

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Cyclopropylammonium 4-iodobenzoate

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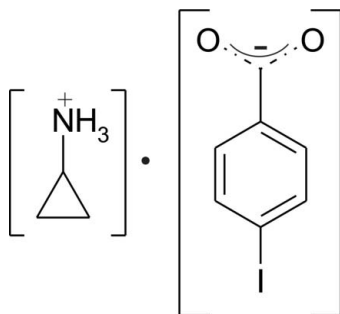
Received 28 May 2012; accepted 29 May 2012

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.023; wR factor = 0.047; data-to-parameter ratio = 19.5.

In the title molecular salt, $\text{C}_3\text{H}_8\text{N}^+\cdot\text{C}_7\text{H}_4\text{IO}_2^-$, the cyclopropanaminium cation forms three hydrogen bonds to the 4-iodobenzoate anion, forming two unique repeating $R_4^4(12)$ hydrogen-bonding rings that result in one-dimensional hydrogen-bonded columns along the crystallographic c axis.

Related literature

For proton-transfer compounds, see: Kinbara *et al.* (1996). For hydrogen bonds between primary ammonium cations and a carboxylate anion, see: Lemmerer (2011). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

 $\text{C}_3\text{H}_8\text{N}^+\cdot\text{C}_7\text{H}_4\text{IO}_2^-$ $M_r = 305.11$ Orthorhombic, $Pbcn$ $a = 30.7877$ (6) Å $b = 9.7608$ (2) Å $c = 7.4757$ (2) Å $V = 2246.54$ (9) Å³ $Z = 8$ Mo $K\alpha$ radiation $\mu = 2.83$ mm⁻¹ $T = 173$ K $0.5 \times 0.15 \times 0.11$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: integration (*XPREP*; Bruker, 2004) $T_{\min} = 0.332$, $T_{\max} = 0.746$

11693 measured reflections

2705 independent reflections

2103 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.037$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.023$ $wR(F^2) = 0.047$ $S = 0.97$

2705 reflections

139 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.42$ e Å⁻³ $\Delta\rho_{\min} = -0.79$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}$	0.86 (3)	1.95 (3)	2.807 (3)	173 (2)
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{i}}$	0.95 (2)	1.90 (2)	2.807 (2)	161 (2)
$\text{N1}-\text{H1C}\cdots\text{O2}^{\text{ii}}$	0.83 (3)	1.92 (3)	2.739 (2)	171 (2)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

The University of Cape Town is thanked for providing the infrastructure required to do this work and the National Research Fund (SFP2006061500015) for the award of a post-doctoral fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5937).

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supporting information

Acta Cryst. (2012). E68, o2096 [https://doi.org/10.1107/S160053681202449X]

Cyclopropylammonium 4-iodobenzoate

Andreas Lemmerer

S1. Comment

Ammonium carboxylate salts are molecular salts formed by mixing a primary amine and a carboxylic acid containing molecule, thus resulting in proton transfer from the acid to the amine (Kinbara *et al.*, 1996). This forms a primary ammonium cation and a carboxylate anion. The three H atoms on the cation can then form three charge-assisted hydrogen bonds to the two O atoms on the anion. In the literature, three kinds of hydrogen bonded rings are most commonly formed by these hydrogen bonds, described using graph-set notation (Bernstein *et al.*, 1995): $R^2_4(8)$, $R^3_4(10)$ and $R^4_4(12)$ (Lemmerer, 2011).

In molecular salt (I), shown in Fig. 1, formed by dissolving cyclopropylamine and *p*-iodobenzoic acid in methanol, only a $R^4_4(12)$ ring is formed. However, two such rings are formed, one by using the N1—H1A \cdots O1 and N1—H1B \cdots O1 hydrogen bonds, and the second one by using the N1—H1B \cdots O1 and N1—H1C \cdots O2 hydrogen bonds. As the N1—H1B \cdots O1 hydrogen bond is common to both rings, a repetition of the two types of rings results, forming a 1-D hydrogen bonded column along the *c* axis (Fig. 2).

S2. Experimental

All chemicals were purchased from commercial sources and used as received. (I) was prepared by slowly evaporating a solution of cyclopropylamine (0.050 g, 0.88 mmol) and *p*-iodobenzoic acid (0.217 g, 0.886 mmol) dissolved in 5 ml methanol.

S3. Refinement

All C—H atoms were refined using a riding model, with a distance of 0.95 Å (Ar—H), 0.99 Å, (CH₂) and 1.00 Å, (CH) and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. N—H atoms on the ammonium group were located in the difference Fourier map and their coordinates and isotropic displacement parameters were refined freely.

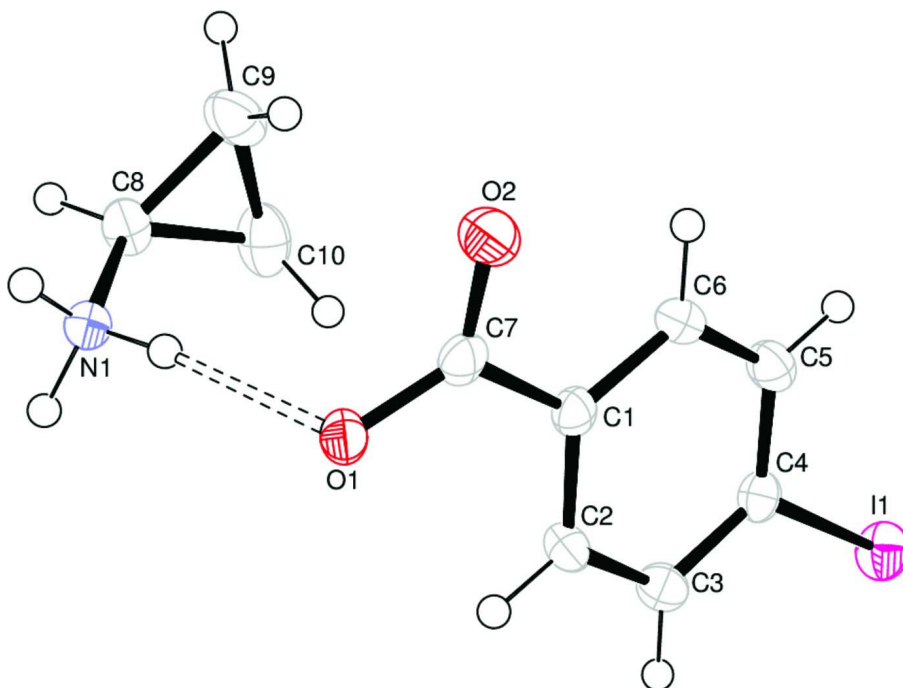


Figure 1

The asymmetric unit and atom numbering scheme of the title compound. Displacement ellipsoids are shown at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

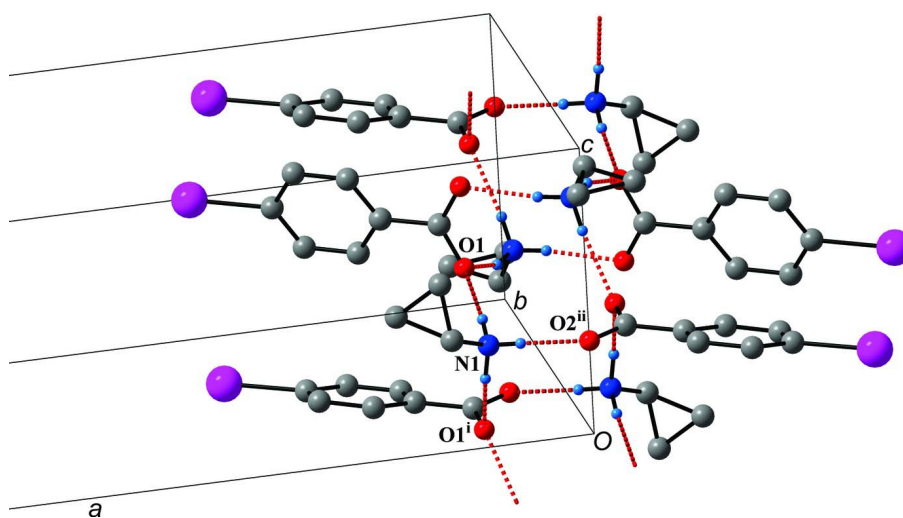


Figure 2

The hydrogen bonding pattern of the title compound. H atoms not involved in hydrogen bonding are omitted for clarity. Atoms marked with superscript *i* and *ii* are at the symmetry positions $(-x, -y + 1, z - 1/2)$ and $(-x, y, -z + 1/2)$ respectively.

Cyclopropylammonium 4-iodobenzoate

Crystal data

$C_3H_8N^+ \cdot C_7H_4IO_2^-$

$M_r = 305.11$

Orthorhombic, *Pbcn*

Hall symbol: $-P\ 2n\ 2ab$

$a = 30.7877(6)\ \text{\AA}$

$b = 9.7608(2)\ \text{\AA}$

$c = 7.4757 (2) \text{ \AA}$
 $V = 2246.54 (9) \text{ \AA}^3$
 $Z = 8$
 $F(000) = 1184$
 $D_x = 1.804 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4806 reflections
 $\theta = 2.7\text{--}28.1^\circ$
 $\mu = 2.83 \text{ mm}^{-1}$
 $T = 173 \text{ K}$
 Needle, colourless
 $0.5 \times 0.15 \times 0.11 \text{ mm}$

Data collection

Bruker APEXII CCD area-detector
 diffractometer
 ω scans
 Absorption correction: integration
 (XPREP; Bruker, 2004)
 $T_{\min} = 0.332$, $T_{\max} = 0.746$
 11693 measured reflections

2705 independent reflections
 2103 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
 $\theta_{\max} = 28^\circ$, $\theta_{\min} = 2.2^\circ$
 $h = -37 \rightarrow 40$
 $k = -12 \rightarrow 11$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.047$
 $S = 0.97$
 2705 reflections
 139 parameters
 0 restraints

H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0206P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.42 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.79 \text{ e \AA}^{-3}$

Special details

Experimental. Numerical integration absorption corrections based on indexed crystal faces were applied using the XPREP routine (Bruker, 2004)

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.12089 (7)	0.65046 (19)	0.5124 (3)	0.0213 (4)
C2	0.14737 (7)	0.5436 (2)	0.4583 (3)	0.0247 (5)
H2	0.1355	0.4708	0.3895	0.03*
C3	0.19096 (7)	0.5418 (2)	0.5035 (3)	0.0268 (5)
H3	0.2089	0.4674	0.4683	0.032*
C4	0.20809 (7)	0.6497 (2)	0.6003 (3)	0.0225 (4)
C5	0.18231 (7)	0.7577 (2)	0.6552 (3)	0.0262 (5)
H5	0.1944	0.8312	0.7221	0.031*
C6	0.13877 (7)	0.7572 (2)	0.6114 (3)	0.0241 (5)
H6	0.1208	0.8306	0.6493	0.029*
C7	0.07329 (7)	0.6516 (2)	0.4662 (3)	0.0235 (5)
O1	0.06027 (5)	0.56982 (14)	0.34560 (18)	0.0263 (3)
O2	0.04919 (5)	0.73454 (15)	0.5467 (2)	0.0332 (4)
I1	0.274626 (5)	0.652799 (16)	0.66287 (2)	0.03283 (6)
C8	0.05737 (7)	0.8095 (2)	-0.0226 (3)	0.0257 (5)

H8	0.0536	0.8472	-0.146	0.031*
C9	0.05800 (8)	0.9120 (2)	0.1242 (3)	0.0353 (6)
H9A	0.0452	0.8852	0.2405	0.042*
H9B	0.0542	1.0096	0.0918	0.042*
C10	0.09938 (8)	0.8420 (2)	0.0668 (3)	0.0383 (6)
H10A	0.1209	0.8967	-0.0008	0.046*
H10B	0.1119	0.7723	0.1479	0.046*
N1	0.03677 (6)	0.67743 (18)	0.0111 (3)	0.0227 (4)
H1A	0.0424 (9)	0.650 (2)	0.118 (4)	0.042 (8)*
H1B	0.0454 (7)	0.607 (2)	-0.068 (3)	0.034 (7)*
H1C	0.0101 (8)	0.686 (2)	-0.001 (3)	0.032 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0246 (10)	0.0218 (10)	0.0174 (10)	-0.0011 (9)	-0.0012 (9)	0.0029 (9)
C2	0.0308 (12)	0.0221 (11)	0.0214 (11)	-0.0006 (9)	-0.0023 (9)	-0.0045 (9)
C3	0.0282 (11)	0.0266 (11)	0.0255 (11)	0.0046 (9)	0.0017 (10)	-0.0037 (10)
C4	0.0212 (10)	0.0273 (11)	0.0190 (10)	-0.0010 (9)	-0.0001 (9)	0.0021 (10)
C5	0.0287 (11)	0.0240 (11)	0.0258 (12)	-0.0040 (9)	-0.0015 (10)	-0.0028 (10)
C6	0.0265 (11)	0.0207 (11)	0.0249 (11)	0.0018 (9)	-0.0003 (9)	-0.0036 (9)
C7	0.0287 (11)	0.0199 (10)	0.0220 (11)	-0.0018 (9)	-0.0040 (9)	0.0065 (10)
O1	0.0329 (8)	0.0222 (8)	0.0238 (8)	-0.0030 (6)	-0.0087 (7)	0.0008 (7)
O2	0.0240 (8)	0.0341 (9)	0.0416 (10)	0.0048 (7)	-0.0064 (7)	-0.0119 (8)
I1	0.02229 (8)	0.04054 (10)	0.03566 (10)	-0.00054 (6)	-0.00137 (7)	-0.00002 (8)
C8	0.0310 (12)	0.0249 (11)	0.0212 (11)	-0.0076 (9)	0.0002 (10)	0.0037 (9)
C9	0.0491 (16)	0.0231 (12)	0.0337 (14)	-0.0057 (11)	0.0002 (12)	-0.0024 (11)
C10	0.0373 (14)	0.0449 (15)	0.0327 (14)	-0.0172 (12)	-0.0037 (11)	0.0041 (12)
N1	0.0240 (10)	0.0213 (10)	0.0229 (11)	0.0007 (8)	-0.0038 (9)	-0.0004 (8)

Geometric parameters (Å, °)

C1—C2	1.384 (3)	C7—O1	1.269 (2)
C1—C6	1.391 (3)	C8—N1	1.459 (3)
C1—C7	1.506 (3)	C8—C9	1.485 (3)
C2—C3	1.384 (3)	C8—C10	1.490 (3)
C2—H2	0.95	C8—H8	1
C3—C4	1.382 (3)	C9—C10	1.508 (3)
C3—H3	0.95	C9—H9A	0.99
C4—C5	1.383 (3)	C9—H9B	0.99
C4—I1	2.101 (2)	C10—H10A	0.99
C5—C6	1.380 (3)	C10—H10B	0.99
C5—H5	0.95	N1—H1A	0.86 (3)
C6—H6	0.95	N1—H1B	0.95 (2)
C7—O2	1.253 (2)	N1—H1C	0.83 (3)
C2—C1—C6	119.13 (19)	C9—C8—C10	60.91 (15)
C2—C1—C7	120.76 (18)	N1—C8—H8	115.8

C6—C1—C7	120.11 (18)	C9—C8—H8	115.8
C3—C2—C1	120.59 (19)	C10—C8—H8	115.8
C3—C2—H2	119.7	C8—C9—C10	59.71 (14)
C1—C2—H2	119.7	C8—C9—H9A	117.8
C4—C3—C2	119.3 (2)	C10—C9—H9A	117.8
C4—C3—H3	120.4	C8—C9—H9B	117.8
C2—C3—H3	120.4	C10—C9—H9B	117.8
C3—C4—C5	121.2 (2)	H9A—C9—H9B	114.9
C3—C4—H1	119.99 (15)	C8—C10—C9	59.38 (15)
C5—C4—H1	118.84 (15)	C8—C10—H10A	117.8
C6—C5—C4	118.9 (2)	C9—C10—H10A	117.8
C6—C5—H5	120.5	C8—C10—H10B	117.8
C4—C5—H5	120.5	C9—C10—H10B	117.8
C5—C6—C1	120.91 (19)	H10A—C10—H10B	115
C5—C6—H6	119.5	C8—N1—H1A	110.5 (16)
C1—C6—H6	119.5	C8—N1—H1B	114.5 (14)
O2—C7—O1	124.1 (2)	H1A—N1—H1B	107 (2)
O2—C7—C1	118.10 (19)	C8—N1—H1C	108.7 (16)
O1—C7—C1	117.78 (19)	H1A—N1—H1C	109 (2)
N1—C8—C9	118.27 (19)	H1B—N1—H1C	107 (2)
N1—C8—C10	119.23 (19)		
C6—C1—C2—C3	0.7 (3)	C2—C1—C6—C5	0.2 (3)
C7—C1—C2—C3	-178.98 (19)	C7—C1—C6—C5	179.90 (19)
C1—C2—C3—C4	-1.4 (3)	C2—C1—C7—O2	166.03 (19)
C2—C3—C4—C5	1.1 (3)	C6—C1—C7—O2	-13.6 (3)
C2—C3—C4—H1	-177.88 (16)	C2—C1—C7—O1	-15.2 (3)
C3—C4—C5—C6	-0.2 (3)	C6—C1—C7—O1	165.19 (18)
H1—C4—C5—C6	178.79 (15)	N1—C8—C9—C10	109.6 (2)
C4—C5—C6—C1	-0.5 (3)	N1—C8—C10—C9	-108.0 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O1	0.86 (3)	1.95 (3)	2.807 (3)	173 (2)
N1—H1B \cdots O1 ⁱ	0.95 (2)	1.90 (2)	2.807 (2)	161 (2)
N1—H1C \cdots O2 ⁱⁱ	0.83 (3)	1.92 (3)	2.739 (2)	171 (2)

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