Electronic Supplementary Information (ESI)

TiO_2 Modified with A Ru(II)–N′NN′ 8-Hydroxyquinolyl Complex for Efficient Gaseous Photoreduction of CO_2

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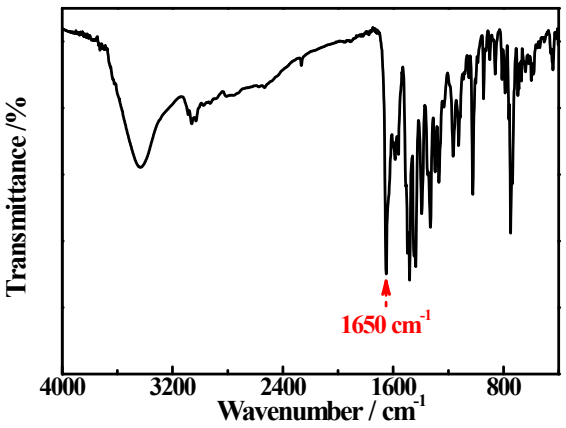

Fig. S1 FTIR spectra of the Ru(II) pincer complex [Ru(N′NN′)(ONO)].

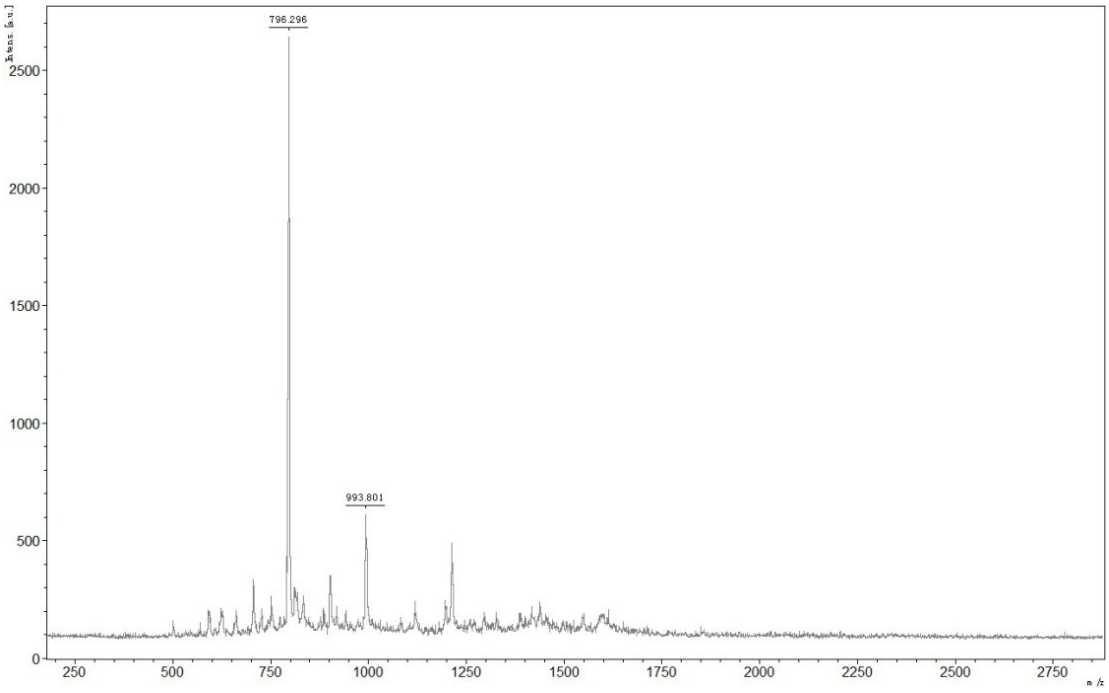

Fig. S2 MALDI-TOF mass spectrum of the Ru(II) pincer complex [Ru(N′NN′)(ONO)].

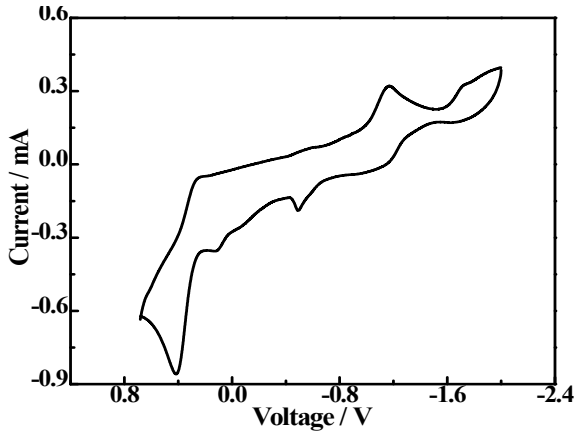

Fig. S3 Cyclic voltammogram of the Ru(II) pincer complex [Ru(N′NN′)(ONO)].

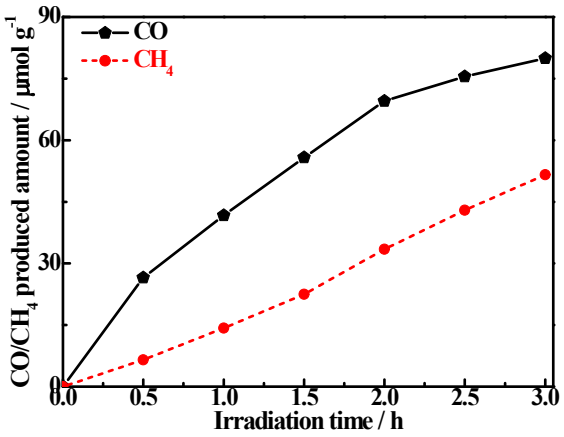

Fig. S4 Time courses of CO/CH_4 production over 0.25wt% Ru(N′NN′)(ONO)-TiO_2 during 3 h full spectrum irradiation of Xe-lamp.

Table S1 Effect of Ru(N′NN′)(ONO)-loading amount on the CO/CH_4 production activity over various photocatalysts

Catalyst	CO produced rate / μmol g^{-1} h^{-1}	CH_4 produced rate / μmol g^{-1} h^{-1}	TCEN / μmol g^{-1} h^{-1}
TiO_2	1.82	0.82	10.20
0.05wt% Ru(N′NN′)(ONO)-TiO_2	16.25	8.17	97.86
0.13wt% Ru(N′NN′)(ONO)-TiO_2	18.37	12.52	136.90
0.25wt% Ru(N′NN′)(ONO)-TiO_2	26.6	17.2	190.80
0.50wt% Ru(N′NN′)(ONO)-TiO_2	18.82	12.14	134.76
1.00wt% Ru(N′NN′)(ONO)-TiO_2	10.14	7.65	81.48
2.00wt% Ru(N′NN′)(ONO)-TiO_2	9.79	5.89	66.70

CIF file of [Ru(N′NN′)(ONO)] CCDC 1536357

data_1

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_chemical_name_systematic; ? ; _chemical_name_common ? _chemical_melting_point ?

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_chemical_formula_weight 794.79

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loop_ _symmetry_equiv_pos_as_xyz

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Refinement of F^2^ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2^, conventional R-factors R are based on F, with F set to zero for negative F^2^. The threshold expression of F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2^ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger. ;

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H9 H 0.1408 1.1808 0.2056 0.073 Uiso 1 1 calc R A . C10 C 0.2737(9) 1.2385(7) 0.2524(4) 0.065(3) Uani 1 1 d . A . H10 H 0.2442 1.2950 0.2468 0.078 Uiso 1 1 calc R . . C11 C 0.3762(9) 1.2286(6) 0.2837(4) 0.057(2) Uani 1 1 d . . . H11 H 0.4194 1.2763 0.2991 0.068 Uiso 1 1 calc R A . C12 C 0.4133(7) 1.1436(7) 0.2915(3) 0.050(2) Uani 1 1 d . A . C13 C 0.5952(7) 1.1599(6) 0.3590(3) 0.050(2) Uani 1 1 d . A . H13A H 0.5861 1.2216 0.3491 0.060 Uiso 1 1 calc R . . H13B H 0.6714 1.1421 0.3540 0.060 Uiso 1 1 calc R . . C14 C 0.5794(5) 1.1468(5) 0.41940(19) 0.059(3) Uani 1 1 d G . . C15 C 0.4785(4) 1.1207(6) 0.4343(2) 0.097(4) Uani 1 1 d G . . H15 H 0.4163 1.1100 0.4063 0.116 Uiso 1 1 calc R . . C16 C 0.4684(5) 1.1103(6) 0.4901(3) 0.141(7) Uani 1 1 d G . . H16 H 0.3994 1.0925 0.5003 0.169 Uiso 1 1 calc R . . C17 C 0.5594(7) 1.1259(6) 0.53106(19) 0.117(5) Uani 1 1 d G . . H17 H 0.5525 1.1188 0.5692 0.140 Uiso 1 1 calc R . . C18 C 0.6603(6) 1.1520(6) 0.5162(2) 0.125(6) Uani 1 1 d G . . H18 H 0.7225 1.1626 0.5441 0.150 Uiso 1 1 calc R . . C19 C 0.6704(5) 1.1624(6) 0.4603(3) 0.116(5) Uani 1 1 d G . . H19 H 0.7394 1.1802 0.4502 0.140 Uiso 1 1 calc R . . C20 C 0.5162(7) 0.7273(6) 0.3333(3) 0.045(2) Uani 1 1 d . A . C21 C 0.3699(7) 0.6616(7) 0.2907(3) 0.052(3) Uani 1 1 d . . . C22 C 0.2675(7) 0.6376(7) 0.2595(4) 0.056(2) Uani 1 1 d . A . H22 H 0.2193 0.6786 0.2392 0.067 Uiso 1 1 calc R . . C23 C 0.2407(9) 0.5526(7) 0.2598(5) 0.070(3) Uani 1 1 d . . . H23 H 0.1712 0.5344 0.2398 0.084 Uiso 1 1 calc R A . C24 C 0.3118(9) 0.4906(7) 0.2885(4) 0.071(3) Uani 1 1 d . A . H24 H 0.2892 0.4320 0.2867 0.085 Uiso 1 1 calc R . . C25 C 0.4140(9) 0.5119(7) 0.3195(4) 0.063(3) Uani 1 1 d . . . H25 H 0.4618 0.4699 0.3389 0.075 Uiso 1 1 calc R A . C26 C 0.4416(8) 0.5983(7) 0.3202(4) 0.052(2) Uani 1 1 d . A . C27 C 0.6241(8) 0.6028(7) 0.3855(4) 0.064(3) Uani 1 1 d . A . H27A H 0.6967 0.6254 0.3782 0.077 Uiso 1 1 calc R . . H27B H 0.6232 0.5400 0.3783 0.077 Uiso 1 1 calc R . . C28 C 0.6150(8) 0.6174(6) 0.4457(4) 0.058(3) Uani 1 1 d . . .

C29 C 0.5164(11) 0.6200(10) 0.4652(5) 0.119(5) Uani 1 1 d . . .

H29 H 0.4508 0.6168 0.4385 0.143 Uiso 1 1 calc R . .

C30 C 0.5027(13) 0.6267(11) 0.5187(5) 0.130(6) Uani 1 1 d . . .

H30 H 0.4309 0.6237 0.5295 0.156 Uiso 1 1 calc R . .

C31 C 0.6014(15) 0.6385(8) 0.5589(5) 0.101(5) Uani 1 1 d . . .

H31 H 0.5977 0.6490 0.5971 0.121 Uiso 1 1 calc R . .

C32 C 0.7014(14) 0.6336(10) 0.5389(5) 0.117(5) Uani 1 1 d . . .

H32 H 0.7685 0.6358 0.5647 0.141 Uiso 1 1 calc R . .
C33 C 0.7081(9) 0.6260(8) 0.4850(4) 0.096(4) Uani 1 1 d . . .
H33 H 0.7794 0.6265 0.4735 0.115 Uiso 1 1 calc R . .
C34 C 0.1987(8) 0.8593(15) 0.1899(4) 0.066(3) Uani 0.645(9) 1 d PDU A 1
C35 C 0.0830(8) 0.8626(14) 0.1689(5) 0.064(5) Uani 0.645(9) 1 d PDU A 1
H35 H 0.0594 0.8591 0.1299 0.077 Uiso 0.645(9) 1 calc PR A 1
C36 C 0.0015(7) 0.8711(14) 0.2051(4) 0.062(4) Uani 0.645(9) 1 d PDU A 1
C37 C 0.0363(6) 0.8760(8) 0.2637(3) 0.056(4) Uani 0.645(9) 1 d PGDU A 1
C38 C -0.0364(5) 0.8860(8) 0.3022(4) 0.066(5) Uani 0.645(9) 1 d PGDU A 1
H38 H -0.1146 0.8851 0.2902 0.079 Uiso 0.645(9) 1 calc PR A 1
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C40 C 0.1198(8) 0.8986(10) 0.3758(3) 0.103(5) Uani 0.645(9) 1 d PGDU A 1
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C42 C 0.1508(6) 0.8773(8) 0.2812(3) 0.045(3) Uani 0.645(9) 1 d PGDU A 1
C43 C 0.3025(8) 0.8577(15) 0.1668(4) 0.060(4) Uani 0.645(9) 1 d PDU A 1
N6 N 0.2305(7) 0.8730(10) 0.2463(4) 0.037(3) Uani 0.645(9) 1 d PDU A 1
O1 O 0.3956(8) 0.857(2) 0.2002(5) 0.052(3) Uani 0.645(9) 1 d PDU A 1
O2 O 0.2965(10) 0.8603(10) 0.1141(4) 0.081(3) Uani 0.645(9) 1 d PDU A 1
O3 O 0.3003(8) 0.8864(8) 0.3537(4) 0.050(4) Uani 0.645(9) 1 d PDU A 1
O4 O -0.1022(8) 0.8722(10) 0.1844(4) 0.102(4) Uani 0.645(9) 1 d PDU A 1
C34' C 0.1312(12) 0.856(2) 0.2703(5) 0.045(3) Uani 0.355(9) 1 d PDU A 2
C35' C 0.0253(13) 0.866(3) 0.2373(6) 0.081(11) Uani 0.355(9) 1 d PDU A 2
H35' H -0.0387 0.8707 0.2552 0.097 Uiso 0.355(9) 1 calc PR A 2
C36' C 0.0115(12) 0.870(3) 0.1784(6) 0.073(10) Uani 0.355(9) 1 d PDU A 2
C37' C 0.1099(12) 0.870(2) 0.1537(7) 0.079(11) Uani 0.355(9) 1 d PGDU A 2
C38' C 0.1049(15) 0.8729(17) 0.0962(6) 0.119(14) Uani 0.355(9) 1 d PGDU A 2
H38' H 0.0347 0.8719 0.0728 0.143 Uiso 0.355(9) 1 calc PR A 2
C39' C 0.2025(18) 0.8771(19) 0.0730(6) 0.133(7) Uani 0.355(9) 1 d PGDU A 2
H39' H 0.1991 0.8790 0.0337 0.160 Uiso 0.355(9) 1 calc PR A 2
C40' C 0.3052(15) 0.879(2) 0.1072(7) 0.081(3) Uani 0.355(9) 1 d PGDU A 2
H40' H 0.3720 0.8814 0.0914 0.097 Uiso 0.355(9) 1 calc PR A 2
C41' C 0.3103(12) 0.876(3) 0.1647(7) 0.060(4) Uani 0.355(9) 1 d PGDU A 2
C42' C 0.2126(12) 0.872(2) 0.1880(6) 0.066(3) Uani 0.355(9) 1 d PGDU A 2
C43' C 0.1757(12) 0.855(2) 0.3292(6) 0.055(9) Uani 0.355(9) 1 d PDU A 2
N6' N 0.2244(13) 0.8475(19) 0.2437(7) 0.037(3) Uani 0.355(9) 1 d PDU A 2
O1' O 0.2815(12) 0.8572(14) 0.3435(7) 0.034(6) Uani 0.355(9) 1 d PDU A 2
O2' O 0.1080(15) 0.8636(19) 0.3642(7) 0.103(5) Uani 0.355(9) 1 d PDU A 2
O3' O 0.4073(13) 0.869(4) 0.1968(9) 0.052(3) Uani 0.355(9) 1 d PDU A 2

O4' O -0.0850(12) 0.8692(18) 0.1491(7) 0.102(4) Uani 0.355(9) 1 d PDU A 2

N1 N 0.5312(5) 0.8753(5) 0.3297(3) 0.0440(17) Uani 1 1 d . A .

N2 N 0.4065(5) 0.9976(5) 0.2853(3) 0.0444(18) Uani 1 1 d . A .

N3 N 0.5112(5) 1.1076(5) 0.3218(3) 0.0457(19) Uani 1 1 d . . .

N4 N 0.4186(5) 0.7407(5) 0.3010(3) 0.0469(19) Uani 1 1 d . A .

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C1 0.025(5) 0.078(7) 0.037(5) -0.001(5) 0.012(4) 0.006(5)

C2 0.026(5) 0.098(8) 0.053(6) 0.001(5) 0.011(5) 0.004(5)

C3 0.036(6) 0.123(11) 0.050(6) -0.004(6) -0.022(5) -0.001(7)

C4 0.034(6) 0.102(10) 0.060(6) 0.002(6) -0.004(5) 0.001(6)

C5 0.034(5) 0.068(7) 0.053(5) 0.003(5) 0.017(5) 0.003(5)

C6 0.033(5) 0.064(7) 0.035(5) -0.011(4) 0.016(4) -0.005(5)

C7 0.039(6) 0.055(7) 0.057(6) -0.001(5) 0.026(5) 0.006(5)

C8 0.049(6) 0.061(7) 0.063(6) 0.001(5) 0.014(5) 0.003(5)

C9 0.040(6) 0.072(7) 0.072(7) 0.008(6) 0.015(5) 0.005(6)

C10 0.057(7) 0.064(8) 0.075(7) 0.010(6) 0.008(6) 0.019(6)

C11 0.069(7) 0.039(6) 0.066(6) -0.002(5) 0.019(5) -0.013(6)

C12 0.041(6) 0.067(7) 0.048(5) -0.005(5) 0.021(4) -0.009(5)

C13 0.037(5) 0.070(7) 0.043(5) -0.013(4) 0.000(4) -0.014(4)

C14 0.044(6) 0.094(8) 0.042(5) -0.003(5) 0.017(4) 0.004(5)

C15 0.050(7) 0.187(14) 0.055(6) 0.016(8) 0.013(5) 0.000(8)

C16 0.052(7) 0.33(2) 0.050(7) 0.025(10) 0.022(6) 0.030(11)

C17 0.135(13) 0.164(14) 0.057(7) 0.008(8) 0.036(9) 0.009(11)

C18 0.108(11) 0.233(18) 0.034(6) 0.003(8) 0.016(7) -0.064(11)

C19 0.052(7) 0.220(16) 0.077(8) -0.034(9) 0.013(6) -0.053(9)

C20 0.035(5) 0.063(7) 0.040(5) -0.003(5) 0.014(4) 0.007(5)

C21 0.031(5) 0.084(8) 0.044(5) -0.005(5) 0.017(4) -0.007(5)

C22 0.038(5) 0.063(7) 0.069(6) -0.007(5) 0.019(5) -0.006(5)

C23 0.057(7) 0.060(8) 0.099(8) -0.012(6) 0.031(6) -0.003(6)

C24 0.060(7) 0.060(7) 0.095(8) -0.009(6) 0.021(6) 0.000(6)

C25 0.070(8) 0.060(8) 0.064(6) -0.012(5) 0.027(6) 0.005(6)

C26 0.047(6) 0.061(7) 0.055(5) 0.004(5) 0.029(5) 0.016(5)

C27 0.040(6) 0.081(8) 0.071(6) 0.004(5) 0.011(5) 0.016(5)

C28 0.043(6) 0.077(8) 0.051(5) 0.012(5) 0.000(5) 0.012(5)

C29 0.081(9) 0.220(17) 0.057(7) -0.001(9) 0.010(7) 0.017(10)

C30 0.099(11) 0.24(2) 0.050(7) 0.015(9) 0.015(7) 0.029(12)

C31 0.174(15) 0.083(9) 0.049(7) 0.010(6) 0.027(9) 0.016(10)
C32 0.118(12) 0.184(16) 0.043(7) 0.016(8) -0.011(7) -0.039(12)
C33 0.039(6) 0.180(14) 0.065(7) 0.020(8) -0.003(5) 0.022(8)
C34 0.054(5) 0.062(6) 0.081(6) -0.004(5) 0.003(5) 0.001(5)
C35 0.042(7) 0.078(9) 0.078(8) -0.012(7) 0.024(7) 0.000(7)
C36 0.040(7) 0.072(8) 0.072(9) 0.002(9) 0.002(7) -0.005(7)
C37 0.047(7) 0.058(8) 0.062(8) -0.005(7) 0.007(6) 0.014(7)
C38 0.032(6) 0.079(9) 0.093(8) 0.015(7) 0.030(6) 0.004(6)
C39 0.125(9) 0.143(10) 0.132(9) 0.003(7) 0.018(7) 0.003(7)
C40 0.098(7) 0.112(9) 0.106(7) 0.005(6) 0.033(6) -0.003(6)
C41 0.041(8) 0.049(9) 0.065(8) 0.003(6) 0.032(6) -0.001(6)
C42 0.014(5) 0.042(7) 0.076(6) -0.001(5) 0.001(4) -0.007(5)
C43 0.071(6) 0.055(8) 0.052(5) 0.005(5) 0.000(4) 0.003(5)
N6 0.032(4) 0.034(8) 0.039(4) 0.000(4) -0.016(3) -0.003(4)
O1 0.045(4) 0.054(8) 0.062(4) 0.001(3) 0.025(3) 0.000(4)
O2 0.097(5) 0.088(6) 0.056(4) 0.004(4) 0.004(4) 0.015(5)
O3 0.050(6) 0.056(8) 0.044(6) -0.013(5) 0.011(5) 0.000(5)
O4 0.068(5) 0.113(6) 0.123(7) 0.003(6) 0.006(5) 0.013(5)
C34' 0.014(5) 0.042(7) 0.076(6) -0.001(5) 0.001(4) -0.007(5)
C35' 0.085(14) 0.082(14) 0.068(14) 0.003(10) -0.015(9) 0.006(10)
C36' 0.055(12) 0.081(13) 0.076(13) -0.006(10) -0.012(9) -0.004(10)
C37' 0.083(14) 0.074(14) 0.076(13) -0.001(10) 0.000(10) -0.001(10)
C38' 0.125(16) 0.114(16) 0.114(16) 0.003(10) 0.004(10) 0.007(10)
C39' 0.125(9) 0.143(10) 0.132(9) 0.003(7) 0.018(7) 0.003(7)
C40' 0.097(5) 0.088(6) 0.056(4) 0.004(4) 0.004(4) 0.015(5)
C41' 0.071(6) 0.055(8) 0.052(5) 0.005(5) 0.000(4) 0.003(5)
C42' 0.054(5) 0.062(6) 0.081(6) -0.004(5) 0.003(5) 0.001(5)
C43' 0.046(12) 0.051(13) 0.067(12) -0.008(9) 0.008(9) 0.004(9)
N6' 0.032(4) 0.034(8) 0.039(4) 0.000(4) -0.016(3) -0.003(4)
O1' 0.033(9) 0.036(10) 0.036(8) -0.014(7) 0.017(7) 0.002(7)
O2' 0.098(7) 0.112(9) 0.106(7) 0.005(6) 0.033(6) -0.003(6)
O3' 0.045(4) 0.054(8) 0.062(4) 0.001(3) 0.025(3) 0.000(4)
O4' 0.068(5) 0.113(6) 0.123(7) 0.003(6) 0.006(5) 0.013(5)
N1 0.030(4) 0.062(5) 0.040(4) -0.001(4) 0.005(3) -0.003(4)
N2 0.025(4) 0.063(5) 0.045(4) -0.001(4) 0.002(3) 0.003(4)
N3 0.032(4) 0.068(6) 0.039(4) -0.010(4) 0.013(3) -0.010(4)
N4 0.024(4) 0.076(6) 0.039(4) 0.000(4) 0.001(3) 0.001(4)
N5 0.036(4) 0.068(6) 0.048(4) 0.010(4) 0.007(4) 0.004(4)
_geom_special_details ;

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.
; loop_ _geom_bond_atom_site_label_1 _geom_bond_atom_site_label_2 _geom_bond_distance _geom_bond_site_symmetry_2 _geom_bond_publ_flag
Ru1 N6 1.900(9) . ? Ru1 N1 2.004(6) . ? Ru1 N6' 2.010(18) . ? Ru1 O1' 2.025(17) . ? Ru1 N2 2.037(8) . ? Ru1 N4 2.063(8) . ? Ru1 O1 2.072(13) . ? Ru1 O3 2.099(11) . ? Ru1 O3' 2.19(2) . ? C1 N1 1.371(11) . ? C1 C2 1.383(11) . ? C1 C6 1.450(12) . ? C2 C3 1.370(14) . ? C2 H2 0.9500 . ? C3 C4 1.350(14) . ? C3 H3 0.9500 . ? C4 C5 1.403(12) . ? C4 H4 0.9500 . ? C5 N1 1.351(11) . ? C5 C20 1.482(12) . ? C6 N3 1.349(11) . ? C6 N2 1.364(10) . ? C7 N2 1.383(11) . ? C7 C8 1.395(12) . ? C7 C12 1.409(12) . ? C8 C9 1.362(13) . ? C8 H8 0.9500 . ? C9 C10 1.391(13) . ? C9 H9 0.9500 . ? C10 C11 1.364(12) . ? C10 H10 0.9500 . ? C11 C12 1.398(13) . ? C11 H11 0.9500 . ? C12 N3 1.414(11) . ? C13 N3 1.496(10) . ? C13 C14 1.523(9) . ? C13 H13A 0.9900 . ? C13 H13B 0.9900 . ? C14 C15 1.3900 . ? C14 C19 1.3900 . ? C15 C16 1.3900 . ? C15 H15 0.9500 . ? C16 C17 1.3900 . ? C16 H16 0.9500 . ? C17 C18 1.3900 . ? C17 H17 0.9500 . ? C18 C19 1.3900 . ? C18 H18 0.9500 . ? C19 H19 0.9500 . ? C20 N4 1.334(10) . ? C20 N5 1.363(11) . ? C21 N4 1.369(11) . ? C21 C22 1.405(12) . ? C21 C26 1.431(13) . ? C22 C23 1.359(12) . ? C22 H22 0.9500 . ? C23 C24 1.406(14) . ? C23 H23 0.9500 . ? C24 C25 1.389(13) . ? C24 H24 0.9500 . ? C25 C26 1.382(13) . ? C25 H25 0.9500 . ? C26 N5 1.408(12) . ? C27 N5 1.452(11) . ? C27 C28 1.501(12) . ? C27 H27A 0.9900 . ? C27 H27B 0.9900 . ? C28 C29 1.353(15) . ? C28 C33 1.371(13) . ? C29 C30 1.338(15) . ? C29 H29 0.9500 . ? C30 C31 1.438(19) . ? C30 H30 0.9500 . ? C31 C32 1.377(19) . ? C31 H31 0.9500 . ? C32 C33 1.329(15) . ? C32 H32 0.9500 . ? C33 H33 0.9500 . ? C34 N6 1.383(8) . ? C34 C35 1.420(8) . ? C34 C43 1.454(8) . ? C35 C36 1.426(8) . ? C35 H35 0.9500 . ? C36 O4 1.282(8) . ? C36 C37 1.425(8) . ? C37 C38 1.3900 . ? C37 C42 1.3900 . ? C38 C39 1.3900 . ? C38 H38 0.9500 . ? C39 C40 1.3900 . ? C39 H39 0.9500 . ? C40 C41 1.3900 . ? C40 H40 0.9500 . ? C41 O3 1.308(7) . ? C41 C42 1.3900 . ? C42 N6 1.382(7) . ? C43 O2 1.274(6) . ? C43 O1 1.287(6) . ? C34' N6' 1.394(9) . ? C34' C35' 1.417(8) . ? C34' C43' 1.449(8) . ? C35' C36' 1.415(8) . ? C35' H35' 0.9500 . ? C36' O4' 1.276(8) . ? C36' C37' 1.417(8) . ? C37' C38' 1.3900 . ? C37' C42' 1.3900 . ? C38' C39' 1.3900 . ? C38' H38' 0.9500 . ? C39' C40' 1.3900 . ? C39' H39' 0.9500 . ? C40' C41' 1.3900 . ? C40' H40' 0.9500 . ? C41' O3' 1.313(8) . ? C41' C42' 1.3900 . ? C42' N6' 1.390(9) . ? C43' O2' 1.277(6) . ? C43' O1' 1.279(7) . ?

loop_ _geom_angle_atom_site_label_1 _geom_angle_atom_site_label_2 _geom_angle_atom_site_label_3 _geom_angle _geom_angle_site_symmetry_1 _geom_angle_site_symmetry_3 _geom_angle_publ_flag
N6 Ru1 N1 172.4(4) . . ? N6 Ru1 N6' 11.5(12) . . ? N1 Ru1 N6' 172.5(7) . . ? N6 Ru1 O1' 73.9(5) . . ? N1 Ru1 O1' 101.2(4) . . ? N6' Ru1 O1' 73.9(6) . . ? N6 Ru1 N2 96.2(5) . . ? N1 Ru1 N2 78.7(3) . . ? N6' Ru1 N2 107.6(8) . . ? O1' Ru1 N2 100.2(6) . . ? N6 Ru1 N4 107.9(5) . . ? N1 Ru1 N4 77.1(3) . . ? N6' Ru1 N4 96.6(9) . . ? O1' Ru1 N4 85.1(6) . . ? N2 Ru1 N4 155.8(3) . . ? N6 Ru1 O1 76.1(4) . . ? N1 Ru1 O1 109.6(3) . . ? N6' Ru1 O1 74.6(5) . . ? O1' Ru1 O1 148.1(5) . . ? N2 Ru1 O1 93.8(9) . . ? N4 Ru1 O1 93.8(9) . . ? N6 Ru1 O3 81.9(4) . . ? N1 Ru1 O3 92.1(3) . . ? N6' Ru1 O3 84.3(5) . . ? O1' Ru1 O3 15.2(6) . . ? N2 Ru1 O3 86.7(4) . . ? N4 Ru1 O3 94.8(4) . . ? O1 Ru1 O3 158.0(4) . . ? N6 Ru1 O3' 79.3(5) . . ? N1 Ru1 O3' 106.0(5) . . ? N6' Ru1 O3' 78.7(7) . . ? O1' Ru1 O3' 152.6(7) . . ? N2 Ru1 O3' 88.1(16) . . ? N4 Ru1 O3' 97.9(16) . . ? O1 Ru1 O3' 6(2) . . ? O3 Ru1 O3' 159.8(7) . . ? N1 C1 C2 121.4(9) . . ? N1 C1 C6 109.4(7) . . ? C2 C1 C6 129.2(10) . . ? C3 C2 C1 117.5(10) . . ? C3 C2 H2 121.3 . . ? C1 C2 H2 121.3 . . ? C4 C3 C2 123.2(9) . . ?

C4 C3 H3 118.4 . . ? C2 C3 H3 118.4 . . ? C3 C4 C5 117.4(10) . . ? C3 C4 H4 121.3 . . ? C5 C4 H4 121.3 . . ? N1 C5 C4 121.5(9) . . ? N1 C5 C20 109.1(8) . . ? C4 C5 C20 129.4(9) . . ? N3 C6 N2 110.5(8) . . ? N3 C6 C1 130.7(8) . . ? N2 C6 C1 118.7(8) . . ? N2 C7 C8 131.7(9) . . ? N2 C7 C12 109.0(8) . . ? C8 C7 C12 119.2(9) . . ? C9 C8 C7 118.3(9) . . ? C9 C8 H8 120.8 . . ? C7 C8 H8 120.8 . . ? C8 C9 C10 121.2(10) . . ? C8 C9 H9 119.4 . . ? C10 C9 H9 119.4 . . ? C11 C10 C9 123.3(10) . . ? C11 C10 H10 118.4 . . ? C9 C10 H10 118.4 . . ? C10 C11 C12 115.4(9) . . ? C10 C11 H11 122.3 . . ? C12 C11 H11 122.3 . . ? C11 C12 C7 122.6(9) . . ? C11 C12 N3 132.2(9) . . ? C7 C12 N3 105.1(8) . . ? N3 C13 C14 109.6(6) . . ? N3 C13 H13A 109.7 . . ? C14 C13 H13A 109.7 . . ? N3 C13 H13B 109.7 . . ? C14 C13 H13B 109.7 . . ? H13A C13 H13B 108.2 . . ? C15 C14 C19 120.0 . . ? C15 C14 C13 122.4(5) . . ? C19 C14 C13 117.6(5) . . ? C16 C15 C14 120.0 . . ? C16 C15 H15 120.0 . . ? C14 C15 H15 120.0 . . ? C15 C16 C17 120.0 . . ? C15 C16 H16 120.0 . . ? C17 C16 H16 120.0 . . ? C18 C17 C16 120.0 . . ? C18 C17 H17 120.0 . . ? C16 C17 H17 120.0 . . ? C17 C18 C19 120.0 . . ? C17 C18 H18 120.0 . . ? C19 C18 H18 120.0 . . ? C18 C19 C14 120.0 . . ? C18 C19 H19 120.0 . . ? C14 C19 H19 120.0 . . ? N4 C20 N5 113.5(8) . . ? N4 C20 C5 117.3(8) . . ? N5 C20 C5 129.2(8) . . ? N4 C21 C22 131.2(9) . . ? N4 C21 C26 108.1(8) . . ? C22 C21 C26 120.6(10) . . ? C23 C22 C21 116.6(10) . . ? C23 C22 H22 121.7 . . ? C21 C22 H22 121.7 . . ? C22 C23 C24 122.5(11) . . ? C22 C23 H23 118.7 . . ? C24 C23 H23 118.7 . . ? C25 C24 C23 122.5(11) . . ? C25 C24 H24 118.8 . . ? C23 C24 H24 118.8 . . ? C26 C25 C24 115.6(10) . . ? C26 C25 H25 122.2 . . ? C24 C25 H25 122.2 . . ? C25 C26 N5 131.4(9) . . ? C25 C26 C21 122.2(10) . . ? N5 C26 C21 106.4(9) . . ? N5 C27 C28 114.1(7) . . ? N5 C27 H27A 108.7 . . ? C28 C27 H27A 108.7 . . ? N5 C27 H27B 108.7 . . ? C28 C27 H27B 108.7 . . ? H27A C27 H27B 107.6 . . ? C29 C28 C33 115.5(9) . . ? C29 C28 C27 123.2(9) . . ? C33 C28 C27 121.3(9) . . ? C30 C29 C28 126.2(13) . . ? C30 C29 H29 116.9 . . ? C28 C29 H29 116.9 . . ?
C29 C30 C31 117.1(13) . . ? C29 C30 H30 121.4 . . ? C31 C30 H30 121.4 . . ? C32 C31 C30 116.2(10) . . ? C32 C31 H31 121.9 . . ? C30 C31 H31 121.9 . . ? C33 C32 C31 122.8(12) . . ? C33 C32 H32 118.6 . . ? C31 C32 H32 118.6 . . ? C32 C33 C28 121.9(11) . . ? C32 C33 H33 119.1 . . ? C28 C33 H33 119.1 . . ? N6 C34 C35 117.5(8) . . ? N6 C34 C43 104.9(7) . . ? C35 C34 C43 136.8(7) . . ? C34 C35 C36 121.6(8) . . ? C34 C35 H35 119.2 . . ? C36 C35 H35 119.2 . . ? O4 C36 C37 121.0(7) . . ? O4 C36 C35 119.4(7) . . ? C37 C36 C35 119.5(7) . . ? C38 C37 C42 120.0 . . ? C38 C37 C36 123.8(6) . . ? C42 C37 C36 116.0(6) . . ? C39 C38 C37 120.0 . . ? C39 C38 H38 120.0 . . ? C37 C38 H38 120.0 . . ? C38 C39 C40 120.0 . . ? C38 C39 H39 120.0 . . ? C40 C39 H39 120.0 . . ? C39 C40 C41 120.0 . . ? C39 C40 H40 120.0 . . ? C41 C40 H40 120.0 . . ? O3 C41 C40 120.4(4) . . ? O3 C41 C42 119.6(4) . . ? C40 C41 C42 120.0 . . ? N6 C42 C41 115.0(5) . . ? N6 C42 C37 124.9(5) . . ? C41 C42 C37 120.0 . . ? O2 C43 O1 123.0(8) . . ? O2 C43 C34 117.9(6) . . ? O1 C43 C34 119.1(6) . . ? C42 N6 C34 120.0(7) . . ? C42 N6 Ru1 114.5(6) . . ? C34 N6 Ru1 124.7(7) . . ? C43 O1 Ru1 114.3(7) . . ? C41 O3 Ru1 108.5(5) . . ? N6' C34' C35' 118.5(9) . . ? N6' C34' C43' 104.5(8) . . ? C35' C34' C43' 136.9(9) . . ? C36' C35' C34' 122.3(10) . . ? C36' C35' H35' 118.8 . . ? C34' C35' H35' 118.8 . . ? O4' C36' C35' 121.4(8) . . ? O4' C36' C37' 121.7(8) . . ? C35' C36' C37' 116.8(9) . . ? C38' C37' C42' 120.0 . . ? C38' C37' C36' 121.1(8) . . ? C42' C37' C36' 118.9(8) . . ? C39' C38' C37' 120.0 . . ? C39' C38' H38' 120.0 . . ? C37' C38' H38' 120.0 . . ? C40' C39' C38' 120.0 . . ? C40' C39' H39' 120.0 . . ? C38' C39' H39' 120.0 . . ? C39' C40' C41' 120.0 . . ? C39' C40' H40' 120.0 . . ? C41' C40' H40' 120.0 . . ? O3' C41' C42' 119.9(5) . . ? O3' C41' C40' 119.8(5) . . ? C42' C41' C40' 120.0 . . ? C41' C42' C37' 120.0 . . ? C41' C42' N6' 116.1(9) . . ? C37' C42' N6' 121.9(10) . . ? O2' C43' O1' 122.5(9) . . ? O2' C43' C34' 118.4(7) . . ? O1' C43' C34' 118.4(7) . . ? C42' N6' C34' 117.1(10) . . ? C42' N6' Ru1 112.2(11) . . ? C34' N6' Ru1 121.9(12) . . ? C43' O1' Ru1 118.9(9) . . ? C41' O3' Ru1 108.5(8) . . ? C5 N1 C1 119.0(7) . . ? C5 N1 Ru1 121.3(6) . . ? C1 N1 Ru1 119.6(6) . . ? C6 N2 C7 107.0(8) . . ? C6 N2 Ru1 113.5(6) . . ? C7 N2 Ru1 139.0(6) . . ? C6

N3 C12 108.3(7) . . ? C6 N3 C13 128.2(8) . . ? C12 N3 C13 122.7(8) . . ? C20 N4 C21 106.7(8) . . ? C20 N4 Ru1 115.1(6) . . ? C21 N4 Ru1 138.1(6) . . ? C20 N5 C26 105.2(7) . . ? C20 N5 C27 130.8(8) . . ? C26 N5 C27 123.6(9) . . ?

loop_ _geom_torsion_atom_site_label_1 _geom_torsion_atom_site_label_2 _geom_torsion_atom_site_label_3 _geom_torsion_atom_site_label_4 _geom_torsion _geom_torsion_site_symmetry_1 _geom_torsion_site_symmetry_2 _geom_torsion_site_symmetry_3 _geom_torsion_site_symmetry_4 _geom_torsion_publ_flag
N1 C1 C2 C3 -1.3(12) . . . . ? C6 C1 C2 C3 178.5(8) . . . . ? C1 C2 C3 C4 0.0(14) . . . . ? C2 C3 C4 C5 0.9(15) . . . . ? C3 C4 C5 N1 -0.5(13) . . . . ? C3 C4 C5 C20 -178.8(9) . . . . ? N1 C1 C6 N3 173.3(7) . . . . ? C2 C1 C6 N3 -6.5(14) . . . . ? N1 C1 C6 N2 -2.4(10) . . . . ? C2 C1 C6 N2 177.8(8) . . . . ? N2 C7 C8 C9 178.4(9) . . . . ? C12 C7 C8 C9 -0.1(12) . . . . ? C7 C8 C9 C10 -1.5(14) . . . . ? C8 C9 C10 C11 2.5(16) . . . . ? C9 C10 C11 C12 -1.7(14) . . . . ? C10 C11 C12 C7 0.0(13) . . . . ? C10 C11 C12 N3 -178.3(8) . . . . ? N2 C7 C12 C11 -177.9(7) . . . . ? C8 C7 C12 C11 0.9(13) . . . . ? N2 C7 C12 N3 0.7(9) . . . . ? C8 C7 C12 N3 179.6(7) . . . . ? N3 C13 C14 C15 -23.8(10) . . . . ? N3 C13 C14 C19 156.6(6) . . . . ? C19 C14 C15 C16 0.0 . . . . ? C13 C14 C15 C16 -179.6(8) . . . . ? C14 C15 C16 C17 0.0 . . . . ? C15 C16 C17 C18 0.0 . . . . ? C16 C17 C18 C19 0.0 . . . . ? C17 C18 C19 C14 0.0 . . . . ? C15 C14 C19 C18 0.0 . . . . ? C13 C14 C19 C18 179.6(7) . . . . ? N1 C5 C20 N4 1.9(10) . . . . ? C4 C5 C20 N4 -179.7(8) . . . . ? N1 C5 C20 N5 -176.5(7) . . . . ? C4 C5 C20 N5 1.9(15) . . . . ? N4 C21 C22 C23 -176.7(9) . . . . ? C26 C21 C22 C23 0.8(12) . . . . ?
C21 C22 C23 C24 -1.4(14) . . . . ? C22 C23 C24 C25 1.0(16) . . . . ? C23 C24 C25 C26 0.0(14) . . . . ? C24 C25 C26 N5 179.5(8) . . . . ? C24 C25 C26 C21 -0.5(13) . . . . ? N4 C21 C26 C25 178.1(8) . . . . ? C22 C21 C26 C25 0.1(12) . . . . ? N4 C21 C26 N5 -1.9(9) . . . . ? C22 C21 C26 N5 -179.9(7) . . . . ? N5 C27 C28 C29 36.6(16) . . . . ? N5 C27 C28 C33 -145.8(10) . . . . ? C33 C28 C29 C30 -3(2) . . . . ? C27 C28 C29 C30 174.9(14) . . . . ? C29 C28 C33 C32 2.0(19) . . . . ? C27 C28 C33 C32 -175.7(12) . . . . ? N6 C34 C35 C36 -6(3) . . . . ? C43 C34 C35 C36 -174(2) . . . . ? C34 C35 C36 O4 -178.7(19) . . . . ? C34 C35 C36 C37 0(3) . . . . ? O4 C36 C37 C38 -3(3) . . . . ? C35 C36 C37 C38 178.7(14) . . . . ? O4 C36 C37 C42 -177.8(16) . . . . ? C35 C36 C37 C42 4(2) . . . . ? C42 C37 C38 C39 0.0 . . . . ? C36 C37 C38 C39 -174.7(15) . . . . ? C37 C38 C39 C40 0.0 . . . . ? C38 C39 C40 C41 0.0 . . . . ? C39 C40 C41 O3 -177.3(12) . . . . ? C39 C40 C41 C42 0.0 . . . . ? O3 C41 C42 N6 -6.2(14) . . . . ? C40 C41 C42 N6 176.5(12) . . . . ? O3 C41 C42 C37 177.3(12) . . . . ? C40 C41 C42 C37 0.0 . . . . ? C38 C37 C42 N6 -176.1(13) . . . . ? C36 C37 C42 N6 -1.0(15) . . . . ? C38 C37 C42 C41 0.0 . . . . ? C36 C37 C42 C41 175.1(14) . . . . ? N6 C34 C43 O2 -168.5(18) . . . . ? C41 C42 N6 C34 178.1(14) . . . . ? C37 C42 N6 C34 -5.7(19) . . . . ? C41 C42 N6 Ru1 8.0(13) . . . . ? C37 C42 N6 Ru1 -175.8(7) . . . . ? C43 C34 N6 C42 -179.5(14) . . . . ? C35 C34 N6 Ru1 178.0(13) . . . . ? C43 C34 N6 Ru1 -10(2) . . . . ? O1' Ru1 N6 C34 -161.9(16) . . . . ? N2 Ru1 N6 C34 99.2(15) . . . . ? N4 Ru1 N6 C34 -82.7(15) . . . . ? O1 Ru1 N6 C34 6.9(17) . . . . ? O3 Ru1 N6 C34 -175.1(15) . . . . ? O3' Ru1 N6 C34 12(2) . . . . ? O2 C43 O1 Ru1 172.6(15) . . . . ? O3' Ru1 O1 C43 -121(15) . . . . ? C40 C41 O3 Ru1 178.8(6) . . . . ? C42 C41 O3 Ru1 1.5(12) . . . . ? N6 Ru1 O3 C41 2.2(10) . . . . ? N1 Ru1 O3 C41 177.5(9) . . . . ? N6' Ru1 O3 C41 -9.1(12) . . . . ? O1' Ru1 O3 C41 -55(2) . . . . ? N2 Ru1 O3 C41 99.0(9) . . . . ? N4 Ru1 O3 C41 -105.2(9) . . . . ? C40' C41' O3' Ru1 179.4(18) . . . . ? C4 C5 N1 C1 -0.7(12) . . . . ? C20 C5 N1 C1 177.9(7) . . . . ? C4 C5 N1 Ru1 -178.7(6) . . . . ? C20 C5 N1 Ru1 -0.2(9) . . . . ? C2 C1 N1 C5 1.6(11) . . . . ? C6 C1 N1 C5 -178.2(7) . . . . ? C2 C1 N1 Ru1 179.7(6) . . . . ? C6 C1 N1 Ru1 -0.2(8) . . . . ? O1' Ru1 N1 C5 81.3(8) . . . . ? N2 Ru1 N1 C5 179.7(6) . . . . ? N4 Ru1 N1 C5 -0.9(6) . . . . ? O1 Ru1 N1 C5 -90.4(11) . . . . ? O3 Ru1 N1 C5 93.5(7) . . . . ? O3' Ru1 N1 C5 -95.6(18) . . . . ? O1' Ru1 N1 C1 -96.7(8) . . . . ? N2 Ru1 N1 C1 1.6(5) . . . . ? N4 Ru1 N1 C1 -179.0(6) . . . . ? O1 Ru1 N1 C1 91.6(11) . . . . ? O3 Ru1 N1 C1 -84.5(6) . . . . ? O3' Ru1 N1 C1 86.4(18) . . . . ? N3 C6 N2 C7 0.9(9) . . . . ?

C1 C6 N2 C7 177.4(7) . . . . ? N3 C6 N2 Ru1 -172.8(5) . . . . ? C1 C6 N2 Ru1 3.8(9) . . . . ? C8 C7 N2 C6 -179.6(8) . . . . ? C12 C7 N2 C6 -1.0(9) . . . . ? C8 C7 N2 Ru1 -8.5(15) . . . . ? C12 C7 N2 Ru1 170.1(6) . . . . ? N6 Ru1 N2 C6 171.4(6) . . . . ? N1 Ru1 N2 C6 -2.8(5) . . . . ? N6' Ru1 N2 C6 172.9(7) . . . . ? O1' Ru1 N2 C6 96.7(6) . . . . ? N4 Ru1 N2 C6 -4.2(9) . . . . ? O1 Ru1 N2 C6 -112.1(6) . . . . ? O3 Ru1 N2 C6 89.9(6) . . . . ? O3' Ru1 N2 C6 -109.6(7) . . . . ? N6 Ru1 N2 C7 0.7(9) . . . . ? N1 Ru1 N2 C7 -173.5(8) . . . . ? N6' Ru1 N2 C7 2.2(10) . . . . ? O1' Ru1 N2 C7 -74.0(9) . . . . ? N4 Ru1 N2 C7 -175.0(7) . . . . ? O1 Ru1 N2 C7 77.2(9) . . . . ? O3 Ru1 N2 C7 -80.8(8) . . . . ? O3' Ru1 N2 C7 79.7(9) . . . . ? N2 C6 N3 C12 -0.4(9) . . . . ? C1 C6 N3 C12 -176.5(8) . . . . ? N2 C6 N3 C13 170.2(7) . . . . ? C1 C6 N3 C13 -5.8(13) . . . . ? C11 C12 N3 C6 178.3(9) . . . . ? C7 C12 N3 C6 -0.2(8) . . . . ? C11 C12 N3 C13 7.0(13) . . . . ? C7 C12 N3 C13 -171.5(7) . . . . ? C14 C13 N3 C6 -68.4(10) . . . . ? C14 C13 N3 C12 101.1(8) . . . . ? N5 C20 N4 C21 -1.6(9) . . . . ? C5 C20 N4 C21 179.7(7) . . . . ? N5 C20 N4 Ru1 176.0(5) . . . . ? C5 C20 N4 Ru1 -2.7(9) . . . . ? C22 C21 N4 C20 179.8(8) . . . . ? C26 C21 N4 C20 2.1(9) . . . . ? C22 C21 N4 Ru1 3.1(14) . . . . ? C26 C21 N4 Ru1 -174.6(6) . . . . ? N6 Ru1 N4 C20 -172.1(6) . . . . ? N1 Ru1 N4 C20 1.9(5) . . . . ? N6' Ru1 N4 C20 -173.9(7) . . . . ? O1' Ru1 N4 C20 -100.8(7) . . . . ? N2 Ru1 N4 C20 3.4(9) . . . . ? O1 Ru1 N4 C20 111.3(6) . . . . ? O3 Ru1 N4 C20 -89.1(6) . . . . ? O3' Ru1 N4 C20 106.7(8) . . . . ? N6 Ru1 N4 C21 4.4(9) . . . . ? N1 Ru1 N4 C21 178.4(8) . . . . ? N6' Ru1 N4 C21 2.6(9) . . . . ? O1' Ru1 N4 C21 75.7(9) . . . . ? N2 Ru1 N4 C21 179.8(7) . . . . ? O1 Ru1 N4 C21 -72.3(9) . . . . ? O3 Ru1 N4 C21 87.4(8) . . . . ? O3' Ru1 N4 C21 -76.9(10) . . . . ? N4 C20 N5 C26 0.4(9) . . . . ? C5 C20 N5 C26 178.9(8) . . . . ? N4 C20 N5 C27 -172.0(8) . . . . ? C5 C20 N5 C27 6.5(14) . . . . ? C25 C26 N5 C20 -179.1(9) . . . . ? C21 C26 N5 C20 0.9(8) . . . . ? C25 C26 N5 C27 -6.0(14) . . . . ? C21 C26 N5 C27 174.0(7) . . . . ? C28 C27 N5 C20 72.1(12) . . . . ? C28 C27 N5 C26 -99.1(10) . . . . ?

_diffrn_measured_fraction_theta_max 0.988 _diffrn_reflns_theta_full 22.58 _diffrn_measured_fraction_theta_full 0.988 _refine_diff_density_max 0.928 _refine_diff_density_min -1.045 _refine_diff_density_rms 0.106

start Validation Reply Form _vrf_THETM01_1;

PROBLEM: The value of sine(theta_max)/wavelength is less than 0.550

RESPONSE: This is due to the weak diffraction ability of the crystal selected for the X-ray experiment. Also the crystal is easily deteriorated when the samples are taken out from the mother liquid, even though the crystal is tested at low temperature(100K).

;_vrf_PLAT411_1;

PROBLEM: Short Inter H...H Contact H30 .. H40' .. 1.76 Ang. RESPONSE: This is due to the disorder of the ligand in the coordination molecue which shoud be omitted.

;_vrf_PLAT602_1;

PROBLEM: VERY LARGE Solvent Accessible VOID(S) in Structure ! Info

RESPONSE: Beacuse of the weak diffraction ability of the crystal selected for the X-ray experiment, it is difficult to locate the position of the solvent molecules.In the final refinement, the procedure 'SQUEEZE/PLATON' was applied to treatthe highly disordered solvent.;# end Validation Reply Form