Table 1. *Final positional parameters of the non-H atoms with e.s.d.'s in parentheses*

| | $x$ | $y$ | $z$ | $B_{eq}$(Å$^2$) |
|---|---|---|---|---|
| Ru | 0·12557 (2) | 0·45800 (5) | 0·33090 (4) | 1·703 (7) |
| Cl(1) | 0·08912 (8) | 0·4047 (2) | 0·5099 (2) | 3·45 (4) |
| Cl(2) | 0·20048 (7) | 0·4050 (2) | 0·4194 (2) | 3·00 (3) |
| S(1) | 0·12788 (8) | 0·6568 (2) | 0·3854 (2) | 2·80 (3) |
| S(2) | 0·05397 (6) | 0·4790 (2) | 0·2446 (2) | 2·43 (3) |
| S(3) | 0·16670 (6) | 0·4915 (2) | 0·1681 (2) | 2·12 (3) |
| S(4) | 0·13014 (7) | 0·1614 (2) | 0·3617 (1) | 2·11 (3) |
| O(1) | 0·1412 (2) | 0·7525 (6) | 0·3023 (6) | 4·3 (1) |
| O(2) | 0·0361 (2) | 0·6023 (6) | 0·2146 (6) | 4·6 (1) |
| O(3) | 0·1417 (2) | 0·5389 (5) | 0·0655 (4) | 2·8 (1) |
| O(4) | 0·1206 (2) | 0·2689 (4) | 0·2801 (4) | 2·40 (9) |
| C(11) | 0·0738 (4) | 0·7077 (8) | 0·4495 (9) | 5·0 (2) |
| C(12) | 0·1694 (5) | 0·6758 (9) | 0·500 (1) | 6·5 (3) |
| C(21) | 0·0083 (3) | 0·4039 (9) | 0·3240 (9) | 4·2 (2) |
| C(22) | 0·0503 (3) | 0·388 (1) | 0·1191 (7) | 4·2 (2) |
| C(31) | 0·1963 (3) | 0·3533 (8) | 0·1255 (7) | 3·3 (2) |
| C(32) | 0·2173 (3) | 0·5872 (8) | 0·1883 (8) | 3·5 (2) |
| C(41) | 0·1779 (3) | 0·0792 (8) | 0·2997 (9) | 4·0 (2) |
| C(42) | 0·0851 (3) | 0·0560 (7) | 0·3228 (8) | 3·3 (2) |

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as:

$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table 2. *Bond lengths* (Å) *and angles* (°) *with their e.s.d.'s*

| | | | |
|---|---|---|---|
| Ru–Cl(1) | 2·413 (2) | S(1)–C(11) | 1·793 (11) |
| Ru–Cl(2) | 2·432 (2) | S(1)–C(12) | 1·804 (13) |
| Ru–S(1) | 2·248 (2) | S(2)–C(21) | 1·789 (10) |
| Ru–S(2) | 2·279 (2) | S(2)–C(22) | 1·779 (9) |
| Ru–S(3) | 2·269 (2) | S(3)–C(31) | 1·787 (9) |
| Ru–O(4) | 2·138 (4) | S(3)–C(32) | 1·785 (9) |
| S(1)–O(1) | 1·473 (7) | O(4)–S(4) | 1·533 (5) |
| S(2)–O(2) | 1·472 (7) | S(4)–C(41) | 1·777 (9) |
| S(3)–O(3) | 1·489 (6) | S(4)–C(42) | 1·772 (8) |
| Cl(1)–Ru–Cl(2) | 86·8 (1) | Ru–S(3)–C(31) | 110·1 (3) |
| Cl(1)–Ru–S(1) | 89·6 (1) | Ru–S(3)–C(32) | 113·1 (3) |
| Cl(1)–Ru–S(2) | 91·8 (1) | Ru–S(1)–O(1) | 119·5 (3) |
| Cl(1)–Ru–S(3) | 173·1 (1) | Ru–S(2)–O(2) | 120·3 (3) |
| Cl(1)–Ru–O(4) | 89·2 (1) | Ru–S(3)–O(3) | 119·6 (2) |
| Cl(2)–Ru–S(1) | 94·5 (1) | C(11)–S(1)–C(12) | 101·9 (5) |
| Cl(2)–Ru–S(2) | 172·0 (1) | C(21)–S(2)–C(22) | 97·9 (5) |
| Cl(2)–Ru–S(3) | 87·2 (1) | C(31)–S(3)–C(32) | 98·4 (4) |
| Cl(2)–Ru–O(4) | 87·2 (1) | C(11)–S(1)–O(1) | 106·3 (4) |
| S(1)–Ru–S(2) | 93·3 (1) | C(12)–S(1)–O(1) | 104·4 (5) |
| S(1)–Ru–S(3) | 94·1 (1) | C(21)–S(2)–O(2) | 106·7 (4) |
| S(1)–Ru–O(4) | 177·9 (2) | C(22)–S(2)–O(2) | 106·6 (4) |
| S(2)–Ru–S(3) | 93·8 (1) | C(31)–S(3)–O(3) | 106·6 (4) |
| S(2)–Ru–O(4) | 85·0 (2) | C(32)–S(3)–O(3) | 106·8 (4) |
| S(3)–Ru–O(4) | 87·2 (1) | Ru–O(4)–S(4) | 122·8 (3) |
| Ru–S(1)–C(11) | 112·9 (3) | O(4)–S(4)–C(41) | 105·0 (4) |
| Ru–S(1)–C(12) | 110·0 (4) | O(4)–S(4)–C(42) | 101·6 (4) |
| Ru–S(2)–C(21) | 111·5 (3) | C(41)–S(4)–C(42) | 96·9 (4) |
| Ru–S(2)–C(22) | 111·5 (3) | | |

**Related literature.** The structure represents a second modification of $(Me_2SO)_4RuCl_2$ with interatomic parameters in essential agreement with those of the previously reported monoclinic, $P2_1/n$, modification (Mercer & Trotter, 1975).

Financial support from CNR (Rome) and ICTP (Trieste) to WMA is acknowledged. Crystals were kindly supplied by Dr E. Alessio.

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# A Chloro-Bridged Palladium Dimer: *trans*-Di-$\mu$-chloro-bis[(2,2-dimethyl-2-phenylethyl)-(triphenylphosphine)palladium] Bis(dichloromethylate)

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(*Received* 21 *November* 1986; *accepted* 13 *January* 1987)

**Abstract.** $[Pd_2Cl_2\{(C_6H_5)_3P\}_2(C_{10}H_{13})_2].2CH_2Cl_2$, $M_r$ = 1244·6, triclinic, $P\bar{1}$, $a$ = 9·361 (2), $b$ = 12·982 (3), $c$ = 13·873 (3) Å, $\alpha$ = 114·13 (1), $\beta$ = 103·55 (1), $\gamma$ = 99·17 (1)°, $V$ = 1432·7 (5) Å$^3$, $Z$ = 1, $D_x$ = 1·442 g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71073 Å, $\mu$ = 9·9 cm$^{-1}$, $F(000)$ = 632, 296 K, $R_F$ = 3·59% for 3807 reflections with $F_o \geq 3\sigma(F_o)$ and 259 parameters. The compound is a chlorine-bridged *trans* dimer centered on a site of inversion. The coordination geometry is approximately square-planar about Pd with asymmetric chloro bridges; in keeping with expected *trans*-labilization trends, the Pd–Cl bond *trans* to the alkyl group, 2·416 (1) Å, is considerably shorter than that *trans* to the phosphine, 2·494 (1) Å.

**Experimental.** Yellow block-shaped crystals (0·30 × 0·35 × 0·40 mm) were recrystallized from $CH_2Cl_2$. Nicolet $R3m/\mu$, Wyckoff scans, lattice parameters from

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0108-2701/87/071427-02$01.50

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Pd | 994·8 (3) | 829·2 (3) | 1491·6 (2) | 30·4 (1) |
| P | 2451 (1) | 420 (1) | 2746 (1) | 33 (1) |
| Cl(1) | −1115 (1) | 892 (1) | 147 (1) | 42 (1) |
| Cl(2) | 6547 (7) | 3902 (4) | 9430 (4) | 273 (4) |
| Cl(3) | 5507 (7) | 2980 (5) | 10689 (5) | 300 (6) |
| C(1) | 963 (5) | 2401 (3) | 2677 (3) | 36 (2) |
| C(2) | 1961 (5) | 3494 (4) | 2725 (4) | 41 (2) |
| C(3) | 3661 (5) | 3571 (4) | 3160 (5) | 61 (3) |
| C(4) | 1648 (7) | 3488 (4) | 1587 (4) | 61 (3) |
| C(5) | 6155 (15) | 2694 (9) | 9600 (11) | 179 (10) |
| C(11) | 97 (3) | 4662 (3) | 3235 (3) | 57 (3) |
| C(12) | −292 | 5607 | 3955 | 72 (4) |
| C(13) | 799 | 6449 | 4976 | 79 (4) |
| C(14) | 2279 | 6346 | 5278 | 71 (3) |
| C(15) | 2668 | 5401 | 4559 | 58 (2) |
| C(16) | 1577 | 4559 | 3537 | 44 (2) |
| C(21) | 1491 (3) | 1077 (3) | 4595 (2) | 49 (2) |
| C(22) | 1613 | 1751 | 5709 | 61 (3) |
| C(23) | 2984 | 2609 | 6466 | 67 (3) |
| C(24) | 4232 | 2793 | 6109 | 67 (3) |
| C(25) | 4109 | 2119 | 4995 | 53 (2) |
| C(26) | 2739 | 1261 | 4238 | 39 (2) |
| C(31) | 4743 (3) | 639 (3) | 1828 (2) | 47 (2) |
| C(32) | 6191 | 644 | 1725 | 60 (3) |
| C(33) | 7270 | 437 | 2454 | 68 (3) |
| C(34) | 6902 | 225 | 3287 | 72 (4) |
| C(35) | 5454 | 219 | 3390 | 59 (3) |
| C(36) | 4375 | 426 | 2661 | 38 (2) |
| C(41) | 43 (3) | −1660 (3) | 1663 (3) | 55 (2) |
| C(42) | −702 | −2797 | 1429 | 72 (3) |
| C(43) | 13 | −3351 | 1993 | 73 (3) |
| C(44) | 1474 | −2767 | 2792 | 80 (4) |
| C(45) | 2219 | −1629 | 3027 | 67 (3) |
| C(46) | 1504 | −1076 | 2462 | 38 (2) |

Equivalent isotropic $U$ defined as one-third of the trace of the orthogonalized $U_{ij}$ tensor.

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

| | | | |
|---|---|---|---|
| Pd–P | 2·246 (1) | Pd–Cl(1) | 2·416 (1) |
| Pd–C(1) | 2·041 (4) | Pd–Cl(1′) | 2·494 (1) |
| P–C(26) | 1·830 (3) | P–C(36) | 1·831 (3) |
| P–C(46) | 1·837 (4) | Cl(1)–Pd′ | 2·494 (1) |
| Cl(2)–C(5) | 1·675 (16) | Cl(3)–C(5) | 1·683 (18) |
| C(1)–C(2) | 1·538 (7) | C(2)–C(3) | 1·535 (7) |
| C(2)–C(4) | 1·533 (8) | C(2)–C(16) | 1·544 (5) |
| P–Pd–Cl(1) | 164·5 (1) | P–Pd–C(1) | 94·0 (1) |
| Cl(1)–Pd–C(1) | 89·4 (1) | P–Pd–Cl(′) | 93·5 (1) |
| Cl(1)–Pd–Cl(1′) | 85·2 (1) | C(1)–Pd–Cl(1′) | 169·7 (2) |
| Pd–P–C(26) | 120·3 (1) | Pd–P–C(36) | 114·0 (1) |
| C(26)–P–C(36) | 105·3 (1) | Pd–P–C(46) | 107·9 (1) |
| C(26)–P–C(46) | 101·4 (2) | C(36)–P–C(46) | 106·6 (2) |
| Pd–Cl(1)–Pd′ | 94·8 (1) | Pd–C(1)–C(2) | 114·7 (3) |
| C(1)–C(2)–C(3) | 109·4 (4) | C(1)–C(2)–C(4) | 113·6 (3) |
| C(3)–C(2)–C(4) | 107·7 (5) | C(1)–C(2)–C(16) | 105·7 (4) |
| C(3)–C(2)–C(16) | 111·1 (3) | C(4)–C(2)–C(16) | 109·3 (4) |
| Cl(2)–C(5)–Cl(3) | 110·0 (7) | C(2)–C(16)–C(11) | 118·5 (2) |
| C(2)–C(16)–C(15) | 121·5 (2) | P–C(26)–C(21) | 117·5 (1) |
| P–C(26)–C(25) | 122·3 (1) | P–C(36)–C(31) | 117·9 (1) |
| P–C(36)–C(35) | 122·1 (1) | P–C(46)–C(41) | 118·8 (1) |
| P–C(46)–C(45) | 121·2 (1) | | |

25 reflections, $20 \le 2\theta \le 25°$, least-squares fit, absorption correction unnecessary ($\mu = 9·9$ cm$^{-1}$, less than 10% variation in $\psi$ scans, uniform crystal size), $2\theta_{max} = 48°$ ($h = \pm 11$, $k = \pm 15$, $l = +16$), three standard reflections ($\bar{4}\bar{1}7$, $\bar{2}\bar{6}1$, $\bar{5}\bar{1}4$), 10% linear decay, 4693 reflections collected, 4482 unique, $R_{int} = 2·19\%$, 675 unobserved reflections, 3807 reflections with $F_o \ge 3\sigma(F_o)$, Patterson solution, refinement on $F$ for 259 parameters, phenyls constrained to fit rigid hexagons [$d$(C–C) = 1·395 Å], all non-hydrogen atoms anisotropic, hydrogen atoms calculated and fixed in idealized positions [$d$(C–H) = 0·96 Å, $U = 1·2\ U_{iso}$ of attached C], $R_F = 3·59\%$, $wR_F = 4·09\%$, $S = 1·229$, $w^{-1} = \sigma^2(F_o) + g F_o^2$, $g = 0·001$, $(\Delta/\sigma)_{max} = 0·083$, $(\Delta\rho)_{max} = 0·66$, $(\Delta\rho)_{min} = -0·80$ e Å$^{-3}$, atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computer programs, Sheldrick (1984). Atomic parameters are given in Table 1, bond distances and angles in Table 2. Fig. 1 is a view of the palladium complex with atom numbering.

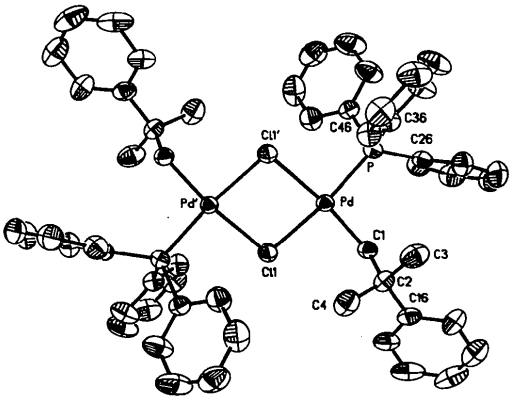


Fig. 1. Molecular structure and labeling scheme for the palladium complex. Thermal ellipsoids are drawn at the 40% probability level.

**Related literature.** There are many examples of asymmetric di-$\mu$-chloro-bridged palladium compounds (Constable, McDonald, Sawkins & Shaw, 1980; Alyea, Dias, Ferguson, McAlees, McCrindle & Roberts, 1977; Zocchi & Tieghi, 1979).

Crystals of the title compound were the gift of Dr Richard Heck, University of Delaware.

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43724 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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