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(*S*)-Methyl 2-(2-iodobenzamido)-propionate

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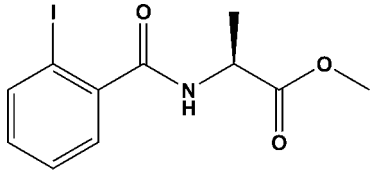
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.008 Å; R factor = 0.038; wR factor = 0.097; data-to-parameter ratio = 20.1.

In the title compound, $C_{11}H_{12}INO_3$, molecules are linked into one-dimensional chains along the crystallographic a axis by strong intermolecular N—H···O hydrogen bonds. The one-dimensional chains are additionally stabilized by weak intermolecular C_{ar}—H···O interactions. Unlike its iodine(V) analogues, the organic chain substituent in the title iodine(I) compound is not coplanar with the aromatic core but exhibits a C—C—C—O torsion angle of 50.3 (7)°.

Related literature

For the synthesis of the title compound, see: Zhdankin *et al.* (2000). For the related iodine(V) amide, see: Zhdankin *et al.* (2003). For other related literature, see: Koposov *et al.* (2006); Zhdankin *et al.* (2005); Chung *et al.* (2003).



Experimental

Crystal data

$C_{11}H_{12}INO_3$
M_r = 333.13
Orthorhombic, $P2_12_12_1$
a = 4.990 (3) Å
b = 12.120 (4) Å
c = 21.176 (10) Å
V = 1280.7 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.49 mm^{-1}
T = 295 K
0.30 × 0.15 × 0.10 mm

Data collection

Rigaku AFC-7R diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.65, T_{max} = 0.78
3032 measured reflections
2940 independent reflections
2721 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.097
S = 0.99
2934 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Absolute structure: Flack (1983), with 408 Friedel pairs
Flack parameter: −0.01 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2···O3^{i}	0.88	1.99	2.801 (6)	154 (4)
C4—H62···O1^{ii}	0.93	2.56	3.46 (2)	161 (8)

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

Data collection: *AFC-7R Software* (Rigaku, 1997); cell refinement: *WinAFC* (Rigaku/MSC, 2000); data reduction: *TEXSAN* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997) and *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2072).

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supplementary materials

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(*S*)-Methyl 2-(2-iodobenzamido)propionate

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Comment

Iodine(V) compounds are useful reagents for numerous oxidative transformations of different organic substrates (Koposov *et al.*, 2006; Zhdankin *et al.*, 2005; Chung *et al.*, 2003). The title compound represents an iodine(I) precursor for the preparation of the corresponding iodine(V) reagents. X-ray crystal structure analysis of the iodine(V) amide revealed the presence of a three-dimensional polymeric network formed by various types of intermolecular interactions. In particular, very strong intermolecular I···O bonding plays an important role in the formation of the tetrameric supramolecular motifs in the structure of the iodine(V) amide (Zhdankin *et al.*, 2003).

We now report the structure of the (*S*)-2-(2-iodobenzoylamino)propionic acid methyl ester, which crystalyses in the orthorhombic $P2_12_12_1$ space group (Fig. 1). In this molecule, the amidic moiety deviates from the phenyl ring plane (the C1—C6—C7—O3 torsion angle is 50.3 (7)°), thus contrasting with the structures of corresponding iodine(V) compounds where the amidic substituent was observed within the aromatic plane. The intramolecular bond distances and angles in the presented iodine(I) molecule are almost indistinguishable from those found in the corresponding iodine(V) compounds.

The molecules of (*S*)-2-(2-iodobenzoylamino)propionic acid methyl ester are linked together into one-dimensional polymeric chains by strong N—H···O hydrogen bonds (Table 1) formed between amide hydrogen atoms and the carbonyl oxygen atom O3 of a neighboring molecule at (*x+1, y, z*), generated by a translation along *a* axis (Fig. 2). The one-dimensional polymeric chain is stabilized by additional weak C—H···O hydrogen bonds formed between the phenyl ring hydrogen atom H62 and the carboxylic oxygen atom O1 of a neighboring molecule at (*-x + 3, y + 1/2, −z + 3/2*) (Fig. 2).

Experimental

The title compound was prepared according to a published procedure (Zhdankin *et al.*, 2000). Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a methylene chloride solution of the compound.

Refinement

The amidic H atom was located in a difference density Fourier map but was ultimately positioned geometrically with a N—H distance of 0.875 Å. The other H atoms were placed in calculated positions with C—H distances of 0.930 (aromatic) and 0.980 Å (alkyl). All hydrogen atoms were refined with U_{iso}(H) = 1.3 U_{eq} of their respective carrier atom. 6 reflections with [sin theta/lambda]**2 with values smaller than 0.01 were eliminated.

Figures

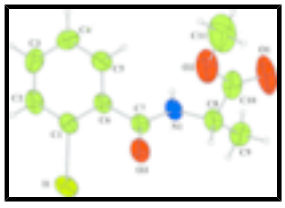

Fig. 1. Thermal ellipsoid representation of the title compound, with anisotropic displacement parameters for the non-H atoms of 50% probability.

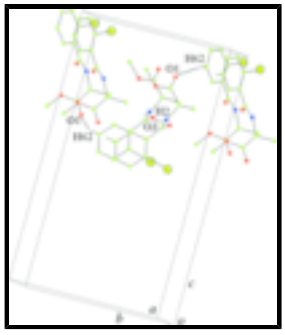

Fig. 2. Thermal ellipsoid representation showing the NH···O and CH···O H-bonding at 50% probability.

(*S*)-Methyl 2-(2-iodobenzamido)propionate

Crystal data

$C_{11}H_{12}I_1N_1O_3$	F_{000} = 648
M_r = 333.13	D_x = 1.728 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 25 reflections
a = 4.990 (3) Å	θ = 15–18º
b = 12.120 (4) Å	μ = 2.49 mm^{-1}
c = 21.176 (10) Å	T = 295 K
V = 1280.7 (10) Å^3	Block, colorless
Z = 4	0.30 × 0.15 × 0.10 mm

Data collection

Rigaku AFC-7R diffractometer	R_{int} = 0.035
Monochromator: graphite	θ_{max} = 27.5º
T = 295 K	θ_{min} = 2.6º
ω/2θ scans	h = −6→6
Absorption correction: ψ scan Azimuthal absorption correction (North *et al.*, 1968)	k = 0→15
T_{min} = 0.65, T_{max} = 0.78	l = 0→27
3032 measured reflections	3 standard reflections
2940 independent reflections	every 150 reflections
2721 reflections with $I > 2\sigma(I)$	intensity decay: 0.00%

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	P = $P(6)$*max(F_o^2,0) + (1-$P(6)$)F_c^2 Method = SHELXL97 (Sheldrick, 1997) W = 1/[Sigma^2(F*) + ($P(1)$p)^2 + $P(2)$p] P(i) are: 0.519E-01 2.03 0.333
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.097	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
S = 0.99	$\Delta\rho_{min}$ = −0.44 e Å^{-3}
2934 reflections	Extinction correction: none
146 parameters	Absolute structure: Flack (1983), with 408 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.01 (5)
Hydrogen site location: inferred from neighbouring sites	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.61721 (8)	0.35486 (3)	0.538896 (17)	0.0580
N1	1.1480 (9)	0.3848 (4)	0.70627 (19)	0.0502
C3	1.1280 (14)	0.6402 (5)	0.5224 (3)	0.0653
O3	0.7040 (7)	0.4075 (4)	0.6935 (2)	0.0652
C1	0.8807 (11)	0.4846 (4)	0.5611 (2)	0.0441
C8	1.1316 (13)	0.3301 (5)	0.7674 (2)	0.0600
C7	0.9335 (9)	0.4247 (4)	0.6762 (2)	0.0424
C6	0.9982 (9)	0.4959 (4)	0.6203 (2)	0.0401
C5	1.1812 (11)	0.5820 (5)	0.6293 (2)	0.0512
C4	1.2461 (13)	0.6542 (5)	0.5809 (3)	0.0617
C2	0.9463 (12)	0.5566 (5)	0.5123 (2)	0.0579
O2	1.1717 (16)	0.5073 (5)	0.8118 (3)	0.1014
C9	1.2777 (15)	0.2191 (6)	0.7664 (4)	0.0757
O1	1.386 (2)	0.3732 (6)	0.8604 (3)	0.1411
C11	1.254 (3)	0.5851 (9)	0.8601 (4)	0.1409
C10	1.2453 (17)	0.4043 (7)	0.8184 (3)	0.0753
H61	1.2598	0.5911	0.6688	0.0606*
H62	1.3668	0.7114	0.5879	0.0742*
H63	1.1732	0.6875	0.4894	0.0780*
H64	0.8672	0.5481	0.4728	0.0698*
H81	0.9424	0.3168	0.7772	0.0718*
H91	1.2646	0.1845	0.8070	0.0897*
H92	1.4629	0.2307	0.7561	0.0897*
H93	1.1970	0.1725	0.7351	0.0897*
H2	1.3076	0.3941	0.6900	0.0500*
H111	1.1864	0.6598	0.8500	0.1845*
H112	1.1811	0.5611	0.9013	0.1845*

H113	1.4524	0.5867	0.8623	0.1845*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.05217 (18)	0.05985 (19)	0.0620 (2)	−0.00088 (18)	−0.00941 (17)	−0.01249 (16)
N1	0.0281 (18)	0.077 (3)	0.046 (2)	−0.0009 (19)	−0.0024 (17)	0.0160 (19)
C3	0.072 (3)	0.070 (3)	0.053 (3)	−0.004 (4)	−0.002 (3)	0.023 (2)
O3	0.0289 (17)	0.104 (3)	0.062 (2)	−0.0042 (19)	−0.0006 (16)	0.030 (2)
C1	0.039 (2)	0.049 (2)	0.044 (2)	0.007 (2)	−0.003 (2)	−0.0040 (18)
C8	0.043 (2)	0.083 (4)	0.054 (3)	−0.007 (3)	−0.004 (2)	0.023 (3)
C7	0.029 (2)	0.056 (3)	0.042 (2)	−0.0003 (19)	0.0010 (17)	0.004 (2)
C6	0.031 (2)	0.053 (3)	0.037 (2)	0.0034 (19)	0.0016 (18)	0.0005 (19)
C5	0.048 (3)	0.060 (3)	0.046 (2)	−0.007 (2)	−0.001 (2)	−0.001 (2)
C4	0.065 (3)	0.057 (3)	0.064 (3)	−0.016 (3)	−0.001 (3)	0.006 (3)
C2	0.062 (4)	0.072 (3)	0.040 (2)	0.003 (3)	−0.007 (2)	0.005 (2)
O2	0.145 (6)	0.090 (3)	0.069 (3)	0.025 (4)	−0.025 (4)	0.000 (3)
C9	0.066 (4)	0.077 (4)	0.084 (5)	−0.007 (3)	−0.013 (3)	0.028 (4)
O1	0.217 (9)	0.121 (5)	0.085 (4)	0.031 (6)	−0.082 (5)	0.004 (3)
C11	0.234 (18)	0.112 (7)	0.077 (5)	0.014 (9)	−0.037 (7)	−0.020 (5)
C10	0.088 (5)	0.087 (5)	0.051 (3)	0.006 (4)	−0.005 (3)	0.018 (3)

Geometric parameters (Å, °)

I1—C1	2.103 (5)	C8—C10	1.516 (10)
C6—C7	1.501 (7)	C8—H81	0.980
O3—C7	1.220 (6)	C6—C5	1.401 (7)
N1—C7	1.335 (6)	C5—C4	1.385 (8)
N1—C8	1.457 (6)	C5—H61	0.930
O1—C10	1.192 (9)	C4—H62	0.930
O2—C10	1.309 (9)	C2—H64	0.930
N1—H2	0.875	O2—C11	1.451 (11)
C3—C4	1.384 (8)	C9—H91	0.960
C3—C2	1.376 (9)	C9—H92	0.960
C3—H63	0.930	C9—H93	0.960
C1—C6	1.390 (7)	C11—H111	0.990
C1—C2	1.393 (7)	C11—H112	0.990
C8—C9	1.530 (10)	C11—H113	0.990
N1—C7—O3	123.2 (5)	C6—C5—H61	119.041
O2—C10—O1	123.1 (8)	C4—C5—H61	119.422
C8—N1—C7	122.9 (4)	C5—C4—C3	119.0 (5)
C8—N1—H2	117.357	C5—C4—H62	120.359
C7—N1—H2	119.675	C3—C4—H62	120.599
C4—C3—C2	120.7 (5)	C1—C2—C3	120.1 (5)
C4—C3—H63	119.557	C1—C2—H64	119.834
C2—C3—H63	119.776	C3—C2—H64	120.069
I1—C1—C6	122.6 (4)	C11—O2—C10	117.7 (7)
I1—C1—C2	116.7 (4)	C8—C9—H91	109.869

C6—C1—C2	120.5 (5)	C8—C9—H92	109.438
N1—C8—C9	111.2 (5)	H91—C9—H92	109.476
N1—C8—C10	110.0 (5)	C8—C9—H93	109.093
C9—C8—C10	110.7 (5)	H91—C9—H93	109.476
N1—C8—H81	108.442	H92—C9—H93	109.476
C9—C8—H81	108.543	O2—C11—H111	110.118
C10—C8—H81	107.883	O2—C11—H112	109.043
N1—C7—C6	114.3 (4)	H111—C11—H112	109.475
O3—C7—C6	122.5 (4)	O2—C11—H113	109.237
C7—C6—C1	124.3 (4)	H111—C11—H113	109.476
C7—C6—C5	117.5 (4)	H112—C11—H113	109.476
C1—C6—C5	118.1 (5)	C8—C10—O2	112.6 (6)
C6—C5—C4	121.5 (5)	C8—C10—O1	124.3 (7)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H2···O3^{i}	0.88	1.99	2.801 (6)	154 (4)
C4—H62···O1^{ii}	0.93	2.56	3.46 (2)	161 (8)

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

Fig. 1

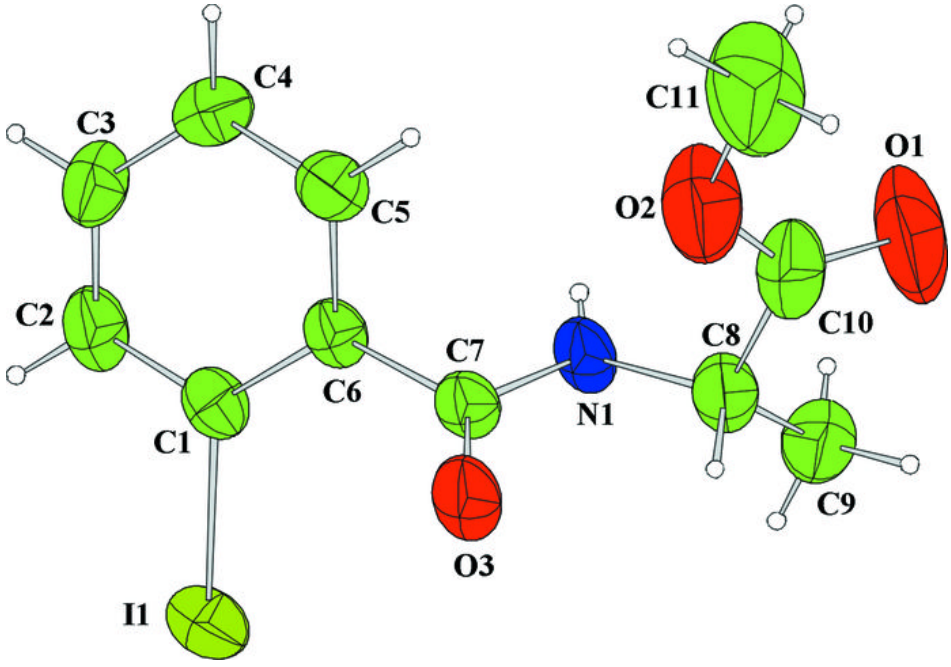

Fig. 2