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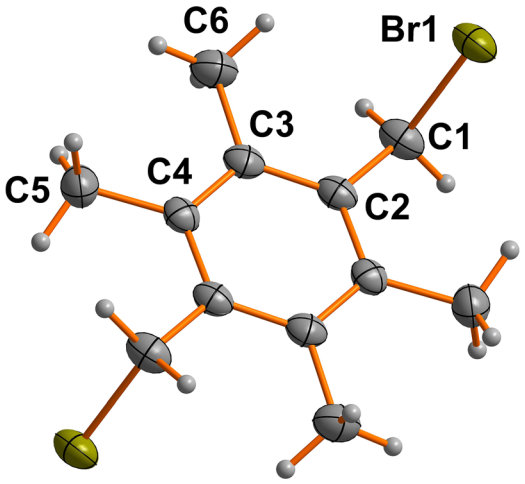
Crystal structre of 1,4-bis(bromomethyl)-2,3,5,6-tetramethylbenzene, $C_{12}H_{16}Br_2$



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Abstract

$C_{12}H_{16}Br_2$, monoclinic, $P2_1/c$ (no. 14), a = 7.9423(5) Å, b = 4.4163(3) Å, c = 16.8127(10) Å, β = 94.534(4)°, V = 587.87(6) Å^3, Z = 2, $R_{gt}(F)$ = 0.0376, $wR_{ref}(F^2)$ = 0.1016, T = 173 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:	Block
Size:	0.12 × 0.11 × 0.10 mm
Wavelength:	Ga $K\alpha$ radiation (1.34139 Å)
μ:	5.57 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE Metaljet PHOTON II, φ and ω
θ_{max}, completeness:	60.3°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4095, 1333, 0.039
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1066
$N(param)_{refined}$:	66
Programs:	Bruker [1], Olex2 [2, 3], SHELX [4, 5]

Source of material

1.4-Bis(bromomethyl)-2,3,5,6-tetramethylbenzene was synthesized and carried out according to the literature [6]. In brief, 13.42 g (0.1 mol) of durene were first dissolved in 50 mL of hot glacial acetic acid before paraformaldehyde 6.15 g (0.20 mol) and 40 mL of a 31% HBr/acetic acid solution were added. The mixture was kept for 8 h at 120 °C and then poured into 100 mL of water. The product was filtered off on a G3 glass frit and dried in vacuum.

Experimental details

All the H atoms were placed geometrically and refined without any constraints or restraints.

Comment

Bromomethyl-substituted benzene derivatives are important synthetic materials for construction of heterocyclic frameworks which have been extensively and widely used [6]. 1.4-Bis(bromomethyl)-2,3,5,6-tetramethylbenzene was one of the used compounds that is important. For example, 1.4-bis(bromomethyl)-2,3,5,6-tetramethylbenzene can be used for the construction of a new organic piperidine derivative named as 4-methyl-1-({2,3,5,6-tetramethyl-4-[(4-methylpiperidinyl) methyl]phenyl}methyl)piperidine, which possesses better antidiabetic, antioxidant and DNA binding abilities [7]. Similarly, Caroline Daisy et al. reported the synthesis of the biologically important 2-(2,4,6-trimethylbenzylthio)benzo[*d*]

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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}
Br1	0.86356 (5)	0.34688 (10)	0.68181 (2)	0.03993 (19)
C1	0.6912 (5)	0.1198 (8)	0.6139 (2)	0.0344 (8)
H1A	0.747474	−0.040050	0.584292	0.041*
H1B	0.611904	0.020101	0.648147	0.041*
C2	0.5945 (4)	0.3245 (8)	0.5557 (2)	0.0277 (7)
C3	0.6510 (4)	0.3664 (8)	0.4794 (2)	0.0291 (7)
C4	0.5549 (4)	0.5378 (8)	0.42288 (19)	0.0281 (7)
C5	0.6072 (5)	0.5692 (11)	0.3389 (2)	0.0410 (9)
H5A	0.507074	0.605676	0.302307	0.061*
H5B	0.662945	0.382554	0.323535	0.061*
H5C	0.685578	0.739705	0.336443	0.061*
C6	0.8168 (5)	0.2266 (10)	0.4600 (2)	0.0399 (9)
H6A	0.899374	0.245834	0.506158	0.060*
H6B	0.858855	0.331573	0.414151	0.060*
H6C	0.799434	0.011979	0.447000	0.060*

thiazole by using 1.4-bis(bromomethyl)-2,3,5,6-tetramethylbenzene [8]. Liu et al. reported two macrocyclic bis-benzimidazolium salts based sensors for anions sensing prepared by using 1.4-bis(bromomethyl)-2,3,5,6-tetramethylbenzene [9]. However, there is no report on crystal structure of the title compound.

The title compound crystalizes in the monoclinic space group $P2_1/c$. The asymmetric unit is defined by one half of the title molecule. The molecules reside around an inversion center of the aforementioned space group (see the Figure). Bond lengths and angles in crystal structure are within normal ranges [10].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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