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{2,4-Bis[1,1-dimethyl-2-(methylthio)ethyl]-3-furyl-*S*,*S*}chloropalladium(II), [Pd($C_{14}H_{23}OS_2$)Cl]

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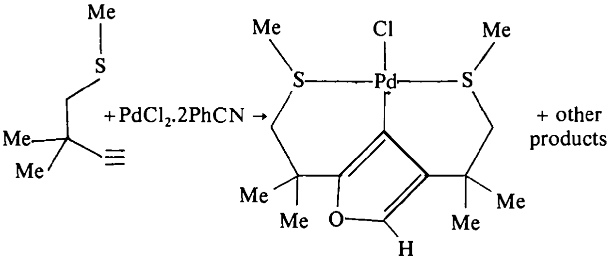
Abstract. $M_r = 413{\cdot}3$, monoclinic, $P2_1/n$, $a = 10{\cdot}332\,(3)$, $b = 9{\cdot}957\,(2)$, $c = 16{\cdot}189\,(5)$ Å, $\beta = 103{\cdot}16\,(2)°$, $V = 1621{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}69$ g cm^{-3}, $F(000) = 840$, $T = 293$ K, μ(Mo $K\alpha$) = 15·3 cm^{-1}, $\lambda = 0{\cdot}71069$ Å, $R = 0{\cdot}018$ for 2589 observed data. The Pd atom is four coordinate in a close to square-planar environment, with Pd—Cl 2·388 (1), Pd—S 2·299 (1) and 2·307 (1), and Pd—C 2·007 (2) Å. The two six-membered rings adopt different conformations, one a twist-boat and the other a twist-chair.

Introduction. The reaction of 2,2-dimethyl-3-butynyl methyl sulphide (1) with dichlorobis(benzonitrile)palladium results in a novel product, (2) (the title compound) along with four other products (McCrindle, Ferguson, Arsenault, McAlees & Parvez, 1983). The structure of (2) was determined by our X-ray analysis which shows it to be an entirely new type of product obtained from the oligomerization of the acetylene (1).



(1) (2)

Experimental. Yellow crystals from dichloromethane, 0·25 × 0·22 × 0·20 mm, CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, 25 reflections with θ in the range $10 < \theta < 15°$ used for measuring lattice constants; for data collection $2 < \theta < 25°$ (h −12–11, k 0–11, l 1–19), ω–2θ scans, ω-scan width $(0{\cdot}80 + 0{\cdot}35\tan\theta)°$; intensities of three reflections monitored every 3 h of exposure time showed no significant variation; Lp corrections, 2853 unique reflections, 2589 (90·7%) with $I > 3\sigma(I)$, where $\sigma^2(I) = S + 2B + [0{\cdot}04(S-B)]^2$, S = scan count and B = time-averaged background count; absorption correction was not considered necessary. Heavy-atom method using the *NRC Crystal Structure Package* (Larson & Gabe, 1978), refinement by block-diagonal least-squares calculations on F, H atoms from difference syntheses included at their idealized positions (C—H 0·95 Å) but not refined. Final $R = 0{\cdot}018$, $R_\omega = 0{\cdot}021$ for 2589 observed data, $R = 0{\cdot}021$ for all data, $\omega = 1/\sigma^2(F)$, GOF = 3·17, $\Delta\rho = \pm 0{\cdot}1$ e Å^{-3} in final difference map, maximum shift/e.s.d. = −0·30 for U_{23} of C(12), mean shift/e.s.d. = 0·07; atomic scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). Other computer programs used included *XANADU* (Roberts & Sheldrick, 1975) and *ORTEP* II (Johnson, 1976). Final fractional coordinates with mean isotropic temperature factors are in Table 1. Table 2 contains molecular dimensions.*

Discussion. The crystal structure contains discrete monomeric molecules (Fig. 1) separated by normal van der Waals distances. The Pd atom is four-coordinate in a close to square-planar environment; deviations of the

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* Lists of structure factors, anisotropic thermal parameters and the calculated hydrogen coordinates, as well as ring torsion angles, have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38524 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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atoms from the PdS_2ClC plane are Pd 0·023 (1), S(1) −0·113 (1), S(2) −0·115 (1), Cl 0·091 (1), C(1) 0·114 (2) Å. The furyl ring is planar, the six-membered ring Pd,S(1),C(12),C(13),C(3),C(1) has a twist-boat conformation and the other six-membered ring, Pd, S(2),C(1),C(2),C(23),C(22), a twist-chair conformation.

Table 1. *Final fractional coordinates for* (2) *with estimated standard deviations in parentheses, and the mean* B_{iso} (Å^2) [$8\pi^2(U_{11}U_{22}U_{33})^{1/3}$]

	x	y	z	B_{iso}
Pd	0·00954 (2)	0·03238 (2)	0·24608 (1)	1·823 (8)
Cl	0·15704 (6)	0·02052 (8)	0·38285 (4)	3·25 (4)
S(1)	0·18835 (6)	0·07426 (6)	0·18660 (4)	2·45 (3)
S(2)	−0·17090 (6)	−0·03498 (6)	0·29652 (4)	2·40 (3)
O	−0·3024 (2)	0·0505 (2)	0·0267 (1)	2·42 (8)
C(1)	−0·1188 (2)	0·0560 (2)	0·1337 (1)	1·95 (10)
C(2)	−0·2406 (2)	−0·0010 (2)	0·1051 (1)	2·05 (10)
C(3)	−0·1042 (2)	0·1497 (2)	0·0677 (1)	1·92 (10)
C(4)	−0·2158 (2)	0·1417 (2)	0·0066 (1)	2·32 (11)
C(11)	0·2958 (3)	0·1967 (3)	0·2501 (2)	3·92 (16)
C(12)	0·1408 (2)	0·1605 (2)	0·0858 (1)	2·42 (11)
C(13)	0·0111 (2)	0·2424 (2)	0·0686 (1)	2·17 (10)
C(14)	0·0154 (3)	0·3531 (2)	0·1347 (2)	2·93 (12)
C(15)	−0·0000 (3)	0·3070 (3)	−0·0192 (2)	3·23 (13)
C(21)	−0·1148 (3)	−0·1386 (3)	0·3885 (2)	4·07 (18)
C(22)	−0·2450 (2)	−0·1629 (2)	0·2209 (1)	2·57 (11)
C(23)	−0·3255 (2)	−0·1011 (3)	0·1385 (1)	2·46 (12)
C(24)	−0·4500 (3)	−0·0307 (3)	0·1546 (2)	3·60 (16)
C(25)	−0·3697 (3)	−0·2151 (3)	0·0748 (2)	3·57 (14)

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

Pd—Cl	2·388 (1)	C(1)—C(2)	1·362 (3)
Pd—S(1)	2·307 (1)	C(1)—C(3)	1·452 (3)
Pd—S(2)	2·299 (1)	C(2)—O	1·384 (3)
Pd—C(1)	2·007 (2)	C(3)—C(4)	1·340 (3)
S(1)—C(11)	1·804 (3)	C(4)—O	1·364 (3)
S(1)—C(12)	1·810 (2)	S(2)—C(21)	1·796 (3)
C(12)—C(13)	1·538 (3)	S(2)—C(22)	1·811 (2)
C(13)—C(14)	1·530 (3)	C(22)—C(23)	1·531 (3)
C(13)—C(15)	1·539 (3)	C(23)—C(24)	1·538 (4)
C(13)—C(3)	1·506 (3)	C(23)—C(25)	1·532 (4)
		C(23)—C(2)	1·507 (3)
Cl—Pd—S(1)	89·61 (3)	C(1)—C(3)—C(4)	106·8 (2)
Cl—Pd—S(2)	93·11 (3)	C(1)—C(3)—C(13)	127·0 (2)
Cl—Pd—C(1)	175·67 (7)	C(4)—C(3)—C(13)	126·2 (2)
S(1)—Pd—S(2)	172·75 (2)	C(3)—C(4)—O	111·4 (2)
S(1)—Pd—C(1)	91·54 (7)	C(2)—O—C(4)	106·0 (2)
S(2)—Pd—C(1)	86·24 (7)	S(1)—C(12)—C(13)	117·5 (2)
Pd—S(1)—C(11)	109·4 (1)	C(12)—C(13)—C(3)	109·4 (2)
Pd—S(1)—C(12)	112·5 (1)	C(12)—C(13)—C(14)	111·7 (2)
C(11)—S(1)—C(12)	101·5 (1)	C(12)—C(13)—C(15)	105·5 (2)
Pd—S(2)—C(21)	109·1 (1)	C(3)—C(13)—C(14)	110·2 (2)
Pd—S(2)—C(22)	102·5 (1)	C(3)—C(13)—C(15)	110·8 (2)
C(21)—S(2)—C(22)	99·4 (1)	C(14)—C(13)—C(15)	109·2 (2)
Pd—C(1)—C(2)	128·9 (2)	S(2)—C(22)—C(23)	111·6 (2)
Pd—C(1)—C(3)	125·8 (2)	C(22)—C(23)—C(2)	109·5 (2)
C(2)—C(1)—C(3)	105·1 (2)	C(22)—C(23)—C(24)	110·2 (2)
C(1)—C(2)—O	110·7 (2)	C(22)—C(23)—C(25)	107·9 (2)
C(1)—C(2)—C(23)	136·3 (2)	C(2)—C(23)—C(24)	109·7 (2)
O—C(2)—C(23)	113·0 (2)	C(2)—C(23)—C(25)	110·9 (2)
		C(24)—C(23)—C(25)	108·6 (2)

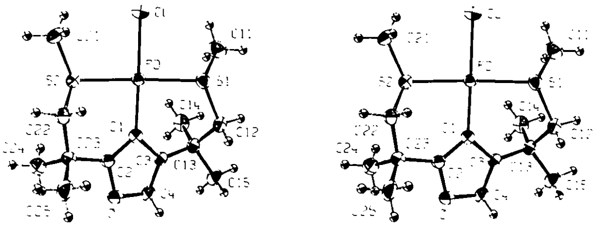

Fig. 1. Stereoview of the molecule (2) with the crystallographic numbering scheme.

The bond lengths at palladium [Pd—S 2·299 (1) and 2·307 (1), Pd—Cl 2·388 (1), Pd—C(sp^2) 2·007 (2) Å] agree well with values reported in a number of related structures (McCrindle, Ferguson, McAlees, Parvez & Stephenson, 1982; McCrindle, Ferguson, McAlees, Parvez & Roberts, 1982; Ferguson, McCrindle, McAlees, Parvez & Stephenson, 1983; Bahsoun, Dehand, Pfeffer, Zinsius, Bouaoud & Le Borne, 1979) with *e.g.* Pd—S 2·290–2·307 (1), Pd—Cl 2·369–2·394 (1), Pd—C(sp^3) 2·063–2·088 (6), Pd—C(sp^2) 1·92–2·00 (2) Å. The remaining bond lengths [C(sp^3)—S 1·796–1·811 (2), C(sp^3)—C(sp^3) 1·530–1·539 (3), C(sp^3)—C(sp^2) 1·506–1·507 (3) C(sp^2)=C(sp^2) 1·340–1·362 (3), C(sp^2)—C(sp^2) 1·452 (3), C(sp^2)—O 1·364–1·384 (3) Å] have normal values.

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