Preliminary communication

The molecular structure of di-μ-acetatobis(2-methylallyl-3-norbornyl)–dipalladium(II)

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The molecular structure of the title compound has been determined by X-ray diffraction from a crystal prepared as described in the preceding communication. The intensities of 3694 independent reflections from this crystal (space group *Pbcm*, $a = 11.82, b = 12.51, c = 17.61$ Å) were measured by a three-circle diffractometer up to a $(\sin \theta)/\lambda$ value of 0.70 Å^{-1} (Mo-Kα radiation).

The structure was solved by Patterson and Fourier methods and a preliminary refinement by least squares, using isotropic temperature parameters, gave a value of 0.099 for the disagreement factor $R = \dfrac{\Sigma |K/F_0 - /F_c/|}{\Sigma |KF_0|}$ calculated on the basis of the 2218 observed reflections, K being a scale factor.

As shown in Fig. 1, the molecule is dimeric, with two oxygen atoms, belonging to two different acetate groups, one carbon atom of the norbornyl moiety and the vinyl end of the lateral chain of the hydrocarbon ligand forming an approximate square planar coordination about each Pd^{II} ion. The other Pd ion in the dimeric molecule completes the coordination at a distance of 2.96 Å, which can be compared with the distance of 2.94 Å found by Churchill and Mason[1] in a π-allylpalladium acetate dimer.
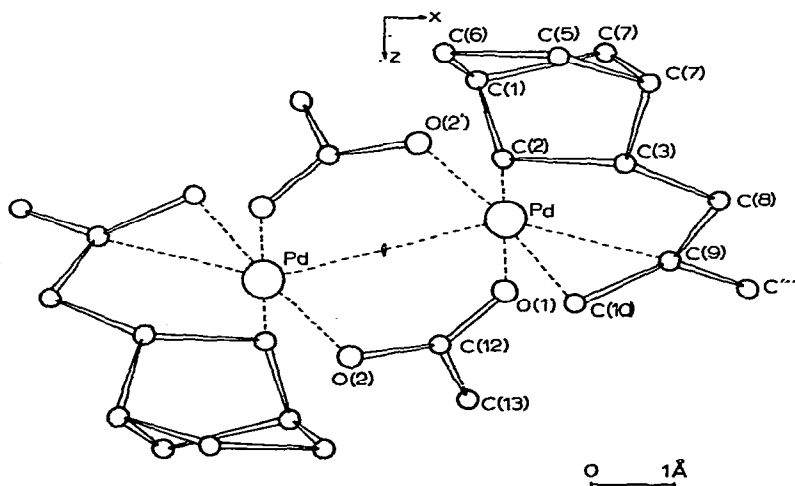

Fig. 1.

TABLE 1 [a]

Distances (Å)		Angles	
Pd–Pd	2.961	O(1)–Pd–O(2')	89.12°
Pd–O(1)	2.20	O(1)–Pd–C(10)	90.88°
Pd–O(2')	2.12	O(2')–Pd–C(2)	93.24°
Pd–C(10)	2.16	C(10)–Pd–C(2)	86.64°
Pd–C(2)	2.04	Pd–C(10)–C(9)	71.72°
Pd–C(9)	2.17	C(8)–C(9)–Pd	104.20°
		C(11)–C(9)–Pd	111.10°
		C(8)–C(9)–C(10)	122.62°
		C(11)–C(9)–C(10)	118.22°

[a]The estimated standard deviations, σ, are as follows:
σ(Pd–Pd) = 0.002 Å, σ(Pd–O) = 0.008 Å, σ(Pd–C) = 0.011 Å, σ(O–Pd–O) = 0.15°, σ(O–Pd–C) = 0.20°, σ(C–Pd–C) = 0.30°, σ(C–C–C) = 0.50°.

The more relevant bond distances and angles are given in the table. The Pd–O(1) and Pd–O(2') distances are significantly different. This difference may be attributed to the *trans* influence of the σ-bonded C(2) atom. The C(9)–C(10) bond distance is 1.40 Å (σ 0.016 Å) and this value is significantly different from both the double-bond and single-bond C–C standard distances.

The plane defined by atoms Pd, C(10) and C(9) is almost perpendicular to the coordination plane, while the C(9)–C(10) bond axis makes an angle of about 72° with the latter plane. This plane passes through the C=C vinyl bond much closer to atom C(10) than to atom C(9). To our knowledge, this geometry for a C=C bond coordinated to a metal atom in a square-planar complex of this type has never been reported before. It is relevant to recent discussions of the Chatt–Dewar model[2], an aspect which we shall consider in some detail in a later paper when the further refinement of the structural analysis is complete.

REFERENCES

1   M.R. Churchill and R. Mason, *Nature*, 204 (1964) 777.
2   *Disc. Faraday Soc.*, 47 (1969).