Crystal structure of monochloro-*syn*-bis[*meso*-ethane-1,2-diylbis(methylphenylphosphane)]nickel(II) monochloride, $[Ni\{(C_6H_5)(CH_3)P(C_2H_4)P(CH_3)(C_6H_5)\}_2Cl]Cl$

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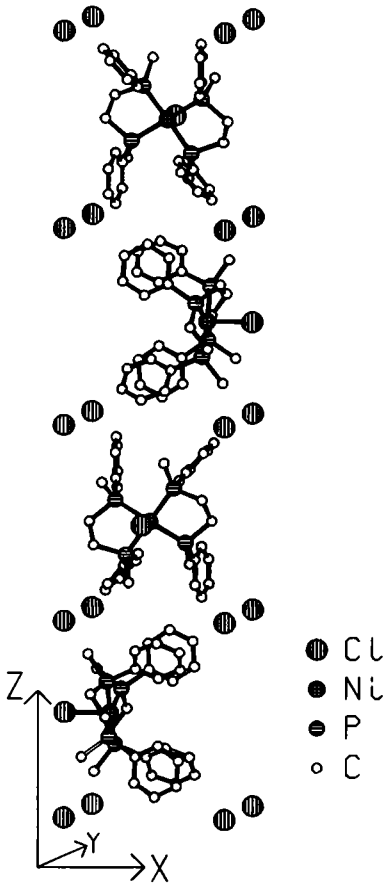

Source of material: Preparation described in ref. 1.
The complexes such as the title compound were synthesized by E. Müller and coworkers using several metal atoms : Ru (see ref. 2), Os (see ref. 3), Pd (see ref. 4), Rh, Pt, Co and Ni (see ref. 1). The Ni-compound was only obtained in form of aggregates of small single crystals. A fragment of about 0.1 mm diameter was mounted on a goniometer head and still turned out to be composed of three individuals. The reflection intensities of two of them were evaluated using the twin integration software of the Stoe IPDS (imaging plate diffraction system). After omitting 9156 overlapped reflexions about 28000 intensity data from each crystal were stored, scaled and merged ($R_{int} = 0.13$) to give 2558 unique reflections.

The Ni-compound differs from the corresponding Ru-compound (see ref. 5) mainly in that only one chlorine atom is bonded to the central atom, resulting in a distorted square pyramidal coordination polyhedron for Ni. The second chlorine atom occupies voids in the packing, which has a remarkably high symmetry. The figure shows four complex cations forming a 4_1 screw, each cation lies on a twofold axis. The nonbonded chlorine atoms reside on $\bar{4}$ axes, four of them surrounding the 4_1 axis as shown. The Ni–Cl distance is 245 pm, the Ni–P distances are 222 pm and 224 pm.

$C_{32}H_{40}Cl_2NiP_4$, tetragonal, $I4_1/acd$ (No. 142), $a = 17.38(1)$ Å, $c = 42.53(1)$ Å, $V = 12847$ Å^3, $Z = 16$, $R(F) = 0.043$, $R_w(F^2) = 0.067$.

Table 1. Parameters used for the X-ray data collection

Crystal:	ruby crystal of irregular shape, size 0.1 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.90 cm^{-1}
Diffractometer:	Stoe IPDS
Scan mode:	180 exposures, $\Delta\varphi = 1.0°$
$T_{measurement}$:	300 K
$2\theta_{max}$:	48.3°
$N(hkl)_{unique}$:	2558
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	179
Programs:	SHELXS-86, SHELXL-93, PLATON, SCHAKAL

Table 2. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{iso}
H(1)	32*g*	−0.0107(3)	0.0427(3)	0.1806(1)	0.050(4)
H(2)	32*g*	0.0608(3)	0.0125(3)	0.1618(1)	0.050(4)
H(3)	32*g*	0.0385(3)	0.0998(3)	0.1603(1)	0.050(4)
H(4)	32*g*	0.0731(3)	0.2063(3)	0.2041(1)	0.050(4)
H(5)	32*g*	0.0142(3)	0.1660(3)	0.2269(1)	0.050(4)
H(6)	32*g*	0.2573(3)	0.0288(3)	0.2197(1)	0.050(4)
H(7)	32*g*	0.3767(3)	0.0610(4)	0.2000(1)	0.050(4)
H(8)	32*g*	0.3880(4)	0.1509(4)	0.1601(1)	0.050(4)
H(9)	32*g*	0.2789(4)	0.2049(3)	0.1390(1)	0.050(4)
H(10)	32*g*	0.1598(4)	0.1710(3)	0.1567(1)	0.050(4)
H(11)	32*g*	−0.0129(3)	0.1309(3)	0.2897(1)	0.050(4)
H(12)	32*g*	0.0395(3)	0.1900(3)	0.3076(1)	0.050(4)
H(13)	32*g*	0.0326(3)	0.1051(3)	0.3197(1)	0.050(4)
H(14)	32*g*	0.0984(3)	0.2372(3)	0.2587(1)	0.050(4)
H(15)	32*g*	0.1704(3)	0.2001(3)	0.2421(1)	0.050(4)
H(16)	32*g*	0.1488(4)	0.1191(3)	0.3444(1)	0.050(4)
H(17)	32*g*	0.2585(4)	0.1324(3)	0.3734(1)	0.050(4)
H(18)	32*g*	0.3760(4)	0.1506(4)	0.3493(2)	0.050(4)
H(19)	32*g*	0.3848(3)	0.1489(3)	0.2953(2)	0.050(4)
H(20)	32*g*	0.2758(3)	0.1298(3)	0.2650(1)	0.050(4)

Table 3. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni(1)	16*e*	0.09911(5)	0	1/4	0.0256(6)	0.0316(6)	0.0304(6)	0	0	–0.0042(6)
Cl(1)	16*e*	–0.0419(1)	0	1/4	0.027(1)	0.064(2)	0.050(1)	0	0	–0.018(1)
Cl(2)	8*a*	0	1/4	3/8	0.081(2)	U_{11}	0.039(2)	0	0	0
Cl(3)	8*b*	0	1/4	1/8	0.060(2)	U_{11}	0.106(3)	–0.020(2)	0	0
P(1)	32*g*	0.10182(9)	0.07433(8)	0.20758(4)	0.031(1)	0.036(1)	0.0328(9)	0.0049(8)	–0.0034(9)	–0.0018(8)
P(2)	32*g*	0.11483(8)	0.10960(8)	0.27691(3)	0.029(1)	0.036(1)	0.032(1)	–0.0010(8)	–0.0010(8)	–0.0071(8)
C(1)	32*g*	0.0403(3)	0.0551(3)	0.1735(1)	0.043(4)	0.050(4)	0.047(4)	–0.006(3)	–0.017(4)	–0.004(3)
C(2)	32*g*	0.0679(3)	0.1687(3)	0.2208(1)	0.043(4)	0.029(4)	0.034(4)	0.011(3)	–0.004(3)	0.006(3)
C(3)	32*g*	0.1951(3)	0.0964(3)	0.1906(1)	0.024(4)	0.033(4)	0.037(4)	–0.000(3)	0.002(3)	–0.005(3)
C(4)	32*g*	0.2612(3)	0.0640(3)	0.2033(1)	0.036(4)	0.034(3)	0.032(4)	0.005(3)	–0.005(3)	0.004(3)
C(5)	32*g*	0.3328(3)	0.0837(4)	0.1916(1)	0.027(4)	0.053(5)	0.072(5)	0.003(3)	–0.009(4)	0.000(4)
C(6)	32*g*	0.3398(4)	0.1370(4)	0.1677(1)	0.044(5)	0.067(5)	0.041(4)	–0.012(4)	0.004(4)	0.008(4)
C(7)	32*g*	0.2744(4)	0.1690(3)	0.1551(1)	0.052(5)	0.058(5)	0.033(4)	–0.010(4)	0.001(4)	0.011(3)
C(8)	32*g*	0.2033(4)	0.1493(3)	0.1658(1)	0.042(4)	0.053(5)	0.034(4)	0.002(4)	–0.000(3)	0.010(4)
C(9)	32*g*	0.0340(3)	0.1371(3)	0.3014(1)	0.038(4)	0.042(4)	0.046(4)	0.003(3)	0.000(3)	–0.012(3)
C(10)	32*g*	0.1179(3)	0.1907(3)	0.2488(1)	0.044(4)	0.030(3)	0.043(3)	0.007(3)	–0.003(4)	–0.006(3)
C(11)	32*g*	0.1993(3)	0.1237(3)	0.3012(1)	0.029(4)	0.023(4)	0.040(4)	–0.000(3)	–0.008(3)	–0.006(3)
C(12)	32*g*	0.1960(4)	0.1245(3)	0.3342(1)	0.041(4)	0.065(5)	0.040(5)	–0.012(3)	–0.003(3)	–0.002(4)
C(13)	32*g*	0.2614(4)	0.1332(3)	0.3516(1)	0.060(5)	0.090(6)	0.035(4)	–0.009(5)	–0.007(4)	0.001(4)
C(14)	32*g*	0.3320(4)	0.1433(4)	0.3372(2)	0.059(6)	0.057(5)	0.054(5)	–0.003(4)	–0.027(4)	0.014(5)
C(15)	32*g*	0.3373(3)	0.1424(3)	0.3050(2)	0.015(4)	0.050(4)	0.082(5)	–0.007(3)	–0.008(4)	0.008(4)
C(16)	32*g*	0.2718(3)	0.1317(3)	0.2868(1)	0.042(4)	0.040(4)	0.036(4)	–0.005(3)	0.005(3)	–0.002(3)

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