The crystal structure of 1-(2-iodobenzoyl)-4-(pyrimidin-2-yl)piperazine: a three-dimensional hydrogen-bonded framework, augmented by π–π stacking interactions and I⋯N halogen bonds

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In 1-(2-iodobenzoyl)-4-(pyrimidin-2-yl)piperazine, $C_{15}H_{15}IN_4O$, the central piperazine ring adopts an almost perfect chair conformation with the pyrimidine substituent in an equatorial site. The planar amide unit makes a dihedral angle of $80.44 (7)°$ with the phenyl ring. A combination of C—H⋯O and C—H⋯π(arene) hydrogen bonds links the molecules into a complex three-dimensional network structure, augmented by a π–π stacking interaction and an I⋯N halogen bond, all involving different pairs of inversion-related molecules. Comparisons are made with the structures of a number of related compounds.

1. Chemical context

Pyrimidine derivatives are well represented amongst the range of heterocyclic compounds that exhibit a broad spectrum of biological activities such as analgesic and anti-inflammatory activity (Amin *et al.*, 2009), antibacterial (Kuyper *et al.*, 1996), antidepressant (Kim *et al.*, 2010), antimicrobial and antioxidant (Padmaja *et al.*, 2009) and antiviral activities (Ibrahim & El-Metwally, 2010). Piperazine-based compounds also exhibit anti-cancer properties (Abdel-Jalil *et al.*, 2005), while the combination of pyrimidine and piperazine units is found in buspirone, 8-[4-(4-pyrimidin-2-ylpiperazin-1-yl)butyl]-8-azaspiro[4.5]decane-7,9-dione (Tollefson *et al.*, 1991), which can be used in the treatment of anxiety. With these considerations in mind, we have now synthesized the title compound (I) (Fig. 1), and we report here its molecular and supramolecular structure. Compound (I) was prepared by reaction of 1-(2-pyrimidyl)piperazine with 2-iodobenzoic acid in the presence of dimethylaminopropyl)-3-ethylcarbodimide as the dehydrating agent.
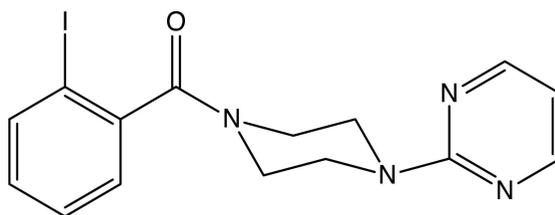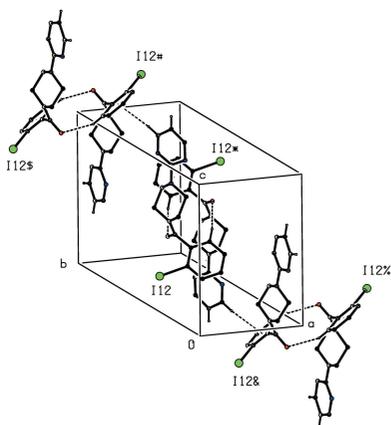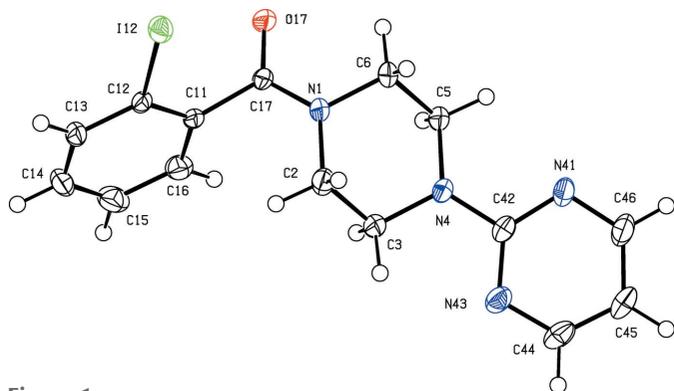
Figure 1
The molecular structure of compound (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2. Structural commentary

Within the molecule of compound (I) (Fig. 1), the piperazine ring adopts an almost perfect chair conformation. The ring-puckering parameters, calculated for the atom sequence (N1, C2, C3, N4, C5, C6) are $Q = 0.557$ (3) Å, $\theta = 1.2$ (3) ° and $\varphi = 258$ (14)°, while for an idealized chair form the value of θ is 0.0° (Boeyens, 1978). The pyrimidine substituent at the pyramidal atom N4 occupies an equatorial site, but the amidic unit at atom N1 is effectively planar, and the r.m.s. deviation from the mean plane of atoms (C2, N1, C6, C17, O17 and C11) is only 0.027 Å. The dihedral angle between this plane and the aryl ring (C11–C16) is 80.44 (7)°. The molecules of (I) thus exhibit no internal symmetry and so are conformationally chiral, as confirmed by the centrosymmetric space group in which the molecule crystallizes.

3. Supramolecular features

The supramolecular assembly of compound (I) is built from two C—H⋯O hydrogen bonds, involving the aryl and pyrimidyl atoms C16 and C45 as the donors (Table 1), and one C—H⋯π(arene) hydrogen bond: there is a further intermolecular C—H⋯O contact, involving atom C13, but here the D—H⋯A angle is less than 140°, and so this contact cannot be regarded as structurally significant (Wood *et al.*, 2009). There are also present in the structure a π–π stacking interaction between pairs of pyrimidine rings and an I⋯N halogen bond.

The hydrogen bonds give rise to a three-dimensional network structure of considerable complexity, but this is readily analysed in terms of three one-dimensional sub-structures (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000). The action of the two C—H⋯O hydrogen bonds in combination links molecules related by inversion and translation into a chain of edge-fused rings running parallel to the [001] direction (Fig. 2), in which $R_2^2(10)$ (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) rings centred at (0.5, 0.5, n + 0.5) alternate with $R_4^2(28)$ rings centred at (0.5, 0.5, n), where n represents an integer in each case.

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

\# $Cg1$ is the centroid of the C11–C16 ring.

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C16—H16⋯O17^{i}	0.95	2.50	3.395 (3)	157
C45—H45⋯O17^{ii}	0.95	2.57	3.508 (3)	169
C46—H46⋯$Cg1^{iii}$	0.95	2.72	3.596 (3)	153

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

A second sub-structure can be identified in which the C—H⋯π(arene) hydrogen bond links molecules related by a 2_1 screw axis to form a chain running parallel to the [010] direction (Fig. 3). The chains parallel to [010] and [001] each

Figure 2
Part of the crystal structure of compound (I) showing the formation of a hydrogen-bonded chain of edge-fused rings parallel to the [001] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H atoms not involved in the motif shown have been omitted. The I atoms marked with an asterisk (*), a hash (#) or a dollar sign ($) are at the symmetry positions $(1 - x, 1 - y, 1 - z)$, $(x, y, 1 + z)$ and $(1 - x, 1 - y, 2 - z)$, respectively.

use only one type of hydrogen bond, but the alternating action of the C—H⋯O and C—H⋯π(arene) hydrogen bonds involving atoms C16 and C46 as the donors (Table 1) links the molecules into a chain of rings running parallel to the [$\bar{1}$11] direction (Fig. 4). The combination of chains running parallel to [010], [001] and [$\bar{1}$11] suffices to generate a continuous three-dimensional network structure.

The formation of the hydrogen-bonded network is augmented by two further intermolecular interactions, each of which involves inversion related pairs of molecules. The pyrimidine rings of the molecules at (x, y, z) and ($1 - x, 1 - y, 2 - z$), which are components of the hydrogen-bonded chain along [001], are strictly parallel with an interplanar spacing of 3.4295 (10) Å and a ring-centroid separation of 3.4924 (6) Å, thus giving rise to a π–π stacking interaction (Fig. 5). Finally, we note a short intermolecular I⋯N contact with geometrical parameters of I12⋯N41^i = 3.168 (2) Å and C12—I12⋯N41^i 174.83 (7)° [symmetry code: (i) $-x, 1 - y, 1 - z$], which can be regarded as a halogen bond (Gilday *et al.*, 2015; Cavallo *et al.*, 2016).

4. Database survey

It is of interest briefly to compare the structure of compound (I) reported here with those of some related structures which have been recently reported. In 2-{4-[(1,3-benzodioxol-5-

yl)methyl]piperazin-1-yl}pyrimidine (II), the molecules are linked into sheets by a combination of C—H⋯π(arene) and C—H⋯π(pyrimidine) hydrogen bonds (Wu *et al.*, 2013). *N*-(4-Chlorophenyl)-4-(pyrimidin-2-yl)piperazine-1-carbox-amide (III) crystallizes with $Z' = 2$ in space group $P2_1/c$ (Li, 2011*b*), and the molecules are linked into chains by two independent N—H⋯O hydrogen bonds: these chains, parallel to [100], are of the $C_2^2(8)$ type rather than of the $C(4)$ type as originally reported. However, the original report overlooked the presence of C—H⋯O hydrogen bonds which, in combination with the N—H⋯O hydrogen bond within the selected asymmetric unit, generates a second chain, this time running parallel to the [010] direction (Fig. 6), so that overall the supramolecular assembly takes the form of a sheet parallel to (001). In the simpler analogue *N*-(4-chlorophenyl)-4-methylpiperidine-1-carboxamide (IV), the assembly was reported (Li, 2011*a*) as consisting of simple $C(4)$ chains built from N—H⋯O hydrogen bonds. However, the presence in (IV) of a C—H⋯O hydrogen bond was overlooked, and the two hydrogen bonds together generate a complex sheet structure lying parallel to (100) (Fig. 7). Finally, we note also the structures of a number of salts of the 4-(pyrimidin-2-yl)piperazin-1-ium cation, including the chloride and nitrate

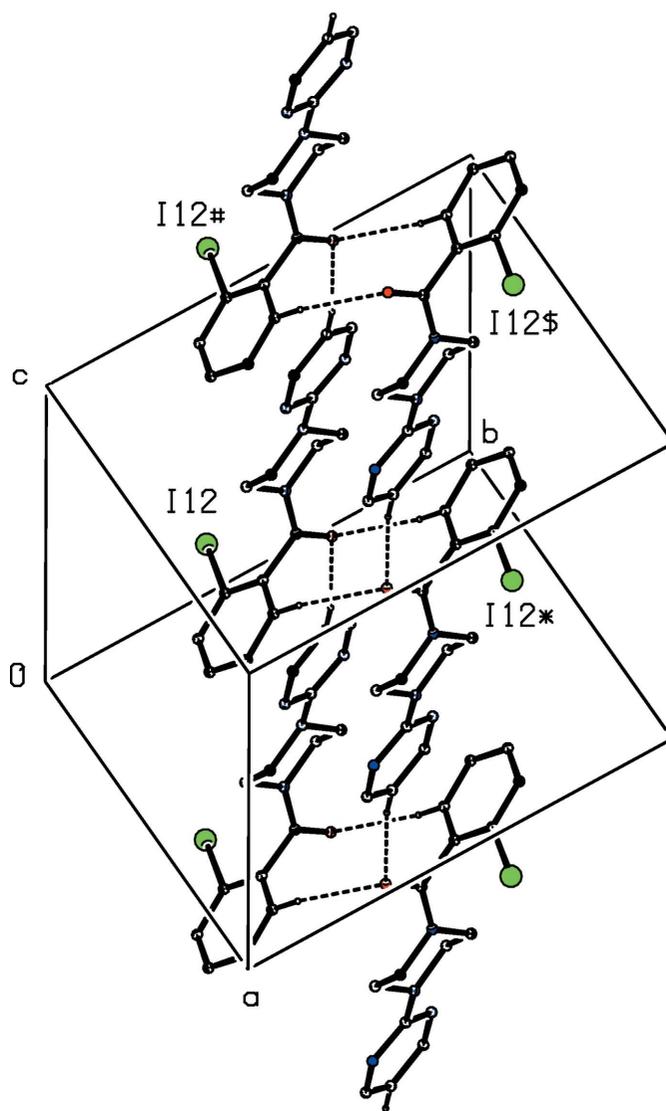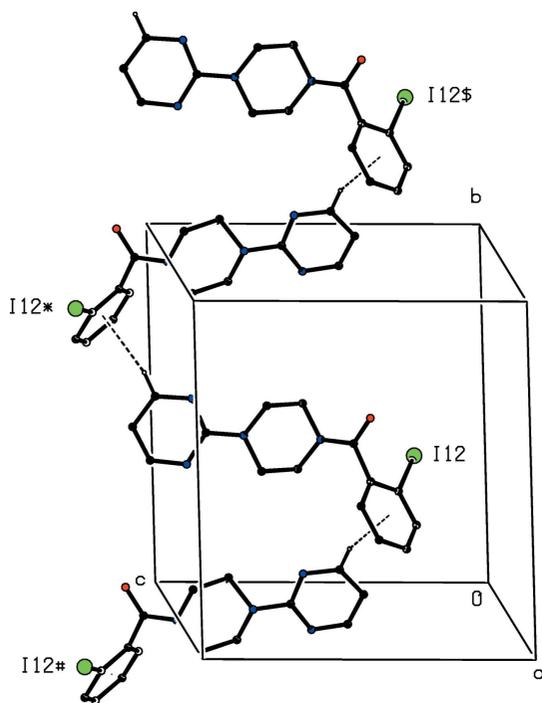**Figure 3**
Part of the crystal structure of compound (I) showing the formation of a hydrogen-bonded chain parallel to the [010] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H atoms not involved in the motif shown have been omitted. The I atoms marked with an asterisk (*), a hash (#) or a dollar sign ($) are at the symmetry positions ($\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$), ($\frac{1}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z$) and ($x, 1 + y, z$), respectively.

Figure 4
Part of the crystal structure of compound (I) showing the formation of a hydrogen-bonded chain of rings parallel to the [$\bar{1}$11] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H atoms not involved in the motif shown have been omitted. The I atoms marked with an asterisk (*), a hash (#), a dollar sign ($), an ampersand (&) or a percent sign (%) are at the symmetry positions ($1 - x, 1 - y, 1 - z$), ($\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$), ($-\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$), ($\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$) and ($1.5 - x, -\frac{1}{2} + y, \frac{1}{2} - z$) respectively.

Figure 5
Part of the crystal structure of compound (I) showing the π–π stacking interaction between adjacent pyrimidine rings. For the sake of clarity, the unit-cell outline and the H atoms have been omitted. The I atom marked with an asterisk (*) is at the symmetry position $(1 - x, 1 - y, 2 - z)$.

(Yamuna *et al.*, 2014*a*), the hydrogenfumarate (Yamuna *et al.*, 2014*b*) and the butanoate (Yamuna *et al.*, 2014*c*).

5. Synthesis and crystallization

1-(2-Pyrimidyl)piperazine was purchased from Sigma–Aldrich. For the synthesis of compound (I), 1-(3-dimethyl-aminopropyl)-3-ethylcarbodimide (52 mg, 0.6 mmol), 1-hydroxybenzotriazole (81 mg, 0.6 mmol) and triethylamine (0.5 ml, 1.8 mmol) were added to a solution of 2-iodobenzoic acid (0.6 mmol) in *N,N*-dimethylformamide (5 ml) and the resulting mixture stirred for 20 mins at 273 K. A solution of 1-(2-pyrimidyl)piperazine (100 mg, 0.6 mmol) in *N,N*-dimethylformamide (5 ml) was then added and stirring was continued overnight at ambient temperature. The reaction was confirmed to be complete using thin-layer chromatography, and the mixture was then quenched with water (10 ml) and extracted with ethyl acetate (20 ml). The organic layer was separated and washed successively with an aqueous hydrochloric acid solution (1 mol dm^{-3}), a saturated solution of sodium hydrogencarbonate and then with brine. The organic phase was dried over anhydrous sodium sulfate and the solvent was removed under reduced pressure. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of a solution in methanol; m. p. 450–452 K.
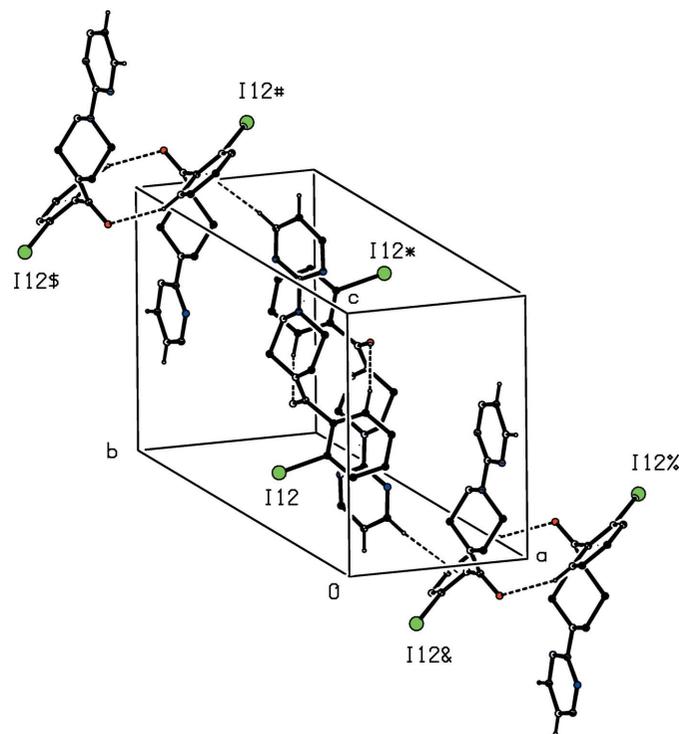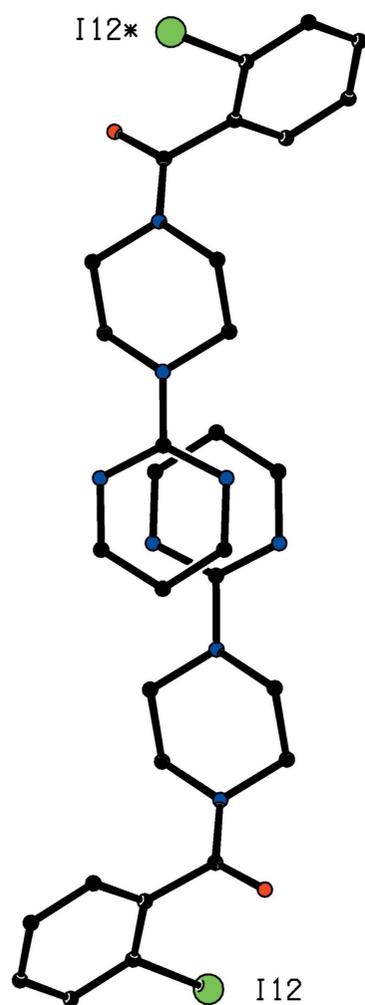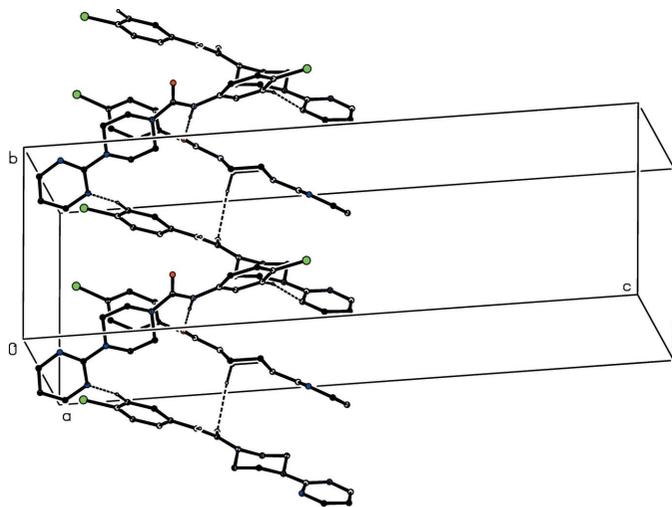

Figure 6
Part of the crystal structure of compound (III) showing the formation of a hydrogen-bonded chain parallel to [010]. The original atomic coordinates (Li, 2011*b*) have been used and, for the sake of clarity, the H atoms not involved in the motif shown have been omitted.
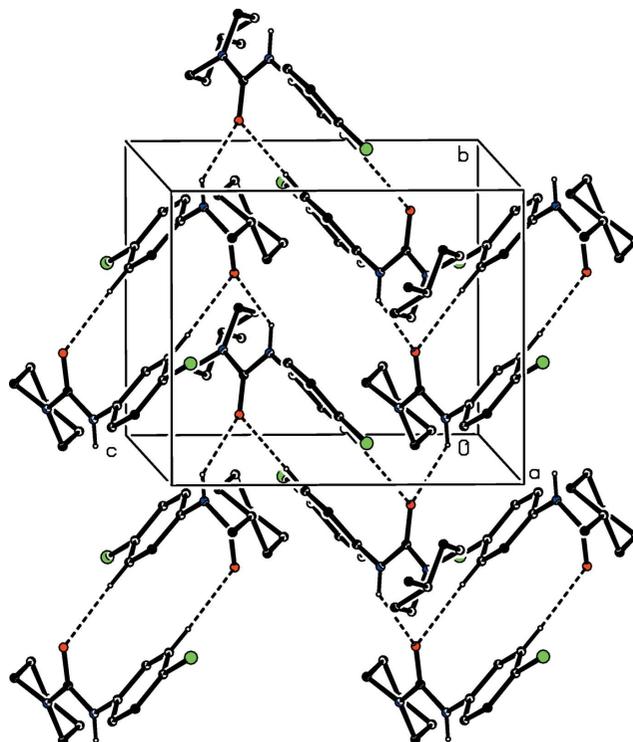

Figure 7
Part of the crystal structure of compound (IV) showing the formation of a hydrogen-bonded sheet parallel to (100). The original atomic coordinates (Li, 2011*a*) have been used and, for the sake of clarity, the H atoms not involved in the motif shown have been omitted.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{15}IN_4O$
M_r	394.21
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	173
a, b, c (Å)	9.6417 (17), 13.604 (2), 12.174 (2)
β (°)	105.155 (2)
V (Å^3)	1541.3 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.08
Crystal size (mm)	$0.67 \times 0.56 \times 0.16$
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.345, 0.717
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8152, 3452, 3188
R_{int}	0.067
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.652
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.028, 0.075, 1.05
No. of reflections	3452
No. of parameters	190
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.96, −0.74

Computer programs: *APEX2* (Bruker, 2004), *SAINT* (Bruker, 2015), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b) and *PLATON* (Spek, 2009).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were located in difference maps, and they were subsequently treated as riding atoms in geometrically idealized positions with C—H distances of 0.95 Å (aromatic) or 0.99 Å (CH$_2$), and with U_{iso}(H) = 1.2U_{eq}(C).

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supporting information

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Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2015); data reduction: *SAINT* (Bruker, 2015); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b) and *PLATON* (Spek, 2009).

1-(2-Iodobenzoyl)-4-(pyrimidin-2-yl)piperazine

Crystal data

$C_{15}H_{15}IN_4O$	$F(000) = 776$
$M_r = 394.21$	$D_x = 1.699$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.6417$ (17) Å	Cell parameters from 3152 reflections
$b = 13.604$ (2) Å	$\theta = 2.3$–27.6°
$c = 12.174$ (2) Å	$\mu = 2.08$ mm^{-1}
$\beta = 105.155$ (2)°	$T = 173$ K
$V = 1541.3$ (4) Å^3	Plate, colour
$Z = 4$	$0.67 \times 0.56 \times 0.16$ mm

Data collection

Bruker APEXII CCD diffractometer	8152 measured reflections
Radiation source: fine focus sealed tube	3452 independent reflections
Graphite monochromator	3188 reflections with $I > 2\sigma(I)$
Detector resolution: 0.3333 pixels mm^{-1}	$R_{int} = 0.067$
φ and ω scans	$\theta_{max} = 27.6°$, $\theta_{min} = 2.3°$
Absorption correction: multi-scan	$h = -12 \rightarrow 10$
(*SADABS*; Bruker, 2015)	$k = -17 \rightarrow 16$
$T_{min} = 0.345$, $T_{max} = 0.717$	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	3452 reflections
Least-squares matrix: full	190 parameters
$R[F^2 > 2\sigma(F^2)] = 0.028$	0 restraints
$wR(F^2) = 0.075$	Hydrogen site location: inferred from
$S = 1.05$	neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0235P)^2 + 0.7279P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.96$ e Å^{-3}

$\Delta\rho_{min} = -0.74$ 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}
N1	0.2388 (2)	0.44984 (14)	0.52539 (16)	0.0253 (4)
C2	0.2263 (3)	0.36447 (15)	0.5950 (2)	0.0282 (6)
H2A	0.2567	0.3046	0.5613	0.034*
H2B	0.1248	0.3559	0.5964	0.034*
C3	0.3195 (3)	0.37777 (17)	0.7159 (2)	0.0290 (6)
H3A	0.3033	0.3225	0.7640	0.035*
H3B	0.4222	0.3777	0.7159	0.035*
N4	0.2839 (2)	0.47043 (14)	0.76229 (16)	0.0260 (4)
C5	0.2977 (3)	0.55610 (16)	0.69257 (18)	0.0247 (5)
H5A	0.3991	0.5637	0.6904	0.030*
H5B	0.2689	0.6163	0.7265	0.030*
C6	0.2028 (3)	0.54263 (17)	0.57233 (19)	0.0273 (5)
H6A	0.1005	0.5421	0.5735	0.033*
H6B	0.2174	0.5980	0.5239	0.033*
C17	0.2929 (3)	0.45026 (16)	0.43404 (17)	0.0211 (4)
O17	0.3118 (2)	0.52552 (12)	0.38448 (14)	0.0334 (4)
C11	0.3328 (3)	0.35268 (15)	0.39109 (19)	0.0195 (5)
C12	0.2434 (2)	0.30692 (15)	0.29625 (17)	0.0191 (4)
I12	0.03709 (2)	0.36186 (2)	0.21850 (2)	0.02547 (8)
C13	0.2883 (3)	0.22226 (16)	0.25098 (19)	0.0257 (5)
H13	0.2268	0.1909	0.1867	0.031*
C14	0.4237 (3)	0.18428 (17)	0.3008 (2)	0.0327 (6)
H14	0.4553	0.1269	0.2699	0.039*
C15	0.5130 (3)	0.22889 (19)	0.3947 (2)	0.0346 (6)
H15	0.6054	0.2021	0.4284	0.042*
C16	0.4677 (3)	0.31314 (18)	0.4401 (2)	0.0279 (5)
H16	0.5293	0.3438	0.5048	0.033*
N41	0.2769 (2)	0.57158 (16)	0.91400 (17)	0.0301 (5)
C42	0.3025 (2)	0.48172 (17)	0.87784 (18)	0.0221 (4)
N43	0.3414 (2)	0.40176 (15)	0.94349 (17)	0.0280 (4)
C44	0.3528 (3)	0.4150 (2)	1.0545 (2)	0.0328 (6)
H44	0.3790	0.3601	1.1039	0.039*
C45	0.3288 (3)	0.5033 (2)	1.1012 (2)	0.0318 (6)
H45	0.3381	0.5108	1.1804	0.038*
C46	0.2902 (3)	0.5804 (2)	1.0258 (2)	0.0338 (6)
H46	0.2721	0.6427	1.0546	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0418 (12)	0.0171 (8)	0.0180 (9)	0.0026 (9)	0.0095 (8)	−0.0025 (7)
C2	0.0455 (17)	0.0190 (11)	0.0247 (13)	−0.0072 (10)	0.0172 (12)	−0.0029 (8)
C3	0.0496 (17)	0.0193 (10)	0.0211 (12)	0.0030 (12)	0.0149 (11)	0.0016 (9)
N4	0.0421 (12)	0.0213 (9)	0.0161 (9)	0.0009 (10)	0.0101 (8)	−0.0012 (7)
C5	0.0377 (13)	0.0191 (10)	0.0182 (11)	0.0013 (11)	0.0088 (9)	−0.0022 (8)
C6	0.0411 (14)	0.0223 (10)	0.0181 (10)	0.0059 (11)	0.0069 (10)	−0.0043 (9)
C17	0.0304 (11)	0.0176 (9)	0.0124 (9)	0.0005 (10)	0.0007 (8)	−0.0020 (8)
O17	0.0646 (13)	0.0173 (7)	0.0196 (8)	0.0013 (9)	0.0133 (8)	0.0014 (6)
C11	0.0302 (12)	0.0145 (9)	0.0150 (10)	−0.0006 (9)	0.0079 (9)	0.0014 (7)
C12	0.0263 (11)	0.0174 (9)	0.0153 (9)	−0.0017 (9)	0.0084 (8)	0.0003 (8)
I12	0.02670 (12)	0.02407 (11)	0.02409 (11)	−0.00088 (5)	0.00387 (8)	−0.00347 (5)
C13	0.0421 (14)	0.0163 (10)	0.0218 (11)	−0.0010 (11)	0.0141 (10)	−0.0016 (8)
C14	0.0491 (16)	0.0192 (11)	0.0374 (14)	0.0070 (12)	0.0250 (12)	0.0005 (10)
C15	0.0345 (13)	0.0304 (13)	0.0408 (14)	0.0108 (13)	0.0132 (11)	0.0083 (11)
C16	0.0313 (12)	0.0261 (11)	0.0238 (11)	0.0009 (11)	0.0031 (9)	0.0020 (9)
N41	0.0378 (12)	0.0343 (11)	0.0186 (10)	0.0082 (10)	0.0081 (8)	−0.0043 (8)
C42	0.0214 (10)	0.0286 (11)	0.0174 (10)	−0.0028 (10)	0.0070 (8)	−0.0009 (9)
N43	0.0368 (12)	0.0279 (10)	0.0214 (10)	−0.0055 (10)	0.0115 (8)	0.0019 (8)
C44	0.0384 (14)	0.0388 (14)	0.0224 (12)	−0.0092 (13)	0.0099 (10)	0.0063 (10)
C45	0.0351 (13)	0.0452 (14)	0.0169 (11)	−0.0073 (13)	0.0098 (9)	−0.0023 (10)
C46	0.0400 (14)	0.0428 (14)	0.0199 (12)	0.0034 (13)	0.0102 (10)	−0.0060 (11)

Geometric parameters (Å, º)

N1—C17	1.346 (3)	C11—C12	1.394 (3)
N1—C2	1.461 (3)	C12—C13	1.393 (3)
N1—C6	1.464 (3)	C12—I12	2.104 (2)
C2—C3	1.521 (4)	C13—C14	1.388 (4)
C2—H2A	0.9900	C13—H13	0.9500
C2—H2B	0.9900	C14—C15	1.379 (4)
C3—N4	1.459 (3)	C14—H14	0.9500
C3—H3A	0.9900	C15—C16	1.391 (3)
C3—H3B	0.9900	C15—H15	0.9500
N4—C42	1.379 (3)	C16—H16	0.9500
N4—C5	1.468 (3)	N41—C46	1.339 (3)
C5—C6	1.521 (3)	N41—C42	1.344 (3)
C5—H5A	0.9900	C42—N43	1.344 (3)
C5—H5B	0.9900	N43—C44	1.340 (3)
C6—H6A	0.9900	C44—C45	1.375 (4)
C6—H6B	0.9900	C44—H44	0.9500
C17—O17	1.226 (3)	C45—C46	1.380 (4)
C17—C11	1.513 (3)	C45—H45	0.9500
C11—C16	1.390 (3)	C46—H46	0.9500
C17—N1—C2	126.25 (18)	C16—C11—C12	119.3 (2)

C17—N1—C6	120.10 (19)	C16—C11—C17	119.1 (2)
C2—N1—C6	113.23 (17)	C12—C11—C17	121.3 (2)
N1—C2—C3	110.4 (2)	C13—C12—C11	120.5 (2)
N1—C2—H2A	109.6	C13—C12—I12	118.05 (18)
C3—C2—H2A	109.6	C11—C12—I12	121.41 (16)
N1—C2—H2B	109.6	C14—C13—C12	119.2 (2)
C3—C2—H2B	109.6	C14—C13—H13	120.4
H2A—C2—H2B	108.1	C12—C13—H13	120.4
N4—C3—C2	109.7 (2)	C15—C14—C13	120.7 (2)
N4—C3—H3A	109.7	C15—C14—H14	119.6
C2—C3—H3A	109.7	C13—C14—H14	119.6
N4—C3—H3B	109.7	C14—C15—C16	119.9 (2)
C2—C3—H3B	109.7	C14—C15—H15	120.0
H3A—C3—H3B	108.2	C16—C15—H15	120.0
C42—N4—C3	120.7 (2)	C11—C16—C15	120.3 (2)
C42—N4—C5	119.60 (18)	C11—C16—H16	119.8
C3—N4—C5	113.33 (17)	C15—C16—H16	119.8
N4—C5—C6	109.75 (19)	C46—N41—C42	116.0 (2)
N4—C5—H5A	109.7	N41—C42—N43	126.0 (2)
C6—C5—H5A	109.7	N41—C42—N4	116.7 (2)
N4—C5—H5B	109.7	N43—C42—N4	117.3 (2)
C6—C5—H5B	109.7	C44—N43—C42	115.3 (2)
H5A—C5—H5B	108.2	N43—C44—C45	123.8 (2)
N1—C6—C5	109.6 (2)	N43—C44—H44	118.1
N1—C6—H6A	109.8	C45—C44—H44	118.1
C5—C6—H6A	109.8	C44—C45—C46	115.8 (2)
N1—C6—H6B	109.8	C44—C45—H45	122.1
C5—C6—H6B	109.8	C46—C45—H45	122.1
H6A—C6—H6B	108.2	N41—C46—C45	123.1 (2)
O17—C17—N1	123.3 (2)	N41—C46—H46	118.5
O17—C17—C11	118.59 (19)	C45—C46—H46	118.5
N1—C17—C11	118.07 (19)		
C17—N1—C2—C3	116.2 (3)	C17—C11—C12—I12	−7.2 (3)
C6—N1—C2—C3	−56.4 (3)	C11—C12—C13—C14	−0.6 (3)
N1—C2—C3—N4	54.1 (3)	I12—C12—C13—C14	−179.83 (16)
C2—C3—N4—C42	151.8 (2)	C12—C13—C14—C15	0.6 (3)
C2—C3—N4—C5	−56.4 (3)	C13—C14—C15—C16	−0.3 (4)
C42—N4—C5—C6	−150.6 (2)	C12—C11—C16—C15	0.1 (3)
C3—N4—C5—C6	57.3 (3)	C17—C11—C16—C15	−173.4 (2)
C17—N1—C6—C5	−116.3 (2)	C14—C15—C16—C11	−0.1 (4)
C2—N1—C6—C5	56.8 (3)	C46—N41—C42—N43	−1.0 (4)
N4—C5—C6—N1	−55.3 (2)	C46—N41—C42—N4	177.6 (2)
C2—N1—C17—O17	−173.7 (3)	C3—N4—C42—N41	175.3 (2)
C6—N1—C17—O17	−1.6 (4)	C5—N4—C42—N41	25.3 (3)
C2—N1—C17—C11	6.0 (4)	C3—N4—C42—N43	−6.0 (3)
C6—N1—C17—C11	178.1 (2)	C5—N4—C42—N43	−156.1 (2)
O17—C17—C11—C16	94.4 (3)	N41—C42—N43—C44	1.1 (4)

N1—C17—C11—C16	−85.3 (3)	N4—C42—N43—C44	−177.5 (2)
O17—C17—C11—C12	−78.9 (3)	C42—N43—C44—C45	−0.7 (4)
N1—C17—C11—C12	101.4 (3)	N43—C44—C45—C46	0.4 (4)
C16—C11—C12—C13	0.2 (3)	C42—N41—C46—C45	0.5 (4)
C17—C11—C12—C13	173.52 (19)	C44—C45—C46—N41	−0.3 (4)
C16—C11—C12—I12	179.44 (16)		

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C11–C16 ring.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C13—H13⋯O17[i]	0.95	2.40	3.160 (3)	136
C16—H16⋯O17[ii]	0.95	2.50	3.395 (3)	157
C45—H45⋯O17[iii]	0.95	2.57	3.508 (3)	169
C46—H46⋯*Cg*1[iv]	0.95	2.72	3.596 (3)	153

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