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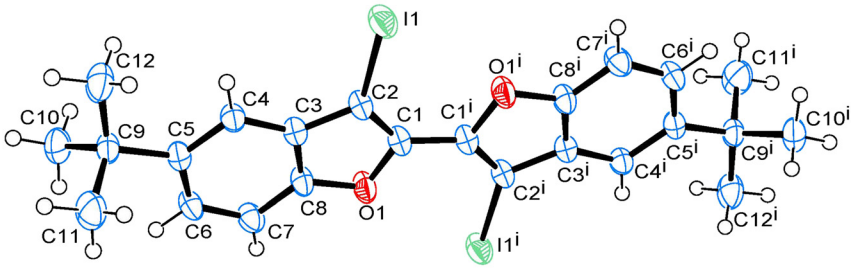
The crystal structure of 5-*tert*-butyl-2-(5-*tert*-butyl-3-iodo-benzofuran-2-yl)-3-iodobenzofuran, $C_{24}H_{24}I_2O_2$



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Abstract

$C_{24}H_{24}I_2O_2$, monoclinic, $P2_1/n$ (no. 14), a = 11.128(2) Å, b = 5.7775(12) Å, c = 17.607(4) Å, β = 98.87(3)°, V = 1118.4(4) Å^3, Z = 2, $R_{gt}(F)$ = 0.0246, w$R_{ref}(F^2)$ = 0.0537, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.23 × 0.21 × 0.19 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	2.83 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.9°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6656, 2641, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 $\sigma(I_{obs})$, 2189
$N(param)_{refined}$:	130
Programs:	Bruker [1], Shelx [2, 3]

1 Source of materials

The 1,4-bis(5-*tert*-butyl-2-methoxyphenyl)buta-1,3-diyne (synthesized by the literature reported procedure [4]) (1.20 g, 3.20 mmol) was dissolved in dry dichloromethane (5 mL), and a dichloromethane solution (20 mL) of I_2 (3.26 g, 12.82 mmol) was added dropwise to the reaction mixture at room temperature. Reaction progress was detected by TLC for 10 h, the reaction was quenched with saturated sodium sulfite solution (40 mL), extracted with dichloromethane (50 mL × 3), dried with anhydrous magnesium sulfate, filtered and concentrated to obtain crude residues. The yellow solid, namely, 5-*tert*-butyl-2-(5-*tert*-butyl-3-iodobenzofuran-2-yl)-3-iodobenzofuran (1.72 g, 90 %), was obtained by silica gel column chromatography. Melting point: 175–176 °C. Crystals of the title compound were obtained in a mixture of dichloromethane and diethyl ether. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.50–7.46 (*m*, 3H), 1.42 (*d*, *J* = 1.2 Hz, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 152.66, 147.53, 146.38, 131.12, 125.03, 118.37, 111.12, 66.20, 35.08, 31.93, 29.85.

2 Experimental details

The crystal structure was solved using the Shelxs [2] structure solution program and refined with the Shelxl [3].

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5017 (2)	0.4394 (5)	−0.03552 (13)	0.0322 (6)
C2	0.5701 (2)	0.2659 (5)	−0.05807 (13)	0.0308 (6)
C3	0.5269 (2)	0.2237 (5)	−0.13819 (13)	0.0296 (5)
C4	0.5602 (2)	0.0753 (5)	−0.19381 (13)	0.0300 (5)
H4	0.6228	−0.0311	−0.1810	0.036*
C5	0.4987 (2)	0.0881 (5)	−0.26852 (13)	0.0297 (5)
C6	0.4031 (2)	0.2460 (5)	−0.28461 (13)	0.0349 (6)
H6	0.3616	0.2527	−0.3346	0.042*
C7	0.3667 (2)	0.3926 (5)	−0.23064 (14)	0.0366 (6)
H7	0.3017	0.4943	−0.2426	0.044*
C8	0.4330 (2)	0.3786 (5)	−0.15752 (14)	0.0326 (6)
C9	0.5366 (3)	−0.0552 (5)	−0.33396 (14)	0.0342 (6)
C10	0.4253 (3)	−0.1699 (6)	−0.38181 (16)	0.0481 (8)
H10A	0.3838	−0.2631	−0.3489	0.072*
H10B	0.4514	−0.2658	−0.4207	0.072*
H10C	0.3713	−0.0524	−0.4057	0.072*
C11	0.5951 (3)	0.1080 (6)	−0.38652 (18)	0.0551 (9)
H11A	0.5367	0.2217	−0.4080	0.083*
H11B	0.6215	0.0203	−0.4273	0.083*
H11C	0.6638	0.1839	−0.3572	0.083*
C12	0.6275 (3)	−0.2438 (6)	−0.30448 (16)	0.0486 (8)
H12A	0.7011	−0.1741	−0.2790	0.073*
H12B	0.6451	−0.3353	−0.3469	0.073*
H12C	0.5936	−0.3409	−0.2689	0.073*
I1	0.70674 (2)	0.07277 (3)	0.00468 (2)	0.04253 (7)
O1	0.41526 (17)	0.5130 (3)	−0.09584 (9)	0.0373 (4)

All hydrogen atoms were placed in calculated positions and refined as riding on their parent atom.

3 Comment

3,3′-Diiodo-2,2′-bibenzofuran derivatives are an important class of organic reaction intermediates [4] and used to synthesize heteropentacenes with π-conjugated systems in material science [5–7]. In this paper, a *para*-*tert*-butyl substituted 3,3′-diiodo-2,2′-bibenzofuran derivative, namely, 5-*tert*-butyl-2-(5-*tert*-butyl-3-iodobenzofuran-2-yl)-3-iodobenzofuran was synthesized by using the iodine electrophilic cyclization reaction of 1,4-bis(2-methoxy-3,5-di*tert*-butylphenyl)-1,3-diyne with I_2 [4].

The crystal structure of the title compound indicates that it is a centrosymmetric structure with the inversion center of the C–C bond as the center of symmetry. Its molecular structure contains two benzofuran rings, two iodine atoms and two *tert*-butyl groups. As illustrated in the figure, an anti-arrangement of two benzofuran moieties and two iodine atoms were observed. In the crystal structure, the carbon-carbon double bond length of C1–C2 is 1.355(4) Å, which is typical for carbon-carbon double bond [8]. The bond lengths of O1–C1 and O1–C8 are 1.385(3) and 1.374(3) Å, respectively. The bond distance of C2–I1 is 2.064(2) Å. These bond lengths are within the normal range [9]. In addition, the plane (C3/C4/C8) of benzene ring and the plane (C1/C2/O1) of furan ring are approximately coplanar and their dihedral angle is only 0.81°, which is similar to the reported related structure containing a benzofuran moiety [9]. The two furan rings lie in a highly coplanar arrangement with the dihedral angle of about 0.81° by connection of C1–C1^i.

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