Nianyuan Tan*, Shuisheng Wu, Yang Huiqiong, Donghui Lan, Chak-Tong Au and Bing Yi*

Crystal structure of (2-bromobenzyl)((1-bromonaphthalen-2-yl)methyl)sulfane, $C_{18}H_{14}Br_2S$

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Abstract

$C_{18}H_{14}Br_2S$, triclinic, $P\bar{1}$ (no. 2), $a = 7.1658(10)$ Å, $b = 7.6868(11)$ Å, $c = 14.971(2)$ Å, $\alpha = 76.328(3)^\circ$, $\beta = 84.313(3)^\circ$, $\gamma = 81.952(3)^\circ$, $V = 791.51(19)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0436$, $wR_{ref}(F^2) = 0.1237$, $T = 296(2)$ 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.

Source of material

The title compound was prepared according to a reported procedure [5]: a solution of (2-bromophenyl)methanethiol (15 mmol) in THF was added dropwise to a suspension of NaH (18 mmol) in THF at 0 °C and stirred for 2 h. Then, a solution of 1-bromo-2-(bromomethyl)naphthalene (15 mmol) in THF was added. The mixture was heated at reflux for 4 h. After cooling, the mixture was diluted with Et₂O and quenched with water. The organic layer was separated, washed with brine, and dried over anhydrous Na₂SO₄. The solvent was removed

Table 1: Data collection and handling.

Crystal:	Colourless prismatic
Size:	0.28 × 0.21 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	5.24 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	26.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	4745, 3073, 0.030
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2452
$N(param)_{refined}$:	190
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

under reduced pressure and the residue was recrystallized from CH₂Cl₂–hexane to give the title compound.

Experimental details

All H atoms were generated geometrically and included in the final stages of the refinement using the riding model, with C–H = 0.93 Å for aryl and 0.97 Å for methylene H atoms, respectively. $U_{iso}(H)$ set to $1.2U_{eq}(C)$ for all H atoms.

Comment

Organosulfur compounds have received widespread attention because of their various applications in areas such as medicine, agriculture, materials and organic synthesis [5–12]. Most notably among these are thioethers (compounds containing C–S–C bonds), which have been often used as valuable precursors in organic synthesis [6], as ligands in organometallic chemistry [5, 7] and in catalysis [8]. To our best knowledge, the crystal structures of the organosulfur compounds containing C–S–C bonds are less reported [9–12]. We herein report the crystal structure of an asymmetric benzyl thioether.

The bond lengths and angles in the title compound are within the expected ranges. The S–C bond lengths [S1–C7 1.818(4), S1–C8 1.814(4) Å] are comparable with the S–C(benzyl) bond lengths in the previously reported organosulfur compounds with the S-benzyl group, such as 2-benzylsulfanyl-benzoic acid benzyl ester [S–C 1.797(3) Å] [9], 2,2'-bis(benzylthio)-1,1'-biphenyl [S–C 1.822(3) Å] [10], benzyl 2-(benzylsulfanyl)benzoate [S–C 1.8159(18) Å] [11] and [2-(4,4-dimethyl-2-oxazolonyl)phenyl] benzyl sulfide [S–C 1.822(2) Å] [12]. The C8–S1–C7 angle is 99.49(19)°

*Corresponding authors: Nianyuan Tan and Bing Yi, Hunan Provincial Key Laboratory of Environmental Catalysis and Waste Regeneration, Hunan Institute of Engineering, Xiangtan 411104, China, e-mail: nytan@sina.com (N. Tan); bingyi2005@126.com (B. Yi)

Shuisheng Wu, Donghui Lan and Chak-Tong Au: Hunan Provincial Key Laboratory of Environmental Catalysis and Waste Regeneration, Hunan Institute of Engineering, Xiangtan 411104, China

Yang Huiqiong: College of Chemistry and Chemical Engineering, Hunan Institute of Engineering, Xiangtan 411104, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	−0.28526(7)	0.69476(6)	0.94958(3)	0.06198(19)
Br2	0.72489(7)	0.73202(7)	0.68654(3)	0.06452(19)
S1	0.19380(15)	0.68900(13)	0.80588(7)	0.0508(3)
C1	−0.2055(5)	0.9277(5)	0.9053(2)	0.0428(8)
C2	−0.3432(6)	1.0735(5)	0.8915(3)	0.0503(10)
H2	−0.470105	1.056329	0.902675	0.060*
C3	−0.2923(7)	1.2448(6)	0.8610(3)	0.0575(11)
H3	−0.384673	1.343727	0.850233	0.069*
C4	−0.1029(7)	1.2695(6)	0.8463(3)	0.0559(11)
H4	−0.067528	1.384980	0.826199	0.067*
C5	0.0329(6)	1.1217(6)	0.8618(3)	0.0503(10)
H5	0.159803	1.139402	0.852551	0.060*
C6	−0.0148(5)	0.9465(5)	0.8909(2)	0.0404(8)
C7	0.1381(6)	0.7895(6)	0.9052(3)	0.0485(9)
H7A	0.099003	0.697911	0.957681	0.058*
H7B	0.251509	0.829467	0.919876	0.058*
C8	0.2951(7)	0.8737(5)	0.7258(3)	0.0599(12)
H8A	0.204136	0.981259	0.718446	0.072*
H8B	0.406137	0.898562	0.750423	0.072*
C9	0.3484(5)	0.8291(5)	0.6341(3)	0.0435(9)
C10	0.2055(6)	0.8515(5)	0.5728(3)	0.0541(10)
H10	0.082264	0.890909	0.591329	0.065*
C11	0.2421(6)	0.8176(5)	0.4879(3)	0.0536(10)
H11	0.144289	0.834683	0.449119	0.064*
C12	0.4262(5)	0.7569(5)	0.4575(3)	0.0433(8)
C13	0.4683(7)	0.7190(6)	0.3696(3)	0.0582(11)
H13	0.371381	0.734370	0.330439	0.070*
C14	0.6436(8)	0.6615(6)	0.3410(3)	0.0657(13)
H14	0.667161	0.636337	0.282819	0.079*
C15	0.7914(7)	0.6394(6)	0.3982(3)	0.0667(14)
H15	0.913557	0.601349	0.377540	0.080*
C16	0.7578(6)	0.6732(5)	0.4847(3)	0.0527(10)
H16	0.857257	0.656004	0.522605	0.063*
C17	0.5746(5)	0.7337(4)	0.5168(2)	0.0384(8)
C18	0.5271(5)	0.7692(5)	0.6050(2)	0.0397(8)

(non-linear geometry around the sulfur atom). The torsion angle of C8–S1–C7–C6 and C7–S1–C8–C9 are 67.0(3)° and −177.8(3)°, respectively.

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