

Crystal structure of 7,15-bis(4-*tert*-butylphenyl)-1,9-dimethylheptazethrene

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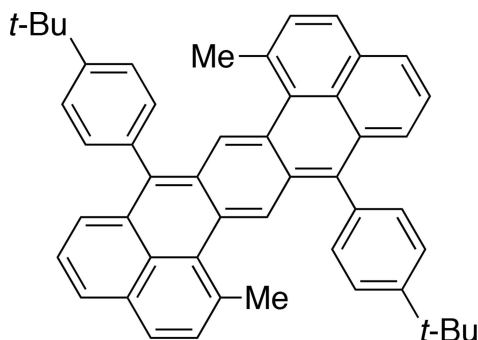
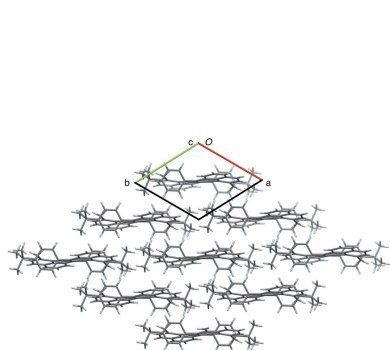
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The title compound, C₅₀H₄₄, **1**, was synthesized as a derivative of heptazethrene bearing two methyl and two *tert*-butylphenyl substituents, respectively, at the 1,9- and 7,15-positions. The asymmetric unit consists of one half of the molecule, which lies about an inversion centre. Albeit remotely located, the substituents contort the heptazethrene plane. The *tert*-butylphenyl substituents stand approximately perpendicular to the core plane, with a dihedral angle of 79.09 (5)° between the phenylene ring system and the substituted benzene ring, and prevent direct intermolecular contacts of the heptazethrene cores.

1. Chemical context

Heptazethrene is a polycyclic aromatic hydrocarbon with a characteristic Z-shaped molecular structure. A series of heptazethrene derivatives have been synthesized by one of the authors, and a derivative, **2**, with methyl and silylethynyl substituents at the 1,9- and 7,15-positions has been reported as the first closed-shell congener (Li *et al.*, 2012). In the crystal structure of **2**, we noticed that the silylethynyl substituents are distorted into a non-linear geometry. Considering that the distorted structure originated from steric interactions between the 1,9- and 7,15-positions, we investigated substituent effects on the molecular structure. Replacing the silylethynyl groups with *tert*-butylphenyl groups, we designed the title compound, **1**, and synthesized it *via* a route recently established for other heptazethrene derivatives (Hu *et al.*, 2016).



2. Structural commentary

The molecular structure of **1** (Fig. 1) consists of a heptazethrene unit at the core, two methyl substituents at the

Table 1

Hydrogen-bond geometry (Å, °).

*Cg*₁ and *Cg*₂ are the centroids of the C1–C3/C3A/C3A1/C8B and C3A1/C3A/C4–C6/C6A rings, respectively.

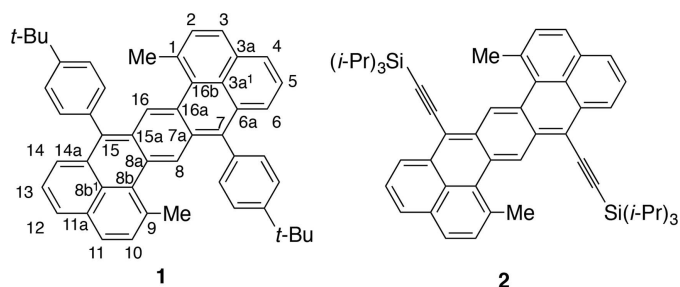
<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C3P–H3P··· <i>Cg</i> ₁ ⁱ	0.95	2.80	3.6278 (15)	147
C3T–H3T2··· <i>Cg</i> ₂ ⁱ	0.98	2.97	3.7929 (17)	143

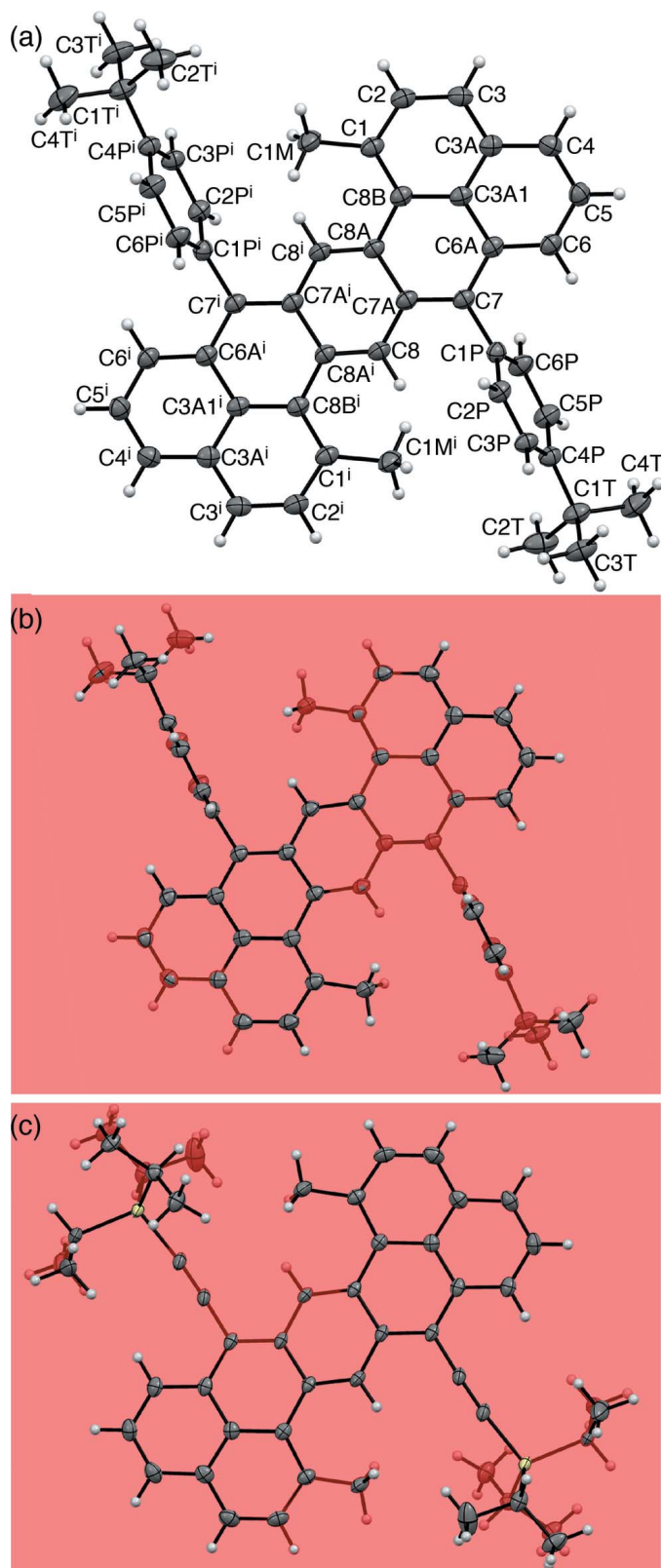
 Symmetry code: (i) *x*, *y* + 1, *z*.

1,9-positions and two *tert*-butylphenyl substituents at the 7,15-positions. One-half of the molecule is generated by the symmetry operation (1 – *x*, 1 – *y*, 1 – *z*), and carbon atoms at the 1/9-, 7/15- or 8/16-positions, for instance, are symmetrically equivalent (Fig. 2*a*). As is the case with **2**, a typical bond-length alternation in the central hexagon is observed, indicating a quinoidal character for **1** (Li *et al.*, 2012). Unlike **2**, however, the heptazethrene core of **1** is not flat but contorted. The mean plane of the heptazethrene core is generated by adopting 28 carbon atoms of the core (*OLEX2*; Dolomanov *et al.*, 2009) and the deviation of the atoms from the mean plane is visualized in Fig. 2*b*. The maximum deviation of 0.2969 (10) Å is recorded (by using *OLEX2*) for the carbon atoms at the 7- and 15-positions. The same analysis was applied to **2** (Fig. 2*c*), and the maximum distance from the mean plane is 0.103 (3) Å for the carbon atoms at the 8- and 16-positions. The contorted structure of **1** is also evidenced by the torsion angle at the 1–16b–16a–16 (see Fig. 1) positions, is –16.91 (19)°. For **2**, the torsion angle at the same position is 5.8 (3)°, which indicates that steric interactions between the 1-methyl and 7-phenyl groups may result in the contorted structure.

3. Supramolecular features

As is the case of **2** (Li *et al.*, 2012), the molecules of **1** form layers with the heptazethrene cores assembled in a parallel manner (Fig. 3). However, due to the bulky phenyl groups at the 7,15-positions, the heptazethrene cores do not directly contact each other. C–H···π interactions are instead observed between the heptazethrene core and the phenyl substituent (Table 1).


Figure 1

 Chemical structures of heptazethrene derivatives. Numbers of positions are displayed for **1**.

Figure 2

The molecular structures of heptazethrene derivatives. Displacement ellipsoids are drawn at the 50% probability level. (a) **1** with the atom-numbering scheme [symmetry code: (i) 1 – *x*, 1 – *y*, 1 – *z*]. (b) **1** with a mean plane of 28 C atoms, viewed perpendicular to the plane shown in red. (c) **2** with a mean plane of 28 C atoms, viewed perpendicular to the plane shown in red.

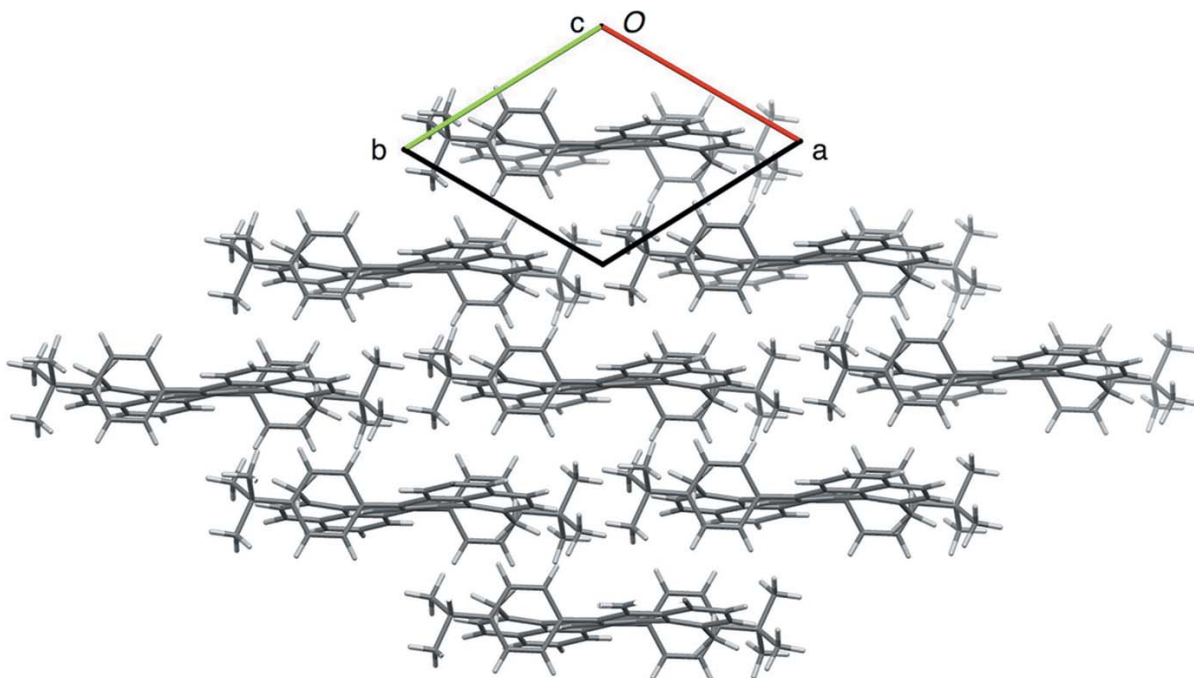


Figure 3
Packing diagram of **1**, viewed along the *c* axis.

4. Database survey

A search of the Cambridge Structural Database (version 5.37 Update 2; Groom *et al.*, 2016) for heptazethrene derivatives returns one result, compound **2** (Li *et al.*, 2012). Two newer derivatives, 1,9-bis(hexyloxy)-7,15-dimesityl-heptazethrene

and 1,9-bis(hexyloxy)-7,15-bis(pentafluorophenyl)-heptazethrene, have recently been reported (Hu *et al.*, 2016). Detailed comparisons with compound **2** are described above. The other two derivatives possessing 7,15-phenyl groups and 1,9-alkoxy substituents are also contorted. Two crystallographically independent molecules are observed in 1,9-bis(hexyloxy)-7,15-dimesityl-heptazethrene, and the 1–16b–16a–16 torsion angles are 2.55 (19) and 13.94 (19)°. One molecule is observed in 1,9-bis(hexyloxy)-7,15-bis(pentafluorophenyl)-heptazethrene, and the corresponding torsion angle is 6.44 (18)°.

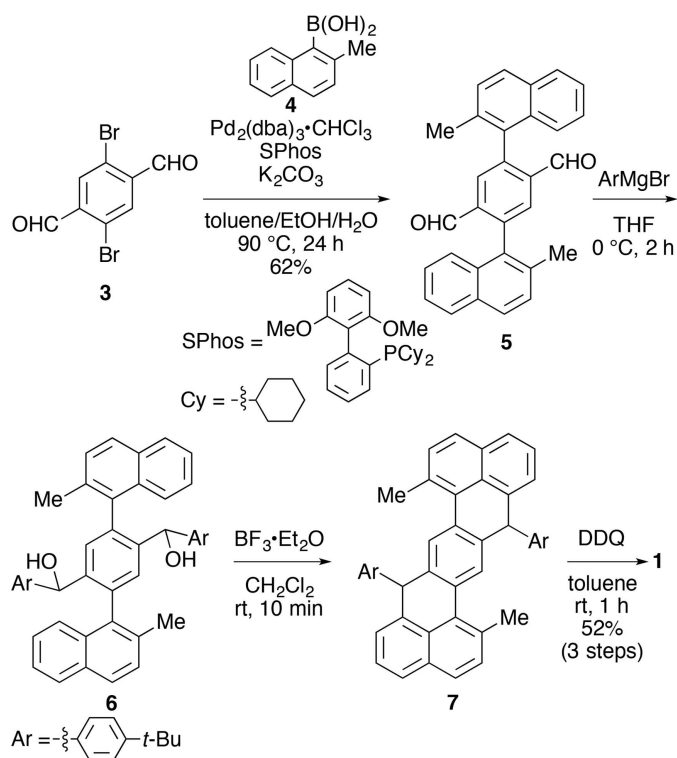


Figure 4
Synthesis of the title compound, **1**.

5. Synthesis and crystallization

The title compound **1** was synthesized by a method reported in a literature (Hu *et al.*, 2016) with different starting materials for the introduction of different substituents (Fig. 4). A mixture of 2,5-dibromo-terephthalaldehyde **3** (2.30 g, 7.89 mmol), 2-methylnaphthylboronic acid **4** (4.41 g, 23.7 mmol), $\text{Pd}_2(\text{dba})_3 \cdot \text{CHCl}_3$ (407 mg, 0.393 mmol), SPhos (648 mg, 1.58 mmol) and K_2CO_3 (5.46 g, 39.4 mmol) was stirred in a deaerated solvent composed of toluene (70.8 ml), ethanol (17.2 ml) and water (19.0 ml) at 363 K for 24 h. The reaction was quenched by addition of saturated aqueous NH_4Cl (50 ml). Organic materials were extracted with CHCl_3 (30 ml \times 4), and the combined organic phase was washed with brine, dried over MgSO_4 and concentrated *in vacuo*. Crude materials were purified by silica gel column chromatography (eluent: 30% CHCl_3 /hexane) to afford the coupling product **5** in 2.02 g (4.87 mmol, 62% yield) as a yellow powder. The compound **5** (1.64 g, 3.97 mmol) was dissolved in THF (80.0 mL), and to the solution was added 4-*tert*-butylphenyl-

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₅₀ H ₄₄
<i>M</i> _r	644.90
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	93
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.7644 (2), 9.2002 (3), 13.1212 (3)
α , β , γ (°)	105.874 (2), 95.080 (2), 115.249 (3)
<i>V</i> (Å ³)	894.47 (5)
<i>Z</i>	1
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	0.51
Crystal size (mm)	0.15 × 0.08 × 0.03
Data collection	
Diffractometer	Rigaku XtaLAB P200
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku Oxford Diffraction, 2015)
<i>T</i> _{min} – <i>T</i> _{max}	0.878, 0.985
No. of measured, independent and observed [<i>F</i> ² > 2.0σ(<i>F</i> ²)] reflections	22866, 3256, 2930
<i>R</i> _{int}	0.026
(sin θ /λ) _{max} (Å ⁻¹)	0.602
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.044, 0.131, 1.08
No. of reflections	3256
No. of parameters	230
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.37, -0.23

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXT* (Sheldrick, 2015a), *SHELXL2016* (Sheldrick, 2015b), *CrystalStructure* (Rigaku, 2016), *Mercury* (Macrae *et al.*, 2008), *OLEX2* (Dolomanov *et al.*, 2009) and *pubCIF* (Westrip, 2010).

magnesium bromide (30.0 ml, 0.66 *M* in diethyl ether, 19.8 mmol) at 273 K. The mixture was stirred for 2 h, and saturated aqueous NH₄Cl (20 ml) was added. Organic materials were extracted with ethyl acetate (50 ml × 3), and the combined organic phase was washed with brine, dried over Na₂SO₄ and concentrated *in vacuo* to give a yellow oil containing diol **6**. Without purification, the crude material was dissolved in CH₂Cl₂ (200 ml), and BF₃·Et₂O (5.10 ml, 39.5 mmol) was added at ambient temperature. After 10 min, methanol (10 ml) was added, and volatile materials were removed *in vacuo*. The crude material was washed with methanol (50 ml), and a purple solid containing the cyclized compound **7** was obtained. Without purification, the crude material was dissolved in toluene (400 ml), and to the solution was added a solution of 2,3-dichloro-5,6-dicyano-*para*-benzoquinone (DDQ; 70.0 ml, 79.5 mM in toluene, 5.57 mmol) at ambient temperature. After 1 h, the mixture was poured onto a pad of silica gel (250 g) and eluted with toluene to afford the title compound **1**. A small amount of contaminants was noted and was removed by washing the compound with methanol (50 ml) to afford the title compound **1** (1.33 g, 2.06 mmol, 52% in three steps from **5**) as a purple solid. Suitable single crystals

were grown from slow liquid–liquid diffusion of acetonitrile into a toluene solution of **1**.

Physical data: m.p. *ca* 643 K (decomposed); IR (ATR, neat): 567, 587, 764, 803, 825, 837, 1017, 1108, 1269, 1362, 1447, 1461, 2864, 2901, 2951 cm⁻¹; ¹H NMR (400 MHz, C₆D₆) δ 1.32 (*s*, 18H), 2.36 (*s*, 6H), 7.03 (*t*, *J* = 8.0 Hz, 2H), 7.09 (*d*, *J* = 8.4 Hz, 2H), 7.25 (*d*, *J* = 7.8 Hz, 2H), 7.28 (*dt*, *J* = 8.4 Hz, 2.0 Hz, 4H), 7.36 (*d*, *J* = 8.0 Hz, 2H), 7.37 (*dt*, *J* = 8.4 Hz, 2.0 Hz, 4H), 7.38 (*d*, *J* = 8.4 Hz, 2.0 Hz, 4H), 7.72 (*s*, 2H); ¹³C NMR (100 MHz, C₆D₆) δ 26.0 (CH₃), 31.5 (CH₃), 34.6, 125.6 (CH), 126.1* (CH), 127.1 (CH), 127.4 (CH), 129.2 (CH), 129.7, 130.3, 131.0* (CH), 131.7, 132.6 (CH), 133.2, 133.6, 134.7, 136.0, 136.7, 150.3 (Resonances with * appeared with twofold intensities and should contain two overlapping resonances.); HR-MS (DART-TOF, positive) calculated for C₅₀H₄₄ [*M*+H]⁺ 645.3521, found 645.3545.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically and refined as riding, allowing for rotation of the methyl group, with *U*_{iso}(H) = 1.5*U*_{eq}(C) for methyl H atoms and 1.2*U*_{eq}(C) for aromatic H atoms.

Acknowledgements

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References

- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Hu, P., Lee, S., Heng, T. S., Aratani, N., Gonçalves, T. P., Qi, Q., Shi, X., Yamada, H., Huang, K.-H., Ding, J., Kim, D. & Wu, J. (2016). *J. Am. Chem. Soc.* **138**, 1065–1077.
- Li, Y., Heng, W.-K., Lee, B. S., Aratani, N., Zafra, J. L., Bao, N., Lee, R., Sung, Y. M., Sun, Z., Huang, K.-H., Webster, R. R., López Navarrete, J. T., Kim, D., Osuka, A., Casado, J., Ding, J. & Wu, J. (2012). *J. Am. Chem. Soc.* **134**, 14913–14922.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Rigaku (2016). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
- Rigaku Oxford Diffraction (2015). *CrysAlis PRO*. Rigaku Corporation, Tokyo, Japan.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

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Crystal structure of 7,15-bis(4-*tert*-butylphenyl)-1,9-dimethylheptazethrene

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Computing details

Data collection: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); cell refinement: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); data reduction: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: *CrystalStructure* (Rigaku, 2016) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2016), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

7,15-Bis(4-*tert*-butylphenyl)-1,9-dimethylheptazethrene

Crystal data

$C_{50}H_{44}$	$Z = 1$
$M_r = 644.90$	$F(000) = 344.00$
Triclinic, $P\bar{1}$	$D_x = 1.197 \text{ Mg m}^{-3}$
$a = 8.7644 (2) \text{ \AA}$	Cu $K\alpha$ radiation, $\lambda = 1.54187 \text{ \AA}$
$b = 9.2002 (3) \text{ \AA}$	Cell parameters from 15939 reflections
$c = 13.1212 (3) \text{ \AA}$	$\theta = 3.6\text{--}68.2^\circ$
$\alpha = 105.874 (2)^\circ$	$\mu = 0.51 \text{ mm}^{-1}$
$\beta = 95.080 (2)^\circ$	$T = 93 \text{ K}$
$\gamma = 115.249 (3)^\circ$	Block, purple
$V = 894.47 (5) \text{ \AA}^3$	$0.15 \times 0.08 \times 0.03 \text{ mm}$

Data collection

Rigaku XtaLAB P200 diffractometer	22866 measured reflections
Detector resolution: $5.811 \text{ pixels mm}^{-1}$	3256 independent reflections
ω scans	2930 reflections with $F^2 > 2.0\sigma(F^2)$
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Rigaku Oxford Diffraction, 2015)	$R_{\text{int}} = 0.026$
$T_{\text{min}} = 0.878$, $T_{\text{max}} = 0.985$	$\theta_{\text{max}} = 68.3^\circ$, $\theta_{\text{min}} = 3.6^\circ$
	$h = -10 \rightarrow 10$
	$k = -9 \rightarrow 10$
	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.044$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.131$	H-atom parameters constrained
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.0807P)^2 + 0.1872P]$
3256 reflections	where $P = (F_o^2 + 2F_c^2)/3$
230 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

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. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2 . R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0 \text{ sigma}(F^2)$ is used only for calculating R-factor (gt).

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	0.77143 (15)	0.27792 (16)	0.37967 (10)	0.0282 (3)
C2	0.82438 (16)	0.19027 (16)	0.29671 (11)	0.0304 (3)
H2	0.901044	0.149216	0.316696	0.036*
C3	0.77086 (16)	0.16138 (16)	0.18882 (11)	0.0294 (3)
H3	0.802444	0.093561	0.135216	0.035*
C3A	0.66842 (15)	0.23267 (15)	0.15725 (10)	0.0270 (3)
C3A1	0.61706 (14)	0.32728 (14)	0.23942 (10)	0.0247 (3)
C4	0.61937 (16)	0.21352 (16)	0.04656 (10)	0.0299 (3)
H4	0.651358	0.147501	-0.007973	0.036*
C5	0.52627 (16)	0.28945 (16)	0.01768 (10)	0.0308 (3)
H5	0.492798	0.274895	-0.056770	0.037*
C6	0.48038 (16)	0.38832 (16)	0.09751 (10)	0.0284 (3)
H6	0.418515	0.442753	0.076400	0.034*
C6A	0.52289 (15)	0.40922 (15)	0.20719 (10)	0.0254 (3)
C7	0.47383 (15)	0.51085 (15)	0.28851 (10)	0.0253 (3)
C7A	0.49880 (15)	0.51364 (15)	0.39461 (10)	0.0255 (3)
C8	0.42937 (15)	0.59605 (15)	0.47208 (10)	0.0261 (3)
H8	0.386896	0.666232	0.452300	0.031*
C8A	0.57990 (15)	0.41936 (15)	0.42770 (10)	0.0249 (3)
C8B	0.65887 (15)	0.34037 (15)	0.35078 (10)	0.0255 (3)
C1M	0.84692 (17)	0.29973 (19)	0.49464 (11)	0.0359 (3)
H1M1	0.953456	0.288760	0.496403	0.054*
H1M2	0.762462	0.211359	0.517639	0.054*
H1M3	0.873653	0.413186	0.544212	0.054*
C1P	0.37768 (15)	0.59823 (15)	0.25632 (9)	0.0257 (3)
C2P	0.45677 (15)	0.77354 (16)	0.27639 (10)	0.0275 (3)
H2P	0.578450	0.840095	0.305958	0.033*
C3P	0.36053 (16)	0.85288 (16)	0.25386 (10)	0.0278 (3)
H3P	0.418003	0.972795	0.268045	0.033*
C4P	0.18176 (15)	0.76070 (15)	0.21096 (10)	0.0273 (3)
C5P	0.10460 (16)	0.58447 (16)	0.18943 (11)	0.0329 (3)
H5P	-0.016815	0.517320	0.158986	0.039*
C6P	0.19987 (16)	0.50500 (16)	0.21115 (11)	0.0317 (3)
H6P	0.142991	0.384575	0.194958	0.038*
C1T	0.06794 (16)	0.84316 (17)	0.19069 (11)	0.0333 (3)
C2T	-0.05461 (19)	0.8203 (2)	0.26853 (15)	0.0468 (4)

H2T1	-0.118334	0.698859	0.259859	0.070*
H2T2	-0.137001	0.861074	0.251474	0.070*
H2T3	0.013342	0.886463	0.343903	0.070*
C3T	0.17399 (17)	1.03289 (17)	0.20891 (13)	0.0401 (3)
H3T1	0.096047	1.078314	0.191671	0.060*
H3T2	0.255721	1.048167	0.161518	0.060*
H3T3	0.238494	1.094359	0.285279	0.060*
C4T	-0.04149 (19)	0.7524 (2)	0.07259 (13)	0.0465 (4)
H4T1	-0.118581	0.631892	0.061158	0.070*
H4T2	0.035486	0.759603	0.022257	0.070*
H4T3	-0.111104	0.807899	0.059085	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0275 (6)	0.0267 (6)	0.0350 (6)	0.0137 (5)	0.0094 (5)	0.0150 (5)
C2	0.0297 (6)	0.0289 (6)	0.0402 (7)	0.0168 (5)	0.0122 (5)	0.0169 (5)
C3	0.0297 (6)	0.0253 (6)	0.0382 (7)	0.0147 (5)	0.0143 (5)	0.0130 (5)
C3A	0.0256 (6)	0.0229 (6)	0.0331 (6)	0.0092 (5)	0.0105 (5)	0.0129 (5)
C3A1	0.0226 (5)	0.0204 (6)	0.0323 (6)	0.0087 (5)	0.0088 (5)	0.0124 (5)
C4	0.0327 (6)	0.0255 (6)	0.0324 (6)	0.0129 (5)	0.0126 (5)	0.0112 (5)
C5	0.0357 (7)	0.0287 (7)	0.0286 (6)	0.0132 (5)	0.0095 (5)	0.0131 (5)
C6	0.0306 (6)	0.0263 (6)	0.0319 (6)	0.0130 (5)	0.0088 (5)	0.0153 (5)
C6A	0.0239 (6)	0.0221 (6)	0.0312 (6)	0.0088 (5)	0.0082 (5)	0.0132 (5)
C7	0.0248 (6)	0.0223 (6)	0.0309 (6)	0.0106 (5)	0.0074 (5)	0.0128 (5)
C7A	0.0256 (6)	0.0229 (6)	0.0307 (6)	0.0114 (5)	0.0077 (5)	0.0131 (5)
C8	0.0272 (6)	0.0247 (6)	0.0320 (6)	0.0145 (5)	0.0078 (5)	0.0137 (5)
C8A	0.0247 (6)	0.0224 (6)	0.0290 (6)	0.0112 (5)	0.0058 (4)	0.0110 (5)
C8B	0.0239 (6)	0.0218 (6)	0.0315 (6)	0.0093 (5)	0.0078 (5)	0.0124 (5)
C1M	0.0378 (7)	0.0472 (8)	0.0390 (7)	0.0293 (6)	0.0127 (6)	0.0218 (6)
C1P	0.0294 (6)	0.0279 (6)	0.0255 (6)	0.0152 (5)	0.0097 (5)	0.0137 (5)
C2P	0.0245 (6)	0.0276 (6)	0.0327 (6)	0.0117 (5)	0.0083 (5)	0.0144 (5)
C3P	0.0283 (6)	0.0237 (6)	0.0351 (6)	0.0123 (5)	0.0098 (5)	0.0144 (5)
C4P	0.0277 (6)	0.0269 (6)	0.0315 (6)	0.0142 (5)	0.0091 (5)	0.0135 (5)
C5P	0.0256 (6)	0.0279 (7)	0.0434 (7)	0.0104 (5)	0.0041 (5)	0.0146 (6)
C6P	0.0311 (6)	0.0229 (6)	0.0409 (7)	0.0114 (5)	0.0055 (5)	0.0139 (5)
C1T	0.0261 (6)	0.0300 (7)	0.0477 (7)	0.0144 (5)	0.0088 (5)	0.0171 (6)
C2T	0.0397 (8)	0.0401 (8)	0.0768 (11)	0.0247 (7)	0.0285 (7)	0.0292 (8)
C3T	0.0315 (7)	0.0308 (7)	0.0654 (9)	0.0172 (6)	0.0110 (6)	0.0232 (7)
C4T	0.0373 (7)	0.0427 (8)	0.0607 (9)	0.0206 (7)	-0.0017 (7)	0.0205 (7)

Geometric parameters (Å, °)

C1—C2	1.4068 (18)	C1M—H1M2	0.9800
C1—C8B	1.4084 (17)	C1M—H1M3	0.9800
C1—C1M	1.5159 (17)	C1P—C2P	1.3902 (17)
C2—C3	1.3655 (18)	C1P—C6P	1.3905 (17)
C2—H2	0.9500	C2P—C3P	1.3896 (17)

C3—C3A	1.4117 (17)	C2P—H2P	0.9500
C3—H3	0.9500	C3P—C4P	1.3928 (17)
C3A—C4	1.4178 (17)	C3P—H3P	0.9500
C3A—C3A1	1.4247 (17)	C4P—C5P	1.3950 (17)
C3A1—C8B	1.4335 (17)	C4P—C1T	1.5331 (17)
C3A1—C6A	1.4394 (17)	C5P—C6P	1.3804 (17)
C4—C5	1.3689 (18)	C5P—H5P	0.9500
C4—H4	0.9500	C6P—H6P	0.9500
C5—C6	1.3970 (18)	C1T—C3T	1.5220 (18)
C5—H5	0.9500	C1T—C2T	1.5361 (19)
C6—C6A	1.3926 (17)	C1T—C4T	1.538 (2)
C6—H6	0.9500	C2T—H2T1	0.9800
C6A—C7	1.4409 (17)	C2T—H2T2	0.9800
C7—C7A	1.3813 (17)	C2T—H2T3	0.9800
C7—C1P	1.4952 (16)	C3T—H3T1	0.9800
C7A—C8	1.4353 (17)	C3T—H3T2	0.9800
C7A—C8A	1.4572 (16)	C3T—H3T3	0.9800
C8—C8A ⁱ	1.3664 (17)	C4T—H4T1	0.9800
C8—H8	0.9500	C4T—H4T2	0.9800
C8A—C8B	1.4756 (17)	C4T—H4T3	0.9800
C1M—H1M1	0.9800		
C2—C1—C8B	118.76 (11)	C1—C1M—H1M3	109.5
C2—C1—C1M	115.40 (11)	H1M1—C1M—H1M3	109.5
C8B—C1—C1M	125.82 (11)	H1M2—C1M—H1M3	109.5
C3—C2—C1	123.15 (12)	C2P—C1P—C6P	117.68 (11)
C3—C2—H2	118.4	C2P—C1P—C7	122.52 (11)
C1—C2—H2	118.4	C6P—C1P—C7	119.66 (11)
C2—C3—C3A	119.76 (12)	C3P—C2P—C1P	121.00 (11)
C2—C3—H3	120.1	C3P—C2P—H2P	119.5
C3A—C3—H3	120.1	C1P—C2P—H2P	119.5
C3—C3A—C4	121.12 (11)	C2P—C3P—C4P	121.54 (11)
C3—C3A—C3A1	118.58 (11)	C2P—C3P—H3P	119.2
C4—C3A—C3A1	120.30 (11)	C4P—C3P—H3P	119.2
C3A—C3A1—C8B	120.68 (11)	C3P—C4P—C5P	116.84 (11)
C3A—C3A1—C6A	118.01 (11)	C3P—C4P—C1T	123.67 (11)
C8B—C3A1—C6A	121.30 (11)	C5P—C4P—C1T	119.45 (11)
C5—C4—C3A	120.49 (11)	C6P—C5P—C4P	121.80 (11)
C5—C4—H4	119.8	C6P—C5P—H5P	119.1
C3A—C4—H4	119.8	C4P—C5P—H5P	119.1
C4—C5—C6	120.12 (11)	C5P—C6P—C1P	121.12 (12)
C4—C5—H5	119.9	C5P—C6P—H6P	119.4
C6—C5—H5	119.9	C1P—C6P—H6P	119.4
C6A—C6—C5	121.78 (12)	C3T—C1T—C4P	112.58 (10)
C6A—C6—H6	119.1	C3T—C1T—C2T	109.31 (12)
C5—C6—H6	119.1	C4P—C1T—C2T	108.22 (11)
C6—C6A—C3A1	119.22 (11)	C3T—C1T—C4T	108.22 (11)
C6—C6A—C7	121.14 (11)	C4P—C1T—C4T	109.63 (11)

C3A1—C6A—C7	119.64 (11)	C2T—C1T—C4T	108.82 (12)
C7A—C7—C6A	120.21 (11)	C1T—C2T—H2T1	109.5
C7A—C7—C1P	119.24 (11)	C1T—C2T—H2T2	109.5
C6A—C7—C1P	120.20 (10)	H2T1—C2T—H2T2	109.5
C7—C7A—C8	120.18 (11)	C1T—C2T—H2T3	109.5
C7—C7A—C8A	121.18 (11)	H2T1—C2T—H2T3	109.5
C8—C7A—C8A	118.35 (11)	H2T2—C2T—H2T3	109.5
C8A ⁱ —C8—C7A	124.58 (11)	C1T—C3T—H3T1	109.5
C8A ⁱ —C8—H8	117.7	C1T—C3T—H3T2	109.5
C7A—C8—H8	117.7	H3T1—C3T—H3T2	109.5
C8 ⁱ —C8A—C7A	116.82 (11)	C1T—C3T—H3T3	109.5
C8 ⁱ —C8A—C8B	124.06 (11)	H3T1—C3T—H3T3	109.5
C7A—C8A—C8B	119.05 (11)	H3T2—C3T—H3T3	109.5
C1—C8B—C3A1	118.48 (11)	C1T—C4T—H4T1	109.5
C1—C8B—C8A	124.42 (11)	C1T—C4T—H4T2	109.5
C3A1—C8B—C8A	117.10 (11)	H4T1—C4T—H4T2	109.5
C1—C1M—H1M1	109.5	C1T—C4T—H4T3	109.5
C1—C1M—H1M2	109.5	H4T1—C4T—H4T3	109.5
H1M1—C1M—H1M2	109.5	H4T2—C4T—H4T3	109.5
C8B—C1—C2—C3	-0.49 (19)	C2—C1—C8B—C3A1	-6.23 (17)
C1M—C1—C2—C3	-179.09 (12)	C1M—C1—C8B—C3A1	172.20 (11)
C1—C2—C3—C3A	5.13 (19)	C2—C1—C8B—C8A	173.80 (11)
C2—C3—C3A—C4	176.25 (11)	C1M—C1—C8B—C8A	-7.76 (19)
C2—C3—C3A—C3A1	-2.76 (17)	C3A—C3A1—C8B—C1	8.50 (17)
C3—C3A—C3A1—C8B	-4.01 (17)	C6A—C3A1—C8B—C1	-171.30 (10)
C4—C3A—C3A1—C8B	176.97 (10)	C3A—C3A1—C8B—C8A	-171.53 (10)
C3—C3A—C3A1—C6A	175.79 (10)	C6A—C3A1—C8B—C8A	8.67 (16)
C4—C3A—C3A1—C6A	-3.22 (17)	C8 ⁱ —C8A—C8B—C1	-16.91 (19)
C3—C3A—C4—C5	-177.21 (11)	C7A—C8A—C8B—C1	166.07 (11)
C3A1—C3A—C4—C5	1.78 (18)	C8 ⁱ —C8A—C8B—C3A1	163.12 (11)
C3A—C4—C5—C6	0.71 (18)	C7A—C8A—C8B—C3A1	-13.90 (16)
C4—C5—C6—C6A	-1.71 (19)	C7A—C7—C1P—C2P	81.89 (15)
C5—C6—C6A—C3A1	0.18 (18)	C6A—C7—C1P—C2P	-104.89 (14)
C5—C6—C6A—C7	-179.66 (11)	C7A—C7—C1P—C6P	-93.64 (14)
C3A—C3A1—C6A—C6	2.25 (16)	C6A—C7—C1P—C6P	79.58 (14)
C8B—C3A1—C6A—C6	-177.94 (10)	C6P—C1P—C2P—C3P	1.16 (17)
C3A—C3A1—C6A—C7	-177.91 (10)	C7—C1P—C2P—C3P	-174.46 (10)
C8B—C3A1—C6A—C7	1.89 (17)	C1P—C2P—C3P—C4P	0.31 (18)
C6—C6A—C7—C7A	172.31 (11)	C2P—C3P—C4P—C5P	-1.40 (18)
C3A1—C6A—C7—C7A	-7.52 (17)	C2P—C3P—C4P—C1T	176.39 (11)
C6—C6A—C7—C1P	-0.83 (17)	C3P—C4P—C5P—C6P	1.05 (19)
C3A1—C6A—C7—C1P	179.33 (10)	C1T—C4P—C5P—C6P	-176.83 (12)
C6A—C7—C7A—C8	-171.72 (10)	C4P—C5P—C6P—C1P	0.4 (2)
C1P—C7—C7A—C8	1.50 (17)	C2P—C1P—C6P—C5P	-1.51 (18)
C6A—C7—C7A—C8A	2.04 (17)	C7—C1P—C6P—C5P	174.24 (11)
C1P—C7—C7A—C8A	175.25 (10)	C3P—C4P—C1T—C3T	7.25 (18)
C7—C7A—C8—C8A ⁱ	168.00 (12)	C5P—C4P—C1T—C3T	-175.02 (12)

C8A—C7A—C8—C8A ⁱ	-5.93 (19)	C3P—C4P—C1T—C2T	-113.67 (14)
C7—C7A—C8A—C8 ⁱ	-168.40 (12)	C5P—C4P—C1T—C2T	64.06 (15)
C8—C7A—C8A—C8 ⁱ	5.47 (18)	C3P—C4P—C1T—C4T	127.77 (13)
C7—C7A—C8A—C8B	8.83 (17)	C5P—C4P—C1T—C4T	-54.50 (15)
C8—C7A—C8A—C8B	-177.30 (10)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 and *Cg2* are the centroids of the C1—C3/C3A/C3A1/C8B and C3A1/C3A/C4—C6/C6A rings, respectively.

<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>
C3P—H3P \cdots Cg1 ⁱⁱ	0.95	2.80	3.6278 (15)	147
C3T—H3T2 \cdots Cg2 ⁱⁱ	0.98	2.97	3.7929 (17)	143

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