Spek, A. L. (1988). *J. Appl. Cryst.* **20**, 264–269.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Spek, A. L. (1993). *HELENA. Program for Data Reduction.* University of Utrecht, The Netherlands.
Spek, A. L. (1994). *Am. Crystallogr. Assoc. Abstr.* **22**, 66.

Acta Cryst. (1996). C**52**, 177–180

Extremely Long C—C Bonds in Strained 1,1,2,2-Tetraphenylcyclobutaarenes: 3,8-Dichloro-1,1,2,2-tetraphenylcyclobuta[*b*]naphthalene, $C_{36}H_{24}Cl_2$, and 3,6,9,10-Tetrachloro-4,5-dimethyl-1,1,2,2,7,7,8,8-octaphenyldicyclobuta[*b*,*h*]phenanthrene Toluene Solvate, $C_{68}H_{46}Cl_4.1.5C_7H_8$

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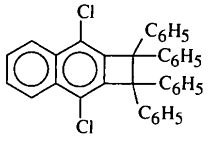
Abstract

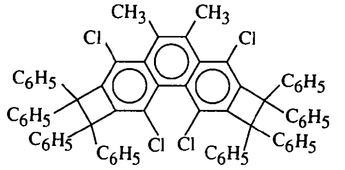
Cyclobutaarenes are highly strained compounds which exhibit unusual geometric features. Introduction of diphenyl substituents on the peripheral C atoms of the cyclobutene ring adds stereoelectronic effects and causes further distortion of the ring structure. The interatomic distances observed for the peripheral C—C bonds in the cyclobutene moiety of the two compounds are in the range 1.71–1.72 Å, among the longest of C_{sp^3}—C_{sp^3} bonds previously reported for similar compounds. The core structure of the phenanthrodicyclobutene derivative is severely twisted due to non-bonding interactions between the chlorine substituents.

Comment

A series of low-temperature crystallographic studies of arenes containing fused strained cyclobutene and cyclopropene rings, characterizing the effect of the angular strain on the electronic structure of benzene, have recently been published (Boese & Bläser, 1988; Bläser *et al.*, 1989; Boese *et al.*, 1994). For the benzocyclobutene species, the interatomic dimensions of the four-membered rings were typically found to be near C_{sp^2}—C_{sp^2} = 1.34, C_{sp^2}—C_{sp^3} = 1.52 and C_{sp^3}—C_{sp^3} = 1.58 Å. More recently, a new synthetic route to benzocyclobutene derivatives has been reported, leading to the preparation of a series of highly strained aromatic compounds which consist of substituted benzocyclobutene, naphthocyclobutene and anthracyclobutene fragments (Toda, Tanaka, Sano & Isozaki, 1994). The title compounds were subjected to a detailed structural analysis (at room temperature) in order to establish the molecular structure of the new materials and to characterize the effect of the severe intramolecular overcrowding on the covalent parameters of the cyclobutene ring.



(I)



(II)

The cyclobutene structure observed in this study is characterized by C_{sp^3}—C_{sp^3} bond distances in the range 1.710 (5)–1.724 (5) Å and C_{sp^3}—C_{sp^2} bond distances varying from 1.526 (4) to 1.538 (4) Å. These values have not been corrected for thermal motion effects and do not account for the presence of 'bent bonds' in the four-membered ring (Boese *et al.*, 1994). In the planar naphthocyclobutene compound, the cyclobutene fusion bond has a normal length of 1.409 (4) Å, while the adjacent bonds in the benzene ring are considerably shortened [1.351 (5) and 1.360 (4) Å]. On the other hand, in the twisted phenanthrodicyclobutene derivative, the fusion-annelated bonds and the adjacent aryl bonds have lengths which are closer in magnitude, within 1.365 (6)–1.368 (5) and 1.369 (5)–1.374 (5) Å, respectively. The intramolecular twist in the latter is best characterized by the C(Cl)—C—C—C(Cl) torsion angle along the concave surface of the molecule of 37.6 (5)°, which represents a severe distortion of the aryl core from planarity. Moreover, while on each side of the molecule the fused cyclobutene and benzene rings are almost coplanar, the dihedral angle formed between the mean planes of the two eight-membered ring pairs is 34.6 (1)°. This allows accommodation of the two inner Cl substituents at a non-bonding distance of 3.099 (2) Å. The stretched C—C outer bonds of the cyclobutene moiety facilitate cleavage of the strained ring at this site by photoirradiation.

The significant stretching of the C_{sp^3}—C_{sp^3} bonds in the cyclobutene fragment is most probably effected by a combination of steric and electronic effects. Some elongation should, undoubtedly, be attributed to steric hindrance between the diphenyl substituents on the peripheral bond. This has been demonstrated by crystal structure analyses of two 1,1,2,2-tetraphenylethane-1,2-diol derivatives, in which the central C_{sp^3}—C_{sp^3} bond distances are 1.59 Å, as compared with the standard

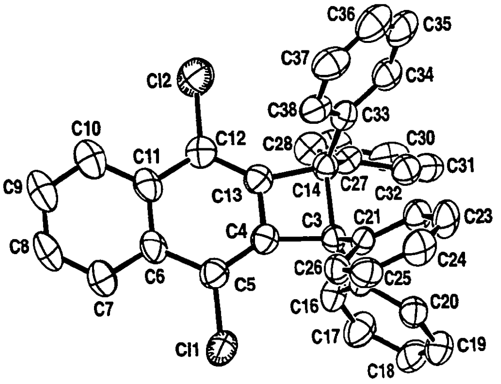


Fig. 1. Molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

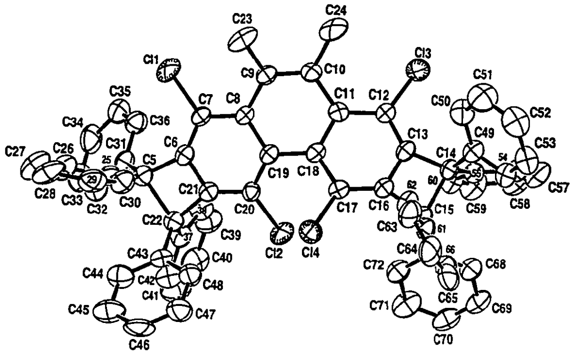


Fig. 2. Molecular structure of (II) showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

C—C bond length of 1.54 Å (Toda, Tanaka, Wang & Lee, 1986; Bourne, Nassimbeni & Toda, 1991). Angular strain in the cyclobutaarene fragment also contributes at least 0.04 Å to the elongation of the saturated C—C bond (see above; Boese *et al.*, 1994). The unique stereochemistry of these species thus accounts for a C—C stretching of *ca* 0.1 Å. Electronic effects (*e.g.* hyperconjugation and through-bond coupling) could be responsible for the additional lengthening, as reported for the bis(anthracene-9,10-dimethylene) photodimer (Zhou, Liu & Allinger, 1993). Within this context, the authors are extremely grateful to one of the referees of this paper for confirming the unusual length of the C—C bond in question by semi-empirical methods. His MNDO and PM3 calculations on the title compounds indicated consistently that the cyclobutene C_{sp^3}—C_{sp^3} bond lengths cluster within the range 1.670–1.684 Å, higher than the computational results obtained for the bis(anthracene-9,10-dimethylene) photodimer (Battersby, Gantzel, Baldridge & Siegel, 1995; Zhou *et al.*, 1993). Incidentally, the first structure determination of the latter compound reported a C—C distance of 1.77 Å within the bridging four-membered ring fragment (Ehrenberg, 1966). However, more recent experimental, as well as theoretical, evaluations indicated the correct value of this bond length to be near 1.64–1.66 Å (Battersby *et al.*, 1995; Harada, Ogawa & Tomoda, 1995; Zhou *et al.*, 1993). Consequently, it appears that the tetraphenylcyclobutaarene species contains the longest C—C bond (near 1.7 Å) reliably determined thus far for ground-state structures of organic compounds. Further studies are still required, however, to fully account for the experimental observations.

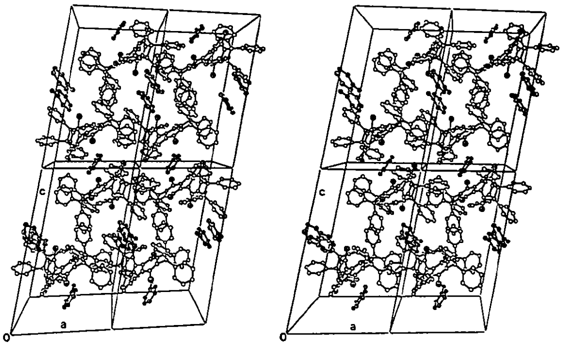


Fig. 3. Crystal structure of the toluene solvate (II) viewed down the *b* axis (four unit cells are shown). The toluene molecules are accommodated in channels formed between the phenanthrodicyclobutene species, which extend along the $[0,y,\frac{1}{2}]$ and $[\frac{1}{2},y,0]$ axes; those located near, and disordered about, the inversion centres at $\frac{1}{2}$,0,0 are shown only in one orientation. The Cl substituents are indicated by crossed circles.

Experimental

The title compounds were crystallized from toluene. 3,8-Dichloro-1,1,2,2-tetraphenylcyclobuta[*b*]naphthalene, (I), crystallized as a pure material, while 3,6,9,10-tetrachloro-4,5-dimethyl-1,1,2,2,7,7,8,8-octaphenyldicyclobuta[*b*,*h*]phenanthrene, (II), formed co-crystals with 1.5 equivalents of toluene.

Compound (I)

Crystal data

$C_{36}H_{24}Cl_2$
M_r = 527.45
Monoclinic
$P2_1/n$
a = 8.988 (6) Å
b = 16.861 (4) Å
c = 17.737 (2) Å
β = 96.48 (3)°
V = 2670.8 (19) Å^3
Z = 4
D_x = 1.312 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 7.6–10.3°
μ = 0.267 mm^{-1}
T = 298 (2) K
Prism
0.40 × 0.25 × 0.20 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none

$R_{\rm int}$ = 0.0209
$\theta_{\rm max}$ = 24.97°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 21$

4463 measured reflections
4192 independent reflections
3122 observed reflections
$[I > 2\sigma(I)]$

3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0480$
$wR(F^2) = 0.1560$
$S = 0.924$
4192 reflections
343 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.002$
$\Delta\rho_{\max} = 0.214$ e Å^{-3}
$\Delta\rho_{\min} = -0.243$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for* (I)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl1	0.12610 (10)	0.52109 (5)	0.37383 (5)	0.0619 (3)
Cl2	0.73664 (10)	0.34948 (5)	0.33397 (5)	0.0678 (3)
C3	0.3185 (3)	0.4953 (2)	0.21177 (14)	0.0357 (6)
C4	0.3485 (3)	0.4699 (2)	0.2954 (2)	0.0396 (7)
C5	0.2918 (3)	0.4712 (2)	0.3627 (2)	0.0433 (7)
C6	0.3689 (4)	0.4293 (2)	0.4262 (2)	0.0470 (8)
C7	0.3096 (4)	0.4222 (2)	0.4959 (2)	0.0605 (9)
C8	0.3809 (5)	0.3780 (2)	0.5540 (2)	0.0715 (11)
C9	0.5153 (5)	0.3401 (2)	0.5457 (2)	0.0712 (11)
C10	0.5781 (4)	0.3464 (2)	0.4792 (2)	0.0608 (9)
C11	0.5072 (4)	0.3904 (2)	0.4170 (2)	0.0491 (8)
C12	0.5678 (3)	0.3955 (2)	0.3465 (2)	0.0459 (7)
C13	0.4888 (3)	0.4351 (2)	0.2883 (2)	0.0405 (7)
C14	0.4915 (3)	0.4543 (2)	0.20439 (15)	0.0365 (6)
C15	0.3114 (3)	0.5852 (2)	0.20046 (14)	0.0360 (6)
C16	0.4038 (4)	0.6345 (2)	0.2488 (2)	0.0485 (8)
C17	0.4100 (4)	0.7146 (2)	0.2364 (2)	0.0591 (9)
C18	0.3227 (4)	0.7488 (2)	0.1766 (2)	0.0604 (9)
C19	0.2268 (4)	0.7024 (2)	0.1306 (2)	0.0574 (9)
C20	0.2203 (3)	0.6216 (2)	0.1423 (2)	0.0474 (7)
C21	0.1919 (3)	0.4527 (2)	0.16445 (15)	0.0357 (6)
C22	0.1940 (3)	0.4443 (2)	0.0865 (2)	0.0465 (7)
C23	0.0769 (4)	0.4086 (2)	0.0425 (2)	0.0552 (8)
C24	−0.0443 (3)	0.3796 (2)	0.0747 (2)	0.0563 (9)
C25	−0.0487 (3)	0.3874 (2)	0.1509 (2)	0.0510 (8)
C26	0.0690 (3)	0.4233 (2)	0.1958 (2)	0.0419 (7)
C27	0.6057 (3)	0.5154 (2)	0.1870 (2)	0.0383 (6)
C28	0.7280 (3)	0.5346 (2)	0.2385 (2)	0.0503 (8)
C29	0.8263 (4)	0.5942 (2)	0.2230 (2)	0.0614 (9)
C30	0.8050 (4)	0.6352 (2)	0.1563 (2)	0.0628 (9)
C31	0.6855 (4)	0.6173 (2)	0.1044 (2)	0.0561 (8)
C32	0.5869 (3)	0.5582 (2)	0.1191 (2)	0.0462 (7)
C33	0.4908 (3)	0.3783 (2)	0.1573 (2)	0.0386 (7)
C34	0.5640 (3)	0.3716 (2)	0.0928 (2)	0.0489 (8)
C35	0.5600 (4)	0.3009 (2)	0.0521 (2)	0.0621 (9)
C36	0.4849 (4)	0.2358 (2)	0.0751 (2)	0.0659 (10)
C37	0.4124 (4)	0.2416 (2)	0.1386 (2)	0.0608 (9)
C38	0.4155 (3)	0.3114 (2)	0.1794 (2)	0.0491 (7)

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

Cl1—C5	1.740 (3)	C6—C11	1.432 (5)
Cl2—C12	1.740 (3)	C7—C8	1.371 (5)
C3—C4	1.538 (4)	C8—C9	1.389 (6)
C3—C14	1.720 (4)	C9—C10	1.369 (5)
C4—C5	1.351 (4)	C10—C11	1.420 (4)
C4—C13	1.409 (4)	C11—C12	1.422 (4)
C5—C6	1.439 (4)	C12—C13	1.360 (4)
C6—C7	1.406 (4)	C13—C14	1.526 (4)
C4—C3—C14	84.2 (2)	C4—C13—C14	96.4 (2)
C13—C4—C3	95.2 (2)	C13—C14—C3	84.1 (2)

Compound (II)

Crystal data

$C_{68}H_{46}Cl_4.1.5C_7H_8$
$M_r = 1143.13$
Triclinic
$P\bar{1}$
$a = 11.415$ (4) Å
$b = 14.316$ (2) Å
$c = 18.785$ (4) Å
$\alpha = 85.29$ (2)°
$\beta = 79.11$ (3)°
$\gamma = 82.63$ (3)°
$V = 2984.3$ (13) Å^3
$Z = 2$
$D_x = 1.272$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 8.3$–$11.2°$
$\mu = 0.242$ mm^{-1}
$T = 298$ (2) K
Prism
0.40 × 0.30 × 0.30 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
7631 measured reflections
7630 independent reflections
6281 observed reflections
$[I > 2\sigma(I)]$

$\theta_{\max} = 22.97°$
$h = -12 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = 0 \rightarrow 20$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0610$
$wR(F^2) = 0.1953$
$S = 1.317$
7630 reflections
711 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.668$
$\Delta\rho_{\max} = 0.687$ e Å^{-3}
$\Delta\rho_{\min} = -0.417$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for* (II)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl1	0.42822 (11)	−0.32897 (7)	0.15863 (7)	0.0584 (3)
Cl2	0.46839 (9)	0.06188 (7)	0.26638 (6)	0.0485 (3)
Cl3	0.14569 (10)	0.14666 (8)	0.00850 (5)	0.0519 (3)
Cl4	0.19810 (10)	0.07813 (7)	0.33528 (5)	0.0490 (3)
C5	0.5884 (4)	−0.2660 (3)	0.2811 (2)	0.0385 (9)
C6	0.4900 (3)	−0.2109 (3)	0.2439 (2)	0.0354 (9)
C7	0.4125 (3)	−0.2215 (3)	0.1983 (2)	0.0377 (9)
C8	0.3304 (3)	−0.1420 (3)	0.1815 (2)	0.0349 (9)
C9	0.2493 (3)	−0.1431 (3)	0.1301 (2)	0.0385 (9)
C10	0.2112 (3)	−0.0602 (3)	0.0954 (2)	0.0363 (9)
C11	0.2158 (3)	0.0298 (3)	0.1248 (2)	0.0336 (9)
C12	0.1646 (3)	0.1195 (3)	0.0976 (2)	0.0351 (9)
C13	0.1328 (3)	0.1909 (2)	0.1445 (2)	0.0346 (9)
C14	0.0740 (3)	0.2931 (3)	0.1471 (2)	0.0370 (9)
C15	0.0967 (3)	0.2805 (3)	0.2358 (2)	0.0350 (9)
C16	0.1504 (3)	0.1809 (3)	0.2148 (2)	0.0341 (9)

C17	0.2056 (3)	0.0982 (3)	0.2420 (2)	0.0349 (9)
C18	0.2516 (3)	0.0259 (2)	0.1936 (2)	0.0326 (8)
C19	0.3340 (3)	−0.0548 (2)	0.2120 (2)	0.0329 (9)
C20	0.4252 (3)	−0.0461 (3)	0.2521 (2)	0.0351 (9)
C21	0.4938 (3)	−0.1263 (3)	0.2715 (2)	0.0347 (9)
C22	0.5921 (3)	−0.1598 (3)	0.3157 (2)	0.0377 (9)
C23	0.1998 (5)	−0.2332 (3)	0.1192 (3)	0.0638 (14)
C24	0.1463 (5)	−0.0640 (3)	0.0328 (3)	0.0600 (13)
C25	0.5372 (4)	−0.3436 (3)	0.3345 (2)	0.0411 (10)
C26	0.5985 (4)	−0.4325 (3)	0.3444 (3)	0.0538 (12)
C27	0.5449 (5)	−0.5016 (3)	0.3901 (3)	0.0678 (14)
C28	0.4317 (6)	−0.4844 (4)	0.4282 (3)	0.071 (2)
C29	0.3692 (5)	−0.3968 (4)	0.4201 (3)	0.0650 (14)
C30	0.4195 (4)	−0.3272 (3)	0.3731 (2)	0.0514 (11)
C31	0.7080 (4)	−0.2985 (3)	0.2348 (2)	0.0410 (10)
C32	0.8100 (4)	−0.3176 (3)	0.2655 (2)	0.0517 (11)
C33	0.9199 (4)	−0.3443 (3)	0.2248 (3)	0.0617 (13)
C34	0.9321 (4)	−0.3536 (3)	0.1524 (3)	0.0664 (14)
C35	0.8326 (5)	−0.3358 (3)	0.1203 (3)	0.0638 (14)
C36	0.7211 (4)	−0.3078 (3)	0.1609 (2)	0.0494 (11)
C37	0.7064 (3)	−0.1137 (3)	0.2879 (2)	0.0401 (10)
C38	0.7391 (4)	−0.0896 (3)	0.2143 (2)	0.0494 (11)
C39	0.8425 (5)	−0.0499 (4)	0.1863 (3)	0.0657 (13)
C40	0.9175 (5)	−0.0313 (4)	0.2318 (3)	0.0723 (15)
C41	0.8862 (5)	−0.0520 (4)	0.3046 (3)	0.0723 (15)
C42	0.7815 (4)	−0.0927 (3)	0.3327 (2)	0.0536 (11)
C43	0.5517 (4)	−0.1592 (3)	0.3976 (2)	0.0412 (10)
C44	0.6117 (4)	−0.2187 (3)	0.4439 (2)	0.0555 (12)
C45	0.5788 (5)	−0.2166 (4)	0.5183 (3)	0.0700 (15)
C46	0.4845 (5)	−0.1532 (4)	0.5474 (3)	0.072 (2)
C47	0.4228 (5)	−0.0944 (4)	0.5029 (3)	0.0674 (14)
C48	0.4559 (4)	−0.0974 (3)	0.4280 (2)	0.0516 (11)
C49	−0.0594 (3)	0.3085 (3)	0.1441 (2)	0.0403 (10)
C50	−0.1163 (4)	0.2415 (3)	0.1196 (3)	0.0587 (12)
C51	−0.2391 (5)	0.2537 (4)	0.1220 (3)	0.079 (2)
C52	−0.3077 (4)	0.3358 (4)	0.1479 (3)	0.073 (2)
C53	−0.2522 (4)	0.4024 (4)	0.1723 (3)	0.0593 (13)
C54	−0.1298 (4)	0.3885 (3)	0.1710 (2)	0.0471 (11)
C55	0.1511 (3)	0.3601 (3)	0.0976 (2)	0.0389 (9)
C56	0.1057 (4)	0.4440 (3)	0.0649 (2)	0.0480 (11)
C57	0.1808 (5)	0.5027 (3)	0.0221 (3)	0.0584 (12)
C58	0.3015 (5)	0.4805 (4)	0.0104 (3)	0.0691 (14)
C59	0.3499 (4)	0.3980 (4)	0.0428 (3)	0.0689 (14)
C60	0.2761 (4)	0.3388 (3)	0.0851 (2)	0.0527 (11)
C61	−0.0155 (3)	0.2814 (3)	0.2936 (2)	0.0389 (9)
C62	−0.0872 (4)	0.2091 (3)	0.3009 (3)	0.0558 (12)
C63	−0.1912 (5)	0.2083 (4)	0.3525 (3)	0.0693 (15)
C64	−0.2254 (5)	0.2798 (4)	0.3986 (3)	0.075 (2)
C65	−0.1562 (5)	0.3529 (4)	0.3919 (3)	0.075 (2)
C66	−0.0520 (4)	0.3536 (3)	0.3409 (3)	0.0564 (12)
C67	0.1863 (3)	0.3428 (3)	0.2492 (2)	0.0385 (9)
C68	0.1638 (4)	0.4408 (3)	0.2388 (2)	0.0463 (10)
C69	0.2464 (5)	0.4983 (3)	0.2480 (3)	0.0588 (12)
C70	0.3527 (5)	0.4605 (4)	0.2690 (3)	0.0652 (14)
C71	0.3758 (4)	0.3652 (4)	0.2801 (3)	0.0631 (13)
C72	0.2932 (4)	0.3062 (3)	0.2708 (2)	0.0491 (11)
C73†	0.4490 (7)	0.0353 (5)	0.9488 (5)	0.120 (11)
C74†	0.4431 (6)	−0.0522 (6)	0.9234 (3)	0.076 (3)
C75†	0.4884 (5)	−0.1341 (4)	0.9588 (4)	0.108 (2)
C76†	0.5396 (5)	−0.1286 (4)	1.0196 (4)	0.103 (5)
C77†	0.5455 (7)	−0.0412 (6)	1.0450 (4)	0.062 (6)
C78†	0.5002 (8)	0.0408 (4)	1.0096 (5)	0.076 (3)
C79†	0.8742 (17)	0.5956 (10)	0.4997 (10)	0.356 (11)
C80†	0.9368 (7)	0.6705 (6)	0.4706 (5)	0.337 (10)
C81†	0.9322 (7)	0.7539 (7)	0.5045 (4)	0.165 (4)
C82†	1.0072 (9)	0.8213 (5)	0.4738 (5)	0.247 (7)
C83†	1.0869 (8)	0.8053 (6)	0.4090 (6)	0.277 (8)
C84†	1.0915 (7)	0.7218 (7)	0.3751 (4)	0.225 (6)
C85†	1.0164 (8)	0.6545 (5)	0.4059 (5)	0.161 (4)

† Atoms C73–C78 represent the toluene molecule located near, and disordered about, the inversion centre at $\frac{1}{2}$,0,1. In the disordered model, atoms C73, C74, C76, C77 and C78 were assigned an occupancy factor of 0.5, and atom C75 an occupancy factor of 1. Atoms C79–C85 represent another toluene moiety located at a general position and partly disordered. Due to the apparent disorder of the toluene molecules, they were included in the refinement with constrained geometries.

Table 4. *Selected geometric parameters (Å, °) for* (II)

Cl1—C7	1.738 (4)	C10—C24	1.513 (6)
Cl2—C20	1.737 (4)	C11—C18	1.422 (5)
Cl3—C12	1.735 (4)	C11—C12	1.437 (5)
Cl4—C17	1.741 (4)	C12—C13	1.371 (5)
C5—C6	1.533 (5)	C13—C16	1.368 (5)
C5—C22	1.710 (5)	C13—C14	1.531 (5)
C6—C21	1.365 (5)	C14—C15	1.724 (5)
C6—C7	1.372 (5)	C15—C16	1.529 (5)
C7—C8	1.433 (5)	C16—C17	1.374 (5)
C8—C19	1.424 (5)	C17—C18	1.417 (5)
C8—C9	1.460 (5)	C18—C19	1.455 (5)
C9—C10	1.368 (5)	C19—C20	1.419 (5)
C9—C23	1.517 (6)	C20—C21	1.369 (5)
C10—C11	1.452 (5)	C21—C22	1.526 (5)
C6—C5—C22	83.6 (3)	C16—C15—C14	83.2 (3)
C21—C6—C5	96.1 (3)	C13—C16—C15	96.9 (3)
C16—C13—C14	96.4 (3)	C6—C21—C22	96.8 (3)
C13—C14—C15	83.5 (3)	C21—C22—C5	83.5 (3)
C8—C9—C10—C11	20.7 (6)	C17—C18—C19—C20	37.6 (6)
C23—C9—C10—C24	15.5 (6)		

For both compounds, data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CADINT* (locally developed software); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*93 and *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1061). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Battersby, T. R., Gantzel, P., Baldridge, K. K. & Siegel, J. S. (1995). *Tetrahedron Lett.* **36**, 845–848.

Bläser, D., Boese, R., Brett, W. A., Rademacher, P., Schwager, H, Stanger, M. & Vollhardt, K. P. C. (1989). *Angew. Chem. Int. Ed. Engl.* **28**, 206–208.

Boese, R. & Bläser, D. (1988). *Angew. Chem. Int. Ed. Engl.* **27**, 304–305.

Boese, R., Bläser, D., Billups, W. E., Haley, M. M., Maulitz, A. H., Mohler, D. L. & Vollhardt, K. P. C. (1994). *Angew. Chem. Int. Ed. Engl.* **33**, 313–317.

Bourne, S. A., Nassimbeni, L. R. & Toda, F. (1991). *J. Chem. Soc. Perkin Trans* 2, pp. 1335–1341.

Ehrenberg, M. (1966). *Acta Cryst.* **20**, 182–186.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Harada, J., Ogawa, K. & Tomoda, S. (1995). *Chem. Lett.* pp. 751–752.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak-Ridge National Laboratory, Tennessee, USA.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Toda, F., Tanaka, K., Sano, I. & Isozaki, T. (1994). *Angew. Chem. Int. Ed. Engl.* **33**, 1757–1758.

Toda, F., Tanaka, K., Wang, Y. & Lee, G.-H. (1986). *Chem. Lett.* pp. 109–112.

Zhou, Z., Liu, R. & Allinger, N. L. (1993). *J. Am. Chem. Soc.* **115**, 7525–7526.