1.4669 (16)	C11—H11C	0.9800
N2—C2	1.4674 (16)	O2—C12	1.2759 (16)
C1—C2	1.5111 (17)	O3—C12	1.2461 (16)
C1—H1C	0.9900	C12—C13	1.5032 (18)
C1—H1D	0.9900	C13—C18	1.3917 (18)
C2—H2A	0.9900	C13—C14	1.3935 (18)
C2—H2B	0.9900	C14—C15	1.3897 (19)
C3—C4	1.5131 (18)	C14—H14	0.9500
C3—H3A	0.9900	C15—C16	1.3894 (19)
C3—H3B	0.9900	C15—H15	0.9500
C4—H4A	0.9900	C16—C17	1.3937 (19)
C4—H4B	0.9900	C16—C19	1.5074 (19)
C5—C6	1.3918 (18)	C17—C18	1.3840 (18)
C5—C10	1.3974 (18)	C17—H17	0.9500
C6—C7	1.3920 (18)	C18—H18	0.9500
C6—H6	0.9500	C19—H19A	0.9800
C7—C8	1.3777 (19)	C19—H19B	0.9800
C7—H7	0.9500	C19—H19C	0.9800
C8—O1	1.3809 (16)	O1W—H1W	0.97 (2)
C8—C9	1.381 (2)	O1W—H2W	0.94 (2)
C9—C10	1.3809 (19)		
C1—N1—C4	109.09 (10)	C7—C8—C9	118.96 (12)
C1—N1—H1A	113.3 (9)	O1—C8—C9	115.52 (12)
C4—N1—H1A	109.7 (9)	C10—C9—C8	121.06 (13)
C1—N1—H1B	109.9 (10)	C10—C9—H9	119.5
C4—N1—H1B	108.5 (10)	C8—C9—H9	119.5
H1A—N1—H1B	106.2 (13)	C9—C10—C5	121.16 (13)
C5—N2—C3	114.15 (10)	C9—C10—H10	119.4
C5—N2—C2	114.57 (10)	C5—C10—H10	119.4
C3—N2—C2	112.07 (10)	C8—O1—C11	117.39 (11)
N1—C1—C2	110.21 (11)	O1—C11—H11A	109.5
N1—C1—H1C	109.6	O1—C11—H11B	109.5
C2—C1—H1C	109.6	H11A—C11—H11B	109.5
N1—C1—H1D	109.6	O1—C11—H11C	109.5
C2—C1—H1D	109.6	H11A—C11—H11C	109.5
H1C—C1—H1D	108.1	H11B—C11—H11C	109.5
N2—C2—C1	111.9 (1)	O3—C12—O2	124.23 (12)
N2—C2—H2A	109.2	O3—C12—C13	117.92 (12)
C1—C2—H2A	109.2	O2—C12—C13	117.85 (11)
N2—C2—H2B	109.2	C18—C13—C14	118.71 (12)
C1—C2—H2B	109.2	C18—C13—C12	121.19 (11)
H2A—C2—H2B	107.9	C14—C13—C12	120.10 (12)
N2—C3—C4	112.90 (11)	C15—C14—C13	120.20 (12)
N2—C3—H3A	109.0	C15—C14—H14	119.9
C4—C3—H3A	109.0	C13—C14—H14	119.9

N2—C3—H3B	109.0	C16—C15—C14	121.25 (13)
C4—C3—H3B	109.0	C16—C15—H15	119.4
H3A—C3—H3B	107.8	C14—C15—H15	119.4
N1—C4—C3	110.50 (11)	C15—C16—C17	118.16 (12)
N1—C4—H4A	109.5	C15—C16—C19	121.89 (13)
C3—C4—H4A	109.5	C17—C16—C19	119.95 (13)
N1—C4—H4B	109.5	C18—C17—C16	120.95 (12)
C3—C4—H4B	109.5	C18—C17—H17	119.5
H4A—C4—H4B	108.1	C16—C17—H17	119.5
C6—C5—C10	116.85 (12)	C17—C18—C13	120.71 (12)
C6—C5—N2	121.63 (12)	C17—C18—H18	119.6
C10—C5—N2	121.48 (12)	C13—C18—H18	119.6
C5—C6—C7	122.01 (13)	C16—C19—H19A	109.5
C5—C6—H6	119.0	C16—C19—H19B	109.5
C7—C6—H6	119.0	H19A—C19—H19B	109.5
C8—C7—C6	119.92 (13)	C16—C19—H19C	109.5
C8—C7—H7	120.0	H19A—C19—H19C	109.5
C6—C7—H7	120.0	H19B—C19—H19C	109.5
C7—C8—O1	125.52 (12)	H1W—O1W—H2W	109.5 (16)
C4—N1—C1—C2	−59.83 (13)	C8—C9—C10—C5	1.5 (2)
C5—N2—C2—C1	175.50 (11)	C6—C5—C10—C9	0.1 (2)
C3—N2—C2—C1	−52.38 (14)	N2—C5—C10—C9	177.80 (13)
N1—C1—C2—N2	57.29 (14)	C7—C8—O1—C11	2.0 (2)
C5—N2—C3—C4	−176.52 (11)	C9—C8—O1—C11	−177.61 (14)
C2—N2—C3—C4	51.15 (14)	O3—C12—C13—C18	−172.58 (12)
C1—N1—C4—C3	58.20 (14)	O2—C12—C13—C18	8.03 (18)
N2—C3—C4—N1	−54.40 (15)	O3—C12—C13—C14	8.23 (19)
C3—N2—C5—C6	−162.71 (12)	O2—C12—C13—C14	−171.15 (12)
C2—N2—C5—C6	−31.58 (17)	C18—C13—C14—C15	−1.47 (19)
C3—N2—C5—C10	19.66 (17)	C12—C13—C14—C15	177.74 (12)
C2—N2—C5—C10	150.78 (12)	C13—C14—C15—C16	0.8 (2)
C10—C5—C6—C7	−1.0 (2)	C14—C15—C16—C17	0.6 (2)
N2—C5—C6—C7	−178.78 (12)	C14—C15—C16—C19	179.81 (14)
C5—C6—C7—C8	0.5 (2)	C15—C16—C17—C18	−1.2 (2)
C6—C7—C8—O1	−178.52 (13)	C19—C16—C17—C18	179.54 (13)
C6—C7—C8—C9	1.1 (2)	C16—C17—C18—C13	0.5 (2)
C7—C8—C9—C10	−2.0 (2)	C14—C13—C18—C17	0.85 (19)
O1—C8—C9—C10	177.58 (13)	C12—C13—C18—C17	−178.35 (12)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2	1.028 (17)	1.714 (17)	2.7391 (15)	174.1 (15)
N1—H1B···O1W[i]	0.939 (16)	1.873 (17)	2.7977 (16)	167.8 (14)
C1—H1C···O1W	0.99	2.46	3.2617 (17)	138
C2—H2B···O3[i]	0.99	2.52	3.5088 (17)	174
C4—H4A···O2[ii]	0.99	2.55	3.5294 (17)	169

C4—H4*B*···O1*W*[iii]	0.99	2.54	3.3184 (17)	135
O1*W*—H1*W*···O3	0.97 (2)	1.67 (2)	2.6418 (14)	176.5 (18)
O1*W*—H2*W*···O2[iv]	0.94 (2)	1.83 (2)	2.7626 (15)	170.5 (17)

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