

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-[4-(2,6-Dimethoxyphenyl)butyl]-1,3-dimethoxybenzene

Christopher M. Kane, Stephen D. Drake and K. Travis Holman*

Department of Chemistry, Georgetown University, 37th and O St NW, Washington, DC 20057, USA

Correspondence e-mail: kth7@georgetown.edu

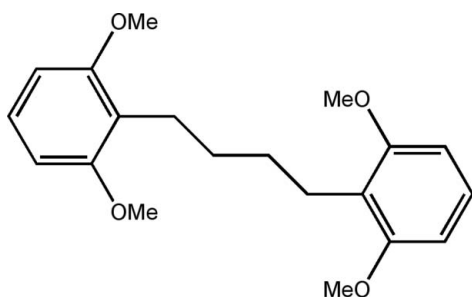
Received 3 June 2010; accepted 28 June 2010

 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.035; wR factor = 0.098; data-to-parameter ratio = 12.9.

The title compound, $\text{C}_{20}\text{H}_{26}\text{O}_4$, crystallizes such that the alkyl chain adopts an all-*anti* conformation. The crystal packing displays edge-to-face arene–arene interactions with a dihedral angle of 87° . The complete molecule is generated by inversion symmetry.

Related literature

For related compounds containing tethered 2,6-dimethoxybenzene fragments, see: Ionkin *et al.* (2003); Evans *et al.* (1991); Yoshimura *et al.* (2008); Shinohara *et al.* (2008); Ono *et al.* (2008). For a related structure, see: Fleck *et al.* (2005). For the synthesis and further studies, see: Lettré *et al.* (1952); Tanaka *et al.* (1989). The rather large crystal used for data collection was chosen in order to optimize data intensity. For weakly absorbing materials, *SADABS* is known to be effective at correcting for crystal sizes larger than the beam without introducing systematic errors, see, for example: Görbitz (1999).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{26}\text{O}_4$	$V = 1725.4 (3) \text{ \AA}^3$
$M_r = 330.41$	$Z = 4$
Orthorhombic, <i>Pbcn</i>	Mo $K\alpha$ radiation
$a = 22.692 (2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 5.5460 (5) \text{ \AA}$	$T = 100 \text{ K}$
$c = 13.7099 (13) \text{ \AA}$	$0.98 \times 0.36 \times 0.22 \text{ mm}$

Data collection

Bruker SMART APEXII CCD diffractometer	14196 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2001)	2071 independent reflections
$T_{\min} = 0.916$, $T_{\max} = 0.981$	1853 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$	161 parameters
$wR(F^2) = 0.098$	All H-atom parameters refined
$S = 1.05$	$\Delta\rho_{\max} = 0.29 \text{ e \AA}^{-3}$
2071 reflections	$\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *X-SEED* and *POV-RAY* (Persistence of Vision, 2004).

The authors acknowledge grant support from the National Science Foundation (DMR-0349316).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2784).

References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
 Bruker (2001). *SADABS*, *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Evans, K. L., Fronczek, F. R. & Gandour, R. D. (1991). *Acta Cryst.* **C47**, 2729–2731.
 Fleck, M. & Walter, M. (2005). *Acta Cryst.* **E61**, o4099–o4100.
 Görbitz, C. H. (1999). *Acta Cryst.* **B55**, 1090–1098.
 Ionkin, A. S. & Marshall, W. J. (2003). *Heteroat. Chem.* **14**, 360–364.
 Lettré, H. & Jahn, A. (1952). *Chem. Ber.* **85**, 346–350.
 Ono, K., Tsukamoto, K., Tomura, M. & Saito, K. (2008). *Acta Cryst.* **E64**, o1069.
 Persistence of Vision (2004). Persistence of Vision (TM) Raytracer (*POV-RAY*). Persistence of Vision Pty Ltd, Williamstown, Victoria, Australia. <http://www.povray.org/>.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Shinohara, Y. & Arai, T. (2008). *Bull. Chem. Soc. Jpn.* **81**, 1500–1504.
 Tanaka, Y., Ubukata, Y. & Aoyama, Y. (1989). *Chem. Lett.* pp. 1905–1908.
 Yoshimura, N., Momotake, A., Shinohara, Y., Nishimura, Y. & Arai, T. (2008). *Chem. Lett.* **37**, 174–175.

supporting information

Acta Cryst. (2010). E66, o1921 [https://doi.org/10.1107/S1600536810025420]

2-[4-(2,6-Dimethoxyphenyl)butyl]-1,3-dimethoxybenzene**Christopher M. Kane, Stephen D. Drake and K. Travis Holman****S1. Comment**

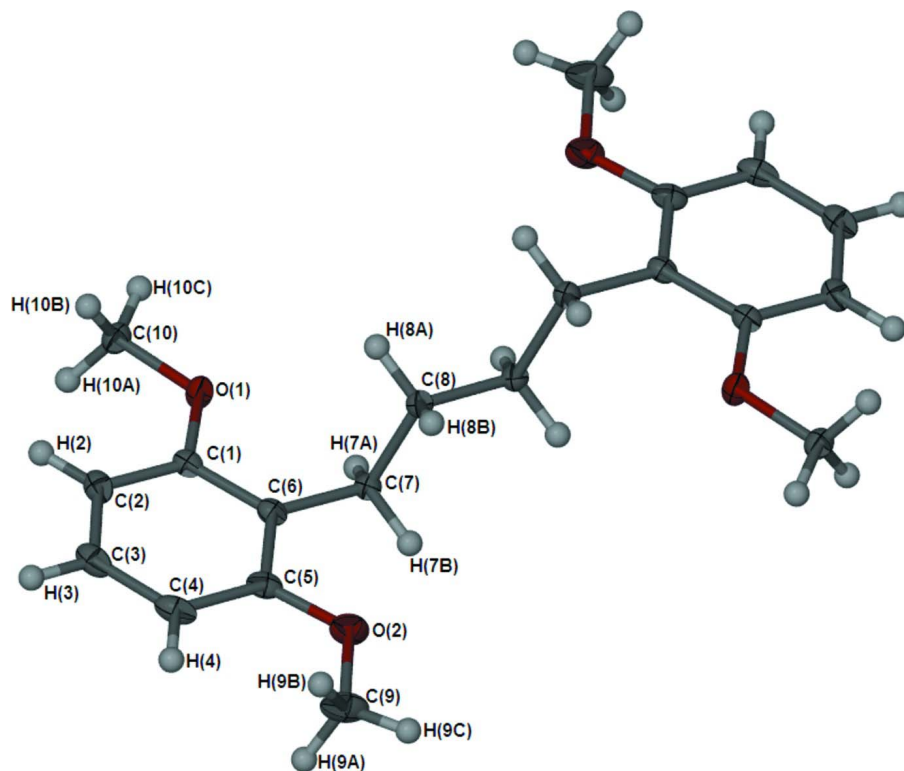
Several tethered 2,6-dimethoxyphenyl derivatives have been synthesized containing conjugated linkers comprised of alkenyl and alkynyl units (Yoshimura *et al.*, 2008, Shinohara *et al.*, 2008, and Ono *et al.*, 2008).

The conformation of the title compound is similar to the hydrocarbon 1,4-diphenylbutane (Fleck *et al.*, 2005). Both molecules exhibit an all *anti* aliphatic conformation. The title compound maintains aromatic C—C bond distances in the range of 1.3844 (16)–1.4042 (13) Å, and aliphatic C—C bonds from 1.5096 (12)–1.5349 (12) Å. One striking difference between the compounds is the crystal packing, which adopts a herringbone pattern for the title compound, whereas in 1,4-diphenylbutane, neither edge-to-face nor π - π stacking interactions are observed.

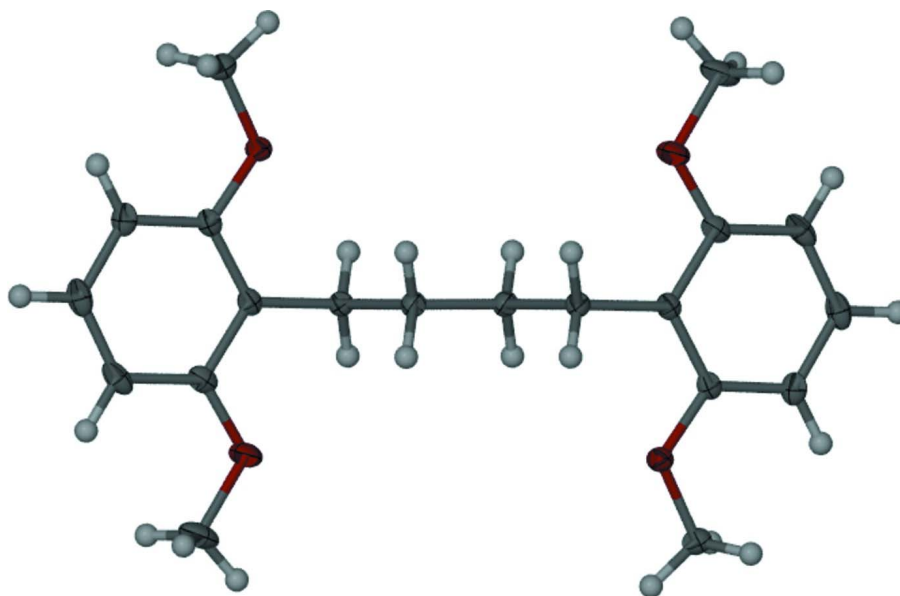
The rather large crystal (~1 mm) used for data collection was chosen in order to optimize data intensity. For weakly absorbing materials, *SADABS* is known to be effective at correcting for crystal sizes larger than the beam, without introducing systematic errors. See, for example: Görbitz (1999).

S2. Experimental

The title compound was obtained by lithiation (10 ml, 2.5 M *n*-BuLi in hexanes) of 1,3-dimethoxybenzene (3.45 g, 25 mmol) under nitrogen atmosphere. Following distillation of hexanes and subsequent addition of 1,4-dibromohexane (2.16 g, 10 mmol), the mixture was heated to 150°C for 2 days. After cooling, the mixture was quenched with water (150 ml) and the product was removed and was recrystallized with a 3:1 hexanes/ethyl acetate solution to afford an off-white compound in 72% yield. Single crystals were obtained by slow evaporation from ethanol.

**Figure 1**

Title compound with atom labels and 50% probability displacement ellipsoids for non-H atoms.

**Figure 2**

View of title compound along C4 carbon chain.

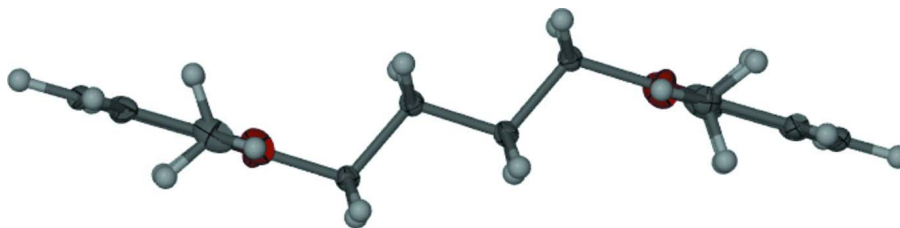


Figure 3
Side view of title compound.

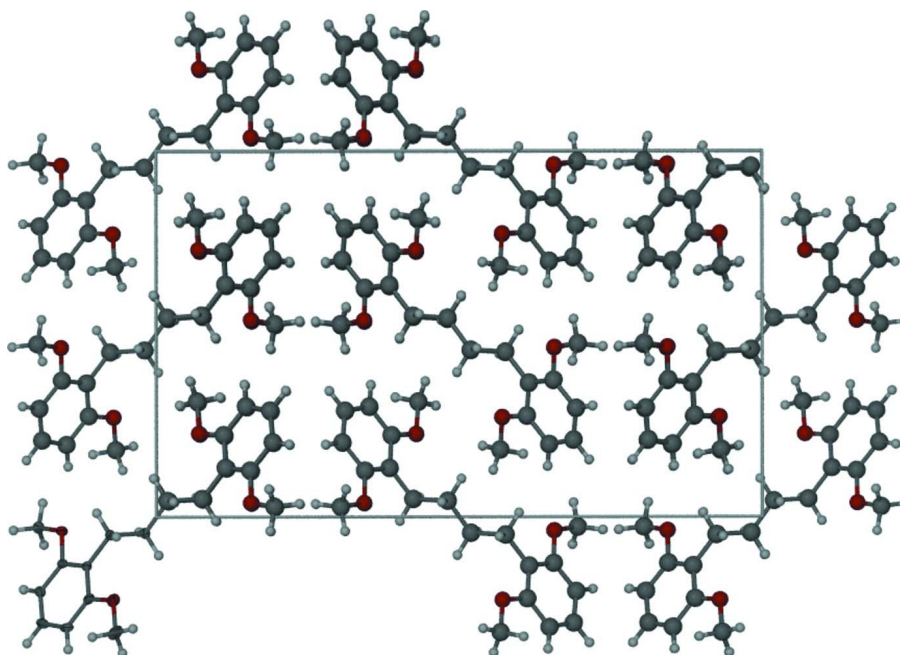


Figure 4
Packing of title compound as viewed down the *b* axis.

2-[4-(2,6-Dimethoxyphenyl)butyl]-1,3-dimethoxybenzene

Crystal data

$C_{20}H_{26}O_4$

$M_r = 330.41$

Orthorhombic, *Pbcn*

Hall symbol: -P 2yn

$a = 22.692$ (2) Å

$b = 5.5460$ (5) Å

$c = 13.7099$ (13) Å

$V = 1725.4$ (3) Å³

$Z = 4$

$F(000) = 712$

$D_x = 1.272$ Mg m⁻³

Melting point = 429–431 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 6702 reflections

$\theta = 3.0$ – 28.5°

$\mu = 0.09$ mm⁻¹

$T = 100$ K

Prism, colorless

$0.98 \times 0.36 \times 0.22$ mm

Data collection

Bruker SMART APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2001)

$T_{\min} = 0.916$, $T_{\max} = 0.981$

14196 measured reflections
 2071 independent reflections
 1853 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 28.0^\circ$, $\theta_{\text{min}} = 1.8^\circ$
 $h = -29 \rightarrow 29$
 $k = -7 \rightarrow 7$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.098$
 $S = 1.05$
 2071 reflections
 161 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 0.4726P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.29 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$

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.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.15557 (4)	0.35467 (17)	0.12621 (6)	0.0182 (2)
C2	0.19265 (4)	0.29025 (19)	0.20325 (7)	0.0225 (2)
C3	0.18839 (5)	0.4180 (2)	0.28983 (7)	0.0260 (2)
C4	0.14842 (5)	0.6043 (2)	0.30147 (7)	0.0257 (2)
C5	0.11173 (4)	0.66605 (18)	0.22332 (7)	0.0216 (2)
C6	0.11502 (4)	0.54382 (17)	0.13379 (6)	0.0182 (2)
C7	0.07619 (4)	0.61358 (17)	0.04894 (7)	0.0185 (2)
C8	0.01905 (4)	0.46605 (17)	0.04386 (7)	0.0190 (2)
C9	0.06764 (6)	0.9829 (2)	0.31571 (9)	0.0354 (3)
C10	0.19556 (4)	0.04010 (18)	0.02716 (8)	0.0229 (2)
H2	0.2201 (6)	0.161 (2)	0.1972 (9)	0.028 (3)*
H3	0.2143 (5)	0.375 (2)	0.3442 (10)	0.030 (3)*
H4	0.1459 (6)	0.689 (3)	0.3613 (10)	0.032 (3)*
H7A	0.0984 (5)	0.588 (2)	-0.0112 (9)	0.021 (3)*
H7B	0.0660 (5)	0.785 (2)	0.0535 (8)	0.021 (3)*
H8A	0.0292 (5)	0.292 (2)	0.0404 (8)	0.021 (3)*
H8B	-0.0035 (5)	0.491 (2)	0.1046 (8)	0.021 (3)*
H9A	0.1055 (6)	1.065 (2)	0.3276 (10)	0.031 (3)*
H9B	0.0560 (7)	0.878 (3)	0.3713 (12)	0.051 (4)*
H9C	0.0387 (7)	1.102 (3)	0.3050 (11)	0.043 (4)*
H10A	0.2369 (6)	0.091 (2)	0.0350 (8)	0.025 (3)*

H10B	0.1868 (6)	-0.085 (2)	0.0749 (10)	0.030 (3)*
H10C	0.1886 (6)	-0.025 (3)	-0.0395 (10)	0.035 (4)*
O1	0.15679 (3)	0.24072 (13)	0.03720 (5)	0.02170 (18)
O2	0.07098 (3)	0.84683 (14)	0.22717 (5)	0.0297 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0188 (4)	0.0210 (4)	0.0148 (4)	-0.0041 (3)	0.0011 (3)	0.0013 (3)
C2	0.0208 (5)	0.0261 (5)	0.0207 (5)	-0.0034 (4)	-0.0019 (3)	0.0060 (4)
C3	0.0280 (5)	0.0328 (5)	0.0171 (4)	-0.0109 (4)	-0.0043 (4)	0.0065 (4)
C4	0.0314 (5)	0.0309 (5)	0.0148 (4)	-0.0139 (4)	0.0026 (4)	-0.0024 (4)
C5	0.0209 (5)	0.0234 (5)	0.0205 (5)	-0.0075 (3)	0.0050 (3)	-0.0033 (4)
C6	0.0170 (4)	0.0210 (4)	0.0166 (4)	-0.0043 (3)	0.0009 (3)	0.0001 (3)
C7	0.0181 (4)	0.0181 (4)	0.0192 (4)	0.0000 (3)	-0.0007 (3)	-0.0007 (3)
C8	0.0179 (4)	0.0186 (4)	0.0207 (5)	0.0004 (3)	-0.0006 (3)	-0.0004 (3)
C9	0.0369 (6)	0.0351 (6)	0.0341 (6)	-0.0084 (5)	0.0121 (5)	-0.0179 (5)
C10	0.0225 (5)	0.0207 (5)	0.0254 (5)	0.0030 (4)	0.0008 (4)	0.0000 (4)
O1	0.0247 (4)	0.0241 (4)	0.0163 (3)	0.0066 (3)	-0.0014 (2)	-0.0015 (2)
O2	0.0281 (4)	0.0317 (4)	0.0293 (4)	0.0002 (3)	0.0044 (3)	-0.0139 (3)

Geometric parameters (Å, °)

O1—C1	1.3746 (11)	C7—H7B	0.979 (12)
O1—C10	1.4251 (11)	C7—H7A	0.976 (12)
O2—C5	1.3650 (13)	C10—H10A	0.986 (13)
O2—C9	1.4314 (12)	C10—H10C	0.995 (14)
C5—C4	1.3992 (14)	C10—H10B	0.975 (14)
C5—C6	1.4042 (13)	C4—C3	1.3844 (16)
C8—C8 ⁱ	1.5283 (18)	C4—H4	0.947 (14)
C8—C7	1.5349 (12)	C2—C3	1.3857 (14)
C8—H8A	0.992 (12)	C2—H2	0.955 (13)
C8—H8B	0.987 (11)	C3—H3	0.979 (13)
C6—C1	1.3993 (13)	C9—H9A	0.985 (13)
C6—C7	1.5096 (12)	C9—H9B	0.994 (16)
C1—C2	1.3967 (13)	C9—H9C	0.943 (16)
C1—O1—C10	117.20 (7)	H7B—C7—H7A	108.5 (10)
C5—O2—C9	117.13 (9)	O1—C10—H10A	110.7 (7)
O2—C5—C4	123.58 (9)	O1—C10—H10C	105.8 (8)
O2—C5—C6	115.11 (8)	H10A—C10—H10C	110.8 (10)
C4—C5—C6	121.31 (9)	O1—C10—H10B	111.4 (8)
C8 ⁱ —C8—C7	112.48 (9)	H10A—C10—H10B	109.1 (10)
C8 ⁱ —C8—H8A	109.4 (7)	H10C—C10—H10B	109.0 (11)
C7—C8—H8A	109.0 (7)	C3—C4—C5	118.96 (9)
C8 ⁱ —C8—H8B	109.6 (7)	C3—C4—H4	120.7 (8)
C7—C8—H8B	108.9 (7)	C5—C4—H4	120.4 (8)
H8A—C8—H8B	107.3 (10)	C3—C2—C1	118.36 (9)

C1—C6—C5	117.49 (8)	C3—C2—H2	120.4 (7)
C1—C6—C7	121.24 (8)	C1—C2—H2	121.3 (7)
C5—C6—C7	121.26 (8)	C4—C3—C2	121.73 (9)
O1—C1—C2	122.79 (9)	C4—C3—H3	119.2 (8)
O1—C1—C6	115.08 (8)	C2—C3—H3	119.1 (8)
C2—C1—C6	122.12 (9)	O2—C9—H9A	109.7 (8)
C6—C7—C8	113.04 (7)	O2—C9—H9B	110.9 (9)
C6—C7—H7B	109.7 (7)	H9A—C9—H9B	111.9 (12)
C8—C7—H7B	108.7 (7)	O2—C9—H9C	105.9 (9)
C6—C7—H7A	108.2 (7)	H9A—C9—H9C	108.2 (12)
C8—C7—H7A	108.6 (7)	H9B—C9—H9C	110.1 (12)

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