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(1*Z*,3*E*,5*E*,7*Z*)-1,2,3,4,5,6,7,8-Octaphenylocta-1,3,5,7-tetraene and 3,4-Diphenylisocoumarin

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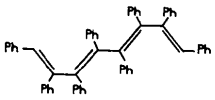
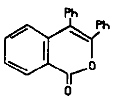
Abstract. (1) 1,2,3,4,5,6,7,8-Octaphenyl-*Z*,*E*,*E*,*Z*-octa-1,3,5,7-tetraene, $C_{56}H_{42}$, $M_r = 714{\cdot}90$, monoclinic, $C2/c$, $a = 23{\cdot}649\,(7)$, $b = 10{\cdot}613\,(3)$, $c = 17{\cdot}015\,(5)$ Å, $\beta = 99{\cdot}17\,(2)^\circ$, $V = 4216\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}126$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}61$ cm^{-1}, $F(000) = 1512$, $T = 296$ K, $R_F = 6{\cdot}37\%$ for 1372 reflections and 205 parameters. The molecule contains crystallographic C_2 symmetry. The central C—C bond is unusually long, 1·644 (8) Å, reflecting the steric crowding of the phenyl rings attached to the 1 and 2′ positions; the H(15)–H(21*a*) contact is particularly short, 2·19 Å. The absence of conjugation is further demonstrated by these distances in the octa-1,3,5,7-tetraene framework: C(1)—C(2) 1·348 (7), C(2)—C(3) 1·487 (6) and C(3)—C(4) 1·346 (7) Å. (2) 3,4-Diphenylisocoumarin, $C_{21}H_{14}O_2$, $M_r = 298{\cdot}37$, monoclinic, $P2_1/n$, $a = 10{\cdot}950\,(4)$, $b = 7{\cdot}913\,(3)$, $c = 17{\cdot}850\,(10)$ Å, $\beta = 93{\cdot}12\,(4)^\circ$, $V = 1544\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}284$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}76$ cm^{-1}, $F(000) = 624$, $T = 296$ K, $R_F = 5{\cdot}71\%$ for 1873 reflections and 240 parameters. The fused six-membered rings are, as expected, nearly coplanar; the dihedral angle is 5·3°. The C—C double bond in the lactone ring is 1·350 (3) Å.

Experimental. (1) Pale yellow crystals from acetone (0·16 × 0·26 × 0·37 mm); Nicolet *R*3*m* diffractometer with graphite monochromator, ω scans; lattice parameters from least-squares fit of 25 reflections ($18 \le 2\theta \le 22^\circ$); $2\theta_{\max} = 42^\circ$, no observed data beyond 42°, ($h = \pm 24$, $k = +11$, $l = +18$), standard reflections $\bar{6}04$, 131, $80\bar{4}$ ($\le 2\%$). 2508 reflections collected, 2252 independent ($R_{\text{int}} = 2{\cdot}5\%$), 1372 observed with $F_o \ge 3\sigma(F_o)$, 880 unobserved reflections, no absorption correction. Direct-methods structure solution (*SOLV*); least-squares refinement on 205 parameters; all non-H atoms anisotropic, H atoms idealized and updated (C—H = 0·96 Å, $U = 1{\cdot}2U$ of attached C), all phenyl rings constrained to rigid, planar hexagons (C—C = 1·395 Å). $R_F = 6{\cdot}37\%$, $wR_F = 7{\cdot}00\%$, $S = 1{\cdot}52$, $w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0{\cdot}001$; $(\Delta/\sigma)_{\max} = 0{\cdot}074$; $\Delta\rho_{\max} = 0{\cdot}20$, $\Delta\rho_{\min} = -0{\cdot}20$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); *SHELXTL* (5.1) computer program (Sheldrick, 1984). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1. Bond lengths and angles are given in Table 3. Phenyl-ring framework torsion angles are given in Table 5. Fig. 1 shows the molecular labeling scheme. A unit-cell packing diagram is shown in Fig. 3.



(1)



(2)

(2) Orange crystals from diethyl ether (0·40 × 0·30 × 0·37 mm); Wyckoff scans; lattice parameters from least-squares fit of 25 reflections ($20 \le 2\theta \le 25^\circ$; $2\theta_{\max} = 50^\circ$, $h = \pm 14$, $k = +10$, $l = +22$, standard reflections $\bar{5}13$, 241, 317 ($\le 1\%$). 3036 reflections collected, 2716 independent ($R_{\text{int}} = 1{\cdot}90\%$), 1873 observed with $F_o \ge 3\sigma(F_o)$, 843 unobserved reflections, no absorption correction. Direct-methods structure solution (*SOLV*), least-squares refinement on 240 parameters; all non-H atoms anisotropic, H atoms located and refined isotropically, phenyl rings constrained to rigid, planar hexagons. $R_F = 5{\cdot}71\%$, $wR_F = 6{\cdot}05\%$, $S = 1{\cdot}29$, $w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0{\cdot}001$; $(\Delta/\sigma)_{\max} = 0{\cdot}044$; $\Delta\rho_{\max} = 0{\cdot}228$, $\Delta\rho_{\min} = -0{\cdot}162$ e Å^{-3}; atomic scattering factors and software as above.

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 2. Bond lengths and angles are given in Table 4.† Interplanar angles are given in Table 6. Fig. 2 shows the molecular structure and numbering scheme. A unit-cell packing diagram is shown in Fig. 4.

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† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52126 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for* (1)

	x	*y*	*z*	*U**
C(1)	349 (2)	−104 (5)	2518 (3)	52 (2)
C(2)	604 (2)	833 (4)	2172 (3)	43 (2)
C(3)	1240 (2)	894 (4)	2284 (3)	43 (2)
C(4)	1530 (2)	1053 (5)	3026 (3)	53 (2)
C(11)	1036 (2)	−1894 (4)	2620 (2)	65 (2)
C(12)	1294	−2951	3012	84 (3)
C(13)	1125	−3364	3717	93 (3)
C(14)	697	−2721	4031	81 (3)
C(15)	439	−1665	3639	63 (2)
C(16)	608	−1251	2934	45 (2)
C(21)	−130 (2)	1652 (3)	1047 (2)	65 (2)
C(22)	−392	2651	596	84 (3)
C(23)	−222	3886	787	88 (3)
C(24)	211	4123	1428	82 (3)
C(25)	474	3124	1878	67 (2)
C(26)	303	1888	1688	48 (2)
C(31)	1243 (1)	157 (3)	888 (2)	56 (2)
C(32)	1481	125	189	77 (3)
C(33)	1984	786	142	86 (3)
C(34)	2250	1478	794	89 (3)
C(35)	2013	1510	1494	61 (2)
C(36)	1509	850	1540	46 (2)
C(41)	2487 (2)	−12 (4)	3124 (2)	72 (3)
C(42)	3067	−78	3444	94 (3)
C(43)	3317	865	3956	115 (4)
C(44)	2986	1875	4148	124 (4)
C(45)	2406	1941	3827	92 (3)
C(46)	2157	998	3315	55 (2)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for* (2)

	x	*y*	*z*	*U**
C(1)	2130 (2)	3238 (3)	7012 (1)	43 (1)
C(2)	1308 (2)	2835 (3)	6449 (1)	40 (1)
C(3)	1738 (2)	2717 (3)	5683 (1)	41 (1)
C(4)	994 (3)	2122 (4)	5069 (2)	51 (1)
C(5)	1446 (3)	2016 (4)	4366 (2)	66 (1)
C(6)	2634 (3)	2521 (4)	4245 (2)	68 (1)
C(7)	3391 (3)	3079 (4)	4837 (2)	62 (1)
C(8)	2950 (2)	3156 (3)	5559 (1)	46 (1)
C(9)	3800 (2)	3608 (4)	6182 (2)	57 (1)
O(10)	3352 (1)	3568 (3)	6886 (1)	56 (1)
O(12)	4872 (2)	3961 (3)	6151 (1)	85 (1)
C(11)	2803 (1)	2683 (3)	8341 (1)	60 (1)
C(12)	2636	2773	9109	74 (1)
C(13)	1609	3584	9368	75 (1)
C(14)	750	4306	8858	66 (1)
C(15)	917	4216	8090	52 (1)
C(16)	1944	3405	7831	46 (1)
C(21)	−290 (1)	1168 (2)	7080 (1)	48 (1)
C(22)	−1509	820	7210	61 (1)
C(23)	−2441	1759	6844	65 (1)
C(24)	−2153	3045	6349	59 (1)
C(25)	−934	3392	6219	48 (1)
C(26)	−2	2455	6585	37 (1)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Related literature. The title compounds were obtained as minor by-products of the reaction of methyl *o*-iodobenzoate and diphenylacetylene in the presence of $Pd(OAc)_2/PPh_3$ catalyst (Wu, Rheingold & Heck 1987).

Table 3. *Bond lengths* (Å) *and angles* (°) *for* (1)

C(1)—C(2)	1·346 (7)	C(1)—C(16)	1·489 (6)
C(1)—C(1*a*)	1·644 (8)	C(2)—C(3)	1·487 (6)
C(2)—C(26)	1·501 (6)	C(3)—C(4)	1·348 (7)
C(3)—C(36)	1·504 (6)	C(4)—C(46)	1·485 (6)
C(2)—C(1)—C(16)	129·3 (4)	C(2)—C(1)—C(1*a*)	120·1 (3)
C(16)—C(1)—C(1*a*)	110·6 (3)	C(1)—C(2)—C(3)	119·4 (4)
C(1)—C(2)—C(26)	125·8 (4)	C(3)—C(2)—C(26)	114·7 (4)
C(2)—C(3)—C(4)	118·5 (5)	C(2)—C(3)—C(36)	116·5 (4)
C(4)—C(3)—C(36)	124·9 (4)	C(3)—C(4)—C(46)	129·5 (5)
C(1)—C(16)—C(11)	119·1 (2)	C(1)—C(16)—C(15)	120·9 (2)
C(2)—C(26)—C(21)	121·3 (2)	C(2)—C(26)—C(25)	118·7 (2)
C(3)—C(36)—C(31)	119·2 (2)	C(3)—C(36)—C(35)	120·8 (2)
C(4)—C(46)—C(41)	121·3 (2)	C(4)—C(46)—C(45)	118·6 (2)

Table 4. *Bond lengths* (Å) *and angles* (°) *for* (2)

C(1)—C(2)	1·350 (3)	C(4)—C(5)	1·376 (4)
C(1)—O(10)	1·394 (3)	C(5)—C(6)	1·389 (4)
C(1)—C(16)	1·493 (3)	C(6)—C(7)	1·380 (4)
C(2)—C(3)	1·473 (3)	C(7)—C(8)	1·402 (4)
C(2)—C(26)	1·499 (3)	C(8)—C(9)	1·456 (4)
C(3)—C(4)	1·410 (4)	C(9)—O(10)	1·373 (3)
C(3)—C(8)	1·401 (3)	C(9)—O(12)	1·211 (3)
C(2)—C(1)—O(10)	122·1 (2)	C(6)—C(7)—C(8)	119·7 (3)
C(2)—C(1)—C(16)	129·0 (2)	C(3)—C(8)—C(7)	120·7 (2)
O(10)—C(1)—C(16)	108·9 (2)	C(3)—C(8)—C(9)	120·8 (2)
C(1)—C(2)—C(3)	118·2 (2)	C(7)—C(8)—C(9)	118·4 (2)
C(1)—C(2)—C(26)	122·1 (2)	C(8)—C(9)—O(10)	116·5 (2)
C(3)—C(2)—C(26)	119·6 (2)	C(8)—C(9)—O(12)	127·1 (3)
C(2)—C(3)—C(4)	122·9 (2)	O(10)—C(9)—O(12)	116·4 (2)
C(2)—C(3)—C(8)	118·9 (2)	C(1)—O(10)—C(9)	123·1 (2)
C(4)—C(3)—C(8)	118·2 (2)	C(1)—C(16)—C(11)	118·7 (1)
C(3)—C(4)—C(5)	120·5 (3)	C(1)—C(16)—C(15)	121·3 (1)
C(4)—C(5)—C(6)	120·7 (3)	C(2)—C(26)—C(21)	120·0 (1)
C(5)—C(6)—C(7)	120·1 (3)	C(2)—C(26)—C(25)	120·0 (1)

Table 5. *Torsion angles* (°), *astom deviations from plane I* (Å) *and interplanar angles* (°), *for* (1)

C(2)—C(1)—C(1*a*)—C(2*a*)	−62·7	C(26)—C(2)—C(3)—C(4)	115·8
C(1*a*)—C(1)—C(2)—C(3)	173·5	C(46)—C(4)—C(3)—C(2)	172·1
C(1)—C(2)—C(3)—C(4)	−62·4	C(16)—C(1)—C(2)—C(26)	172·9
C(16)—C(1)—C(2)—C(3)	−9·1	C(16)—C(1)—C(1*a*)—C(16*a*)	−58·3
C(26)—C(2)—C(1)—C(1*a*)	−4·4	C(26)—C(2)—C(3)—C(36)	−60·6
C(16)—C(1)—C(1*a*)—C(2*a*)	119·5	C(36)—C(3)—C(4)—C(46)	−11·8
C(36)—C(3)—C(2)—C(1)	121·2		

Plane I [C(1), C(2), C(3), C(4)]

C(1)	−0·115	C(3)	−0·245
C(2)	0·247	C(4)	0·113
Plane II	[C(11)–C(16)]	Plane IV	[C(31)–C(36)]
Plane III	[C(21)–C(26)]	Plane V	[C(41)–C(46)]
I–II	65·4	I–IV	140·5
I–III	125·7	I–V	88·5

Table 6. *Interplanar angles* (°) *for* (2)

Plane I	[C(1), C(2), C(3), C(8), C(9), O(10)]		
Plane II	[C(3), C(4), C(5), C(6), C(7), C(8)]		
Plane III	[C(11)–C(16)]	Plane IV	[C(21)–C(26)]
I–II	5·3	I–IV	57·1
I–III	44·9		

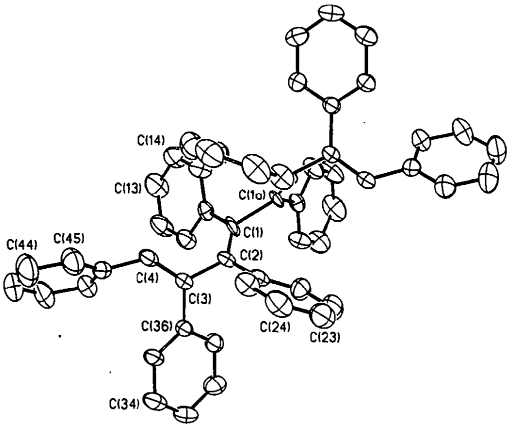

Fig. 1. Molecular structure and labeling scheme for (1) with 40% probability thermal ellipsoids. H atoms have been deleted for clarity.

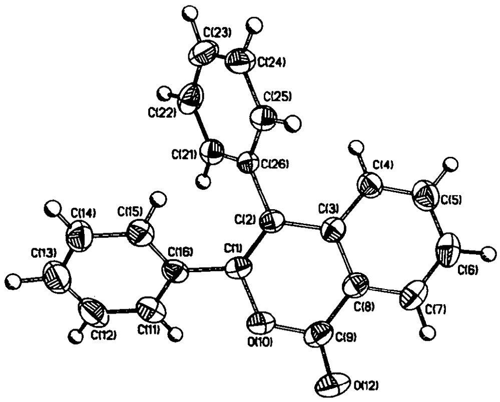

Fig. 2. Molecular structure and labeling scheme for (2) with 40% probability thermal ellipsoids. H atoms are shown with arbitrary radii.

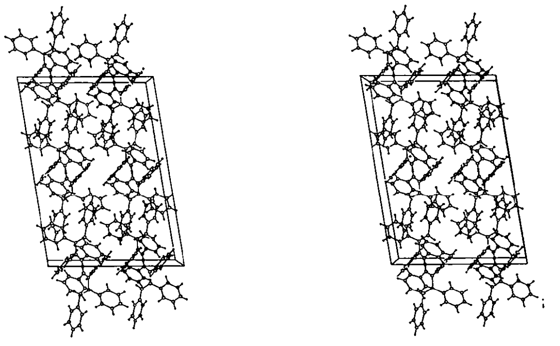

Fig. 3. Unit-cell packing diagram for (1) as viewed down the *b* axis with *a* vertical.

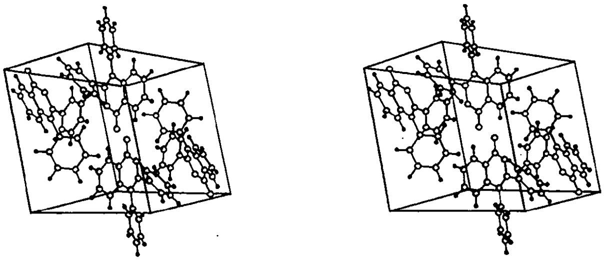

Fig. 4. Unit-cell packing diagram for (2); the *a* axis is nearly vertical and the *c* axis nearly horizontal.

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Structure of 4,5-Diphenyl-1-triphenylmethoxy-1,2,3-triazole

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Abstract. $C_{33}H_{25}N_3O$, $M_r = 479{\cdot}58$, monoclinic, $P2_1/n$, $a = 10{\cdot}599\,(5)$, $b = 14{\cdot}286\,(4)$, $c = 16{\cdot}175\,(3)$ Å, $\beta = 94{\cdot}31\,(4)°$, $V = 2442{\cdot}4\,(7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}304$ g cm^{-3}, λ(Mo $K\overline{\alpha}$) = 0·71073 Å, $\mu = 0{\cdot}742$ cm^{-1}, $F(000) = 1008$, $T = 105$ K, $R = 0{\cdot}034$, $wR = 0{\cdot}044$ for 3052 unique observed reflections. The triazole ring is planar. There are no unusual bond distances or angles.

Experimental. The title compound was prepared by the reaction of silver phenylcyanomethane nitronate with trityl chloride in toluene at 278 K (Boyer, Manimaran & Ramakrishnan, 1987) and recrystallized from benzene yielding colorless crystals. A tabular crystal with approximate dimensions 0·30 × 0·30 × 0·40 mm was mounted on an Enraf–Nonius CAD-4 diffractometer with graphite crystal monochromator

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