IUCrData

ISSN 2414-3146

1,1-Diphenyl-4-(thiophen-2-yl)but-3-yn-1-ol

Christian A. Umaña,[a] Leslie W. Pineda[a,b] and Jorge A. Cabezas[a]*

[a]Escuela de Química, Universidad de Costa Rica, 2060 San José, Costa Rica, and [b]Centro de Electroquímica y Energía Química (CELEQ), Universidad de Costa Rica, 2060 San José, Costa Rica. *Correspondence e-mail: jorge.cabezas@ucr.ac.cr

Received 20 October 2018
Accepted 14 November 2018

Edited by C. Rizzoli, Universita degli Studi di Parma, Italy

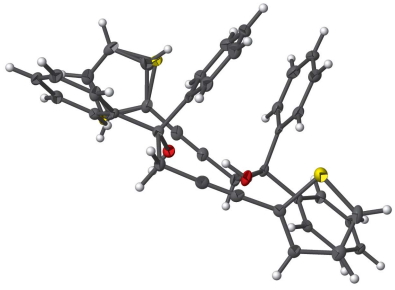
Keywords: crystal structure; homopropargyl alcohols; 1,3-dilithiopropyne; propargylation; alkynes.

CCDC reference: 1873455

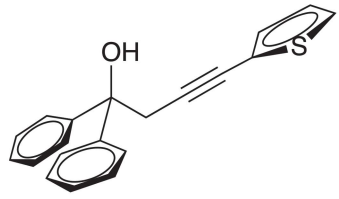
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title homopropargyl alcohol, $C_{20}H_{16}OS$, contains two independent molecules comprising a hydroxy group, a 3-(2-thiophenyl)-propargylic moiety and two aromatic rings linked to a central carbon atom. The two unique molecules are linked into a dimer by an O—H···O hydrogen bond. In one molecule, the thiophene ring is disordered over two orientations rotated by 180° with a refined occupancy ratio of 0.575 (4):0.425 (4). The crystal structure is stabilized by O—H···π and C—H···π hydrogen-bond interactions. The crystal studied was a two-component non-merohedral twin, the refined ratio of the twin components being 0.575 (4):0.425 (4).

3D view



Chemical scheme



Structure description

1,1,4-Aromatic trisubstituted homopropargylic alcohols are difficult to synthesize efficiently. They are very useful intermediates in the synthesis of a variety of organic compounds (Kim *et al.*, 2017; Foley & Leighton, 2015; Francais *et al.*, 2010; Hosseyni *et al.*, 2016; Gao *et al.*, 2014; Trost & Rhee 2003; Yadav & Maiti, 2002). In this work, the crystal structure of the homopropargyl alcohol 1,1-diphenyl-4-(2-thiophenyl)-3-butyn-1-ol is reported.

The title compound crystallizes with two molecules in the asymmetric unit. Each molecule contains a central carbon atom (C1_1 or C1_2) which is bound to a hydroxy group, a 3-(2-thiophenyl)propargylic fragment and two phenyl substituents, leading to a distorted tetrahedral geometry as it departs from the ideal value (109.5°) with angles spanning from 105.1 (3) to 114.0 (3)° (Fig. 1). The lengths of the carbon–carbon triple bonds (C3_1—C4_1 and C3_2—C4_2) are 1.193 (5) and 1.189 (5) Å, respectively. The propargylic units (C4_1—C3_1—C2_1 and C4_2—C3_2—C2_2) exhibit angles of 176.4 (4) and 178.5 (4)°, slightly distorted from the expected linear geometry. The two molecules are linked by an O1_2—H1_2···O1_1 hydrogen bond (Fig. 1, Table 1), forming

OPEN ACCESS

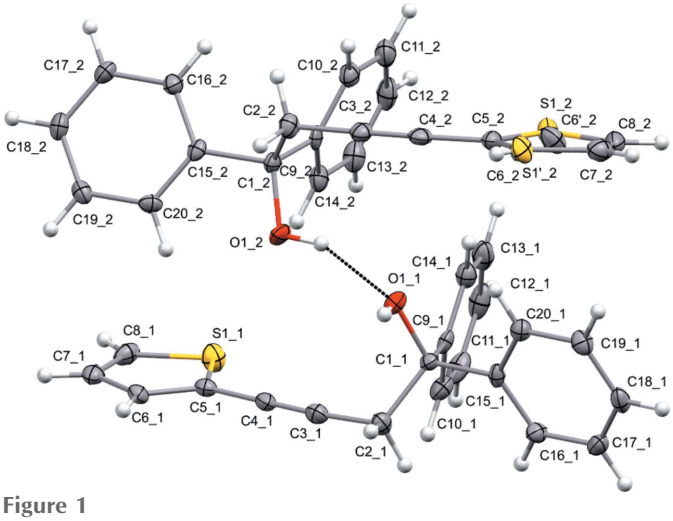


Figure 1
The title molecules with 50% probability ellipsoids. The intermolecular O—H···O hydrogen bond is shown as a black dotted line.

a dimeric unit. In the crystal (Fig. 2), molecules are linked into a three-dimensional network by O—H···π and C—H···π interactions (Table 1).

Synthesis and crystallization

The title compound was prepared in a one-pot reaction according to the previously reported procedure (Umaña & Cabezas, 2017). The product was purified by column chromatography and recrystallized from an ethyl ether:hexanes (1:1 *v*/*v*) solvent mixture to give colourless block-shaped crystals.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. In the last cycles of refinement an outlier ($\bar{4}47$) was omitted and enhanced rigid-bond restraints (RIGU instruction in *SHELXL*) were applied. The crystal used for the X-ray diffraction experiment was a two-component non-merohedral twin, the refined ratio of the twin components being 0.575 (4):0.425 (4). One thiophene ring is rotationally disordered by approximately 180° over two positions with a refined occupancy ratio 0.575 (4):0425 (4).

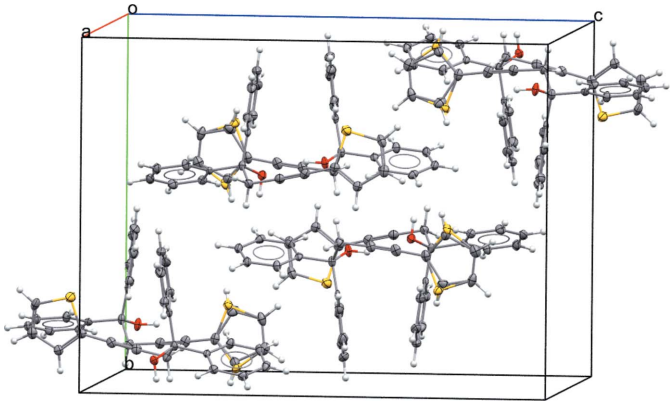


Figure 2
Crystal packing of the title compound viewed approximately down the *a* axis.

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1, *Cg*2 and *Cg*3 are the centroids of the C15_2–C20_2, C15_1–C20_1 and S1_1/C5_1–C8_1 rings, respectively; *Cg*4 is the centroid of the disordered S1_2/S1′_2/C6_2/C6′_2/C5_2/C7_2/C8_2 thiophene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1_2—H1_2···O1_1	0.85 (4)	2.08 (4)	2.864 (4)	153 (4)
O1_1—H1_1···*Cg*1^{i}	0.86 (5)	2.69 (5)	3.516 (3)	162 (4)
C8_1—H8_1···*Cg*2^{ii}	0.95	2.88	3.489 (5)	123
C8_2—H8_2···*Cg*1^{iii}	0.95	2.92	3.519 (5)	123
C16_2—H16_2···*Cg*3^{iv}	0.95	2.86	3.648 (4)	141
C20_2—H20_2···*Cg*3	0.95	2.99	3.658 (4)	129
C16_1—H16_1···*Cg*4^{v}	0.95	2.92	3.679 (6)	137

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $x-1, y, z$; (v) $x+1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{16}OS$
M_r	304.39
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, *b*, *c* (Å)	9.4240 (4), 16.0262 (7), 20.8913 (10)
β (°)	99.042 (1)
V (Å^3)	3116.0 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.21
Crystal size (mm)	0.35 × 0.25 × 0.15
Data collection	
Diffractometer	Bruker D8 VENTURE
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.665, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6312, 6312, 3628
R_{int}	0.020
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.603
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.074, 0.137, 1.04
No. of reflections	6312
No. of parameters	423
No. of restraints	363
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.36

Computer programs: *APEX3* and *SAINT* (Bruker, 2017), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2017* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2006) and *PLATON* (Spek, 2009).

Acknowledgements

Dr Bruce Noll (Bruker, Madison, USA) is greatly acknowledged for his assistance in solving the disorder in the crystal structure.

Funding information

We thank the Sistema de Estudios de Posgrado (SEP), Universidad de Costa Rica (UCR) for a stipend to CAU, and the Vicerrectoría de Investigación (UCR) for financial support.

References

Bruker (2017). *APEX3* and *SAINT*. Bruker AXS Inc., Madison, WI, USA.
Foley, C. N. & Leighton, J. L. (2015). *Org. Lett.* **17**, 5858–5861.
Francais, A., Leyva, A., Etxebarria-Jardi, G. & Ley, S. V. (2010). *Org. Lett.* **12**, 340–343.
Gao, P., Li, H. X., Hao, X. H., Jin, D. P., Chen, D. Q., Yan, X. B., Wu, X. X., Song, X. R., Liu, X. Y. & Liang, Y. M. (2014). *Org. Lett.* **16**, 6298–6301.
Hosseyni, S., Wojtas, L., Li, M. & Shi, X. (2016). *J. Am. Chem. Soc.* **138**, 3994–3997.
Kim, J., Jeong, W. & Rhee, Y. H. (2017). *Org. Lett.* **19**, 242–245.
Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Trost, B. M. & Rhee, Y. H. (2003). *J. Am. Chem. Soc.* **125**, 7482–7483.
Umaña, C. A. & Cabezas, J. A. (2017). *J. Org. Chem.* **82**, 9505–9514.
Yadav, J. S. & Maiti, A. (2002). *Tetrahedron*, **58**, 4955–4961.

full crystallographic data

IUCrData (2018). **3**, x181616 [https://doi.org/10.1107/S2414314618016164]

1,1-Diphenyl-4-(thiophen-2-yl)but-3-yn-1-ol

Christian A. Umaña, Leslie W. Pineda and Jorge A. Cabezas

1,1-Diphenyl-4-(thiophen-2-yl)but-3-yn-1-ol

Crystal data

$C_{20}H_{16}OS$
M_r = 304.39
Monoclinic, $P2_1/n$
a = 9.4240 (4) Å
b = 16.0262 (7) Å
c = 20.8913 (10) Å
β = 99.042 (1)°
V = 3116.0 (2) Å^3
Z = 8
$F(000)$ = 1280
D_x = 1.298 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9969 reflections
θ = 2.5–24.9°
μ = 0.21 mm^{-1}
T = 100 K
Block, colorless
0.35 × 0.25 × 0.15 mm

Data collection

Bruker D8 VENTURE diffractometer
Radiation source: sealed tube, Siemens KFFMO2K-90
Curved graphite monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.665, T_{max} = 0.746
6312 measured reflections
6312 independent reflections
3628 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.4°, θ_{min} = 2.5°
h = −11→11
k = 0→19
l = 0→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.137
S = 1.04
6312 reflections
423 parameters
363 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0291P)^2 + 3.993P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Special details

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

Refinement. Refined as a 2-component non-merohedral twin. Twin ratio: 0.1944 (18), Disorder is present in the sulfur position of the second molecule. This sulfur is also present at the site of C6, and C6 is present at S1. The SOF for the primary positions of S1 and C6 is 0.575 (4),
The hydroxy H atoms were located in a difference Fourier map and refined isotropically with U_{iso}(H) = 1.5 U_{eq}(O). All other H atoms were placed geometrically and refined using a riding atom approximation, with C–H = 0.95–0.99 Å and with U_{iso}(H) = 1.2 U_{eq}(C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1_1	0.89408 (11)	0.25822 (7)	0.55877 (5)	0.0256 (3)	
O1_1	0.5644 (3)	0.40979 (17)	0.34584 (12)	0.0193 (6)	
H1_1	0.569 (4)	0.463 (3)	0.3412 (18)	0.029*	
C1_1	0.6758 (4)	0.3760 (2)	0.31374 (17)	0.0149 (8)	
C2_1	0.8186 (4)	0.4171 (2)	0.34297 (16)	0.0196 (9)	
H2A_1	0.896007	0.396352	0.320101	0.024*	
H2AB_1	0.811096	0.478233	0.33639	0.024*	
C3_1	0.8565 (4)	0.3994 (2)	0.41259 (18)	0.0199 (9)	
C4_1	0.8800 (4)	0.3829 (2)	0.46898 (18)	0.0177 (9)	
C5_1	0.9078 (4)	0.3615 (2)	0.53574 (17)	0.0161 (8)	
C6_1	0.9475 (4)	0.4114 (2)	0.58869 (17)	0.0175 (9)	
H6_1	0.96169	0.469944	0.586297	0.021*	
C7_1	0.9651 (4)	0.3658 (2)	0.64742 (18)	0.0210 (9)	
H7_1	0.991389	0.390781	0.688811	0.025*	
C8_1	0.9406 (4)	0.2829 (3)	0.63877 (18)	0.0229 (9)	
H8_1	0.948374	0.243351	0.673037	0.027*	
C9_1	0.6712 (4)	0.2825 (2)	0.32478 (16)	0.0160 (8)	
C10_1	0.7942 (4)	0.2332 (2)	0.33579 (17)	0.0222 (9)	
H10_1	0.886056	0.258288	0.337602	0.027*	
C11_1	0.7836 (5)	0.1480 (3)	0.34412 (18)	0.0279 (10)	
H11_1	0.868351	0.115014	0.351931	0.034*	
C12_1	0.6509 (5)	0.1103 (3)	0.34117 (18)	0.0301 (10)	
H12_1	0.643741	0.051672	0.346339	0.036*	
C13_1	0.5290 (5)	0.1592 (3)	0.33062 (18)	0.0281 (10)	
H13_1	0.437325	0.133861	0.328996	0.034*	
C14_1	0.5385 (4)	0.2441 (2)	0.32240 (17)	0.0212 (9)	
H14_1	0.453305	0.276686	0.31503	0.025*	
C15_1	0.6432 (4)	0.3940 (2)	0.24053 (16)	0.0153 (8)	
C16_1	0.7410 (4)	0.3718 (2)	0.20010 (17)	0.0185 (9)	
H16_1	0.829414	0.346388	0.21813	0.022*	
C17_1	0.7116 (4)	0.3860 (2)	0.13419 (17)	0.0216 (9)	
H17_1	0.7792	0.370121	0.107161	0.026*	
C18_1	0.5834 (4)	0.4235 (2)	0.10747 (18)	0.0214 (9)	
H18_1	0.563912	0.43478	0.062299	0.026*	
C19_1	0.4846 (4)	0.4444 (2)	0.14678 (17)	0.0206 (9)	
H19_1	0.395939	0.469208	0.128469	0.025*	
C20_1	0.5136 (4)	0.4295 (2)	0.21297 (17)	0.0178 (9)	
H20_1	0.444227	0.443605	0.239592	0.021*	

S1_2	0.1569 (7)	0.2927 (3)	0.2463 (2)	0.0216 (9)	0.575 (4)
S1′_2	0.1245 (12)	0.4662 (5)	0.2240 (4)	0.0219 (14)	0.425 (4)
O1_2	0.5308 (3)	0.37527 (17)	0.47711 (12)	0.0218 (7)	
H1_2	0.519 (4)	0.373 (2)	0.436 (2)	0.033*	
C1_2	0.4005 (4)	0.3526 (2)	0.49912 (16)	0.0169 (8)	
C2_2	0.2812 (4)	0.4146 (2)	0.47095 (16)	0.0198 (9)	
H2A_2	0.194854	0.404279	0.491382	0.024*	
H2AB_2	0.314341	0.472185	0.482032	0.024*	
C3_2	0.2420 (4)	0.4077 (2)	0.40064 (18)	0.0192 (9)	
C4_2	0.2097 (4)	0.4002 (2)	0.34357 (18)	0.0199 (9)	
C5_2	0.1691 (4)	0.3879 (3)	0.27521 (17)	0.0200 (9)	
C6_2	0.133 (3)	0.4500 (14)	0.2293 (12)	0.023 (4)	0.575 (4)
H6_2	0.131394	0.50824	0.237789	0.028*	0.575 (4)
C6′_2	0.162 (4)	0.3085 (17)	0.2360 (13)	0.027 (5)	0.425 (4)
H6′_2	0.189015	0.254826	0.253066	0.032*	0.425 (4)
C7_2	0.0954 (4)	0.4089 (3)	0.16213 (19)	0.0252 (10)	
H7_2	0.066807	0.437284	0.122265	0.03*	
C8_2	0.1100 (4)	0.3262 (3)	0.16962 (18)	0.0242 (10)	
H8_2	0.094788	0.288821	0.133892	0.029*	
C9_2	0.3636 (4)	0.2613 (2)	0.48320 (16)	0.0166 (8)	
C10_2	0.2235 (4)	0.2333 (2)	0.46501 (17)	0.0217 (9)	
H10_2	0.146325	0.27216	0.459849	0.026*	
C11_2	0.1956 (5)	0.1489 (3)	0.45433 (18)	0.0285 (10)	
H11_2	0.099719	0.130536	0.440926	0.034*	
C12_2	0.3065 (5)	0.0914 (3)	0.46307 (18)	0.0284 (10)	
H12_2	0.28691	0.033578	0.457095	0.034*	
C13_2	0.4461 (5)	0.1192 (3)	0.48061 (18)	0.0268 (10)	
H13_2	0.522966	0.080209	0.485778	0.032*	
C14_2	0.4746 (4)	0.2031 (2)	0.49067 (17)	0.0219 (9)	
H14_2	0.571003	0.221311	0.502822	0.026*	
C15_2	0.4281 (4)	0.3632 (2)	0.57297 (16)	0.0151 (8)	
C16_2	0.3218 (4)	0.3432 (2)	0.60937 (17)	0.0186 (9)	
H16_2	0.231595	0.323301	0.588148	0.022*	
C17_2	0.3449 (4)	0.3519 (2)	0.67595 (17)	0.0189 (9)	
H17_2	0.271062	0.337854	0.700221	0.023*	
C18_2	0.4758 (4)	0.3811 (2)	0.70726 (17)	0.0199 (9)	
H18_2	0.491857	0.387674	0.753018	0.024*	
C19_2	0.5828 (4)	0.4005 (2)	0.67169 (17)	0.0185 (9)	
H19_2	0.673068	0.420244	0.693028	0.022*	
C20_2	0.5590 (4)	0.3914 (2)	0.60476 (17)	0.0166 (8)	
H20_2	0.633345	0.404736	0.580596	0.02*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1_1	0.0310 (6)	0.0225 (6)	0.0220 (5)	−0.0020 (5)	0.0003 (4)	0.0024 (5)
O1_1	0.0234 (15)	0.0185 (15)	0.0175 (14)	0.0029 (13)	0.0080 (11)	−0.0002 (12)
C1_1	0.0135 (19)	0.018 (2)	0.0140 (18)	0.0011 (16)	0.0045 (15)	−0.0005 (16)

C2_1	0.022 (2)	0.021 (2)	0.016 (2)	−0.0040 (18)	0.0014 (16)	0.0030 (16)
C3_1	0.017 (2)	0.021 (2)	0.021 (2)	−0.0035 (18)	0.0023 (16)	−0.0008 (17)
C4_1	0.012 (2)	0.022 (2)	0.0185 (19)	−0.0017 (17)	0.0012 (16)	−0.0031 (16)
C5_1	0.012 (2)	0.017 (2)	0.0192 (19)	−0.0001 (17)	0.0027 (16)	0.0004 (15)
C6_1	0.013 (2)	0.021 (2)	0.017 (2)	−0.0002 (17)	−0.0005 (16)	−0.0007 (16)
C7_1	0.014 (2)	0.030 (2)	0.019 (2)	0.0028 (18)	0.0025 (16)	−0.0004 (17)
C8_1	0.020 (2)	0.031 (2)	0.018 (2)	0.0059 (19)	0.0054 (17)	0.0096 (17)
C9_1	0.023 (2)	0.018 (2)	0.0081 (19)	0.0033 (16)	0.0045 (16)	0.0003 (15)
C10_1	0.026 (2)	0.024 (2)	0.018 (2)	0.0050 (18)	0.0085 (17)	0.0014 (17)
C11_1	0.039 (3)	0.026 (2)	0.019 (2)	0.015 (2)	0.0068 (19)	0.0023 (18)
C12_1	0.057 (3)	0.016 (2)	0.019 (2)	0.001 (2)	0.009 (2)	−0.0002 (17)
C13_1	0.040 (3)	0.024 (2)	0.021 (2)	−0.010 (2)	0.006 (2)	−0.0034 (18)
C14_1	0.027 (2)	0.020 (2)	0.017 (2)	−0.0009 (18)	0.0034 (17)	0.0002 (17)
C15_1	0.023 (2)	0.012 (2)	0.0101 (18)	−0.0034 (16)	0.0011 (15)	0.0004 (15)
C16_1	0.015 (2)	0.024 (2)	0.0160 (19)	0.0024 (17)	0.0014 (15)	0.0036 (17)
C17_1	0.023 (2)	0.026 (2)	0.017 (2)	−0.0051 (18)	0.0077 (17)	−0.0025 (17)
C18_1	0.027 (2)	0.023 (2)	0.014 (2)	−0.0082 (19)	−0.0016 (17)	0.0008 (17)
C19_1	0.021 (2)	0.019 (2)	0.020 (2)	0.0028 (18)	−0.0015 (16)	0.0022 (17)
C20_1	0.020 (2)	0.017 (2)	0.0166 (19)	−0.0005 (17)	0.0027 (16)	−0.0002 (16)
S1_2	0.0247 (18)	0.0207 (19)	0.0177 (16)	0.0029 (14)	−0.0013 (12)	−0.0034 (11)
S1′_2	0.024 (3)	0.023 (3)	0.016 (2)	0.0003 (19)	−0.0058 (17)	0.0051 (15)
O1_2	0.0211 (15)	0.0330 (17)	0.0128 (13)	−0.0020 (13)	0.0068 (12)	0.0039 (13)
C1_2	0.017 (2)	0.023 (2)	0.0107 (18)	0.0007 (17)	0.0027 (15)	0.0012 (16)
C2_2	0.022 (2)	0.022 (2)	0.0146 (19)	0.0030 (18)	−0.0002 (17)	0.0030 (16)
C3_2	0.014 (2)	0.024 (2)	0.019 (2)	0.0025 (18)	0.0030 (16)	0.0025 (17)
C4_2	0.010 (2)	0.029 (2)	0.022 (2)	0.0003 (17)	0.0027 (16)	0.0042 (17)
C5_2	0.014 (2)	0.029 (2)	0.0165 (19)	−0.0015 (18)	0.0024 (16)	0.0023 (17)
C6_2	0.028 (10)	0.023 (7)	0.020 (6)	−0.004 (6)	0.008 (6)	−0.003 (4)
C6′_2	0.026 (10)	0.024 (7)	0.029 (6)	−0.007 (7)	−0.003 (7)	0.003 (5)
C7_2	0.019 (2)	0.033 (3)	0.023 (2)	0.000 (2)	0.0023 (17)	0.0048 (18)
C8_2	0.017 (2)	0.038 (3)	0.017 (2)	0.000 (2)	0.0010 (17)	−0.0071 (18)
C9_2	0.023 (2)	0.021 (2)	0.0053 (18)	0.0002 (17)	0.0001 (15)	−0.0001 (15)
C10_2	0.023 (2)	0.026 (2)	0.015 (2)	0.0020 (18)	0.0014 (17)	0.0008 (17)
C11_2	0.032 (2)	0.034 (3)	0.018 (2)	−0.005 (2)	0.0007 (18)	−0.0039 (19)
C12_2	0.046 (3)	0.019 (2)	0.020 (2)	−0.004 (2)	0.004 (2)	−0.0049 (18)
C13_2	0.036 (2)	0.024 (2)	0.019 (2)	0.004 (2)	0.0011 (19)	−0.0017 (18)
C14_2	0.026 (2)	0.024 (2)	0.014 (2)	0.0026 (18)	−0.0010 (17)	0.0001 (17)
C15_2	0.018 (2)	0.014 (2)	0.0136 (18)	0.0049 (16)	0.0034 (15)	0.0002 (15)
C16_2	0.016 (2)	0.024 (2)	0.0152 (19)	0.0015 (18)	0.0016 (15)	0.0012 (17)
C17_2	0.022 (2)	0.022 (2)	0.015 (2)	0.0012 (18)	0.0070 (16)	0.0007 (16)
C18_2	0.028 (2)	0.019 (2)	0.0126 (19)	0.0027 (18)	0.0017 (16)	−0.0017 (16)
C19_2	0.018 (2)	0.017 (2)	0.019 (2)	0.0014 (17)	−0.0025 (16)	−0.0032 (16)
C20_2	0.015 (2)	0.016 (2)	0.020 (2)	−0.0015 (17)	0.0042 (16)	0.0035 (16)

Geometric parameters (Å, º)

S1_1—C8_1	1.706 (4)	S1′_2—C5_2	1.660 (7)
S1_1—C5_1	1.734 (4)	O1_2—C1_2	1.424 (4)

O1_1—C1_1	1.437 (4)	O1_2—H1_2	0.85 (4)
O1_1—H1_1	0.85 (4)	C1_2—C9_2	1.528 (5)
C1_1—C9_1	1.518 (5)	C1_2—C15_2	1.533 (5)
C1_1—C2_1	1.536 (5)	C1_2—C2_2	1.547 (5)
C1_1—C15_1	1.539 (5)	C2_2—C3_2	1.461 (5)
C2_1—C3_1	1.470 (5)	C2_2—H2A_2	0.99
C2_1—H2A_1	0.99	C2_2—H2AB_2	0.99
C2_1—H2AB_1	0.99	C3_2—C4_2	1.189 (5)
C3_1—C4_1	1.193 (5)	C4_2—C5_2	1.433 (5)
C4_1—C5_1	1.420 (5)	C5_2—C6_2	1.388 (18)
C5_1—C6_1	1.369 (5)	C5_2—C6′_2	1.51 (3)
C6_1—C7_1	1.415 (5)	C6_2—C7_2	1.54 (3)
C6_1—H6_1	0.95	C6_2—H6_2	0.95
C7_1—C8_1	1.356 (5)	C6′_2—C8_2	1.43 (3)
C7_1—H7_1	0.95	C6′_2—H6′_2	0.95
C8_1—H8_1	0.95	C7_2—C8_2	1.339 (5)
C9_1—C14_1	1.389 (5)	C7_2—H7_2	0.95
C9_1—C10_1	1.392 (5)	C8_2—H8_2	0.95
C10_1—C11_1	1.383 (5)	C9_2—C10_2	1.390 (5)
C10_1—H10_1	0.95	C9_2—C14_2	1.391 (5)
C11_1—C12_1	1.381 (6)	C10_2—C11_2	1.389 (5)
C11_1—H11_1	0.95	C10_2—H10_2	0.95
C12_1—C13_1	1.379 (6)	C11_2—C12_2	1.383 (6)
C12_1—H12_1	0.95	C11_2—H11_2	0.95
C13_1—C14_1	1.376 (5)	C12_2—C13_2	1.382 (6)
C13_1—H13_1	0.95	C12_2—H12_2	0.95
C14_1—H14_1	0.95	C13_2—C14_2	1.381 (5)
C15_1—C20_1	1.387 (5)	C13_2—H13_2	0.95
C15_1—C16_1	1.391 (5)	C14_2—H14_2	0.95
C16_1—C17_1	1.380 (5)	C15_2—C20_2	1.382 (5)
C16_1—H16_1	0.95	C15_2—C16_2	1.387 (5)
C17_1—C18_1	1.386 (5)	C16_2—C17_2	1.381 (5)
C17_1—H17_1	0.95	C16_2—H16_2	0.95
C18_1—C19_1	1.376 (5)	C17_2—C18_2	1.384 (5)
C18_1—H18_1	0.95	C17_2—H17_2	0.95
C19_1—C20_1	1.387 (5)	C18_2—C19_2	1.379 (5)
C19_1—H19_1	0.95	C18_2—H18_2	0.95
C20_1—H20_1	0.95	C19_2—C20_2	1.388 (5)
S1_2—C5_2	1.640 (7)	C19_2—H19_2	0.95
S1_2—C8_2	1.681 (6)	C20_2—H20_2	0.95
S1′_2—C7_2	1.574 (11)		

C8_1—S1_1—C5_1	91.83 (19)	C9_2—C1_2—C15_2	108.8 (3)
C1_1—O1_1—H1_1	106 (3)	O1_2—C1_2—C2_2	109.0 (3)
O1_1—C1_1—C9_1	105.1 (3)	C9_2—C1_2—C2_2	113.9 (3)
O1_1—C1_1—C2_1	108.2 (3)	C15_2—C1_2—C2_2	108.0 (3)
C9_1—C1_1—C2_1	114.0 (3)	C3_2—C2_2—C1_2	112.8 (3)
O1_1—C1_1—C15_1	110.3 (3)	C3_2—C2_2—H2A_2	109.0

C9_1—C1_1—C15_1	109.3 (3)	C1_2—C2_2—H2A_2	109.0
C2_1—C1_1—C15_1	109.9 (3)	C3_2—C2_2—H2AB_2	109.0
C3_1—C2_1—C1_1	111.8 (3)	C1_2—C2_2—H2AB_2	109.0
C3_1—C2_1—H2A_1	109.3	H2A_2—C2_2—H2AB_2	107.8
C1_1—C2_1—H2A_1	109.3	C4_2—C3_2—C2_2	178.5 (4)
C3_1—C2_1—H2AB_1	109.3	C3_2—C4_2—C5_2	177.8 (4)
C1_1—C2_1—H2AB_1	109.3	C6_2—C5_2—C4_2	126.1 (12)
H2A_1—C2_1—H2AB_1	107.9	C4_2—C5_2—C6′_2	129.6 (10)
C4_1—C3_1—C2_1	176.4 (4)	C6_2—C5_2—S1_2	114.7 (11)
C3_1—C4_1—C5_1	178.7 (4)	C4_2—C5_2—S1_2	119.2 (3)
C6_1—C5_1—C4_1	129.6 (4)	C4_2—C5_2—S1′_2	122.7 (4)
C6_1—C5_1—S1_1	110.9 (3)	C6′_2—C5_2—S1′_2	107.6 (10)
C4_1—C5_1—S1_1	119.6 (3)	C5_2—C6_2—C7_2	108.6 (16)
C5_1—C6_1—C7_1	112.2 (4)	C5_2—C6_2—H6_2	125.7
C5_1—C6_1—H6_1	123.9	C7_2—C6_2—H6_2	125.7
C7_1—C6_1—H6_1	123.9	C8_2—C6′_2—C5_2	109.8 (16)
C8_1—C7_1—C6_1	113.4 (4)	C8_2—C6′_2—H6′_2	125.1
C8_1—C7_1—H7_1	123.3	C5_2—C6′_2—H6′_2	125.1
C6_1—C7_1—H7_1	123.3	C8_2—C7_2—C6_2	108.2 (8)
C7_1—C8_1—S1_1	111.7 (3)	C8_2—C7_2—S1′_2	118.7 (4)
C7_1—C8_1—H8_1	124.2	C8_2—C7_2—H7_2	125.9
S1_1—C8_1—H8_1	124.2	C6_2—C7_2—H7_2	125.9
C14_1—C9_1—C10_1	118.4 (4)	C7_2—C8_2—C6′_2	108.9 (12)
C14_1—C9_1—C1_1	118.7 (3)	C7_2—C8_2—S1_2	115.7 (4)
C10_1—C9_1—C1_1	122.9 (3)	C7_2—C8_2—H8_2	122.1
C11_1—C10_1—C9_1	120.5 (4)	S1_2—C8_2—H8_2	122.1
C11_1—C10_1—H10_1	119.8	C10_2—C9_2—C14_2	118.7 (4)
C9_1—C10_1—H10_1	119.8	C10_2—C9_2—C1_2	122.8 (3)
C12_1—C11_1—C10_1	120.6 (4)	C14_2—C9_2—C1_2	118.4 (3)
C12_1—C11_1—H11_1	119.7	C11_2—C10_2—C9_2	120.3 (4)
C10_1—C11_1—H11_1	119.7	C11_2—C10_2—H10_2	119.8
C13_1—C12_1—C11_1	119.0 (4)	C9_2—C10_2—H10_2	119.8
C13_1—C12_1—H12_1	120.5	C12_2—C11_2—C10_2	120.5 (4)
C11_1—C12_1—H12_1	120.5	C12_2—C11_2—H11_2	119.7
C14_1—C13_1—C12_1	120.8 (4)	C10_2—C11_2—H11_2	119.7
C14_1—C13_1—H13_1	119.6	C13_2—C12_2—C11_2	119.2 (4)
C12_1—C13_1—H13_1	119.6	C13_2—C12_2—H12_2	120.4
C13_1—C14_1—C9_1	120.7 (4)	C11_2—C12_2—H12_2	120.4
C13_1—C14_1—H14_1	119.7	C14_2—C13_2—C12_2	120.5 (4)
C9_1—C14_1—H14_1	119.7	C14_2—C13_2—H13_2	119.7
C20_1—C15_1—C16_1	118.5 (3)	C12_2—C13_2—H13_2	119.7
C20_1—C15_1—C1_1	121.0 (3)	C13_2—C14_2—C9_2	120.7 (4)
C16_1—C15_1—C1_1	120.5 (3)	C13_2—C14_2—H14_2	119.7
C17_1—C16_1—C15_1	121.0 (3)	C9_2—C14_2—H14_2	119.7
C17_1—C16_1—H16_1	119.5	C20_2—C15_2—C16_2	118.6 (3)
C15_1—C16_1—H16_1	119.5	C20_2—C15_2—C1_2	121.1 (3)
C16_1—C17_1—C18_1	120.0 (4)	C16_2—C15_2—C1_2	120.2 (3)
C16_1—C17_1—H17_1	120.0	C17_2—C16_2—C15_2	121.1 (4)

C18_1—C17_1—H17_1	120.0	C17_2—C16_2—H16_2	119.5
C19_1—C18_1—C17_1	119.6 (3)	C15_2—C16_2—H16_2	119.5
C19_1—C18_1—H18_1	120.2	C16_2—C17_2—C18_2	119.9 (4)
C17_1—C18_1—H18_1	120.2	C16_2—C17_2—H17_2	120.1
C18_1—C19_1—C20_1	120.5 (4)	C18_2—C17_2—H17_2	120.1
C18_1—C19_1—H19_1	119.8	C19_2—C18_2—C17_2	119.7 (3)
C20_1—C19_1—H19_1	119.8	C19_2—C18_2—H18_2	120.2
C19_1—C20_1—C15_1	120.4 (4)	C17_2—C18_2—H18_2	120.2
C19_1—C20_1—H20_1	119.8	C18_2—C19_2—C20_2	120.2 (4)
C15_1—C20_1—H20_1	119.8	C18_2—C19_2—H19_2	119.9
C5_2—S1_2—C8_2	92.7 (3)	C20_2—C19_2—H19_2	119.9
C7_2—S1′_2—C5_2	94.7 (5)	C15_2—C20_2—C19_2	120.6 (3)
C1_2—O1_2—H1_2	109 (3)	C15_2—C20_2—H20_2	119.7
O1_2—C1_2—C9_2	110.6 (3)	C19_2—C20_2—H20_2	119.7
O1_2—C1_2—C15_2	106.2 (3)		

Hydrogen-bond geometry (Å, º)

Cg1, Cg2 and Cg3 are the centroids of the C15_2–C20_2, C15_1–C20_1 and S1_1/C5_1–C8_1 rings, respectively; Cg4 is the centroid of the disordered S1_2/S1′_2/C6_2/C6′_2/C5_2/C7_2/C8_2 thiophene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1_2—H1_2···O1_1	0.85 (4)	2.08 (4)	2.864 (4)	153 (4)
O1_1—H1_1···*Cg*1^{i}	0.86 (5)	2.69 (5)	3.516 (3)	162 (4)
C8_1—H8_1···*Cg*2^{ii}	0.95	2.88	3.489 (5)	123
C8_2—H8_2···*Cg*1^{iii}	0.95	2.92	3.519 (5)	123
C16_2—H16_2···*Cg*3^{iv}	0.95	2.86	3.648 (4)	141
C20_2—H20_2···*Cg*3	0.95	2.99	3.658 (4)	129
C16_1—H16_1···*Cg*4^{v}	0.95	2.92	3.679 (6)	137

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*+1/2, −*y*+1/2, *z*+1/2; (iii) *x*−1/2, −*y*+1/2, *z*−1/2; (iv) *x*−1, *y*, *z*; (v) *x*+1, *y*, *z*.