Crystal structure of 1,5-cyclooctadiene-bis[(4-methyl-pyridin-2-yl)-trimethylsilanyl-amido]ruthenium(II), $Ru(C_8H_{12})(C_9H_{15}N_2Si)_2$

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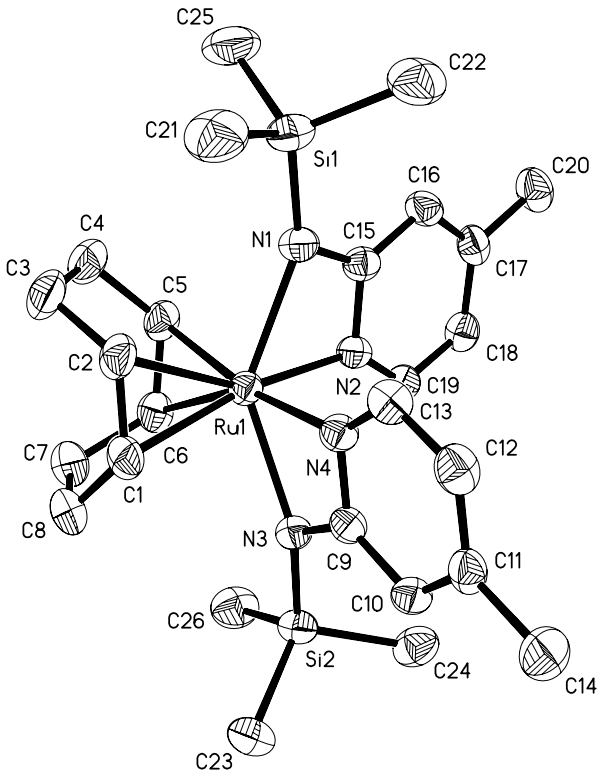

Abstract

$C_{26}H_{42}N_4RuSi_2$, monoclinic, *C*12/*c*1 (no. 15), a = 16.399(2) Å, b = 16.138(2) Å, c = 21.349(2) Å, β = 96.735(7)°, V = 5611.0 Å^3, Z = 8, $R_{gt}(F)$ = 0.034, $wR_{ref}(F^2)$ = 0.097, T = 193 K.

Source of material

To a stirred solution of 0.4 g (2.2 mmol) of lithiated (4-methyl-pyridin-2-yl)-trimethylsilanyl-amine in 8 mL diethyl ether a suspension of 0.308 g (1.1 mmol) [(cod)$RuCl_2$] (cod = 1,5-cyclooctadiene) in 10 mL thf was added at –30 °C and the solution was stirred for six hours at this temperature. The reaction mixture was then stored at –25 °C over night. The solvent was removed in vacuum and the residue was extracted with hexane and filtered. The yellow-brown filtrate was stored at –25 °C to afford yellow crystals suitable for X-ray crystal structure analysis (yield 0.38 g, 61 %).

Discussion

The development of amido metal chemistry of the early transition metals increased dramatically in the last decades. In comparison late transition amido metal chemistry is much less developed [1]. The amido-metal bond in the complexes can be stabilized by additional N-donor functions, for instance by a pyridine moiety [2]. Recently, we have reported examples of amido complexes of late transition metals (nickel and palladium) stabilized by deprotonated 2-aminopyridines [3,4]. The first aminopyridinato complex of ruthenium, a dinuclear complex, was reported by Cotton et al. [5].

The title compound, a mononuclear ruthenium complex, consists of two aminopyridinato ligands and one cod ligand. The bulky trimethylsilyl substituents at the amido N atoms force the aminopyridinato ligands into a *transoid* arrangement. An unusual binding situation is notified by the small chelating angles of 63.00° (∠N1–Ru1–N2) and of 63.10° (∠N3–Ru1–N4). Both pyridine rings are in plane with the ruthenium atom and the appropriate amido N atoms. The deviations from the planes are 0.0047 Å and 0.0141 Å. The angle between these planes is 87.3°. For each of the two aminopyridinato ligands, the Ru—$N_{pyridine}$ distance (d(Ru1—N2) = 2.071 Å, d(Ru1—N4) = 2.082 Å) is significantly shorter than the corresponding Ru—N_{amido} distance (d(Ru1—N1) = 2.179 Å, d(Ru1—N3) = 2.173 Å).

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.05 × 0.05 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	6.65 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{max}$:	52.44°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	37728, 5568
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4356
$N(param)_{refined}$:	298
Programs:	SIR97 [6], SHELXL-97 [7]

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

Atom	Site	x	y	z	U_{iso}
H(1)	8*f*	0.7663	0.1751	0.6370	0.043
H(2)	8*f*	0.8212	0.1423	0.7372	0.044
H(3A)	8*f*	0.7839	0.0208	0.7870	0.056
H(3B)	8*f*	0.7135	0.0024	0.7304	0.056
H(4A)	8*f*	0.6321	0.0345	0.8025	0.054
H(4B)	8*f*	0.7003	0.0899	0.8424	0.054
H(5)	8*f*	0.6279	0.2016	0.8017	0.041
H(6)	8*f*	0.5680	0.2161	0.6995	0.041
H(7A)	8*f*	0.5383	0.0957	0.6376	0.056
H(7B)	8*f*	0.5930	0.0408	0.6889	0.056
H(8A)	8*f*	0.6736	0.0395	0.6121	0.056
H(8B)	8*f*	0.6443	0.1291	0.5866	0.056
H(10)	8*f*	0.7566	0.4005	0.5566	0.038
H(12)	8*f*	0.9700	0.3969	0.6731	0.045
H(13)	8*f*	0.9090	0.3109	0.7412	0.039
H(14A)	8*f*	0.9641	0.4760	0.5789	0.072
H(14B)	8*f*	0.8760	0.5165	0.5582	0.072
H(14C)	8*f*	0.9051	0.4377	0.5211	0.072

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(16)	8*f*	0.7412	0.3688	0.9137	0.039
H(18)	8*f*	0.5613	0.4912	0.8042	0.040
H(19)	8*f*	0.5764	0.3983	0.7230	0.036
H(20A)	8*f*	0.5991	0.5382	0.9086	0.065
H(20B)	8*f*	0.6940	0.5254	0.9341	0.065
H(20C)	8*f*	0.6260	0.4653	0.9575	0.065
H(21A)	8*f*	0.8913	0.1079	0.8259	0.092
H(21B)	8*f*	0.9606	0.1405	0.8792	0.092
H(21C)	8*f*	0.9453	0.1872	0.8128	0.092
H(22A)	8*f*	0.8612	0.3562	0.9293	0.084
H(22B)	8*f*	0.9260	0.3471	0.8792	0.084
H(22C)	8*f*	0.9414	0.3003	0.9456	0.084

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(23A)	8*f*	0.6763	0.3111	0.4878	0.068
H(23B)	8*f*	0.5803	0.3061	0.4637	0.068
H(23C)	8*f*	0.6271	0.2266	0.4950	0.068
H(24A)	8*f*	0.6347	0.4644	0.5682	0.065
H(24B)	8*f*	0.5672	0.4541	0.6159	0.065
H(24C)	8*f*	0.5403	0.4515	0.5415	0.065
H(25A)	8*f*	0.7592	0.2045	0.9599	0.094
H(25B)	8*f*	0.8445	0.1563	0.9755	0.094
H(25C)	8*f*	0.7734	0.1181	0.9261	0.094
H(26A)	8*f*	0.4852	0.2911	0.6305	0.073
H(26B)	8*f*	0.5034	0.2140	0.5871	0.073
H(26C)	8*f*	0.4566	0.2937	0.5562	0.073

Table 3. 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}
Ru(1)	8*f*	0.70948(2)	0.23202(2)	0.72276(1)	0.0269(1)	0.0252(1)	0.0246(1)	0.0011(1)	0.00509(9)	0.0006(1)
Si(2)	8*f*	0.59940(6)	0.32128(6)	0.57563(4)	0.0321(5)	0.0371(5)	0.0265(4)	0.0026(4)	0.0016(4)	–0.0002(4)
Si(1)	8*f*	0.84209(6)	0.22589(7)	0.87669(5)	0.0376(5)	0.0461(6)	0.0330(5)	0.0034(4)	–0.0022(4)	0.0083(4)
N(1)	8*f*	0.7654(2)	0.2557(2)	0.8188(1)	0.033(1)	0.034(1)	0.026(1)	0.004(1)	0.002(1)	0.001(1)
N(3)	8*f*	0.6778(2)	0.2950(2)	0.6333(1)	0.031(1)	0.029(1)	0.024(1)	–0.002(1)	0.004(1)	0.001(1)
N(4)	8*f*	0.8007(2)	0.3039(2)	0.6896(1)	0.030(1)	0.029(1)	0.027(1)	0.001(1)	0.005(1)	–0.001(1)
C(1)	8*f*	0.7391(2)	0.1342(2)	0.6589(2)	0.049(2)	0.028(2)	0.034(2)	0.006(1)	0.014(2)	–0.007(1)
C(2)	8*f*	0.7735(2)	0.1133(2)	0.7195(2)	0.045(2)	0.025(2)	0.040(2)	0.009(1)	0.009(2)	0.000(1)
C(3)	8*f*	0.7385(3)	0.0462(2)	0.7589(2)	0.061(2)	0.027(2)	0.053(2)	0.008(2)	0.011(2)	0.009(2)
C(4)	8*f*	0.6734(3)	0.0785(2)	0.7993(2)	0.058(2)	0.032(2)	0.045(2)	–0.005(2)	0.011(2)	0.010(2)
N(2)	8*f*	0.6661(2)	0.3330(2)	0.7687(1)	0.029(1)	0.029(1)	0.024(1)	0.000(1)	0.002(1)	0.000(1)
C(5)	8*f*	0.6299(2)	0.1559(2)	0.7739(2)	0.037(2)	0.030(2)	0.037(2)	–0.009(1)	0.012(1)	0.004(1)
C(6)	8*f*	0.5928(2)	0.1647(2)	0.7125(2)	0.032(2)	0.033(2)	0.039(2)	–0.010(1)	0.006(1)	0.001(1)
C(7)	8*f*	0.5909(3)	0.0939(2)	0.6656(2)	0.055(2)	0.036(2)	0.047(2)	–0.013(2)	–0.001(2)	–0.005(2)
C(8)	8*f*	0.6616(3)	0.0966(2)	0.6252(2)	0.065(3)	0.032(2)	0.042(2)	–0.001(2)	0.005(2)	–0.011(2)
C(9)	8*f*	0.7536(2)	0.3289(2)	0.6362(1)	0.030(2)	0.028(2)	0.025(2)	0.002(1)	0.006(1)	–0.003(1)
C(10)	8*f*	0.7885(2)	0.3822(2)	0.5941(2)	0.038(2)	0.032(2)	0.025(2)	0.000(1)	0.007(1)	–0.001(1)
C(11)	8*f*	0.8693(2)	0.4076(2)	0.6075(2)	0.039(2)	0.029(2)	0.033(2)	–0.005(1)	0.014(1)	–0.006(1)
C(12)	8*f*	0.9145(2)	0.3802(2)	0.6630(2)	0.031(2)	0.040(2)	0.042(2)	–0.007(1)	0.008(1)	–0.005(2)
C(13)	8*f*	0.8782(2)	0.3289(2)	0.7031(2)	0.026(2)	0.037(2)	0.035(2)	0.002(1)	0.004(1)	–0.002(1)
C(14)	8*f*	0.9069(3)	0.4645(3)	0.5625(2)	0.052(2)	0.049(2)	0.047(2)	–0.014(2)	0.017(2)	0.001(2)
C(15)	8*f*	0.7160(2)	0.3218(2)	0.8236(2)	0.029(2)	0.033(2)	0.026(2)	–0.001(1)	0.006(1)	0.002(1)
C(16)	8*f*	0.7078(2)	0.3759(2)	0.8747(2)	0.037(2)	0.037(2)	0.024(2)	–0.006(1)	0.007(1)	–0.002(1)
C(17)	8*f*	0.6508(2)	0.4391(2)	0.8674(2)	0.037(2)	0.031(2)	0.034(2)	–0.008(1)	0.013(1)	–0.006(1)
C(18)	8*f*	0.6010(2)	0.4481(2)	0.8100(2)	0.033(2)	0.029(2)	0.038(2)	0.003(1)	0.011(1)	–0.001(1)
C(19)	8*f*	0.6105(2)	0.3931(2)	0.7620(2)	0.030(2)	0.029(2)	0.031(2)	0.001(1)	0.007(1)	0.000(1)
C(20)	8*f*	0.6417(2)	0.4971(2)	0.9216(2)	0.051(2)	0.040(2)	0.041(2)	–0.007(2)	0.017(2)	–0.011(2)
C(21)	8*f*	0.9189(3)	0.1572(3)	0.8449(2)	0.049(2)	0.069(3)	0.062(3)	0.020(2)	–0.008(2)	0.005(2)
C(22)	8*f*	0.8995(3)	0.3184(3)	0.9119(2)	0.042(2)	0.070(3)	0.053(3)	–0.003(2)	–0.009(2)	0.004(2)
C(23)	8*f*	0.6237(3)	0.2872(3)	0.4961(2)	0.054(2)	0.051(2)	0.030(2)	–0.001(2)	0.001(2)	–0.005(2)
C(24)	8*f*	0.5835(2)	0.4366(2)	0.5753(2)	0.047(2)	0.044(2)	0.041(2)	0.011(2)	0.010(2)	0.007(2)
C(25)	8*f*	0.7997(3)	0.1695(3)	0.9424(2)	0.076(3)	0.072(3)	0.037(2)	–0.010(2)	–0.006(2)	0.019(2)
C(26)	8*f*	0.4993(2)	0.2745(3)	0.5889(2)	0.039(2)	0.059(3)	0.046(2)	–0.002(2)	–0.001(2)	0.006(2)

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