Complexes of the Platinum Metals. Part 38.[1] The Synthesis and *X*-Ray Crystal Structure of an Asymmetric Binuclear Ruthenium(I, I) Di-*p*-Tolyltriazenido Complex $[Ru_2(\mu\text{-}RNNNR)(RNNNR)(\mu\text{-}CO)_2(CO)_2(bipy)]$(R = *p*-tolyl, bipy = 2,2′-bipyridyl)†

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The binuclear ruthenium(I, I) triazenido complex $[Ru_2(RNNNR)_2(CO)_6]$(R = *p*-tolyl) reacts with excess of 2,2′-bipyridyl (bipy) to afford a substitution product $[Ru_2(RNNNR)_2(CO)_4(bipy)]$ which has been shown by *X*-ray diffraction methods to contain a pair of non-equivalent ruthenium atoms linked by a metal–metal bond (2.706Å) and bridged by a 1,3-di-*p*-tolyltriazenide ligand and a pair of carbonyls. The co-ordination sphere of the first ruthenium atom is completed by a chelate triazenide ligand and a terminal carbonyl, that of the second ruthenium atom by a chelate bipy ligand and a terminal carbonyl.

As part of an on-going study of platinum-group metal 1,3-diaryltriazenido complexes we recently prepared 1,3-diaryltriazenido species $[Ru_2(\mu\text{-}RNNNR)_2(CO)_6]$, analogous to the well known binuclear ruthenium(I,I) carboxylates $[Ru_2(\mu\text{-}O_2CR)_2(CO)_6]$,[2] and showed that they readily undergo axial carbonyl-substitution reactions to give products of the form $[Ru_2(\mu\text{-}RNNNR)_2(CO)_4L_2]$.[3,4] One of these products $[Ru_2(\mu\text{-}RNNNR)_2(CO)_4(NH_2R)_2]$ (R = *p*-tolyl) has been characterised by *X*-ray diffraction methods.[4] We now report a reaction between $[Ru_2(\mu\text{-}RNNNR)_2(CO)_6]$ (R = *p*-tolyl) and 2,2′-bipyridyl leading to a binuclear product $[Ru_2(RNNNR)_2(CO)_4(bipy)]$ which has been shown by *X*-ray diffraction methods to possess an asymmetric structure with bridging and chelate triazenide ligands. The relationship between this product and the recently reported[5] cationic complex $[Ru_2(\mu\text{-}O_2CMe)(\mu\text{-}CO)_2(CO)_2(bipy)_2]^+$ is discussed.

Experimental

The complex $[Ru_2(\mu\text{-}RNNNR)_2(CO)_6]$(R = *p*-tolyl) was synthesised as previously described.[4]

$[Ru_2(\mu\text{-}RNNNR)(RNNNR)(\mu\text{-}CO)_2(CO)_2(bipy)]$.—A mixture of $[Ru_2(\mu\text{-}RNNNR)_2(CO)_6]$ (0.1 g) and 2,2′-bipyridyl (0.05 g) in benzene (15 cm^3) was heated under reflux for 2 h to form an orange solution which was cooled, filtered, and then evaporated to dryness under reduced pressure. The residual oil was triturated with ethanol to yield an orange solid (0.08 g) which was crystallised from methylene chloride-ethanol to form orange plates, m.p. (sealed under nitrogen) 130—134 °C (decomp.) (Found: C, 55.45; H, 3.85; N, 12.2. Calc. for $C_{42}H_{36}N_8O_4Ru_2$: C, 54.9, H, 3.95; N, 12.2%). Infrared ($CHCl_3$ solution): ν(CO) 2 030vs, 1 980m (terminal); 1 790w, 1 730vs (bridging); other bands *ca.* 2 900vbr, 1 500m, 1 365s, 1 300s, and 1 280m cm^{-1}. N.m.r. (CD_2Cl_2 internal standard $SiMe_4$): ^1H, *asym.* triazenide, δ 2.00 and 2.15 (each 3 H, s, MeC_6H_4), 5.50, 6.27, 6.42, and 6.64 [each 2 H, d, $^3J(HH')$ *ca.* 10, MeC_6H_4]; *sym.* triazenide, 2.40 (6 H, s, MeC_6H_4), 7.17 and 7.55 [each 4 H, d, $^3J(HH')$ *ca.* 10 Hz, MeC_6H_4]; 2,2′-bipyridyl, 7.87 (4 H, m), 8.05 (2 H, m), and 10.22 (2 H, m). $^{13}C\text{-}\{^1H\}$ (62.9 MHz), terminal CO, δ 196.3, 194.7; bridging CO, 258.8; *asym.* triazenide, 147.5, 144.9, 135.5, 134.9, 128.2, 127.5, 125.5, 124.6, 21.0, and 20.7 (all singlets); *sym.* triazenide, 155.6, 132.7, 129.4, 118.8, and 21.1 (all singlets); 2,2′-bipyridyl, 148.7, 148.3, 139.2, 126.8, and 122.7 p.p.m. (all singlets).

Crystal Structure Determination of $[Ru_2(RNNNR)_2(CO)_4(bipy)]$ (R = *p-tolyl*).—*Crystal data.* $C_{42}H_{36}N_8O_4Ru_2$, M = 919.0, monoclinic, space group $P2_1/n$, a = 12.498(5), b = 29.364(11), c = 23.026(10) Å, β = 102.55(3)°, Z = 8, U = 8 250(5) Å^3, $F(000)$ = 3 712, $\mu(Mo\text{-}K_\alpha)$ = 7.39 cm^{-1}, D_c = 1.48 g cm^{-3}, orange air-stable plate-shaped crystal, dimensions 0.25 × 0.20 × 0.05 mm.

Data collection and processing. Nicolet R3mV diffractometer, graphite-monochromated Mo-K_α radiation (λ = 0.710 73 Å); lattice vectors identified by application of the automatic indexing routine of the diffractometer to positions of 18 reflections ($21 < 2\theta < 30°$) taken from a rotation photograph and centred by the diffractometer; 10 243 reflections measured ($5 < 2\theta < 50°$, 0 to h, 0 to k, $-l$ to $+l$), 9 433 unique reflections corrected for Lorentz, polarisation, and absorption effects,[6] giving 3 680 reflections with $I > 3\sigma(I)$. No systematic variation in the intensity of the standard reflections was found.

Structure analysis and refinement. Direct methods for four ruthenium atoms (two independent molecules in the asymmetric unit) followed by Fourier-difference synthesis to locate remaining non-hydrogen atoms; full-matrix least-squares refinement for these atoms.[6] In the final refinement, ruthenium, oxygen, and nitrogen atoms were refined anisotropically. Hydrogen atoms were not included in the refinement. At the end of the refinement (589 parameters) the final R and R' values were 0.0881 and 0.0702 respectively [weighting scheme $w^{-1} = \sigma^2(F) + 0.000\,063F^2$]. The last cycle did not shift any parameter by more than 0.002 times its estimated standard deviation (e.s.d.) and the largest peak in the final Fourier-difference synthesis was less than 0.75e Å^{-3}. Atomic positional parameters are given in Table 1, selected bond lengths and interbond angles in Table 2.

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† 1-(2,2′-Bipyridyl)-di-μ-carbonyl-1,2-dicarbonyl-μ-1,3-di-*p*-tolyltriazenido-N^1N^3-2-(1,3-di-*p*-tolyltriazenido-N^1N^3)diruthenium (*Ru–Ru*).

Supplementary data available: see Instructions for Authors, *J. Chem. Soc. Dalton Trans.*, 1990, Issue 1, pp. xix—xxii.

Table 1. Atomic co-ordinates (× 10^4) for the complex [Ru_2(μ-RNNNR)(RNNNR)(μ-CO)$_2$(CO)$_2$(bipy)](R = *p*-tolyl)

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
Ru(1)	6 807(2)	123(1)	3 051(1)	Ru(11)	9 913(2)	1 917(1)	−397(1)
Ru(2)	5 765(2)	781(1)	3 539(1)	Ru(12)	10 153(2)	1 230(1)	−1 142(1)
O(1)	5 349(17)	749(6)	2 195(7)	O(101)	12 002(16)	1 396(5)	−83(8)
O(2)	7 372(17)	207(6)	4 356(8)	O(103)	10 944(19)	2 670(7)	−987(10)
O(3)	8 684(18)	689(7)	2 852(9)	O(102)	8 246(17)	1 853(6)	−1 540(8)
O(4)	7 499(18)	1 485(7)	3 621(7)	O(104)	11 496(16)	1 764(6)	−1 825(8)
N(1)	7 961(20)	−437(7)	3 148(10)	N(101)	8 782(23)	2 366(6)	−33(10)
N(2)	7 719(19)	−607(7)	2 621(10)	N(102)	9 434(26)	2 505(8)	419(11)
N(3)	6 903(18)	−348(6)	2 307(10)	N(103)	10 434(26)	2 344(8)	449(10)
N(4)	4 521(17)	274(8)	3 469(9)	N(104)	9 246(18)	1 393(7)	59(10)
N(5)	4 586(21)	−152(8)	3 343(10)	N(105)	8 873(17)	1 008(7)	−169(9)
N(6)	5 441(20)	−303(7)	3 182(9)	N(106)	9 119(16)	890(6)	−652(9)
N(7)	5 413(19)	982(7)	4 408(10)	N(107)	9 362(18)	876(7)	−1 961(8)
N(8)	4 486(17)	1 325(7)	3 269(11)	N(108)	11 091(17)	595(7)	−1 135(9)
C(1)	5 758(21)	618(8)	2 680(11)	C(101)	11 138(25)	1 483(9)	−406(12)
C(2)	6 887(23)	314(9)	3 870(13)	C(102)	9 028(23)	1 717(9)	−1 186(12)
C(3)	7 996(28)	478(10)	2 938(14)	C(103)	10 534(26)	2 373(11)	−757(13)
C(4)	6 823(25)	1 203(10)	3 587(12)	C(104)	10 993(25)	1 540(10)	−1 573(13)
C(5)	8 836(24)	−645(9)	3 529(12)	C(105)	7 592(27)	2 446(9)	−113(13)
C(6)	9 138(22)	−1 105(8)	3 433(11)	C(106)	7 252(30)	2 700(11)	342(14)
C(7)	10 054(25)	−1 300(9)	3 838(13)	C(107)	6 064(38)	2 730(13)	211(18)
C(8)	10 627(25)	−1 045(10)	4 344(13)	C(108)	5 398(33)	2 558(12)	−208(17)
C(9)	10 280(25)	−590(10)	4 433(12)	C(109)	5 771(28)	2 320(10)	−661(14)
C(10)	9 407(25)	−400(10)	4 025(13)	C(110)	6 963(26)	2 265(9)	−605(13)
C(11)	11 603(28)	−1 254(11)	4 798(14)	C(111)	4 094(41)	2 566(15)	−327(20)
C(12)	6 367(25)	−501(10)	1 723(13)	C(112)	11 336(28)	2 458(9)	845(13)
C(13)	6 534(22)	−946(9)	1 549(12)	C(113)	11 221(24)	2 750(9)	1 340(12)
C(14)	6 039(25)	−1 084(10)	957(13)	C(114)	12 210(29)	2 854(10)	1 742(13)
C(15)	5 407(24)	−767(10)	587(12)	C(115)	13 191(27)	2 690(10)	1 709(13)
C(16)	5 766(23)	−191(9)	1 360(12)	C(116)	13 339(27)	2 396(10)	1 228(14)
C(17)	5 204(24)	−335(10)	782(12)	C(117)	12 316(28)	2 289(10)	808(13)
C(18)	4 829(29)	−915(11)	−98(15)	C(118)	14 311(27)	2 806(10)	2 174(13)
C(19)	3 467(24)	382(9)	3 623(12)	C(119)	8 868(25)	1 473(9)	623(13)
C(20)	3 422(21)	306(8)	4 236(11)	C(120)	7 766(25)	1 472(9)	576(12)
C(21)	2 464(26)	471(10)	4 380(12)	C(121)	7 370(25)	1 578(10)	1 129(14)
C(22)	1 669(27)	708(11)	3 986(14)	C(122)	8 266(31)	1 649(11)	1 658(15)
C(23)	1 723(24)	750(10)	3 389(13)	C(123)	9 302(26)	1 627(9)	1 673(12)
C(24)	2 697(23)	580(8)	3 215(11)	C(124)	9 677(25)	1 538(9)	1 103(14)
C(25)	613(29)	922(12)	4 168(14)	C(125)	7 792(29)	1 734(12)	2 244(15)
C(26)	5 377(22)	−781(9)	3 012(11)	C(126)	8 677(22)	442(8)	−843(10)
C(27)	4 548(23)	−891(9)	2 560(12)	C(127)	9 265(21)	48(9)	−690(10)
C(28)	4 466(23)	−1 361(9)	2 375(12)	C(128)	8 822(23)	−381(9)	−858(11)
C(29)	5 313(26)	−1 652(10)	2 657(13)	C(129)	7 737(23)	−409(9)	−1 192(11)
C(30)	6 100(24)	−1 536(9)	3 112(13)	C(130)	7 141(25)	−22(10)	−1 340(12)
C(31)	6 214(22)	−1 073(9)	3 326(11)	C(131)	7 618(25)	422(9)	−1 177(12)
C(32)	5 256(29)	−2 181(12)	2 443(15)	C(132)	7 200(24)	−885(9)	−1 380(12)
C(33)	5 877(26)	785(11)	4 933(15)	C(133)	8 474(25)	1 043(9)	−2 378(14)
C(34)	5 547(25)	940(10)	5 477(13)	C(134)	8 122(23)	841(10)	−2 930(12)
C(35)	4 744(28)	1 272(11)	5 402(15)	C(135)	8 633(24)	434(10)	−3 037(12)
C(36)	4 320(26)	1 469(10)	4 883(15)	C(136)	9 489(22)	251(8)	−2 615(11)
C(37)	4 600(22)	1 318(9)	4 359(12)	C(137)	9 848(23)	470(9)	−2 069(12)
C(38)	4 135(24)	1 493(10)	3 785(14)	C(138)	10 692(22)	302(9)	−1 577(12)
C(39)	3 352(26)	1 833(10)	3 705(13)	C(139)	11 060(24)	−145(10)	−1 594(12)
C(40)	2 789(26)	1 951(10)	3 097(14)	C(140)	11 837(26)	−308(10)	−1 087(14)
C(41)	3 238(25)	1 783(10)	2 633(13)	C(141)	12 219(23)	12(10)	−616(12)
C(42)	4 086(26)	1 463(10)	2 734(14)	C(142)	11 840(24)	447(9)	−653(13)

tallographic Data Centre comprises H-atom co-ordinates, thermal parameters, and remaining bond lengths and angles.

Results and Discussion

Treatment of [Ru_2(μ-RNNNR)$_2$(CO)$_6$] (R = *p*-tolyl) with excess of 2,2′-bipyridyl in boiling benzene over a period of 2 h affords an orange, air-stable, crystalline product of stoicheiometry Ru_2(RNNNR)$_2$(CO)$_4$(bipy) in good yield. Spectroscopic data (Experimental section) provide evidence for the presence of terminal and bridging carbonyl ligands, and of triazenide ligands in two different environments which in turn suggest the adoption of an asymmetric binuclear structure. However, they are not sufficient to permit an unambiguous structural assignment. An *X*-ray diffraction study undertaken to resolve this problem revealed the asymmetric triazenide-bridged structure [Ru_2(μ-RNNNR)(RNNNR)(μ-CO)$_2$(CO)$_2$(bipy)], shown in perspective view with atomic numbering scheme in the Figure. The structure (**1a**) is closely related to that displayed by the cation of the ruthenium(I,I) carboxylate

Table 2. Selected bond lengths (Å) and angles (°)

	Molecule 1	Molecule 2		Molecule 1	Molecule 2
Ru(1)–Ru(2)	2.707(3)	2.706(3)	Ru(2)–C(2)	1.992(28)	1.993(28)
Ru(1)–N(1)	2.166(23)	2.226(23)	Ru(2)–C(4)	1.797(32)	1.836(31)
Ru(1)–N(3)	2.225(20)	2.290(22)	O(1)–C(1)	1.187(24)	1.199(27)
Ru(1)–N(6)	2.190(22)	2.133(19)	O(2)–C(2)	1.192(27)	1.197(26)
Ru(1)–C(1)	2.017(25)	1.996(28)	O(3)–C(3)	1.110(30)	1.192(30)
Ru(1)–C(2)	1.950(28)	1.999(27)	O(4)–C(4)	1.175(29)	1.150(28)
Ru(1)–C(3)	1.878(34)	1.834(33)	N(1)–N(2)	1.287(24)	1.242(26)
Ru(2)–N(4)	2.133(21)	2.139(20)	N(2)–N(3)	1.348(23)	1.323(29)
Ru(2)–N(7)	2.220(22)	2.192(19)	N(4)–N(5)	1.292(25)	1.264(21)
Ru(2)–N(8)	2.243(22)	2.201(21)	N(5)–N(6)	1.284(26)	1.291(22)
Ru(2)–C(1)	2.032(25)	2.007(28)			
N(1)–Ru(1)–Ru(2)	150.2(6)	147.9(7)	C(1)–Ru(2)–N(4)	85.0(8)	93.3(9)
N(3)–Ru(1)–Ru(2)	151.0(6)	153.8(8)	C(1)–Ru(2)–N(7)	168.4(10)	168.7(10)
N(3)–Ru(1)–N(1)	57.4(7)	56.0(9)	C(1)–Ru(2)–N(8)	94.2(9)	94.5(9)
N(6)–Ru(1)–Ru(2)	83.7(6)	83.3(6)	C(2)–Ru(2)–Ru(1)	46.0(8)	47.4(8)
N(6)–Ru(1)–N(1)	94.2(8)	83.9(8)	C(2)–Ru(2)–N(4)	89.4(10)	82.0(9)
N(6)–Ru(1)–N(3)	85.7(8)	92.6(8)	C(2)–Ru(2)–N(7)	95.6(10)	96.6(10)
C(1)–Ru(1)–Ru(2)	48.3(7)	47.6(8)	C(2)–Ru(2)–N(8)	172.1(10)	167.8(9)
C(1)–Ru(1)–N(1)	161.3(9)	159.0(10)	C(2)–Ru(2)–C(1)	93.7(11)	94.3(11)
C(1)–Ru(1)–N(3)	105.1(9)	106.4(11)	C(4)–Ru(2)–Ru(1)	95.9(9)	97.4(9)
C(1)–Ru(1)–N(6)	90.8(9)	85.8(9)	C(4)–Ru(2)–N(4)	179.0(11)	177.5(10)
C(2)–Ru(1)–Ru(2)	47.3(8)	47.2(8)	C(4)–Ru(2)–N(7)	92.8(10)	88.5(10)
C(2)–Ru(1)–N(1)	103.0(10)	104.2(11)	C(4)–Ru(2)–N(8)	90.0(10)	93.3(10)
C(2)–Ru(1)–N(3)	157.4(10)	158.9(11)	C(4)–Ru(2)–C(1)	94.1(11)	87.4(12)
C(2)–Ru(1)–N(6)	84.7(9)	92.2(9)	C(4)–Ru(2)–C(2)	90.2(11)	95.5(11)
C(2)–Ru(1)–C(1)	95.4(11)	94.4(11)	N(2)–N(1)–Ru(1)	100.4(16)	99.0(20)
C(3)–Ru(1)–Ru(2)	97.3(9)	98.2(9)	N(3)–N(2)–N(1)	106.4(21)	111.5(28)
C(3)–Ru(1)–N(1)	84.7(11)	95.9(11)	N(2)–N(3)–Ru(1)	95.7(14)	93.4(17)
C(3)–Ru(1)–N(3)	93.6(11)	85.1(11)	N(5)–N(4)–Ru(2)	127.8(19)	125.2(17)
C(3)–Ru(1)–N(6)	178.9(11)	177.3(11)	N(6)–N(5)–N(4)	119.7(25)	117.6(21)
C(3)–Ru(1)–C(1)	90.2(12)	93.5(12)	N(5)–N(6)–Ru(1)	124.5(17)	128.8(15)
C(3)–Ru(1)–C(2)	95.7(12)	90.5(12)	Ru(2)–C(1)–Ru(1)	83.9(10)	85.1(12)
N(4)–Ru(2)–Ru(1)	83.1(6)	81.4(5)	Ru(2)–C(2)–Ru(1)	86.7(12)	85.4(11)
N(7)–Ru(2)–Ru(1)	140.5(6)	143.9(6)	O(1)–C(1)–Ru(1)	137.6(20)	135.9(21)
N(7)–Ru(2)–N(4)	88.2(7)	91.2(7)	O(1)–C(1)–Ru(2)	138.5(20)	138.6(21)
N(8)–Ru(2)–Ru(1)	141.8(6)	139.4(5)	O(2)–C(2)–Ru(1)	137.7(22)	136.8(21)
N(8)–Ru(2)–N(4)	90.6(8)	89.0(7)	O(2)–C(2)–Ru(2)	135.5(22)	137.5(21)
N(8)–Ru(2)–N(7)	76.4(8)	75.2(8)	O(3)–C(3)–Ru(1)	177.8(30)	179.5(25)
C(1)–Ru(2)–Ru(1)	47.8(7)	47.3(8)	O(4)–C(4)–Ru(2)	178.6(26)	174.8(25)

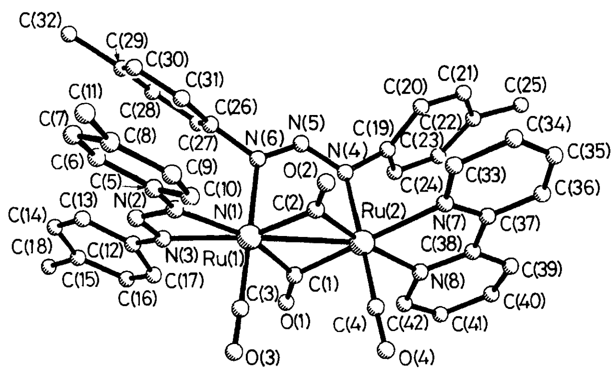
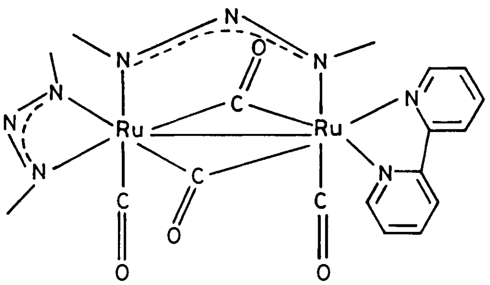


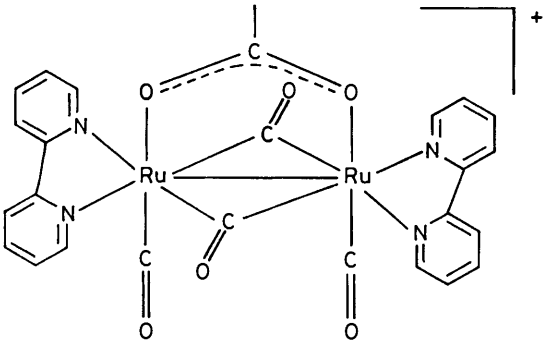
Figure. Molecular structure of one of the crystallographically independent [$Ru_2(\mu$-RNNNR)(RNNNR)(μ-CO)$_2$(CO)$_2$(bipy)] (R = *p*-tolyl) molecules (the other is essentially identical) showing the inner co-ordination sphere

substitution product [$Ru_2(\mu$-$O_2CMe)(\mu$-CO)$_2$(CO)$_2$(bipy)$_2$][BPh_4] (**1b**) reported by Steyn and Singleton[5] while the present work was in progress. The two structures have the same overall geometry with an edge-sharing bioctahedral configuration for the core atoms and very similar Ru–Ru distances, 2.707/2.706 and 2.709 Å respectively, typical of Ru^I–Ru^I single bonds.

The poor diffracting quality of the crystal used in the present work adversely affected the quality of the structural data obtained; this is reflected in the high final *R* values and e.s.d.s reported. However comparison of our data for compound (**1a**) with those reported by Steyn and Singleton[5] for the cation (**1b**) reveals consistent bond lengths and angles for those groups which are common to both structures. Bond angles subtended at the ruthenium centres Ru(1) and Ru(2) in the triazenide structure (**1a**) reflect distortion of the underlying octahedral co-ordination geometry imposed by the small bites of the chelating triazenide and bipyridyl ligands. Distortion is most marked for the Ru(1) centre where the dominant feature is the very small angle (57.4/56.0°) subtended by the chelate triazenide ligand. This angle, though considerably less than those found in simple tris(triazenide) chelates (*ca.* 62–66°), is similar to those encountered for isolated triazenide chelate ligands in a range of mono- and bi-nuclear complexes (*ca.* 56–58°).[7] The co-ordination geometry about the Ru(2) centre likewise reflects the rather less severe distortion imposed by the chelate 2,2′-bipyridyl ligand [subtended angle 76.4(8)/75.2(8)° and is very similar to those found for the ruthenium centres in the cationic complex (**1b**). The angles N–N–N observed for the bridging and chelating triazenide ligands (117.6/119.7°) and (111.5/106.4°)



(1a)



(1b)

respectively reflect the additional angular strain imposed on the triazenide ligand by the steric requirements of the chelate co-ordination mode.

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