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1,4-Dimethoxy-2,5-bis{2-[4-(trifluoromethyl)phenyl]ethynyl}benzene

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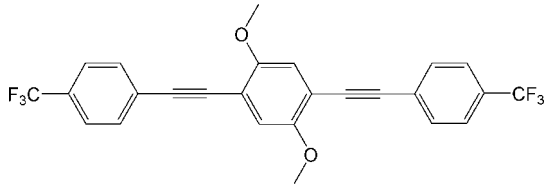
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.042; wR factor = 0.117; data-to-parameter ratio = 13.5.

The asymmetric unit of the title compound, $C_{26}H_{16}F_6O_2$, contains one half of the molecule situated on an inversion centre. In the rod-like molecule, the two terminal benzene rings form a dihedral angle of 71.9 (1)° with the central benzene ring. The trifluoromethyl group is rotationally disordered over two orientations in a 0.53 (1):0.47 (1) ratio. The crystal packing exhibits no classical intermolecular interactions.

Related literature

For applications and details of the synthesis of (arylene)-ethynylene derivatives, see: Dirk *et al.* (2001); Miljanić *et al.* (2005); Morin *et al.* (2007). For the crystal structure of a related 1,4-bis(*p*-tolylethynyl)benzene, see: Filatov & Petrukhina (2005).



Experimental

Crystal data

$C_{26}H_{16}F_6O_2$
M_r = 474.39
Monoclinic, $P2_1/c$
a = 11.1473 (4) Å
b = 13.0795 (6) Å
c = 7.5875 (4) Å
β = 97.467 (3)°
V = 1096.88 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.973, T_{max} = 0.977
9899 measured reflections
2484 independent reflections
1753 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.117
S = 1.04
2484 reflections
184 parameters
30 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5093).

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1,4-Dimethoxy-2,5-bis{2-[4-(trifluoromethyl)phenyl]ethynyl}benzene

Baohai Zhao, Jimao Lin, Cuihua Zhao and Ziying Wang

S1. Comment

Recently, the synthesis and applications of new aryleneethynylene derivatives were reported (Dirk *et al.*, 2001; Miljanić *et al.*, 2005; Morin *et al.*, 2007). To make our own contribution in this field of material science, herewith we report the synthesis and crystal structure of the title compound, (I), which can be used as luminescent material.

In (I) (Fig. 1), all bond lengths and angles are normal and correspond to those reported for 1,4-bis(*p*-tolyl-ethynyl)benzene (Filatov & Petrukhina, 2005). The asymmetric unit of (I) contains a half of the rod-like molecule. The centroid of the central benzene ring is situated on an inversion centre. The central benzene ring and C2–C7 ring form a dihedral angle of 71.9 (1)°. The crystal packing exhibits no classical intermolecular interactions.

S2. Experimental

1,4-Dimethoxy-2,5-diethynylbenzene (93 mg, 0.5 mmol), $Pd(PPh_3)_2Cl_2$ (17.5 mg)and CuI (9.5 mg) were added to tri-ethylamine (3 ml) and tetrahydrofuran (9 ml)in a Schlenk flask under N_2 atmosphere. The mixture was stirred at room temperature overnight. Then the solution was cooled to room temperature and the solvent was removed in vacuum. CH_2Cl_2 (15 ml) was added and the suspension was filtered. The filtrate was washed with HCl (1 mol l^{-1}), ammonium chloride solution and water. Then organic phase was dried with $MgSO_4$ and concentrated. The crude product was purified by column chromatography on silica gel to afford the title compound (185.3 mg, 78%). Crystals suitable for X-ray structure analysis were obtained by slowly evaporating dichloromethane solution of the title compound at room temperature.

S3. Refinement

All the H atoms were treated as riding atoms in geometrically idealized positions (C—H 0.93–0.96 Å), with $U_{iso}(H)$ = 1.2–1.5U_{eq}(C). Trifluoromethyl fragment was treated as rotationally disordered over two orientations with the refined occupancies of 0.53 (1):0.47 (1), respectively.

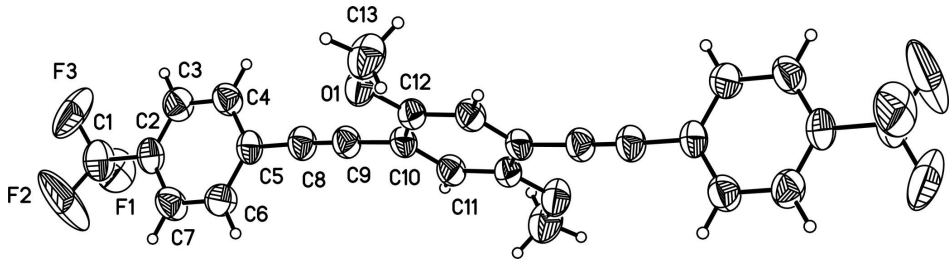

Figure 1

The molecular structure of (I) showing the atom-numbering scheme and 50% probabilty displacement ellipsoids. Unlabelled atoms are related with the labelled ones by symmetry operation (-*x*, 2 - *y*, 2 - *z*). For the disordered F atoms, only major parts are shown.

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Crystal data

$C_{26}H_{16}F_6O_2$
M_r = 474.39
Monoclinic, $P2_1/c$
a = 11.1473 (4) Å
b = 13.0795 (6) Å
c = 7.5875 (4) Å
β = 97.467 (3)°
V = 1096.88 (9) Å^3
Z = 2
$F(000)$ = 484
D_x = 1.436 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
θ = 2.4–27.4°
μ = 0.13 mm^{-1}
T = 293 K
Block, colourless
0.22 × 0.20 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.973, T_{max} = 0.977
9899 measured reflections
2484 independent reflections
1753 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.4°, θ_{min} = 2.4°
h = −14→14
k = −15→16
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.117
S = 1.04
2484 reflections
184 parameters
30 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.235P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.011 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.75647 (18)	0.83209 (17)	0.5592 (3)	0.0718 (6)	
C2	0.63855 (14)	0.85402 (14)	0.6275 (2)	0.0527 (4)	
C3	0.54917 (16)	0.78099 (15)	0.6153 (3)	0.0588 (5)	
H3	0.5617	0.7178	0.5647	0.071*	
C4	0.44076 (15)	0.80134 (14)	0.6781 (2)	0.0562 (5)	
H4	0.3805	0.7518	0.6697	0.067*	
C5	0.42165 (13)	0.89548 (14)	0.7537 (2)	0.0473 (4)	
C6	0.51220 (16)	0.96868 (14)	0.7635 (3)	0.0589 (5)	
H6	0.4999	1.0322	0.8131	0.071*	
C7	0.62038 (16)	0.94824 (15)	0.7003 (3)	0.0608 (5)	
H7	0.6806	0.9978	0.7069	0.073*	
C8	0.31058 (14)	0.91755 (14)	0.8243 (2)	0.0525 (4)	
C9	0.22045 (14)	0.93931 (13)	0.8846 (2)	0.0497 (4)	
C10	0.10877 (13)	0.96910 (12)	0.9454 (2)	0.0452 (4)	
C11	0.03456 (14)	1.03921 (12)	0.8455 (2)	0.0477 (4)	
H11	0.0584	1.0656	0.7417	0.057*	
C12	0.07381 (13)	0.92971 (12)	1.1021 (2)	0.0455 (4)	
C13	0.1199 (2)	0.82346 (18)	1.3576 (3)	0.0769 (6)	
H13A	0.1109	0.8791	1.4372	0.115*	
H13B	0.1823	0.7782	1.4105	0.115*	
H13C	0.0449	0.7867	1.3348	0.115*	
O1	0.15207 (10)	0.86246 (10)	1.19450 (17)	0.0605 (4)	
F1	0.7461 (6)	0.8553 (4)	0.3877 (5)	0.0928 (16)	0.530 (10)
F2	0.8484 (5)	0.8832 (9)	0.6296 (14)	0.183 (5)	0.530 (10)
F3	0.7807 (6)	0.7351 (3)	0.5523 (9)	0.125 (3)	0.530 (10)
F1′	0.7695 (6)	0.7396 (4)	0.5079 (11)	0.136 (4)	0.470 (10)
F2′	0.8458 (4)	0.8398 (6)	0.6905 (7)	0.0953 (18)	0.470 (10)
F3′	0.7830 (9)	0.8979 (10)	0.4453 (17)	0.205 (6)	0.470 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0550 (12)	0.0973 (17)	0.0678 (14)	0.0107 (11)	0.0252 (10)	−0.0029 (13)
C2	0.0413 (8)	0.0727 (12)	0.0462 (9)	0.0070 (8)	0.0135 (7)	−0.0022 (8)
C3	0.0545 (10)	0.0638 (12)	0.0603 (11)	0.0052 (8)	0.0152 (8)	−0.0127 (9)

C4	0.0465 (9)	0.0623 (11)	0.0613 (11)	−0.0033 (8)	0.0128 (8)	−0.0070 (9)
C5	0.0374 (8)	0.0606 (10)	0.0454 (9)	0.0062 (7)	0.0118 (7)	0.0025 (8)
C6	0.0543 (10)	0.0562 (11)	0.0703 (12)	0.0008 (8)	0.0239 (9)	−0.0095 (9)
C7	0.0468 (9)	0.0697 (12)	0.0696 (12)	−0.0083 (8)	0.0213 (9)	−0.0073 (10)
C8	0.0438 (9)	0.0616 (11)	0.0542 (10)	0.0045 (8)	0.0146 (7)	0.0039 (8)
C9	0.0409 (8)	0.0552 (10)	0.0552 (10)	0.0014 (7)	0.0142 (7)	0.0028 (8)
C10	0.0354 (7)	0.0494 (9)	0.0531 (9)	−0.0011 (7)	0.0144 (7)	−0.0027 (7)
C11	0.0422 (8)	0.0532 (10)	0.0503 (9)	−0.0016 (7)	0.0162 (7)	0.0039 (8)
C12	0.0379 (8)	0.0464 (9)	0.0532 (10)	0.0014 (6)	0.0099 (7)	0.0029 (7)
C13	0.0759 (13)	0.0846 (15)	0.0732 (14)	0.0208 (11)	0.0207 (11)	0.0317 (12)
O1	0.0510 (7)	0.0679 (8)	0.0653 (8)	0.0144 (6)	0.0170 (6)	0.0167 (6)
F1	0.089 (3)	0.120 (3)	0.081 (2)	0.005 (2)	0.0551 (18)	0.006 (2)
F2	0.056 (3)	0.278 (10)	0.228 (8)	−0.065 (5)	0.068 (4)	−0.175 (8)
F3	0.119 (4)	0.109 (4)	0.168 (5)	0.074 (3)	0.095 (4)	0.067 (4)
F1′	0.071 (3)	0.180 (8)	0.163 (5)	0.005 (3)	0.031 (3)	−0.118 (6)
F2′	0.0321 (19)	0.142 (4)	0.115 (3)	0.0013 (19)	0.0200 (17)	0.000 (3)
F3′	0.142 (8)	0.247 (10)	0.260 (10)	0.105 (7)	0.157 (8)	0.190 (9)

Geometric parameters (Å, º)

C1—F2	1.281 (4)	C6—C7	1.380 (2)
C1—F3′	1.281 (4)	C6—H6	0.9300
C1—F1′	1.285 (4)	C7—H7	0.9300
C1—F3	1.300 (4)	C8—C9	1.191 (2)
C1—F2′	1.318 (4)	C9—C10	1.437 (2)
C1—F1	1.327 (4)	C10—C11	1.391 (2)
C1—C2	1.502 (2)	C10—C12	1.397 (2)
C2—C3	1.375 (3)	C11—C12^{i}	1.381 (2)
C2—C7	1.376 (3)	C11—H11	0.9300
C3—C4	1.381 (2)	C12—O1	1.3664 (19)
C3—H3	0.9300	C12—C11^{i}	1.381 (2)
C4—C5	1.386 (2)	C13—O1	1.427 (2)
C4—H4	0.9300	C13—H13A	0.9600
C5—C6	1.386 (2)	C13—H13B	0.9600
C5—C8	1.441 (2)	C13—H13C	0.9600
F2—C1—F3′	71.9 (5)	C5—C4—H4	119.9
F2—C1—F1′	120.1 (5)	C6—C5—C4	119.06 (14)
F3′—C1—F1′	112.5 (6)	C6—C5—C8	119.78 (16)
F2—C1—F3	111.5 (5)	C4—C5—C8	121.15 (16)
F3′—C1—F3	124.1 (6)	C7—C6—C5	120.70 (17)
F1′—C1—F3	15.5 (5)	C7—C6—H6	119.6
F2—C1—F2′	32.8 (6)	C5—C6—H6	119.6
F3′—C1—F2′	103.9 (7)	C2—C7—C6	119.57 (17)
F1′—C1—F2′	101.3 (4)	C2—C7—H7	120.2
F3—C1—F2′	87.9 (5)	C6—C7—H7	120.2
F2—C1—F1	104.7 (6)	C9—C8—C5	177.5 (2)
F3′—C1—F1	35.2 (8)	C8—C9—C10	175.89 (19)

F1′—C1—F1	85.2 (4)	C11—C10—C12	119.64 (13)
F3—C1—F1	100.1 (4)	C11—C10—C9	118.86 (14)
F2′—C1—F1	133.2 (3)	C12—C10—C9	121.50 (15)
F2—C1—C2	116.4 (3)	C12^{i}—C11—C10	121.18 (15)
F3′—C1—C2	113.2 (3)	C12^{i}—C11—H11	119.4
F1′—C1—C2	115.0 (4)	C10—C11—H11	119.4
F3—C1—C2	113.4 (3)	O1—C12—C11^{i}	124.45 (15)
F2′—C1—C2	109.5 (3)	O1—C12—C10	116.37 (13)
F1—C1—C2	109.1 (3)	C11^{i}—C12—C10	119.18 (15)
C3—C2—C7	120.41 (15)	O1—C13—H13A	109.5
C3—C2—C1	120.20 (17)	O1—C13—H13B	109.5
C7—C2—C1	119.38 (17)	H13A—C13—H13B	109.5
C2—C3—C4	120.11 (17)	O1—C13—H13C	109.5
C2—C3—H3	119.9	H13A—C13—H13C	109.5
C4—C3—H3	119.9	H13B—C13—H13C	109.5
C3—C4—C5	120.14 (17)	C12—O1—C13	117.37 (13)
C3—C4—H4	119.9		

F2—C1—C2—C3	−154.8 (8)	C8—C5—C6—C7	178.50 (18)
F3′—C1—C2—C3	124.7 (9)	C3—C2—C7—C6	0.9 (3)
F1′—C1—C2—C3	−6.6 (5)	C1—C2—C7—C6	−179.94 (18)
F3—C1—C2—C3	−23.5 (4)	C5—C6—C7—C2	−0.3 (3)
F2′—C1—C2—C3	−119.9 (4)	C6—C5—C8—C9	−1 (5)
F1—C1—C2—C3	87.1 (3)	C4—C5—C8—C9	178 (100)
F2—C1—C2—C7	26.0 (8)	C5—C8—C9—C10	85 (5)
F3′—C1—C2—C7	−54.5 (10)	C8—C9—C10—C11	−11 (3)
F1′—C1—C2—C7	174.2 (5)	C8—C9—C10—C12	169 (3)
F3—C1—C2—C7	157.3 (4)	C12—C10—C11—C12^{i}	−0.4 (3)
F2′—C1—C2—C7	60.9 (4)	C9—C10—C11—C12^{i}	179.90 (16)
F1—C1—C2—C7	−92.1 (3)	C11—C10—C12—O1	−179.04 (15)
C7—C2—C3—C4	−0.8 (3)	C9—C10—C12—O1	0.6 (2)
C1—C2—C3—C4	−179.95 (18)	C11—C10—C12—C11^{i}	0.4 (3)
C2—C3—C4—C5	0.0 (3)	C9—C10—C12—C11^{i}	−179.92 (16)
C3—C4—C5—C6	0.6 (3)	C11^{i}—C12—O1—C13	−1.1 (3)
C3—C4—C5—C8	−178.37 (17)	C10—C12—O1—C13	178.26 (17)
C4—C5—C6—C7	−0.5 (3)		

Symmetry code: (i) $-x$, $-y+2$, $-z+2$.