Crystal structure of tetrakis (μ -acetato)-bis[5-nitro-2-(chromone-2-carboxyl-amino)-1,3-thiazole]dirhodium(II) dichloroform solvate, $\text{Rh}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_{13}\text{H}_7\text{N}_3\text{O}_5\text{S})_2 \cdot 2\text{CHCl}_3$

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Source of material: 2/1 amounts of reactant ligand and $\text{Rh}_2(\text{acetate})_4$ were used and dissolved in methanol at 60°C. The formed precipitate was filtered, washed with methanol solution and then dried with diethyl ether and air. The complex was recrystallized in $\text{CHCl}_3/\text{CCl}_4$ mixture.

$\text{C}_{36}\text{H}_{28}\text{Cl}_6\text{N}_6\text{O}_{18}\text{Rh}_2\text{S}_2$, monoclinic, $C12/c1$ (No. 15), $a = 27.885(3) \text{ \AA}$, $b = 11.075(3) \text{ \AA}$, $c = 19.496(4) \text{ \AA}$, $\beta = 125.60(4)^\circ$, $V = 4895.6 \text{ \AA}^3$, $Z = 4$, $\rho_m = 1.795 \text{ g cm}^{-3}$, $R(F) = 0.044$, $R_w(F^2) = 0.157$.

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

Crystal:	red parallelepiped, size 0.215 x 0.30 x 0.15 mm
Wavelength:	Cu K_α radiation (1.54183 Å)
μ :	99.50 cm ⁻¹
Diffractometer:	Philips PW1100
Scan mode:	ω
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	126°
$N(hkl)_{\text{unique}}$:	3806
Criterion for F_o :	$F_o > 4 \sigma(F_o)$
$N(\text{param})_{\text{refined}}$:	317
Programs:	SHELXS-86, SHELXL-93

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(5)	8f	0.2080(2)	-0.0565(4)	0.3423(3)	0.100(7)
H(9)	8f	0.3681(2)	0.1817(4)	0.4701(2)	0.100(7)
H(18)	8f	0.5298(3)	0.1715(5)	0.4946(4)	0.100(7)
H(19)	8f	0.4154(3)	0.4785(6)	0.5822(4)	0.100(7)
H(20)	8f	0.4742(4)	0.6378(6)	0.6684(5)	0.100(7)
H(21)	8f	0.5681(4)	0.6616(7)	0.7046(5)	0.100(7)
H(22)	8f	0.6074(3)	0.5251(7)	0.6631(4)	0.100(7)
H(32A)	8f	0.0551(3)	0.2998(7)	0.3011(4)	0.100(7)
H(32B)	8f	0.0622(3)	0.1668(7)	0.2807(4)	0.100(7)
H(32C)	8f	0.0764(3)	0.2752(7)	0.2436(4)	0.100(7)
H(52A)	8f	0.2947(3)	0.5031(5)	0.3640(4)	0.100(7)
H(52B)	8f	0.3131(3)	0.5834(5)	0.4420(4)	0.100(7)
H(52C)	8f	0.2470(3)	0.5790(5)	0.3634(4)	0.100(7)
H	8f	0.3473(4)	0.0343(7)	0.0792(5)	0.100(7)

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Rh	8f	0.26226(1)	0.18021(3)	0.46576(2)	0.0229(3)	0.0229(3)	0.0220(3)	-0.0025(1)	0.0129(2)	-0.0062(1)
N(1)	8f	0.2828(2)	0.0446(4)	0.3997(2)	0.035(2)	0.028(2)	0.033(2)	0.001(2)	0.020(2)	-0.001(2)
C(2)	8f	0.3294(2)	0.0427(4)	0.3992(3)	0.032(3)	0.025(2)	0.022(2)	0.006(2)	0.015(2)	0.000(2)
S(3)	8f	0.33153(6)	-0.0711(1)	0.34135(8)	0.0494(8)	0.0339(7)	0.0350(7)	0.0036(5)	0.0273(6)	-0.0074(5)
C(4)	8f	0.2637(2)	-0.1131(4)	0.3150(3)	0.053(3)	0.025(3)	0.030(2)	-0.007(2)	0.022(2)	-0.004(2)
C(5)	8f	0.2440(2)	-0.0446(4)	0.3504(3)	0.041(3)	0.029(2)	0.030(2)	-0.005(2)	0.019(2)	-0.006(2)
N(6)	8f	0.2340(3)	-0.2181(4)	0.2616(3)	0.078(4)	0.029(2)	0.033(2)	-0.010(3)	0.031(3)	-0.011(2)
O(7)	8f	0.1822(3)	-0.2341(5)	0.2350(3)	0.077(4)	0.066(3)	0.079(3)	-0.036(3)	0.047(3)	-0.035(3)
O(8)	8f	0.2623(2)	-0.2816(4)	0.2473(3)	0.102(4)	0.040(2)	0.051(3)	-0.004(3)	0.044(3)	-0.021(2)
N(9)	8f	0.3725(2)	0.1271(4)	0.4429(2)	0.035(2)	0.031(2)	0.034(2)	-0.004(2)	0.021(2)	-0.008(2)
C(10)	8f	0.4224(2)	0.1291(5)	0.4455(3)	0.041(3)	0.039(3)	0.033(3)	0.006(2)	0.024(2)	0.002(2)
O(11)	8f	0.4325(2)	0.0530(4)	0.4109(3)	0.051(2)	0.056(3)	0.061(3)	0.003(2)	0.037(2)	-0.014(2)
C(12)	8f	0.4654(2)	0.2276(5)	0.4984(3)	0.038(3)	0.037(3)	0.039(3)	0.004(2)	0.025(2)	0.007(2)
O(13)	8f	0.4428(2)	0.3094(3)	0.5242(3)	0.041(2)	0.040(2)	0.050(2)	-0.005(2)	0.027(2)	-0.008(2)
C(14)	8f	0.4786(3)	0.4042(5)	0.5741(3)	0.049(3)	0.033(3)	0.037(3)	0.000(2)	0.021(3)	0.003(2)
C(15)	8f	0.5354(3)	0.4172(5)	0.5950(4)	0.047(4)	0.046(3)	0.039(3)	-0.014(3)	0.014(3)	0.012(3)
C(16)	8f	0.5584(3)	0.3307(6)	0.5649(5)	0.040(4)	0.051(4)	0.062(4)	-0.001(3)	0.027(3)	0.019(3)
O(17)	8f	0.6076(2)	0.3392(6)	0.5803(4)	0.054(3)	0.086(4)	0.121(5)	-0.020(3)	0.052(3)	0.002(3)
C(18)	8f	0.5180(3)	0.2319(5)	0.5149(4)	0.043(3)	0.045(3)	0.061(4)	0.000(3)	0.036(3)	0.006(3)
C(19)	8f	0.4540(3)	0.4869(6)	0.5989(4)	0.071(4)	0.052(4)	0.043(3)	0.005(3)	0.023(3)	-0.003(3)
C(20)	8f	0.4890(4)	0.5824(6)	0.6494(5)	0.107(7)	0.042(4)	0.062(4)	0.001(4)	0.041(5)	-0.007(3)
C(21)	8f	0.5456(4)	0.5960(7)	0.6717(5)	0.098(7)	0.058(5)	0.052(4)	-0.032(4)	0.021(4)	-0.012(4)
C(22)	8f	0.5688(3)	0.5153(7)	0.6463(4)	0.069(5)	0.060(4)	0.055(4)	-0.022(4)	0.025(4)	0.001(3)
O(20)	8f	0.1526(2)	0.3135(3)	0.4291(2)	0.028(2)	0.043(2)	0.030(2)	0.002(1)	0.010(2)	-0.012(1)
O(30)	8f	0.1759(2)	0.1820(3)	0.3650(2)	0.030(2)	0.039(2)	0.026(2)	-0.004(1)	0.011(2)	-0.012(1)
C(31)	8f	0.1402(2)	0.2464(5)	0.3672(3)	0.029(3)	0.040(3)	0.029(3)	-0.003(2)	0.013(2)	-0.007(2)
C(32)	8f	0.0779(3)	0.2471(7)	0.2914(4)	0.033(3)	0.083(5)	0.043(3)	0.004(3)	0.004(3)	-0.023(3)
O(40)	8f	0.2808(2)	0.3254(3)	0.4206(3)	0.052(3)	0.034(2)	0.047(2)	-0.012(2)	0.039(2)	-0.010(2)
O(50)	8f	0.2556(2)	0.4551(3)	0.4828(2)	0.046(2)	0.031(2)	0.038(2)	-0.004(2)	0.029(2)	-0.004(2)
C(51)	8f	0.2719(2)	0.4301(5)	0.4357(3)	0.027(3)	0.034(3)	0.031(2)	-0.011(2)	0.013(2)	-0.004(2)
C(52)	8f	0.2826(3)	0.5329(5)	0.3980(4)	0.074(4)	0.038(3)	0.055(4)	-0.009(3)	0.047(3)	0.003(3)
C	8f	0.3747(4)	0.0983(7)	0.1158(5)	0.101(6)	0.065(5)	0.055(4)	-0.043(4)	0.036(4)	-0.011(4)
Cl(1)	8f	0.3883(1)	0.0804(2)	0.2140(1)	0.116(2)	0.093(2)	0.072(1)	-0.002(1)	0.062(1)	-0.010(1)
Cl(2)	8f	0.4374(1)	0.0800(3)	0.1207(2)	0.136(2)	0.123(2)	0.120(2)	-0.056(2)	0.095(2)	-0.029(2)
Cl(3)	8f	0.3400(2)	0.2343(3)	0.0716(2)	0.140(3)	0.104(2)	0.150(3)	-0.013(2)	0.036(2)	0.059(2)

References

- Viostat, B.; Nguyen-Huy Dung; Robert, F.: Complexation d'un Agent Trichomonacide (Ténitroazole) par l'Acétate de Rhodium: Structure Cristalline du Tétrakis(μ-acétato)-bis[5-nitro-2-(2-thénylamino)-1,3-thiazole]dirhodium(II) Dichlorométhane. *Acta Crystallogr. C* **47** (1991) 2550-2553.
- Taudou, A.; Delmas, F.; Timond-David, P.; Ecalte, R.: Recherche d'activité anti-parasitaire de carboxamido-2 benzopyrones-4. *Eur. J. Med. Chem.* **22** (1987) 583-585.
- Sheldrick, G. M.: Phase annealing in SHELXS-90. Direct methods for larger structures. *Acta Crystallogr. A* **46** (1990) 467-473.
- Sheldrick, G. M.: SHELXL-93. Program for refining crystal structures. University of Göttingen, Germany 1993.