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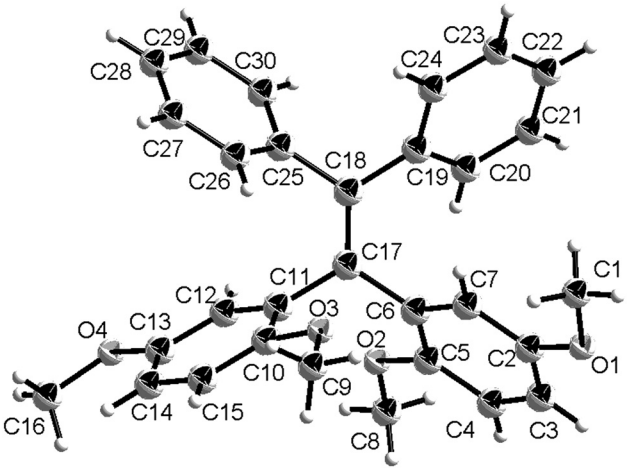
The crystal structure of 2,2′-(2,2-diphenylethene-1,1-diyl)bis(1,4-dimethoxybenzene), $C_{30}H_{28}O_4$



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Abstract

$C_{30}H_{28}O_4$, triclinic, $P\bar{1}$ (no. 2), a = 9.473(14) Å, b = 9.775(14) Å, c = 14.43(2) Å, α = 100.354(17)°, β = 91.053(17)°, γ = 94.062(17)°, V = 1310(1) Å^3, Z = 2, $R_{gt}(F)$ = 0.0517, w$R_{ref}(F^2)$ = 0.1803, 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.

1 Source of material

A mixture of (2,5-dimethoxyphenyl)boronic acid (910 mg, 5.0 mmol), Na_2CO_3 (690 mg, 5.0 mmol) (2,2-dibromoethene-1,1-diyl)dibenzene (336 mg, 1.0 mmol) and tetrakis(triphenylphosphine)palladium (139 mg, 0.12 mmol) in 50 mL 1,4-dioxane/water (v/v = 4:1) in a flask was stirred at 90 °C for 24 h under N_2. After evaporating the solvents, resulting mixture was extracted with dichloromethane (3 × 50 mL) and then washed with water and brine successively. The organic layer was dried over anhydrous Na_2SO_4 and removed in vacuo and the residue was separated by column chromatography on silica gel (eluent: 1:2 DCM/petroleum ether) to give the title compound (285 mg, yield 63 %) as yellow solid. Crystals of the title compound were obtained by slow vapour of solution of title compound in CH_2Cl_2 within 1 week.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.40 × 0.40 × 0.40 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.7°, 98 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,630, 5,562, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3,449
$N(param)_{refined}$:	311
Programs:	Bruker,[1] SHELX[2]

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

In 2001, Tang et al. reported a totally new class of organic fluorophore molecules, which are non-emissive or weakly emissive in solution but display strong fluorescence upon aggregation or in the solid state.[3] This novel photophysical phenomenon is coined the aggregation-induced emission (AIE) effect, and it steers clear of the notorious aggregation-caused quenching (ACQ) problem of conventional fluorophores in a solid emitter.[4–6] Tetraphenylethylene (TPE) is

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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}
O1	0.11892 (16)	0.20842 (15)	0.15378 (10)	0.0884 (5)
O2	0.20897 (13)	0.72726 (14)	0.39574 (10)	0.0766 (4)
O3	0.56330 (13)	0.41804 (12)	0.31448 (8)	0.0654 (3)
O4	0.66003 (16)	0.92739 (13)	0.56554 (9)	0.0844 (4)
C1	0.2182 (3)	0.1652 (3)	0.08190 (18)	0.1082 (8)
H1A	0.229963	0.234497	0.042614	0.162*
H1B	0.182980	0.077919	0.044204	0.162*
H1C	0.307707	0.154518	0.110958	0.162*
C2	0.14620 (19)	0.33887 (19)	0.21215 (12)	0.0636 (5)
C3	0.04142 (19)	0.3805 (2)	0.27478 (13)	0.0718 (5)
H3	−0.040671	0.322575	0.275360	0.086*
C4	0.05751 (18)	0.5086 (2)	0.33718 (13)	0.0687 (5)
H4	−0.014150	0.535477	0.378254	0.082*
C5	0.18367 (17)	0.59799 (18)	0.33787 (12)	0.0566 (4)
C6	0.29186 (15)	0.55657 (17)	0.27501 (10)	0.0491 (4)
C7	0.27081 (17)	0.42688 (17)	0.21213 (11)	0.0550 (4)
H7	0.340830	0.399259	0.169914	0.066*
C8	0.1026 (2)	0.7754 (3)	0.46214 (16)	0.0938 (7)
H8A	0.017660	0.788138	0.428364	0.141*
H8B	0.136998	0.862447	0.500516	0.141*
H8C	0.082517	0.707525	0.501570	0.141*
C9	0.6208 (2)	0.29301 (19)	0.33198 (15)	0.0765 (5)
H9A	0.588648	0.216550	0.282930	0.115*
H9B	0.589864	0.273889	0.391670	0.115*
H9C	0.722301	0.304828	0.333052	0.115*
C10	0.59019 (16)	0.53893 (16)	0.38218 (10)	0.0478 (4)
C11	0.52516 (15)	0.65875 (16)	0.36359 (10)	0.0463 (4)
C12	0.55273 (17)	0.78517 (17)	0.42659 (10)	0.0532 (4)
H12	0.511011	0.864001	0.415017	0.064*
C13	0.64256 (18)	0.79571 (17)	0.50741 (11)	0.0573 (4)
C14	0.70382 (18)	0.67726 (18)	0.52588 (11)	0.0590 (4)
H14	0.761665	0.682814	0.579423	0.071*
C15	0.67782 (17)	0.54937 (17)	0.46325 (11)	0.0545 (4)
H15	0.719143	0.470772	0.475656	0.065*
C16	0.7404 (3)	0.9393 (2)	0.65257 (15)	0.1052 (9)
H16A	0.835791	0.916791	0.639220	0.158*
H16B	0.697972	0.875967	0.689619	0.158*
H16C	0.741002	1.033029	0.686968	0.158*
C17	0.43024 (15)	0.64684 (15)	0.27585 (10)	0.0457 (4)
C18	0.46991 (15)	0.70787 (15)	0.20068 (10)	0.0458 (4)
C19	0.37613 (16)	0.69912 (16)	0.11325 (10)	0.0500 (4)
C20	0.23297 (19)	0.7248 (2)	0.11748 (12)	0.0669 (5)
H20	0.193700	0.752956	0.175807	0.080*
C21	0.1477 (2)	0.7088 (2)	0.03554 (14)	0.0855 (6)
H21	0.052635	0.726556	0.039948	0.103*
C22	0.2044 (3)	0.6662 (2)	−0.05337 (14)	0.0873 (7)
H22	0.146822	0.652885	−0.107634	0.105*
C23	0.3463 (2)	0.6444 (2)	−0.05932 (13)	0.0808 (6)
H23	0.385214	0.618074	−0.117970	0.097*
C24	0.4323 (2)	0.66188 (19)	0.02278 (11)	0.0640 (5)
H24	0.528318	0.648690	0.017623	0.077*
C25	0.61532 (16)	0.78470 (16)	0.19834 (10)	0.0499 (4)
C26	0.74078 (18)	0.7247 (2)	0.21832 (12)	0.0646 (5)
H26	0.734858	0.635603	0.232827	0.078*
C27	0.8739 (2)	0.7957 (3)	0.21691 (15)	0.0823 (6)
H27	0.955040	0.754142	0.230935	0.099*
C28	0.8849 (2)	0.9295 (3)	0.19435 (15)	0.0886 (7)
H28	0.973294	0.977482	0.194593	0.106*
C29	0.7644 (3)	0.9902 (2)	0.17170 (14)	0.0812 (6)
H29	0.771835	1.078382	0.155617	0.097*
C30	0.6290 (2)	0.91769 (17)	0.17307 (13)	0.0677 (5)
H30	0.548408	0.958543	0.157071	0.081*

a simple small molecule with its π electrons in four phenyl rings delocalizing to the double bond. Due to the crowded phenyl substituents, the resultant repulsion makes the phenyl rings deviate from the π-π delocalization plane, resulting in a propeller-like arrangement. TPE derivatives have accounted for more than half of all AIEgen compounds, showing that they are the most quickly developed.[7] In 2001, Miller and coworkers reported the synthesis of 4,4′-(2,2-diphenylethene-1,1-diyl)bis(methoxybenzene) through the Suzuki reaction of (2,2-dibromoethene-1,1-diyl)dibenzene and (4-methoxyphenyl)boronic acid.[8] Bai and coworkers reported the preparation of 2,2′-(2,2-diphenylethene-1,1-diyl)bis(methoxybenzene) by the reaction between 2,2′-dimethyloxybenzophenone and the lithiated diphenylmethane at 70 °C for 24 h in THF, followed by dehydration in refluxing toluene with a Dean-Stark trap using p-toluenesulfonic acid as a catalyst.[9] Herein, we reported the synthesis of a new tetraphenylethylene derivative, which may enrich the tetraphenylethylene chemistry. The title compound, built up by the $C_{30}H_{28}O_4$ molecules, has been synthesized. The single-crystal structure verifies that all bond lengths are in normal ranges.

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

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