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.*

Acta Cryst. (1981). B**37**, 490

The crystal and molecular structure of 2-oxo-2-phenoxy-4*H*-1,3,2-benzodioxaphosphorin: errata.

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Abstract

E.s.d.'s of bond lengths and angles in Tables 3 and 4 of Gałdecki & Główka [*Acta Cryst.* (1978). B**34**, 160–163] are corrected. The mean σ's should be: 0·003 for P—O, 0·004 for O—C, 0·006 for C—C and 0·06 Å for C—H; 0·2 for O—P—O and P—O—C, 0·3 for O—C—C and 0·3° for C—C—C.

All relevant information is contained in the *Abstract.*

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Comparison of two independent structure determinations of (1–3-η-2-methylallyl)palladium chloride dimer.

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Abstract

Two independent structure analyses of (1–3-η-2-methylallyl)palladium chloride dimer are compared by means of half-normal probability plots. No systematic errors are detected in the derived atomic positions, while the thermal parameters differ significantly between the two studies; this can be attributed to absorption effects. [This work: $C_8H_{14}Cl_2Pd_2$, $P\bar{1}$, $a = 9{\cdot}266\ (9)$, $b = 6{\cdot}332\ (6)$, $c = 4{\cdot}985\ (4)$ Å, $\alpha = 92{\cdot}01\ (3)$, $\beta = 90{\cdot}77\ (3)$, $\gamma = 95{\cdot}94\ (5)°$; $R = 0{\cdot}035$ for 1155 reflections.]

Introduction

The crystal structure of the title compound was first reported and thoroughly discussed by Mason & Wheeler (1968) (hereinafter MW). The earlier work had been carried out using visual intensity estimates from film; the present study employs diffractometer X-ray data. Comparison of our results with those of MW gives some information on the precision and accuracy available from diffractometer data.

Experimental

Crystals are triclinic, space group $P\bar{1}$, with $a = 9{\cdot}266\ (9)$, $b = 6{\cdot}332\ (6)$, $c = 4{\cdot}985\ (4)$ Å, $\alpha = 92{\cdot}01\ (3)$, $\beta = 90{\cdot}77\ (3)$, $\gamma = 95{\cdot}94\ (5)°$; crystal size 0·22 × 0·16 × 0·06 mm. Intensity data within a Bragg limit of 27° were collected by the use of a Philips PW 1100 diffractometer (Mo $K\alpha$ radiation). The number of independent reflections was 1270; 1155 reflections

Table 1. *Coordinates* ($\times 10^4$) *and* U_{eq} ($\times 10^2$) *values of the non-hydrogen atoms*

$U_{eq} = (U_1 U_2 U_3)^{1/3}$, where U_1, U_2 and U_3 are the mean-square displacements along the principal axes of the thermal ellipsoids.

	x	y	z	U_{eq} (Å^2)
Pd	−1234 (1)	912 (1)	2191 (1)	3·5
Cl	1046 (2)	2313 (3)	403 (4)	4·4
C(1)	−1753 (10)	3424 (14)	4823 (16)	4·7
C(2)	−3018 (8)	2298 (13)	3806 (15)	4·2
C(3)	−3148 (9)	127 (14)	4219 (17)	5·1
C(4)	−3999 (10)	3296 (16)	1927 (20)	5·9

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

E.s.d.'s in parentheses take into account the accuracy of the cell dimensions.

Pd–Cl	2·401 (3)	Cl–Pd–Cl′	88·5 (0·1)
Pd–Cl′	2·401 (3)	Cl–Pd–C(1)	102·6 (0·2)
Pd–C(1)	2·124 (9)	Cl′–Pd–C(3)	101·0 (0·2)
Pd–C(2)	2·107 (8)	C(1)–Pd–C(3)	67·9 (0·3)
Pd–C(3)	2·077 (9)	Pd–Cl–Pd′	91·5 (0·1)
C(1)–C(2)	1·387 (11)	Pd–C(1)–C(2)	70·2 (0·5)
C(2)–C(3)	1·390 (12)	Pd–C(3)–C(2)	71·7 (0·5)
C(2)–C(4)	1·497 (13)	C(1)–C(2)–C(3)	115·3 (0·8)
		C(1)–C(2)–C(4)	120·9 (0·8)
		C(3)–C(2)–C(4)	122·6 (0·7)

Non-bonded distances ≤3·5 Å

C(1)···C(3)	2·347 (12)	Pd···C(4)	3·109 (10)
C(3)···C(4)	2·533 (14)	Cl···Cl′	3·350 (4)
C(1)···C(4)	2·510 (13)	Pd···Pd′	3·441 (2)

Dihedral angles (°)

Cl–Pd–Cl′/C(1)–C(2)–C(3)	111·6
C(1)–Pd–C(3)/C(1)–C(2)–C(3)	109·1

with $I \geq 3\sigma(I)$ were used in the refinement procedure. Starting parameters, scattering factors and refinement conditions were the same as those of MW. The final difference Fourier synthesis (spurious peaks less than 0·6 e Å^{-3}) permitted the location of the H atoms which were included in the structure factor calculation. The improvement in R was from 0·078 (MW) to 0·035. Table 1 lists final positional and thermal parameters and Table 2 shows bond distances and angles. The C–H distances are not reported; they are in the range 0·97–1·13 Å. The atom numbering scheme is the same as that of MW.

All calculations were carried out with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) on the CDC 6600/7600 computer of the Consorzio Interuniversitario Italia Nord-Orientale, Casalecchio (Bologna).*

Discussion

Table 3. *Values obtained from half-normal probability plots*

Values compared	Number of values	Range of δP	Slope	Intercept
Positional parameters	18	0·03–2·20	1·37	0·01
Distances between 2·4 and 5·0 Å	63	0·01–2·65	1·17	0·04
Intramolecular distances less 2·4 Å and angles	30	0·02–2·31	1·00	0·31

The accuracy of the structure has been improved by our determination, as evidenced by the value of R, the considerably lower standard deviations in coordinates and in bond distances and angles and by the location of the H atoms. It is apparent that the positional parameters from the two analyses are, in general, in good agreement. On the other hand, the differences between the thermal parameters are considerable and the U_{ii} values of the present study are systematically lower than those reported by MW. However, too much significance should not be attached to the values in either set, because of systematic errors in the data, mainly arising from neglect of absorption.

Table 3 details the data obtained from the half-normal probability plots (Abrahams & Keve, 1971); the last, including the distances less than 5·0 Å, is more generally useful for studies of molecular geometry (De Camp, 1973). The bond lengths and angles plot indicates minimal systematic error in one or both sets of results; furthermore, examination of the intercepts suggests that no systematic error is present either in the positional parameters or in the non-bonded distances, while the ≥1 slopes point to slight underestimation of the associated standard deviations.

In summary, the two data sets have given essentially the same positional description of the structure, the differences being adequately described by their e.s.d.'s, and the results are indeed typical of the material.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35718 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

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