Crystal structures of dimethyl 5-iodoisophthalate and dimethyl 5-ethynylisophthalate

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In dimethyl 5-iodoisophthalate, $C_{10}H_9IO_4$, (I), the planes through the methyl carboxylate moieties are tilted with respect to the benzene ring, whereas the molecular framework of dimethyl 5-ethynylisophthalate, $C_{12}H_{10}O_4$, (II), is perfectly planar. The crystal structure of (I) is stabilized by a three-dimensional supramolecular network comprising C—H···O=C hydrogen bonds, as well as I···O=C interactions. In the crystal of (II), the molecules are connected via C—$H_{ethynyl}$···O=C hydrogen bonds to infinite strands. Moreover, π–π arene stacking interactions connect the molecular chains into two-dimensional supramolecular aggregates.
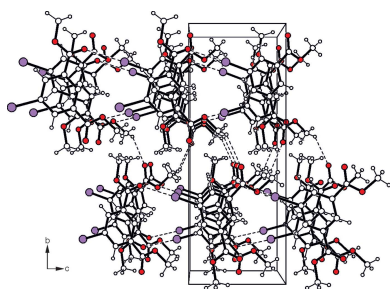
1. Chemical context

In recent years, the design of solid porous framework materials (MacGillivray, 2010; Furukawa *et al.*, 2013; Eddaoudi *et al.*, 2015) has become a very important topic in the field of supramolecular crystal engineering (Desiraju *et al.*, 2011). Associated with it, so-called linker molecules featuring a geometrically rigid structure frequently being of linear, trigonal or tetrahedral shape and having carboxylic acid functions as terminal groups play a key role in building such systems (Lin *et al.*, 2006; Hausdorf *et al.*, 2009; Zheng *et al.*, 2010). In the course of the synthesis of the respective linkers, the title compounds (I) and (II), both being 5-substituted dimethyl isophthalates, are much used intermediates. However, these compounds are not only synthetically significant but also show interesting structures in the crystalline state, as demonstrated herein.

(I)   (II)

2. Structural commentary

The molecular structures of the title compounds, (I) and (II), are illustrated in Fig. 1*a* and 1*b*, respectively. Taking into account experimental error, the bond distances within the isophthalate framework agree well with those found in the crystal structure of dimethyl isophthalate (Gallagher, 2012). Compound (I) crystallizes in the orthorhombic space group $Pna2_1$ with one molecule in the asymmetric unit. The molecule adopts a twisted conformation with the mean planes defined
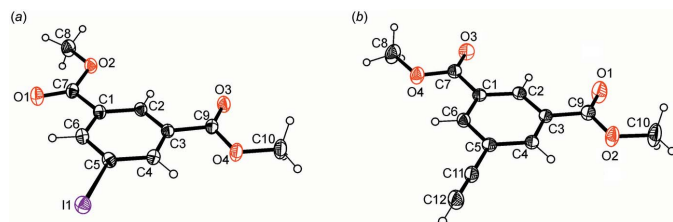
Figure 1
Perspective view of the molecular structures of the title compounds, (*a*) (I) and (*b*) (II), with atom labelling. Anisotropic displacement ellipsoids are drawn at the 40% probability level.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8—H8$A\cdots$O1^i	0.98	2.55	3.257 (4)	129

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C12—H12$\cdots$O1^i	0.94	2.29	3.223 (1)	172

Symmetry code: (i) $x - 1, y, z$.

by the methyl carboxylate moieties inclined at angles of 12.6 (2) and 6.0 (2)° with respect to the plane of the benzene ring. Compound (II) crystallizes in the orthorhombic space group *Pnma* with the molecule located on a symmetry plane, *i.e.* the molecule is perfectly planar. However, the molecule adopts approximate C_{2v} symmetry with the atoms C2, C5, C11 and C12 lying on a non-crystallographic bisecting symmetry plane.

3. Supramolecular features

Infinite strands with the molecules connected *via* I$\cdots$O=C interactions [I1$\cdots$O3—C9($x - \frac{1}{2}, y + \frac{3}{2}, z - 1$; $D\cdots A = 3.129$ (2) (Desiraju & Steiner, 1999) (Politzer *et al.* 2007; Desiraju *et al.*, 2013), represent the basic supramolecular aggregates of the crystal structure of (I). Association of the molecular strands by C—H$\cdots$O=C type hydrogen bonds (Table 1) (Desiraju & Steiner, 1999) and π–π stacking interactions [centroid–centroid distance = 4.149 (2) Å] (Tiekink & Zukerman-Schpector, 2012) generate a three-dimensional supramolecular network (Fig. 2). In the crystal structure of (II), the molecules are connected *via* $C_{ethynyl}$—H$\cdots$O=C bonds (Table 2) into infinite strands, which are further arranged into molecular sheets that extend parallel to the *ac* plane (Fig. 3).
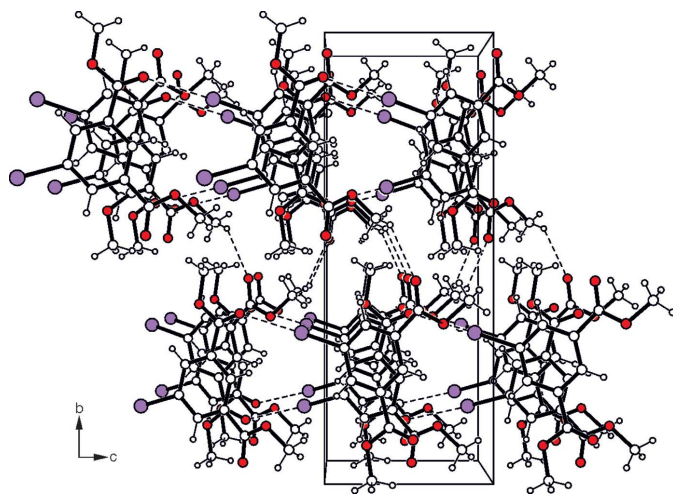
Furthermore, π–π arene interactions with a centroid–centroid distance of 3.566 (1) Å and a slippage of 1.325 Å between the interacting aromatic rings stabilize the crystal structure along the stacking axis of the molecular sheets.

4. Database survey

The search in the Cambridge Structural Database (CSD, Version 5.38, update May 2017; Groom *et al.*, 2016) for *meta*-substituted derivatives of dimethyl isophthalate excluding their metal complexes, solvates and salts gave 18 hits. None of these compounds represents a 5-halogen- and 5-alkynyl-substituted dimethyl isophalate. The parent compound, dimethyl isophthalate (CSD refcode GOHRUS; Gallagher & Mocilac, 2012) crystallizes in space group *Pna*2$_1$ with two conformationally similar molecules in the asymmetric unit. The independent molecules participate in different ways in non-covalent bonding. One of them is involved in the formation of linear strands with the molecules connected by C—H$_{aryl}\cdots$O=C bonds. Interstrand association is accomplished by π–π arene stacking. Molecules related by the twofold screw axis are also linked *via* C—H$_{aryl}\cdots$O=C

Figure 2
Packing diagram of compound (I) viewed down the *a* axis. Dashed lines represent hydrogen-bonding interactions.
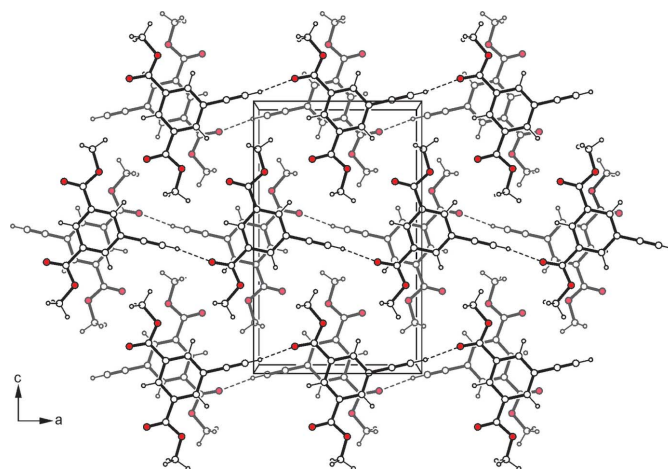

Figure 3
Packing excerpt of compound (II) viewed down the *b* axis. Dashed lines represent hydrogen-bonding interactions.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{10}H_9IO_4$	$C_{12}H_{10}O_4$
M_r	320.07	218.20
Crystal system, space group	Orthorhombic, $Pna2_1$	Orthorhombic, $Pnma$
Temperature (K)	143	223
a, b, c (Å)	7.7483 (2), 19.3451 (6), 7.2338 (2)	10.1206 (5), 6.6219 (4), 16.3658 (8)
V (Å^3)	1084.29 (5)	1096.80 (10)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.94	0.10
Crystal size (mm)	$0.30 \times 0.22 \times 0.15$	$0.54 \times 0.12 \times 0.10$
Data collection		
Diffractometer	Bruker APEXII CCD area detector	Bruker APEXII CCD area detector
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2008a)	Multi-scan (*SADABS*; Sheldrick, 2008a)
T_{min}, T_{max}	0.472, 0.666	0.948, 0.990
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22794, 2909, 2806	12397, 1292, 932
R_{int}	0.026	0.033
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.684	0.638
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.015, 0.038, 1.05	0.039, 0.110, 1.03
No. of reflections	2909	1292
No. of parameters	139	87
No. of restraints	1	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.47, −0.44	0.17, −0.18
Absolute structure	Flack x determined using 1255 quotients [$(I^+)-(I^-)$]/[$(I^+)+(I^-)$] (Parsons *et al.*, 2013)	–
Absolute structure parameter	−0.004 (8)	–

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015a), *SHELXL2018* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012) and *SHELXTL* (Sheldrick, 2008b).

bonding to form helical strands. In addition, these strands are stabilized by π–π stacking forces.

5. Synthesis and crystallization

Compounds (I) and (II) were synthesized following literature procedures. This involves a diazotization/iodination reaction of dimethyl 5-aminoisophthalate (Mazik & König, 2006) to give compound (I). Subsequent reaction of (I) with 2-methylbut-3-yne-2-ol (MEBYNOL) using a Pd-catalysed Sonogashira coupling procedure (Doucet & Hierso, 2007; Rafael & Carmen, 2007) yielded the corresponding blocked acetylenic diester as an intermediate (Hauptvogel *et al.*, 2011). Removal of the 2-hydroxypropyl blocking group was undertaken using sodium hydride in toluene and quenching with water to result in the title compound (II) (Havens & Hergenrother, 1985; Hauptvogel *et al.*, 2011).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Hydrogen atoms were positioned geometrically and refined using a riding model with C—H distances of 0.94–0.98 Å and $U_{iso}(H) = 1.5U_{eq}$(C-methyl) or $U_{iso}(H) = 1.2U_{eq}$(C) for other H atoms.

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supporting information

Crystal structures of dimethyl 5-iodoisophthalate and dimethyl 5-ethynylisophthalate

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Computing details

For both structures, data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: SHELXT (Sheldrick, 2015a). Program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b) for (I); *SHELXL2014* (Sheldrick, 2015b) for (II). For both structures, molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008b).

1,3-Dimethyl 1-iodocyclohexa-3,5-diene-1,3-dicarboxylate (I)

Crystal data

$C_{10}H_9IO_4$	$D_x = 1.961$ Mg m^{-3}
$M_r = 320.07$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, $Pna2_1$	Cell parameters from 5755 reflections
$a = 7.7483$ (2) Å	$\theta = 3.0–33.7°$
$b = 19.3451$ (6) Å	$\mu = 2.94$ mm^{-1}
$c = 7.2338$ (2) Å	$T = 143$ K
$V = 1084.29$ (5) Å^3	Irregular, colourless
$Z = 4$	$0.30 \times 0.22 \times 0.15$ mm
$F(000) = 616$	

Data collection

Bruker APEXII CCD area detector diffractometer	2909 independent reflections
	2806 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.026$
Absorption correction: multi-scan (SADABS; Sheldrick, 2008a)	$\theta_{max} = 29.1°$, $\theta_{min} = 1.1°$
	$h = -10 \rightarrow 10$
$T_{min} = 0.472$, $T_{max} = 0.666$	$k = -26 \rightarrow 26$
22794 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.019P)^2 + 0.3689P]$
$R[F^2 > 2\sigma(F^2)] = 0.015$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.038$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.05$	$\Delta\rho_{max} = 0.47$ e Å^{-3}
2909 reflections	$\Delta\rho_{min} = -0.44$ e Å^{-3}
139 parameters	Absolute structure: Flack x determined using 1255 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
1 restraint	
Hydrogen site location: inferred from neighbouring sites	Absolute structure parameter: -0.004 (8)

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.

Refinement. Refined as a 2-component twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.81504 (2)	0.66115 (2)	0.83115 (6)	0.02386 (5)
O1	0.5202 (3)	0.54751 (11)	0.1864 (3)	0.0327 (5)
O2	0.3831 (3)	0.63417 (11)	0.0411 (3)	0.0260 (4)
O3	0.4971 (3)	0.87789 (10)	0.2029 (3)	0.0281 (5)
O4	0.6557 (3)	0.89701 (11)	0.4576 (3)	0.0282 (5)
C1	0.5514 (4)	0.66267 (12)	0.2993 (4)	0.0190 (8)
C2	0.5347 (3)	0.73272 (14)	0.2585 (4)	0.0198 (5)
H2	0.479369	0.746965	0.147720	0.024*
C3	0.5994 (3)	0.78152 (14)	0.3805 (3)	0.0194 (5)
C4	0.6796 (3)	0.76086 (15)	0.5445 (4)	0.0206 (5)
H4	0.723931	0.794439	0.627770	0.025*
C5	0.6940 (3)	0.69088 (15)	0.5850 (4)	0.0207 (5)
C6	0.6295 (4)	0.64128 (15)	0.4637 (4)	0.0209 (5)
H6	0.638485	0.593502	0.492478	0.025*
C7	0.4856 (4)	0.60819 (14)	0.1721 (4)	0.0223 (5)
C8	0.3221 (5)	0.58545 (18)	−0.0951 (5)	0.0330 (7)
H8A	0.418544	0.571014	−0.173823	0.049*
H8B	0.232932	0.607288	−0.171341	0.049*
H8C	0.273509	0.544937	−0.032593	0.049*
C9	0.5768 (3)	0.85636 (11)	0.3323 (8)	0.0214 (4)
C10	0.6363 (5)	0.97101 (16)	0.4293 (5)	0.0338 (7)
H10A	0.688803	0.984036	0.311034	0.051*
H10B	0.693716	0.995947	0.529896	0.051*
H10C	0.513396	0.982919	0.427800	0.051*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02804 (8)	0.02336 (8)	0.02019 (8)	−0.00006 (6)	−0.00121 (12)	0.00381 (11)
O1	0.0500 (14)	0.0174 (10)	0.0308 (12)	0.0032 (9)	−0.0060 (10)	−0.0006 (9)
O2	0.0300 (11)	0.0187 (10)	0.0293 (11)	0.0017 (8)	−0.0066 (9)	−0.0048 (8)
O3	0.0347 (12)	0.0194 (10)	0.0301 (11)	0.0027 (9)	−0.0082 (9)	0.0019 (8)
O4	0.0382 (12)	0.0165 (9)	0.0299 (11)	−0.0011 (8)	−0.0073 (9)	−0.0004 (9)
C1	0.0216 (11)	0.0183 (10)	0.017 (2)	0.0009 (9)	0.0020 (10)	−0.0004 (9)
C2	0.0196 (12)	0.0190 (12)	0.0209 (11)	0.0022 (10)	0.0042 (10)	0.0019 (10)
C3	0.0191 (11)	0.0183 (11)	0.0208 (13)	0.0013 (9)	0.0025 (9)	0.0012 (8)
C4	0.0217 (13)	0.0191 (13)	0.0211 (12)	−0.0014 (10)	0.0017 (10)	−0.0012 (10)
C5	0.0224 (13)	0.0217 (13)	0.0181 (12)	0.0022 (10)	0.0023 (10)	0.0017 (10)

C6	0.0242 (13)	0.0183 (12)	0.0201 (12)	0.0016 (10)	0.0024 (11)	0.0013 (10)
C7	0.0256 (13)	0.0197 (12)	0.0217 (13)	−0.0011 (10)	0.0035 (11)	−0.0011 (10)
C8	0.0409 (19)	0.0257 (15)	0.0323 (15)	0.0005 (13)	−0.0088 (13)	−0.0075 (13)
C9	0.0218 (10)	0.0173 (9)	0.0250 (10)	−0.0001 (8)	0.0096 (18)	−0.002 (2)
C10	0.0453 (19)	0.0174 (14)	0.0387 (19)	0.0014 (13)	−0.0064 (15)	−0.0021 (12)

Geometric parameters (Å, º)

I1—C5	2.093 (3)	C3—C4	1.398 (4)
O1—C7	1.209 (3)	C3—C9	1.499 (4)
O2—C7	1.335 (4)	C4—C5	1.390 (4)
O2—C8	1.443 (4)	C4—H4	0.9500
O3—C9	1.196 (5)	C5—C6	1.393 (4)
O4—C9	1.347 (5)	C6—H6	0.9500
O4—C10	1.454 (4)	C8—H8A	0.9800
C1—C2	1.393 (3)	C8—H8B	0.9800
C1—C6	1.398 (4)	C8—H8C	0.9800
C1—C7	1.489 (4)	C10—H10A	0.9800
C2—C3	1.386 (4)	C10—H10B	0.9800
C2—H2	0.9500	C10—H10C	0.9800
C7—O2—C8	115.7 (2)	C1—C6—H6	120.4
C9—O4—C10	115.7 (3)	O1—C7—O2	124.0 (3)
C2—C1—C6	120.5 (3)	O1—C7—C1	124.0 (3)
C2—C1—C7	121.8 (3)	O2—C7—C1	112.1 (2)
C6—C1—C7	117.7 (2)	O2—C8—H8A	109.5
C3—C2—C1	119.7 (3)	O2—C8—H8B	109.5
C3—C2—H2	120.2	H8A—C8—H8B	109.5
C1—C2—H2	120.2	O2—C8—H8C	109.5
C2—C3—C4	120.4 (3)	H8A—C8—H8C	109.5
C2—C3—C9	117.9 (3)	H8B—C8—H8C	109.5
C4—C3—C9	121.7 (3)	O3—C9—O4	123.9 (2)
C5—C4—C3	119.5 (3)	O3—C9—C3	125.3 (3)
C5—C4—H4	120.2	O4—C9—C3	110.7 (3)
C3—C4—H4	120.2	O4—C10—H10A	109.5
C4—C5—C6	120.6 (3)	O4—C10—H10B	109.5
C4—C5—I1	118.9 (2)	H10A—C10—H10B	109.5
C6—C5—I1	120.5 (2)	O4—C10—H10C	109.5
C5—C6—C1	119.2 (3)	H10A—C10—H10C	109.5
C5—C6—H6	120.4	H10B—C10—H10C	109.5
C6—C1—C2—C3	1.3 (4)	C8—O2—C7—O1	−4.1 (4)
C7—C1—C2—C3	−179.4 (2)	C8—O2—C7—C1	176.3 (3)
C1—C2—C3—C4	−0.6 (4)	C2—C1—C7—O1	168.0 (3)
C1—C2—C3—C9	−179.3 (3)	C6—C1—C7—O1	−12.7 (4)
C2—C3—C4—C5	−0.1 (4)	C2—C1—C7—O2	−12.5 (4)
C9—C3—C4—C5	178.5 (3)	C6—C1—C7—O2	166.8 (3)
C3—C4—C5—C6	0.1 (4)	C10—O4—C9—O3	1.1 (5)

C3—C4—C5—I1	179.86 (19)	C10—O4—C9—C3	−177.5 (3)
C4—C5—C6—C1	0.7 (4)	C2—C3—C9—O3	5.3 (5)
I1—C5—C6—C1	−179.1 (2)	C4—C3—C9—O3	−173.3 (3)
C2—C1—C6—C5	−1.3 (4)	C2—C3—C9—O4	−176.1 (3)
C7—C1—C6—C5	179.4 (2)	C4—C3—C9—O4	5.3 (4)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O1^i	0.98	2.55	3.257 (4)	129

Symmetry code: (i) −x+1, −y+1, z−1/2.

1,3-Dimethyl 1-ethynylcyclohexa-3,5-diene-1,3-dicarboxylate (II)

Crystal data

$C_{12}H_{10}O_4$	$D_x = 1.321$ Mg m^{-3}
$M_r = 218.20$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, $Pnma$	Cell parameters from 2950 reflections
$a = 10.1206$ (5) Å	$\theta = 2.4$–23.1°
$b = 6.6219$ (4) Å	$\mu = 0.10$ mm^{-1}
$c = 16.3658$ (8) Å	$T = 223$ K
$V = 1096.80$ (10) Å^3	Column, colourless
$Z = 4$	0.54 × 0.12 × 0.10 mm
$F(000) = 456$	

Data collection

Bruker APEXII CCD area detector diffractometer	1292 independent reflections
	932 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.033$
Absorption correction: multi-scan (SADABS; Sheldrick, 2008a)	$\theta_{max} = 27.0°$, $\theta_{min} = 2.5°$
	$h = -12 \rightarrow 12$
$T_{min} = 0.948$, $T_{max} = 0.990$	$k = -8 \rightarrow 5$
12397 measured reflections	$l = -20 \rightarrow 19$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.110$	$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.2932P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
1292 reflections	$(\Delta/\sigma)_{max} < 0.001$
87 parameters	$\Delta\rho_{max} = 0.17$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	1.25864 (13)	0.2500	0.40733 (10)	0.0552 (4)	
O2	1.08848 (14)	0.2500	0.32081 (9)	0.0519 (4)	
O3	1.15356 (16)	0.2500	0.70802 (10)	0.0759 (6)	
O4	0.94247 (15)	0.2500	0.74402 (9)	0.0617 (5)	
C1	0.98919 (18)	0.2500	0.60451 (11)	0.0339 (4)	
C2	1.08100 (18)	0.2500	0.54143 (12)	0.0350 (4)	
H2	1.1718	0.2500	0.5536	0.042*	
C3	1.03958 (18)	0.2500	0.46063 (12)	0.0339 (4)	
C4	0.90505 (19)	0.2500	0.44325 (12)	0.0361 (4)	
H4	0.8767	0.2500	0.3886	0.043*	
C5	0.81192 (17)	0.2500	0.50582 (12)	0.0348 (4)	
C6	0.85496 (18)	0.2500	0.58682 (12)	0.0340 (4)	
H6	0.7929	0.2500	0.6296	0.041*	
C7	1.0392 (2)	0.2500	0.68995 (13)	0.0428 (5)	
C8	0.9818 (3)	0.2500	0.82935 (14)	0.0791 (9)	
H8A	1.0203	0.1202	0.8431	0.119*	0.5
H8B	0.9050	0.2740	0.8634	0.119*	0.5
H8C	1.0465	0.3558	0.8385	0.119*	0.5
C9	1.1418	0.2500	0.3949	0.039	
C10	1.1813	0.2500	0.2530	0.067	
H10A	1.1353	0.2149	0.2030	0.101*	0.5
H10B	1.2505	0.1519	0.2633	0.101*	0.5
H10C	1.2202	0.3832	0.2474	0.101*	0.5
C11	0.67260 (19)	0.2500	0.48649 (12)	0.0406 (5)	
C12	0.5604 (2)	0.2500	0.47022 (14)	0.0532 (6)	
H12	0.4700	0.2500	0.4571	0.064*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0293 (8)	0.0828 (11)	0.0536 (10)	0.000	0.0049 (7)	0.000
O2	0.0409 (9)	0.0783 (11)	0.0364 (8)	0.000	0.0066 (7)	0.000
O3	0.0329 (9)	0.1475 (18)	0.0473 (10)	0.000	−0.0079 (8)	0.000
O4	0.0362 (9)	0.1153 (14)	0.0336 (8)	0.000	−0.0026 (7)	0.000
C1	0.0294 (10)	0.0371 (10)	0.0352 (11)	0.000	−0.0006 (8)	0.000
C2	0.0257 (9)	0.0385 (10)	0.0408 (11)	0.000	−0.0029 (8)	0.000
C3	0.0295 (10)	0.0338 (9)	0.0385 (11)	0.000	0.0023 (8)	0.000
C4	0.0343 (11)	0.0404 (10)	0.0334 (10)	0.000	−0.0020 (9)	0.000
C5	0.0275 (9)	0.0379 (10)	0.0389 (11)	0.000	−0.0002 (8)	0.000
C6	0.0276 (9)	0.0398 (10)	0.0347 (11)	0.000	0.0011 (8)	0.000
C7	0.0299 (11)	0.0568 (12)	0.0416 (12)	0.000	−0.0013 (9)	0.000
C8	0.0550 (16)	0.151 (3)	0.0315 (13)	0.000	−0.0049 (12)	0.000
C9	0.034	0.043	0.039	0.000	0.003	0.000
C10	0.063	0.098	0.041	0.000	0.018	0.000
C11	0.0341 (11)	0.0558 (12)	0.0319 (10)	0.000	0.0003 (9)	0.000

| C12 | 0.0340 (12) | 0.0849 (17) | 0.0409 (13) | 0.000 | −0.0041 (10) | 0.000 |

Geometric parameters (Å, º)

O1—C9	1.1998 (14)	C4—C5	1.392 (3)
O2—C9	1.3272 (15)	C4—H4	0.9400
O2—C10	1.4544 (14)	C5—C6	1.395 (3)
O3—C7	1.195 (3)	C5—C11	1.445 (3)
O4—C7	1.319 (3)	C6—H6	0.9400
O4—C8	1.452 (3)	C8—H8A	0.9700
C1—C2	1.389 (3)	C8—H8B	0.9700
C1—C6	1.389 (3)	C8—H8C	0.9700
C1—C7	1.487 (3)	C10—H10A	0.9700
C2—C3	1.387 (3)	C10—H10B	0.9700
C2—H2	0.9400	C10—H10C	0.9700
C3—C4	1.391 (3)	C11—C12	1.166 (3)
C3—C9	1.4925 (18)	C12—H12	0.9400
C9—O2—C10	115.75 (10)	O3—C7—O4	123.6 (2)
C7—O4—C8	116.19 (18)	O3—C7—C1	124.21 (19)
C2—C1—C6	119.96 (18)	O4—C7—C1	112.23 (17)
C2—C1—C7	118.13 (17)	O4—C8—H8A	109.5
C6—C1—C7	121.91 (17)	O4—C8—H8B	109.5
C3—C2—C1	120.42 (17)	H8A—C8—H8B	109.5
C3—C2—H2	119.8	O4—C8—H8C	109.5
C1—C2—H2	119.8	H8A—C8—H8C	109.5
C2—C3—C4	119.39 (18)	H8B—C8—H8C	109.5
C2—C3—C9	118.53 (15)	O1—C9—O2	123.76 (10)
C4—C3—C9	122.09 (16)	O1—C9—C3	124.12 (11)
C3—C4—C5	120.83 (18)	O2—C9—C3	112.12 (9)
C3—C4—H4	119.6	O2—C10—H10A	109.5
C5—C4—H4	119.6	O2—C10—H10B	109.5
C4—C5—C6	119.18 (17)	H10A—C10—H10B	109.5
C4—C5—C11	119.98 (18)	O2—C10—H10C	109.5
C6—C5—C11	120.84 (17)	H10A—C10—H10C	109.5
C1—C6—C5	120.21 (17)	H10B—C10—H10C	109.5
C1—C6—H6	119.9	C12—C11—C5	179.5 (2)
C5—C6—H6	119.9	C11—C12—H12	180.0
C6—C1—C2—C3	0.000 (1)	C8—O4—C7—O3	0.000 (1)
C7—C1—C2—C3	180.000 (1)	C8—O4—C7—C1	180.000 (1)
C1—C2—C3—C4	0.000 (1)	C2—C1—C7—O3	0.000 (1)
C1—C2—C3—C9	180.000 (1)	C6—C1—C7—O3	180.000 (1)
C2—C3—C4—C5	0.000 (1)	C2—C1—C7—O4	180.000 (1)
C9—C3—C4—C5	180.000 (1)	C6—C1—C7—O4	0.000 (1)
C3—C4—C5—C6	0.000 (1)	C10—O2—C9—O1	0.000 (1)
C3—C4—C5—C11	180.000 (1)	C10—O2—C9—C3	180.000 (1)
C2—C1—C6—C5	0.000 (1)	C2—C3—C9—O1	0.000 (1)

C7—C1—C6—C5	180.000 (1)	C4—C3—C9—O1	180.000 (1)
C4—C5—C6—C1	0.000 (1)	C2—C3—C9—O2	180.000 (1)
C11—C5—C6—C1	180.000 (1)	C4—C3—C9—O2	0.000 (1)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1[i]	0.94	2.29	3.223 (1)	172

Symmetry code: (i) *x*−1, *y*, *z*.