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Chlorido(η^6-*p*-cymene)[2-(5-phenyl-4*H*-1,2,4-triazol-3-yl-κN^2)pyridine-κN]ruthenium(II) chloride. Addendum

IUCr Editorial Office

5 Abbey Square, Chester CH1 2HU, England

The paper by Sikalov [*IUCrData* (2018), **3**, x180625] is updated.

As requested by the author's institution, the Taras Shevchenko National University of Kyiv, two additional authors and an acknowledgement are added to the data report of Sikalov (2018). The revised author list is Alexander Sikalov, Dmytro M. Khomenko and Roman O. Doroschuk. The affiliation for the two additional authors is the Department of Chemistry, Taras Shevchenko National University of Kyiv, Volodymyrska Street 64/13, Kyiv 01601, Ukraine. In addition, Dr S. Shova, 'Petra Poni' Institute of Macromolecular Chemistry, Iasi, Romania, is thanked for the data collection and structure solution.

References

Sikalov, A. (2018). *IUCrData*, **3**, x180625.

full crystallographic data

IUCrData (2018). **3**, x181073 [https://doi.org/10.1107/S2414314618010738]

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Chlorido(η^6-*p*-cymene)[2-(5-phenyl-4*H*-1,2,4-triazol-3-yl-κN^2)pyridine-κN]ruthenium(II) chloride

Crystal data

$[RuCl(C_{10}H_{14})(C_{13}H_{10}N_4)]Cl$
M_r = 528.44
Triclinic, $P\overline{1}$
Hall symbol: P 1
a = 6.4704 (2) Å
b = 8.7593 (3) Å
c = 10.1975 (4) Å
α = 101.425 (3)°
β = 97.379 (3)°
γ = 95.337 (3)°
V = 557.62 (3) Å^3
Z = 1
$F(000)$ = 267.999
D_x = 1.574 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8029 reflections
θ = 2–32°
μ = 0.96 mm^{-1}
T = 200 K
Prism, clear intense red
0.35 × 0.20 × 0.20 mm

Data collection

Agilent Xcalibur, Eos with CCD area detector diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)
T_{min} = 0.81, T_{max} = 0.83
13071 measured reflections
7295 independent reflections
7220 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.028
θ_{max} = 32.7°, θ_{min} = 2.1°
h = −9→9
k = −13→13
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.062
S = 0.98
7295 reflections
276 parameters
7 restraints
Primary atom site location: other
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.02P)^2 + 0.15P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Absolute structure: Flack (1983), 3505 Friedel-pairs
Absolute structure parameter: −0.03 (2)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ru1	0.86321 (8)	0.38802 (6)	0.54014 (6)	0.0176

Cl2	0.67364 (12)	0.60046 (10)	0.49812 (9)	0.0272
Cl3	0.38532 (13)	0.18193 (12)	−0.10651 (9)	0.0382
N4	0.6317 (4)	0.2491 (3)	0.3951 (2)	0.0217
N5	0.9711 (4)	0.4024 (3)	0.3535 (2)	0.0205
N6	0.4495 (3)	0.1562 (3)	0.3969 (2)	0.0224
N7	0.4792 (3)	0.1708 (3)	0.1855 (2)	0.0220
C8	1.0648 (5)	0.5307 (4)	0.7248 (3)	0.0242
C9	0.6471 (4)	0.2555 (3)	0.2682 (2)	0.0197
C10	0.7853 (5)	0.3174 (4)	0.7245 (3)	0.0269
C11	1.1560 (4)	0.4760 (3)	0.3399 (3)	0.0252
C12	0.3595 (4)	0.1103 (3)	0.2695 (3)	0.0212
C13	0.8319 (4)	0.3402 (3)	0.2389 (3)	0.0217
C14	1.1769 (4)	0.4165 (3)	0.6582 (3)	0.0266
C15	0.8723 (4)	0.3530 (3)	0.1111 (3)	0.0294
C16	0.8650 (4)	0.4755 (3)	0.7562 (3)	0.0269
C17	0.1609 (4)	0.0092 (3)	0.2237 (3)	0.0227
C18	−0.1477 (6)	−0.0882 (5)	0.0591 (4)	0.0390
C19	1.0659 (4)	0.4275 (4)	0.0989 (3)	0.0318
C20	1.2075 (4)	0.4896 (4)	0.2144 (3)	0.0299
C21	0.9010 (4)	0.2016 (3)	0.6564 (3)	0.0276
C22	1.1445 (4)	0.7045 (3)	0.7648 (3)	0.0300
C23	0.0847 (5)	−0.0834 (4)	0.3077 (3)	0.0350
C24	1.0990 (4)	0.2552 (4)	0.6249 (3)	0.0279
C25	−0.2238 (5)	−0.1779 (4)	0.1438 (3)	0.0421
C26	0.0434 (4)	0.0051 (4)	0.0982 (3)	0.0310
C27	1.2355 (5)	0.7703 (4)	0.6552 (3)	0.0395
C28	−0.1071 (5)	−0.1770 (4)	0.2670 (3)	0.0433
C29	0.8085 (6)	0.0338 (4)	0.6203 (4)	0.0445
C30	1.3064 (6)	0.7305 (4)	0.8939 (4)	0.0505
H71	0.451 (3)	0.160 (3)	0.1007 (18)	0.0278 (19)*
H111	1.2519	0.5190	0.4165	0.0292*
H141	1.3038	0.4499	0.6226	0.0345*
H151	0.7718	0.3117	0.0351	0.0363*
H161	0.7756	0.5545	0.7899	0.0341*
H181	−0.2263	−0.0894	−0.0232	0.0473*
H191	1.1020	0.4339	0.0136	0.0391*
H201	1.3382	0.5360	0.2072	0.0359*
H221	1.0243	0.7611	0.7874	0.0381*
H231	0.1642	−0.0822	0.3918	0.0421*
H241	1.1754	0.1828	0.5704	0.0352*
H251	−0.3543	−0.2396	0.1181	0.0508*
H261	0.0930	0.0653	0.0394	0.0384*
H271	1.3590	0.7220	0.6351	0.0610*
H272	1.2764	0.8813	0.6860	0.0609*
H273	1.1347	0.7520	0.5733	0.0611*
H281	−0.1561	−0.2397	0.3228	0.0532*
H291	0.8511	−0.0189	0.5382	0.0682*
H293	0.8554	−0.0149	0.6927	0.0682*

H292	0.6578	0.0253	0.6089	0.0680*
H301	1.2398	0.7033	0.9668	0.0790*
H303	1.4155	0.6662	0.8771	0.0789*
H302	1.3657	0.8396	0.9189	0.0788*
H101	0.6409	0.2853	0.7369	0.0348*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.01437 (7)	0.02394 (8)	0.01423 (7)	0.00019 (5)	0.00201 (5)	0.00478 (6)
Cl2	0.0199 (3)	0.0299 (3)	0.0327 (4)	0.0052 (3)	0.0026 (3)	0.0090 (3)
Cl3	0.0336 (4)	0.0593 (5)	0.0200 (3)	−0.0087 (3)	0.0005 (3)	0.0134 (3)
N4	0.0213 (11)	0.0260 (11)	0.0172 (12)	−0.0043 (9)	0.0043 (9)	0.0054 (9)
N5	0.0156 (10)	0.0278 (12)	0.0182 (10)	0.0016 (9)	0.0031 (8)	0.0055 (9)
N6	0.0204 (10)	0.0244 (10)	0.0204 (10)	−0.0055 (8)	0.0013 (8)	0.0052 (8)
N7	0.0222 (10)	0.0261 (11)	0.0165 (10)	−0.0041 (8)	0.0020 (8)	0.0052 (8)
C8	0.0198 (13)	0.0308 (15)	0.0185 (15)	−0.0021 (11)	−0.0049 (10)	0.0041 (12)
C9	0.0194 (11)	0.0237 (12)	0.0156 (11)	−0.0009 (9)	0.0022 (8)	0.0053 (9)
C10	0.0234 (14)	0.0414 (18)	0.0177 (12)	−0.0001 (12)	0.0040 (10)	0.0121 (13)
C11	0.0186 (11)	0.0336 (14)	0.0226 (12)	−0.0014 (10)	0.0041 (9)	0.0058 (11)
C12	0.0193 (11)	0.0235 (12)	0.0211 (12)	0.0004 (9)	0.0043 (9)	0.0056 (10)
C13	0.0185 (11)	0.0261 (12)	0.0198 (12)	−0.0011 (9)	0.0022 (9)	0.0055 (10)
C14	0.0184 (11)	0.0393 (15)	0.0224 (12)	0.0025 (10)	−0.0030 (9)	0.0119 (11)
C15	0.0288 (14)	0.0392 (15)	0.0193 (12)	−0.0043 (12)	0.0043 (10)	0.0073 (11)
C16	0.0259 (13)	0.0371 (15)	0.0159 (11)	0.0025 (11)	0.0019 (9)	0.0030 (11)
C17	0.0212 (11)	0.0231 (12)	0.0224 (12)	−0.0026 (9)	0.0029 (9)	0.0038 (10)
C18	0.0271 (14)	0.052 (3)	0.032 (2)	−0.0112 (16)	−0.0040 (14)	0.0070 (18)
C19	0.0312 (14)	0.0440 (17)	0.0217 (13)	−0.0024 (12)	0.0088 (11)	0.0106 (12)
C20	0.0210 (12)	0.0405 (16)	0.0293 (14)	−0.0038 (11)	0.0085 (10)	0.0103 (12)
C21	0.0326 (14)	0.0282 (13)	0.0220 (12)	0.0020 (11)	−0.0011 (10)	0.0094 (10)
C22	0.0247 (13)	0.0341 (15)	0.0262 (13)	0.0011 (11)	−0.0008 (10)	−0.0013 (11)
C23	0.0360 (15)	0.0386 (16)	0.0280 (14)	−0.0129 (13)	−0.0004 (12)	0.0127 (12)
C24	0.0260 (14)	0.0356 (16)	0.0238 (14)	0.0114 (12)	0.0006 (11)	0.0088 (12)
C25	0.0306 (15)	0.0471 (19)	0.0424 (18)	−0.0161 (13)	0.0014 (13)	0.0071 (15)
C26	0.0255 (13)	0.0375 (16)	0.0279 (14)	−0.0049 (11)	0.0017 (11)	0.0073 (12)
C27	0.0418 (17)	0.0321 (15)	0.0435 (18)	−0.0019 (13)	0.0135 (14)	0.0037 (14)
C28	0.0392 (17)	0.0482 (19)	0.0393 (18)	−0.0180 (15)	0.0049 (14)	0.0131 (15)
C29	0.051 (2)	0.0342 (16)	0.050 (2)	0.0026 (15)	0.0041 (16)	0.0180 (15)
C30	0.051 (2)	0.048 (2)	0.0396 (19)	−0.0053 (17)	−0.0179 (16)	−0.0004 (16)

Geometric parameters (Å, º)

Ru1—Cl2	2.399 (3)	C15—H151	0.933
Ru1—N4	2.072 (3)	C16—H161	0.981
Ru1—N5	2.132 (2)	C17—C23	1.394 (4)
Ru1—C8	2.223 (3)	C17—C26	1.394 (4)
Ru1—C10	2.195 (3)	C18—C25	1.382 (5)
Ru1—C14	2.189 (3)	C18—C26	1.383 (5)

Ru1—C16	2.183 (3)	C18—H181	0.922
Ru1—C21	2.213 (3)	C19—C20	1.378 (4)
Ru1—C24	2.195 (3)	C19—H191	0.940
N4—N6	1.373 (3)	C20—H201	0.922
N4—C9	1.322 (3)	C21—C24	1.421 (4)
N5—C11	1.342 (3)	C21—C29	1.490 (4)
N5—C13	1.363 (4)	C22—C27	1.513 (4)
N6—C12	1.320 (3)	C22—C30	1.538 (4)
N7—C9	1.345 (3)	C22—H221	0.988
N7—C12	1.374 (3)	C23—C28	1.388 (4)
N7—H71	0.846 (17)	C23—H231	0.939
C8—C14	1.407 (4)	C24—H241	0.972
C8—C16	1.435 (4)	C25—C28	1.379 (5)
C8—C22	1.518 (4)	C25—H251	0.936
C9—C13	1.444 (3)	C26—H261	0.940
C10—C16	1.391 (4)	C27—H271	0.964
C10—C21	1.438 (4)	C27—H272	0.961
C10—H101	0.982	C27—H273	0.967
C11—C20	1.388 (4)	C28—H281	0.930
C11—H111	0.924	C29—H291	0.959
C12—C17	1.458 (3)	C29—H293	0.955
C13—C15	1.386 (3)	C29—H292	0.961
C14—C24	1.414 (4)	C30—H301	0.963
C14—H141	0.983	C30—H303	0.955
C15—C19	1.390 (4)	C30—H302	0.967

Cl2—Ru1—N4	84.77 (8)	Ru1—C14—H141	122.1
Cl2—Ru1—N5	83.97 (7)	C8—C14—H141	119.4
N4—Ru1—N5	76.14 (9)	C24—C14—H141	118.0
Cl2—Ru1—C8	94.13 (9)	C13—C15—C19	118.5 (3)
N4—Ru1—C8	168.20 (9)	C13—C15—H151	120.5
N5—Ru1—C8	115.48 (10)	C19—C15—H151	121.0
Cl2—Ru1—C10	109.97 (9)	Ru1—C16—C8	72.50 (16)
N4—Ru1—C10	101.13 (11)	Ru1—C16—C10	71.95 (16)
N5—Ru1—C10	165.66 (9)	C8—C16—C10	122.3 (3)
C8—Ru1—C10	68.16 (12)	Ru1—C16—H161	121.8
Cl2—Ru1—C14	124.65 (8)	C8—C16—H161	117.5
N4—Ru1—C14	148.83 (11)	C10—C16—H161	119.7
N5—Ru1—C14	95.31 (9)	C12—C17—C23	119.5 (2)
C8—Ru1—C14	37.19 (11)	C12—C17—C26	121.3 (2)
C10—Ru1—C14	79.71 (11)	C23—C17—C26	119.2 (2)
Cl2—Ru1—C16	88.32 (8)	C25—C18—C26	120.5 (4)
N4—Ru1—C16	130.20 (10)	C25—C18—H181	119.6
N5—Ru1—C16	151.76 (10)	C26—C18—H181	119.9
C8—Ru1—C16	38.00 (11)	C15—C19—C20	118.8 (2)
C10—Ru1—C16	37.05 (11)	C15—C19—H191	121.1
Cl2—Ru1—C21	146.94 (8)	C20—C19—H191	120.1
N4—Ru1—C21	93.12 (10)	C11—C20—C19	119.9 (2)

N5—Ru1—C21	127.60 (10)	C11—C20—H201	120.5
C8—Ru1—C21	81.38 (11)	C19—C20—H201	119.6
C10—Ru1—C21	38.07 (11)	Ru1—C21—C10	70.29 (16)
Cl2—Ru1—C24	161.72 (8)	Ru1—C21—C24	70.50 (15)
N4—Ru1—C24	113.51 (12)	C10—C21—C24	117.4 (3)
N5—Ru1—C24	100.26 (10)	Ru1—C21—C29	129.3 (2)
C8—Ru1—C24	67.92 (12)	C10—C21—C29	119.4 (3)
C10—Ru1—C24	67.60 (11)	C24—C21—C29	123.3 (3)
C14—Ru1—C16	67.15 (10)	C8—C22—C27	114.4 (2)
C14—Ru1—C21	68.15 (10)	C8—C22—C30	107.7 (3)
C16—Ru1—C21	68.00 (11)	C27—C22—C30	111.2 (3)
C14—Ru1—C24	37.62 (11)	C8—C22—H221	107.5
C16—Ru1—C24	79.57 (11)	C27—C22—H221	108.0
C21—Ru1—C24	37.60 (11)	C30—C22—H221	107.9
Ru1—N4—N6	135.48 (17)	C17—C23—C28	120.1 (3)
Ru1—N4—C9	115.74 (18)	C17—C23—H231	119.5
N6—N4—C9	108.6 (2)	C28—C23—H231	120.3
Ru1—N5—C11	125.85 (19)	Ru1—C24—C21	71.89 (15)
Ru1—N5—C13	116.14 (17)	Ru1—C24—C14	70.97 (15)
C11—N5—C13	117.8 (2)	C21—C24—C14	121.0 (3)
N4—N6—C12	106.0 (2)	Ru1—C24—H241	123.9
C9—N7—C12	105.0 (2)	C21—C24—H241	120.0
C9—N7—H71	127.5 (12)	C14—C24—H241	118.6
C12—N7—H71	127.5 (12)	C18—C25—C28	120.0 (3)
Ru1—C8—C14	70.11 (16)	C18—C25—H251	120.5
Ru1—C8—C16	69.50 (16)	C28—C25—H251	119.5
C14—C8—C16	116.6 (3)	C17—C26—C18	120.0 (3)
Ru1—C8—C22	131.5 (2)	C17—C26—H261	120.6
C14—C8—C22	123.8 (3)	C18—C26—H261	119.4
C16—C8—C22	119.6 (3)	C22—C27—H271	109.8
N7—C9—N4	109.8 (2)	C22—C27—H272	109.7
N7—C9—C13	130.9 (2)	H271—C27—H272	108.0
N4—C9—C13	119.2 (2)	C22—C27—H273	111.2
Ru1—C10—C16	71.00 (16)	H271—C27—H273	109.1
Ru1—C10—C21	71.64 (15)	H272—C27—H273	109.0
C16—C10—C21	120.7 (3)	C23—C28—C25	120.2 (3)
Ru1—C10—H101	122.9	C23—C28—H281	119.8
C16—C10—H101	120.0	C25—C28—H281	120.0
C21—C10—H101	118.8	C21—C29—H291	110.7
N5—C11—C20	122.1 (3)	C21—C29—H293	108.3
N5—C11—H111	118.9	H291—C29—H293	109.8
C20—C11—H111	119.0	C21—C29—H292	110.6
N7—C12—N6	110.7 (2)	H291—C29—H292	109.1
N7—C12—C17	124.4 (2)	H293—C29—H292	108.3
N6—C12—C17	124.9 (2)	C22—C30—H301	110.0
C9—C13—N5	111.6 (2)	C22—C30—H303	109.2
C9—C13—C15	125.5 (2)	H301—C30—H303	109.5
N5—C13—C15	122.8 (2)	C22—C30—H302	109.4

Ru1—C14—C8	72.71 (16)	H301—C30—H302	109.3
Ru1—C14—C24	71.41 (15)	H303—C30—H302	109.5
C8—C14—C24	122.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H71···Cl3	0.85	2.15	2.984	169

IUCrData

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Chlorido(η^6-*p*-cymene)[2-(5-phenyl-4*H*-1,2,4-triazol-3-yl-κN^2)pyridine-κN]ruthenium(II) chloride

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Keywords: crystal structure; ruthenium; *p*-cymene; coordination chemistry; hydrogen bonding; pyridyltriazoles.

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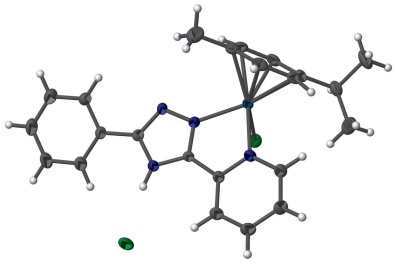
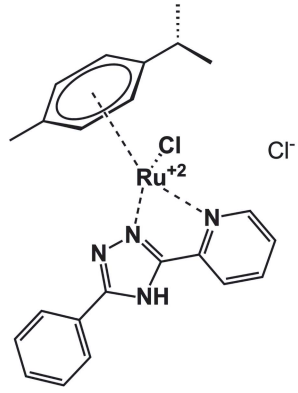
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $[RuCl(C_{10}H_{14})(C_{13}H_{10}N_4)]Cl$, the pyridyltriazole fragment of the bidentate ligand is essentially planar [dihedral angle = 0.8 (1)°], while the phenyl substituent is inclined at 19.4 (1)° to the 1,2,4-triazole ring. In the crystal, the complex cations are packed in sheets with no particularly strong interactions between them. The Cl^- anions are bound to the cations by unusually strong N—H···Cl hydrogen bonds.

3D view



Chemical scheme



Structure description

Recently, ruthenium(II) π-arene complexes have been of increasing interest – mostly due to their anticancer properties (Süss-Fink, 2010) and their catalytic activity in transfer hydrogenation reactions (Hohloch *et al.*, 2013). Therefore, it is of great importance to know the structural details of these compounds. We have synthesized an example of a ruthenium(II) *p*-cymene complex with a pyridyl-1,2,4-triazole ligand in the 4*H*-tautomeric form. The molecular structure of the title compound is shown in Fig. 1. As is traditional for this kind of compounds, the cation exhibits a distorted octahedral piano-stool geometry with *p*-cymene as a π-bound hexahapto ligand, a chloride anion as a monodentate ligand and the 3-(2-pyridyl)-5-phenyl-1,2,4-triazole as a bidentate chelating *N*,*N*-donor ligand. It is interesting that while the pyridyltriazole moiety is virtually planar with a dihedral angle of a mere 0.8 (1)° between the pyridine and triazole rings, the phenyl ring does not lie in this plane and is inclined at 19.4 (1)° to the triazole ring plane, even though there is no apparent steric hindrance to cause this. We surmise that the fact that the 2-(5-phenyl-4*H*-1,2,4-triazol-3-yl)pyridine ligand is coordinated *via* N4 arises from unfavourable steric interactions (between the phenyl group and the *p*-cymene ligand), which would take place in a hypothetical analogue of the title compound where the ligand is coordinated *via* N7.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N7—H71···Cl3	0.85	2.15	2.984	169

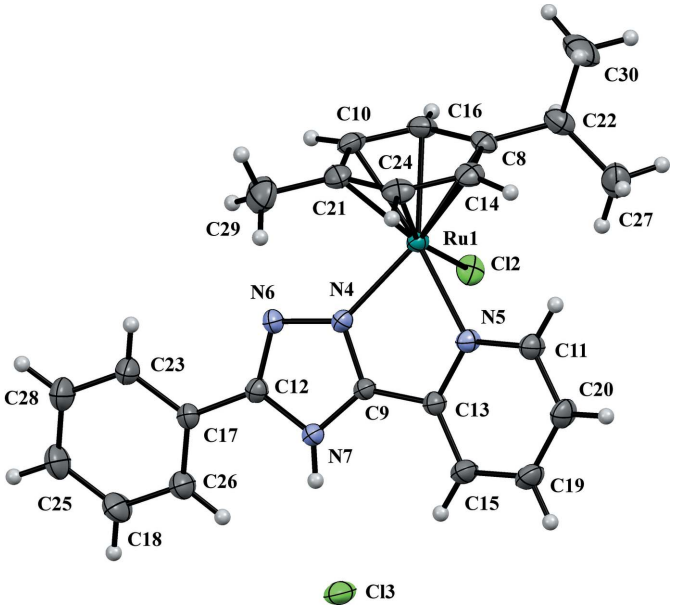


Figure 1
The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

As shown in Fig. 2, the title compound features an intriguing hydrogen bond formed between the N—H group of the 1,2,4-triazole ring and the chloride counter-ion (Table 1). The relatively short H···Cl distance and an NH···Cl angle close to 180° suggest an atypical strength of the bond as compared to other N—H···Cl hydrogen bonds, excluding charge-assisted

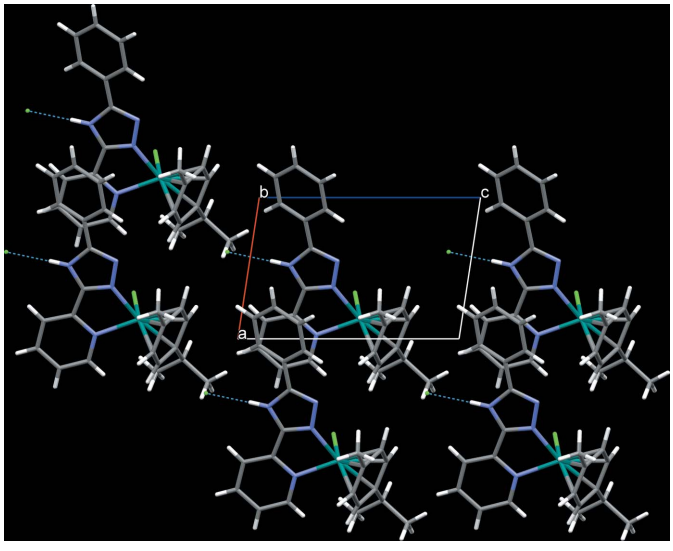


Figure 2
A crystal packing diagram (view along the *b* axis) of the title compound. N—H···Cl hydrogen bonds are depicted as blue dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[RuCl(C_{10}H_{14})(C_{13}H_{10}N_4)]Cl$
M_r	528.44
Crystal system, space group	Triclinic, $P1$
Temperature (K)	200
a, b, c (Å)	6.4704 (2), 8.7593 (3), 10.1975 (4)
α, β, γ (°)	101.425 (3), 97.379 (3), 95.337 (3)
V (Å^3)	557.62 (3)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.96
Crystal size (mm)	0.35 × 0.20 × 0.20
Data collection	
Diffractometer	Agilent Xcalibur, Eos with CCD area detector
Absorption correction	Multi-scan (*DENZO*/*SCALE-PACK*; Otwinowski & Minor, 1997)
T_{min}, T_{max}	0.81, 0.83
No. of measured, independent and observed [$I > 2.0\sigma(I)$] reflections	13071, 7295, 7220
R_{int}	0.028
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.760
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.062, 0.98
No. of reflections	7295
No. of parameters	276
No. of restraints	7
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.56, −0.40
Absolute structure	Flack (1983), 3505 Friedel-pairs
Absolute structure parameter	−0.03 (2)

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* (Sheldrick, 2008), *CRYSTALS* (Betteridge *et al.*, 2003), *Mercury* (Macrae *et al.*, 2006), *publCIF* (Westrip, 2010).

hydrogen bonds (Steed & Atwood, 2009). Unexpectedly, the title compound does not exhibit any significant π-stacking interactions [the shortest centroid–centroid distance is 4.122 Å (offset) and the shortest C—H···centroid distance is 3.453 Å].

It appears that crystal structures of ruthenium(II) *p*-cymene complexes bearing 3-(2-pyridyl)-1,2,4-triazoles have not been published until now. The two most closely related crystal structures are those of η^5-cyclopentadienyl analogues, which also differ from the title compound by variations in the 3-(2-pyridyl)-1,2,4-triazole ligands and by replacement of the

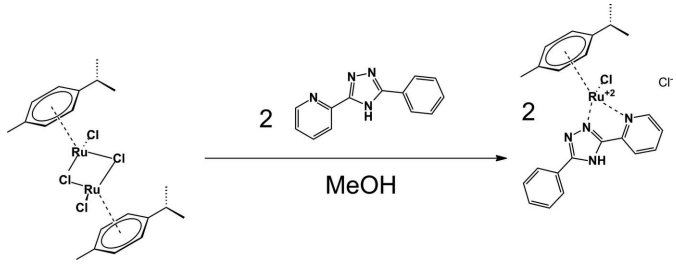


Figure 3
Synthesis of the title compound.

chlorido ligand by PPh_3 in the inner coordination sphere. The Ru1—N4 and Ru1—N5 bond lengths [2.072 (3) and 2.132 (2) Å, respectively] in the title compound are comparable to those in the related compounds mentioned above [respective bond distances are 2.069 (3) and 2.125 (3) Å (Gupta *et al.*, 2010) and 2.089 (4) and 2.119 (3) Å (Gupta *et al.*, 2012)].

Synthesis and crystallization

The ruthenium(II) *p*-cymene dichloride dimer (244.8 mg, 0.4 mmol) was dissolved in about 3 ml of methanol. After complete dissolution of the starting compound, 2-(5-phenyl-4*H*-1,2,4-triazol-3-yl)pyridine (355.2 mg, 0.8 mmol) was added to the clear red solution. Within 30 minutes, the reaction mixture adopted a reddish–orange hue and red prismatic crystals suitable for single-crystal XRD analysis began to form on the bottom and sides of the reaction vessel. Several crystals were harvested, and then the solvent was distilled off with the use of a rotary evaporator. Yield: 93%. Schematic representation of the synthesis is given in Fig. 3.

^{1}H NMR (400 MHz, DMSO-δ_6): δ(p.p.m.) 9.43 (*d*, *J* = 4.2 Hz, 1H, py$^{\alpha}$H), 8.30–8.08 (*m*, 3H, py$^{\beta}$H, py$^{\beta'}$H and py$^{\gamma}$H), 7.83–7.26 (*m*, 5H, phenyl), 6.14 (*d*, *J* = 5.0 Hz, 1H, *p*-cymene CH), 6.02 (*d*, *J* = 4.9 Hz, 1H, *p*-cymene CH), 5.93 (d, *J* = 4.9 Hz, 1H, *p*-cymene CH), 5.80 (*d*, *J* = 5.0 Hz, 1H, *p*-cymene CH), 2.74–2.63 (*m*, 1H, CH from ^{i}Pr), 2.17 (*s*, 3H, Me from *p*-cymene), 1.06 (*d*, *J* = 6.8 Hz, 3H, Me from ^{i}Pr), 0.98 (*d*, *J* = 6.7 Hz, 3H, Me from ^{i}Pr).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2018). **3**, x180625 [https://doi.org/10.1107/S2414314618006259]

Chlorido(η^6-*p*-cymene)[2-(5-phenyl-4*H*-1,2,4-triazol-3-yl-κN^2)pyridine-κN]ruthenium(II) chloride

Alexander Sikalov

Chlorido(η^6-*p*-cymene)[2-(5-phenyl-4*H*-1,2,4-triazol-3-yl-κN^2)pyridine-κN]ruthenium(II) chloride

Crystal data

$[RuCl(C_{10}H_{14})(C_{13}H_{10}N_4)]Cl$
M_r = 528.44
Triclinic, $P\overline{1}$
Hall symbol: P 1
a = 6.4704 (2) Å
b = 8.7593 (3) Å
c = 10.1975 (4) Å
α = 101.425 (3)°
β = 97.379 (3)°
γ = 95.337 (3)°
V = 557.62 (3) Å^3
Z = 1
$F(000)$ = 267.999
D_x = 1.574 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8029 reflections
θ = 2–32°
μ = 0.96 mm^{-1}
T = 200 K
Prism, clear intense red
0.35 × 0.20 × 0.20 mm

Data collection

Agilent Xcalibur, Eos with CCD area detector diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)
T_{min} = 0.81, T_{max} = 0.83
13071 measured reflections
7295 independent reflections
7220 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.028
θ_{max} = 32.7°, θ_{min} = 2.1°
h = −9→9
k = −13→13
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.062
S = 0.98
7295 reflections
276 parameters
7 restraints
Primary atom site location: other
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.02P)^2 + 0.15P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Absolute structure: Flack (1983), 3505 Friedel-pairs
Absolute structure parameter: −0.03 (2)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ru1	0.86321 (8)	0.38802 (6)	0.54014 (6)	0.0176

Cl2	0.67364 (12)	0.60046 (10)	0.49812 (9)	0.0272
Cl3	0.38532 (13)	0.18193 (12)	−0.10651 (9)	0.0382
N4	0.6317 (4)	0.2491 (3)	0.3951 (2)	0.0217
N5	0.9711 (4)	0.4024 (3)	0.3535 (2)	0.0205
N6	0.4495 (3)	0.1562 (3)	0.3969 (2)	0.0224
N7	0.4792 (3)	0.1708 (3)	0.1855 (2)	0.0220
C8	1.0648 (5)	0.5307 (4)	0.7248 (3)	0.0242
C9	0.6471 (4)	0.2555 (3)	0.2682 (2)	0.0197
C10	0.7853 (5)	0.3174 (4)	0.7245 (3)	0.0269
C11	1.1560 (4)	0.4760 (3)	0.3399 (3)	0.0252
C12	0.3595 (4)	0.1103 (3)	0.2695 (3)	0.0212
C13	0.8319 (4)	0.3402 (3)	0.2389 (3)	0.0217
C14	1.1769 (4)	0.4165 (3)	0.6582 (3)	0.0266
C15	0.8723 (4)	0.3530 (3)	0.1111 (3)	0.0294
C16	0.8650 (4)	0.4755 (3)	0.7562 (3)	0.0269
C17	0.1609 (4)	0.0092 (3)	0.2237 (3)	0.0227
C18	−0.1477 (6)	−0.0882 (5)	0.0591 (4)	0.0390
C19	1.0659 (4)	0.4275 (4)	0.0989 (3)	0.0318
C20	1.2075 (4)	0.4896 (4)	0.2144 (3)	0.0299
C21	0.9010 (4)	0.2016 (3)	0.6564 (3)	0.0276
C22	1.1445 (4)	0.7045 (3)	0.7648 (3)	0.0300
C23	0.0847 (5)	−0.0834 (4)	0.3077 (3)	0.0350
C24	1.0990 (4)	0.2552 (4)	0.6249 (3)	0.0279
C25	−0.2238 (5)	−0.1779 (4)	0.1438 (3)	0.0421
C26	0.0434 (4)	0.0051 (4)	0.0982 (3)	0.0310
C27	1.2355 (5)	0.7703 (4)	0.6552 (3)	0.0395
C28	−0.1071 (5)	−0.1770 (4)	0.2670 (3)	0.0433
C29	0.8085 (6)	0.0338 (4)	0.6203 (4)	0.0445
C30	1.3064 (6)	0.7305 (4)	0.8939 (4)	0.0505
H71	0.451 (3)	0.160 (3)	0.1007 (18)	0.0278 (19)*
H111	1.2519	0.5190	0.4165	0.0292*
H141	1.3038	0.4499	0.6226	0.0345*
H151	0.7718	0.3117	0.0351	0.0363*
H161	0.7756	0.5545	0.7899	0.0341*
H181	−0.2263	−0.0894	−0.0232	0.0473*
H191	1.1020	0.4339	0.0136	0.0391*
H201	1.3382	0.5360	0.2072	0.0359*
H221	1.0243	0.7611	0.7874	0.0381*
H231	0.1642	−0.0822	0.3918	0.0421*
H241	1.1754	0.1828	0.5704	0.0352*
H251	−0.3543	−0.2396	0.1181	0.0508*
H261	0.0930	0.0653	0.0394	0.0384*
H271	1.3590	0.7220	0.6351	0.0610*
H272	1.2764	0.8813	0.6860	0.0609*
H273	1.1347	0.7520	0.5733	0.0611*
H281	−0.1561	−0.2397	0.3228	0.0532*
H291	0.8511	−0.0189	0.5382	0.0682*
H293	0.8554	−0.0149	0.6927	0.0682*

H292	0.6578	0.0253	0.6089	0.0680*
H301	1.2398	0.7033	0.9668	0.0790*
H303	1.4155	0.6662	0.8771	0.0789*
H302	1.3657	0.8396	0.9189	0.0788*
H101	0.6409	0.2853	0.7369	0.0348*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.01437 (7)	0.02394 (8)	0.01423 (7)	0.00019 (5)	0.00201 (5)	0.00478 (6)
Cl2	0.0199 (3)	0.0299 (3)	0.0327 (4)	0.0052 (3)	0.0026 (3)	0.0090 (3)
Cl3	0.0336 (4)	0.0593 (5)	0.0200 (3)	−0.0087 (3)	0.0005 (3)	0.0134 (3)
N4	0.0213 (11)	0.0260 (11)	0.0172 (12)	−0.0043 (9)	0.0043 (9)	0.0054 (9)
N5	0.0156 (10)	0.0278 (12)	0.0182 (10)	0.0016 (9)	0.0031 (8)	0.0055 (9)
N6	0.0204 (10)	0.0244 (10)	0.0204 (10)	−0.0055 (8)	0.0013 (8)	0.0052 (8)
N7	0.0222 (10)	0.0261 (11)	0.0165 (10)	−0.0041 (8)	0.0020 (8)	0.0052 (8)
C8	0.0198 (13)	0.0308 (15)	0.0185 (15)	−0.0021 (11)	−0.0049 (10)	0.0041 (12)
C9	0.0194 (11)	0.0237 (12)	0.0156 (11)	−0.0009 (9)	0.0022 (8)	0.0053 (9)
C10	0.0234 (14)	0.0414 (18)	0.0177 (12)	−0.0001 (12)	0.0040 (10)	0.0121 (13)
C11	0.0186 (11)	0.0336 (14)	0.0226 (12)	−0.0014 (10)	0.0041 (9)	0.0058 (11)
C12	0.0193 (11)	0.0235 (12)	0.0211 (12)	0.0004 (9)	0.0043 (9)	0.0056 (10)
C13	0.0185 (11)	0.0261 (12)	0.0198 (12)	−0.0011 (9)	0.0022 (9)	0.0055 (10)
C14	0.0184 (11)	0.0393 (15)	0.0224 (12)	0.0025 (10)	−0.0030 (9)	0.0119 (11)
C15	0.0288 (14)	0.0392 (15)	0.0193 (12)	−0.0043 (12)	0.0043 (10)	0.0073 (11)
C16	0.0259 (13)	0.0371 (15)	0.0159 (11)	0.0025 (11)	0.0019 (9)	0.0030 (11)
C17	0.0212 (11)	0.0231 (12)	0.0224 (12)	−0.0026 (9)	0.0029 (9)	0.0038 (10)
C18	0.0271 (14)	0.052 (3)	0.032 (2)	−0.0112 (16)	−0.0040 (14)	0.0070 (18)
C19	0.0312 (14)	0.0440 (17)	0.0217 (13)	−0.0024 (12)	0.0088 (11)	0.0106 (12)
C20	0.0210 (12)	0.0405 (16)	0.0293 (14)	−0.0038 (11)	0.0085 (10)	0.0103 (12)
C21	0.0326 (14)	0.0282 (13)	0.0220 (12)	0.0020 (11)	−0.0011 (10)	0.0094 (10)
C22	0.0247 (13)	0.0341 (15)	0.0262 (13)	0.0011 (11)	−0.0008 (10)	−0.0013 (11)
C23	0.0360 (15)	0.0386 (16)	0.0280 (14)	−0.0129 (13)	−0.0004 (12)	0.0127 (12)
C24	0.0260 (14)	0.0356 (16)	0.0238 (14)	0.0114 (12)	0.0006 (11)	0.0088 (12)
C25	0.0306 (15)	0.0471 (19)	0.0424 (18)	−0.0161 (13)	0.0014 (13)	0.0071 (15)
C26	0.0255 (13)	0.0375 (16)	0.0279 (14)	−0.0049 (11)	0.0017 (11)	0.0073 (12)
C27	0.0418 (17)	0.0321 (15)	0.0435 (18)	−0.0019 (13)	0.0135 (14)	0.0037 (14)
C28	0.0392 (17)	0.0482 (19)	0.0393 (18)	−0.0180 (15)	0.0049 (14)	0.0131 (15)
C29	0.051 (2)	0.0342 (16)	0.050 (2)	0.0026 (15)	0.0041 (16)	0.0180 (15)
C30	0.051 (2)	0.048 (2)	0.0396 (19)	−0.0053 (17)	−0.0179 (16)	−0.0004 (16)

Geometric parameters (Å, º)

Ru1—Cl2	2.399 (3)	C15—H151	0.933
Ru1—N4	2.072 (3)	C16—H161	0.981
Ru1—N5	2.132 (2)	C17—C23	1.394 (4)
Ru1—C8	2.223 (3)	C17—C26	1.394 (4)
Ru1—C10	2.195 (3)	C18—C25	1.382 (5)
Ru1—C14	2.189 (3)	C18—C26	1.383 (5)

Ru1—C16	2.183 (3)	C18—H181	0.922
Ru1—C21	2.213 (3)	C19—C20	1.378 (4)
Ru1—C24	2.195 (3)	C19—H191	0.940
N4—N6	1.373 (3)	C20—H201	0.922
N4—C9	1.322 (3)	C21—C24	1.421 (4)
N5—C11	1.342 (3)	C21—C29	1.490 (4)
N5—C13	1.363 (4)	C22—C27	1.513 (4)
N6—C12	1.320 (3)	C22—C30	1.538 (4)
N7—C9	1.345 (3)	C22—H221	0.988
N7—C12	1.374 (3)	C23—C28	1.388 (4)
N7—H71	0.846 (17)	C23—H231	0.939
C8—C14	1.407 (4)	C24—H241	0.972
C8—C16	1.435 (4)	C25—C28	1.379 (5)
C8—C22	1.518 (4)	C25—H251	0.936
C9—C13	1.444 (3)	C26—H261	0.940
C10—C16	1.391 (4)	C27—H271	0.964
C10—C21	1.438 (4)	C27—H272	0.961
C10—H101	0.982	C27—H273	0.967
C11—C20	1.388 (4)	C28—H281	0.930
C11—H111	0.924	C29—H291	0.959
C12—C17	1.458 (3)	C29—H293	0.955
C13—C15	1.386 (3)	C29—H292	0.961
C14—C24	1.414 (4)	C30—H301	0.963
C14—H141	0.983	C30—H303	0.955
C15—C19	1.390 (4)	C30—H302	0.967

Cl2—Ru1—N4	84.77 (8)	Ru1—C14—H141	122.1
Cl2—Ru1—N5	83.97 (7)	C8—C14—H141	119.4
N4—Ru1—N5	76.14 (9)	C24—C14—H141	118.0
Cl2—Ru1—C8	94.13 (9)	C13—C15—C19	118.5 (3)
N4—Ru1—C8	168.20 (9)	C13—C15—H151	120.5
N5—Ru1—C8	115.48 (10)	C19—C15—H151	121.0
Cl2—Ru1—C10	109.97 (9)	Ru1—C16—C8	72.50 (16)
N4—Ru1—C10	101.13 (11)	Ru1—C16—C10	71.95 (16)
N5—Ru1—C10	165.66 (9)	C8—C16—C10	122.3 (3)
C8—Ru1—C10	68.16 (12)	Ru1—C16—H161	121.8
Cl2—Ru1—C14	124.65 (8)	C8—C16—H161	117.5
N4—Ru1—C14	148.83 (11)	C10—C16—H161	119.7
N5—Ru1—C14	95.31 (9)	C12—C17—C23	119.5 (2)
C8—Ru1—C14	37.19 (11)	C12—C17—C26	121.3 (2)
C10—Ru1—C14	79.71 (11)	C23—C17—C26	119.2 (2)
Cl2—Ru1—C16	88.32 (8)	C25—C18—C26	120.5 (4)
N4—Ru1—C16	130.20 (10)	C25—C18—H181	119.6
N5—Ru1—C16	151.76 (10)	C26—C18—H181	119.9
C8—Ru1—C16	38.00 (11)	C15—C19—C20	118.8 (2)
C10—Ru1—C16	37.05 (11)	C15—C19—H191	121.1
Cl2—Ru1—C21	146.94 (8)	C20—C19—H191	120.1
N4—Ru1—C21	93.12 (10)	C11—C20—C19	119.9 (2)

N5—Ru1—C21	127.60 (10)	C11—C20—H201	120.5
C8—Ru1—C21	81.38 (11)	C19—C20—H201	119.6
C10—Ru1—C21	38.07 (11)	Ru1—C21—C10	70.29 (16)
Cl2—Ru1—C24	161.72 (8)	Ru1—C21—C24	70.50 (15)
N4—Ru1—C24	113.51 (12)	C10—C21—C24	117.4 (3)
N5—Ru1—C24	100.26 (10)	Ru1—C21—C29	129.3 (2)
C8—Ru1—C24	67.92 (12)	C10—C21—C29	119.4 (3)
C10—Ru1—C24	67.60 (11)	C24—C21—C29	123.3 (3)
C14—Ru1—C16	67.15 (10)	C8—C22—C27	114.4 (2)
C14—Ru1—C21	68.15 (10)	C8—C22—C30	107.7 (3)
C16—Ru1—C21	68.00 (11)	C27—C22—C30	111.2 (3)
C14—Ru1—C24	37.62 (11)	C8—C22—H221	107.5
C16—Ru1—C24	79.57 (11)	C27—C22—H221	108.0
C21—Ru1—C24	37.60 (11)	C30—C22—H221	107.9
Ru1—N4—N6	135.48 (17)	C17—C23—C28	120.1 (3)
Ru1—N4—C9	115.74 (18)	C17—C23—H231	119.5
N6—N4—C9	108.6 (2)	C28—C23—H231	120.3
Ru1—N5—C11	125.85 (19)	Ru1—C24—C21	71.89 (15)
Ru1—N5—C13	116.14 (17)	Ru1—C24—C14	70.97 (15)
C11—N5—C13	117.8 (2)	C21—C24—C14	121.0 (3)
N4—N6—C12	106.0 (2)	Ru1—C24—H241	123.9
C9—N7—C12	105.0 (2)	C21—C24—H241	120.0
C9—N7—H71	127.5 (12)	C14—C24—H241	118.6
C12—N7—H71	127.5 (12)	C18—C25—C28	120.0 (3)
Ru1—C8—C14	70.11 (16)	C18—C25—H251	120.5
Ru1—C8—C16	69.50 (16)	C28—C25—H251	119.5
C14—C8—C16	116.6 (3)	C17—C26—C18	120.0 (3)
Ru1—C8—C22	131.5 (2)	C17—C26—H261	120.6
C14—C8—C22	123.8 (3)	C18—C26—H261	119.4
C16—C8—C22	119.6 (3)	C22—C27—H271	109.8
N7—C9—N4	109.8 (2)	C22—C27—H272	109.7
N7—C9—C13	130.9 (2)	H271—C27—H272	108.0
N4—C9—C13	119.2 (2)	C22—C27—H273	111.2
Ru1—C10—C16	71.00 (16)	H271—C27—H273	109.1
Ru1—C10—C21	71.64 (15)	H272—C27—H273	109.0
C16—C10—C21	120.7 (3)	C23—C28—C25	120.2 (3)
Ru1—C10—H101	122.9	C23—C28—H281	119.8
C16—C10—H101	120.0	C25—C28—H281	120.0
C21—C10—H101	118.8	C21—C29—H291	110.7
N5—C11—C20	122.1 (3)	C21—C29—H293	108.3
N5—C11—H111	118.9	H291—C29—H293	109.8
C20—C11—H111	119.0	C21—C29—H292	110.6
N7—C12—N6	110.7 (2)	H291—C29—H292	109.1
N7—C12—C17	124.4 (2)	H293—C29—H292	108.3
N6—C12—C17	124.9 (2)	C22—C30—H301	110.0
C9—C13—N5	111.6 (2)	C22—C30—H303	109.2
C9—C13—C15	125.5 (2)	H301—C30—H303	109.5
N5—C13—C15	122.8 (2)	C22—C30—H302	109.4

Ru1—C14—C8	72.71 (16)	H301—C30—H302	109.3
Ru1—C14—C24	71.41 (15)	H303—C30—H302	109.5
C8—C14—C24	122.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H71···Cl3	0.85	2.15	2.984	169