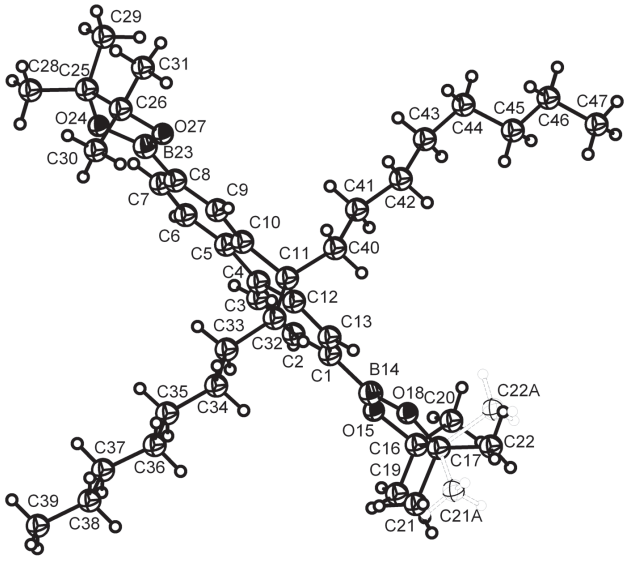
Yingfan Liu, Kaiming Mao, Xiaochuan Li* and Young-A Son

Crystal structure of bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane)-9,9-dioctylfluorene, $C_{41}H_{64}B_2O_4$



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Abstract

$C_{41}H_{64}B_2O_4$, triclinic $P\bar{1}$ (no. 2), $a = 12.8189(8)$ Å, $b = 13.4808(9)$ Å, $c = 14.1592(12)$ Å, $\alpha = 110.394(3)°$, $\beta = 90.170(1)°$; $\gamma = 115.623(3)°$; $V = 2033.5(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0562$, $wR_{ref}(F^2) = 0.1629$, T = 296 K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

***Corresponding author: Xiaochuan Li,** College of Chemistry and Chemical Engineering, Henan Normal University, Xinxiang, Henan, 453007, P.R. China, e-mail: lixiaochuan@htu.cn
Yingfan Liu and Kaiming Mao: College of Material and Chemical Engineering, Zhengzhou University of Light Industry, Henan Provincial Key Lab of Surface and Interface Science, Zhengzhou 450002, P.R. China
Young-A Son: Department of Advanced Organic Materials and Textile System Engineering, Chungnam National University, Daejeon 305-764, South Korea

Table 1: Data collection and handling.

Crystal:	Block, colourless
Size:	0.10 × 0.17 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.06 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	20.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	40607, 8001, 0.093
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4054
$N(param)_{refined}$:	445
Programs:	Bruker programs [1], SHELX [2], DIAMOND [3]

Source of material

The title structure was synthesized according to published procedures [4, 5]. The colorless crystals of the title compound were obtained by slow evaporation of a hexane solution at room temperature.

Experimental details

The hydrogen atoms were placed geometrically and refined using a riding model with d(C–H) = 0.93 Å (aromatic), 0.96 Å (-CH_3), 0.97 Å (-CH_2-), $U_{iso}(H) = 1.2\ U_{eq}(C)$ for CH and CH_2 groups or $U_{iso}(H) = 1.5\ U_{eq}(C)$ for CH_3 groups. Two methyl groups bonded to the oxygen atom are disorded.

Discussion

Small fluorescent molecules have been developed due to the easy structural modification and tunable photophysical properties in various functional design, such as molecular machine, logic operation, OLEDs [6–9]. Fluorene is a typical blue fluorescent molecule and used as building block in functional polymers. The title crystal structure was built up from $C_{41}H_{64}B_2O_4$ molecules only. Two eight-membered alkyl chains stretch to both sides of the fluorene ring, which is configured by intramolecular C–H···π interactions: C33–H33A···π^{i1} (C1/C2/C3/C4/C12/C13, i: x, y, x), C33–H33B···π^{i1} (C5/C6/C7/C8/C9/C10), C44–H41A···π^{i1} (C5/C6/C7/C8/C9/C10), C44–H41B···π^{i1} (C1/C2/C3/C4/C12/C13). The H··· centroid distances range from 3.301 to 3.479 Å, which is inside the interval classified by Malone of 2.65–4.00 Å, based on a survey of the Cambridge Structural Database together with semi-empirical and ab initio

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
B14	0.4188(2)	0.3316(3)	0.0820(2)	0.0507(7)
B23	1.1053(2)	0.2639(2)	0.32668(18)	0.0424(6)
C1	0.53085(18)	0.34476(19)	0.13781(15)	0.0428(5)
C2	0.59989(18)	0.44522(19)	0.22562(16)	0.0460(5)
H2	0.5763	0.5042	0.2520	0.055*
C3	0.70155(18)	0.46007(18)	0.27455(15)	0.0436(5)
H3	0.7462	0.5280	0.3329	0.052*
C4	0.73628(16)	0.37159(17)	0.23516(14)	0.0355(5)
C5	0.83877(16)	0.36287(17)	0.26961(13)	0.0345(5)
C6	0.93078(18)	0.43936(18)	0.35139(14)	0.0423(5)
H6	0.9356	0.5117	0.3950	0.051*
C7	1.01530(18)	0.40692(18)	0.36736(15)	0.0432(5)
H7	1.0772	0.4587	0.4226	0.052*
C8	1.01160(17)	0.29945(18)	0.30392(14)	0.0391(5)
C9	0.91865(17)	0.22411(18)	0.21986(15)	0.0401(5)
H9	0.9148	0.1526	0.1752	0.048*
C10	0.83319(16)	0.25494(17)	0.20289(14)	0.0359(5)
C11	0.72402(16)	0.18556(17)	0.11911(14)	0.0367(5)
C12	0.66874(17)	0.26940(17)	0.14778(14)	0.0358(5)
C13	0.56718(17)	0.25651(18)	0.09969(15)	0.0419(5)
H13	0.5225	0.1885	0.0414	0.050*
C16	0.2766(2)	0.3773(2)	0.04381(19)	0.0605(7)
C17	0.2540(3)	0.2544(3)	−0.0353(2)	0.0860(9)
C19	0.1819(3)	0.3770(4)	0.1045(3)	0.1287(14)
H19A	0.1115	0.3528	0.0597	0.193*
H19B	0.1668	0.3222	0.1376	0.193*
H19C	0.2063	0.4557	0.1553	0.193*
C20	0.3073(3)	0.4731(3)	0.0026(3)	0.1307(14)
H20A	0.2410	0.4531	−0.0451	0.196*
H20B	0.3275	0.5478	0.0580	0.196*
H20C	0.3731	0.4795	−0.0317	0.196*
C21	0.1453(7)	0.1481(8)	−0.0010(6)	0.097(2)
H21A	0.1651	0.1669	0.0707	0.145*
H21B	0.1391	0.0709	−0.0402	0.145*
H21C	0.0715	0.1473	−0.0145	0.145*
C21A	0.1502(9)	0.1600(9)	−0.0709(10)	0.149(4)
H21D	0.1168	0.1411	−0.0151	0.224*
H21E	0.1586	0.0936	−0.1177	0.224*
H21F	0.0993	0.1764	−0.1061	0.224*
C22	0.2226(8)	0.2116(10)	−0.1427(6)	0.107(3)
H22A	0.1638	0.2321	−0.1590	0.160*
H22B	0.1918	0.1265	−0.1715	0.160*
H22C	0.2908	0.2472	−0.1706	0.160*
C22A	0.3034(8)	0.2875(9)	−0.1406(6)	0.112(3)
H22D	0.2654	0.3284	−0.1575	0.168*
H22E	0.2851	0.2148	−0.1980	0.168*
H22F	0.3870	0.3376	−0.1240	0.168*
C25	1.2684(2)	0.2753(2)	0.40343(17)	0.0545(6)
C26	1.2018(2)	0.1535(2)	0.31271(17)	0.0534(6)
C28	1.3033(3)	0.2699(3)	0.50190(19)	0.0934(10)
H28A	1.3605	0.2408	0.4937	0.140*
H28B	1.3366	0.3483	0.5553	0.140*
H28C	1.2353	0.2172	0.5198	0.140*
C29	1.3734(2)	0.3652(2)	0.3767(2)	0.0840(9)
H29A	1.4336	0.3401	0.3669	0.126*
H29B	1.3487	0.3696	0.3149	0.126*
H29C	1.4040	0.4423	0.4316	0.126*
C30	1.1272(3)	0.0520(3)	0.3440(3)	0.0944(10)
H30A	1.1772	0.0358	0.3804	0.142*
H30B	1.0768	0.0736	0.3875	0.142*
H30C	1.0801	−0.0180	0.2841	0.142*
C31	1.2774(3)	0.1186(3)	0.2397(2)	0.0857(9)
H31A	1.3318	0.1056	0.2743	0.129*
H31B	1.2282	0.0467	0.1820	0.129*
H31C	1.3203	0.1815	0.2165	0.129*
C32	0.64306(18)	0.06334(18)	0.12105(15)	0.0451(5)
H32A	0.5726	0.0262	0.0700	0.054*
H32B	0.6825	0.0140	0.1002	0.054*
C33	0.6059(2)	0.0619(2)	0.22192(17)	0.0582(6)
H33A	0.5606	0.1055	0.2411	0.070*
H33B	0.6754	0.1021	0.2746	0.070*
C34	0.5324(2)	−0.0643(2)	0.21643(19)	0.0709(7)
H34A	0.4594	−0.1008	0.1687	0.085*
H34B	0.5744	−0.1099	0.1887	0.085*
C35	0.5037(3)	−0.0741(2)	0.3143(2)	0.0841(9)
H35A	0.4543	−0.0360	0.3384	0.101*
H35B	0.5761	−0.0302	0.3643	0.101*
C36	0.4411(3)	−0.2016(2)	0.3098(2)	0.0811(8)
H36A	0.3660	−0.2441	0.2637	0.097*
H36B	0.4879	−0.2416	0.2816	0.097*
C37	0.4203(2)	−0.2088(2)	0.4117(2)	0.0770(8)
H37A	0.3723	−0.1700	0.4392	0.092*
H37B	0.4953	−0.1646	0.4582	0.092*
C38	0.3612(2)	−0.3331(2)	0.4095(2)	0.0750(8)
H38A	0.4097	−0.3718	0.3837	0.090*
H38B	0.2865	−0.3782	0.3624	0.090*
C39	0.3398(3)	−0.3364(3)	0.5130(2)	0.0921(9)
H39A	0.3019	−0.4178	0.5071	0.138*
H39B	0.4136	−0.2935	0.5596	0.138*
H39C	0.2903	−0.2999	0.5383	0.138*
C40	0.75755(18)	0.16616(18)	0.01183(14)	0.0440(5)
H40A	0.8015	0.1211	0.0017	0.053*
H40B	0.6855	0.1170	−0.0388	0.053*
C41	0.8289(2)	0.27638(19)	−0.00976(16)	0.0500(6)
H41A	0.8982	0.3297	0.0435	0.060*
H41B	0.7824	0.3177	−0.0077	0.060*
C42	0.8669(2)	0.2475(2)	−0.11318(16)	0.0537(6)
H42A	0.9119	0.2049	−0.1147	0.064*
H42B	0.7969	0.1938	−0.1656	0.064*
C43	0.9392(2)	0.3519(2)	−0.14103(18)	0.0635(7)
H43A	1.0040	0.4108	−0.0847	0.076*
H43B	0.8908	0.3883	−0.1492	0.076*
C44	0.9887(2)	0.3206(2)	−0.23837(18)	0.0670(7)
H44A	1.0462	0.3942	−0.2427	0.080*
H44B	1.0299	0.2765	−0.2329	0.080*
C45	0.8997(2)	0.2484(2)	−0.33625(17)	0.0636(7)
H45A	0.8659	0.2968	−0.3465	0.076*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H45B	0.8366	0.1800	−0.3286	0.076*
C46	0.9472(2)	0.2041(3)	−0.4314(2)	0.0755(8)
H46A	1.0088	0.2725	−0.4403	0.091*
H46B	0.9825	0.1571	−0.4209	0.091*
C47	0.8568(3)	0.1301(3)	−0.5276(2)	0.1083(12)
H47A	0.8932	0.1055	−0.5842	0.162*
H47B	0.7963	0.0610	−0.5204	0.162*
H47C	0.8227	0.1764	−0.5399	0.162*
O15	0.37965(15)	0.41385(17)	0.11384(14)	0.0836(6)
O18	0.35014(15)	0.23786(16)	−0.00405(13)	0.0765(5)
O24	1.18142(12)	0.31967(13)	0.41708(10)	0.0534(4)
O27	1.12032(13)	0.17268(13)	0.25826(11)	0.0572(4)

molecular orbital calculations [10]. There is no doubt that the fluorine ring is planar with a mean deviation of 0.007 Å. However, the two oxaborolane rings are totally different in configuration. One of the oxaborolane rings is planar with a mean deviation of 0.014 Å. While the other one is folded and the mean deviation is calculated to be 0.108 Å. The above intermolecular interactions pack molecules layer by layer with the plane of fluorine and oxaborolane ring parallel to each other.

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