^{13}C NMR SPECTROSCOPY OF SUBSTITUTED IMIDAZOLES

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Abstract – A simple equation can be used for the evaluation of ^{13}C chemical shifts of substituted imidazoles.

Carbon - 13 spectral correlations of substituted imidazoles have not been too abundant in recent literature[1] and no general equations for the evaluation of spectral parameters have been reported. In the course of another already deceased synthetic project, a ^{13}C NMR spectroscopic analysis of the title compounds was required and a simplistic model for calculating the chemical shifts was developed.

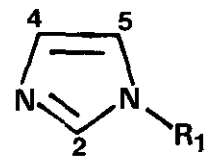
The procedure is based on the assumption that the chemical shifts can be calculated using the simple mathematical expression[2]:

$$\delta_C(k) = C_k + \sum_i A_{ik}(R_i)$$

The observed and calculated values for the resonances of nearly 30 compounds were found to be in satisfactory agreement (ca. ± 2 ppm). In the case of multiple substitution, uncertainty increases, as expected, but the calculated values can still be used to distinguish the imidazole ring carbons if it is not possible on multiplicity grounds.

Recently, a more elaborate statistical method for similar purposes has been reported[3].

$$\delta_C(k) = C_k + \sum_i A_{ik}(R_i)$$



C_2 = 134.8 ppm

$C_4 = C_5$ = 121.5 ppm

Substituent increments A_{ik}

	i = 1			i = 2	
R_i	A_{12}	A_{14}	A_{15}	A_{22}	$A_{24} = A_{25}$
Me	1.8	6.7	-2.2	9.4	-0.5
Et	0.4	5.4	-2.1	12.5	-1.4
Pr	0.9	5.3	-1.8		
n-Hex	1.1	6.0	-1.5		

	i = 4 = 5		
R_4	A_{42}	A_{44}	A_{45}
Me	-0.2	10.3	-2.8
Pr	5.6	15.0	0.6
CH_2OR	-0.8	10.9	-3.5
CH_2Cl	-6.8	9.7	-11.8
CH_2CN	-0.9	7.0	-12.5
CH_2CH_2OR	0.2	12.5	-4.4
COOR	-5.2	21.5	-8.2
Ar	0.7	16.9	-6.2
CH_2Ph	0.3	15.3	-3.6

In connection with this work, the following ^{13}C NMR spectra were recorded (JEOL JNM-FX 60 spectrometer, 15.04 MHz, DMSO-d_6, TMS as internal standard):

4(5)-Phenylimidazole: 111.5, 124.9 (2), 126.9, 128.7 (2), 132.8, 135.6, 138.6

4(5)-*p*-Biphenylimidazole: 112.3, 123.2 (2), 124.7 (2), 125.1 (2), 125.4 (2), 125.4, 127.1 (2), 134.1, 136.5, 138.6

4,5-Diphenylimidazole: 126.7 (2), 127.5 (4), 128.1 (4), 131.4 (2), 133.3, 135.1 (2)

4(5)-Phenyl-5(4)-methylimidazole: 12.1, 125.5, 125.9 (2), 128.2 (2), 132.4, 133.6, 134.5

4(5)-*p*-Biphenyl-5(4)-Hydroxymethylimidazole: 54.8, 126.4 (4), 126.5 (2), 126.8, 127.1, 128.8 (2), 133.4, 134.3 (2), 137.7, 139.9

4(5)-Propyl-5(4)-carboxyethylimidazole: 13.6, 14.3, 22.3, 42.3, 59.3, 128.1, 135.4, 137.1, 165.8

REFERENCES AND NOTES

1. ^{13}C NMR chemical shifts of substituted imidazoles have been reported in the following papers:

a) H.-J. Sattler, V. Stoeck and W. Schunack, *Arch. Pharm. (Weinheim)*, 1975, 308, 795.

b) M. Utaka, J. Koyama and A. Takeda, *J. Am. Chem. Soc.*, 1978, 98, 984.

c) K. Wegner and W. Schunack, *Arch. Pharm. (Weinheim)*, 1977, 310, 380.

d) V. Stoeck and W. Schunack, *Arch. Pharm. (Weinheim)*, 1977, 310, 677.

e) H.-J. Sattler, V. Stoeck and W. Schunack, *Arch. Pharm. (Weinheim)*, 1978, 311, 736.

2. cf. eg. F.W. Wehrli and T. Wirthlin, *Interpretation of Carbon-13 NMR Spectra*, Heyden and Son Ltd., London, 1978.

3. B.L. Lam, G.R. Wellman, C.S. Labaw and E.S. Pepper, *J. Org. Chem.*, 1982, 47, 144.

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