Cyclohexa-2,5-diene-1,4-dione–1,2,4,5-tetrafluoro-3,6-diiodobenzene (1/1)

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The asymmetric unit of the title co-crystal adduct, $C_6H_4O_2 \cdot C_6F_4I_2$, comprises a half-molecule each of cyclohexa-2,5-diene-1,4-dione and 1,2,4,5-tetrafluoro-3,6-diiodobenzene. The $C_6F_4I_2$ molecule is almost planar (r.m.s. deviation = 0.0062 Å). In the crystal, the components are connected through O···I halogen bonds [3.017 (11) Å], leading to the formation of wavelike chains along the a axis. The crystal packing also features C—H···F interactions.

Related literature

For related studies on co-crystal formation, see: Bhogala & Nangia (2008); Ji *et al.* (2011); Arman *et al.* (2010); Cardillo *et al.* (2000). For background to halogen bonding, see: Metrangolo *et al.* (2008).
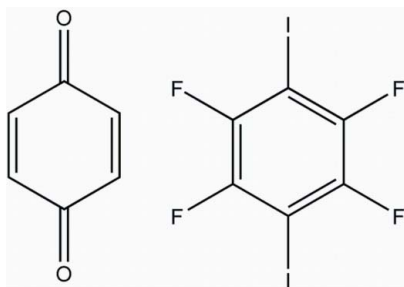
Experimental

Crystal data

$C_6H_4O_2 \cdot C_6F_4I_2$

$M_r = 509.95$

Triclinic, $P\bar{1}$
$a = 5.778$ (3) Å
$b = 6.354$ (3) Å
$c = 10.013$ (5) Å
$\alpha = 102.295$ (5)°
$\beta = 93.861$ (5)°
$\gamma = 97.781$ (5)°

$V = 354.1$ (3) Å^3
$Z = 1$
Mo $K\alpha$ radiation
$\mu = 4.48$ mm^{-1}
$T = 296$ K
$0.43 \times 0.30 \times 0.26$ mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.249$, $T_{max} = 0.389$

2585 measured reflections
1291 independent reflections
1096 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.07$
1291 reflections

91 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 1.34$ e Å^{-3}
$\Delta\rho_{min} = -0.76$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···F1[i]	0.93	2.64	3.562	171

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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supplementary materials

Cyclohexa-2,5-diene-1,4-dione–1,2,4,5-tetrafluoro-3,6-diiodobenzene (1/1)

Peng Liu, Chuansheng Ruan, Tiesheng Li and Baoming Ji

Comment

The title co-crystal is part of a study on the halogen bond, which is a powerful intermolecular interaction we and others have used extensively to produce a variety of structures involving perfuorinated compounds (Ji *et al.* 2011; Arman *et al.* 2010; Cardillo *et al.* 2000), usually very diffcult to crystallize.

In the crystal structure, 1,2,4,5-tetrafluoro-3,6-diiodobenzene molecule is flat with the *r.m.s.* deviation of the 12 constituent atoms being 0.0062 Å (Fig. 1). It is noted that the cyclohexa-2,5-diene-1,4-dione molecule acts as a bidentate donor towards 1,2,4,5-tetrafluoro-3,6-diiodobenzene molecule, giving rise to chains extended throughout the whole crystal, in which the bond length of O···I halogen bond is 3.017 Å, as observed in the previous reports (Metrangolo *et al.* 2008; Ji *et al.* 2011).

In addition, the molecules are further stabilized in the crystal packing *via* a combination of C—H···F contacts (Table. 1), as shown in Fig. 2.

Experimental

The starting materials were commercial obtained from Aldrich. The 1:1 adduct was obtained by dissolving in chloroform, at room temperature and in a vial, equimolecular amounts of cyclohexa-2,5-diene-1,4-dione and 1,2,4,5-tetrafluoro-3,6-diiodobenzene. The open vial was closed in a cylindrical bottle containing vaseline oil. Volatile solvents were allowed to diffuse at room temperature and, after one day, the yellow block crystals were obtained.

Refinement

All H atoms were positioned geometrically and treated as riding, with C—H bond lengths constrained to 0.93 Å (aromatic CH), and with Uiso~(H) = 1.2Ueq(C).

Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).
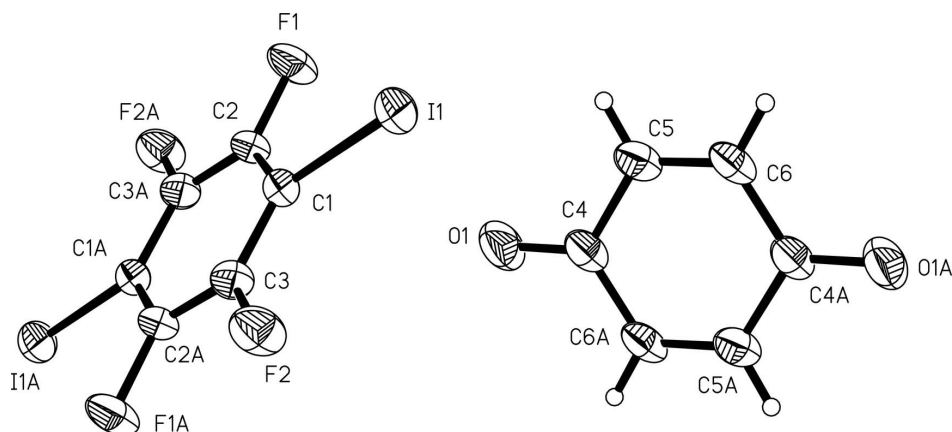
Figure 1

View of the title molecular structure with atom numbering scheme and 30% probability displacement ellipsoids for non-hydrogen atoms.
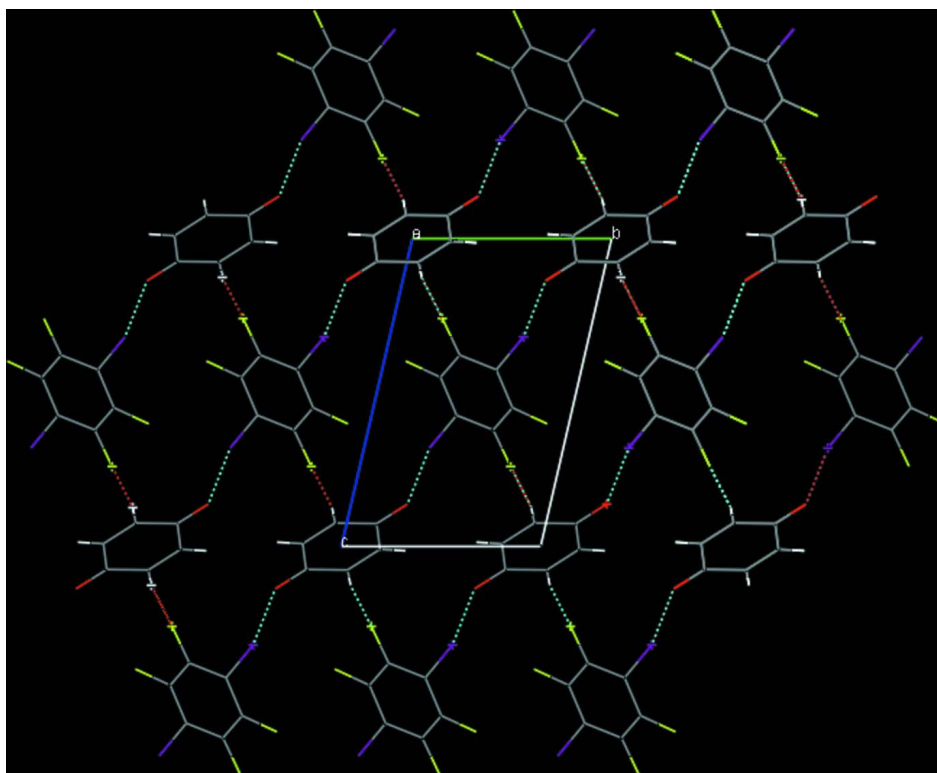
Figure 2

A view in projection down the *b* axis showing the unit-cell contents. The O—I and C—H···F are shown as bule and purple dashed lines.

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Crystal data

$C_6H_4O_2 \cdot C_6F_4I_2$	Triclinic, $P\overline{1}$
$M_r = 509.95$	Hall symbol: -P 1

$a = 5.778$ (3) Å
$b = 6.354$ (3) Å
$c = 10.013$ (5) Å
$\alpha = 102.295$ (5)°
$\beta = 93.861$ (5)°
$\gamma = 97.781$ (5)°
$V = 354.1$ (3) Å^3
$Z = 1$
$F(000) = 234$

$D_x = 2.391$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1573 reflections
$\theta = 3.3–25.5°$
$\mu = 4.48$ mm^{-1}
$T = 296$ K
Block, yellow
$0.43 \times 0.30 \times 0.26$ mm

Data collection

Bruker APEXII CCD
  diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan
  (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.249$, $T_{max} = 0.389$

2585 measured reflections
1291 independent reflections
1096 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.5°$, $\theta_{min} = 3.3°$
$h = -6 \rightarrow 6$
$k = -7 \rightarrow 7$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.07$
1291 reflections
91 parameters
0 restraints
Primary atom site location: structure-invariant
  direct methods

Secondary atom site location: difference Fourier
  map
Hydrogen site location: inferred from
  neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.34$ e Å^{-3}
$\Delta\rho_{min} = -0.76$ e Å^{-3}

Special details

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.3224 (8)	0.5732 (8)	0.4260 (5)	0.0453 (11)
C2	0.3930 (9)	0.3726 (8)	0.3786 (5)	0.0466 (11)
C3	0.4339 (9)	0.6981 (8)	0.5494 (5)	0.0501 (12)
C4	0.5734 (10)	0.8493 (9)	0.0745 (6)	0.0593 (14)
C5	0.3440 (10)	0.8029 (10)	−0.0092 (6)	0.0652 (15)
H5	0.2464	0.6719	−0.0141	0.078*
C6	0.2763 (10)	0.9415 (10)	−0.0761 (6)	0.0612 (14)
H6	0.1296	0.9086	−0.1263	0.073*
F1	0.2901 (6)	0.2428 (6)	0.2593 (3)	0.0702 (9)

F2	0.3718 (6)	0.8922 (5)	0.6021 (4)	0.0712 (9)
I1	0.05048 (6)	0.67187 (6)	0.31973 (4)	0.06065 (19)
O1	0.6309 (10)	0.7170 (8)	0.1376 (5)	0.0898 (15)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.045 (3)	0.048 (3)	0.043 (3)	0.0023 (19)	−0.003 (2)	0.014 (2)
C2	0.047 (3)	0.047 (3)	0.039 (3)	−0.002 (2)	−0.007 (2)	0.003 (2)
C3	0.054 (3)	0.045 (3)	0.047 (3)	0.003 (2)	0.001 (2)	0.004 (2)
C4	0.064 (3)	0.058 (3)	0.051 (3)	0.022 (3)	−0.013 (3)	0.000 (3)
C5	0.062 (3)	0.068 (4)	0.055 (3)	0.004 (3)	−0.011 (3)	0.001 (3)
C6	0.054 (3)	0.069 (4)	0.053 (3)	0.020 (3)	−0.017 (2)	−0.002 (3)
F1	0.076 (2)	0.067 (2)	0.0527 (18)	0.0096 (16)	−0.0214 (16)	−0.0097 (15)
F2	0.084 (2)	0.0544 (19)	0.068 (2)	0.0227 (16)	−0.0092 (17)	−0.0052 (16)
I1	0.0554 (3)	0.0678 (3)	0.0610 (3)	0.00885 (17)	−0.00837 (17)	0.02435 (19)
O1	0.108 (4)	0.073 (3)	0.085 (3)	0.027 (3)	−0.035 (3)	0.016 (2)

Geometric parameters (Å, º)

C1—C3	1.381 (7)	C4—O1	1.221 (7)
C1—C2	1.388 (7)	C4—C6^{ii}	1.483 (9)
C1—I1	2.079 (5)	C4—C5	1.478 (8)
C2—F1	1.346 (5)	C5—C6	1.298 (9)
C2—C3^{i}	1.373 (8)	C5—H5	0.9300
C3—F2	1.340 (6)	C6—C4^{ii}	1.483 (9)
C3—C2^{i}	1.373 (8)	C6—H6	0.9300
C3—C1—C2	116.9 (5)	O1—C4—C6^{ii}	123.3 (5)
C3—C1—I1	122.0 (4)	O1—C4—C5	119.7 (6)
C2—C1—I1	121.0 (4)	C6^{ii}—C4—C5	117.0 (5)
F1—C2—C3^{i}	118.8 (5)	C6—C5—C4	121.2 (6)
F1—C2—C1	119.7 (4)	C6—C5—H5	119.4
C3^{i}—C2—C1	121.6 (4)	C4—C5—H5	119.4
F2—C3—C2^{i}	118.5 (4)	C5—C6—C4^{ii}	121.8 (5)
F2—C3—C1	120.0 (5)	C5—C6—H6	119.1
C2^{i}—C3—C1	121.6 (5)	C4^{ii}—C6—H6	119.1
C3—C1—C2—F1	179.7 (5)	C2—C1—C3—C2^{i}	0.2 (8)
I1—C1—C2—F1	2.5 (7)	I1—C1—C3—C2^{i}	177.4 (4)
C3—C1—C2—C3^{i}	−0.2 (8)	O1—C4—C5—C6	179.1 (6)
I1—C1—C2—C3^{i}	−177.4 (4)	C6^{ii}—C4—C5—C6	−1.2 (10)
C2—C1—C3—F2	−179.0 (5)	C4—C5—C6—C4^{ii}	1.3 (10)
I1—C1—C3—F2	−1.9 (7)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

| C6—H6···F1[iii] | 0.93 | 2.64 | 3.562 | 171 |

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