Crystal structure of 2,4-bis((4-bromophenoxy)methyl)-1,3,5-trimethylbenzene, $C_{23}H_{22}Br_2O_2$

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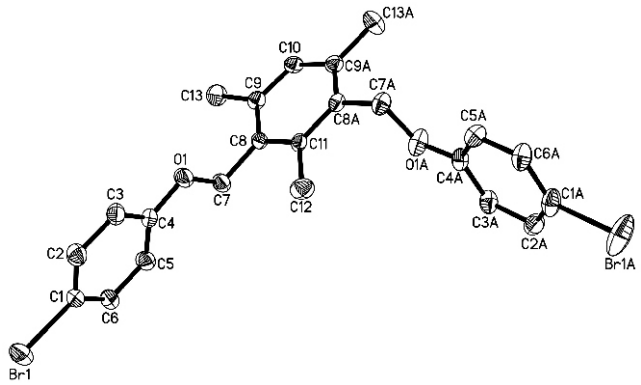
Received March 6, 2011, accepted and available on-line July 12, 2011; CCDC no. 1267/3427



Abstract

$C_{23}H_{22}Br_2O_2$, monoclinic, *C*121 (no. 5), a = 8.6594(7) Å, b = 7.9730(6) Å, c = 15.1266(8) Å, β = 94.539(6)°, V = 1041.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.037, $wR_{ref}(F^2)$ = 0.073, T = 293 K.

Source of material

All chemicals were purchased commercially and used without purification. 2,4-bis(bromomethyl)-1,3,5-trimethylbenzene was prepared according to reference [1]. The title compound was synthesized by the following procedure. A mixture of 4-bromophenol (3.46 g, 0.02 mol), 2,4-bis(bromomethyl)-1,3,5-trimethylbenzene(3.06 g, 0.01 mol), K_2CO_3 (5.52 g, 0.04 mol) and *N*,*N*-dimethylformamide (30 mL) was heated at 80 °C for 12 hours. Then the mixture was poured into 100 mL cold water. The white precipitate obtained was filtered and dried. Crystals of the title compound were obtained by slow evaporation of the methanol solution (yield 41%).

Experimental details

All hydrogen atoms on carbons were generated at idealized positions with d(C—H) = 0.93 - 0.97 Å, and U_{iso}(H) = 1.2U_{eq}(C) for CH_2 groups and U_{iso}(H) = 1.5U_{eq}(C) for CH_3 groups. The occupations for H11 positions were fixed to 0.50.

Discussion

The asymmetric unit of the title crystal structure contains a half 2,4-bis((4-bromophenoxy)methyl)-1,3,5-trimethylbenzene molecule. In oder to stabilize the whole structure, the two 4-bromophenoxy groups were loctated above and below the plane of the central benzene ring. The dihedral angles between the benzene ring of the 4-bromophenoxy groups and the central benzene ring are 72.17°.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.15 × 0.20 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	39.08 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.18°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2374, 1868
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1149
$N(param)_{refined}$:	124
Program:	SHELXS-97 [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(2)	4*c*		0.4771	0.7944	0.4016	0.058
H(3)	4*c*		0.4109	0.5921	0.2943	0.056
H(5)	4*c*		0.8546	0.5465	0.2450	0.056
H(6)	4*c*		0.9221	0.7461	0.3506	0.062
H(7A)	4*c*		0.7519	0.2922	0.1711	0.055
H(7B)	4*c*		0.7326	0.4561	0.1134	0.055
H(11A)	4*c*	0.50	0.5624	0.5874	0.0509	0.084
H(11B)	4*c*	0.50	0.3958	0.5874	0.0021	0.084
H(11C)	4*c*	0.50	0.5417	0.5874	–0.0530	0.084
H(13A)	4*c*		0.6425	–0.1260	0.1321	0.091
H(13B)	4*c*		0.6196	0.0199	0.1993	0.091
H(13C)	4*c*		0.7695	0.0133	0.1477	0.091
H(10)	2*a*		½	–0.1036	0	0.046

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	4*c*	0.75836(8)	0.95540(8)	0.47227(4)	0.1270(5)	0.0854(4)	0.0764(4)	–0.0539(4)	0.0420(3)	–0.0441(4)
C(1)	4*c*	0.7066(6)	0.7902(6)	0.3845(3)	0.076(4)	0.045(3)	0.036(3)	–0.022(3)	0.007(3)	–0.005(2)
C(2)	4*c*	0.5526(5)	0.7450(5)	0.3697(3)	0.056(3)	0.046(3)	0.044(3)	0.003(2)	0.010(2)	–0.006(2)
C(3)	4*c*	0.5140(5)	0.6233(6)	0.3059(3)	0.041(2)	0.064(3)	0.035(3)	–0.006(2)	0.004(2)	–0.002(2)
C(4)	4*c*	0.6251(5)	0.5490(5)	0.2599(3)	0.049(3)	0.045(2)	0.029(3)	–0.007(2)	0.005(2)	0.001(2)
C(5)	4*c*	0.7787(5)	0.5961(5)	0.2765(3)	0.048(3)	0.045(3)	0.048(3)	–0.003(2)	0.009(2)	–0.009(2)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	4*c*	0.8188(5)	0.7156(6)	0.3390(3)	0.055(3)	0.060(3)	0.040(3)	–0.014(2)	0.005(3)	–0.010(2)
C(7)	4*c*	0.6770(5)	0.3654(5)	0.1396(3)	0.045(3)	0.051(3)	0.041(3)	–0.007(2)	0.006(2)	–0.011(2)
C(8)	4*c*	0.5846(4)	0.2691(5)	0.0685(3)	0.033(2)	0.038(2)	0.032(2)	–0.003(2)	0.007(2)	–0.005(2)
C(9)	4*c*	0.5802(5)	0.0945(5)	0.0702(3)	0.047(3)	0.032(2)	0.041(3)	0.004(2)	0.008(2)	0.002(2)
C(11)	2*a*	½	0.3567(8)	0	0.052(4)	0.028(3)	0.029(4)	0	0.010(3)	0
C(12)	2*a*	½	0.5473(8)	0	0.065(5)	0.037(4)	0.066(6)	0	0.011(4)	0
C(13)	4*c*	0.6603(5)	–0.0090(7)	0.1441(3)	0.067(3)	0.058(4)	0.054(3)	–0.003(3)	–0.012(2)	0.015(3)
C(10)	2*a*	½	0.0131(6)	0	0.044(4)	0.024(3)	0.047(4)	0	0.002(3)	0
O(1)	4*c*	0.5732(3)	0.4311(5)	0.1990(2)	0.050(2)	0.071(2)	0.046(2)	–0.020(2)	0.014(2)	–0.018(2)

Acknowledgments. We thank the Science Foundation for Young Teachers of Northeast Normal University (no. 20060304) and the Analysis and Testing Foundation of Northeast Normal University for support.

References

1. Van der Made, A. W.; Van der Made, R. H.: A Convenient Procedure for Bromomethylation of Aromatic Compounds. Selective Mono-, Bis-, or Trisbromomethylation. J. Org. Chem. **58** (1993) 1262-1263.
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.