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3,3′,4,4′-Tetrabutyl-5,5′-(biphenyl-4,4′-diyldiethynyl)bis(thiophene-2-carbonitrile)

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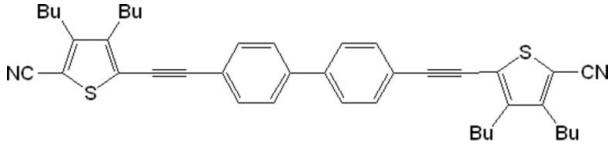
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.071; wR factor = 0.235; data-to-parameter ratio = 19.6.

The molecule of the title compound, $C_{42}H_{44}N_2S_2$, is centrosymmetric. Thus, an asymmetric unit comprises half of the molecule. The torsion angle of the C—C≡C—C backbone is −165.6 (4)°. The crystal packing is dominated by van der Waals interactions.

Related literature

For related literature, see: Bloor (1995); Bumm *et al.* (1996); Carroll & Gorman (2002); Liu *et al.* (2006); Martin *et al.* (1997, 1999).



Experimental

Crystal data

$C_{42}H_{44}N_2S_2$
M_r = 640.91
Triclinic, $P\overline{1}$
a = 8.1428 (16) Å
b = 9.1856 (18) Å
c = 13.606 (3) Å
α = 90.37 (3)°
β = 100.53 (3)°
γ = 112.32 (3)°
V = 922.3 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 293 (2) K
0.41 × 0.39 × 0.28 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: empirical (using intensity measurements) (*ABSCOR*; Higashi, 1995)
T_{min} = 0.879, T_{max} = 0.996
5834 measured reflections
4080 independent reflections
3287 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.235
S = 1.01
4080 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); 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: KP2126).

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supplementary materials

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3,3',4,4'-Tetrabutyl-5,5'-(biphenyl-4,4'-diyldiethynyl)bis(thiophene-2-carbonitrile)

L. Liu, W. Xu, X.-W. Xiao and D.-B. Zhu

Comment

By developing new techniques to fabricate miniature components, and to fit more components into each cm ^{2} of silicon, engineers have driven the speed and capabilities of computing at a predictably fast pace. But the reduction in size of components, and thus increase in speed, can only continue for some finite time. New processes are under development in order to extend the useful functionality of silicon integrated circuits (Carroll *et al.*, 2002).

Linear, π-conjugated molecules have attracted great attention due to their potential in molecular scale electronic devices (Bloor, 1995; Bumm *et al.*, 1996). Molecular wires typically consist of two electroactive functional groups linked by an extended π-electron network. Rainer E. Martin synthesized a series of monodisperse Me_3Si -endcapped poly(triacetylene) oligomers (Martin *et al.*, 1997; Martin *et al.*, 1999). Here, the synthesis and crystal structures of 4,4'-dicyano- (3,4-dibutylthiophenylethynyl)biphenyl (I) are presented.

The molecular structure of the title compound (Fig. 1) is centrosymmetric. Thus, an asymmetric unit comprises a half on the molecule. The inversion centre is located in the middle of C3—$C3_i$ bond. The two phenyl rings and carbon triple bonds are coplanar. The torsion angle of C9—C8—C7—C6 is −165.6 (4)° whereas the diphenyl moiety is linear due to an inversion symmetry of the molecule. The crystal packing (Fig. 2) is dominated by van der Waals interactions.

Experimental

The synthesis of the title compound was performed as described by Liu *et al.* (2006).

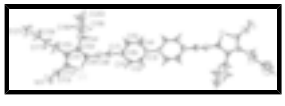
Colourless plate-like crystals of 4,4'-dicyano-(3,4-dibutyl-thienyl ethynyl) biphenyl were grown from ethanol /hexane mixture by slow evaporation of the solution.

Refinement

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(*s*) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(*s*) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 1998); software used

to prepare material for publication: *SHELXTL* (Siemens, 1994).

Figures



Scheme 1. The structural diagram of the title compound (I).
Fig. 1. The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability. To generate the molecule symmetry code $-x, -y, -z + 2$ is applied.

Fig. 2. The crystal packing along *a* axis of the title compound.

3,3',4,4'-Tetrabutyl-5,5'-(biphenyl-4,4'-diyldiethynyl)bis(thiophene-2-\ carbonitrile)

Crystal data

$C_{42}H_{44}N_2S_2$	$V = 922.3\ (3)$ Å^3
$M_r = 640.91$	$Z = 1$
Triclinic, $P\overline{1}$	$F_{000} = 342$
$a = 8.1428\ (16)$ Å	$D_x = 1.154$ Mg m^{-3}
$b = 9.1856\ (18)$ Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$c = 13.606\ (3)$ Å	$\mu = 0.18$ mm^{-1}
$\alpha = 90.37\ (3)^o$	$T = 293\ (2)$ K
$\beta = 100.53\ (3)^o$	Plate, colourless
$\gamma = 112.32\ (3)^o$	0.41 × 0.39 × 0.28 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer	4080 independent reflections
Radiation source: fine-focus sealed tube	3287 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.029$
Detector resolution: 0.76 pixels mm^{-1}	$\theta_{max} = 27.5^o$
$T = 293(2)$ K	$\theta_{min} = 1.5^o$
Oscillation scans	$h = -9\rightarrow10$
Absorption correction: empirical (using intensity measurements) (ABSCOR; Higashi, 1995)	$k = -9\rightarrow11$
$T_{min} = 0.879$, $T_{max} = 0.996$	$l = -17\rightarrow17$
5834 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.071$	H-atom parameters constrained
$wR(F^2) = 0.235$	$w = 1/[\sigma^2(F_o^2) + (0.1503P)^2 + 0.3412P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
4080 reflections	$\Delta\rho_{max} = 0.81$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.28848 (9)	0.32421 (9)	0.44787 (5)	0.0608 (3)
C1	0.1734 (4)	0.2078 (4)	0.8390 (2)	0.0766 (9)
H1A	0.2773	0.2920	0.8308	0.092*
N1	0.4301 (5)	0.4639 (4)	0.2102 (3)	0.1016 (11)
C2	0.1583 (4)	0.1546 (4)	0.9336 (2)	0.0752 (9)
H2A	0.2524	0.2049	0.9878	0.090*
C3	0.0075 (3)	0.0290 (3)	0.94934 (16)	0.0469 (5)
C4	−0.1306 (4)	−0.0409 (3)	0.86606 (19)	0.0639 (7)
H4A	−0.2353	−0.1245	0.8740	0.077*
C5	−0.1156 (4)	0.0112 (3)	0.77223 (19)	0.0662 (7)
H5A	−0.2095	−0.0393	0.7179	0.079*
C6	0.0349 (4)	0.1361 (3)	0.75692 (17)	0.0533 (6)
C7	0.0492 (4)	0.1910 (3)	0.65898 (19)	0.0574 (6)
C8	0.0589 (4)	0.2360 (3)	0.57725 (18)	0.0556 (6)
C9	0.0843 (3)	0.2910 (3)	0.48107 (17)	0.0506 (5)
C10	−0.0335 (3)	0.3280 (3)	0.40904 (17)	0.0492 (5)
C11	0.0437 (4)	0.3854 (3)	0.32413 (18)	0.0534 (6)
C12	0.2171 (4)	0.3890 (3)	0.3356 (2)	0.0589 (6)
C13	0.3348 (5)	0.4322 (4)	0.2656 (2)	0.0717 (8)
C14	−0.2209 (4)	0.3068 (3)	0.4213 (2)	0.0589 (6)
H14A	−0.2454	0.3970	0.3970	0.071*
H14B	−0.2242	0.3058	0.4922	0.071*
C15	−0.0488 (5)	0.4360 (4)	0.2334 (2)	0.0686 (8)
H15A	0.0366	0.5335	0.2151	0.082*
H15B	−0.1478	0.4583	0.2507	0.082*
C16	−0.3690 (4)	0.1584 (4)	0.3671 (3)	0.0848 (10)
H16A	−0.4820	0.1504	0.3849	0.102*
H16B	−0.3809	0.1699	0.2957	0.102*
C17	−0.1229 (5)	0.3166 (5)	0.1432 (2)	0.0803 (9)
H17A	−0.1959	0.2149	0.1632	0.096*
H17B	−0.0227	0.3054	0.1193	0.096*
C18	−0.3470 (6)	0.0111 (5)	0.3857 (4)	0.1018 (13)
H18A	−0.3299	0.0015	0.4574	0.122*

H18B	−0.2369	0.0170	0.3650	0.122*
C19	−0.2385 (7)	0.3634 (6)	0.0574 (3)	0.1009 (13)
H19A	−0.3402	0.3718	0.0810	0.121*
H19B	−0.1663	0.4668	0.0392	0.121*
C20	−0.5024 (6)	−0.1393 (4)	0.3335 (4)	0.0994 (12)
H20A	−0.4765	−0.2298	0.3532	0.149*
H20B	−0.5152	−0.1357	0.2621	0.149*
H20C	−0.6130	−0.1468	0.3526	0.149*
C21	−0.3088 (8)	0.2507 (7)	−0.0336 (3)	0.1312 (19)
H21A	−0.3806	0.2867	−0.0838	0.197*
H21B	−0.3823	0.1483	−0.0167	0.197*
H21C	−0.2091	0.2443	−0.0590	0.197*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0507 (4)	0.0685 (5)	0.0586 (4)	0.0192 (3)	0.0086 (3)	0.0062 (3)
C1	0.0565 (15)	0.092 (2)	0.0554 (15)	0.0035 (15)	0.0041 (12)	0.0216 (14)
N1	0.109 (3)	0.090 (2)	0.111 (2)	0.0240 (19)	0.067 (2)	0.0237 (18)
C2	0.0547 (14)	0.092 (2)	0.0499 (14)	0.0031 (14)	−0.0033 (11)	0.0171 (14)
C3	0.0491 (12)	0.0484 (12)	0.0435 (11)	0.0205 (10)	0.0063 (9)	0.0037 (9)
C4	0.0622 (15)	0.0614 (16)	0.0479 (13)	0.0051 (12)	0.0037 (11)	0.0033 (11)
C5	0.0703 (17)	0.0661 (16)	0.0439 (13)	0.0121 (13)	−0.0013 (11)	−0.0005 (11)
C6	0.0623 (14)	0.0603 (14)	0.0433 (12)	0.0305 (12)	0.0100 (10)	0.0085 (10)
C7	0.0645 (15)	0.0634 (15)	0.0490 (13)	0.0300 (13)	0.0115 (11)	0.0066 (11)
C8	0.0613 (14)	0.0601 (14)	0.0454 (12)	0.0242 (12)	0.0089 (10)	0.0054 (10)
C9	0.0520 (12)	0.0518 (12)	0.0437 (11)	0.0157 (10)	0.0092 (9)	0.0033 (9)
C10	0.0549 (13)	0.0455 (12)	0.0443 (11)	0.0168 (10)	0.0086 (9)	0.0014 (9)
C11	0.0645 (14)	0.0464 (12)	0.0472 (12)	0.0188 (11)	0.0119 (10)	0.0059 (9)
C12	0.0639 (15)	0.0546 (14)	0.0547 (14)	0.0160 (12)	0.0189 (12)	0.0096 (11)
C13	0.0767 (19)	0.0607 (16)	0.0757 (19)	0.0164 (14)	0.0325 (16)	0.0100 (13)
C14	0.0606 (15)	0.0583 (14)	0.0621 (15)	0.0266 (12)	0.0149 (12)	0.0025 (11)
C15	0.094 (2)	0.0677 (17)	0.0520 (14)	0.0403 (16)	0.0142 (14)	0.0149 (12)
C16	0.0577 (17)	0.082 (2)	0.110 (3)	0.0246 (16)	0.0124 (17)	−0.0179 (19)
C17	0.101 (2)	0.092 (2)	0.0537 (16)	0.050 (2)	0.0030 (15)	0.0039 (15)
C18	0.080 (2)	0.074 (2)	0.148 (4)	0.028 (2)	0.016 (2)	−0.006 (2)
C19	0.122 (3)	0.133 (4)	0.064 (2)	0.075 (3)	0.001 (2)	0.004 (2)
C20	0.092 (3)	0.067 (2)	0.122 (3)	0.0202 (19)	0.004 (2)	−0.008 (2)
C21	0.151 (5)	0.169 (5)	0.080 (3)	0.087 (4)	−0.016 (3)	−0.010 (3)

Geometric parameters (Å, °)

S1—C9	1.718 (3)	C14—H14A	0.9700
S1—C12	1.725 (3)	C14—H14B	0.9700
C1—C6	1.386 (4)	C15—C17	1.511 (4)
C1—C2	1.389 (4)	C15—H15A	0.9700
C1—H1A	0.9300	C15—H15B	0.9700
N1—C13	1.140 (4)	C16—C18	1.448 (5)
C2—C3	1.381 (4)	C16—H16A	0.9700

C2—H2A	0.9300	C16—H16B	0.9700
C3—C4	1.392 (4)	C17—C19	1.531 (5)
C3—C3^{i}	1.490 (4)	C17—H17A	0.9700
C4—C5	1.376 (4)	C17—H17B	0.9700
C4—H4A	0.9300	C18—C20	1.532 (6)
C5—C6	1.375 (4)	C18—H18A	0.9700
C5—H5A	0.9300	C18—H18B	0.9700
C6—C7	1.437 (3)	C19—C21	1.484 (6)
C7—C8	1.195 (4)	C19—H19A	0.9700
C8—C9	1.428 (3)	C19—H19B	0.9700
C9—C10	1.380 (4)	C20—H20A	0.9600
C10—C11	1.425 (3)	C20—H20B	0.9600
C10—C14	1.504 (4)	C20—H20C	0.9600
C11—C12	1.380 (4)	C21—H21A	0.9600
C11—C15	1.497 (4)	C21—H21B	0.9600
C12—C13	1.425 (4)	C21—H21C	0.9600
C14—C16	1.506 (4)		
C9—S1—C12	90.36 (13)	C11—C15—H15A	108.6
C6—C1—C2	120.4 (3)	C17—C15—H15A	108.6
C6—C1—H1A	119.8	C11—C15—H15B	108.6
C2—C1—H1A	119.8	C17—C15—H15B	108.6
C3—C2—C1	121.9 (3)	H15A—C15—H15B	107.6
C3—C2—H2A	119.1	C18—C16—C14	117.0 (3)
C1—C2—H2A	119.1	C18—C16—H16A	108.0
C2—C3—C4	116.9 (2)	C14—C16—H16A	108.0
C2—C3—C3^{i}	121.7 (3)	C18—C16—H16B	108.0
C4—C3—C3^{i}	121.4 (3)	C14—C16—H16B	108.0
C5—C4—C3	121.4 (3)	H16A—C16—H16B	107.3
C5—C4—H4A	119.3	C15—C17—C19	112.6 (3)
C3—C4—H4A	119.3	C15—C17—H17A	109.1
C6—C5—C4	121.5 (3)	C19—C17—H17A	109.1
C6—C5—H5A	119.2	C15—C17—H17B	109.1
C4—C5—H5A	119.2	C19—C17—H17B	109.1
C5—C6—C1	117.9 (2)	H17A—C17—H17B	107.8
C5—C6—C7	121.3 (2)	C16—C18—C20	116.4 (4)
C1—C6—C7	120.8 (3)	C16—C18—H18A	108.2
C8—C7—C6	179.2 (3)	C20—C18—H18A	108.2
C7—C8—C9	175.7 (3)	C16—C18—H18B	108.2
C10—C9—C8	128.5 (2)	C20—C18—H18B	108.2
C10—C9—S1	113.10 (18)	H18A—C18—H18B	107.4
C8—C9—S1	118.34 (19)	C21—C19—C17	114.0 (4)
C9—C10—C11	112.0 (2)	C21—C19—H19A	108.8
C9—C10—C14	122.5 (2)	C17—C19—H19A	108.8
C11—C10—C14	125.5 (2)	C21—C19—H19B	108.8
C12—C11—C10	111.3 (2)	C17—C19—H19B	108.8
C12—C11—C15	123.5 (2)	H19A—C19—H19B	107.7
C10—C11—C15	125.2 (3)	C18—C20—H20A	109.5
C11—C12—C13	128.0 (3)	C18—C20—H20B	109.5

C11—C12—S1	113.28 (19)	H20A—C20—H20B	109.5
C13—C12—S1	118.7 (2)	C18—C20—H20C	109.5
N1—C13—C12	178.3 (4)	H20A—C20—H20C	109.5
C10—C14—C16	114.4 (2)	H20B—C20—H20C	109.5
C10—C14—H14A	108.7	C19—C21—H21A	109.5
C16—C14—H14A	108.7	C19—C21—H21B	109.5
C10—C14—H14B	108.7	H21A—C21—H21B	109.5
C16—C14—H14B	108.7	C19—C21—H21C	109.5
H14A—C14—H14B	107.6	H21A—C21—H21C	109.5
C11—C15—C17	114.6 (2)	H21B—C21—H21C	109.5
C12—C11—C10—C9	0.1 (3)	C1—C2—C3—C4	−0.9 (5)
C12—S1—C9—C10	0.0 (3)	C5—C4—C3—C2	1.1 (6)
C10—C11—C12—S1	−0.1 (1)	C5—C6—C1—C2	−0.4 (5)
C11—C12—S1—C9	0.0 (5)	C12—C11—C15—C17	77.9 (4)
C11—C10—C9—S1	−0.1 (3)	C11—C15—C17—C19	172.0 (7)
C11—C10—C9—C8	177.5 (5)	C15—C17—C19—C21	178.2 (5)
C12—S1—C9—C8	−177.8 (8)	C10—C11—C15—C17	−102.1 (3)
C7—C6—C5—C4	−179.4 (1)	C11—C10—C14—C16	79.8 (7)
C7—C6—C1—C2	179.6 (8)	C9—C10—C14—C16	−99.4 (5)
C6—C5—C4—C3	−1.1 (5)	C10—C14—C16—C18	52.3 (7)
C6—C1—C2—C3	0.5 (7)	C14—C16—C18—C20	177.5 (2)
C4—C5—C6—C1	0.7 (2)	C4—C3—C3—C4	90.00 (3)

Symmetry codes: (i) −*x*, −*y*, −*z*+2.

Fig. 1

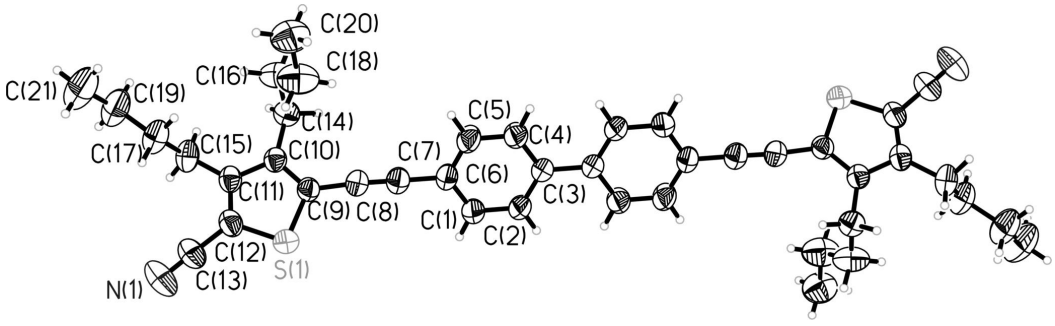

C(20)
C(18)
C(16)
C(21)
C(19)
C(14)
C(17)
C(15)
C(10)
C(11)
C(9)
C(8)
C(7)
C(5)
C(4)
C(6)
C(3)
C(1)
C(2)
C(12)
S(1)
N(1)
C(13)

Fig. 2