

# Crystal structure of (Z)-1-phenyl-3-styrylundeca-2-en-4,10-diyne-1-ol

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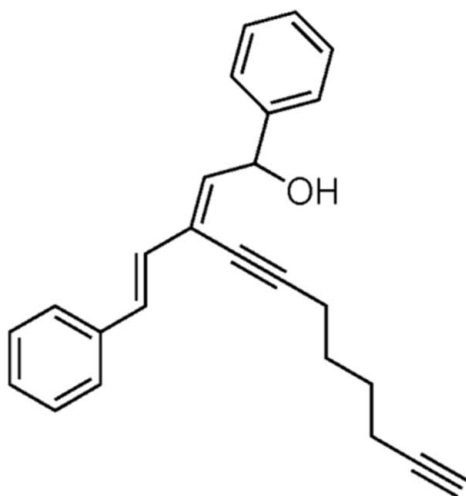
The molecule of the title compound, C<sub>25</sub>H<sub>24</sub>O, obtained by acid-catalysed 1,3-migration of an alcohol group, is T-shaped. The planes of the two phenyl rings are inclined to one another by 81.9 (2)°. In the crystal, molecules are linked by O—H···O hydrogen bonds, forming chains along [001].

**Keywords:** crystal structure; 1,3-migration; alcohol group; catalytic cyclization; styrylundecane.

**CCDC reference:** 1009363

## 1. Related literature

For the 1,3-migration of an alcoholic group adjacent to a vinyl group in the presence of a Lewis acid, see: Piotti & Alper (1997); Poloukhine & Popik (2005). For catalytic cyclization of alcohols containing a number of unsaturated groups, see: Teo *et al.* (2014).



## 2. Experimental

### 2.1. Crystal data

C <sub>25</sub> H <sub>24</sub> O	Z = 3
M <sub>r</sub> = 340.44	Mo Kα radiation
Trigonal, P3 <sub>2</sub>	μ = 0.07 mm <sup>-1</sup>
a = 17.867 (2) Å	T = 103 K
c = 5.3290 (6) Å	0.34 × 0.04 × 0.04 mm
V = 1473.3 (4) Å <sup>3</sup>	

### 2.2. Data collection

Bruker Kappa APEXII CCD diffractometer	14182 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2013)	4846 independent reflections
T <sub>min</sub> = 0.74, T <sub>max</sub> = 1.00	2945 reflections with I > 2σ(I)
	R <sub>int</sub> = 0.078

### 2.3. Refinement

R[F <sup>2</sup> > 2σ(F <sup>2</sup> )] = 0.058	1 restraint
wR(F <sup>2</sup> ) = 0.129	H-atom parameters constrained
S = 0.98	Δρ <sub>max</sub> = 0.39 e Å <sup>-3</sup>
4846 reflections	Δρ <sub>min</sub> = -0.24 e Å <sup>-3</sup>
235 parameters	

**Table 1**

Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O1 <sup>i</sup>	0.84	1.83	2.652 (3)	166

Symmetry code: (i) -y + 1, x - y + 1, z - 1/3.

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINTE* (Bruker, 2013); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014*, *PLATON* (Spek, 2009) and *pubCIF* (Westrip, 2010).

Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5038).

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## supporting information

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## Crystal structure of (Z)-1-phenyl-3-styrylundeca-2-en-4,10-diyn-1-ol

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### S1. Comment

1,3-migration of an alcoholic group adjacent to a vinyl group in the presence of a Lewis-acid is widely known (Piotti & Alper, 1997). One such example was demonstrated recently in the preparation of 4-( $\alpha$ -hydroxybenzyl)-1-*tert*-butyl-dimethylsilyloxy-4-cyclodecene-2,6-diyne (Poloukhtine & Popik, 2005). In addition, alcohols containing many unsaturated groups provide an access to a myriad of types of functionalization such as catalytic cyclization (Teo *et al.*, 2014). Herein, we report on the synthesis and crystal structure of the title compound, obtained by the acid-catalyzed 1,3-migration of an alcoholic group.

The molecular structure of the title compound is illustrated in Fig. 1. The molecule is T-shaped with the two phenyl ring inclined to one another by 81.9 (2) °.

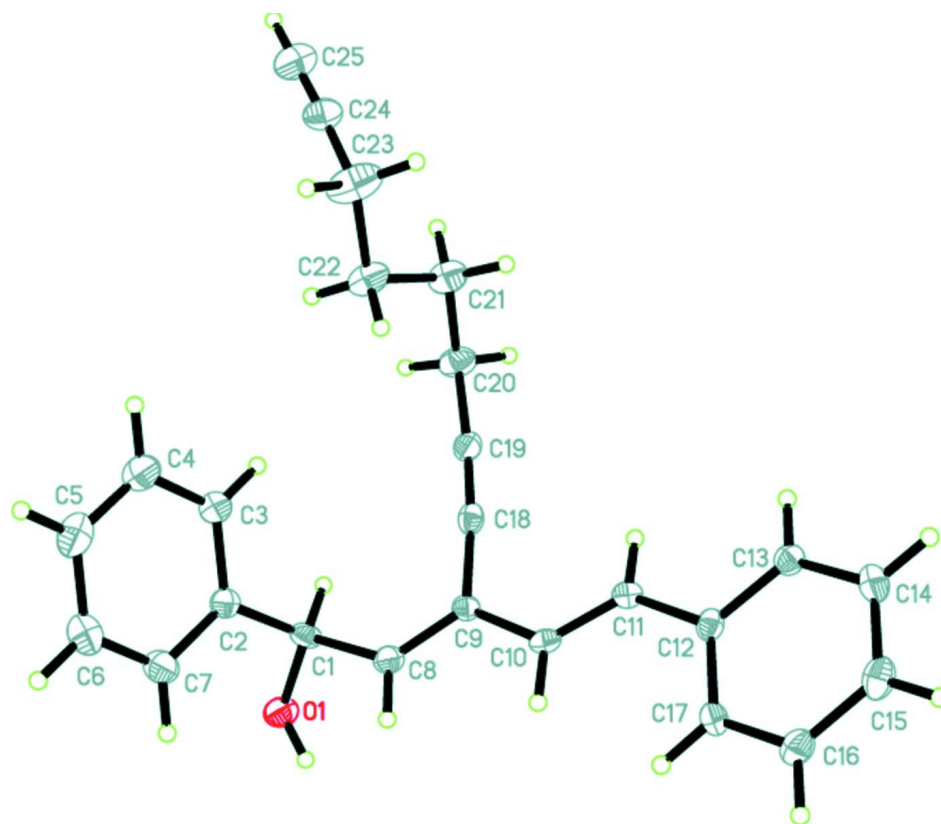
In the crystal, molecules are linked by O—H $\cdots$ O hydrogen bonds forming chains along the *c* axis direction (Table 1 and Fig. 2).

### S2. Synthesis and crystallization

The synthesis of the title compound is illustrated in Fig. 3. (Z)-1-phenyl-3-styrylundeca-1-en-4,10-diyn-3-ol (1 mmol, 340.5 mg) was dissolved in 10 ml of CH<sub>2</sub>Cl<sub>2</sub>. DMAP [4-(dimethylamino)pyridine; 0.1 mmol, 12 mg], triethylamine (5 mmol, 0.70 ml) and acetic anhydride (5 mmol, 0.47 ml) were added sequentially and the reaction mixture was stirred overnight. It was then washed with saturated sodium bicarbonate and extracted twice with CH<sub>2</sub>Cl<sub>2</sub>. The organic layers were combined, dried with MgSO<sub>4</sub> and the solvent was removed under reduced pressure. The resulting oil was purified by column chromatography with hexane/ethylacetate as eluent. The product was recrystallized with ethylacetate to give a colourless compound in 80% yield. Slow evaporation of a solution in ethylacetate gave needle-like crystals. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) 1.71–1.83 (m, 4H), 1.97 (t, 1H), 2.20 (s, 1H), 2.26–2.30 (m, 2H), 2.55 (t, 2), 5.90 (d, 1H), 6.09 (d, 1H), 6.68 (d, 1H), 6.99 (d, 1H), 7.21–7.47 (m, 10H) <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) 18.0, 19.2, 27.7, 27.7, 68.8, 72.5, 75.3, 84.0, 97.9, 124.1, 125.9, 126.8, 127.7, 127.9, 128.6, 128.6, 132.4, 136.8, 140.4, 142.7

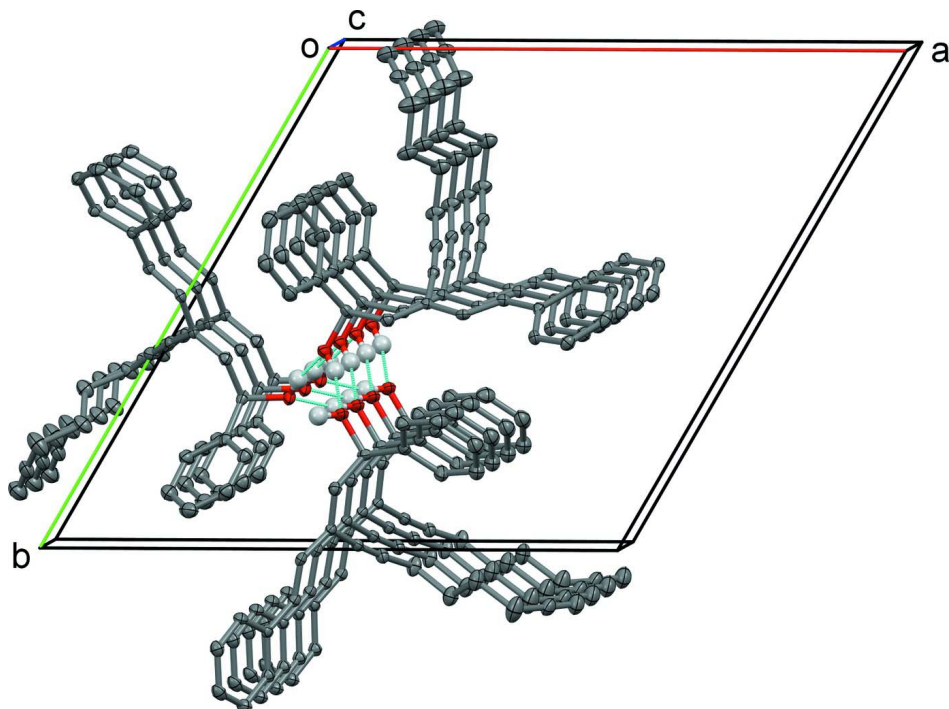
### S3. Refinement

The OH and C-bound H atoms were included in calculated positions and treated as riding atoms: O—H = 0.84 Å, C—H = 0.95 - 1.00 Å with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$  for the OH H atom and =  $1.2U_{\text{eq}}(\text{C})$  for other H atoms.

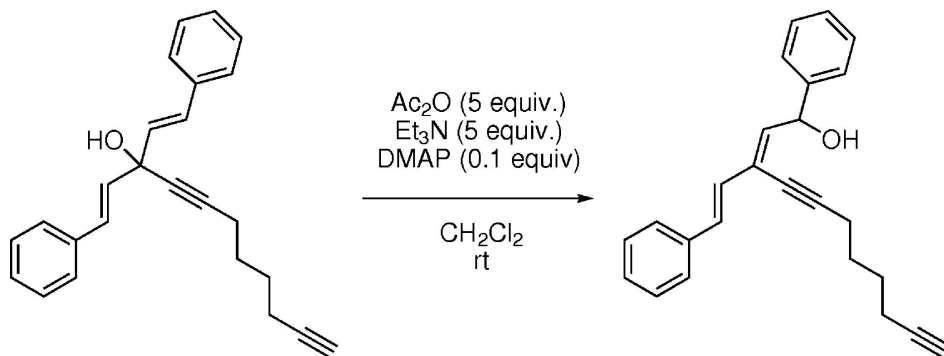


**Figure 1**

A view of the molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

A partial view along the *c* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1 for details).

**Figure 3**

Reaction scheme.

### (*Z*)-1-Phenyl-3-styrylundeca-2-en-4,10-diyn-1-ol

#### Crystal data

$C_{25}H_{24}O$

$M_r = 340.44$

Trigonal,  $P3_2$

$a = 17.867(2) \text{ \AA}$

$c = 5.3290(6) \text{ \AA}$

$V = 1473.3(4) \text{ \AA}^3$

$Z = 3$

$F(000) = 546$

$D_x = 1.151 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1285 reflections

$\theta = 2.3\text{--}20.5^\circ$

$\mu = 0.07 \text{ mm}^{-1}$

$T = 103 \text{ K}$

Needle, colourless

$0.34 \times 0.04 \times 0.04 \text{ mm}$

*Data collection*

Bruker Kappa APEXII CCD  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
 $\omega$  and  $\varphi$  scan  
Absorption correction: multi-scan  
(*SADABS*; Bruker, 2013)  
 $T_{\min} = 0.74$ ,  $T_{\max} = 1.00$

14182 measured reflections  
4846 independent reflections  
2945 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.078$   
 $\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.6^\circ$   
 $h = -23 \rightarrow 14$   
 $k = -22 \rightarrow 23$   
 $l = -7 \rightarrow 7$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.058$   
 $wR(F^2) = 0.129$   
 $S = 0.98$   
4846 reflections  
235 parameters  
1 restraint  
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  
 $w = 1/[\sigma^2(F_o^2) + (0.0486P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.30859 (17)	0.59437 (17)	0.3483 (5)	0.0251 (7)
H1A	0.3463	0.6311	0.2521	0.038*
C1	0.3069 (3)	0.5136 (2)	0.3226 (7)	0.0199 (9)
H1	0.3048	0.4898	0.4940	0.024*
C2	0.2281 (2)	0.4484 (3)	0.1774 (7)	0.0197 (9)
C3	0.1897 (3)	0.3614 (3)	0.2407 (8)	0.0281 (10)
H3	0.2105	0.3441	0.3801	0.034*
C4	0.1209 (3)	0.2999 (3)	0.1007 (9)	0.0341 (12)
H4	0.0948	0.2407	0.1461	0.041*
C5	0.0899 (3)	0.3235 (3)	-0.1032 (9)	0.0333 (11)
H5	0.0434	0.2809	-0.1998	0.040*
C6	0.1275 (3)	0.4099 (3)	-0.1651 (8)	0.0305 (11)
H6	0.1060	0.4269	-0.3038	0.037*
C7	0.1962 (3)	0.4721 (3)	-0.0270 (8)	0.0272 (10)
H7	0.2216	0.5313	-0.0722	0.033*
C8	0.3878 (2)	0.5282 (2)	0.1948 (7)	0.0196 (9)
H8	0.4050	0.5639	0.0497	0.024*
C9	0.4386 (2)	0.4964 (2)	0.2634 (7)	0.0160 (8)
C10	0.5143 (2)	0.5153 (2)	0.1121 (7)	0.0171 (8)
H10	0.5253	0.5510	-0.0311	0.021*

C11	0.5691 (2)	0.4869 (2)	0.1575 (7)	0.0171 (8)
H11	0.5608	0.4552	0.3082	0.021*
C12	0.6413 (2)	0.5000 (2)	-0.0039 (7)	0.0173 (9)
C13	0.6850 (2)	0.4550 (2)	0.0458 (7)	0.0184 (9)
H13	0.6692	0.4182	0.1882	0.022*
C14	0.7511 (3)	0.4630 (3)	-0.1091 (8)	0.0220 (9)
H14	0.7805	0.4324	-0.0718	0.026*
C15	0.7739 (3)	0.5160 (3)	-0.3182 (7)	0.0245 (10)
H15	0.8184	0.5209	-0.4264	0.029*
C16	0.7321 (2)	0.5620 (3)	-0.3704 (7)	0.0231 (9)
H16	0.7482	0.5985	-0.5134	0.028*
C17	0.6665 (3)	0.5545 (3)	-0.2137 (7)	0.0204 (9)
H17	0.6385	0.5867	-0.2492	0.024*
C18	0.4152 (2)	0.4385 (3)	0.4758 (8)	0.0184 (8)
C19	0.3892 (2)	0.3880 (2)	0.6457 (7)	0.0201 (9)
C20	0.3519 (3)	0.3267 (3)	0.8553 (8)	0.0275 (10)
H20A	0.3052	0.3336	0.9313	0.033*
H20B	0.3973	0.3426	0.9844	0.033*
C21	0.3157 (3)	0.2328 (3)	0.7913 (8)	0.0310 (11)
H21A	0.2888	0.1977	0.9434	0.037*
H21B	0.3637	0.2235	0.7405	0.037*
C22	0.2484 (3)	0.2009 (3)	0.5807 (8)	0.0317 (11)
H22A	0.2021	0.2137	0.6248	0.038*
H22B	0.2760	0.2322	0.4237	0.038*
C23	0.2091 (3)	0.1044 (3)	0.5383 (9)	0.0467 (14)
H23A	0.1740	0.0881	0.3830	0.056*
H23B	0.2562	0.0913	0.5127	0.056*
C24	0.1549 (3)	0.0521 (3)	0.7450 (9)	0.0326 (11)
C25	0.1097 (3)	0.0105 (3)	0.9069 (10)	0.0431 (13)
H25	0.0727	-0.0235	1.0394	0.052*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0252 (16)	0.0188 (15)	0.0341 (17)	0.0130 (13)	0.0087 (14)	0.0019 (13)
C1	0.024 (2)	0.021 (2)	0.021 (2)	0.0159 (19)	0.0076 (18)	0.0067 (18)
C2	0.019 (2)	0.021 (2)	0.024 (2)	0.0129 (18)	0.0084 (18)	0.0036 (18)
C3	0.022 (2)	0.025 (2)	0.036 (3)	0.010 (2)	0.002 (2)	0.008 (2)
C4	0.026 (2)	0.026 (3)	0.047 (3)	0.010 (2)	0.003 (2)	0.005 (2)
C5	0.024 (2)	0.034 (3)	0.038 (3)	0.011 (2)	-0.001 (2)	-0.006 (2)
C6	0.031 (3)	0.035 (3)	0.030 (3)	0.020 (2)	-0.005 (2)	-0.002 (2)
C7	0.028 (2)	0.027 (2)	0.029 (2)	0.016 (2)	0.003 (2)	0.005 (2)
C8	0.020 (2)	0.018 (2)	0.021 (2)	0.0093 (18)	0.0026 (17)	0.0022 (17)
C9	0.016 (2)	0.0124 (19)	0.019 (2)	0.0071 (17)	-0.0012 (17)	-0.0009 (16)
C10	0.018 (2)	0.0110 (19)	0.019 (2)	0.0051 (17)	0.0007 (17)	0.0011 (16)
C11	0.019 (2)	0.015 (2)	0.017 (2)	0.0078 (18)	0.0010 (17)	0.0004 (16)
C12	0.014 (2)	0.017 (2)	0.020 (2)	0.0066 (17)	-0.0008 (17)	-0.0034 (17)
C13	0.018 (2)	0.019 (2)	0.018 (2)	0.0087 (18)	0.0014 (17)	0.0001 (17)

C14	0.021 (2)	0.025 (2)	0.024 (2)	0.0145 (19)	-0.0020 (18)	-0.0037 (19)
C15	0.019 (2)	0.031 (2)	0.023 (2)	0.012 (2)	0.0035 (18)	-0.0035 (19)
C16	0.021 (2)	0.025 (2)	0.022 (2)	0.0096 (19)	0.0035 (18)	0.0020 (19)
C17	0.017 (2)	0.021 (2)	0.025 (2)	0.0119 (18)	-0.0006 (18)	0.0014 (18)
C18	0.015 (2)	0.020 (2)	0.021 (2)	0.0094 (17)	-0.0002 (17)	-0.0042 (18)
C19	0.017 (2)	0.020 (2)	0.021 (2)	0.0073 (18)	0.0002 (18)	0.0011 (18)
C20	0.031 (3)	0.024 (2)	0.020 (2)	0.008 (2)	-0.001 (2)	0.0045 (19)
C21	0.033 (3)	0.026 (2)	0.027 (2)	0.010 (2)	0.002 (2)	0.002 (2)
C22	0.032 (3)	0.024 (2)	0.029 (3)	0.006 (2)	0.003 (2)	0.003 (2)
C23	0.055 (3)	0.028 (3)	0.039 (3)	0.007 (2)	0.006 (3)	-0.002 (2)
C24	0.033 (3)	0.019 (2)	0.043 (3)	0.010 (2)	-0.004 (2)	-0.003 (2)
C25	0.037 (3)	0.029 (3)	0.054 (4)	0.009 (2)	0.001 (3)	0.006 (3)

*Geometric parameters (Å, °)*

O1—C1	1.435 (4)	C13—C14	1.388 (5)
O1—H1A	0.8400	C13—H13	0.9500
C1—C8	1.498 (5)	C14—C15	1.385 (6)
C1—C2	1.516 (6)	C14—H14	0.9500
C1—H1	1.0000	C15—C16	1.388 (6)
C2—C3	1.390 (5)	C15—H15	0.9500
C2—C7	1.390 (5)	C16—C17	1.390 (5)
C3—C4	1.387 (6)	C16—H16	0.9500
C3—H3	0.9500	C17—H17	0.9500
C4—C5	1.378 (6)	C18—C19	1.197 (5)
C4—H4	0.9500	C19—C20	1.470 (5)
C5—C6	1.381 (6)	C20—C21	1.504 (6)
C5—H5	0.9500	C20—H20A	0.9900
C6—C7	1.385 (6)	C20—H20B	0.9900
C6—H6	0.9500	C21—C22	1.531 (6)
C7—H7	0.9500	C21—H21A	0.9900
C8—C9	1.341 (5)	C21—H21B	0.9900
C8—H8	0.9500	C22—C23	1.519 (6)
C9—C18	1.446 (5)	C22—H22A	0.9900
C9—C10	1.461 (5)	C22—H22B	0.9900
C10—C11	1.332 (5)	C23—C24	1.456 (7)
C10—H10	0.9500	C23—H23A	0.9900
C11—C12	1.467 (5)	C23—H23B	0.9900
C11—H11	0.9500	C24—C25	1.161 (6)
C12—C13	1.399 (5)	C25—H25	0.9500
C12—C17	1.401 (5)		
C1—O1—H1A	109.5	C12—C13—H13	119.4
O1—C1—C8	109.5 (3)	C15—C14—C13	119.5 (4)
O1—C1—C2	111.5 (3)	C15—C14—H14	120.2
C8—C1—C2	110.3 (3)	C13—C14—H14	120.2
O1—C1—H1	108.5	C14—C15—C16	120.3 (4)
C8—C1—H1	108.5	C14—C15—H15	119.9

C2—C1—H1	108.5	C16—C15—H15	119.9
C3—C2—C7	118.8 (4)	C15—C16—C17	120.1 (4)
C3—C2—C1	119.0 (4)	C15—C16—H16	120.0
C7—C2—C1	122.0 (4)	C17—C16—H16	120.0
C2—C3—C4	120.2 (4)	C16—C17—C12	120.6 (4)
C2—C3—H3	119.9	C16—C17—H17	119.7
C4—C3—H3	119.9	C12—C17—H17	119.7
C5—C4—C3	120.9 (4)	C19—C18—C9	174.8 (4)
C5—C4—H4	119.5	C18—C19—C20	176.0 (4)
C3—C4—H4	119.5	C19—C20—C21	116.2 (4)
C4—C5—C6	119.0 (4)	C19—C20—H20A	108.2
C4—C5—H5	120.5	C21—C20—H20A	108.2
C6—C5—H5	120.5	C19—C20—H20B	108.2
C5—C6—C7	120.7 (4)	C21—C20—H20B	108.2
C5—C6—H6	119.6	H20A—C20—H20B	107.4
C7—C6—H6	119.6	C20—C21—C22	113.6 (4)
C6—C7—C2	120.4 (4)	C20—C21—H21A	108.8
C6—C7—H7	119.8	C22—C21—H21A	108.8
C2—C7—H7	119.8	C20—C21—H21B	108.8
C9—C8—C1	126.9 (3)	C22—C21—H21B	108.8
C9—C8—H8	116.5	H21A—C21—H21B	107.7
C1—C8—H8	116.5	C23—C22—C21	111.3 (4)
C8—C9—C18	120.1 (3)	C23—C22—H22A	109.4
C8—C9—C10	119.7 (3)	C21—C22—H22A	109.4
C18—C9—C10	120.0 (3)	C23—C22—H22B	109.4
C11—C10—C9	125.7 (4)	C21—C22—H22B	109.4
C11—C10—H10	117.1	H22A—C22—H22B	108.0
C9—C10—H10	117.1	C24—C23—C22	113.4 (4)
C10—C11—C12	125.9 (4)	C24—C23—H23A	108.9
C10—C11—H11	117.0	C22—C23—H23A	108.9
C12—C11—H11	117.0	C24—C23—H23B	108.9
C13—C12—C17	118.2 (3)	C22—C23—H23B	108.9
C13—C12—C11	119.7 (4)	H23A—C23—H23B	107.7
C17—C12—C11	122.1 (3)	C25—C24—C23	178.0 (5)
C14—C13—C12	121.3 (4)	C24—C25—H25	180.0
C14—C13—H13	119.4		
O1—C1—C2—C3	-145.9 (3)	C8—C9—C10—C11	-179.1 (4)
C8—C1—C2—C3	92.2 (4)	C18—C9—C10—C11	-3.5 (6)
O1—C1—C2—C7	38.1 (5)	C9—C10—C11—C12	175.0 (3)
C8—C1—C2—C7	-83.8 (4)	C10—C11—C12—C13	-169.7 (4)
C7—C2—C3—C4	0.2 (6)	C10—C11—C12—C17	8.1 (6)
C1—C2—C3—C4	-175.9 (4)	C17—C12—C13—C14	-0.6 (5)
C2—C3—C4—C5	0.4 (6)	C11—C12—C13—C14	177.2 (3)
C3—C4—C5—C6	-1.0 (7)	C12—C13—C14—C15	-0.6 (6)
C4—C5—C6—C7	1.0 (7)	C13—C14—C15—C16	1.1 (6)
C5—C6—C7—C2	-0.3 (7)	C14—C15—C16—C17	-0.5 (6)
C3—C2—C7—C6	-0.2 (6)	C15—C16—C17—C12	-0.8 (6)



C1—C2—C7—C6	175.8 (4)	C13—C12—C17—C16	1.3 (5)
O1—C1—C8—C9	133.8 (4)	C11—C12—C17—C16	-176.5 (4)
C2—C1—C8—C9	-103.1 (4)	C19—C20—C21—C22	-54.5 (5)
C1—C8—C9—C18	2.6 (6)	C20—C21—C22—C23	-175.6 (4)
C1—C8—C9—C10	178.2 (3)	C21—C22—C23—C24	68.5 (6)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1—H1A $\cdots$ O1 <sup>i</sup>	0.84	1.83	2.652 (3)	166

Symmetry code: (i)  $-y+1, x-y+1, z-1/3$ .