Fig. 1. General view (*SHELXTL-Plus* graphic) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries.
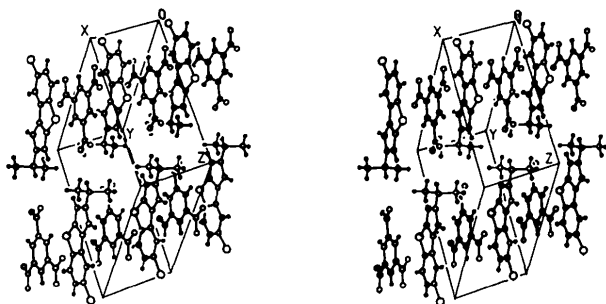

Fig. 2. Stereoscopic view (*SHELXTL-Plus* graphic) of the unit cell. Dashed lines correspond to short intermolecular contacts.

Vol. IV). The programs used were *PARST* (Nardelli, 1983), *SHELX76* (Sheldrick, 1976), *SHELXTL-Plus* (Sheldrick, 1987), *PCK83* (Williams, 1984), *PLATON* (Spek, 1982), and *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit-cell contents is in Fig. 2. Positional parameters and the equivalent values of the anisotropic dis-

placement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles, least-squares planes, dihedral angles, possible hydrogen bonds and short intermolecular contacts are given in Table 2.

Related literature. For charge-transfer complexes see Parini (1962), Thesing, Melchior & Schäfer (1964), Foster (1976), Bleidelis, Shvets & Freimanis (1976) and Jelitto (1989). For preparation and spectroscopic data of tricyclic 14π-hetarenes see Dyker (1988) and Götte (1990).

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The Diels–Alder Diadduct of Hexachlorocyclopentadiene with Cyclooctatetraene

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Abstract. *endo,endo*-1,5,6,7,8,14,15,16,17,17,18,18-Dodecachlorohexacyclo[12.2.1.1^{5,8}.0^{2,13}.0^{3,12}.0^{4,9}]octa-deca-6,10,15-triene, $C_{18}H_8Cl_{12}$, $M_r = 649.7$, ortho-rhombic, $P2_12_12_1$, $a = 13.028$ (2), $b = 13.0214$ (12), $c = 14.252$ (2) Å, $V = 2417.7$ (5) Å^3, $Z = 4$, $D_x = 1.785$ g cm^{-3}; λ(Cu $K\alpha$) = 1.54184 Å, $\mu =$

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130·2 cm^{-1}, $F(000) = 1280$, $T = 297$ K, $R = 0·058$ for 2514 observations with $I > 3\sigma(I)$ (of 2899 unique data). The cyclobutane ring is almost planar with maximum deviations $\pm 0·030$ (7) Å and endocyclic torsion angles having identical magnitudes 4·4 (4)° and alternating in sign. The cyclohexene ring is also quite flattened, with average deviation 0·052 Å from the best plane, and maximum deviation 0·098 (6) Å. The dihedral angle formed by these two planes is 66·2 (3)°. The C=C bonds have lengths 1·31 (1)– 1·35 (1) Å, while C(sp^2)—Cl distances range from 1·679 (7) to 1·699 (7) Å and C(sp^3)—Cl distances range from 1·732 (7) to 1·774 (7) Å.

Experimental. The title compound was prepared by slowly heating cyclooctatetraene with excess hexachlorocyclopentadiene (Fray & Smith, 1969) to 508 K for 5 min. Crystals grown by slow cooling of ethanol, m.p. 445–448 K, were suitable; a clear colorless crystal of dimensions 0·30 × 0·33 × 0·42 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from the setting angles of 25 reflections having $30 > \theta > 25°$. The ω–2θ scans were designed for $I = 25\sigma(I)$, subject to maximum scan time = 90 s, scan rates varied from 0·66–3·30° min^{-1}. A full sphere of data having $2 < \theta < 22°$, $-6 \leq h \leq 6$, $-6 \leq k \leq 6$, $-6 \leq l \leq 6$ and an octant having $22 < \theta < 75°$, $0 \leq h \leq 16$, $0 \leq k \leq 16$, $0 \leq l \leq 17$ were measured and corrected for background, Lorentz, polarization and absorption. Absorption corrections were based on ψ scans, with minimum relative transmission coefficient 62·54%. Three standard reflections (400, 020, 002) measured every 10 000 s exhibited only random fluctuations, so no correction for decay was needed. 3776 reflections measured, 2514 observed $[I > 3\sigma(I)]$. The space group was determined by Laue symmetry mmm and systematic absences $h00$ with h odd, $0k0$ with k odd, and $00l$ with l odd. Redundant data were averaged; $R_{int} = 0·050$. Merging data in Laue group $4/m$ yielded $R_{int} = 0·053$. The structure was solved by direct methods, refined by full-matrix least squares based on F, using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0·02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) and anomalous coefficients from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1). Heavy-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF and were refined with isotropic thermal parameters. Final $R = 0·058$ for 2514 observed data (0·076 for all 2899 data), $wR = 0·073$ and $S = 3·643$ for 304 variables. Maximum shift was 0·14σ in the final cycle,

Table 1. *Coordinates and equivalent isotropic thermal parameters for the title compound*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i{}^*a_j{}^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Cl(1)	0·2387 (2)	0·5225 (2)	0·6263 (2)	6·03 (5)
Cl(2)	0·2791 (2)	0·6554 (2)	0·4353 (2)	5·53 (5)
Cl(3)	0·4643 (2)	0·8349 (2)	0·4773 (2)	6·19 (5)
Cl(4)	0·5286 (2)	0·8230 (2)	0·6996 (2)	7·04 (5)
Cl(5)	0·3963 (2)	0·6161 (2)	0·8043 (1)	7·26 (6)
Cl(6)	0·2729 (2)	0·7651 (2)	0·7123 (2)	5·75 (4)
Cl(7)	0·7951 (2)	0·2431 (2)	0·4475 (2)	6·23 (5)
Cl(8)	0·8228 (2)	0·4722 (2)	0·3529 (2)	5·39 (4)
Cl(9)	0·5845 (2)	0·5559 (2)	0·2820 (1)	5·17 (4)
Cl(10)	0·4144 (1)	0·3813 (2)	0·3385 (1)	4·52 (4)
Cl(11)	0·5369 (2)	0·1764 (1)	0·4483 (2)	4·98 (4)
Cl(12)	0·6192 (2)	0·2342 (2)	0·2703 (1)	5·24 (4)
C1	0·4520 (6)	0·5365 (5)	0·6143 (4)	3·3 (1)
C2	0·5343 (6)	0·6195 (6)	0·6382 (4)	3·9 (1)
C3	0·5946 (6)	0·5916 (5)	0·5494 (5)	3·5 (1)
C4	0·6936 (5)	0·5420 (6)	0·5773 (5)	3·9 (1)
C5	0·7143 (6)	0·4427 (6)	0·5713 (5)	3·8 (1)
C6	0·6438 (6)	0·3644 (5)	0·5281 (5)	3·3 (1)
C7	0·5353 (5)	0·4031 (4)	0·4984 (4)	2·6 (1)
C8	0·5083 (5)	0·5149 (5)	0·5208 (4)	3·0 (1)
C9	0·3499 (5)	0·5969 (5)	0·6152 (5)	3·3 (1)
C10	0·3519 (5)	0·6709 (5)	0·5325 (5)	3·4 (1)
C11	0·4218 (6)	0·7411 (5)	0·5504 (5)	3·7 (1)
C12	0·4678 (6)	0·7179 (5)	0·6441 (5)	3·9 (1)
C13	0·3700 (7)	0·6747 (6)	0·6945 (5)	4·3 (1)
C14	0·6901 (5)	0·3237 (5)	0·4348 (5)	3·3 (1)
C15	0·7078 (5)	0·4179 (5)	0·3722 (4)	3·2 (1)
C16	0·6143 (5)	0·4511 (5)	0·3458 (4)	3·2 (1)
C17	0·5350 (5)	0·3809 (5)	0·3903 (4)	2·9 (1)
C18	0·5945 (6)	0·2782 (5)	0·3847 (5)	3·5 (1)

max. residual density 0·52, min. $-0·51$ e Å^{-3}. The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a structural diagram, Fig. 2 is a perspective drawing showing the atom numbering for the title molecule, and Fig. 3 shows the unit cell. Bond distances, angles, and selected torsion angles are presented in Table 2.* Refinement of the mirror-image structure under identical circumstances yielded $R = 0·064$, $wR = 0·081$, $S = 4·009$; thus the absolute configuration is determined.

Related literature. Single-bond distances analogous to C1—C9 and C2—C12 in 1,6,7,8,9,14,15,16-octachloro-17,17,18,18-tetramethoxypentacyclo[12.2.1.-$1^{6,9}.0^{2,13}.0^{5,10}$]octadeca-7,15-diene 1·561 (3) and 1·555 (4) Å (Garcia, Fronczek & McLaughlin, 1991a); 17,17,18,18-tetramethoxypentacyclo-[12.2.1.1$^{6,9}.0^{2,13}.0^{5,10}$]octadeca-7,15-diene 1·566 (1) and 1·564 (1) Å (Garcia, Fronczek & McLaughlin, 1991a); *endo,endo,anti*-1,6,7,8,9,14,15,16-octachloropentacyclo[12.2.1.1$^{6,9}.0^{2,13}.0^{5,10}$]octadeca-7,15-diene 1·553 (4) and 1·560 (3) Å (Garcia, Fronczek & McLaughlin, 1991b); and (5,4:7,8-e,e')-[bis(phenan-

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thro(9,10b)-1,4-diphenylbicyclo[2.2.1]heptan-7-one]-bicyclo[4.2.0]oct-2-ene benzene methanol solvate 1·556 (19) and 1·531 (19) Å (Yasuda, Harano & Kanematsu, 1981) are similar to those in the title compound, 1·54 (1) and 1·55 (1) Å, respectively. The bond angles in the four-membered ring of the title
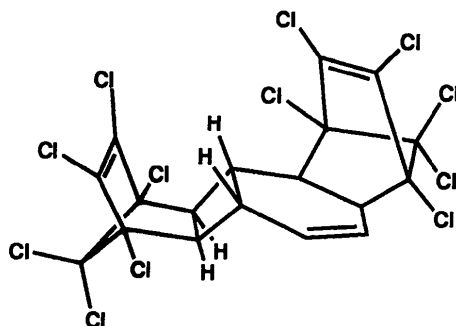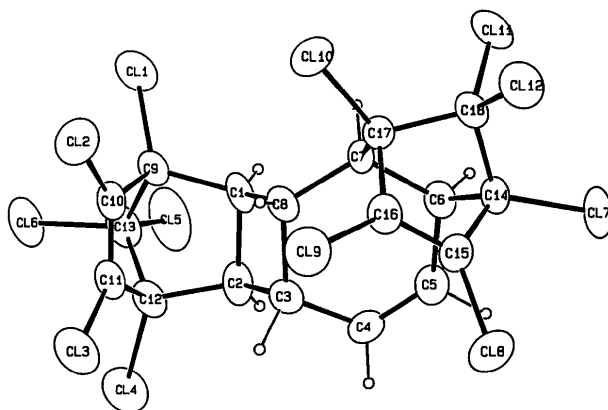

Fig. 1. Title molecule.

Fig. 2. *ORTEP* drawing (Johnson, 1965) of the title molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius.
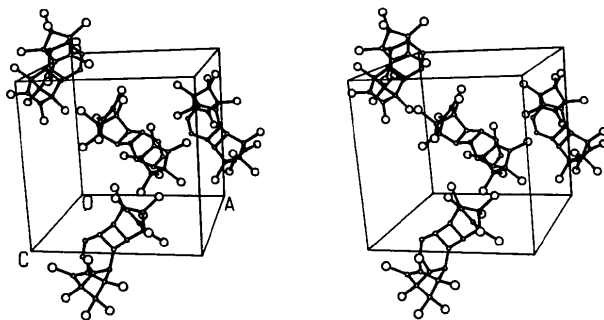

Fig. 3. Stereoview of the unit cell.

Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

Cl(1)—C9	1·751 (7)	C3—C8	1·56 (1)
Cl(2)—C10	1·691 (7)	C4—C5	1·32 (1)
Cl(3)—C11	1·699 (7)	C5—C6	1·50 (1)
Cl(4)—C12	1·768 (7)	C6—C7	1·560 (9)
Cl(5)—C13	1·774 (7)	C6—C14	1·554 (9)
Cl(6)—C13	1·747 (8)	C7—C8	1·530 (8)
Cl(7)—C14	1·732 (7)	C7—C17	1·568 (8)
Cl(8)—C15	1·679 (7)	C9—C10	1·523 (9)
Cl(9)—C16	1·685 (7)	C9—C13	1·54 (1)
Cl(10)—C17	1·736 (7)	C10—C11	1·31 (1)
Cl(11)—C18	1·772 (7)	C11—C12	1·49 (1)
Cl(12)—C18	1·758 (7)	C12—C13	1·57 (1)
C1—C2	1·56 (1)	C14—C15	1·534 (9)
C1—C8	1·547 (9)	C14—C18	1·55 (1)
C1—C9	1·54 (1)	C15—C16	1·35 (1)
C2—C3	1·53 (1)	C16—C17	1·518 (9)
C2—C12	1·55 (1)	C17—C18	1·547 (9)
C3—C4	1·50 (1)		

C2—C1—C8	89·3 (5)	C10—C9—C13	98·6 (5)
C2—C1—C9	103·7 (5)	Cl(2)—C10—C9	123·3 (5)
C8—C1—C9	120·5 (5)	Cl(2)—C10—C11	129·1 (6)
C1—C2—C3	90·5 (5)	C9—C10—C11	107·5 (6)
C1—C2—C12	101·6 (6)	Cl(3)—C11—C10	127·3 (6)
C3—C2—C12	121·8 (6)	Cl(3)—C11—C12	124·2 (5)
C2—C3—C4	108·9 (6)	C10—C11—C12	108·1 (6)
C2—C3—C8	89·9 (5)	Cl(4)—C12—C2	114·4 (5)
C4—C3—C8	114·5 (6)	Cl(4)—C12—C11	115·0 (5)
C3—C4—C5	125·4 (7)	Cl(4)—C12—C13	115·9 (5)
C4—C5—C6	124·4 (7)	C2—C12—C11	110·1 (5)
C5—C6—C7	116·5 (5)	C2—C12—C13	100·5 (5)
C5—C6—C14	110·2 (6)	C11—C12—C13	99·0 (6)
C7—C6—C14	103·3 (5)	Cl(5)—C13—Cl(6)	107·5 (4)
C6—C7—C8	117·3 (5)	Cl(5)—C13—C9	113·4 (5)
C6—C7—C17	102·1 (5)	Cl(5)—C13—C12	113·7 (6)
C8—C7—C17	112·3 (5)	Cl(6)—C13—C9	115·2 (5)
C1—C8—C3	90·0 (5)	Cl(6)—C13—C12	114·4 (5)
C1—C8—C7	117·5 (5)	C9—C13—C12	92·2 (5)
C3—C8—C7	119·9 (6)	Cl(7)—C14—C6	115·1 (5)
Cl(1)—C9—C1	115·5 (4)	Cl(7)—C14—C15	115·2 (5)
Cl(1)—C9—C10	115·7 (5)	Cl(7)—C14—C18	116·8 (5)
Cl(1)—C9—C13	116·0 (5)	C6—C14—C15	106·4 (5)
Cl—C9—C10	107·5 (5)	C6—C14—C18	102·2 (5)
Cl—C9—C13	101·2 (6)	C15—C14—C18	99·1 (5)
Cl(8)—C15—C14	124·5 (5)	C7—C17—C16	107·3 (5)
Cl(8)—C15—C16	128·8 (5)	C7—C17—C18	102·0 (5)
C14—C15—C16	106·5 (6)	C16—C17—C18	99·1 (5)
Cl(9)—C16—C15	128·3 (6)	Cl(11)—C18—Cl(12)	107·9 (4)
Cl(9)—C16—C17	123·8 (5)	Cl(11)—C18—C14	112·9 (5)
C15—C16—C17	107·8 (6)	Cl(11)—C18—C17	114·1 (5)
Cl(10)—C17—C7	114·8 (4)	Cl(12)—C18—C14	113·8 (5)
Cl(10)—C17—C16	115·9 (4)	Cl(12)—C18—C17	114·9 (5)
Cl(10)—C17—C18	115·7 (5)	C14—C18—C17	92·8 (5)

C1—C2—C3—C8	4·4 (5)	C2—C3—C8—C1	−4·4 (5)
C8—C1—C2—C3	−4·4 (5)	C2—C1—C8—C3	4·3 (5)
C4—C3—C8—C7	−15·7 (8)	C3—C4—C5—C6	5·0 (12)
C4—C5—C6—C7	−6·5 (10)	C5—C6—C7—C8	−3·3 (8)
C6—C7—C8—C3	14·2 (8)	C8—C3—C4—C5	6·3 (10)

compound are all equal, ranging from 89·3 (5) to 90·5 (5)°, similar to the (5,4:7,8-*e,e'*)-[bis(phenanthro(9,10b)-1,4-diphenylbicyclo[2.2.1]heptan-7-one]-bicyclo[4.2.0]oct-2-ene benzene methanol solvate (Yasuda, Harano & Kanematsu, 1981) bond-angle variation [from 88·5 (10) to 91·5 (10)°]. Bond angles between the cyclobutane and the cyclohexene rings, C1—C8—C7 and C2—C3—C4, in the title compound, 117·5 (5) and 108·9 (6)°, respectively are similar to those of (5,4:7,8-*e,e'*)-[bis(phenanthro(9,10b)-1,4-diphenylbicyclo[2.2.1]heptan-7-one]-bicyclo[4.2.0]oct-2-ene benzene methanol solvate (Yasuda, Harano & Kanematsu, 1981), 117·3 (11) and 113·5 (12)°, respectively.

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

Structure of bis(diphenylboron-dimethylglyoximato)nickel(II). Corrigendum.

By RICHARD E. MARSH, *The Beckman Institute, California Institute of Technology, Pasadena, California* 91125, *USA**

Abstract

The structure of $Ni(C_{32}H_{32}B_2N_4O_4)$ has been described as triclinic, space group $P\bar{1}$ [Xu, Lei, Cheng, Xu, Chen & Tang (1990). *Acta Cryst.* C46, 1447–1450]. It is properly described as monoclinic, space group $C2/c$. Revised coordinates are given.

The triclinic cell dimensions were reported as $a = 8.385$ (2), $b = 14.068$ (3), $c = 14.234$ (3) Å, $\alpha = 75.20$ (2), $\beta = 72.74$ (2), $\gamma = 72.53°$, $Z = 2$. The lattice vectors $[1\bar{2}0]$, $[\bar{1}00]$ and $[01\bar{1}]$ define a C-centered cell with $a' = 26.838$, $b' = 8.385$, $c' = 17.269$ Å, $\alpha' = 90.00$, $\beta' = 129.29$, $\gamma' = 89.87°$, $Z = 4$. The corresponding coordinate transformations are: $x' = \frac{1}{2}(-y-z) + 0.25$, $y' = -x - \frac{1}{2}(y+z) + 0.25$, $z' = -z$. After averaging over pairs of symmetry-related atoms, the $C2/c$ coordinates in Table 1 result. Included in Table 1 are the shifts in the coordinates necessary to attain the higher symmetry; none of these shifts is greater than the corresponding e.s.d. Since the shifts are so small, there are no significant changes in the bond lengths or angles. Xu, Lei, Cheng, Xu, Chen & Tang (1990) noted that 'the molecule has approximate $C2$ symmetry' and also noted 'the approximate systematic absence $k + l = 2n + 1$ for $0kl$ reflections in the triclinic system.' These absences are characteristic of the c-glide plane in $C2/c$, and the molecule lies on a crystallographic twofold axis.

The revised angle γ' appears to differ from the expected value of 90° by several e.s.d.'s. However, the atomic coordinates leave no doubt that the monoclinic description is

* Contribution No. 8224.

Table 1. *Coordinates* ($\times 10^4$), *space group* $C2/c$

Numbers in square brackets are shifts in the $P\bar{1}$ coordinates necessary to attain the symmetry of $C2/c$.

	x'	y'	z'
Ni	0 [·3]	−2126	−2500 [·1]
O(1)	1260 [0]	−1928 [0]	−1929 [1]
O(2)	−348 [0]	−1998 [1]	−1244 [0]
N(1)	897 [1]	−1951 [2]	−1635 [1]
N(2)	128 [0]	−1968 [1]	−1313 [0]
C(1)	1166 [0]	−1616 [0]	−716 [0]
C(2)	710 [1]	−1629 [0]	−516 [0]
C(11)	1869 [1]	−1268 [4]	55 [0]
C(21)	891 [3]	−1290 [3]	478 [4]
C(101)	1439 [1]	−2772 [1]	−3140 [2]
C(102)	1981 [1]	−1779 [1]	−2638 [2]
C(103)	2382 [1]	−1762 [0]	−2888 [0]
C(104)	2251 [0]	−2713 [1]	−3642 [0]
C(105)	1715 [2]	−3690 [1]	−4160 [2]
C(106)	1322 [0]	−3723 [0]	−3900 [0]
C(107)	805 [2]	−4662 [2]	−2766 [1]
C(108)	1249 [0]	−5510 [3]	−1886 [0]
C(109)	1160 [2]	−7110 [0]	−1802 [2]
C(110)	620 [0]	−7908 [2]	−2594 [2]
C(111)	168 [2]	−7088 [1]	−3461 [3]
C(112)	260 [1]	−5498 [4]	−3544 [0]
B	958 [2]	−2877 [0]	−2888 [3]

correct, and it seems clear that the accuracies in the triclinic cell dimensions are appreciably worse than represented by the reported e.s.d.'s. It is the usual procedure – and, presumably, the one followed by Xu *et al.* (1990) – to report the precision values obtained during an automated centering routine on a computer-controlled diffractometer; such values need have little relationship to the true accuracies, and it is misleading to quote them as values of 'estimated standard deviations'. Many factors – such as absorption, mis-centering of the crystal, misalign-