

FORMATION OF TRINUCLEAR COMPLEXES WITH BISACYL- π -ALLYL LIGANDS FROM PALLADIUM ACETATE AND PYRYLIUM SALTS

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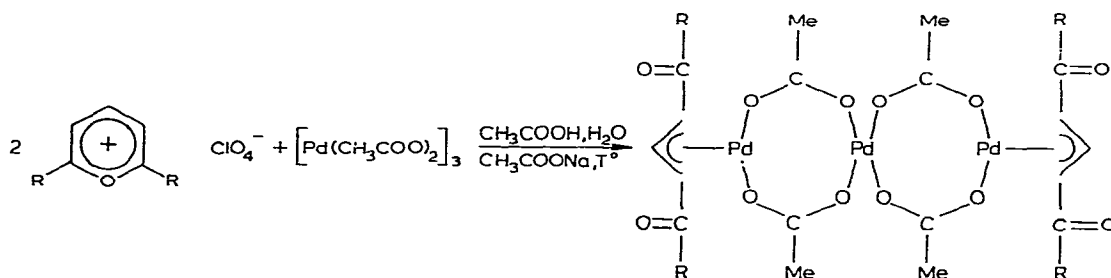
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Summary

Trimeric palladium acetate reacts with 2,6-disubstituted pyrylium salts to give trinuclear bis-acyl-allylpalladium acetates. The structure of bis(1,3-dipivaloyl- π -allyl)trispalladium(II) μ -tetraacetate is determined by an X-ray study.

Introduction

As reported earlier by us [1,2], 2,6-disubstituted pyrylium salts react on heating with palladium chloride in water-containing organic solvents to give binuclear complexes with 1,3-bisacyl- π -allyl ligands. Binuclear π -allylpalladium acetate obtained by the interaction of the bis- π -allyldipalladium μ -dichloride with silver acetate has been described in the literature [3,4]. The interaction of trimeric palladium acetate [5] with 2,6-disubstituted pyrylium salts in refluxing acetic acid in the presence of water and sodium acetate was found to lead to the formation of trinuclear bis(acylallyl)palladium acetates (I) [6]:



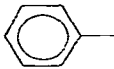
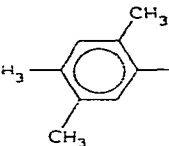
Some characteristics of the compounds obtained are summarized in Table 1.

An X-ray study of bis(1,3-dipivaloyl- π -allyl)trispalladium(II) μ -tetraacetate (Ia) has been carried out to confirm its structure and determine the molecular geometry.

Results and discussion

The molecular structure with bond lengths is shown in Fig. 1; bond angles are given in Table 2. The molecule represents a trinuclear centrosymmetric complex where the Pd atoms located on the same straight line are linked pairwise by pairs of acetate bridges. Thus the central Pd(2) atom has a square-planar coordination by oxygen atoms of four bridging acetate groups. The terminal Pd(1) and Pd(1') atoms, in addition to two oxygen atoms of the acetate bridges, are coordinated by the conventional η^3 -type bonding with the allyl fragments of the acyclic diketo ligands and thus acquire also a square-planar coordination. The coordination planes of the palladium atoms Pd(2)O(1)O(3) and Pd(1)O(2)O(4) (I and II in Table 3) make an angle of 25.3° with each other. The Pd(1)...Pd(2) distance, equal to $2.864(1) \text{ \AA}$, exceeds the Pd—Pd bond length in the previously studied palladium complexes with such bonding (2.57 – $2.699 \text{ \AA}$) [7–12] and the shortest interatomic distance in metallic palladium ($2.751 \text{ \AA}$) [13], but is nevertheless shorter than the doubled covalent radius of Pd ($2.98 \text{ \AA}$) [14]. In other polynuclear complexes where Pd atoms are linked by two bridging acetate groups but without metal–metal bonding the Pd...Pd distances are somewhat greater. For example, in the tetranuclear complex $[\text{Pd}_2(\text{CH}_3\text{COO})_2(\text{CO})_2]_2$ the Pd...Pd distance is $2.909(1) \text{ \AA}$ [12], and in dimeric π -allylpalladium acetate $[(\eta^3\text{-C}_3\text{H}_5)(\text{CH}_3\text{COO})\text{Pd}]_2$ it is $2.94(2) \text{ \AA}$ [3]. Thus the proximity of the Pd atoms cannot be attributed to the effect of the bridging acetate groups.

TABLE 1
PROPERTIES OF COMPOUNDS I

R	Color	M.p. ($^\circ\text{C}$)	IR spectrum (cm^{-1}), sol. in CHCl_3 UR 20 instrument			
			$\nu(\text{C}=\text{O})$	$\nu_{\text{as}}(\text{COO})$	$\nu_{\text{s}}(\text{COO})$	$\nu(\text{C}\equiv\text{O})$
Ia $\text{tert-C}_4\text{H}_9$	brick-red	187–195 (dec.)	1690	1567	1406	
Ib 	brown-yellow	220–222 (dec.)	1675	1565	1402	
Ic 	pale yellow	191–193 (dec.)	1655	1562	1405	
Id $(\text{CO}_3)_2\text{Mn}$ 	yellow	198–202 (dec.)	1665	1565	1410	1955, 2033

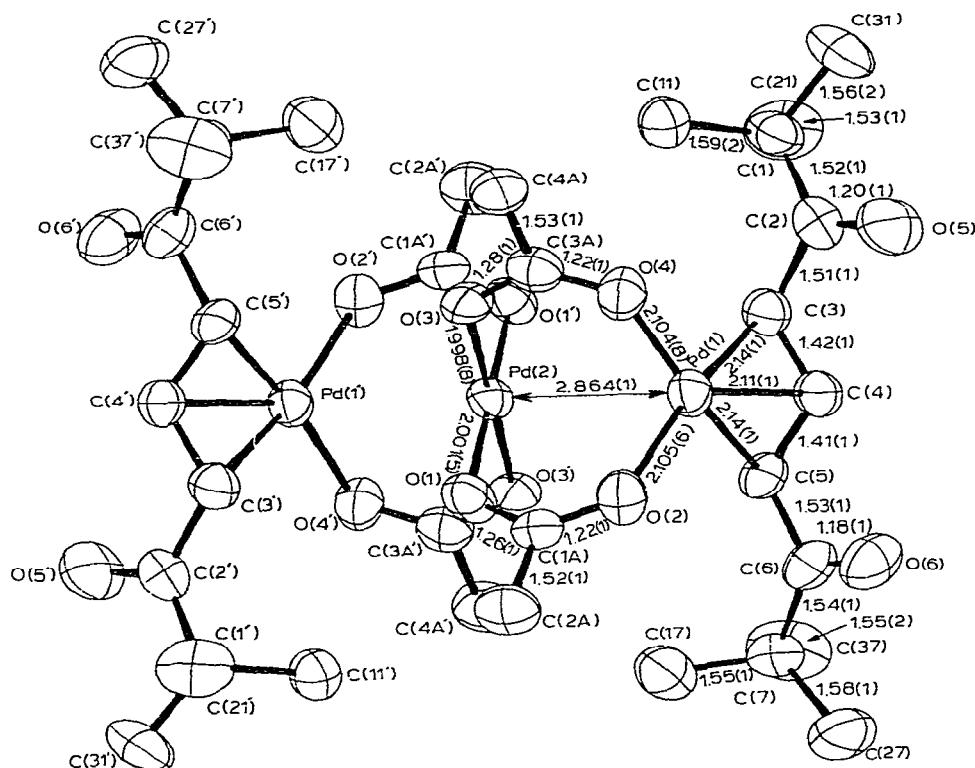


Fig. 1. The molecular structure of bis(1,3-dipivaloyl- π -allyl)tris(palladium(II) μ -tetraacetate (Ia).

TABLE 2

BOND ANGLES ω (deg.)

Angle	ω	Angle	ω
O(2)Pd(1)O(4)	90.3(3)	C(2)C(3)Pd(1)	118.3(7)
O(4)Pd(1)C(3)	102.2(3)	Pd(1)C(3)C(4)	69.5(6)
O(4)Pd(1)C(4)	134.1(3)	C(3)C(4)C(5)	116.3(9)
O(4)Pd(1)C(5)	170.2(3)	C(3)C(4)Pd(1)	71.5(6)
O(2)Pd(1)C(5)	99.2(3)	C(5)C(4)Pd(1)	72.0(6)
O(2)Pd(1)C(4)	130.8(3)	C(4)C(5)C(6)	115.5(8)
O(2)Pd(1)C(3)	167.3(3)	C(6)C(5)Pd(1)	114.5(6)
O(1)Pd(2)O(3)	88.9(3)	Pd(1)C(5)C(4)	69.4(5)
O(1)Pd(2)O(3')	91.1(3)	O(5)C(6)C(5)	121.5(9)
Pd(1)O(2)C(1A)	121.8(6)	O(6)C(6)C(7)	122.7(9)
Pd(1)O(4)C(3A)	125.7(6)	C(5)C(6)C(7)	115.8(8)
Pd(2)O(1)C(1A)	123.6(6)	C(2)C(1)C(11)	108.8(8)
Pd(2)O(3)C(3A)	123.8(6)	C(2)C(1)C(21)	109.2(9)
O(1)C(1A)O(2)	126.6(8)	C(2)C(1)C(31)	108.9(9)
O(1)C(1A)C(2A)	115.4(8)	C(11)C(1)C(21)	113.4(9)
O(2)C(1A)C(2A)	118.0(8)	C(11)C(1)C(31)	108.3(9)
O(3)C(3A)O(4)	127.7(9)	C(21)C(1)C(31)	108.1(9)
O(3)C(3A)C(4A)	113.9(8)	C(6)C(7)C(17)	107.7(8)
O(4)C(3A)C(4A)	118.4(8)	C(6)C(7)C(27)	108.7(9)
O(5)C(2)C(1)	123(1)	C(6)C(7)C(37)	110.3(9)
O(5)C(2)C(3)	120(1)	C(17)C(7)C(27)	110.0(9)
C(1)C(2)C(3)	117.6(9)	C(17)C(7)C(37)	111.1(9)
C(2)C(3)C(4)	116.7(9)	C(27)C(7)C(37)	108.9(9)

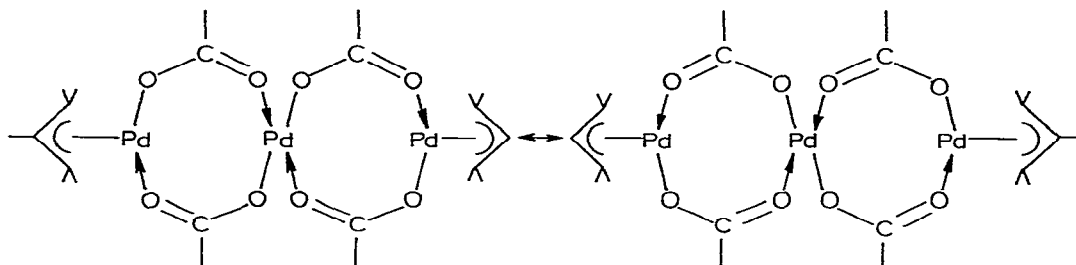
TABLE 3
EQUATIONS $Ax + By + Cz - D = 0$ OF SOME OF THE MOLECULAR PLANES AND DISPLACEMENTS OF ATOMS FROM THE CORRESPONDING PLANES (Å)

Plane	Atoms and their displacement				A	B	C	D
I	Pd(1) C(1A) ^a -0.952(8)	O(2) C(3A) ^a -0.969(8)	O(4) C(3) ^a -0.085(8)	C(4) ^a 0.618(8)	0.230	0.453	-0.861	-6.499
II	Pd(2) C(3)	O(1) C(4)	O(3) C(5)	C(5) ^a -0.087(8)	-0.166	0.285	-0.944	-12.100
III	Pd(1) ^a -1.6976(8)	C(2) ^a -0.23(1)	C(6) ^a -0.33(1)		-0.919	-0.389	-0.064	-3.530
IV	Pd(1) -0.0016(8)	O(2) 0.013(8)	C(1A) -0.023(9)	O(1) 0.016(8)	0.943	-0.302	-0.137	2.797
V	Pd(1) -0.0025(6)	O(4) 0.000(6)	C(3A) 0.006(8)	O(3) 0.008(6)	-0.380	-0.871	-0.310	5.496
Angle between the planes I-II 25.3; I-III 109.5; IV-V 86.6°								

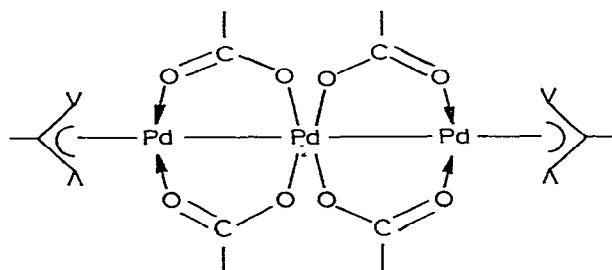
^a Atoms not taken into account in calculation of corresponding plane equations.

As in other Pd complexes with bridging acetate groups, both bridging systems Pd(1)O(2)C(1A)O(1)Pd(2) and Pd(1)O(4)C(3A)O(3)Pd(2) (IV and V in Table 3) are virtually planar and nearly perpendicular to each other.

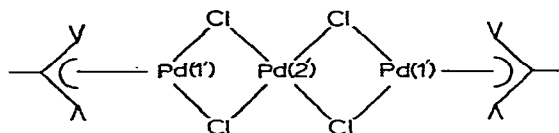
The Pd(1)—O(2) and Pd(1)—O(4) bond lengths (2.105(6) and 2.104(8) Å, respectively) of the terminal Pd atoms agree with the values of 2.10–2.12 Å in other carboxylate complexes [3,12]. At the same time, the Pd(2)—O(1) and Pd(2)—O(3) bond lengths (2.001(5) and 1.998(8) Å, respectively) of the central atom are significantly shorter. The short Pd—O distances (2.028 and 2.039 Å) have also been found earlier in the complex Pd[P(C₆H₅)₃]₂(O₂C₆Cl₄), where the Pd atom is chelated with an *o*-tetrachlorobenzoquinone ligand [15]. Along with shortening of the Pd(2)—O(1) and Pd(2)—O(3) bonds in molecule Ia one can observe a certain elongation of the O(1)—C(1A) and O(3)—C(3A) bonds (1.26(1) and 1.28(1) Å, respectively) as compared with the O(2)—C(1A) and O(4)—C(3A) bonds (1.22(1) Å in both cases). The distribution of the bond lengths in the bridging systems and the proximity of the Pd atoms are consistent with the fact that, along with the main electronic structure with 16-electron shells for all the Pd atoms:



there is a certain contribution from the structure with a Pd—Pd bond and an 18-electron shell for the terminal Pd atoms and a 16-electron shell for the central atom:



A difference between the bond lengths of the terminal and central Pd atoms with the bridging atoms has been also found in the molecule of the trinuclear complex Pd₃(η³-2-methylallyl)₂Cl₄ [16]:



In this molecule the Pd(2)—Cl bonds (2.313(9) and 2.297(8) Å) are considerably shorter than the Pd(1)—Cl bonds (2.418(8) and 2.416(9) Å), the former

being typical of terminal Cl atoms and the latter of bridging ones. Such an unusual geometry of the complex is attributed to a substantial contribution from the ionic form $[\eta^3\text{-2-MeC}_3\text{H}_4\text{Pd}]^+[\text{PdCl}_4]^{2-}[\eta^3\text{-2-MeC}_3\text{H}_4\text{Pd}]^+$.

The allyl group is coordinated by the Pd(1) atom in such a way that the central atom, C(4), of this ligand is situated on the other side of the coordination plane relatively to the C(1A) and C(3A) atoms of the acetate bridges (I in Table 3). Such orientation of the allyl group is probably caused by steric factors, as in the geometry found for the complex there are no shortened contacts between atoms of the acetate and dipivaloyl- π -allyl ligands.

The difference between the Pd—C(3) and Pd—C(5) distances (2.14(1) Å in both cases) to the terminal atoms and the Pd—C(4) distance (2.11(1) Å) to the central atom of the π -allyl is insignificant as it lies within 3σ , although the values found agree with those observed in the binuclear complex with the same ligand [1], where the greater accuracy has shown a difference in these distances: Pd—C(terminal) 2.137(3) and 2.148(3) Å, Pd—C(central) 2.096(3) Å.

The π -allyl plane is tilted to the coordination plane of Pd(1) at an angle of 109.5° , which is close to the theoretically calculated value ($102\text{--}114^\circ$) [17] and found in other complexes with a similar coordination of the Pd atom (e.g. 107.5° in $[\eta^3\text{-C}_{13}\text{H}_{21}\text{O}_2\text{PdCl}]_2$ [1]).

The bond lengths in the dipivaloyl- π -allyl ligand coincide with those reported in ref. 1. Its two chemically equivalent halves show some differences in the torsion angles (C(4)C(3)C(2)O(5) 12.1° , C(4)C(5)C(6)O(6) 19.8° , C(4)C(3)C(2)C(1) 13.4° and C(4)C(5)C(6)C(7) 21.9°), which are probably due to crystal field effects.

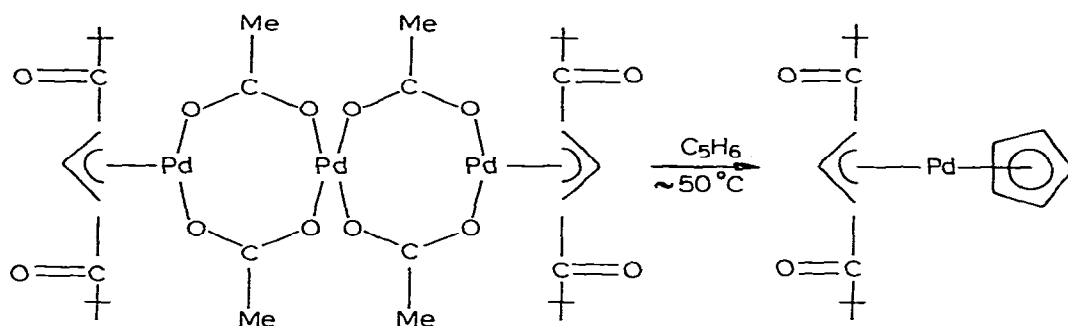
Studies of the chemical properties of complexes I have shown that their acetate residues, like those in the starting palladium acetate [5] are very mobile. Under the action of acids they are readily replaced by other acidic ligands. The metal—carbon bond is normally unaffected in these reactions. With benzoic acid Ia converts to bis(1,3-dipivaloyl- π -allyl)trispalladium(II) μ -tetrabenzoate; IR spectrum: $\nu(\text{C=O}) = 1688$, $\nu_{\text{as}}(\text{COO}) = 1556$, $\nu_{\text{s}}(\text{COO}) = 1392\text{ cm}^{-1}$ (solution in CHCl_3 , UR 20 instrument).

From a solution of Ia in CF_3COOH a complex was isolated in which there was one Pd atom per one bispyvaloyl- π -allyl ligand and one trifluoroacetate residue ($\nu(\text{C=O}) = 1685\text{ cm}^{-1}$). Also there is a very strong band at 1660 cm^{-1} which is likely to be due to the antisymmetric valence vibrations of the trifluoroacetate group: $\nu(\text{C—F}) = 1165, 1205\text{ cm}^{-1}$ (vaseline oil, Specord IR 71 instrument).

Under the same conditions Ib gives trifluoroacetate with the bisbenzoyl- π -allyl ligand. IR spectrum: $\nu(\text{C=O}) = 1665$, $\nu(\text{COO}) = 1645$, $\nu(\text{C—F}) = 1180\text{ cm}^{-1}$ (vaseline oil, Specord IR-71).

The IR spectrum of the azide complex, probably dimeric with bridging azido groups, produced by the interaction of a toluene solution of HN_3 with Ia, contains a very strong band $\nu(\text{N}_3) = 2100\text{ cm}^{-1}$, and $\nu(\text{C=O}) = 1680\text{ cm}^{-1}$ (vaseline oil, Specord IR 71).

Complex Ia metallates cyclopentadiene when heated in it, to give a fully organometallic compound:



Heating of Ia in acetylacetone results in the substitution of both the acetate and bispyvaloyl- π -allyl ligands and the formation of palladium acetylacetonate.

Experimental

The starting pyrylium salts were synthesized by the known methods [18,19].

1. Bis(1,3-dipyvaloyl- π -allyl)tris(palladium(II) μ -tetraacetate (Ia)

0.6 g of 2,6-di-*t*-butylpyrylium perchlorate and 0.7 g of palladium acetate in 20 ml of glacial acetic acid were heated to reflux under stirring. A solution of 0.2 g of CH_3COONa in 4 ml of water was then added, boiling was continued for 12 min, and the solution was filtered while hot. After 3 h the precipitate formed was filtered off. Large red-brown crystals were obtained, 0.75 g (75%). Found: C, 40.75; H, 5.69. $\text{C}_{34}\text{H}_{54}\text{C}_{12}\text{Pd}_3$ calcd.: C, 41.10; H, 5.54%.

2. Bis(1,3-dibenzoyl- π -allyl)tris(palladium(II) μ -tetraacetate (Ib)

0.33 g of 2,6-diphenylpyrylium perchlorate and 0.22 g of palladium acetate in 10 ml of glacial acetic acid were heated under stirring for 10 min to 100°C , then a solution of 1 g of CH_3COONa in 1 ml of water was added. The resulting voluminous precipitate was filtered off, washed with an ether/hexane mixture and dried on the filter. The product was purified by reprecipitation from CHCl_3 with hexane. Yield: 0.3 g (90%). Found: C, 17.72; H, 3.52; Pd, 30.91. $\text{C}_{42}\text{H}_{38}\text{O}_{12}\text{Pd}_3$ calcd.: C, 47.91; H, 3.61; Pd, 30.30%.

3. Bis(1,3-di(2,4,5-trimethylbenzoyl)- π -allyl)tris(palladium(II) μ -tetraacetate (Ic)

0.2 g of 2,6-di(2,4,5-trimethylphenyl)pyrylium perchlorate and 0.16 g of palladium acetate in 7 ml of glacial acetic acid were heated to reflux, and then a solution of 0.05 g of CH_3COONa in 1 ml of water was added, boiling was continued for 3 min, and the solution was filtered while hot. The golden-coloured precipitate obtained on cooling was isolated and recrystallized from acetic acid. Yield: 0.1 g (28%). Found: C, 52.44; H, 5.74; Pd, 25.42. $\text{C}_{54}\text{H}_{62}\text{O}_{12}\text{Pd}_3$ calcd.: C, 53.01, H, 5.01, Pd, 26.22%.

4. Bis(1,3-dicymantrenoyl- π -allyl)tris(palladium(II) μ -tetraacetate (Id)

1.95 g of 2,6-dicymantrenoylpyrylium perchlorate and 1.12 g of palladium acetate in 100 ml of glacial acetic acid were heated to reflux under stirring. 0.5 g of sodium acetate in 20 ml of water was added to the resulting dark

cherry-coloured solution and heating was discontinued. After change of the reaction mixture colour to yellow, the precipitate was filtered off, washed with ether and hexane, and dried on the filter. The product was purified by recrystallization from chloroform. The complex is sensitive to light. Yield of the crude product: 1.7 g (60%). Found: C, 39.10; H, 2.53, Mn + Pd, 34.89. $C_{50}H_{34}O_{24}Mn_4Pd_3$ calcd.: C, 38.56; H, 2.19; Mn + Pd, 34.62%.

5. *Bis(1,3-dipyvaloyl- π -allyl)trispalladium(II) μ -tetrabenzoate*

0.2 g of Ia and 0.12 g of benzoic acid in 7 ml of benzene were refluxed for 2 min; the solution was filtered off while hot from the precipitated metallic palladium. After concentration by evaporation in air, 2 ml of hexane was added to the resinous residue formed with grinding with a glass rod. After evaporation of hexane the grey-orange solid product (0.2 g) was recrystallized from nitromethane while cooling on an ice/salt bath. Yield of the analytically pure product: 0.04 g (16%). Found: C, 52.83; H, 5.42; Pd, 26.37. $C_{54}H_{62}O_{12}Pd_3$ calcd.: C, 53.29; H, 5.09; Pd, 25.82%.

6. *Interaction of Ia with CF_3COOH*

0.4 g of Ia was dissolved in 2 ml of trifluoroacetic acid. Water was added and a yellow precipitate obtained was filtered off, washed with water to neutral reaction, dried in air and recrystallized from isooctane. Yield: 0.29 g, m.p. 195°C. Found: C, 42.34; H, 4.65; F, 13.77%. $C_{15}H_{21}O_4F_3Pd$ calcd.: C, 42.02, H, 4.90; F, 13.30%.

7. *Interaction of Ia with HN_3*

1 ml of a toluene solution of HN_3 ($C = 0.036$ g/ml) was added to 0.08 g of Ia in 4 ml of toluene, then the solution was filtered off from the dark-grey precipitate formed (Handle with care! Explosive palladium azide). From the filtrate, while adding hexane, an orange precipitate was obtained which was filtered off, washed with hexane, dissolved on the filter in $CHCl_3$ without drying and reprecipitated with hexane. Yield: 0.03 g (49%), m.p. 90–91°C. Found: C, 43.99; H, 6.43; N, 11.23. $C_{13}H_{21}O_2N_3Pd$ calcd.: C, 43.70; H, 5.89; N, 11.76%.

8. *Interaction of Ia with cyclopentadiene*

0.2 g of Ia in 3.6 ml of freshly distilled cyclopentadiene was kept on a water bath at 50°C for 30 min. The dark blue crystals precipitated after cooling were filtered off, dissolved in the minimum amount of chloroform and then a 10-fold excess of petroleum ether was added. After concentration by evaporation in air to the initial volume, the crystals precipitated were filtered off.

The compound was identified by elemental analysis and by comparison of its IR spectrum with that of the authentic sample [2]. Yield: 0.06 g (40%), m.p. 168–173°C. Found: C, 56.18; H, 7.24. $C_{18}H_{26}O_2Pd$ calcd.: C, 56.78; H, 6.84%.

9. *X-ray study of Ia*

Crystals of Ia are monoclinic, at room temperature $a = 14.748(4)$, $b = 11.464(2)$, $c = 15.639(4)$ Å, $\beta = 129.9(1)^\circ$, $V = 2027.0(8)$ Å³, $d_{calc.} = 3.01$ g/cm³, space group $P2_1/c$. Cell parameters and intensities of 2840 reflections with $F^2 > 2\sigma$ were measured with a Syntex P2₁ 4-circle automatic diffractome-

TABLE 4

ATOMIC COORDINATES ($\times 10^4$, FOR Pd $\times 10^5$) AND B_{ij} PARAMETERS OF THE ANISOTROPIC TEMPERATURE FACTORS

$$T = \exp[-1/4(B_{11}c^2h^2 + B_{22}b^2k^2 + \dots + 2B_{23}b^*c^*hk)]$$

Atom	x	y	z	B ₁₁	B ₂₂	B ₃₃	B ₁₂	B ₁₃	B ₂₃
Pd(1)	84216(5)	8542(5)	77563(4)	3.62(3)	4.01(3)	3.06(2)	0.14(2)	2.01(2)	0.19(2)
Pd(2)	100000(0)	0(0)	100000(0)	3.76(4)	3.27(3)	2.87(3)	0.03(3)	2.25(3)	0.02(3)
O(1)	10703(5)	1600(4)	10363(4)	4.9(3)	3.3(2)	3.5(2)	-0.8(3)	2.5(2)	-0.4(2)
O(2)	9506(5)	2313(5)	8647(4)	3.8(3)	3.9(2)	4.0(2)	-0.5(2)	2.3(2)	0.3(2)
O(3)	9039(5)	522(5)	10420(4)	4.7(3)	4.4(2)	3.6(2)	0.9(2)	3.1(2)	0.0(2)
O(4)	9757(5)	103(5)	7806(4)	3.6(3)	5.3(3)	3.4(2)	0.9(2)	2.5(2)	0.4(2)
O(5)	6444(8)	-594(6)	5111(6)	10.9(6)	7.1(4)	4.6(3)	1.4(4)	5.0(4)	0.7(3)
O(6)	6375(6)	3096(5)	6599(5)	6.7(4)	5.6(3)	5.5(3)	1.8(3)	4.3(3)	1.6(3)
C(1A)	10304(7)	2399(6)	9651(6)	3.7(4)	2.9(3)	4.2(4)	-0.1(3)	2.7(3)	-0.3(3)
C(2A)	10911(8)	3576(7)	10110(8)	5.9(5)	3.2(4)	6.3(5)	-1.6(3)	3.7(4)	-0.8(3)
C(3A)	10640(7)	-390(7)	8607(7)	3.82(4)	3.2(3)	4.4(4)	-0.7(3)	3.0(3)	-0.9(3)
C(4A)	11488(8)	-935(8)	8473(7)	4.25(4)	6.4(5)	4.8(4)	0.6(4)	3.3(4)	-0.7(4)
C(1)	7158(8)	-2397(8)	6112(7)	7.7(4)	4.5(4)	4.2(4)	-0.4(3)	2.9(3)	-1.2(3)
C(2)	6859(8)	-1102(8)	5959(7)	4.2(4)	5.4(4)	3.7(4)	-0.8(3)	2.3(3)	-0.6(3)
C(3)	7081(7)	-459(8)	6924(7)	3.3(4)	4.6(4)	4.2(4)	-0.3(3)	2.3(3)	-0.8(3)
C(4)	6566(7)	665(7)	6691(7)	2.6(3)	4.4(4)	4.4(4)	-0.2(3)	1.8(3)	-0.8(3)
C(5)	6927(6)	1330(7)	7619(7)	2.5(3)	4.2(4)	4.4(4)	0.4(3)	2.1(3)	-0.5(3)
C(6)	6606(7)	2629(8)	7392(7)	3.3(4)	4.5(4)	4.8(4)	0.6(3)	2.7(3)	0.6(3)
C(7)	6563(8)	3263(7)	8236(7)	5.2(4)	3.8(4)	4.8(4)	0.5(3)	3.2(4)	-0.3(3)
C(11)	8538(8)	-2551(8)	7097(7)	3.8(4)	6.1(4)	4.6(4)	0.3(4)	1.8(4)	-1.3(4)
C(21)	6402(10)	-3043(9)	6309(10)	7.4(6)	6.1(5)	9.0(7)	-1.3(5)	6.9(6)	-0.8(6)
C(31)	6850(9)	-2881(9)	5020(8)	6.2(5)	7.0(6)	4.7(4)	-1.7(4)	3.6(4)	-2.6(4)
C(17)	7846(8)	3352(9)	9352(8)	5.2(5)	6.2(5)	5.0(4)	-0.4(4)	2.9(4)	-1.3(4)
C(27)	6031(11)	4522(9)	7775(9)	9.2(7)	4.4(4)	6.4(5)	1.7(5)	5.0(5)	1.0(4)
C(37)	5764(11)	2587(10)	8386(10)	8.3(7)	7.0(6)	8.7(7)	-1.8(5)	6.9(6)	-1.3(5)

ter (λ Mo- K_{α} , graphite monochromator, $2\theta_{\max} = 54^{\circ}$).

The structure was solved by a heavy-atom method and refined by a least-squares technique in a full-matrix anisotropic approximation. No attempts to localize the hydrogen atoms have been made. The final R factor is 0.043 ($R_G = 0.064$) for 2731 reflections with $|F| > 6\sigma$. Atomic coordinates and B_{ij} parameters of anisotropic temperature factors are given in Table 4.

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