

This article was downloaded by:

On: 23 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713455674>

Syntheses and Crystal Structures of Two Ion Pair Complexes, [Ru(bpy)₃]₂[Fe(CN)₆]I · 7H₂O and [Ru(bpy)₃]- [Fe(CN)₅NO]CH₃OH · H₂O

Wen Dong^a; Si Shu-Feng^a; Dia-Zheng Liao^a; Zong-Hui Jiang^{ab}; Shi-Ping Yan^a

^a Department of Chemistry, Nankai University, Tianjin, P.R. China ^b State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing, P.R. China

Online publication date: 15 September 2010

To cite this Article Dong, Wen , Shu-Feng, SI , Liao, Dia-Zheng , Jiang, Zong-Hui and Yan, Shi-Ping(2003) 'Syntheses and Crystal Structures of Two Ion Pair Complexes, [Ru(bpy)₃]₂[Fe(CN)₆]I · 7H₂O and [Ru(bpy)₃]- [Fe(CN)₅NO]CH₃OH · H₂O', *Journal of Coordination Chemistry*, 56: 6, 531 – 538

To link to this Article: DOI: 10.1080/0095897031000101448

URL: <http://dx.doi.org/10.1080/0095897031000101448>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

Note

SYNTHESES AND CRYSTAL STRUCTURES OF TWO ION PAIR COMPLEXES, [Ru(bpy)₃]₂[Fe(CN)₆]I · 7H₂O AND [Ru(bpy)₃]- [Fe(CN)₅NO]CH₃OH · H₂O

WEN DONG^a, SHU-FENG SI^a, DIA-ZHENG LIAO^{a,*},
ZONG-HUI JIANG^{a,b} and SHI-PING YAN^a

^aDepartment of Chemistry, Nankai University, Tianjin 300071, P.R. China; ^bState Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093 P.R. China

(Received 15 October 2002; In final form 30 January 2003)

Two ion pair complexes, [Ru(bpy)₃]₂[Fe(CN)₆]I · 7H₂O (**1**) and [Ru(bpy)₃][Fe(CN)₅NO](CH₃OH) · H₂O (**2**) (bpy = 2,2-bipyridine) have been synthesized and structurally characterized. X-Ray crystallographic structures of **1** and **2** both show Fe(III) and Ru(II) in distorted octahedral environments. In both complexes, H-bonding interactions between an uncoordinated water molecule and the nitrogen atom of a cyano group exist.

Keywords: Crystal structures; Ruthenium(II); H-bonding

INTRODUCTION

The photophysical properties of [Ru(bpy)₃]²⁺ have been studied intensively during the past four decades [1–8]. It has been found that excited states of [Ru(bpy)₃]²⁺ and other related complexes, having relatively long lifetimes, can be quenched by energy transfer, complex formation, and electron transfer processes. Ayersin *et al.* reported a radiationless energy transfer from [Ru(bpy)₃]²⁺ to [Os(bpy)₃]²⁺ in a crystal of [Ru(bpy)₃]₂X₂ containing [Os(bpy)₃]²⁺ [4]. Intermolecular energy-transfer in a salt composed of ruthenium (II) and chromium(III) has been investigated [9]. Electron-transfer quenching in [Ru(bpy)₃]₂[M(CN)₆]Cl · 8H₂O (M = Co³⁺ or Fe³⁺) was also reported by Ikeda and Ohno [10]. An energy-transfer reaction from ruthenium(II) to osmium(II) in a mixed crystal, {Δ-[Ru(phen)₃]}{Λ-[Os(phen)₃]}(PF₆)₄ (phen = 1,10-phenanthroline) was also reported. In addition, metal cyanide compounds have also attracted great attention because of their remarkable magnetic, magneto-optical and optoelectronic properties [11]. However, bimetallic compounds containing both [Ru(bpy)₃]²⁺ and [M(CN)₆]⁻⁽⁶⁻ⁿ⁾ or [M(CN)₅NO]⁻⁽⁵⁻ⁿ⁾ are important for quantitative studies of the rate

*Corresponding author.

of electron transfer which an excited ion undergoes with its neighboring ion in ion pair compounds [9,10,12]. Focusing our interests on the compounds of this kind, we have synthesized two novel ion pair compounds, $[\text{Ru}(\text{bpy})_3]_2[\text{Fe}(\text{CN})_6]\text{I} \cdot 7\text{H}_2\text{O}$ (**1**) and $[\text{Ru}(\text{bpy})_3][\text{Fe}(\text{CN})_5\text{NO}](\text{CH}_3\text{OH}) \cdot \text{H}_2\text{O}$ (**2**). They have been characterized and their structures determined.

EXPERIMENTAL

Materials and Synthesis

All starting materials were of analytical grade and purchased from Tianjin Chemical Company. $[\text{Ru}(\text{bpy})_3]\text{I}_2 \cdot 6\text{H}_2\text{O}$ was prepared by a literature method [13].

*$[\text{Ru}(\text{bpy})_3]_2[\text{Fe}(\text{CN})_6]\text{I} \cdot 7\text{H}_2\text{O}$, **1***

A solution of 32.9 mg (0.1 mmol) of $\text{K}_3[\text{Fe}(\text{CN})_6]$ in 10 mL of water was added dropwise to a stirred solution of 170.4 mg (0.2 mmol) of $[\text{Ru}(\text{bpy})_3]\text{I}_2 \cdot 6\text{H}_2\text{O}$ in 40 mL $\text{H}_2\text{O}/\text{CH}_3\text{OH}$ (3:1). Single crystals suitable for X-ray diffraction studies were obtained by slow evaporation of the resulting solution at room temperature. Yield: 134.6 mg (84%). *Anal.* Calcd (found) for $\text{C}_{66}\text{H}_{60}\text{FeN}_{18}\text{O}_7\text{Ru}$ (%): C, 49.41 (49.43); H, 3.93 (3.74); N, 15.58 (15.72).

*$[\text{Ru}(\text{bpy})_3][\text{Fe}(\text{CN})_5\text{NO}]\text{CH}_3\text{OH} \cdot 3\text{H}_2\text{O}$, **2***

The complex was synthesized as described for **1**, with $\text{K}_3[\text{Fe}(\text{CN})_5\text{NO}] \cdot 2\text{H}_2\text{O}$ instead of $\text{K}_3[\text{Fe}(\text{CN})_6]$. Yield: 121.75 mg (76%). *Anal.* Calcd (found) for $\text{C}_{36}\text{H}_{33}\text{FeN}_{12}\text{O}_5\text{Ru}$ (%): C, 49.22 (49.61); H, 3.59 (3.79); N, 18.93 (19.29).

Physical Measurements

IR spectra were recorded in KBr discs on a Shimadzu IR-408 infrared spectrophotometer in the $4000\text{--}600\text{ cm}^{-1}$ region. Elemental analyses were carried out using a Perkin-Elmer model 240 instrument.

X-ray Crystallography

Two selected crystals of dimensions $0.30\text{ mm} \times 0.25\text{ mm} \times 0.20\text{ mm}$ were sealed in two Lindemann capillaries. Determination of the unit cell constants and data collection was performed on a Bruker Smart 1000 area detector diffractometer using graphite-monochromatized Mo- $\text{K}\alpha$ radiation ($\lambda = 0.71073\text{ \AA}$) at 293(2) K. An $\omega\text{--}2\theta$ scan mode was employed for data collection in the range $1.71 \leq \theta \leq 23.32^\circ$ for Complex **1** and $1.63 \leq \theta \leq 23.29^\circ$ Complex **2**. A total of 9974 [$R_{\text{int}} = 0.0786$] independent reflections for **1** were collected, of which 6262 [$I > 2\sigma(I)$] were considered to be observed and used in the succeeding structure determination and refinement. A total of 13053 [$R_{\text{int}} = 0.1000$] independent reflections for **2** were collected from which 5519 [$I > 2\sigma(I)$] of which to be considered observed and used in the succeeding structure determination and refinement. Empirical absorption corrections were applied using

the SADABS program [14]. The structures were solved by direct methods and successive Fourier difference syntheses and refined by full-matrix least-squares procedures on F^2 with anisotropic thermal parameters for all nonhydrogen atoms [15]. Hydrogen atoms were positioned geometrically and refined isotropically. Final atomic coordinates for all nonhydrogen atoms and equivalent thermal parameters are given in Table I for Complex **1** and Table II for Complex **2**.

TABLE I Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Compound **1**

	x/a	y/b	z/c	U_{eq}
Ru(1)	993(1)	3254(1)	3249(1)	32(1)
Ru(2)	995(1)	6623(1)	3131(1)	31(1)
Fe(1)	5887(3)	4968(1)	680(2)	31(1)
I(1)	-128(1)	6729(1)	5588(1)	59(1)
N(1)	-312(11)	3342(7)	3787(6)	30(4)
N(2)	143(13)	3841(6)	2738(6)	31(4)
N(3)	1770(12)	3910(6)	3688(6)	32(4)
N(4)	2314(11)	3305(7)	2708(5)	33(4)
N(5)	1615(11)	2623(6)	3811(7)	31(4)
N(6)	478(11)	2541(6)	2794(6)	31(4)
N(7)	62(13)	6054(6)	3618(6)	31(4)
N(8)	-322(12)	6529(7)	2549(7)	42(5)
N(9)	401(12)	7344(6)	3562(7)	34(4)
N(10)	1616(12)	7251(7)	2599(7)	38(4)
N(11)	2237(11)	6572(6)	3178(6)	28(4)
N(12)	1779(13)	5965(7)	2719(7)	41(5)
N(13)	5839(14)	4817(9)	-707(8)	56(6)
N(14)	5616(16)	6256(8)	612(9)	66(6)
N(15)	3506(17)	4816(9)	700(8)	60(6)
N(16)	5928(17)	5103(9)	2073(9)	76(7)
N(17)	5959(16)	3705(8)	784(9)	63(6)
N(18)	8246(16)	5169(8)	662(9)	65(6)
C(1)	-508(16)	3098(9)	4341(8)	42(6)
C(2)	-1310(16)	3226(10)	4718(10)	48(6)
C(3)	-2013(19)	3622(10)	4523(9)	54(7)
C(4)	-1848(17)	3873(10)	3928(10)	55(7)
C(5)	-1016(17)	3757(9)	3585(8)	42(6)
C(6)	-804(15)	3992(8)	2972(8)	30(5)
C(7)	-1460(16)	4350(8)	2668(9)	42(5)
C(8)	-1189(19)	4565(9)	2104(10)	51(7)
C(9)	-280(20)	4447(8)	1882(10)	53(7)
C(10)	392(17)	4076(8)	2185(8)	40(5)
C(11)	1488(18)	4203(9)	4195(9)	48(6)
C(12)	2120(20)	4573(11)	4496(12)	68(8)
C(13)	3070(20)	4694(9)	4249(9)	48(7)
C(14)	3380(17)	4431(9)	3733(9)	52(6)
C(15)	2734(17)	4038(9)	3456(8)	38(5)
C(16)	2995(14)	3732(8)	2881(8)	32(5)
C(17)	3867(14)	3841(9)	2525(9)	40(6)
C(18)	4043(17)	3575(10)	1991(10)	48(6)
C(19)	3341(19)	3147(10)	1823(8)	52(7)
C(20)	2495(17)	3026(9)	2171(9)	45(6)
C(21)	2105(16)	2701(10)	4346(9)	57(6)
C(22)	2405(17)	2247(12)	4736(10)	66(8)
C(23)	2127(17)	1732(12)	4561(10)	56(7)
C(24)	1647(19)	1623(9)	4023(11)	66(8)
C(25)	1361(15)	2079(9)	3634(8)	40(5)

(continued)

TABLE I Continued

	x/a	y/b	z/c	U_{eq}
C(26)	785(15)	2020(8)	3037(9)	38(6)
C(27)	592(18)	1522(9)	2718(11)	61(7)
C(28)	166(18)	1496(10)	2152(10)	57(7)
C(29)	-131(17)	2030(10)	1920(10)	56(7)
C(30)	-25(16)	2530(9)	2251(9)	45(6)
C(31)	302(16)	5828(7)	4190(7)	33(5)
C(32)	-420(20)	5482(9)	4488(12)	59(7)
C(33)	-1320(20)	5329(10)	4213(10)	55(7)
C(34)	-1548(17)	5536(8)	3630(9)	46(6)
C(35)	-837(16)	5900(8)	3344(8)	33(5)
C(36)	-1040(16)	6140(8)	2730(8)	34(5)
C(37)	-1855(17)	6001(10)	2349(10)	56(7)
C(38)	-2061(19)	6251(10)	1772(10)	59(7)
C(39)	-1313(18)	6642(11)	1602(9)	59(7)
C(40)	-475(16)	6787(10)	1986(8)	44(5)
C(41)	-124(16)	7356(9)	4090(9)	45(6)
C(42)	-255(18)	7889(12)	4407(11)	64(8)
C(43)	140(20)	8387(10)	4161(9)	66(8)
C(44)	611(15)	8360(8)	3636(9)	48(6)
C(45)	760(15)	7842(9)	3328(10)	40(6)
C(46)	1409(17)	7783(10)	2757(9)	49(6)
C(47)	1729(16)	8237(9)	2409(11)	54(6)
C(48)	2250(20)	8189(13)	1875(11)	69(8)
C(49)	2492(15)	7613(11)	1718(9)	59(7)
C(50)	2170(16)	7166(10)	2085(8)	49(6)
C(51)	2397(17)	6867(10)	4254(9)	44(6)
C(52)	3259(16)	6750(10)	4639(9)	46(6)
C(53)	3951(17)	6348(10)	4472(11)	54(7)
C(54)	3827(16)	6064(9)	3924(9)	42(6)
C(55)	2962(15)	6189(8)	3556(8)	34(5)
C(56)	2749(16)	5868(8)	2988(8)	33(5)
C(57)	3408(15)	5506(9)	2673(10)	52(7)
C(58)	3190(20)	5218(10)	2145(10)	55(7)
C(59)	2164(19)	5314(9)	1932(10)	50(7)
C(60)	1529(17)	5676(9)	2241(8)	46(6)
C(61)	5859(16)	4886(9)	-196(9)	36(5)
C(62)	5740(17)	5774(9)	614(9)	44(6)
C(63)	4410(20)	4879(9)	701(10)	46(7)
C(64)	5915(16)	5048(9)	1553(9)	41(6)
C(65)	5992(18)	4192(9)	762(10)	41(6)
C(66)	7408(19)	5094(8)	687(9)	37(6)
O(1)	5021(14)	2567(5)	708(8)	63(5)
O(2)	1436(11)	4506(6)	640(7)	63(4)
O(3)	6933(12)	2734(7)	5693(8)	80(5)
O(4)	6078(16)	4599(5)	3201(8)	72(5)
O(5)	10305(10)	5548(6)	734(8)	72(5)
O(6)	1219(11)	6574(6)	670(8)	82(5)
O(7)	3307(14)	6215(8)	730(11)	136(8)

Crystal Data

[Ru(bpy)₃]₂[Fe(CN)₆]I · 7H₂O (**1**), $M = 1602.21$, monoclinic, $P2_1/c$, $a = 11.178(3)$, $b = 14.824(4)$, $c = 23.166(6)$ Å, $\beta = 91.429(5)^\circ$, $V = 6724(3)$ Å³, $Z = 4$, $F(000) = 3200$, $D_c = 1.583$ g cm⁻³, $\mu = 1.182$ mm⁻¹; [Ru(bpy)₃][Fe(CN)₅NO](CH₃OH) · H₂O (**2**), $M = 688.61$, monoclinic, Cc , $a = 12.959(4)$, $b = 23.733(7)$, $c = 21.828(6)$ Å, $\beta = 91.429(5)^\circ$, $V = 3358.7(14)$ Å³, $Z = 4$, $F(000) = 1388$, $D_c = 1.362$ g cm⁻³, $\mu = 0.966$ mm⁻¹.

TABLE II Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Compound **2**

	x/a	y/b	z/c	U_{eq}
Ru(1)	819(1)	6976(1)	1731(1)	34(1)
Fe(1)	4498(1)	6474(1)	4161(1)	38(1)
N(1)	-269(6)	7305(4)	2406(3)	38(2)
N(2)	-623(6)	6128(5)	1577(3)	43(2)
N(3)	1676(6)	5897(4)	2117(3)	37(2)
N(4)	1867(5)	6519(4)	1070(3)	36(2)
N(5)	2138(5)	7897(4)	1919(3)	34(2)
N(6)	203(5)	8098(5)	1285(2)	33(2)
N(7)	1858(7)	6842(5)	3838(3)	64(2)
N(8)	4977(6)	6322(5)	2858(3)	50(2)
N(9)	4990(7)	8509(6)	3992(3)	64(2)
N(10)	7213(7)	6222(5)	4336(3)	60(2)
N(11)	4105(7)	4431(5)	4062(3)	59(2)
N(12)	4165(6)	6487(5)	4848(3)	46(2)
C(1)	-15(7)	7918(6)	2818(3)	44(2)
C(2)	-799(10)	8113(6)	3259(4)	61(3)
C(3)	-1863(10)	7672(7)	3270(5)	71(3)
C(4)	-2132(8)	7051(7)	2863(4)	61(3)
C(5)	-1336(7)	6872(6)	2416(4)	46(2)
C(6)	-1529(8)	6199(6)	1969(4)	47(2)
C(7)	-2533(9)	5639(8)	1925(5)	76(3)
C(8)	-2615(10)	5007(8)	1503(5)	87(4)
C(9)	-1707(10)	4924(7)	1112(5)	83(4)
C(10)	-714(8)	5505(6)	1163(4)	59(3)
C(11)	1518(7)	5590(6)	2658(4)	44(2)
C(12)	2133(8)	4863(6)	2885(4)	52(3)
C(13)	2939(7)	4427(6)	2559(4)	50(2)
C(14)	3146(7)	4724(6)	1999(4)	42(2)
C(15)	2503(7)	5454(6)	1790(3)	35(2)
C(16)	2609(7)	5800(5)	1194(3)	36(2)
C(17)	3391(7)	5465(6)	796(4)	50(3)
C(18)	3415(8)	5835(6)	257(4)	56(3)
C(19)	2669(8)	6529(6)	114(3)	48(2)
C(20)	1902(7)	6841(6)	531(4)	46(2)
C(21)	3085(7)	7778(6)	2274(3)	42(2)
C(22)	3961(7)	8408(6)	2380(4)	49(2)
C(23)	3882(8)	9200(6)	2086(4)	60(3)
C(24)	2928(8)	9368(6)	1721(3)	45(2)
C(25)	2041(7)	8722(6)	1650(3)	34(2)
C(26)	937(7)	8828(6)	1293(3)	34(2)
C(27)	629(8)	9598(6)	993(4)	50(2)
C(28)	-453(8)	9637(6)	699(3)	49(2)
C(29)	-1206(8)	8906(6)	683(3)	45(2)
C(30)	-846(7)	8147(6)	981(3)	43(2)
C(31)	2867(8)	6696(6)	3941(3)	44(2)
C(32)	4799(7)	6385(5)	3345(4)	35(2)
C(33)	4800(8)	7762(6)	4068(4)	43(3)
C(34)	6210(8)	6306(6)	4269(3)	42(2)
C(35)	4269(7)	5195(6)	4104(4)	43(2)
C(36)	4658(12)	2105(9)	3711(6)	141(5)
O(1)	3845(6)	6448(5)	5313(3)	89(2)
O(2)	829(8)	1006(7)	5300(3)	156(4)
O(3)	9768(6)	7095(5)	4552(3)	98(2)
O(4)	3095(12)	1412(7)	4933(5)	229(6)
O(5)	3684(8)	2585(6)	3921(6)	192(6)

RESULTS AND DISCUSSIONS

Spectroscopic Characterization

IR spectra of **1** and **2** exhibit bands expected for terminal CN stretching at 2110, 2050 cm^{-1} (**1**) and 2140 cm^{-1} (**2**) [16], and bands for 2,2-bipy at 1630, 1600, 1460 and 1420 cm^{-1} . Complex **2** displays an NO stretch at 1920 cm^{-1} indicating an NO^+ complex of iron(II) with extensive π bonding [17]. Studies focusing on ion pair charge transfer of the present compounds are in progress.

Description of the Structures

Perspective views of **1** and **2** are shown in Figs. 1 and 2, respectively. Complex **1** is comprised of $[\text{Ru}(\text{bpy})_3]^{2+}$ cations and $[\text{Fe}(\text{CN})_6]^{3-}$ anions, whose charge is balanced by one I^- ion. $[\text{Ru}(\text{bpy})_3]^{2+}$ and $[\text{Fe}(\text{CN})_6]^{3-}$ are approximately octahedral in Complex **1** and the average Ru(1)–N bond distance is 2.085 Å; Ru(2)–N is 2.087 Å, is longer than (2.056 Å) in $[\text{Ru}(\text{bpy})_3](\text{PF}_6)$ [18]. This bonding difference implies that the structure and excited-state behavior are different. The average Fe(1)–C distance in Complex **1** is 1.923 Å, which is normal. Complex **2** is comprised of $[\text{Ru}(\text{bpy})_3]^{2+}$ cations and $[\text{Fe}(\text{CN})_6]^{3-}$ anions. The average Ru(1)–N bond distance is 2.061 Å, similar to that in $[\text{Ru}(\text{bpy})_3](\text{PF}_6)$ and this has theoretical and photophysical significance. The average Fe(1)–C bond distance is 1.929 Å, and the Fe(1)–N(12) is 1.645 Å, which is normal.

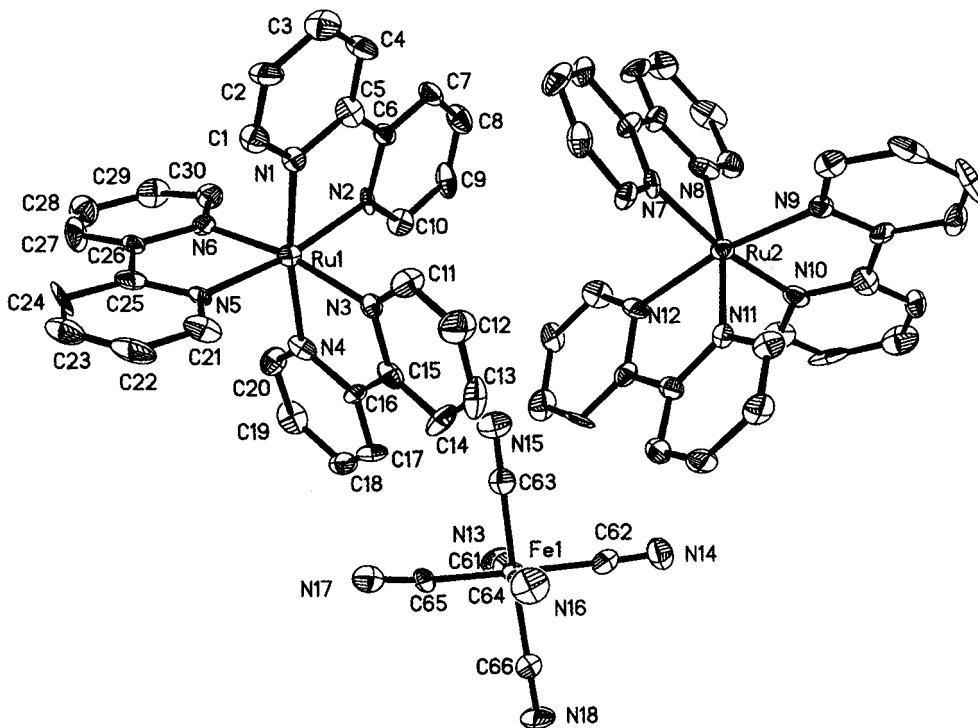


FIGURE 1 Perspective view of Complex **1** in labeling scheme.

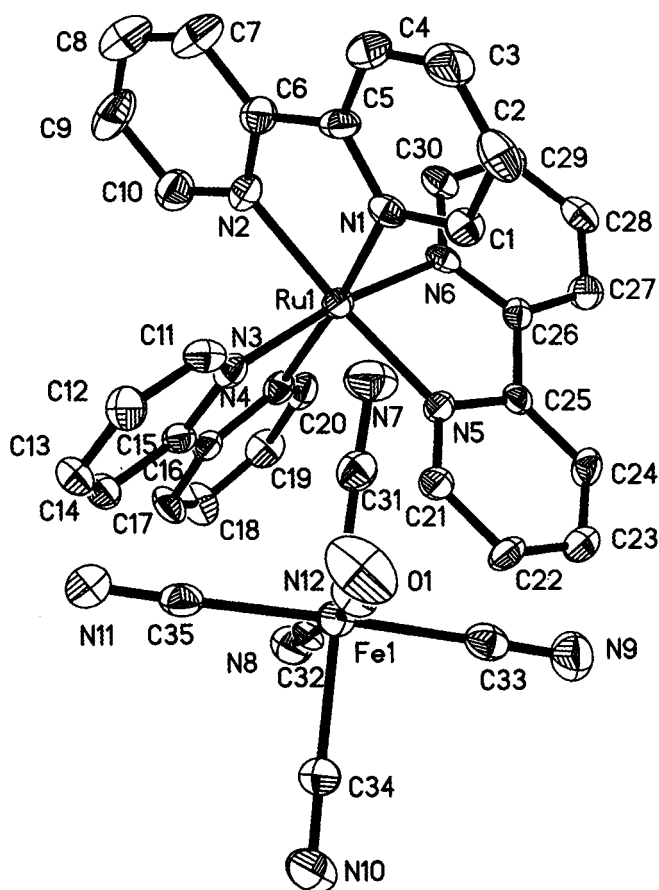


FIGURE 2 Perspective view of Complex 2 in labeling scheme.

TABLE III Selected bondlengths (Å) and angles (°) for Compound 1

Ru(1)–N(1)	2.081(14)	Ru(1)–N(2)	2.091(14)
Ru(2)–N(10)	2.082(16)	Ru(2)–N(9)	2.087(15)
Fe(1)–C(64)	1.91(2)	Fe(1)–C(61)	1.922(19)
Fe(1)–C(63)	1.92(3)	Fe(1)–C(62)	1.93(2)
N(6)–Ru(1)–N(1)	95.4(6)	N(6)–Ru(1)–N(3)	169.8(6)
N(1)–Ru(1)–N(2)	78.9(6)	N(3)–Ru(1)–N(2)	89.7(5)
N(2)–Ru(1)–N(5)	170.8(6)	N(5)–Ru(1)–N(4)	93.5(6)
N(7)–Ru(2)–N(10)	170.5(6)	N(7)–Ru(2)–N(8)	78.4(7)
N(10)–Ru(2)–N(8)	93.7(6)	N(7)–Ru(2)–N(9)	96.1(6)
N(7)–Ru(2)–N(12)	91.3(5)	N(10)–Ru(2)–N(12)	94.6(6)
C(65)–Fe(1)–C(64)	90.1(9)	C(64)–Fe(1)–C(61)	179.8(10)
C(65)–Fe(1)–C(63)	87.7(10)	C(61)–Fe(1)–C(63)	90.3(9)
C(65)–Fe(1)–C(62)	178.1(11)	C(65)–Fe(1)–C(66)	94.4(9)

In both complexes, O–H···N hydrogen bonds between uncoordinated water molecules and CN or ON groups exist. In **1**, a 2D network is formed by H-bonding and [Ru(bpy)₃]²⁺ is symmetrically located on both sides of the formal plane. Selected bond lengths and angles are presented in Table III for **1** and Table IV for **2**, respectively.

TABLE IV Selected bondlengths (Å) and angles (°) for compound 2

Ru(1)–N(3)	2.058(6)	Ru(1)–N(4)	2.065(6)
Ru(1)–N(1)	2.063(6)	Ru(1)–N(2)	2.068(7)
Fe(1)–N(12)	1.645(7)	Fe(1)–C(31)	1.909(9)
Fe(1)–C(33)	1.952(10)	Fe(1)–C(34)	1.940(9)
N(5)–Ru(1)–N(3)	95.8(2)	N(5)–Ru(1)–N(4)	87.5(2)
N(3)–Ru(1)–N(4)	78.5(3)	N(4)–Ru(1)–N(1)	174.5(3)
N(1)–Ru(1)–N(6)	89.6(2)	N(6)–Ru(1)–N(2)	98.9(3)
N(12)–Fe(1)–C(35)	92.6(4)	N(12)–Fe(1)–C(32)	175.6(3)
C(35)–Fe(1)–C(32)	83.8(3)	C(31)–Fe(1)–C(32)	86.5(3)
C(32)–Fe(1)–C(33)	85.7(3)	N(12)–Fe(1)–C(34)	97.0(3)
C(35)–Fe(1)–C(34)	90.7(4)	C(31)–Fe(1)–C(34)	171.6(3)

Acknowledgment

The work was supported by the National Natural Sciences Foundation of China (No.20071019, No.50172021, No.90101028).

Supplementary Data

Full Crystallographic details are available from the authors.

References

- [1] J.N. Demas and A.W. Adanson, *J. Am. Chem. Soc.* **93**, 1801 (1971).
- [2] G. Sprintschnik, H.W. Sprintschnik, P.P. Kirsch and D.G. Whitley, *J. Am. Chem. Soc.* **99**, 4947 (1977).
- [3] H. Yesin, E. Gallhuber, A. Volger and H. Kunkely, *J. Am. Chem. Soc.* **105**, 4155 (1983).
- [4] H. Yesin, E. Braun, E. Gallhuber and G. Hensler, *Ber.* **91**, 1228 (1987).
- [5] E. Gallhuber, G. Hensler and H. Yersin, *J. Am. Chem. Soc.* **109**, 4818 (1987).
- [6] B. Jing, A. Song, M. Zhang and T. Shen, *Chem. Lett.* 789 (1999).
- [7] K. Shinozaki, Y. Hotta, T. Otsuka and Y. Kaizu, *Chem. Lett.* 101 (1999).
- [8] F. Barigelletti and L. Flamigni, *Chem. Soc. Rev.* **29**, 1 (2000).
- [9] I. Fujita and H. Kobayashi, *J. Chem. Phys.* **59**, 2902 (1973).
- [10] T. Iguro, N. Ikeda and T. Ohno, *Inorg. Chem. Acta* **226**, 203, (1994).
- [11] K.R. Dunbar and R.A. Hientz, *Prog. Inorg. Chem.* **45**, 283 (1997); L.M. Baraldo, P. Forlano, A.R. Parise, L.D. Slep and J.A. Olabe, *Coord. Chem. Rev.* **219–221**, 881 (2001).
- [12] M. Ohba and H. Okawa, *Coord. Chem. Rev.* **198**, 313 (2000).
- [13] H. Burstall, *J. Chem. Soc.* 173 (1936).
- [14] SAINT, Version 4.0, Data Integration Software (Bruker AXS. Inc., Madison, WI, 1997).
- [15] G.M. Sheldrick, SHELXS-97 and SHELXL-97, Software for Crystal Structure Analysis. (Seimens Analytical X-ray Instruments Inc., Madison, Wisconsin, 1997).
- [16] G. Wilkinson, R.D. Gillard and J.A. Mcleverty, *Comprehensive Coordination Chemistry*, Middle Transition Elements Vol. 4. (Pergamon Press, Oxford, 1987), pp. 1189–1190.
- [17] D.P. Rileema, D.S. Jones, C. Woods and A.A. Levy, *Inorg. Chem.* **31**, 2935, (1992).
- [18] G.O. Morpurgo, V. Mosini, P. Porta, G. Dessy and V. Fares, *J. Chem. Soc., Dalton Trans.* 1272 (1980).