^{13}C-NMR SPECTRA OF SUBSTITUTED 1,4-DIHYDROPYRIDINES

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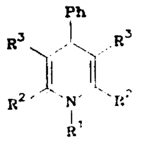
UDC 543.422.25:547.827

It has been found that the electron shift in the $C_{(2)}=C_{(3)}$ bond, under the influence of the substituent attached to the nitrogen atom and the substituents in the 3- and 5-positions, occurs via a π-inductive mechanism. 4-(1,4-Dihydropyridyl) functional groups behave as electron donating substituents via an inductive mechanism.

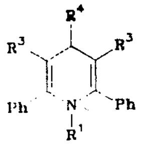
Despite the broad spectrum of practical applications of substituted 1,4-dihydropyridines, their ^{13}C-NMR spectra have been studied very little. Only a few studies are known [1-3], in which the ^{13}C chemical shifts (CS) of several 1,4-dihydropyridines were reported. In another study [4], the ^{13}C CS values of a series of 4-aryl-2,6-dimethyl-3,5-diethoxycarbonyl-1,2-dihydropyridines were analyzed, and a linear correlation was established between δ $C_{(4)}$ and the σ^+-constant for the substituent in the 4-aryl ring:

$$\delta\ C_{(4)}=0.779\sigma^{+}+39.51 \quad (r=0.96). \qquad (1)$$

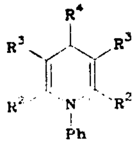
In the present paper we have studied substituted 4-phenyl-1,4-dihydropyridines (I), 2,6-diphenyl-1,4-dihydropyridines (II), and 1-phenyl-1,4-dihydropyridiens (III) by ^{13}C-NMR spectroscopy.



I



II



III

The measured ^{13}C-NMR spectral parameters for these compounds are given in Tables 1 and 2. Signal assignments were made based on literature data [5, 6], as well as using spectra obtained without proton (spin-spin) decoupling.

In the case of N-substituted 4-phenyl-3,5-dicarboethoxy-1,4-dihydropyridines (compounds XVI, XIV-XXX) with strongly electron-withdrawing (acceptor) substituents attached to the nitrogen atom, the signals corresponding to the $C_{(2,6)}$ atoms in the dihydropyridine ring are shifted upfield, while the $C_{(3,5)}$ signals are shifted downfield, reflecting substantial polarization of the $C_{(2)}=C_{(3)}$double bond under the influence of this substituent. It was not possible, however, to obtain a satisfactory correlation of the ^{13}C CS values with substituent constants for the groups attached to the nitrogen atom. We assume that introduction of a substituent to the nitrogen atom significantly changes the role of the nitrogen unshared electron pair in the overall conjugation system of the dihydropyridine ring. This assumption is contradicted, however, by the fact that the sum of the chemical shifts of the $C_{(2)}$ and $C_{(3)}$ carbon atoms is practically constant, i.e., the electron shift in the $C_{(2)}=C_{(3)}$ bond occurs primarily via a π-inductive mechanism. This conclusion is supported by the observation that the effect of the N-substituent on the ^{13}C CS values remains fundamentally unchanged when CH_3 groups are added to the 2,6-positions, thus interfering with conjugation. It should be noted that the greatest substituent effects on the $C_{(2,6)}$ CS values as observed in compounds XXVIII and XXX, in which the upfield shift of the $C_{(2,6)}$ resonance signals can be attributed at least partially to the anisotropic effect of the C=O and S=O functional groups. In the 4-phenyl

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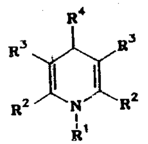
Institute of Organic Synthesis, Academy of Sciences of the Latvian SSR, Riga. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 9, pp. 1232-1238, September, 1989. Original article submitted April 15, 1988.

0009-3122/89/2509-1032$12.50

TABLE 1. ^{13}C-NMR Spectral Parameters of Substituted 1,4-Dihydropyridines

Compound	^{13}C chemical shifts of the nonaromatic carbons, δ, ppm							^{13}C-^{1}H coupling constant, J, Hz
	$C_{(2)}$	$C_{(3)}$	$C_{(4)}$	2-CH_3	C=O	CH_2CH_3	other carbon atoms	
I	146,70	102,82	39,92	19,19	168,59	—	51,59 ($COOCH_3$)	136,9
II	146,44	163,05	40,05	19,19	168,21	60,08; 15,12	—	136,7
III	146,11	103,20	40,10	19,25	167,75	—	67,08 (CH); 23,02 (CH_3); 22.63 (CH_3)	137,0
IV	145,80	103,50	40,22	19,19	167,68	—	28,99 (CH_3); 79,51 ($C(CH_3)_3$)	136,4
V	146,40	103,15	40,10	19,12	168,14	—	62,41 (OCH_2); 38,23 (OCH_2CH_2); 25,56 (CH); 23,22 (CH_3)	—
VI	146,89	102,34	39,76	19,22	168,21	66,69; 16,10	63,70 (OCH_2); 69,03 (OCH_2CH_2)	—
VII	147,49	101,40	39,30	19,45	167,49	—	119,64 (CN); 59,58 (OCH_2); 18,55 (OCH_2CH_2CN)	—
VIII	146,60	103,20	40,00	19,26	168,14	—	73,67 (OCH_2); 32,05 ($C(CH_3)_3$); 27,44 (CH_3)	—
IX	147,68	101,80	39,35	19,38	169,30	—	51,94 (OCH_2); 78,00 (CH); 79,97 ($C{\equiv}CH$)	—
X	146,91	102,40	39,75	19,26	167,68	—	64,71 (OCH_2); 134,37 ($CH{=}CH_2$); 117,95 ($={CH_2}$)	—
XI	147,82	83,93	42,31	18,81	—	—	120,22 (CN)	138,7
XII	145,41	113,73	39,84	20,04	197,55		31,08 ($COCH_3$)	134,5
XIII	148,48	102,60	39,79	—	167,95	60,13; 14,51	—	135,6
XIV	148,32	101,85	39,85	—	168,21	60,13; 14,77	—	—
XV	146,00	104,44	40,62	—	166,97	60,49; 14,52	—	—
XVI	136,07	107,25	38,13	—	167,63	60,29; 15,13	—	137,3
XVII	136,23	106,96	38,03	—	168,05	—	—	—
XVIII	147,77	106,21	39,63	19,19	168,47	60,62; 15,16	—	137,3
XIX	150,61	105,87	38,93	17,11	168,27	60,42; 15,23	34,91 (NCH_2)	—
XX	148,65	106,08	39,53	19,12	168,47	60,59; 15,16	—	—
XXI	148,97	105,07	40,08	19,35	168,08	60,91; 15,16	—	—
XXII	148,01	103,60	30,17	—	167,68	59,84; 14,58	22,70 (CH_3)	—
XXIII	149,26	98,60	26,79	—	167,88	59,90; 14,57	—	—
XXIV	136,69	111,08	38,27	—	167,10	60,91; 15,09	—	137,6
XXV	139,09	108,22	37,90	—	167,36	60,46; 15,16	49,73 (NCH_2); 16,52 (CH_3)	—
XXVI	136,91	108,34	38,41	—	167,23	60,42; 15,17	55,75 (CH); 22,57 (CH_3)	—
XXVII	139,37	108,47	37,83	—	167,16	60,55; 15,24	57,70 (NCH_2)	—
XXVIII	131,00	114,12	38,48	—	165,99	61,53; 15,04	41,72 (CH_3)	—
XXIX	139,97	108,48	37,81	—	167,30	60,49; 15,16	170,74 (CO); 62,18 (CH_2); 55,12 (NCH_2)	—
XXX	132,11	115,31	39,53	—	166,29	—	168,86 (CO)	—
XXXI	146,09	130,12	42,41	20,11	—	—	—	—

TABLE 2. ^{13}C Chemical Shifts of the Aromatic Carbon Atoms in Substituted 1,4-Dihydropyridines



Compound	Substituent				Position of the phenyl group	Chemical shifts of the aromatic carbon atoms, δ, ppm				Δ= $=C_p-C_m$
	R^1	R^2	R^3	R^4		C_o	C_m	C_p	$C_{(i)}$	
I	H	CH_3	$COOCH_3$	C_6H_5	4	128,14	129,05	126,97	149,05	−2,08
II	H	CH_3	$COOCH_2CH_3$	C_6H_5	4	128,57	128,92	126,94	149,43	−1,98
III	H	CH_3	$COOCH(CH_3)_2$	C_6H_5	4	128,82	128,82	126,94	149,62	−1,88
IV	H	CH_3	$COOC(CH_3)_3$	C_6H_5	4	128,53	128,53	126,71	149,57	−1,82
V	H	CH_3	$COOCH_2CH_2CH(CH_3)_2$	C_6H_5	4	128,37	128,82	126,94	149,36	−1,88
VI	H	CH_3	$COOCH_2CH_2OCH_2CH_3$	C_6H_5	4	128,53	129,02	127,01	149,23	−2,01
VII	H	CH_3	$COOCH_2CH_2CN$	C_6H_5	4	128,40	129,12	127,10	148,85	−2,02
VIII	H	CH_3	$COOCH_2C(CH_3)_3$	C_6H_5	4	128,01	128,96	126,95	148,92	−2,01
IX	H	CH_3	$COOCH_2—C{\equiv}CH$	C_6H_5	4	128,21	129,05	127,10	148,59	−1,95
X	H	CH_3	$COOCH_2CH{=}CH_2$	C_6H_5	4	128,34	128,99	126,97	148,98	−2,02
XI	H	CH_3	CN	C_6H_5	4	128,73	129,90	128,73	145,09	−1,17
XII	H	CH_3	$COCH_3$	C_6H_5	4	128,21	129,31	127,10	148,27	−2,21
XIII	H	C_6H_5	$COOCH_2CH_3$	C_6H_5	4	128,11	129,34	127,33	148,48	−2,01
XIV	H	$4\text{-}OCH_3—C_6H_4$	$COOCH_2CH_3$	C_6H_5	4	128,05	129,28	127,20	148,32	−2,08
					2,6	131,94	114,33	161,45	129,28	
XV	H	$4\text{-}NO_2—C_6H_4$	$COOCH_2CH_3$	C_6H_5	4	128,34	129,51	127,69	147,94	−1,82
					2,6	131,65	123,92	148,85	143,85	—
XVI	H	H	$COOCH_2CH_3$	C_6H_5	4	128,86	128,99	127,23	148,55	−1,76
XVII	H	H	$COOCH_3$	C_6H_5	4	128,76	129,12	127,33	148,39	−1,79
XVIII	C_6H_5	CH_3	$COOCH_2CH_3$	C_6H_5	4	128,27	129,34	127,36	148,00	−1,98
					1	131,39	130,77	130,03	141,04	
XIX	CH_3	CH_3	$COOCH_2CH_3$	C_6H_5	4	127,75	129,12	127,04	147,23	−2,08
XX	$4\text{-}OCH_3—C_6H_4$	CH_3	$COOCH_2CH_3$	C_6H_5	4	128,24	129,34	127,33	148,35	−2,01
					1	132,33	115,79	160,31	133,50	—
XXI	C_6H_5	CH_3	$COOCH_2CH_3$	$4\text{-}NO_2—C_6H_4$	4	129,64	124,83	147,41	155,50	—
					1	131,49	130,84	130,22	140,75	—
XXII	H	C_6H_5	$COOCH_2CH_3$	CH_3	2,6	130,02	128,60	129,64	137,36	—
XXIII	H	C_6H_5	$COOCH_2CH_3$	H	2,6	129,80	128,66	129,54	137,50	—
XXIV	C_6H_5	H	$COOCH_2CH_3$	C_6H_5	1	122,03	131,20	127,72	143,87	—
					4	129,12	129,20	127,59	147,09	−1,53
XXV	C_2H_5	H	$COOCH_2CH_3$	C_6H_5	4	128,83	129,09	127,40	148,13	−1,69
XXVI	$CH(CH_3)_2$	H	$COOCH_2CH_3$	C_6H_5	4	128,73	128,99	127,30	148,01	−1,69
XXVII	$CH_2C_6H_5$	H	$COOCH_2CH_3$	C_6H_5	4	128,73	128,99	127,36	147,75	−1,63
					1	128,73	128,99	129,96	138,60	—
XXVIII	SO_2CH_3	H	$COOCH_2CH_3$	C_6H_5	4	129,05	129,38	128,08	145,09	−1,30
XXIX	$CH_2COOCH_2CH_3$	H	$COOCH_2CH_3$	C_6H_5	4	128,96	129,18	127,43	148,00	−1,75
XXX	COC_6H_5	H	$COOCH_2CH_3$	C_6H_5	4	129,38	129,61	128,08	144,94	−1,53
					1	130,03	133,57	130,03	132,95	—
XXXI	H	CH_3	NO_2	C_6H_5	4	129,01	129,30	128,34	143,22	−0,96

substituents in these compounds the π-electron density is shifted considerably in the direction of the $C_{(i)}$ carbon atom, due to the influence of the electronic effect of the N-substituent. This is indicated by the upfield shift of the corresponding resonance signal in the series H → Alk → C_6H_5 → SO_2Me → COPh.

In the case of the 2,6-disubstituted 4-phenyl-3,5-dicarboethoxy-1,4-dihydropyridine derivatives examined (compounds II, XIII, XVI), increasing the electron withdrawing (acceptor) properties of the substituents in the 2,6-positions of the dihydropyridine ring does not lead to a clear dependence of ^{13}C CS values on the electronic properties of the corresponding substituents. Apparently steric factors play a role here. In addition, we also note that the $C_{(p)}$ atom is deshielded and the $C_{(i)}$ ring atom shielded in the 4-phenyl radical, in the series of 2,6-substituents: CH_3—H—C_6H_5; this is associated with an increase in the π-inductive effect of the complex dihydropyridine substituent.

In the case of the 4-phenyl-2,6-dimethyl-1,4-dihydropyridine derivatives studied herein (compounds I-XII), we have found a correlation of CS values with the electronic properties of the substituents located in the 3,5-positions of the dihydropyridine ring. As the electron withdrawing characteristics of the R group in the COOR substituent are increased the signals corresponding to the $C_{(3,5)}$ atoms, as well as the $C_{(4)}$ carbon atom in the dihydropyridine ring, are shifted upfield. The opposite effect was observed for the carbon atoms located in the 2,6-positions of the dihydropyridine ring. Using correlation analysis we derived the following relationships between the ^{13}C CS values and the Taft σ*-constants for the substituents R in the 3,5-COOR groups:

$$\delta\ C_{(2,6)} = 1.38\sigma^* + 146.53 \quad (r = 0.950;\ s = 0.49;\ n = 10). \tag{2}$$

$$\delta\ C_{(3,5)} = -1.72\sigma^* + 102.90 \quad (r = 0.989;\ s = 0.54;\ n = 10). \tag{3}$$

$$\delta\ C_{(4)} = -0.79\sigma^* + 39.94 \quad (r = 0.994;\ s = 0.37;\ n = 10). \tag{4}$$

In the case of the $C_{(4)}$ atom this effect is probably related to the fact that the atom is sp^3-hybridized and thus not involved in the overall π-electron system, which is composed of the $C_{(3)}$, $C_{(2)}$, N, $C_{(6)}$, and $C_{(5)}$ ring atoms, as well as the C=O fragment in the ester functional groups in the 3,5-positions of the dihydropyridine ring. The opposing shifts of the resonance signals for the $C_{(2,6)}$ versus $C_{(3,5)}$ atoms can be explained in terms of π-inductive electron shift under the influence of the 3,5-substituents, in the presence of stable (unchanged) conjugative interaction of the COOR functional groups and the $C_{(3)}=C_{(2)}$ double bond. This interpretation is supported by the observation that the sum of the CS values for the $C_{(3)}$ and $C_{(2)}$ atoms in these compounds is constant. We did not detect a clear relationship between the ^{13}C CS values of the carbonyl group carbon atoms and the inductive Taft substituent constants for the R groups in COOR. The CS values of these nuclei are apparently affected by steric factors, as well as by effects arising from rotation of the carbonyl groups relative to the plane of the dihydropyridine ring.

The ^{13}C CS values within the phenyl ring can be used to analyze the effects of substituents in different positions of the dihydropyridine ring on the phenyl ring itself. We have obtained, for instance, a good correlation of $C_{(p)}$ CS values with the Taft σ*-constants for the substituents R in the 3,5-COOR functional groups:

$$C_{(p)} = 0.17\sigma^* + 126.96 \quad (r = 0.993;\ s = 0.17;\ n = 10). \tag{5}$$

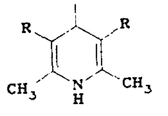
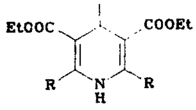
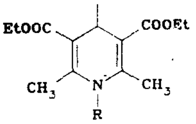
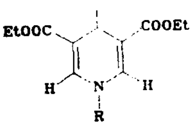
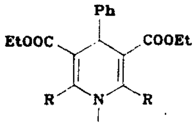
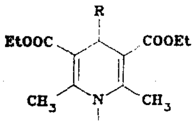
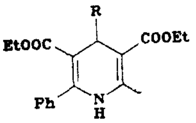
No such correlation was observed for the $C_{(i)}$ atoms. This can be ascribed to the effect of anisotropy of the complex 1,4-dihydropyridine residue on the shielding of the proximate $C_{(i)}$ carbon atom.

Resonance effects between the 4-phenyl ring and the 1,4-dihydropyridin-4-yl residue were estimated using the parameter $\Delta = \delta C_{(p)} - \delta C_{(m)}$, which, according to [7], reflects the degree of resonance interaction between the two ring systems. The fact that this parameter is constant indicates that there is little change in the π-electron density (distribution) as the alkyl substituent in the COOR group is varied.

These results contradict somewhat results obtained earlier [4], since the correlation expressed in Eq. (1) would seem to assume the presence of direct polar conjugation between the 4-phenyl ring and the 4-(1,4-dihydropyridine) substituent.

Using ^{13}C-NMR spectroscopy and known correlation equations [8] for the CS values of the m- and p-carbon atoms in the substituted benzene rings, we were able to determine σ_1 and $\sigma_R{}^0$-

TABLE 3. Calculated Values of σ_I and $\sigma_R{}^0$ for 1,4-Dihydropyridone Substituents

Compound	Substituent	R	σ_I	$\sigma_R{}^0$
I		$COOCH_3$	−0,14	−0,08
		$COOC_2H_5$	−0,18	−0,08
		$COOCH(CH_3)_2$	−0,21	−0,06
		$COOC(CH_3)_3$	−0,30	−0,06
		$COOCH_2CH_2CH(CH_3)_2$	−0,21	−0,06
		$COOCH_2CH_2OC_2H_5$	−0,15	−0,08
		$COOCH_2CH_2CN$	−0,11	−0,08
		$COOCH_2C(CH_3)_3$	−0,16	−0,08
		$COOCH_2CH{=}CH_2$	−0,16	−0,08
		$COOCH_2C{\equiv}CH$	−0,14	−0,08
		CN	0,14	−0,06
		$COCH_3$	−0,06	−0,09
II		CH_3	−0,18	−0,08
		H	−0,15	−0,06
		C_6H_5	−0,04	−0,08
		$4\text{-}OCH_3{-}C_6H_4$	−0,06	−0,09
		$4\text{-}NO_2{-}C_6H_4$	0,01	−0,06
III		H	−0,18	−0,08
		CH_3	−0,11	−0,08
		C_6H_5	−0,04	−0,08
		$4\text{-}OCH_3{-}C_6H_4$	−0,04	−0,08
IV		H	−0,15	−0,06
		COC_6H_5	0,04	−0,07
		C_6H_5	−0,06	−0,06
		C_2H_5	−0,12	−0,06
		$CH(CH_3)_2$	−0,15	−0,06
		$CH_2C_6H_5$	−0,15	−0,06
		SO_2CH_3	−0,02	−0,05
		$CH_2COOCH_2CH_3$	−0,11	−0,07
V		H	0,53	−0,18
		CH_3	0,42	−0,05
VI		C_6H_5	0,42	−0,05
		$4\text{-}NO_2{-}C_6H_4$	0,45	−0,05
VII		H	−0,23	0,05
		CH_3	−0,25	0,06
		C_6H_5	−0,18	0,06

constants for the dihydropyridyl functional groups, which are regarded in this case simply as complex substituents attached to the benzene ring. The values of the derived reactivity constants are summarized in Table 3. Based on an inductive mechanism 4-(1,4-dihydropyridyl) functional groups behave as electron donating substituents. The resonance term has a constant value.

The direct $J_{^{13}C(4)-^{1}H}$ spin-spin coupling constants were measured for the series of compounds (I-IV, XIII, XVI, XVIII, XXIV), and were found to change very little as the substituent R in the COOR group was varied. This suggests that the geometry of the dihydropyridine ring is not affected greatly by substituent effects, with the exception of compound XI, in which the increased value of the $J_{^{13}C(4)-^{1}H}$ coupling constant is due to a more planar conformation of the dihydropyridine ring [9].

EXPERIMENTAL

^{13}C-NMR spectra were recorded at a frequency of 22.63 MHz on a Bruker WH-90/DS pulse spectrometer, using 10% solutions of deuterodimethyl sulfoxide at a temperature of 40-42°C. The accuracy of the CS measurements was ±0.03 ppm. Cyclohexane (δ27.44 ppm) was used as the internal standard. The previously unknown 2,6-dimethyl-4-phenyl-1,4-dihydropyridine-3,5-dicarboxylic acid ester derivatives VIII-X were prepared by the Hantzsch reaction. The products were recrystallized from ethanol.

Dineopentyl Ether (VIII). mp 150-151°C. IR spectrum (nujol): 3315 (br), 3235 (NH), 1703 (C=O), 1648 cm^{-1} (C—C). UV spectrum, λ_{max} (log ε) (ethanol): 2.35 (4.26), 351 nm (3.86). ^{1}H-NMR spectrum ($CDCl_3$): 0.91 (18H, s, $C(CH_3)_3$); 2,37 6H, s, 2,6-CH_3; 3.64 (2H, d) and 3.80 (2H, d, ^{2}J = 10.5 Hz, OCH_2); 5.14 (1H, s, 4-H); 5.64 (1H, br.s, NH); 7.23 ppm (5H, m, 4-C_6H_5).

Dipropargyl Ester (IX). mp 143-144°C. IR spectrum (nujol): 3295, 3270, 3235 (NH), 1686 (C=O), 1650 cm^{-1} (C=C). UV spectrum, λ_{max} (log ε) (ethanol): 239 (4.21), 357 nm (3.83). ^{1}H-NMR spectrum ($CDCl_3$): 2.36 (6H, s, 2,6-CH_3); 2.41 (2H, t, ≡CH); 4.65 (4H, d, ^{4}J = 2.4 Hz, OCH_2); 5.03 (1H, s, 4-H); 5.73 (1H, br.s, NH); 7.27 ppm (5H, m, 4-C_6H_5).

Diallyl Ester (X). mp 104-105°C. IR spectrum (nujol): 3345 (NH), 1700 (C=O), 1662, 1650 cm^{-1} (C=C). UV spectrum, λ_{max} (log ε) (ethanol): 238 (4.24), 3.56 nm (3.84). ^{1}H-NMR spectrum ($CDCl_3$): 2.35 (6H, s, 2,6-CH_3); 4.56 (4H, m, ^{3}H = 5.1 Hz, ^{4}J = 1.2 Hz, OCH_2); 5.07 (1H, s, 4-H); 5.21 (4H, m, ^{3}J = 5.1 Hz, ^{4}J = 1.2 Hz, OCH_2); 5.59 (1H, br. s, NH); 5.88 (2H, m, CH); 7.26 ppm (5H, m, 4-C_6H_5).

2,6-Dimethyl-4-phenyl-3,5-dinitro-1,4-dihydropyridine (XXXI). This was prepared by refluxing for 2 h a mixture of 0.92 g (10 mmole) nitroacetone, 0.54 g (5 mole) benzaldehyde, and 4.3 g (50 mmole) ammonium acetate in 10 ml acetic acid. Yield of compound (XXXI) 0.97 g (71%), mp 178°C (from ethanol). IR spectrum (nujol): 3300 (NH), 3240, 3100, 1650, 1630 C=C) cm^{-1}. UV spectrum, λ_{max} (ethanol): 207, 247, 263 (sh), 304, 432 nm. ^{1}H-NMR spectrum (DMSO-D_6): 2.40 (6H, s, 2,6-DH_3), 5.56 (1H, s, 4-H), 7.22 (5H, m, 4-C_6H_5), 10.02 ppm (1H, s, NH).

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