



Abstracts

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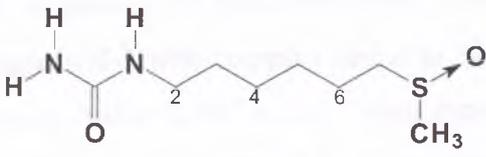
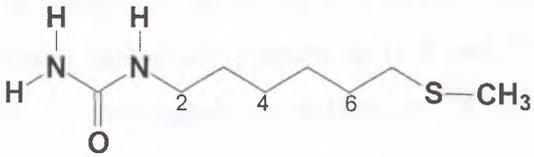
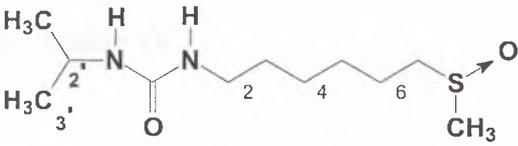
CORRELATION OF DATA NMR ^{13}C AND ^1H SPECTROSCOPY WITH INTERNAL SULPHUR CONTAINING ALKALOIDS OF *DIPPTHYCHOCARPUS* *STRICTUS*

I. I. Okhunov, S. F. Aripova, M. G. Levkovich, N. D. Abdullaev, V. U. Khujaev

*Institute of the Chemistry of Plant Substances named after Academician S. Y. Yunusov,
Academy of Sciences, 1000170, Tashkent, Republic of Uzbekistan,
Fax (99871) 120 64 75, E-mail: salima_aripova@mail.ru*

A number of sulphur containing alkaloids, derivatives of *N*-alkylurea were isolated earlier from the wild growing plant *Dipthychocarpus strictus* of Cruciferae family. There is no data on NMR ^{13}C spectra parameters of sulfur containing alkaloids in the state of the art. We studied spectra of NMR ^{13}C sulfur- containing alkaloids for the first time.

Table 1 Alkaloids of *Dipthychocarpus* structures

A Dipthychocarpaine (3)	
B Desooxydipthochocarpaine (4)	
C Dipthochocarpamine (5)	

<p style="text-align: center;">D Dipthochocarpidine (1)</p>	
<p style="text-align: center;">E Dipthochocarpiline (2)</p>	
<p style="text-align: center;">F Dipthochocarpilidine (6)</p>	

NMR spectra of compounds A-F were registered in CD_3OD with an internal etalons HMDSO (0 m.d.), on spectrometer UNITY 400+ (Varian) with working frequency 400 MHz for protons and 100 MHz for carbon nuclear. Signals of protons were separated on two characteristic groups. Methyls and methylenes groups, neighboring with heteroatom resonated at 2.5-3.1 m.d., but methylenes signals were on H-3 to H-6 - at 1.3-1.8 m.d. In compounds A, B and C the signals from NH-protons at 4.6 m.d. were detected.

In NMR spectrum of compounds A protons of gem-heteroatom formed two clear signals: H-2 (triplet at 3.31 m.d.), two signals H-7 with complex shape as "ddd" (2.775 and 2.708 m.d.) and singlet of methyl group on sulfur (2.567 m.d.). Using methods of double resonance their neighbors - methylenes groups H-3 and H-6 were identified. Double resonance, due to signals of protons of methylene group on H-6 (1.697 m.d), and COSY spectra identified the signals of remaining methylene protons at H-5 and H-4. Using the methods of direct heterocorrelation ^1H - ^{13}C the signals of carbons in ^{13}C NMR identified (Table 1).

It was succeeded to measure KCCB for compound A only for heteroatomic methylenes on H-2 and H-7 (reference in the bottom of table 1).

Table 2 NMR specters of A-F compounds

Compd.	Atoms								
	NH, CO	2	3	4	5	6	7a	7b	CH ₃
A (¹ H)	4.580	3.031	1.449	1.346	1.440	1.697	2.708	2.775	2.567
A (¹³ C)	162.40	4.096	29.51	27.56	31.03	23.71	54.88		38.24
B	4.587	3.015	1.250 – 1.500			1.540	2.420		1.990
C (H)	4.586	3.038	1.376– 1.499	1.337	1.376– 1.499	1.696	2.706	2.773	2.566
C (H')		3.714	1.040						
D		3.044	1.451	1.340	1.429	1.697	2.707	2.774	2.567
E (H)		3.049	1.235 – 1.472			1.697	2.708	2.774	2.568
E (H')		3.032				1.530	2.417		1.993
F		2.396	1.606	1.425 – 1.515		1.713	2.713	2.783	2.570

KCCB: A (H-2, (t, 7.0), H-7a (ddd, 13.0, 8.4, 6.0), H-7b (ddd, 13.0, 8.6, 7.4)), C (H-3', (6×d, 6.5), H-4, (d, 6.5)).

In spectra of compounds B, in connection with inconstant character of sulfur atom, first of all, changing for signals of methyl group (1.990 m.d.) and methylene on H-7 (2.420 m.d.) is observed. All other remaining signals were close to those of molecule A. In compound C right part completely repeats the spectrum of compound A. In spectrum of compounds B right side of molecule completely repeats the spectrum of compound A. Left side of molecule is represented by two peculiar signals of *N*-isopropyl group in 3.714 m.d. (signal methyne proton with 6 KCCB) and in 1.040 m.d (doublet double methyl group). The structure of compound D looks like as dimer of compound A and correspondingly, practically repeats NMR spectrum of compound A. Molecule E can be considered as dimer of molecules A and B, which also was confirmed by NMR specter. In structure of F more considerable changes were observed - in the left side of the molecule triple bond considerably affected on methylen pair H-2 shift to strong field on 2.396 m.d. Other signals remained close to those of compound A.