

## Structure of (Chloranilato)bis(tri-*m*-tolylphosphine)palladium(II)

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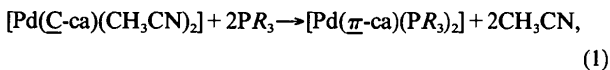
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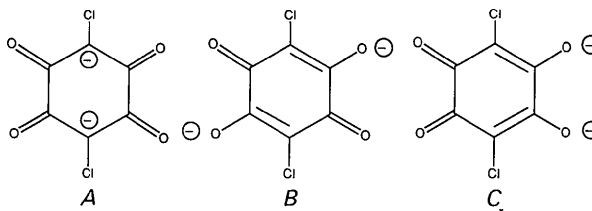
**Abstract.** (Chloranilato)bis(tri-*m*-tolylphosphine)-palladium(II),  $[\text{Pd}(\text{C}_6\text{Cl}_2\text{O}_4)\{\text{P}(\text{C}_7\text{H}_7)_3\}_2]$  (chloranilic acid = 2,5-dichloro-3,6-dihydroxy-*p*-benzoquinone):  $M_r = 922.12$ , monoclinic,  $P2_1/c$ ,  $a = 15.231(5)$ ,  $b = 10.214(5)$ ,  $c = 26.952(7)$  Å,  $\beta = 94.600(20)^\circ$ ,  $V = 4179.40$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.465$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.70930$  Å,  $\mu = 0.68$  mm<sup>-1</sup>,  $F(000) = 1888$ ,  $T = 77$  K, final  $R = 0.039$ , 3916 observed reflections. The title compound exhibits a distorted square-planar  $\text{P}_2\text{O}_2$  ligand field about  $\text{Pd}^{\text{II}}$  characterized by a P(1)—Pd—P(2) angle of  $99.55(6)^\circ$  and a P(1), Pd, O(1), C(1) torsion angle of  $-34.1(3)^\circ$ . Coordinated chloranilate C—C and C—O bond lengths reveal a predominant *o*-quinone resonance form, in contrast to the delocalized *p*-quinone structure characteristic of bridging  $\text{C}_6\text{Cl}_2\text{O}_4^{2-}$  in transition metal complexes.

**Introduction.** Linkage isomerism in chloranilate dianion complexes ( $\text{ca}^{2-}$ ; 3,6-dichloro-2,5-dioxo-1,4-benzenediolate) is of interest as a probe of the steric and electronic factors that influence the interconversion of  $sp^2$ - and  $sp^3$ -hybridized C atoms in the first coordination sphere of palladium(II) (Jeong & Holwerda, 1988*a,b*, 1989*a,b*). Thus, the bis(carbanion) (*A*), *p*-quinone (*B*) and *o*-quinone (*C*) chloranilate resonance forms suggest at least three possibilities for linkage isomerism in  $\text{Pd}^{\text{II}}\text{-ca}^{2-}$  compounds (Jeong & Holwerda, 1988*a*). Tertiary phosphines induce linkage isomerization from  $\text{C-ca}^{2-}$  to  $\pi\text{-ca}^{2-}$  in reactions with  $[\text{Pd}(\text{C-ca})(\text{CH}_3\text{CN})_2]$  [equation (1); Jeong & Holwerda, 1988*a*, 1989*a*],



where  $\text{C-ca}^{2-}$  and  $\pi\text{-ca}^{2-}$  represent chloranilate ligated through C—Cl C atoms (resonance form *A*) and as a quinone (resonance form *B* or *C*). Although

NMR, infrared and electronic spectra of the  $[\text{Pd}(\pi\text{-ca})(\text{PR}_3)_2]$  products are consistent with the presence of chloranilate in its *p*-quinonoid resonance form (Jeong & Holwerda, 1988*a*, 1989*a*), solution-phase measurements cannot distinguish unambiguously between  $\text{ca}^{2-}$  chelated through O atoms or diene C=C electron pairs. For this reason, we report here the crystal structure of (chloranilato)bis-(tri-*m*-tolylphosphine)palladium(II),  $[\text{Pd}(\text{ca})\{\text{P}(\text{Ph-}m\text{-CH}_3)_3\}_2]$ , a representative  $\pi\text{-ca}^{2-}$  complex.



**Experimental.** The title compound was prepared from the reaction of  $[\text{Pd}(\text{ca})(\text{CH}_3\text{CN})_2]$  with  $\text{P}(\text{Ph-}m\text{-CH}_3)_3$ , as described elsewhere (Jeong & Holwerda, 1989*a*). X-ray diffraction studies were carried out on an Enraf-Nonius CAD-4 diffractometer equipped with a liquid-N<sub>2</sub> low-temperature device; 24 reflections with  $7 < \theta < 15^\circ$  throughout all octants of reciprocal space to refine cell constants; systematic absences:  $h0l$ ,  $l = \text{odd}$ ,  $0k0$ ,  $k = \text{odd}$ ; three standard reflections measured after every 7200 s of X-ray exposure showed no deterioration; scan width ( $0.70 + 0.35\tan\theta$ )°, horizontal aperture width ( $3.50 + 0.86\tan\theta$ ) mm and a constant height of 6 mm; scan time  $\leq 50$  s; index range  $-16 \leq h \leq 16$ ,  $0 \leq k \leq 11$ ,  $0 \leq l \leq 29$ ; non-H-atom positions determined from sharpened Patterson function and subsequent difference Fourier synthesis; H atoms from difference Fourier synthesis and refined isotropically; non-H atoms anisotropic. Crystallographic details and atomic coordinates are given in Tables 1 and 2,

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Table 1. *Crystallographic data for*  
[Pd(ca){P(Ph-*m*-CH<sub>3</sub>)<sub>3</sub>]<sub>2</sub>]

Crystal dimensions and shape	0.33 × 0.30 × 0.10 mm; prismatic dark purple crystal
2θ (max) (°)	45.9
Number of reflections	5794
Number of unique reflections	5794
Number of reflections with $F_o > 4.0\sigma(F_o)$	3916
Maximum and minimum transmission factor	0.8214 and 0.9213
Residuals	
for significant reflections	$R$ 0.039, $wR$ 0.049; GoF 1.756
for all reflections	$R$ 0.071, $wR$ 0.049

respectively.\* Intensity data were collected using the  $\theta/2\theta$  scan mode. Correction was made for absorption. The last least-squares cycle was calculated with 99 atoms, 682 parameters and 3916 out of 5794 reflections. Weights based on counting statistics were used. In the final difference map, the deepest hole was  $-1.280 \text{ e } \text{\AA}^{-3}$  and the highest peak  $1.080 \text{ e } \text{\AA}^{-3}$ , which was near palladium; the maximum  $\Delta/\sigma$  ratio was 0.265 (excluding H atoms), refined on  $F$ . Atomic and anomalous scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974). Lorentz, polarization and absorption corrections were applied to the data using the program *SHELX76* (Sheldrick, 1976). All calculations were performed with the NRCVAX crystal structure series of programs (Gabe, Le Page, Charland, Lee & White, 1989).

**Discussion.** The chloranilate ligand in [Pd(ca){P(Ph-*m*-CH<sub>3</sub>)<sub>3</sub>]<sub>2</sub>] chelates the Pd<sup>II</sup> center through ring O atoms (see *ORTEP* drawing in Fig. 1) in a distorted square-planar P<sub>2</sub>O<sub>2</sub> ligand field that includes two tri-*m*-tolylphosphine ligands in the *cis* orientation. Principal bonding parameters are summarized in Table 3. *p*-Benzoquinones have been shown to coordinate through the 1,4-diolefin groups (Glick & Dahl, 1965; Aleksandrov, Struchkov, Khandkarova & Gubin, 1970; Minematsu, Takahashi & Hagihara, 1975), in contrast to the present finding. The only other Pd<sup>II</sup>-ca<sup>2-</sup> compound to be characterized crystallographically, K<sub>2</sub>[Pd(ca)Cl<sub>2</sub>].4H<sub>2</sub>O, falls within the C-ca<sup>2-</sup> classification by virtue of its non-quinonoid boat chloranilate conformation and short Pd—C bond lengths of 2.02 and 2.07 Å (Krasochka, Avilov & Atovmyan, 1974).

Chloranilate typically functions as a bridging ligand which favors polynuclear structures, as in

\* Lists of structure factors, anisotropic thermal parameters, distances from the PdP<sub>2</sub> plane, torsion angles, intramolecular bonding parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52953 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Atomic coordinates and*  $B_{\text{iso}}$  (Å<sup>2</sup>)

Standard deviations for the last digit are in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{iso}}$
Pd	0.71456 (3)	0.55923 (5)	0.333824 (16)	1.465 (18)
Cl(1)	0.53571 (11)	0.26057 (18)	0.21703 (6)	3.00 (7)
Cl(2)	0.85312 (11)	0.63451 (18)	0.17474 (6)	2.98 (7)
P(1)	0.64164 (10)	0.50533 (16)	0.40051 (6)	1.52 (6)
P(2)	0.80932 (10)	0.70884 (15)	0.36935 (6)	1.59 (7)
O(1)	0.7647 (3)	0.6024 (4)	0.26752 (14)	2.15 (18)
O(2)	0.6399 (3)	0.4363 (4)	0.28719 (14)	2.22 (18)
O(3)	0.6118 (3)	0.3081 (5)	0.12001 (16)	3.47 (22)
O(4)	0.7481 (3)	0.4643 (4)	0.10191 (16)	3.23 (22)
C(1)	0.7342 (4)	0.5315 (5)	0.23075 (22)	1.55 (23)
C(2)	0.6606 (4)	0.4408 (6)	0.24166 (20)	1.62 (24)
C(3)	0.6200 (4)	0.3663 (6)	0.20452 (23)	2.3 (3)
C(4)	0.6462 (4)	0.3721 (6)	0.15479 (24)	2.4 (3)
C(5)	0.7249 (4)	0.4613 (6)	0.14458 (21)	2.0 (3)
C(6)	0.7644 (4)	0.5343 (6)	0.18535 (21)	1.79 (25)
C(10)	0.5370 (4)	0.4319 (6)	0.37847 (21)	1.8 (3)
C(11)	0.4585 (4)	0.4975 (7)	0.38110 (25)	2.5 (3)
C(12)	0.3792 (4)	0.4380 (8)	0.3629 (3)	3.1 (3)
C(13)	0.3821 (4)	0.3151 (7)	0.3419 (3)	3.1 (3)
C(14)	0.4601 (4)	0.2494 (6)	0.33934 (25)	2.6 (3)
C(15)	0.5379 (4)	0.3071 (6)	0.35753 (23)	2.1 (3)
C(16)	0.2934 (5)	0.5065 (9)	0.3670 (4)	5.8 (5)
C(17)	0.6171 (4)	0.6434 (6)	0.43875 (22)	1.8 (3)
C(18)	0.5917 (4)	0.7591 (6)	0.41435 (23)	2.2 (3)
C(19)	0.5771 (4)	0.8716 (6)	0.44030 (24)	2.5 (3)
C(20)	0.5827 (4)	0.8666 (6)	0.49125 (25)	2.7 (3)
C(21)	0.6044 (4)	0.7527 (7)	0.51643 (23)	2.2 (3)
C(22)	0.6223 (4)	0.6405 (6)	0.49104 (23)	2.2 (3)
C(23)	0.5559 (5)	0.9993 (7)	0.4132 (3)	3.5 (4)
C(24)	0.6933 (4)	0.3816 (6)	0.44013 (21)	1.62 (25)
C(25)	0.7793 (4)	0.3413 (6)	0.43431 (22)	1.9 (3)
C(26)	0.8222 (4)	0.2508 (6)	0.46642 (24)	2.3 (3)
C(27)	0.7764 (5)	0.2050 (6)	0.50570 (24)	2.8 (3)
C(28)	0.6905 (5)	0.2390 (6)	0.51101 (23)	2.5 (3)
C(29)	0.6478 (4)	0.3270 (6)	0.47809 (22)	2.1 (3)
C(30)	0.9158 (5)	0.2105 (7)	0.4611 (3)	3.6 (3)
C(31)	0.9218 (4)	0.6762 (6)	0.35448 (21)	1.7 (3)
C(32)	0.9407 (4)	0.5798 (6)	0.32060 (23)	2.3 (3)
C(33)	1.0256 (4)	0.5620 (7)	0.3058 (3)	3.1 (3)
C(34)	1.0902 (4)	0.6471 (8)	0.3253 (3)	3.7 (4)
C(35)	1.0734 (4)	0.7402 (8)	0.3597 (3)	4.0 (4)
C(36)	0.9895 (4)	0.7544 (7)	0.37494 (25)	2.9 (3)
C(37)	1.0437 (6)	0.4571 (9)	0.2695 (3)	5.1 (4)
C(38)	0.8229 (4)	0.7360 (6)	0.43602 (22)	1.8 (3)
C(39)	0.8613 (4)	0.6327 (6)	0.46481 (22)	1.9 (3)
C(40)	0.8695 (4)	0.6391 (6)	0.51705 (22)	2.0 (3)
C(41)	0.8423 (4)	0.7539 (7)	0.53845 (23)	2.7 (3)
C(42)	0.8074 (5)	0.8542 (6)	0.51073 (24)	2.8 (3)
C(43)	0.7967 (4)	0.8468 (6)	0.45908 (24)	2.4 (3)
C(44)	0.9062 (5)	0.5256 (7)	0.5467 (3)	3.1 (3)
C(45)	0.7782 (4)	0.8643 (6)	0.33914 (22)	2.0 (3)
C(46)	0.8314 (5)	0.9738 (6)	0.34281 (25)	2.8 (3)
C(47)	0.8071 (5)	1.0903 (6)	0.3178 (3)	3.2 (3)
C(48)	0.7277 (5)	1.0931 (7)	0.2901 (3)	3.8 (4)
C(49)	0.6738 (5)	0.9854 (7)	0.2859 (3)	3.4 (3)
C(50)	0.6983 (4)	0.8705 (7)	0.3101 (3)	3.0 (3)
C(51)	0.8694 (6)	1.2048 (8)	0.3184 (3)	4.9 (4)

$B_{\text{iso}}$  is the mean of the principal axes of the thermal ellipsoid.

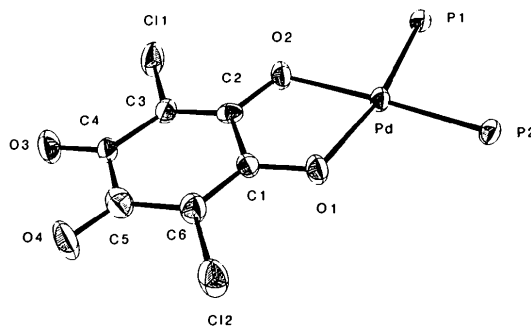


Fig. 1. *ORTEP* (Johnson, 1965) view of the inner coordination sphere of [Pd(ca){P(Ph-*m*-CH<sub>3</sub>)<sub>3</sub>]<sub>2</sub>, excluding phosphine *m*-tolyl substituents. Ellipsoids are drawn at 50% probability.

Table 3. Principal bond lengths (Å) and angles (°)

Standard deviations for the last digit are in parentheses.

Pd—P(1)	2.2554 (17)	Pd—P(2)	2.2612 (17)
Pd—O(1)	2.047 (4)	Pd—O(2)	2.054 (4)
C(1)—C(3)	1.732 (7)	C(2)—C(6)	1.737 (6)
O(1)—C(1)	1.284 (7)	O(2)—C(2)	1.292 (7)
O(3)—C(4)	1.225 (7)	O(4)—C(5)	1.231 (7)
C(1)—C(2)	1.500 (8)	C(1)—C(6)	1.342 (8)
C(2)—C(3)	1.365 (8)	C(3)—C(4)	1.430 (9)
C(4)—C(5)	1.548 (9)	C(5)—C(6)	1.421 (8)
P(1)—Pd—P(2)	99.55 (6)	P(1)—Pd—O(1)	172.02 (12)
P(1)—Pd—O(2)	93.13 (12)	P(2)—Pd—O(1)	87.36 (12)
P(2)—Pd—O(2)	167.24 (12)	O(1)—Pd—O(2)	79.89 (15)
Pd—O(1)—C(1)	114.5 (4)	Pd—O(2)—C(2)	113.2 (3)
O(1)—C(1)—C(2)	115.3 (5)	O(1)—C(1)—C(6)	124.2 (5)
C(2)—C(1)—C(6)	120.6 (5)	O(2)—C(2)—C(1)	116.8 (5)
O(2)—C(2)—C(3)	123.1 (5)	C(1)—C(2)—C(3)	120.1 (5)
C(1)—C(3)—C(2)	120.3 (5)	C(1)—C(3)—C(4)	118.3 (5)
C(2)—C(3)—C(4)	121.4 (6)	O(3)—C(4)—C(3)	124.2 (6)
O(3)—C(4)—C(5)	117.8 (6)	C(3)—C(4)—C(5)	117.9 (5)
O(4)—C(5)—C(4)	117.8 (5)	O(4)—C(5)—C(6)	125.0 (6)
C(4)—C(5)—C(6)	117.2 (5)	C(2)—C(6)—C(1)	119.4 (4)
C(2)—C(6)—C(5)	117.9 (5)	C(1)—C(6)—C(5)	122.6 (5)

complexes with Pr<sup>III</sup> (Riley, Haddad & Raymond, 1983), Ni<sup>II</sup> and Cu<sup>II</sup> (Pierpont, Francesconi & Hendrickson, 1977; Tinti, Verdaguer, Kahn & Savariault, 1987). In [Cu<sub>2</sub>(Me<sub>5</sub>dien)<sub>2</sub>(C<sub>6</sub>O<sub>4</sub>Cl<sub>2</sub>)](BPh<sub>4</sub>)<sub>2</sub> the chloranilate bridge coordinates unsymmetrically (Cu—O bond lengths of 1.956 and 2.196 Å) and exhibits a *p*-quinonoid structure with four short (1.381 Å) and two long (1.536 Å) C—C lengths (Pierpont *et al.*, 1977).

The bonding parameters shown in Table 3 and supplementary Tables 4 and 5\* reveal that the chloranilate ligand of [Pd(ca){P(Ph-*m*-CH<sub>3</sub>)<sub>3</sub>}]<sub>2</sub> is twisted out of the PdP<sub>2</sub> plane. A slight distortion from square planar towards pyramidal coordination geometry is particularly evident in the P(1), Pd, O(1), C(1) torsion angle of -34.1 (3)° and the stereoview of the unit cell in Fig. 2. 6,6'-Disubstitution on bipyridine (bpy) in palladium(II) complexes causes drastic deviations from an overall planar configuration, motivated by steric interactions between bpy substituents (Newkome, Fronczek, Gupta, Puckett, Pantaleo & Kiefer, 1982). Considering the expansion in triphenylphosphine cone angle induced by *m*-CH<sub>3</sub> groups (Tolman, 1977) and the exceptionally large P(1)—Pd—P(2) bond angle of 99.55°, the deviation from square-planar geometry in [Pd(ca){P(Ph-*m*-CH<sub>3</sub>)<sub>3</sub>}]<sub>2</sub> may be attributed to steric repulsion between the *cis* tri-*m*-tolylphosphine ligands.

Although chloranilic acid clearly favours a *p*-quinonoid structure (Andersen, 1967*a*), unsymmetrically coordinated ca<sup>2+</sup> in [Pd(ca){P(Ph-*m*-CH<sub>3</sub>)<sub>3</sub>}]<sub>2</sub> exhibits a bonding pattern that is most consistent with the *o*-quinone resonance form *C*. Thus, the two Pd—O bond lengths are essentially identical (2.05 Å), as are the C(1)—O(1) and C(2)—O(2) distances (1.29 Å). Furthermore, the considerably shorter but equal C(4)—O(3) (1.225) and C(5)—O(4)

(1.231 Å) bond lengths support the formulation of adjacent C=O bonds opposite to the C—O ligating functions. An extensive literature exists on the coordination chemistry of *o*-quinone ligands as well as their semiquinone and catecholate reduction products (Pierpont & Buchanan, 1981), including a recent report of ca<sup>2+</sup> coordinated as an *o*-quinone in [Cu(terpy)(ca)].H<sub>2</sub>O (terpy = 2,2',2''-terpyridine) (Folgado, Ibanez, Coronado, Beltran, Savariault & Galy, 1988). As compared with chloranilic acid dihydrate (C—O, 1.320; C=O, 1.225 Å; Andersen, 1967*a*), the [Pd(ca){P(Ph-*m*-CH<sub>3</sub>)<sub>3</sub>}]<sub>2</sub> C—O single bonds are shorter by ca 0.03 Å while the corresponding double bonds are identical in length. Chloranilate unsymmetrically ligated to the electrophilic Pd<sup>II</sup> center is best viewed as being electronically polarized from the free ca<sup>2+</sup> structure, in which all four C—O bonds are of equal length (1.248 Å) at a value intermediate between those characteristic of C—O and C=O bonds (Andersen, 1967*b*). It should be noted, however, that the bonds from C atoms to the ligated O atoms retain partial double-bond character and therefore cannot be regarded as full phenolate O<sup>-</sup> donors. This point is underscored by a structural comparison with the oxidative addition product of (PPh<sub>3</sub>)<sub>2</sub>Pd<sup>0</sup> with tetrachloro-1,2-benzoquinone, [Pd(PPh<sub>3</sub>)<sub>2</sub>(C<sub>6</sub>Cl<sub>4</sub>O<sub>2</sub>)] (Pierpont & Downs, 1975). This complex, described as a Pd<sup>II</sup>-hydroquinone adduct, exhibits C—O bonds (1.342 and 1.344 Å) longer by 0.05 Å and slightly shorter Pd—O bonds (by 0.02 Å; 2.028 and 2.038 Å) than in the present Pd<sup>II</sup>-ca<sup>2+</sup> complex. Pd—P and C—Cl bond lengths given by Pierpont & Downs (1975) are comparable to those reported here.

A comparison of [Pd(ca){P(Ph-*m*-CH<sub>3</sub>)<sub>3</sub>}]<sub>2</sub> C—C bonds with those of H<sub>2</sub>ca.2H<sub>2</sub>O reinforces our assignment of a predominant *o*-quinone resonance form. The C(2)—C(3) [1.365 (8)] and C(1)—C(6) [1.342 (8) Å] bond lengths compare favorably with the average C=C length in H<sub>2</sub>ca (1.346 Å) and an equally good correspondence exists between the lengths of proposed C(3)—C(4) [1.430 (9)] and C(5)—C(6) [1.421 (8) Å] single bonds and those of the parent acid (1.446 Å) (Andersen, 1967*a*). The appreciable disparity between the longest chloranilate C—C bonds in the Pd<sup>II</sup> complex [1.500 (8) vs 1.548 (9) Å] and the observation of equivalent long C—C bonds in H<sub>2</sub>ca (1.506 Å; Andersen, 1967*a*)

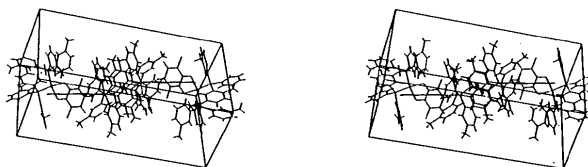


Fig. 2. Stereoview of the [Pd(ca){P(Ph-*m*-CH<sub>3</sub>)<sub>3</sub>}]<sub>2</sub> unit cell. *c* horizontal, *a* vertical and *b* into the plane of the paper.

\* See deposition footnote.

implies a weakening of the C(4)—C(5) linkage owing to the proximal C=O bonds. The existence of only two distinct C—C bond types in  $\text{ca}^{2-}$  [as  $(\text{NH}_4)_2\text{-ca.H}_2\text{O}$ ; 1.401 and 1.535 Å] and in  $\text{ca}^{2-}$  bridged between bivalent first-row transition-metal ions (Pierpont *et al.*, 1977) implies a delocalization of *p*-quinone  $\pi$ -electron density for which there is no equivalent in  $[\text{Pd}(\text{ca})\{\text{P}(\text{Ph-}m\text{-CH}_3)_3\}_2]$ .

The X-ray crystal structure of  $[\text{Pd}(\text{ca})\{\text{P}(\text{Ph-}m\text{-CH}_3)_3\}_2]$  poses an apparent contradiction with electronic and infrared spectra recently reported for this and related  $[\text{Pd}(\text{ca})(\text{PR}_3)_2]$  compounds (Jeong & Holwerda, 1988*a*, 1989*a*). Thus, the title complex exhibits  $\pi$ - $\pi^*$  absorption bands at 344 and 543  $\text{cm}^{-1}$ , quite similar in both position and intensity to those of  $\text{ca}^{2-}$  and  $\text{Hca}^-$ , for which the contribution of the *p*-quinone resonance form cannot be neglected (Jeong & Holwerda, 1989*a*). Similarly, the  $\text{ca}^{2-}$  contribution to the infrared spectrum of  $[\text{Pd}(\text{ca})\{\text{P}(\text{Ph-}m\text{-CH}_3)_3\}_2]$  [C—O and C=O bands at 1519 and 1643  $\text{cm}^{-1}$ , respectively; Jeong & Holwerda (1989*a*)] is virtually indistinguishable from  $\text{K}_2\text{ca}$  IR features. The lack of a strong medium dependence of  $[\text{Pd}(\pi\text{-ca})(\text{PR}_3)_2]$  electronic spectra rules out the possibility of a solvent-induced perturbation in the preferred resonance form of coordinated  $\text{ca}^{2-}$ . Considering that *o*-quinone and *p*-quinone isomers are both consistent with the observed three-line  $^{13}\text{C}$  NMR spectrum of coordinated chloranilate in  $[\text{Pd}(\text{ca})(\text{PR}_3)_2]$  complexes (Jeong & Holwerda, 1988*a*), it appears likely that resonance forms *B* and *C* contribute to the structure of  $[\text{Pd}(\text{ca})\{\text{P}(\text{Ph-}m\text{-CH}_3)_3\}_2]$  in solution.

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## Structure of Bis(1,3-diphenyl-1,3-propanedionato-*O,O'*)(1,2-ethanediamine-*N,N'*)-nickel(II)

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**Abstract.**  $\text{C}_{32}\text{H}_{30}\text{N}_2\text{NiO}_4$ ,  $M_r = 565.3$ , orthorhombic,  $\text{Pna}2_1$ ,  $a = 10.347$  (3),  $b = 11.586$  (3),  $c = 23.694$  (4) Å,  $V = 2840$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.32$ ,  $D_m$

$= 1.31$  (1)  $\text{g cm}^{-3}$ , Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å,  $\mu = 6.84$   $\text{cm}^{-1}$ ,  $F(000) = 1184$ ,  $T = 293$  (1) K,  $R = 0.048$  for 1622 observed reflections with  $I \geq 2.5\sigma(I)$ . The preparation of a Schiff-base condensate *via* the template reaction of 1,2-ethanediamine and 1,3-

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