^{13}C NMR of Tris-(bipyrimidine)ruthenium(II) Dichloride

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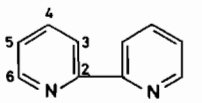
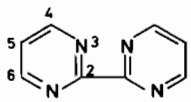
Hunziker and Ludi [1] prepared $Ru(bpym)_3Cl_2$ (bpy = 2,2′-bipyridine). The ^{13}C and proton NMR of $Ru(bpy)_3Cl_2$ in DMSO indicate retention of D_3 symmetry in solution [2]. We prepared $Ru(bpym)_3Cl_2$ by melting $RuCl_3$ with bipyrimidine (1:8 ratio by weight) and maintaining the melt at 170 °C for four h in an oil bath. Chloroform was used to remove excess bipyrimidine and the red brown complex crystallised twice from a small amount of methanol (yield 50%). The ^{13}C-NMR for $Ru(bpy)_3^{2+}$ and $Ru(bpym)_3^{2+}$ in D_2O was taken with dioxane as the reference*. The fully proton-decoupled spectrum gives five and four lines respectively. Off-resonance proton-decoupling of the two spectra gives a ^{13}C–H splitting (doublet) for all the lines except the highest field line, thus unambiguously assigning it to the connecting carbons (2,2′) on both rings. Comparison with ^{13}C data for bipyridine [3] and bipyrimidine* leads to the assignment of lines shown in Table I below. D_3 symmetry is also retained in solution for $Ru(bpym)_3^{2+}$ in water.



Bipyridine



Bipyrimidine

References

1 M. Hunziker and A. Ludi, *J. Am. Chem. Soc., 99,* 7370 (1977).
2 R. J. Watts, *J. Chem. Educ., 60,* 834 (1983).
3 E. Breitmaier, G. Haas and W. Voelter, 'Atlas of Carbon-13 NMR Data, Vol. 2', Heyden, London, 1979.

* ^1H Coupled and ^1H dec-spectra of these compounds where measured on a Bruker WP 80 FT NMR instrument.

TABLE I. ^{13}C NMR Results (ppm).

	C_2	C_3	C_4	C_5	C_6
Bipyridine	155.25(singlet)	124.20	137.25	120.40	149.20
Bipyrimidine	161.25(singlet)	–	158.87	123.30	158.87
$Ru(bpy)_3^{2+}$	157.8(singlet)	128.3	138.7	125.0	152.1
$Ru(bpym)_3^{2+}$	163.3(singlet)	–	159.9	125.7	161.1

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