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X-ray Structure Analysis Online

Crystal Structure of 1,2,3-Tribromobenz[*f*]indane

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The title compound, $C_{13}H_9Br_3$, crsytalizes in the triclinic $P\bar{1}$ space group. The unit-cell parameters at room temperature are a = 6.371(3), b = 9.145(3), c = 10.731(10)Å, α = 91.27(5), β = 99.83(6), γ = 97.49(3)°, V = 610.2(7)Å^3, D_x = 1.314 g/cm^3, Z = 2. The crystal structure has non-classical hydrogen bonding interactions.

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Benz[*f*]indenonens and benz[*f*]indenes are potentially useful building blocks in organic and organometallic synthesis. However, their chemistry has not been much explored. Recently, benz[*f*]indenones were used as important starting materials in the synthesis of kinamycin antibiotics, which showed excellent activity against gram-positive bacteria.[1] The use of benz[*f*]indene as a cyclopentadienyl ligand attracted much attention because an annulated benzo ring might increase both the stereocontrol and the produtivity of catalytic system. Lately, benz[*f*]indenones and benz[*f*]indenes were used as target molecules for computational chemistry.[2] The zirconocene compounds of benz[*f*]indenes are used as catalysts in ethylene polymerization.[3] The bromination of hydrocarbons is an important process, since it leads to useful intermediates for the synthesis of a large variety of bromoorganic compounds.[4-6] Therefore, we herein report on the synthesis and the crystal structure of the title compound, 1,2,3-tribromobenz[*f*]indane (Fig. 1).

To a magnetically stirred solution of benz[*f*]indene (0.2 g, 1.2 mmol) in CH_2Cl_2 (10 ml) cooled to 0°C (ice bath) was added dropwise a solution of bromine (0.21 g, 1.3 mmol) in CH_2Cl_2 (3 ml). The reaction mixture was kept to a refrigerator overnight. After removing the solvent *in vacuo*, the crude product was submitted to column chromatography (silica gel) eluting with hexane to give the product (97 mg, 20%). The structure of tribromobenz[*f*]indane was elucidated by single-crystal X-ray diffraction.

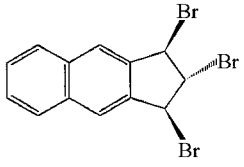


Fig. 1 Chemical diagram.

A summary of the crystallographic information is given in Table 1. The data were collected on a CrysAlis CCD, Oxford Diffraction Ltd. with an imaging-plate detector diffractometer using graphite-monochromated Mo K_α radiation with the ω scan mode. The collected data were reduced by using CrysAlis RED; also, an empirical absorption correction was applied using spherical harmonics, implemented in the SCALE3 ABSPACK scaling algorithm. The title compound was solved by direct methods and refined by a full-matrix least-squares method on

Table 1 Crystal and experimental data

Formula: $C_{13}H_9Br_3$	
Formula weight = 404.90	
Crystal system: triclinic	
Space group: $P\bar{1}$	Z = 2
a = 6.371(3)Å	α = 91.27(5)°
b = 9.145(3)Å	β = 99.83(6)°
c = 10.731(10)Å	γ = 97.49(3)°
V = 610.2(7)Å^3	
D_x = 1.314 g/cm^3	
No. of reflections used = 4146	
$2\theta_{max}$ = 65.6° with Mo K_α	
R = 0.088	
$(\Delta/\sigma)_{max}$ = 0.00	
$(\Delta\rho)_{max}$ = 1.71 e Å^{-3}	
$(\Delta\rho)_{min}$ = –0.75 e Å^{-3}	
Data collection: CrysAlis CCD, Oxford Diffraction Ltd.	
Cell refinement: CrysAlis RED, Oxford Diffraction Ltd.	
Data reduction: CrysAlis RED	
Absorption correction: CrysAlis RED, empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	
Structure determination: direct methods (SIR-97)	
Refinement: full matrix least-squares method (SHELXL-97)	
CCDC deposit number: 638476	

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Table 2 Atomic coordinates and temperature factors

Atom	*x*	*y*	*z*	$U_{(eq)}$[Å^2]
Br1	0.62218(13)	0.69964(11)	0.44748(8)	0.0461(3)
Br2	-0.02212(13)	0.43187(10)	0.28461(10)	0.0487(3)
Br3	0.16510(16)	0.91847(10)	0.39796(9)	0.0504(3)
C1	0.3833(11)	0.6689(8)	0.1970(7)	0.031(2)
C2	0.3902(12)	0.5944(9)	0.3192(7)	0.035(2)
C3	0.1732(12)	0.6095(9)	0.3544(8)	0.037(3)
C4	0.0965(12)	0.7403(9)	0.2856(8)	0.036(2)
C5	0.2128(11)	0.7534(8)	0.1755(7)	0.034(2)
C6	0.1721(12)	0.8253(8)	0.0705(7)	0.034(2)
C7	0.3051(12)	0.8209(9)	-0.0219(7)	0.036(2)
C8	0.2707(15)	0.8971(10)	-0.1349(8)	0.043(3)
C9	0.3976(16)	0.8910(10)	-0.2240(8)	0.050(3)
C10	0.5706(15)	0.8093(10)	-0.2009(8)	0.045(3)
C11	0.6096(13)	0.7332(10)	-0.0956(8)	0.042(3)
C12	0.4784(11)	0.7375(8)	-0.0013(7)	0.032(2)
C13	0.5114(12)	0.6603(9)	0.1085(8)	0.037(3)

$U_{(eq)}$ = 1/3 of the trace of the orthogonalized *U* tensor.

Table 3 Selected bond distances (Å) and angles (°)

Br1 - C2	1.970(8)	C5 - C6	1.322(10)
Br2 - C3	1.962(8)	C6 - C7	1.413(11)
Br3 - C4	1.961(9)	C7 - C8	1.411(12)
C1 - C2	1.488(11)	C7 - C12	1.412(11)
C1 - C5	1.404(10)	C8 - C9	1.358(13)
C1 - C13	1.361(11)	C9 - C10	1.402(14)
C2 - C3	1.516(11)	C10 - C11	1.344(12)
C3 - C4	1.511(12)	C11 - C12	1.421(11)
C4 - C5	1.497(11)	C12 - C13	1.386(11)
C2 - C1 - C5	111.3(6)	C4 - C5 - C6	129.9(7)
C2 - C1 - C13	128.9(7)	C5 - C6 - C7	119.9(7)
C5 - C1 - C13	119.8(7)	C6 - C7 - C8	122.2(7)
Br1 - C2 - C1	109.8(5)	C6 - C7 - C12	119.3(7)
Br1 - C2 - C3	110.1(5)	C8 - C7 - C12	118.5(7)
C1 - C2 - C3	103.5(6)	C7 - C8 - C9	121.9(9)
Br2 - C3 - C2	106.9(5)	C8 - C9 - C10	118.7(8)
Br2 - C3 - C4	108.0(5)	C9 - C10 - C11	122.0(8)
C2 - C3 - C4	106.1(7)	C10 - C11 - C12	120.3(8)
Br3 - C4 - C3	110.4(6)	C7 - C12 - C11	118.6(7)
Br3 - C4 - C5	111.9(5)	C7 - C12 - C13	118.9(7)
C3 - C4 - C5	104.7(6)	C11 - C12 - C13	122.5(7)
C1 - C5 - C4	108.5(6)	C1 - C13 - C12	120.5(7)
C1 - C5 - C6	121.6(7)		

F^2. All H atoms were located geometrically and constrained to ride on their parent atoms with C–H(aromatic) = 0.93 and C–H(methine) = 0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(C). The final conventional R(F) was 0.088. The highest peak of the residual electron density was at 0.93 Å from Br3, and the largest hole was 1.09 Å from Br1. The atomic coordinates and the equivalent isotropic displacement parameters along with the estimated standard deviations for the other atoms, except for H, are listed in Table 2. An ORTEP-3 drawing of the title compound with the atom numbering is shown in Fig. 2.

In the title compound, the almost envelope-type conformation on C3 of the fivemembered ring (C1–C5) was evidenced by the puckering parameters (Cremer & Pople, 1975), Q_2 = 0.238(9)Å and φ_2 = 254(2)°. The mean plane of the two fused-six membered rings is essentially planar, with the maximum deviations for atoms C13, C10 and C5 being –0.017(8), 0.014(9) and 0.011(7)Å, respectively.

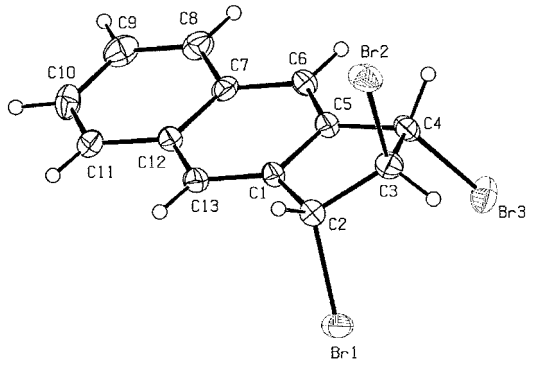


Fig. 2 ORTEP view of the title molecule, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

The C–C–Br angles are in the range 106.9(5) – 111.9(5)°. The C–Br bonds are in the range 1.961(9) – 1.970(8)Å (Table 1). Repulsive interactions between the Br atoms may affect the molecular structure. The values of the other geometric parameters in the title compound are normal.

The intermolecular interaction between Br2···C8^{i} [3.437(10)Å] and Br2···C9^{i} [3.522(10)Å] [symmetry code: (i) –*x*, 1–*y*, –*z*] between two molecules around the inversion center is noteworthy. Dipole–dipole and van der Waals interactions are effective in the molecular packing of the title compound.

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