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(η^5-Cyclopentadienyl)(thiocyanato-*N*)bis(triphenylphosphine)ruthenium Dichloromethane Solvate

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Abstract. $[Ru(\eta^5\text{-}C_5H_5)(NCS)(C_{18}H_{15}P)_2].CH_2Cl_2$, $M_r = 833.7$, orthorhombic, $P2_1nb$ (equivalent positions: x, y, z; $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; $x, \frac{1}{2}+y, \frac{1}{2}-z$; $\frac{1}{2}+x, -y, -z$), $a = 8.887$ (2), $b = 20.495$ (4), $c = 20.780$ (4) Å, $V = 3785.0$ (14) Å^3, $Z = 4$, $D_x = 1.463$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 7.16$ cm^{-1}, $F(000) = 1704$, $T = 231$ (1) K, $R = 0.0709$ for 8644 unique reflections. The molecule has pseudo-octahedral symmetry with the cyclopentadienyl group occupying three sites in a facial arrangement. The thiocyanato ligand is N bound and the angle at the N atom is 168.4 (5)° while the ligand itself is linear with the angle at the C atom being 178.7 (6)°.

Experimental. Obtained by the method of Wilczewski, Bochenska & Biernat (1981) and recrystallized by slow diffusion of a layer of diethyl ether into a dichloromethane solution. Bright yellow block, 0.25 × 0.20 × 0.30 mm, mounted on a glass fibre. Nicolet *R3m/V* diffractometer, graphite monochromator, unit cell from 30 reflections ($17.19 < 2\theta < 29.87°$). 9753 total reflections measured, 8644 unique ($R_{int} = 2.09\%$) with 5977 observed [$F > 6.0\sigma(F)$], collected between $3.0 < 2\theta < 55.0°$, $-2 \le h \le 11$, $0 \le k \le 26$, $0 \le l \le 27$, using θ–2θ scans, variable scan speed 2.93–14.65° min^{-1} in ω, scan range 1.20° plus $K\alpha$ separation. Data were corrected for Lorentz and polarization effects. Neutral-atom scattering factors and complex anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). During the collection of the intensity data, three check reflections were monitored every 197 reflections and indicated no significant deterioration.

Calculations were performed using *SHELXTL-Plus* on a MicroVAX II computer (Sheldrick, 1987). The Ru, S and both P atoms were located from a Patterson synthesis and the Ru atom was constrained to (0, x, z). Subsequent difference maps located all remaining non-H atoms. All non-H atoms were refined with anisotropic thermal parameters and H atoms were placed in calculated positions and allowed to ride. Empirical absorption correction $t_{min}/t_{max} = 0.670/0.728$. Full-matrix least-squares refinement on F using a weighting scheme $\{w = [\sigma^2(F) + 0.0017F^2]^{-1}\}$ converged to give $R = 0.0424$, $wR = 0.0539$ ($R = 0.0709$, $wR = 0.0639$ for all data) and $S = 0.98$ for data/parameters = 13.3. No shift in the

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3)

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

	x	y	z	U_{eq}
Ru(1)	0	2212 (1)	1534 (1)	23 (1)
C(1)	−2198 (7)	2362 (3)	2023 (3)	44 (2)
C(2)	−1205 (7)	2038 (3)	2445 (3)	39 (2)
C(3)	−814 (7)	1421 (3)	2170 (3)	42 (2)
C(4)	−1582 (7)	1374 (3)	1574 (3)	44 (2)
C(5)	−2419 (8)	1950 (4)	1477 (3)	48 (2)
N(1)	−267 (6)	2896 (2)	811 (2)	32 (2)
C(6)	−568 (7)	3328 (3)	484 (3)	34 (2)
S(1)	−971 (3)	3954 (1)	28 (1)	63 (1)
C(7)	8557 (14)	5455 (5)	904 (5)	103 (5)
Cl(1)	7154 (3)	5767 (1)	1340 (1)	80 (1)
Cl(2)	10381 (3)	5694 (1)	1182 (2)	105 (1)
P(1)	1789 (2)	1609 (1)	980 (1)	22 (1)
C(11)	2794 (6)	1045 (3)	1511 (3)	26 (2)
C(12)	4178 (6)	1212 (3)	1786 (3)	29 (2)
C(13)	4854 (9)	809 (3)	2242 (3)	40 (2)
C(14)	4144 (7)	251 (3)	2445 (3)	40 (2)
C(15)	2749 (8)	84 (3)	2183 (3)	42 (2)
C(16)	2089 (7)	469 (3)	1723 (3)	33 (2)
C(21)	3258 (6)	1962 (3)	465 (3)	26 (2)
C(22)	2937 (8)	2533 (3)	136 (3)	42 (2)
C(23)	3932 (10)	2779 (4)	−315 (4)	63 (3)
C(24)	5277 (9)	2479 (4)	−433 (3)	52 (3)
C(25)	5607 (7)	1904 (4)	−117 (3)	45 (2)
C(26)	4613 (6)	1643 (3)	325 (3)	37 (2)
C(31)	1012 (6)	1062 (3)	354 (3)	26 (2)
C(32)	−228 (7)	1260 (3)	−3 (3)	31 (2)
C(33)	−678 (7)	900 (3)	−534 (3)	42 (2)
C(34)	79 (9)	345 (3)	−711 (3)	42 (2)
C(35)	1288 (7)	137 (3)	−354 (3)	42 (2)
C(36)	1771 (8)	494 (3)	176 (3)	39 (2)
P(2)	1584 (2)	2956 (1)	2035 (1)	24 (1)
C(41)	359 (6)	3542 (3)	2457 (3)	28 (2)
C(42)	−748 (7)	3882 (3)	2123 (3)	40 (2)
C(43)	−1753 (8)	4295 (3)	2434 (4)	50 (2)
C(44)	−1700 (9)	4367 (4)	3094 (4)	55 (3)
C(45)	−620 (9)	4022 (4)	3431 (3)	53 (2)
C(46)	406 (7)	3620 (3)	3128 (3)	42 (2)
C(51)	2846 (7)	3469 (3)	1550 (3)	29 (2)
C(52)	2445 (8)	4070 (3)	1299 (3)	39 (2)
C(53)	3468 (8)	4429 (3)	932 (3)	46 (2)
C(54)	4874 (10)	4189 (3)	807 (3)	47 (2)
C(55)	5294 (7)	3591 (3)	1052 (3)	48 (2)
C(56)	4302 (7)	3234 (3)	1421 (3)	40 (2)
C(61)	2855 (7)	2679 (3)	2670 (3)	32 (2)
C(62)	3997 (7)	3083 (3)	2906 (3)	43 (2)
C(63)	4930 (13)	2863 (3)	3403 (3)	51 (2)
C(64)	4721 (8)	2248 (4)	3665 (3)	51 (3)
C(65)	3590 (8)	1860 (3)	3445 (3)	46 (2)
C(66)	2669 (7)	2063 (3)	2941 (3)	36 (2)

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Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

Ru(1)—C(1)	2.224 (7)	Ru(1)—C(2)	2.205 (6)
Ru(1)—C(3)	2.213 (7)	Ru(1)—C(4)	2.221 (7)
Ru(1)—C(5)	2.219 (7)	Ru(1)—N(1)	2.070 (4)
Ru(1)—P(1)	2.318 (1)	Ru(1)—P(2)	2.323 (2)
N(1)—C(6)	1.146 (7)	C(6)—S(1)	1.634 (6)
N(1)—Ru(1)—P(1)	94.6 (1)	N(1)—Ru(1)—P(2)	87.1 (1)
P(1)—Ru(1)—P(2)	99.0 (1)	Ru(1)—N(1)—C(6)	168.4 (5)
N(1)—C(6)—S(1)	178.7 (6)		

final refinement (maximum $\Delta/\sigma = 0.005$, mean $\Delta/\sigma = 0.000$). The largest unassigned electron density peaks (0.87, -0.90 e Å^{-3}) were adjacent to the Ru atom.

Fractional atomic coordinates are given in Table 1 and selected bond distances and bond angles in Table 2.* Fig. 1 shows thermal ellipsoids drawn at the 30% probability level for the title compound.

Related literature. Only one previous structural characterization of a Ru thiocyanate-*N* complex has been reported (Herber, Nan, Potenza, Schugar & Bino, 1989). In the complex bis[(2,2′-bipyridyl-*N*,*N*′)thiocyanato-*N*]ruthenium the Ru—N bond length is 2.055 (5) Å, the angle at the ligand N atom is 168.2 (5)°, and the angle N—C—S is 177.5 (6)°.

* Lists of structure factors, anisotropic displacement parameters, bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54663 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HE0034]

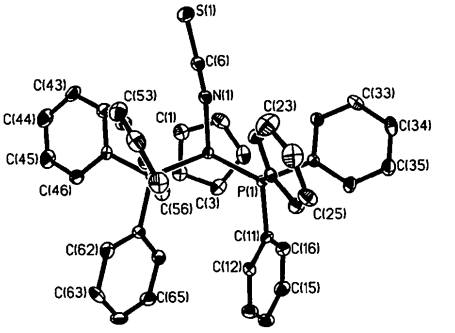

Fig. 1. Plot showing 30% thermal ellipsoids for the title compound.

The angles are consistent with the Ru centre in this complex and in the title compound being a good π donor (Drew, bin-Othman & Nelson, 1976).

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Structure of *cyclo*-Tris{dichloro-μ-[methylenebis(dimethylphosphine)-κP:κP]-[methylenebis(dimethylphosphine)-$\kappa^2 P$]ruthenium(II)}–Ethanol–Toluene (1/1/1)

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Abstract. $[Ru_3(\mu\text{-}C_5H_{14}P_2)_3(C_5H_{14}P_2)_3Cl_6].C_7H_8.C_2H_6O$, $M_r = 1469.9$, triclinic, $P\bar{1}$, $a = 13.208$ (2), $b = 13.316$ (3), $c = 20.828$ (7) Å, $\alpha = 77.04$ (2), $\beta = 72.53$ (2), $\gamma = 72.35$ (2)°, $V = 3294$ (1) Å^3, $Z = 2$, $D_x = 1.48$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 1.13$ mm^{-1}, $F(000) = 1508$, $T = 298$ K, final $R = 0.057$, $wR = 0.048$ for 8141 reflections with $I > 4\sigma(I_o)$ and 614 variable parameters. The 12-membered ring

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