New synthetic routes for the preparation of ruthenium-1,10-phenanthroline complexes. Tests of cytotoxic and antibacterial activity of selected ruthenium complexes.

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Diffraction data for all three compounds were collected on a Nonius Kappa CCD diffractometer with graphite monochromated Mo$K\alpha$ radiation at temperature 150 K. The data were processed using DENZO[1] program. Structures were solved by direct methods using SIR97[2]. Most of the positions of hydrogen atoms were obtained from the difference Fourier maps, the remaining were calculated. We employed full-matrix least-squares refinements on F magnitudes with anisotropic displacement factors for non-hydrogen atoms using Xtal3.6[3]. The exception were C and O atoms of solvate methanol molecules in **3**, which are slightly disordered and are thus refined with isotropic displacement parameter. The position of hydroxyl H atoms of these molecules were not determined. One water molecule (O5W) in **1** is disordered over two positions O5W1 and O5W2. These two maxima were also refined with isotropic displacement parameter and the corresponding H atoms positions were not determined. The parameters of hydrogen atoms were not refined. In the final cycle of the refinement we used 3939, 7201 and 6147 reflections and 217, 397 and 454 parameters for compounds **1**, **2**·4H_2O and **3**·6MeOH, respectively. The final R values were 0.033, 0.030 and 0.068 and R_w values were 0.030, 0.030 and 0.045 for **1**, **2**·4H_2O and **3**·6MeOH respectively. The crystallographic data were deposited in the Cambridge Crystallographic Data Center and were assigned the deposition numbers CCDC 1028368-1028370 for compounds **1-3** respectively.

References

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Table S1: Crystallographic data for compounds **1**, **2**·4H_2O and **3**·6MeOH**.**

	1	**2**·4H_2O	**3**·6MeOH
Empirical formula	$C_{14}H_{14}Cl_3N_2ORuS$	$C_{12}H_{19}Cl_4N_2O_5Ru$	$C_{42}H_{48}Cl_2\ N_6O_6Ru$
M_w	465.76	514.16	904.858
T, K	150(2)	150(2)	150(2)
Crystal system	*triclinic*	*triclinic*	*orthorhombic*
Space group	*P-1*	*P-1*	*Pbca*
a, Å	8.8592(1)	8.1409(2)	18.5607(3)
b, Å	11.7145(2)	9.2715(2)	18.6235(3)
c, Å	16.7731(3)	13.0919(3)	23.7095(5)
α, deg.	92.5915(7)	86.061(1)	90.000
β, deg.	103.1136(7)	77.551(1)	90.000
γ, deg.	93.3115(7)	78.652(1)	90.000
V, Å^3	1689.46(5)	945.70(4)	8195.5(3)
Z	4	2	8
D_{calc}, g/cm^3	1.831	1.806	1.466
μ, mm^{-1}	1.527	1.418	0.568
F(000)	924	514	3672
Crystal size, mm	0.44×0.16×0.14	0.175×0.125×0.10	0.30×0.25×0.20
Color	red	brown	red
Data collected / unique	24614 / 7691	16369 / 4297	30130 / 12959
R_{int}	0.031	0.039	0.10
Restraints / parameters	0 / 397	0 / 217	0 / 454
S	1.218	1.089	1.100
R_1, wR_2 [I>2σ(I)]	0.030 / 0.030	0.033 / 0.030	0.068 / 0.045
R_1, wR_2 (all data)	0.043 / 0.056	0.058 / 0.067	0.197 / 0.085
Largest diff. peak / hole (e Å^{-3})	0.749 / -0.962	0.792 / -0.614	1.857 / -3.293*

*The largest diff. peak and hole in **3** is located in close proximity to the central ruthenium ion

Table S2: Selected geometric parameters (Å, °) in **1**.

Ru1a—Cl1a	2.3528(14)	Ru1b—N2b	2.093(5)
Ru1a—Cl2a	2.3512(13)	Ru1b—Cl3b	2.3350(12)
Ru1a—Cl3a	2.3346(12)	Ru1b—Cl1b	2.3333(13)
Ru1a—S1a	2.2935(12)	Ru1b—Cl2b	2.3461(13)
Ru1a—N1a	2.086(4)	Ru1b—S1b	2.3114(13)
Ru1a—N2a	2.097(5)	Ru1b—N1b	2.102(5)
Cl1a—Ru1a—Cl2a	91.97(5)	Cl1b—Ru1b—N1b	85.79(13)
Cl1a—Ru1a—Cl3a	92.93(5)	Cl1b—Ru1b—N2b	88.21(13)
Cl1a—Ru1a—S1a	86.62(5)	Cl2b—Ru1b—Cl3b	95.18(5)
Cl1a—Ru1a—N1a	174.11(13)	Cl2b—Ru1b—S1b	89.61(5)
Cl1a—Ru1a—N2a	94.74(13)	Cl2b—Ru1b—N1b	86.93(13)
Cl2a—Ru1a—Cl3a	174.42(5)	Cl2b—Ru1b—N2b	89.04(13)
Cl2a—Ru1a—S1a	92.39(4)	Cl3b—Ru1b—S1b	87.22(4)
Cl2a—Ru1a—N1a	86.83(11)	Cl3b—Ru1b—N1b	172.19(13)
Cl2a—Ru1a—N2a	92.18(13)	Cl3b—Ru1b—N2b	93.34(14)
Cl3a—Ru1a—S1a	90.55(4)	S1b—Ru1b—N1b	100.33(13)
Cl3a—Ru1a—N1a	88.03(11)	S1b—Ru1b—N2b	178.58(13)
Cl3a—Ru1a—N2a	84.77(13)	N1b—Ru1b—N2b	79.16(18)
S1a—Ru1a—N1a	99.19(12)	Cl1b—Ru1b—Cl3b	91.85(4)
S1a—Ru1a—N2a	175.18(14)	Cl1b—Ru1b—S1b	93.08(5)
N1a—Ru1a—N2a	79.56(17)	Cl1b—Ru1b—Cl2b	172.59(5)

Table S3: Selected geometric parameters (Å, °) in **2**·4H_2O.

Ru—Cl1	2.3483 (16)	Ru—N2	2.056 (5)
Ru—Cl2	2.3490 (16)	N1—C10	1.377 (7)
Ru—Cl3	2.3890 (16)	N1—C2	1.330 (8)
Ru—Cl4	2.3854 (15)	N2—C9	1.331 (8)
Ru—N1	2.064 (5)	N2—C12	1.365 (8)
Cl1—Ru—Cl2	177.28 (5)	Cl2—Ru—N2	91.46 (14)
Cl1—Ru—Cl3	88.89 (6)	Cl3—Ru—Cl4	89.60 (6)
Cl1—Ru—Cl4	91.17 (5)	Cl3—Ru—N1	95.66 (14)
Cl1—Ru—N1	89.81 (14)	Cl3—Ru—N2	174.78 (15)
Cl1—Ru—N2	87.85 (14)	Cl4—Ru—N1	174.67 (14)
Cl2—Ru—Cl3	91.61 (6)	Cl4—Ru—N2	94.54 (15)
Cl2—Ru—Cl4	91.51 (5)	N1—Ru—N2	80.26 (19)
Cl2—Ru—N1	87.48 (14)		

Table S4: Selected geometric parameters (Å, °) in **3**·6MeOH.

Ru—N1a	2.063(8)	Ru—N10a	2.075(9)
Ru—N1b	2.071(9)	Ru—N10b	2.065(9)
Ru—N1c	2.067(7)	Ru—N10c	2.066(9)
N1a—Ru—N1b	96.6(3)	N1a—Ru—N10c	88.2(3)
N1a—Ru—N1c	93.0(3)	N1b—Ru—N1c	94.9(3)
N1a—Ru—N10a	80.3(4)	N1b—Ru—N10a	90.9(3)
N1a—Ru—N10b	173.9(3)	N1b—Ru—N10b	80.3(3)

Table S5: Hydrogen bond contact distances and angles in compound **2**·4H_2O. O5w1 and O5w2 atoms have 50% occupancy.

Donor (D)	Acceptor (A)	D...A (Å)	D-H...A (°)
O1w	$O3w^{1-x,1-y,2-z}$	2.642(7)	165
O1w	O4w	2.432(7)	174
O1w	$O5w1^{-x,2-y,2-z}$	2.610(10)	174
O1w	$O5w2^{-x,2-y,2-z}$	2.467(16)	154
O2w	$Cl4^{-x,2-y,2-z}$	3.222(5)	163
O2w	$Cl3^{x,1+y,z}$	3.420(4)	162
O3w	$Cl3^{1+x,y,z}$	3.202(5)	164
O3w	Cl2	3.108(5)	166
O4w	O3w	2.737(7)	178
O4w	$O2w^{1-x,2-y,2-z}$	2.725(7)	175
O5w1	O2w	2.791(12)	-
O5w1	Cl1	3.130(15)	-
O5w2	O2w	2.711(17)	-
O5w2	Cl1	3.186(11)	-

Table S6: Minimal inhibitory concentrations (MIC) of the tested compounds against selected bacteria. n.i. = no inhibition.

MIC (ug/ml)						
Microorganism	**1**	**4**	**5**	**P**	**phen**	**Acv**
Klebsiella pneumoniae	800	>1000	>1000	>1000	60	>1000
Staphylococcus aureus	250	>1000	>1000	>1000	20	>1000
Escherichia coli	500	>1000	>1000	>1000	75	>1000
Salmonella typhimurium	1000	>1000	>1000	>1000	50	>1000
Proteus vulgaris	900	>1000	>1000	>1000	35	>1000
Pseudomonas aeruginosa	>1000	>1000	>1000	>1000	>1000	>1000
Micrococcus luteus	>1000	>1000	>1000	>1000	250	>1000
Bacillus subtilis	>1000	>1000	>1000	>1000	250	>1000