atoms results in differences in their hydrogen-bonding capabilities (see below). Surprisingly, both non-coordinated O atoms are situated on the same side of the coordination plane, opposite the elongated Ni—O(4) bond. According to the precision of our data, C(1)—O(5) and C(1)—O(6) bond distances are equivalent although C—O(uncoordinated) bond lengths are expected to be shorter, as found in nickel acetate tetrahydrate and copper hippurate (Brown & Trefonas, 1973). Bonding distances and angles within both carboxylate groups and phenyl rings are similar to values reported in related structures (*International Tables for X-ray Crystallography*, 1968). The complete molecule is roughly centrosymmetric.

The Cl atoms are well isolated in this structure and nearest non-bonded contacts are between Cl(2) and a neighboring-ring atom $C(2^i)$ at 3·5 Å and $O(5^i)$ at 3·8 Å [equivalent positions for neighboring molecules are (i) = $\bar{x}$, $1 - y$, $\bar{z}$; (ii) = $-1 + x, y, z$; (iii) = $\bar{x},\bar{y},\bar{z}$; (iv) = $1 + x,y,z$]. This lack of any direct interaction between Ni and Cl atoms would account for the higher degree of hydration in these *meta*-substituted halobenzoato complexes as a means of satisfying the coordination requirements of the Ni atom.

All water molecules participate in the following hydrogen-bonding network (as illustrated in Fig. 3). O(1) and O(3) are four-coordinate with single bonds to Ni and three hydrogen bonds (donor, donor, acceptor) to $[O(6),O(7^i),O(4^i)]$ and $[O(8),O(6^{ii}),O(2^{iii})]$ respectively; O(2) and O(4) are three-coordinate and hydrogen bonded (donor, donor) to $[O(3^{iii}),O(8^{iv})]$ and $[O(1^i),O(6^{ii})]$ respectively. Average bond parameters in the network are: bonded O—H distance, 0·80 (5) Å; hydrogen-bonded O···O contact, 2·7 (1) Å; and O···H—O angle, 162 (5)°. The eight hydrogen bonds are not equitably distributed since carboxylate atoms O(5) through O(8) participate in zero, three, one and two hydrogen bonds respectively, as shown in Fig. 3.

This represents a significant departure from the hydrogen-bonding scheme in the comparable nickel acetate tetrahydrate, and is due to the unequal disposition of the carboxylate groups (see above). The entire network operates in the *ab* plane and results in marked cleavage properties for the macroscopic crystal.

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Dichloro(1-2:5-6-η-cyclooctatetraene)palladium(II)

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Abstract. $C_8H_8Cl_2Pd$, monoclinic, $P2_1/n$, $a = 11·608$ (2), $b = 9·637$ (2), $c = 7·844$ (2) Å, $\beta = 98·45$ (5)° at 22°C, $Z = 4$, $D_m = 2·13$ (2), $D_x = 2·15$ g cm^{-3}, FW 281·46, $\Sigma|\Delta F|/\Sigma F_o = 0·0189$ from 1508 reflections. The complex is a monomer with the two Cl atoms and the centers of the 1–2 and 5–6 double bonds of the cyclooctatetraene ligand lying in a plane with approximately 90° bond angles. Pd—C = 2·203 (2), 2·211 (3),
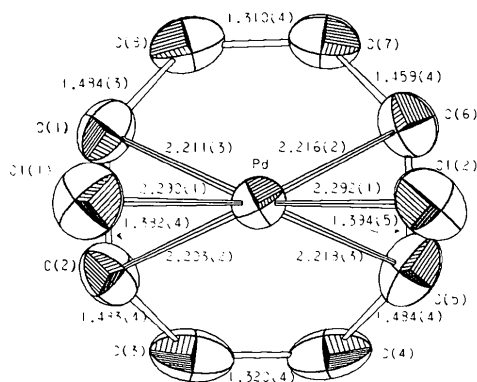
2·218 (3), 2·216 (2) Å; Pd—Cl = 2·290 (1), 2·292 (1) Å.

Introduction. Intensity data (1430 observed present, 78 observed with intensity less than three standard deviations above the background) were collected using a four-circle diffractometer, with θ–2θ step scans, and graphite-monochromatized Mo $K\alpha$ radiation ($\lambda K\alpha_1$ = 0·70926 Å) to 2θ = 50°. The 2θ step size ranged from 0·14 to 0·18°; backgrounds were measured at $\pm 1·60°$ from the peak center. [For further details see Baenziger, Foster, Howells, Howells, Valk & Burton (1977).] Data

were corrected for Lorentz and polarization factors and for absorption [analytical method of de Meulenaer & Tompa (1965) as used in the program *ABSORB* described by Templeton & Templeton (1973) with local adaptation by F. J. Hollander]. The linear absorption coefficient is 26·30 cm^{-1}. The crystal bounding planes and their distances from the crystal center are: $\pm$(110), 0·0154 cm; $\pm$(10$\bar{1}$), 0·0052 cm; $\pm$(3$\bar{3}$1), 0·0318 cm; corrections ranged from 1·300 to 2·19. The red crystals were one of two isomeric crystalline products from the addition of cyclooctatetraene to a benzene solution of bis(benzonitrile)palladium chloride.

The cell dimensions were determined from least-squares extrapolation from back-reflection Weissenberg diagrams at 295 and at 100 (5) K. The cell dimensions found are: at 100 K, a = 11·460 (4), b = 9·534 (15), c = 7·832 (11) Å, β = 98·62 (8)°; at 295 K, a = 11·608 (2), b = 9·637 (2), c = 7·844 (2) Å, β = 98·45 (5)°. The systematic absences, $h0l$ for $h + l$ odd and $0k0$ for k odd, indicated the unique space group $P2_1/n$. The density of the crystals was determined by flotation in a solvent mixture.

A set of diffraction data were also collected at 100 K using a stationary-counter, ω-scan technique with a manually operated proportional counter system (Ni-filtered Cu $K\alpha$ radiation) mounted on an equi-inclination Weissenberg camera. Full-matrix least-squares refinement using these data gave $(R_s/\Sigma wF_o^2)^{1/2}$ = 0·094 for a refinement model in which the H atoms were not refined and the anisotropic temperature factors for three C atoms had to be constrained to keep them at physically reasonable values. The room-temperature data set was collected with modern diffractometer techniques to remove possible systematic errors which appeared to be present in the older low-temperature measurements.

Table 1. *Atom parameters* (*for* Pd, Cl $\times 10^5$, *for* C $\times 10^4$, *for* H $\times 10^3$)

Estimated standard deviations in the last significant digits are in parentheses.

	x	y	z
Pd	34459 (2)	70518 (2)	40406 (2)
Cl(1)	18670 (7)	64232 (9)	53060 (9)
Cl(2)	39403 (8)	87368 (7)	60792 (8)
C(1)	2694 (2)	5981 (3)	1641 (3)
C(2)	3405 (3)	5060 (3)	2655 (3)
C(3)	4623 (3)	4762 (3)	2411 (3)
C(4)	5449 (2)	5712 (3)	2687 (3)
C(5)	5204 (3)	7137 (3)	3255 (4)
C(6)	4485 (3)	8072 (3)	2243 (4)
C(7)	3896 (3)	7744 (3)	518 (3)
C(8)	3091 (3)	6786 (3)	222 (3)
H(1)	192	610	186
H(2)	310	459	356
H(3)	482	386	205
H(4)	622	548	252
H(5)	556	743	437
H(6)	437	897	270
H(7)	411	825	957
H(8)	275	660	907

Table 2. *Interatomic distances* (Å), *bond angles* (°) *and least-squares planes*

Pd—Cl(1)	2·290 (1)	C(1)—C(2)	1·382 (4)	C(1)—C(2)—C(3)	123·4 (2)
Pd—Cl(2)	2·292 (1)	C(2)—C(3)	1·483 (4)	C(2)—C(3)—C(4)	121·9 (2)
Pd—C(1)	2·211 (3)	C(3)—C(4)	1·320 (4)	C(3)—C(4)—C(5)	121·6 (2)
Pd—C(2)	2·203 (2)	C(4)—C(5)	1·484 (4)	C(4)—C(5)—C(6)	123·5 (3)
Pd—C(5)	2·218 (3)	C(5)—C(6)	1·394 (5)	C(5)—C(6)—C(7)	123·2 (2)
Pd—C(6)	2·216 (2)	C(6)—C(7)	1·459 (4)	C(6)—C(7)—C(8)	122·8 (2)
		C(7)—C(8)	1·310 (4)	C(7)—C(8)—C(1)	121·9 (3)
Cl(1)—Pd—Cl(2)	91·11 (3)	C(8)—C(1)	1·484 (3)	C(8)—C(1)—C(2)	122·8 (2)
C(1,2)—Pd—Cl(1)	89·0 (1)				
C(5,6)—Pd—C(1,2)	88·7 (2)				
C(5,6)—Pd—Cl(2)	91·2 (1)				

Planes as designated by $m_1x + m_2y + m_3z = d$, with orthogonal axes chosen along **a**, **b** and **a** × **b**

	m_1	m_2	m_3	d		Deviations (Å) ($\pm 0·003$ Å)		
C(1), C(2), C(5), C(6)	0·4992	−0·3130	−0·8080	−1·368	−0·002	0·002	0·003	−0·003
C(3), C(4), C(7), C(8)	0·5027	−0·3040	−0·8092	−0·344	−0·007	0·007	−0·006	0·007
C(6), C(7), C(8), C(1)	0·7169	−0·6914	−0·0894	−1·990	0·003	−0·006	0·007	−0·003
C(2), C(3), C(4), C(5)	−0·0671	0·2899	−0·9547	−0·798	0·000	0·001	−0·001	0·000
Pd, Cl(1), Cl(2), C(1,2), C(5,6)	−0·4878	0·6590	−0·5725	0·960	0·00	0·004 (1)	−0·001 (1)	−0·014 (3)
								0·060 (3)

Fig. 1. Plan view of the title compound looking down the bisector of the Cl—Pd—Cl angle.

Discussion. The cyclooctatetraene (COT) molecule is very symmetrically coordinated to the Pd atom, the only significant distortion of the cyclooctatetraene molecule being the lengthening of the double bonds which are coordinated to the Pd atom. The bond lengths of the π-coordinated double bonds [1·382 (4), 1·394 (5) Å] are comparable with those found in norbornadienepalladium chloride [1·366 (10) Å] (Baenziger, Richards & Doyle, 1965) and cyclooctatetraene-copper(I) chloride [1·392 (13) Å] (Baenziger, Richards & Doyle, 1964), and can be contrasted with the shorter bond lengths of the uncoordinated double bonds [1·320 (4), 1·310 (4) Å] in the complex and 1·334 (1) Å in the free molecule (Bastiansen, Hedberg & Hedberg, 1957). The single-bond distances in the complex average 1·478 (12) Å, in comparison with 1·462 (1) Å for free COT, 1·48 (1) Å for COT.CuCl and 1·46 (3) Å in COT.AgNO$_3$ (Lipscomb & Mathews, 1959). The ring angles average 122·6 (7)° in the complex, as compared with 126·3 (2)° in free COT.

The structure was solved by Patterson and electron density methods. H atoms were located by difference electron density maps but their parameters were not refined. The function minimized was $R_s = \Sigma w(|F_o| - |F_c|)^2$; the weighted R factor defined by the expression $(R_s/\Sigma wF_o^2)^{1/2}$ was 0·0292. Scattering factors for Pd, C and Cl were from *International Tables for X-ray Crystallography* (1962), those for H from Stewart, Davidson & Simpson (1965). Anomalous-dispersion terms were included for Pd and Cl. Positional parameters are given in Table 1,* bond distances and angles in Table 2. A plan view of the molecule is given in Fig. 1, looking down the bisector of the Cl—Pd—Cl angle.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33233 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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Potassium Ethylenediaminetetraacetatomanganate(III) Dihydrate

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Abstract. KMn(C$_{10}$H$_{12}$N$_2$O$_8$).2H$_2$O, orthorhombic, $P2_12_12_1$, $a = 6·576$ (4), $b = 10·038$ (8), $c = 23·14$ (2) Å, $M_r = 418·3$, $V = 1527·5$ Å^3, $Z = 4$, $D_m = 1·81$, $D_x = 1·82$ g cm^{-3}, μ(Cu $K\alpha$, $\lambda = 1·5418$ Å) = 103·7 cm^{-1}.