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1,4-Bis(triisopropylsilyl)buta-1,3-diyne and 1,4-bis(biphenyl-4-yl)buta-1,3-diyne

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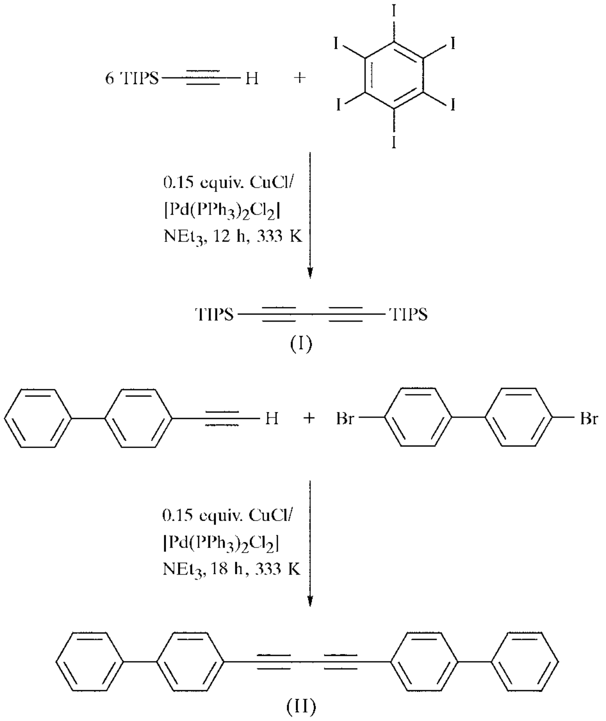
We report the single crystal structures of 1,4-bis(triisopropylsilyl)buta-1,3-diyne, $C_{22}H_{42}Si_2$, and 1,4-bis(biphenyl-4-yl)buta-1,3-diyne, $C_{28}H_{18}$, the packing in both of which illustrates the versatility of weak C—H···π supramolecular interactions in dictating the overall solid-state structures.

Comment

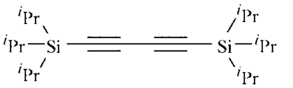
We have been interested in the development of polyalkyne-based stars and dendrimers and their reactions with $Co_2(CO)_8$ to produce organometallic cluster-decorated architectures (Constable *et al.*, 2006). We have used Sonogashira palladium-catalysed cross-coupling reactions (Sonogashira *et al.*, 1975; Sonogashira, 2002) for the divergent assembly of polyalkynes containing rigid frameworks with well-defined structures. Under Sonogashira conditions, reactions between terminal alkynes (RC≡CH) and aryl halides can give rise to diynes (RC≡C—C≡CR) as side-products (these most often arise from bromo precursors) (Sonogashira *et al.* 1975; Sonogashira 2002). Related reactions (Liu & Burton, 1997) or modified Sonogashira conditions (Rossi *et al.*, 1985) have been used for the specific formation of diynes. Two molecular cores that we have investigated are hexakis[(triisopropylsilyl)ethynyl]benzene and 4,4′-bis(biphenyl-4-ylethynyl)biphenyl. During attempts to synthesize these compounds, we found that 1,4-bis(triisopropylsilyl)buta-1,3-diyne, (I), and 1,4-bis(biphenyl-4-yl)buta-1,3-diyne, (II), could be produced quantitatively.

With the aim of preparing $C_6(C{\equiv}CSi^iPr_3)_6$, we treated C_6I_6 with six equivalents of $^iPr_3SiC{\equiv}CH$ under Sonogashira cross-coupling conditions. Instead of the desired product, compound (I) was formed quantitatively under the conditions shown in the scheme. This was also the case when C_6Br_6 was used as the precursor. Similarly (see scheme), the palladium-catalysed cross-coupling reaction between 4-ethynylbiphenyl and 4,4′-dibromobiphenyl led to the quantitative formation of (II). Compounds (I) and (II) have been reported previously (Eisler *et al.*, 2005; Hlavatý *et al.*, 2002; Ried & Saxena, 1970; Toda & Tokumaru, 1990), but have not, to our knowledge, been structurally characterized. We report here their single-crystal structures, which illustrate a number of facets of weak C—H···π interactions in dictating solid-state structures. Such hydrogen bonds are now well established as important components in solid-state supramolecular assemblies (Desiraju, 2002, 2005; Desiraju & Steiner, 1999; Nishio, 2004; Nishio *et al.*, 1998; Steiner, 2002), and their role in organic reactions has recently been assessed (Nishio, 2005).

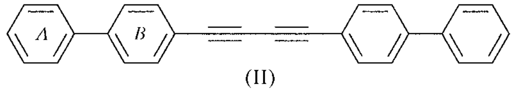


(I)

(II)



(I)



(II)

X-ray quality crystals of (I) were grown from a CH_2Cl_2 solution. Fig. 1 shows the structure of the centrosymmetric molecule of (I). The carbon backbone is linear, as observed for $Me_3Si(C{\equiv}C)_2SiMe_3$ (Carré *et al.*, 2003) and $^iPr_3Si(C{\equiv}C)_n$-Si^iPr_3 (n = 4, 5 or 6; Eisler *et al.*, 2005), in contrast with the curved backbone of $^iPr_3Si(C{\equiv}C)_8Si^iPr_3$ (Eisler *et al.*, 2005). The C—Si—C bond angles lie in the range 105.97 (9)–116.94 (13)°.

Molecules of (I) pack in rows (Fig. 2*a*), such that the distance between the least-squares planes containing adjacent rows of SiCCCCSi chains is 5.8 Å. Adjacent chains are interlocked, with the packing being supported by close methyl C—H···(alkyne π) interactions (C8—H83···C1 = 2.9 Å and C8—

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H83$\cdots$C2 = 3.1 Å). This leads to the presence of close (repulsive) H$\cdots$H contacts (Me$_2$C—H61$\cdots$H61—CMe$_2$ = 2.7 Å). A second set of C—H$\cdots\pi$ interactions operates between molecules within each row (Fig. 2*b*). Their evolution gives rise to short H$\cdots$H contacts between pairs of molecules.

The structures of a number of molecules closely related to (I) have been determined and comparisons of the solid-state packing are instructive. A search of the Cambridge Structural Database (CSD, Version 5.2.7; Allen, 2002; Bruno *et al.*, 2002) for molecules containing an E—C$\equiv$C—C$\equiv$C—E unit (E is Si, Sn, Ge or Pb) gave only 14 hits (Brouty *et al.*, 1980; Brunel *et al.*, 2001; Carré *et al.*, 1999, 2003; Dam *et al.*, 1998; Neugebauer *et al.*, 2000). Among these are two polymorphs of Me$_3$SiC$\equiv$CC$\equiv$CSiMe$_3$ (structures determined at 120 and 203 K; Carré *et al.*, 2003). The packing of the molecules in both polymorphs differs from that in (I). Although the molecules are interlocked by virtue of the close approach of SiMe$_3$ and alkyne groups, molecules in both polymorphs of Me$_3$SiC$\equiv$CC$\equiv$ CSiMe$_3$ form grid-like assemblies, in contrast with the parallel alignment of molecules observed in the solid state of (I).

Compound (I) is a member of a family of polyynes, ^{i}Pr$_3$Si(C$\equiv$C)$_n$Si^{i}Pr$_3$ (n = 4, 5, 6 and 8; Eisler *et al.*, 2005). The solid-state packing of ^{i}Pr$_3$Si(C$\equiv$C)$_4$Si^{i}Pr$_3$ resembles that of (I), with molecules organized in offset rows, while for ^{i}Pr$_3$Si(C$\equiv$C)$_5$Si^{i}Pr$_3$ and ^{i}Pr$_3$Si(C$\equiv$C)$_6$Si^{i}Pr$_3$, a herring-bone

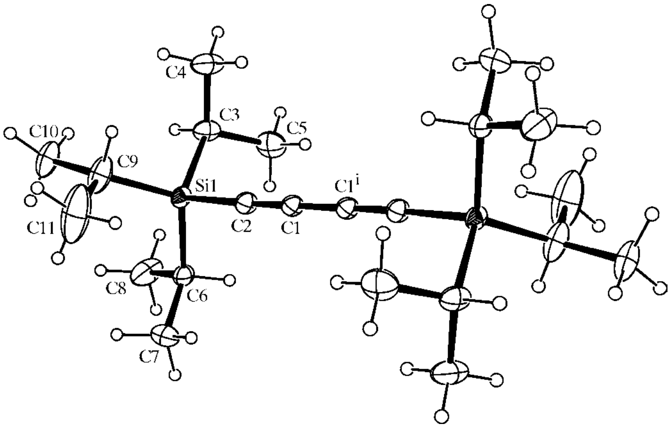


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitary radii. Unlabelled atoms and C1^i are generated by the symmetry operator ($-x + 2$, $-y + 1$, $-z + 1$).

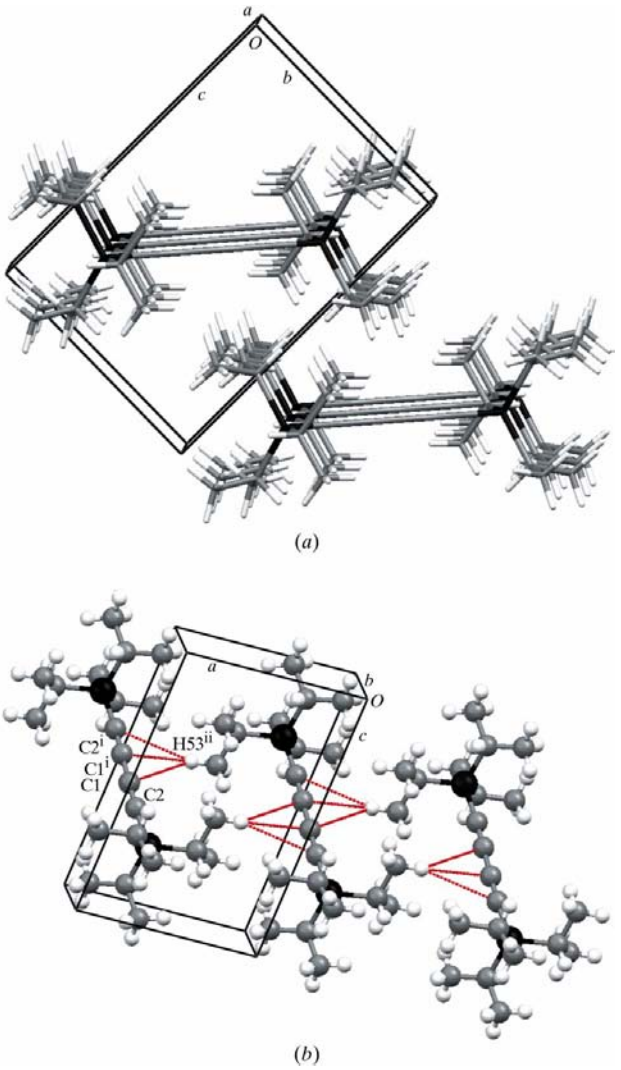


Figure 2
(*a*) The packing of molecules of (I), showing parts of two offset rows. (*b*) C—H$\cdots\pi$ interactions between adjacent molecules in a row (C2$^i\cdots$H53^{ii} = 3.2 Å, C1$^i\cdots$H53^{ii} = 2.9 Å and C1$\cdots$H53^{ii} = 3.1 Å). [Symmetry codes: (i) $-x + 2$, $-y + 1$, $-z + 1$; (ii) $1 - x$, $1 - y$, $1 - z$.]

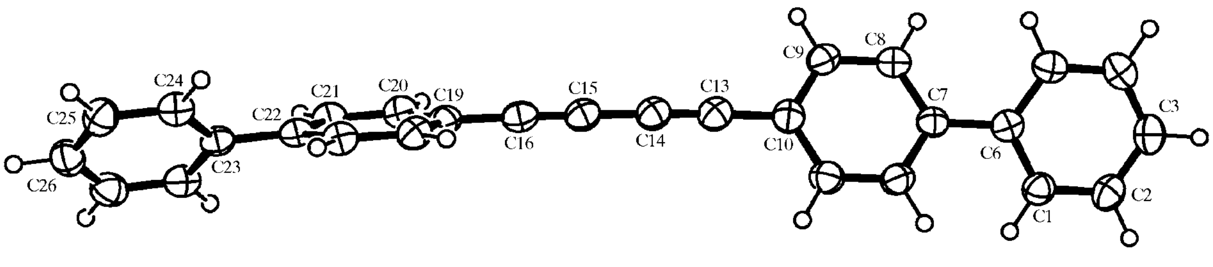


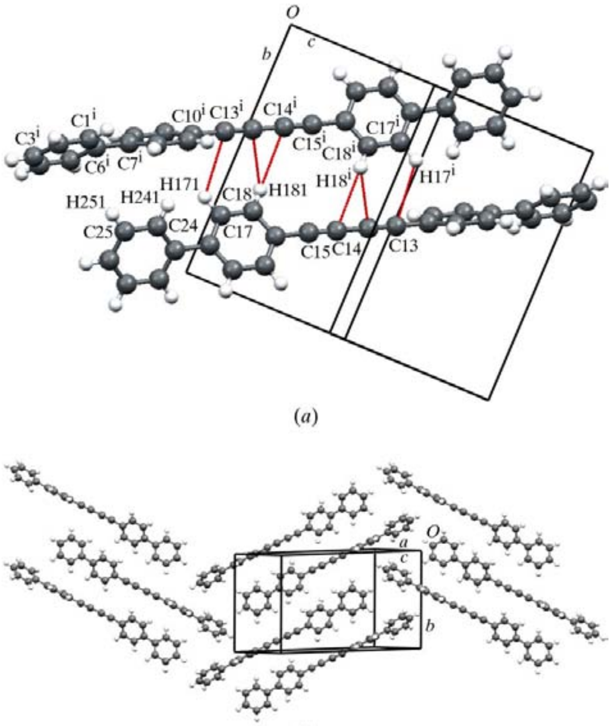
Figure 3
The molecular structure of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitary radii.

assembly is observed. In $^{i}Pr_3Si(C{\equiv}C)_8Si^{i}Pr_3$, the polyyne backbone is significantly curved and the molecular packing is less readily compared with that of the smaller polyynes (Eisler *et al.*, 2005).

Crystals of (II) were grown from a CH_2Cl_2 solution, and the molecular structure is shown in Fig. 3. The molecule is slightly bowed and the aryl rings are twisted with respect to one another, so that the angles between the least-squares planes of the rings containing atoms C6 and C7, atoms C7 and C22, and atoms C22 and C23 are 28.70 (7), 61.07 (6) and 44.22 (6)°, respectively. The origin of these ring orientations can be traced to the intermolecular $C—H\cdots\pi$ interactions listed in Table 3. The basic motif in the solid state is a dimeric unit (Fig. 4*a*), in which both $C—H_{aryl}\cdots\pi_{alkyne}$ and $C—H_{aryl}\cdots\pi_{aryl}$ interactions are present (Table 3).

The dimers further assemble into layers (Fig. 4*b*), again with $C—H\cdots\pi$ interactions playing a role (Table 3). Stacking of planes of molecules into the three-dimensional lattice is also supported by $C—H\cdots\pi$ contacts (Table 3) . The molecular structure of (II) shows interesting contrasts with that of 1,4-diphenylbuta-1,3-diyne (Fronczek & Erickson, 1995; Surette *et al.*, 1994). Molecules of the latter are planar in the solid state and pack in a herring-bone arrangement. Whereas $C—H\cdots\pi$ contacts control the ring orientations and packing in (II), π-stacking interactions are important in 1,4-diphenylbuta-1,3-diyne. Also related to (II) is 4-ethynylbiphenyl (Mague *et al.*, 1997). As in (II), the biphenyl unit of 4-ethynylbiphenyl is non-planar. The authors (Mague *et al.*, 1997) describe the structure as containing 'no significant intermolecular interactions', although inspection of the data indicate the presence of weak $C—H_{alkyne}\cdots\pi_{aryl}$ contacts.

In conclusion, we have investigated the solid-state structures of two simple diynes and in both cases find that weak $C—H\cdots\pi$ contacts control the molecular packing. In the case of 1,4-bis(biphenyl-4-yl)buta-1,3-diyne, a combination of $C—H_{aryl}\cdots\pi_{alkyne}$ and $C—H_{aryl}\cdots\pi_{aryl}$ interactions operate at the expense of π-stacking interactions.



(*a*)

(*b*)

Figure 4
(*a*) The dimeric motif in the solid-state structure of (II). [Symmetry code (i) $1 - x, 1 - y, -z$.] (*b*) The packing of molecules of (II) into layers. Two dimeric units are shown in the middle of the figure.

Experimental

Compound (I), previously prepared directly (Eisler *et al.*, 2005; Hlavatý *et al.*, 2002), was the product of an unsuccessful attempt to prepare $C_6(CCSi^{i}Pr_3)_6$. C_6I_6 (1.00 g, 1.20 mmol), CuCl (17.8 mg, 0.18 mmol) and $[PdCl_2(PPh_3)_2]$ (126 mg, 0.18 mmol) were added to Et_3N (75 ml) and, after addition of $^{i}Pr_3SiC{\equiv}CH$ (2.13 ml, 9.60 mmol), the mixture was stirred at 333 K for 12 h under argon. The solvent was removed and the residue was extracted with 30% CH_2Cl_2 in hexanes (200 ml). The product was purified by column chromatography (alumina, hexanes) and (I) was collected as a dark-yellow solid (1.74 g, 100%; m.p. 369 K). FAB–MS *m*/*z*: 362 ($[M]^+$), 319 ($[M - {}^{i}Pr]$, base peak); ^1H NMR (400 MHz, $CDCl_3$): δ 1.09 (*s*, TIPS); ^{13}C NMR (125 MHz, $CDCl_3$): δ 90.2 ($C{\equiv}C$), 81.6 ($C{\equiv}C$), 18.6 (CH), 11.3 (CH_3); IR (solid, ν, cm^{-1}): 2943 (*s*), 2866 (*s*), 2050 (*s*), 1458 (*s*), 1383 (*s*), 1365 (*s*), 1230 (*m*), 1011 (*s*), 991 (*s*), 881 (*vs*), 663 (*vs*), 625 (*vs*). Crystals were grown from a solution in CH_2Cl_2. The route to compound (II) was optimized during attempts to prepare 4,4′-bis(biphenyl-4-ylethynyl)biphenyl. 4,4′-Dibromobiphenyl (233 mg, 1.00 mmol), CuCl (14.9 mg, 0.15 mmol) and $[PdCl_2(PPh_3)_2]$ (105 mg, 0.15 mmol) were added to Et_3N (40 ml) and, after addition of 4-ethynylbiphenyl (Foroozesh *et al.*, 1997) (196 mg, 1.10 mmol), the mixture was stirred at 333 K for 18 h under argon. The solvent was removed, the residue was redissolved in hexanes (150 ml) and the mixture was filtered. The product was purified by column chromatography (alumina, hexanes–CH_2Cl_2 1:4) to yield (II) as a yellow solid (195 mg, 100%; m.p. 513 K). FAB–MS *m*/*z*: 354 ($[M]^+$, base peak), 177 ($[M - PhC_6H_4CC]^+$); ^1H NMR (400 MHz, $CDCl_3$): δ 7.60 [*m*, 12H, H(*B*2,*B*3,*A*2)], 7.46 [*m*, 4H, H(*A*3)], 7.38 [*t*, 2H, H(*A*4)]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 141.9 [C(1*A*/1*B*)], 140.0 [C(1*B*/1*A*)], 132.9 [C(3*B*)], 128.9 [C(3*A*)], 127.9 [C(4*A*)], 127.1 [C(2*A*)], 127.0 [C(2*B*)], 120.6 [C(4*B*)], 81.8 [C(ArC≡C)], 74.6 [C(ArC≡C); IR (solid, ν, cm^{-1}): 3059 (*w*), 3036 (*w*), 2133 (*w*), 1599 (*m*), 1481 (*s*), 1448 (*s*), 839 (*vs*), 762 (*vs*), 721 (*vs*), 696 (*vs*). Crystals of (II) were grown from a solution in CH_2Cl_2.

Compound (I)

Crystal data

$C_{22}H_{42}Si_2$
$M_r = 362.75$
Triclinic, $P\overline{1}$
$a = 7.2397$ (4) Å
$b = 7.8151$ (5) Å
$c = 10.9548$ (5) Å
$\alpha = 86.680$ (5)°
$\beta = 80.485$ (4)°
$\gamma = 78.542$ (4)°
$V = 598.90$ (6) Å^3
$Z = 1$
$D_x = 1.006$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.15$ mm^{-1}
$T = 173$ K
Plate, colourless
0.30 × 0.16 × 0.14 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\rm min} = 0.98$, $T_{\rm max} = 0.98$

29988 measured reflections
3478 independent reflections
2019 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.069$
$\theta_{\rm max} = 30.0°$

Refinement

Refinement on F
$R[F > 2\sigma(F)] = 0.050$
$wR(F) = 0.054$
$S = 1.09$
2019 reflections
109 parameters
H-atom parameters constrained
$w = [1 - (F_o - F_c)^2/36\sigma^2(F)/1.89T_0(x) - 0.168T_1(x) + 1.21T_2(x)]$, where T_i are the Chebychev polynomials and $x = F_c/F_{\rm max}$ (Watkin, 1994; Prince, 1982)
$(\Delta/\sigma)_{\rm max} = 0.007$
$\Delta\rho_{\rm max} = 0.93$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.64$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I).

C1—C1$^{\rm i}$	1.373 (3)	C3—Si1	1.873 (2)
C1—C2	1.204 (2)	C6—Si1	1.873 (2)
C2—Si1	1.8432 (18)	C9—Si1	1.876 (2)
C1$^{\rm i}$—C1—C2	179.6 (3)	C1—C2—Si1	177.18 (18)

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Compound (II)

Crystal data

$C_{28}H_{18}$
$M_r = 354.45$
Monoclinic, $P2_1/n$
$a = 6.6723$ (2) Å
$b = 11.0320$ (3) Å
$c = 26.0094$ (7) Å
$\beta = 91.2833$ (14)°
$V = 1914.04$ (9) Å^3

$Z = 4$
$D_x = 1.230$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.07$ mm^{-1}
$T = 173$ K
Block, colourless
0.27 × 0.22 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\rm min} = 0.98$, $T_{\rm max} = 0.99$

21205 measured reflections
5625 independent reflections
2992 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.081$
$\theta_{\rm max} = 30.1°$

Refinement

Refinement on F
$R[F > 2\sigma(F)] = 0.040$
$wR(F) = 0.047$
$S = 0.99$
2992 reflections
253 parameters
H-atom parameters constrained

$w = 1/[\sigma(F) + (0.03P)]$, where $P = [\max(F_o,0) + 2F_c]/3$ (Sheldrick, 1997)
$(\Delta/\sigma)_{\rm max} = 0.004$
$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}

Table 2
Selected geometric parameters (Å, °) for (II).

C6—C7	1.4806 (18)	C15—C16	1.2045 (18)
C10—C13	1.431 (2)	C16—C19	1.433 (2)
C13—C14	1.2041 (18)	C22—C23	1.4799 (18)
C14—C15	1.373 (2)		
C10—C13—C14	177.56 (15)	C14—C15—C16	179.36 (15)
C13—C14—C15	178.75 (15)	C15—C16—C19	177.96 (15)

Table 3
Important intermolecular C—H···π contacts (Å, °) in compound (II).

*Cg*1 is the centroid of ring C1–C6, *Cg*2 of ring C7–C12, *Cg*3 of ring C17–C22 and *Cg*4 of ring C23–C28.

	Distance	Angle
Within the dimer (Fig. 4*a*)		
H181···C14$^{\rm i}$	2.9	
H181···C15$^{\rm i}$	3.0	
H171···C13$^{\rm i}$	3.1	
C24—H241···*Cg*2$^{\rm i}$	2.8	147
C25—H251···*Cg*1$^{\rm i}$	3.5	174
Other interactions within a layer (Fig. 4*b*)		
C3—H31···*Cg*2$^{\rm ii}$	2.9	148
C26—H261···*Cg*1$^{\rm iii}$	3.4	170
Interactions between layers		
C1—H11···*Cg*4$^{\rm iv}$	2.9	151
C8—H81···*Cg*3$^{\rm v}$	3.1	127
C11—H111···*Cg*1$^{\rm vi}$	3.4	136
C20—H201···*Cg*4$^{\rm vii}$	2.9	121

Symmetry codes: (i) $1-x, 1-y, -z$; (ii) $-\frac{3}{2}-x, \frac{1}{2}+y, -\frac{1}{2}-z$; (iii) $-\frac{5}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$; (iv) $-\frac{3}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$; (v) $-x, 1-y, -z$; (vi) $-\frac{1}{2}-x, -\frac{1}{2}+y, -\frac{1}{2}-z$; (vii) $-1+x, y, z$.

All H atoms were treated as riding, with C—H = 1.00 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

For both compounds, data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG3022). Services for accessing these data are described at the back of the journal.

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