

1-1-2007

(Z)-2-Phenyl-3-(2,2':5',2"-terthiophen-3'-yl)acrylonitrile

David L. Officer

University of Wollongong, davido@uow.edu.au

Pawel W. Wagner

University of Wollongong, pawel@uow.edu.au

Maciej Kubicki

Adam Mickiewicz university

Follow this and additional works at: <https://ro.uow.edu.au/scipapers>



Part of the [Life Sciences Commons](#), [Physical Sciences and Mathematics Commons](#), and the [Social and Behavioral Sciences Commons](#)

Recommended Citation

Officer, David L.; Wagner, Pawel W.; and Kubicki, Maciej: (Z)-2-Phenyl-3-(2,2':5',2"-terthiophen-3'-yl)acrylonitrile 2007, o3054-o3055.
<https://ro.uow.edu.au/scipapers/3643>

(Z)-2-Phenyl-3-(2,2':5',2"-terthiophen-3'-yl)acrylonitrile

Abstract

In the crystal structure of the title compd., C₂₁H₁₃NS₃, one of the terminal thiophene rings is disordered in approx. a 3:1 ratio of components, and the disorder is of the flip type. The terthiophene units are far from coplanarity, in contrast to terthiophene itself. The conformation might be described, for the unit of greater occupancy, as quasi-cis [the S-C-C-S torsion angle is 41.1 (5)degrees] quasi-trans [the S-C-C-S torsion angle is -140.1 (4)degrees], and for the less-occupied part as quasi-cis-quasi-cis [35.7 (6)degrees]. The dihedral angle between the terminal ring planes is as high as 71.7 (2)degrees. The central C-C=C-C group is almost planar [max. deviation of 0.027 (3) .ANG.] and it is approx. equally twisted with respect to both the central thiophene and the Ph ring planes [26.5 (4) and 23.5 (7)degrees, resp.]. The crystal packing is detd. by van der Waals interactions, and some weak C-H and C-H_p relatively short directional contacts.

Keywords

Phenyl, terthiophen, acrylonitrile

Disciplines

Life Sciences | Physical Sciences and Mathematics | Social and Behavioral Sciences

Publication Details

Wagner, P. W., Officer, D. L. & Kubicki, M. (2007). (Z)-2-Phenyl-3-(2,2':5',2"-terthiophen-3'-yl)acrylonitrile. Acta Crystallographica Section E: Structure Reports Online, 63 (7), o3054-o3055.

(Z)-2-Phenyl-3-(2,2':5',2''-terthiophen-3'-yl)acrylonitrile

Paweł Wagner,^a David L. Officer^b and Maciej Kubicki^{c*}

^aNanomaterials Research Centre, and MacDiarmid Institute for Advanced Materials and Nanotechnology, Massey University, Private Bag 11 222, Palmerston North, New Zealand, ^bARC Centre of Excellence for Electromaterials Science, University of Wollongong, Northfield Avenue, Wollongong, NSW 2522, Australia, and

^cDepartment of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland

Correspondence e-mail: mkubicki@amu.edu.pl

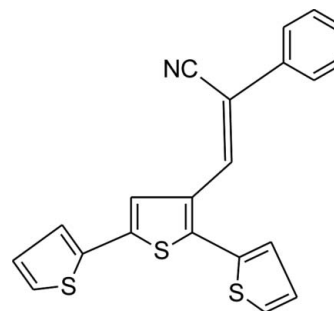
Received 21 May 2007; accepted 24 May 2007

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; disorder in main residue; R factor = 0.066; wR factor = 0.153; data-to-parameter ratio = 13.1.

In the crystal structure of the title compound, $\text{C}_{21}\text{H}_{13}\text{NS}_3$, one of the terminal thiophene rings is disordered in approximately a 3:1 ratio of components, and the disorder is of the flip type. The terthiophene units are far from coplanarity, in contrast to terthiophene itself. The conformation might be described, for the unit of greater occupancy, as quasi-*cis* [the $\text{S}-\text{C}-\text{C}-\text{S}$ torsion angle is $41.1(5)^\circ$] quasi-*trans* [the $\text{S}-\text{C}-\text{C}-\text{S}$ torsion angle is $-140.1(4)^\circ$], and for the less-occupied part as quasi-*cis*-quasi-*cis* [$35.7(6)^\circ$]. The dihedral angle between the terminal ring planes is as high as $71.7(2)^\circ$. The central $\text{C}=\text{C}-\text{C}$ group is almost planar [maximum deviation of $0.027(3)$ Å] and it is approximately equally twisted with respect to both the central thiophene and the phenyl ring planes [$26.5(4)$ and $23.5(7)^\circ$, respectively]. The crystal packing is determined by van der Waals interactions, and some weak $\text{C}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\pi$ relatively short directional contacts.

Related literature

Similar flip-type disorder was observed in other 3'-arylviny derivatives of terthiophene [without additional substituents at the vinyl group, e.g. Wagner *et al.* (2007)], or in simple thiophene derivatives (e.g. Sonar *et al.*, 2004, 2005; Wagner *et al.*, 2006). For related literature, see: Clarke *et al.* (2007); Collis *et al.* (2003); Grant & Officer (2005); Roncali (1999); Skotheim & Reynolds (2007).



Experimental

Crystal data

$\text{C}_{21}\text{H}_{13}\text{NS}_3$
 $M_r = 375.50$
 Monoclinic, $P2_1/c$
 $a = 9.167(1)$ Å
 $b = 19.564(2)$ Å
 $c = 10.424(1)$ Å
 $\beta = 111.30(1)^\circ$

$V = 1741.8(3)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.43$ mm⁻¹
 $T = 173(1)$ K
 $0.26 \times 0.2 \times 0.16$ mm

Data collection

Bruker P4 CCD diffractometer
 Absorption correction: none
 10129 measured reflections

3054 independent reflections
 1861 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.149$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.153$
 $S = 1.02$
 3054 reflections
 233 parameters

51 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.56$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.68$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C13}-\text{H13}\cdots\text{N19}^i$	0.93	2.54	3.435 (7)	161

Symmetry code: (i) $-x + 1, -y, -z - 1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINTE-Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2015).

References

- Bruker (2001). *SAINTE-Plus* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.
 Clarke, T. M., Gordon, K. C., Wagner, P. & Officer, D. L. (2007). *J. Phys. Chem. A*, **111**, 2385–2397.
 Collis, G. A., Burrell, A. K., Scott, S. M. & Officer, D. L. (2003). *J. Org. Chem.* **68**, 8974–8983.
 Grant, D. K. & Officer, D. L. (2005). *Synth. Met.* **154**, 93–95.

- Roncali, J. (1999). *Annu. Rep. Prog. Chem. Sect. C Phys. Chem.* **95**, 47–88.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Siemens (1989). *Stereochemical Workstation Operation Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Skotheim, T. A. & Reynolds, J. R. (2007). *Handbook of Conducting Polymers*, 3rd ed. New York: Taylor & Francis Group.
- Sonar, V. N., Parkin, S. & Crooks, P. A. (2004). *Acta Cryst.* **C60**, o217–o218.
- Sonar, V. N., Parkin, S. & Crooks, P. A. (2005). *Acta Cryst.* **E61**, o933–o935.
- Wagner, P., Officer, D. L. & Kubicki, M. (2006). *Acta Cryst.* **E62**, o5745–o5747.
- Wagner, P., Officer, D. L. & Kubicki, M. (2007). *Acta Cryst.* **C63**. In the press.

supplementary materials

Acta Cryst. (2007). E63, o3054–o3055 [doi:10.1107/S1600536807025366]

(Z)-2-Phenyl-3-(2,2':5',2''-terthiophen-3'-yl)acrylonitrile

P. Wagner, D. L. Officer and M. Kubicki

Comment

Oligo- and polythiophenes, potentially applicable as solar cells, light-emitting diodes or NLO materials (Skotheim & Reynolds, 2007) are widely studied due to their good chemical stability in both oxidized and reduced states, and to relatively easy functionalization of the monomers whether thiophene, bithiophene, or terthiophene (for example: Roncali, 1999; Grant & Officer, 2005 and references therein).

Recently we have proposed an alternative approach to the formation of regioregular styryl-functionalized oligo- and polythiophenes (for example, Collis *et al.*, 2003; Grant & Officer, 2005). We have demonstrated that the styryl functionality can control oligomer regioregularity and provides advantages in some applications. However styrylterthiophenes largely form dimers on oxidative polymerization as a result of "polaron trapping" (Clarke *et al.*, 2007).

There is a flip-disorder of one of the terminal thiophene rings. The disorder is connected with two statistically distributed orientations of the thiophene sulfur atom. In practice that means that there are two molecules in which the thiophene rings are rotated by 180° approximately along the line that bisects the S—C—C angle. These two orientations are not equivalent, but they are distributed approximately in 3:1 proportion. The conformation of the molecule of greater occupancy might be described as quasi-*trans*-quasi-*cis*, while that of smaller occupancy is quasi-*cis*-quasi-*cis*. A disorder of this kind is often observed in the structures of simple thiophene and terthiophene derivatives with one substituent; for example in similar (*E*)-3'-(2-(4-cyanophenyl)ethenyl)-[2,2':5',2'']terthiophene (Collis *et al.*, 2003) or in 3-(2-(anthracen-9-yl)ethenyl)-thiophene (Wagner *et al.*, 2006).

The terthiophene rings are approximately planar (maximum deviation is 0.020 (6) Å), but the dihedral angles between their planes are large (for instance, between the terminal rings it is 71.7 (2)°). Also the planar (within 0.012 (3) Å) phenyl ring is inclined by 49.9 (2)° with respect to the central thiophene ring. In the crystal structure the van der Waals forces seem to determine the packing. Some weak specific C13—H13⋯N19ⁱ and C25—H25⋯π(Cg1ⁱⁱ) (Cg1 is the centroid of C11⋯C15 thiophene ring, symmetry codes as in Table 1) directional interactions also might be of some importance.

Refinement

Hydrogen atoms were placed at calculated positions and refined as 'riding model' with isotropic thermal parameters set at 1.2 (1.3 for methyl groups) times the U_{eq} values of appropriate carrier atoms. In the disordered part, the carbon atoms of the were constrained to have the same components of the displacement tensor as the sulfur atoms occupying the same site. Weak restraints were also applied to the geometry of disordered fragment.

Figures

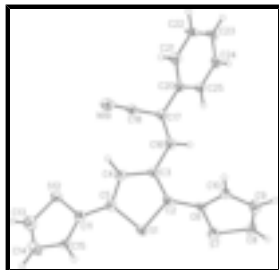


Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level for non H-atoms (Siemens, 1989)

(2Z)-2-Phenyl-3-(2,2':5',2''-terthiophen-3'-yl)acrylonitrile

Crystal data

$C_{21}H_{13}NS_3$

$M_r = 375.50$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.167 (1) \text{ \AA}$

$b = 19.564 (2) \text{ \AA}$

$c = 10.424 (1) \text{ \AA}$

$\beta = 111.30 (1)^\circ$

$V = 1741.8 (3) \text{ \AA}^3$

$Z = 4$

$F_{000} = 776$

$D_x = 1.432 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2212 reflections

$\theta = 5\text{--}15^\circ$

$\mu = 0.43 \text{ mm}^{-1}$

$T = 173 (1) \text{ K}$

Block, yellow

$0.26 \times 0.2 \times 0.16 \text{ mm}$

Data collection

Bruker P4 CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 173(1) \text{ K}$

ω scans

Absorption correction: none

10129 measured reflections

3054 independent reflections

1861 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.149$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.1^\circ$

$h = -10 \rightarrow 10$

$k = -23 \rightarrow 22$

$l = -11 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.066$

$wR(F^2) = 0.153$

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} < 0.001$$

$S = 1.02$ $\Delta\rho_{\max} = 0.56 \text{ e } \text{\AA}^{-3}$
 3054 reflections $\Delta\rho_{\min} = -0.68 \text{ e } \text{\AA}^{-3}$
 233 parameters Extinction correction: none
 51 restraints
 Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.56931 (14)	0.14221 (6)	-0.00033 (13)	0.0210 (3)	
C2	0.6725 (5)	0.0783 (2)	0.1084 (5)	0.0159 (11)	
C3	0.7043 (6)	0.0252 (2)	0.0376 (5)	0.0183 (11)	
C4	0.6420 (5)	0.0369 (2)	-0.1078 (5)	0.0178 (11)	
H4	0.6522	0.0058	-0.1716	0.021*	
C5	0.5666 (5)	0.0978 (2)	-0.1438 (5)	0.0185 (11)	
C6	0.7141 (5)	0.0857 (2)	0.2557 (5)	0.0180 (11)	
S7	0.58362 (16)	0.11909 (7)	0.32299 (13)	0.0260 (4)	
C8	0.7152 (6)	0.1126 (3)	0.4875 (5)	0.0260 (13)	
H8	0.6952	0.1262	0.5650	0.031*	
C9	0.8511 (6)	0.0855 (3)	0.4913 (5)	0.0270 (13)	
H9	0.9350	0.0775	0.5729	0.032*	
C10	0.8555 (5)	0.0704 (2)	0.3611 (4)	0.0158 (11)	
H10	0.9423	0.0524	0.3469	0.019*	
C11	0.4806 (5)	0.1228 (2)	-0.2812 (5)	0.0201 (11)	
S12	0.3782 (5)	0.06990 (10)	-0.4104 (3)	0.0205 (7)	0.725 (5)
C12	0.379 (5)	0.0865 (15)	-0.413 (3)	0.0205 (7)	0.275 (5)
H12	0.3736	0.0391	-0.4138	0.025*	0.275 (5)
C13	0.3084 (6)	0.1337 (3)	-0.5189 (6)	0.0325 (14)	
H13	0.2411	0.1261	-0.6091	0.039*	
C14	0.3516 (6)	0.1956 (3)	-0.4733 (5)	0.0314 (14)	
H14	0.3198	0.2362	-0.5218	0.038*	
C15	0.4584 (14)	0.1880 (5)	-0.3325 (10)	0.0231 (12)	0.725 (5)
H15	0.5091	0.2253	-0.2798	0.028*	0.725 (5)
S15	0.4713 (9)	0.2068 (2)	-0.3123 (6)	0.0231 (12)	0.275 (5)

supplementary materials

C16	0.7830 (5)	-0.0363 (2)	0.1054 (5)	0.0192 (11)
H16	0.7778	-0.0452	0.1913	0.023*
C17	0.8621 (5)	-0.0818 (2)	0.0607 (5)	0.0171 (11)
C18	0.8859 (5)	-0.0716 (2)	-0.0653 (5)	0.0172 (11)
N19	0.9073 (5)	-0.0643 (2)	-0.1664 (4)	0.0265 (11)
C20	0.9287 (5)	-0.1459 (2)	0.1335 (5)	0.0175 (11)
C21	1.0512 (6)	-0.1787 (3)	0.1111 (5)	0.0223 (12)
H21	1.0948	-0.1594	0.0518	0.027*
C22	1.1089 (6)	-0.2397 (3)	0.1758 (5)	0.0266 (13)
H22	1.1898	-0.2618	0.1586	0.032*
C23	1.0471 (6)	-0.2681 (3)	0.2657 (5)	0.0260 (13)
H23	1.0861	-0.3092	0.3095	0.031*
C24	0.9279 (5)	-0.2354 (3)	0.2906 (5)	0.0238 (12)
H24	0.8860	-0.2544	0.3515	0.029*
C25	0.8696 (5)	-0.1743 (2)	0.2257 (5)	0.0196 (11)
H25	0.7898	-0.1521	0.2443	0.024*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0144 (7)	0.0187 (7)	0.0234 (7)	0.0031 (6)	-0.0008 (5)	-0.0007 (5)
C2	0.0102 (18)	0.0141 (18)	0.0197 (17)	0.0004 (15)	0.0008 (14)	0.0010 (14)
C3	0.0141 (19)	0.0172 (18)	0.0217 (18)	-0.0004 (15)	0.0043 (15)	-0.0006 (15)
C4	0.009 (3)	0.020 (3)	0.022 (3)	-0.003 (2)	0.002 (2)	-0.007 (2)
C5	0.010 (3)	0.023 (3)	0.018 (3)	-0.003 (2)	0.000 (2)	0.000 (2)
C6	0.018 (3)	0.011 (2)	0.022 (3)	0.000 (2)	0.003 (2)	0.001 (2)
S7	0.0199 (8)	0.0254 (7)	0.0299 (8)	-0.0009 (6)	0.0056 (6)	-0.0038 (6)
C8	0.031 (3)	0.027 (3)	0.017 (3)	-0.008 (3)	0.005 (2)	-0.007 (2)
C9	0.022 (3)	0.027 (3)	0.023 (3)	-0.008 (3)	-0.003 (2)	-0.001 (2)
C10	0.0124 (18)	0.0140 (17)	0.0192 (18)	-0.0028 (15)	0.0035 (14)	-0.0001 (14)
C11	0.0154 (19)	0.0203 (19)	0.0215 (18)	-0.0012 (16)	0.0032 (15)	-0.0036 (15)
S12	0.0168 (10)	0.0152 (15)	0.0226 (10)	-0.0020 (12)	-0.0010 (7)	0.0007 (9)
C12	0.0168 (10)	0.0152 (15)	0.0226 (10)	-0.0020 (12)	-0.0010 (7)	0.0007 (9)
C13	0.023 (3)	0.038 (4)	0.029 (3)	-0.006 (3)	0.001 (2)	0.000 (3)
C14	0.018 (3)	0.034 (3)	0.038 (3)	0.005 (3)	0.006 (3)	0.014 (3)
C15	0.0199 (16)	0.019 (2)	0.0227 (17)	-0.0020 (17)	-0.0010 (13)	-0.0084 (16)
S15	0.0199 (16)	0.019 (2)	0.0227 (17)	-0.0020 (17)	-0.0010 (13)	-0.0084 (16)
C16	0.011 (3)	0.021 (3)	0.021 (3)	-0.003 (2)	-0.001 (2)	-0.001 (2)
C17	0.0134 (18)	0.0159 (18)	0.0178 (17)	-0.0018 (15)	0.0006 (14)	-0.0025 (14)
C18	0.0126 (18)	0.0162 (18)	0.0193 (18)	0.0005 (15)	0.0016 (14)	-0.0022 (15)
N19	0.014 (2)	0.029 (3)	0.030 (3)	0.003 (2)	0.0006 (19)	-0.004 (2)
C20	0.0141 (18)	0.0163 (18)	0.0171 (17)	-0.0001 (16)	-0.0002 (14)	-0.0016 (15)
C21	0.015 (3)	0.024 (3)	0.026 (3)	-0.001 (2)	0.004 (2)	-0.002 (2)
C22	0.013 (3)	0.024 (3)	0.035 (3)	0.009 (3)	-0.001 (2)	-0.004 (2)
C23	0.017 (3)	0.021 (3)	0.031 (3)	0.008 (3)	-0.003 (2)	-0.001 (2)
C24	0.017 (3)	0.027 (3)	0.019 (3)	0.000 (3)	-0.003 (2)	0.000 (2)
C25	0.007 (3)	0.019 (3)	0.025 (3)	0.002 (2)	-0.003 (2)	-0.008 (2)

Geometric parameters (Å, °)

S1—C2	1.721 (5)	C12—H12	0.9300
S1—C5	1.722 (5)	C13—C14	1.309 (7)
C2—C3	1.366 (6)	C13—H13	0.9300
C2—C6	1.449 (6)	C14—C15	1.446 (11)
C3—C4	1.431 (6)	C14—S15	1.653 (5)
C3—C16	1.448 (6)	C14—H14	0.9300
C4—C5	1.360 (6)	C15—H15	0.9300
C4—H4	0.9300	C16—C17	1.335 (6)
C5—C11	1.447 (6)	C16—H16	0.9300
C6—C10	1.394 (6)	C17—C18	1.422 (6)
C6—S7	1.720 (5)	C17—C20	1.479 (6)
S7—C8	1.706 (5)	C18—N19	1.148 (5)
C8—C9	1.342 (7)	C20—C25	1.380 (6)
C8—H8	0.9300	C20—C21	1.384 (6)
C9—C10	1.404 (6)	C21—C22	1.378 (7)
C9—H9	0.9300	C21—H21	0.9300
C10—H10	0.9300	C22—C23	1.376 (7)
C11—C15	1.369 (10)	C22—H22	0.9300
C11—C12	1.53 (4)	C23—C24	1.370 (6)
C11—S15	1.670 (4)	C23—H23	0.9300
C11—S12	1.688 (5)	C24—C25	1.382 (7)
S12—C13	1.650 (6)	C24—H24	0.9300
C12—C13	1.402 (5)	C25—H25	0.9300
C2—S1—C5	92.1 (2)	C14—C13—H13	121.2
C3—C2—C6	128.5 (4)	C12—C13—H13	129.5
C3—C2—S1	111.7 (3)	S12—C13—H13	121.4
C6—C2—S1	119.8 (3)	C13—C14—C15	106.1 (6)
C2—C3—C4	111.7 (4)	C13—C14—S15	119.5 (4)
C2—C3—C16	122.3 (4)	C13—C14—H14	126.9
C4—C3—C16	125.9 (4)	C15—C14—H14	127.0
C5—C4—C3	113.5 (4)	S15—C14—H14	113.6
C5—C4—H4	123.3	C11—C15—C14	116.2 (7)
C3—C4—H4	123.2	C11—C15—H15	121.9
C4—C5—C11	127.5 (4)	C14—C15—H15	121.9
C4—C5—S1	110.9 (3)	C14—S15—C11	92.0 (3)
C11—C5—S1	121.3 (4)	C17—C16—C3	128.4 (5)
C10—C6—C2	128.7 (4)	C17—C16—H16	115.8
C10—C6—S7	110.2 (3)	C3—C16—H16	115.8
C2—C6—S7	121.0 (3)	C16—C17—C18	120.9 (4)
C8—S7—C6	92.2 (2)	C16—C17—C20	124.3 (4)
C9—C8—S7	111.6 (4)	C18—C17—C20	114.8 (4)
C9—C8—H8	124.2	N19—C18—C17	178.7 (5)
S7—C8—H8	124.2	C25—C20—C21	118.6 (5)
C8—C9—C10	114.1 (5)	C25—C20—C17	120.3 (4)
C8—C9—H9	123.0	C21—C20—C17	121.1 (4)
C10—C9—H9	123.0	C22—C21—C20	120.6 (5)

supplementary materials

C6—C10—C9	111.8 (4)	C22—C21—H21	119.7
C6—C10—H10	124.1	C20—C21—H21	119.7
C9—C10—H10	124.1	C23—C22—C21	120.2 (5)
C15—C11—C5	130.7 (5)	C23—C22—H22	119.9
C5—C11—C12	132.0 (7)	C21—C22—H22	119.9
C5—C11—S15	119.7 (4)	C24—C23—C22	119.7 (5)
C12—C11—S15	108.1 (7)	C24—C23—H23	120.1
C15—C11—S12	107.4 (5)	C22—C23—H23	120.2
C5—C11—S12	121.8 (3)	C23—C24—C25	120.3 (5)
C13—S12—C11	92.7 (3)	C23—C24—H24	119.9
C13—C12—C11	111 (2)	C25—C24—H24	119.9
C13—C12—H12	130.0	C20—C25—C24	120.6 (5)
C11—C12—H12	119.0	C20—C25—H25	119.7
C14—C13—C12	109.3 (17)	C24—C25—H25	119.7
C14—C13—S12	117.4 (4)		
C5—S1—C2—C3	0.3 (4)	C5—C11—S12—C13	175.6 (4)
C5—S1—C2—C6	-179.7 (4)	C5—C11—C12—C13	174.9 (13)
C6—C2—C3—C4	179.5 (4)	S15—C11—C12—C13	0(3)
S1—C2—C3—C4	-0.5 (5)	C11—C12—C13—C14	0(3)
C6—C2—C3—C16	3.2 (8)	C11—S12—C13—C14	0.2 (5)
S1—C2—C3—C16	-176.8 (4)	S12—C13—C14—C15	1.7 (8)
C2—C3—C4—C5	0.5 (6)	C12—C13—C14—S15	0(2)
C16—C3—C4—C5	176.6 (4)	C5—C11—C15—C14	-173.9 (6)
C3—C4—C5—C11	-174.8 (4)	S15—C11—C15—C14	-168 (5)
C3—C4—C5—S1	-0.3 (5)	S12—C11—C15—C14	3.5 (11)
C2—S1—C5—C4	0.0 (4)	C13—C14—C15—C11	-3.4 (12)
C2—S1—C5—C11	174.9 (4)	C13—C14—S15—C11	-0.1 (7)
C3—C2—C6—C10	43.0 (8)	C15—C14—S15—C11	-7(3)
S1—C2—C6—C10	-137.0 (4)	C5—C11—S15—C14	-175.7 (4)
C3—C2—C6—S7	-138.9 (4)	C12—C11—S15—C14	0(2)
S1—C2—C6—S7	41.1 (5)	C2—C3—C16—C17	-158.0 (5)
C10—C6—S7—C8	0.0 (4)	C4—C3—C16—C17	26.3 (8)
C2—C6—S7—C8	-178.5 (4)	C3—C16—C17—C18	3.7 (8)
C6—S7—C8—C9	-0.8 (4)	C3—C16—C17—C20	-175.3 (4)
S7—C8—C9—C10	1.4 (6)	C16—C17—C20—C25	22.4 (7)
C2—C6—C10—C9	179.1 (5)	C18—C17—C20—C25	-156.6 (4)
S7—C6—C10—C9	0.8 (5)	C16—C17—C20—C21	-157.5 (5)
C8—C9—C10—C6	-1.5 (6)	C18—C17—C20—C21	23.4 (6)
C4—C5—C11—C15	-148.9 (9)	C25—C20—C21—C22	2.3 (7)
S1—C5—C11—C15	37.0 (10)	C17—C20—C21—C22	-177.7 (4)
C4—C5—C11—C12	35 (3)	C20—C21—C22—C23	-1.3 (7)
S1—C5—C11—C12	-139 (2)	C21—C22—C23—C24	0.0 (7)
C4—C5—C11—S15	-150.2 (6)	C22—C23—C24—C25	0.1 (7)
S1—C5—C11—S15	35.7 (6)	C21—C20—C25—C24	-2.2 (7)
C4—C5—C11—S12	34.0 (7)	C17—C20—C25—C24	177.9 (4)
S1—C5—C11—S12	-140.1 (4)	C23—C24—C25—C20	1.0 (7)
C15—C11—S12—C13	-2.1 (7)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C13—H13···N19 ⁱ	0.93	2.54	3.435 (7)	161

Symmetry codes: (i) $-x+1, -y, -z-1$.

Fig. 1

