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Differences in proton–proton coupling constants of N^+–CH_2–CH_2 protons of some betaines, N^+–$(CH_2)_{2\text{-}3}$–COO^-, and their complexes in aqueous solution

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Abstract

Synthesis and ^{1}H NMR spectra in D_2O of 4 betaines and 19 betaine complexes with mineral acids containing 2 or 3 CH_2 groups in the tether, N^+–$(CH_2)_n$–COO^-, $n = 2, 3$, and diverse volume of the positively charged groups are reported. In compounds containing three CH_2 groups in the tether and three substituents at the nitrogen atom or α, α′-disubstituted pyridine ring, a characteristic multiplet for an $AA'MM'X_2$ spin system is observed. This is consistent with preference for *trans* conformation (68–85%). In the spectra of compounds with two CH_2 groups in the tether or three CH_2 groups and unsubstituted pyridine ring, the multiplet changes to a triplet and gives apparent A_2X_2 and $A_2M_2X_2$ spectra, respectively, consistent with no significant conformational preference. Both the number of CH_2 groups in tether and the bulkiness of the charged groups are responsible for the observed differences of N^+CH_2 multiplicity and reflect changes in conformational preferences. According to the PM3 calculations, in the gas phase a *gauche*-like conformer is more stable than the *trans*, but in aqueous solution it is reverse.

Keywords: Betaines; ^{1}H NMR spectra; $AA'MM'X_2$; $A_2M_2X_2$; A_2X_2 spin systems; Conformation

1. Introduction

The relation between the magnitude of vicinal coupling constants (J_{vic}), which vary from 0 to 16 Hz (generally 5–8 Hz in freely rotating systems), and dihedral angles in X–CH_2–CH_2–Y systems, usually referred to as a Karplus-type equation, is widely used in structural and conformational analysis [1,2]. Spin system depends not only on the torsion angle, but also on several other factors, which the electronegativity of X and Y (the charges on X and Y) and their volumes are most important [1–4]. In betaines and sulfobetaines (R_3N^+–CH_2–CH_2COO^-/SO_3^-), the attractive electrostatic (Coulombic) interactions between the charged substituents are essentially important.

Recently, Gregoire et al. [5] applied the Karplus relationship in the form developed by Altona et al. [3] to determine conformations of zwitterionic forms of β-alanine (H_3N^+–CH_2–CH_2–COO^-), taurine (H_3N^+–CH_2–CH_2–SO_3^-) and their *N,N,N*-trimethyl derivatives. A statistical equilibrium of the *gauche* and *trans* conformations were found in most compounds, except *N,N,N*,-trimethyl-taurine, where

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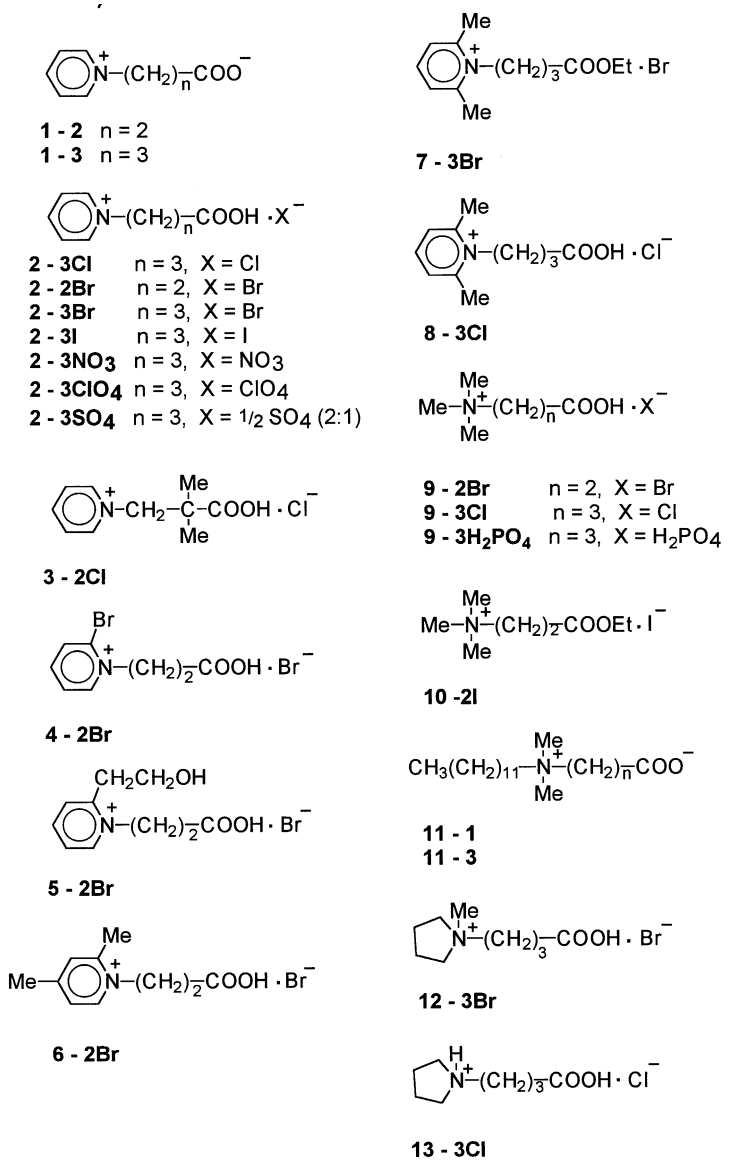


Scheme 1.

the *trans* form predominates. This is probably caused by a steric hindrance (the SO_3 group is more bulkier than the CO_2 one).

Intercharge distance in the homologous series of carboxybetaines, $R_3N^+(CH_2)_nCOO^-$ with $n = 1$–7 ($R = CH_3$ [6]or long alkyl chain of C_{12} or C_{16} [7]), and of sulfobetaines ($R_3N^+(CH_2)_nSO_3^-$ with $n = 1$–4 [8], R-$PyN^+(CH_2)_3SO_3^-$ [8], PyN — pyridine) have also been derived from NMR spectra or molecular and quantum mechanics.

In the present work we investigate ^1H NMR spectra in D_2O of alkylammonio- and pyridininio betaines and their complexes with mineral acids, containing two or three CH_2 groups in tether ($N^+(CH_2)_{2\text{-}3}COO^-$), different size of the positively charged groups (Scheme 1) and focus our attention

Table 1
Melting points and ^{1}H chemical shifts, δ (ppm/DCC) at 300.07 MHz for the investigated compounds in D_2O

Compound	Mp (°C)	Ring					Tether			Substituent
		H-2	H-3	H-4	H-5	H-6	N–CH_2	CH_2	CH_2COO	
1-2	127–128	8.89	8.07	8.56	8.07	8.89	4.83t (6.57)[a]	–	2.93t	
1-3	107–198	8.88	8.09	8.57	8.09	8.88	4.65t (7.25)	2.28	2.28	
2-2Br	140–142	8.94	8.08	8.57	8.08	8.94	4.90t (6.31)	–	3.19t	
2-3Cl	160–161	8.89	8.09	8.57	8.09	8.89	4.69t (7.29)	2.33p	2.51t	
2-3Br	161	8.89	8.09	8.57	8.09	8.89	4.69t (7.42)	2.33p	2.52t	
2-3I	115–117	8.88	8.09	8.56	8.09	8.88	4.66t (7.14)	2.32p	2.40t	
2-3NO_3	77–78	8.88	8.09	8.57	8.09	8.88	4.68t (7.42)	2.33p	2.49t	
2-3ClO_4	100–101	8.88	8.09	8.57	8.09	8.88	4.68t (7.42)	2.32p	2.51t	
2-3SO_4	136–137	8.89	8.10	8.58	8.10	8.89	4.69t (7.28)	2.33p	2.52t	
3-2Cl	179–180	8.85	8.09	8.61	8.09	8.85	4.82s	–	–	1.31s (CH_3)
4-2Br	181–183	–	8.39	8.39	8.04	9.11	5.08t (6.59)	–	3.19t	
5-2Br	133	–	8.04	8.46	7.91	8.87	4.94t (6.87)	–	3.12t	3.44t (CH_3), 4.08t (CH_2O)
6-2Br	189–190	–	7.73	–	7.67	8.59	4.76t (6.93)	–	3.03t	2.57s (4-CH_3), 2.81s (2-CH_3)
7-3Br	171–172	–	8.21	7.76	8.21	–	4.56m	2.20m	2.69t	1.30t (CH_3), 2.90s (CH_3), 4.22q (OCH_2)
8-3Cl	205	–	7.53	7.99	7.53	–	4.35m	1.95m	2.45t	2.67s (CH_3)
9-2Br	173–175	–	–	–	–	–	3.62t (7.69)	–	2.81t	3.14s (CH_3)
9-3Cl	220dec	–	–	–	–	–	3.36m	2.09m	2.47t	3.14s (CH_3)
9-3H_2PO_4	188–189	–	–	–	–	–	3.36m	2.09m	2.46t	3.13s (CH_3)
10-2I	149	–	–	–	–	–	3.50t (7.55)	–	2.80t	1.06t (CH_3), 2.95s $(CH_3)_3$, 4.01q (OCH_2)
11-3	103–108	–	–	–	–	–	3.27m	1.95m	2.23t	0.87t (CH_3), 1.28 $(CH_2)_7$, 1.34 $(CH_2)_2$ 1.73 (CH_2), 3.07s $(CH_3)_2$, 3.27m (NCH_2)[b]
11-1	155	–	–	–	–	–	3.83s	–	3.83s	0.87t (CH_3), 1.29 $(CH_2)_7$, 1.34 $(CH_2)_2$, 1.71 (CH_2), 3.20s $(CH_3)_2$, 3.57m (NCH_2)[b]
12-3Br	162–164	3.53	2.21	2.21	3.53	–	3.37m	2.09m	2.48t	3.07s (CH_3)
13-3Cl	116–121	3.67eq 3.09ax	2.12	2.12	3.67eq 3.09ax	–	3.23m	2.00m	2.49t	

[a] Coupling constant.
[b] Data for C_{11}–CH_2–N^+, s — singlet, m — multiplet, p — pentet (quintet), q — quartet, t — triplet, eq — equatorial, ax — axial

mainly on multiplicity of the N^+CH_2 protons. In most compounds, as expected, the chemical shifts of the N^+–CH_2 protons are equivalent by rapid rotational interchange between the *gauche* and *trans* rotamers and give apparent an A_2X_2 or $A_2M_2X_2$ spectra [9]. In some spectra, however, a multiplet is present, which can be described by typical $AA'MM'X_2$ spin system.

2. Experimental

2.1. Synthesis

Most of the compounds were prepared by slow addition of respective ω-halogenocarboxylic acids or their esters (0.1 mol) to pyridines, *N*-methylpyrrolidine, pyrrolidine, trimethylamine (40% aqueous solution) or *N*-dodecyl-*N,N*-dimethylamine on cooling. Then the mixture was stirred up to precipitation and finally left to stand from 2 d to 2 weeks to increase the yield of the reaction [10,11]. In the case of 2,6-lutidine or 3-chloropivalic acid the reaction was carried out at 100°C. The crude product was filtered off and washed with diethyl ether to remove unreacted materials and recrystallized from acetonitrile or acetonitrile–methanol mixture (10:1). Yields were usually higher than 60%.

In the case of reaction between trimethylamine and

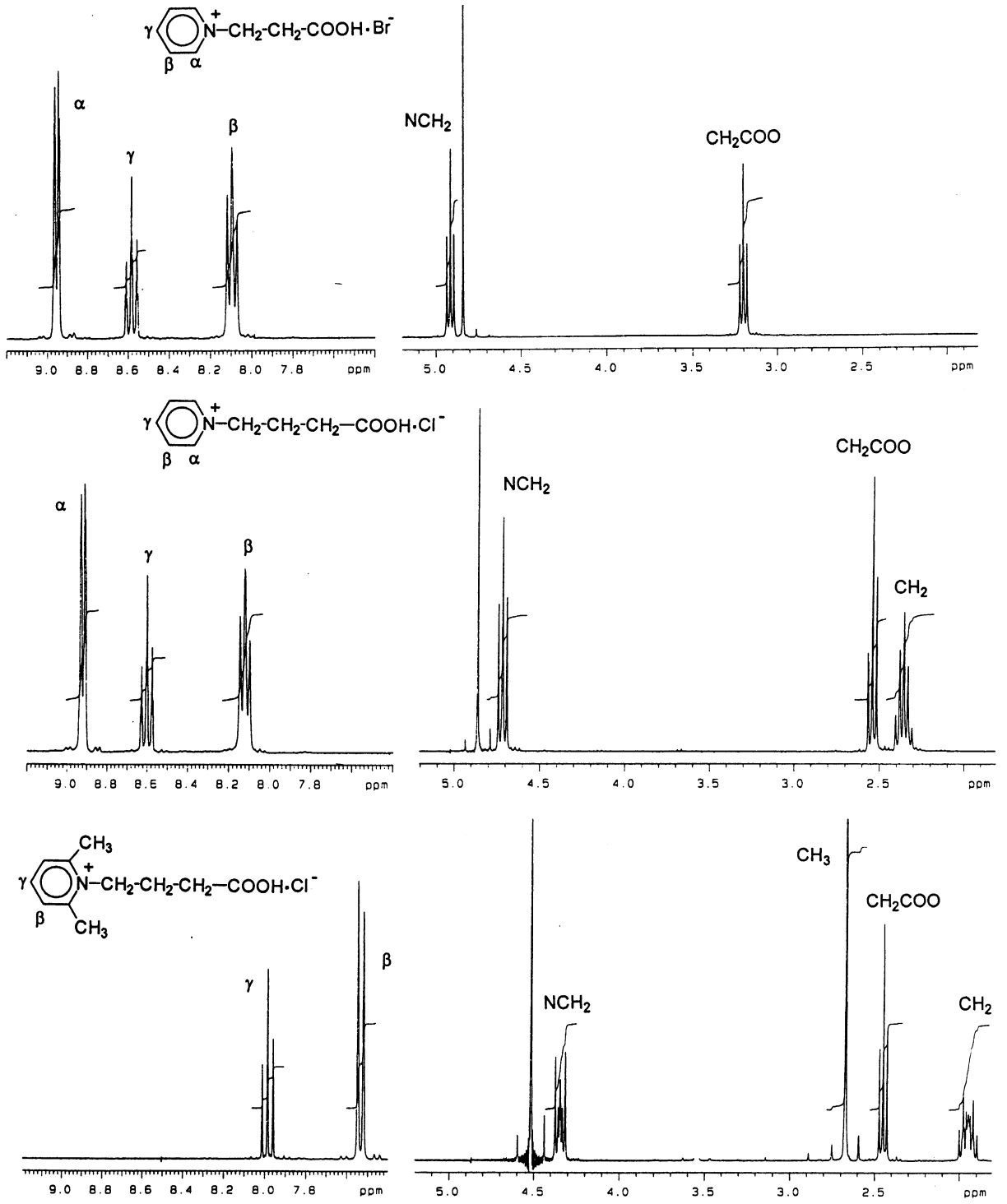


Fig. 1. ^{1}H NMR spectra of **2-2Br**, **2-3Cl** and **8-3Cl** in D_2O solution.

3-bromopropionic acid, we obtained **11-1**, as was described by Chen and Mak [12] and we did not observe the Hoffmann elimination reaction as was reported previously [6,7]. Indeed, the Hoffmann elimination reaction occurs in the case of 2,6-lutidine, which is probably caused by temperature (100°C).

The obtained hydrochlorides or hydrobromides (5 g) were then dissolved in 1:1 water–methanol mixture (50 cm^3) and treated with wet anion-exchange resin, Amberlite 420 (60 g) in its basic form. The mixture was stirred ca. 2 h until the organic layer gave a negative Beilstein test for halide. The residue was separated, washed with methanol and the eluate was evaporated under vacuum below 70°C. Each crude betaine was dried over P_2O_5 in vacuum to get a solid product and recrystallized from acetonitrile–methanol mixture (10:1).

The remaining complexes were prepared by mixing equivalent of betaines with acid in methanol. The precipitates were recrystallized from acetonitrile–methanol mixture (5:1) or methanol. Melting points of investigated compounds are listed in Table 1.

2.2. Spectra

The ^1H NMR spectra were recorded on a Varian Gemini 300 VT spectrometer operating at 300.07 MHz. The spectra were measured in D_2O (20 mg in 0.7 cm^3) relative to internal standard 3-(trimethylsilyl)propionic-d_4 acid sodium salt.

The FTIR spectra were measured on a Bruker IFS 113v instrument in D_2O solution in cell with CaF_2 windows, 0.025 mm thick.

Proton–proton coupling constants were analyzed by a computer simulation with the Varian LAOCOON software.

2.3. PM3 calculations

Structures and energies were calculated by means of the semiempirical PM3 method [13] as implemented in AMPAC 6.55 [14] and MOPAC 5.0 [15] program packages. The solvent effect was taken into account by using the self-consistent reaction field (SCRF) method [16–19]. The following cavity radius 3.3 Å ($n = 2$) and 4.2 Å ($n = 3$) were estimated from the geometries of all *trans* and *gauche* conformers.

3. Results and discussion

All the investigated ^1H NMR spectra vary with concentration. When the solute concentration of **2-2Br** decreases progressively from 0.5 to 0.015 M, the entire spectrum is shifted towards higher field ($\Delta\delta$ is 0.08 ppm for 2,6-H, 0.06 ppm for N^+CH_2 and 3,5-H, 0.15 ppm for 4-H and CH_2COO protons). Correlations of the chemical shifts and concentration is linear with a slope between 0.12 and 0.26 and $r = 0.86 - 0.99$ and reflects the process of aggregation, which is a typical process of zwitterions in water. The effect of counterion on chemical shifts is small (compare data for **2-3X** in Table 1). FTIR spectra of complexes in D_2O show an intense band in the 1720–1710 cm^{-1} region attributed to the $\nu C{=}O$ mode in COOH group.

As follows from Fig. 1, the most interesting feature is the multiplicity of the N^+CH_2 protons. The spectra of compounds containing three CH_2 groups in the tether ($n = 3$) and three substituents at the nitrogen atom (sp^3 hybridization) (**9-3Cl**, **9-3H_2PO_4**, **11-3, 12-3Br** and **13-3Cl**) or 2,6-disubstituted pyridine ring (sp^2 hybridization) (**7-3Br** and **8-3Cl**) are characterised by a multiplet typical of the $AA'MM'X_2$ spin system. In the spectrum of **11-3** the signals of both methylene groups connected with the charged nitrogen atom ($-CH_2-N^+-CH_2-$) show similar multiplets but their chemical shifts are so close that we were not able to separate them. The multiplet is also present in the spectrum of **11-1** for $C-CH_2N^+$. In the spectra of remaining compounds containing two CH_2 (**1-2**, **2-2Br**, **3-2Cl**, **4-2Br**, **5-2Br**, **9-2Br** and **10-2I**) or three CH_2 groups in tether and unsubstituted pyridine ring (**1-3**, **2-3Cl**, **2-3Br**, **2-3I**, **2-3NO_2**, **2-3ClO_4** and **2-3SO_4**) the N^+CH_2 protons display a triplet with the coupling constant of 6.94 ± 0.50 Hz ($n = 2$) and 7.32 ± 0.11 Hz ($n = 3$). These molecules also belong to $AA'XX'$ or $AA'MM'X_2$ systems, respectively, but they give apparent A_2X_2 and $A_2M_2X_2$ spectra.

If the spectrum is representative of an $AA'MM'$ ($M = X$ in $AA'XX'$) spin system, then J_{AM}, $J_{AM'}$, $J_{AA'}$, $J_{MM'}$, ν_A and ν_M must have values such as to

Table 2
Coupling constants and chemical shifts for N^+CH_2–CH_2 protons as part of $AA'MM'$ spin system and average percentage of *trans* conformers at 300.07 MHz and 21°C

	7-3Br	**8-3Cl**	**9-3Cl**	**9-3H$_2$PO$_4$**	**12-3Br**	**13-3Cl**	**9-2Br**	**11-1**
$J_{AA'}$	15.6	16.2	10.2	11.7	11.1	13.2	16.8	18.5
J_{AM}	12.3	12.3	12.1	12.4	12.4	10.7	9.0	12.1
$J_{AM'}$	5.0	5.1	5.1	4.7	4.9	5.5	6.4	4.5
$J_{MM'}$	15.6	16.2	10.2	11.7	11.1	13.2	13.1	18.5
ν_A	1370.1	1304.4	1014.0	1007.9	944.0	970.0	1087.2	1071.6
ν_M	656.6	584.8	628.7	623.3	559.6	602.0	885.0	517.3
% trans	81.5	80.5	85.5	85.5	83.5	68	50	82

give line shapes that are consistent with the observed line widths [20–23]. To solve this problem the Varian LAOCOON software was used to simulate spectra. The coupling constants obtained are listed in Table 2 and the experimental and simulated spectra are compared in Figs. 2 and 3. The percentage of *trans* conformers listed in Table 2 was estimated using the procedure recently published by Gregoire et al.[5]. Our data for **9-2Br** slightly differ from those given in Ref. [5]. Note that the signal of the N^+CH_2 protons in **9-2Br** slightly differs from typical triplet (Fig. 2).

The long-range COSY experiment was used to estimate the long-range couplings [23]. As Fig. 4 shows, in **9-2Br** and **9-3Cl** there is a coupling through four bonds between the CH_3 and N^+CH_2 protons. In **9-3Cl** an additional coupling occurs between the N^+CH_2 and CH_2COO protons. The lack of coupling between CH_3 and CH_2COO protons implies that the distance between them is much longer and this confirmed an extended structure (≥80% of *trans* conformation).

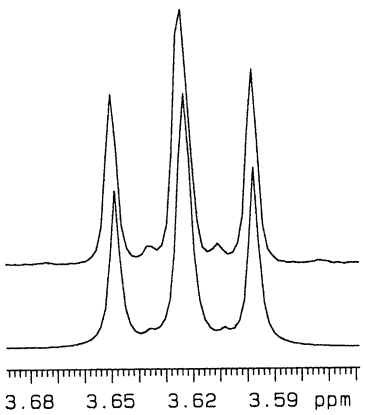


Fig. 2. Observed (upper) and calculated (lower) resonance due to the N^+–CH_2 group of **9-2Br**.

When $n = 2$ (Table 3), the PM3 calculations suggest that in the gas-phase, the *trans* conformer of betaine and β-alanine should be less stable than the *gauche* one. It can be expected that while water would diminish the electrostatic effect by virtue of its high dielectric constant, the strong gas-phase preference for *gauche* conformation would not be maintained in water solution. Indeed, the PM3 data suggest that in aqueous solution the *trans* conformers are more stable than the *gauche* one by 20.4 and 14.0 kcal/mol, respectively. A similar situation is observed for compound with $n = 3$ (Table 4); in the gas-phase the most stable is *gauche–gauche* conformation, while in aqueous solution the *trans–trans* conformation. For such differences of energy, rotation is sufficiently fast such that the resulting isomers cannot be separated in ^{1}H NMR spectra at room temperature [24].

Gregoire et al. [5] carried out ab initio calculations for zwitterionic β-alanine. They found that in aqueous solution the *gauche*-like conformer is more stable than the *trans*-like by 2–3 kcal/mol, while in the gas phase the difference is more than 20 kcal/mol. Generally, the PM3 method reproduces relative energies and geometries of organic molecules comparable to those of ab initio data. The discrepancy between the ab initio data from Ref. [5] and our PM3 data are probably caused by the cavity radius. In our opinion, values of $r_0 = 1.2$ Å [5] used in the ab initio calculations is too small.

The accuracy of determining the conformational preference is at least somewhat compromised by the fact that we have no assurance that the rotational

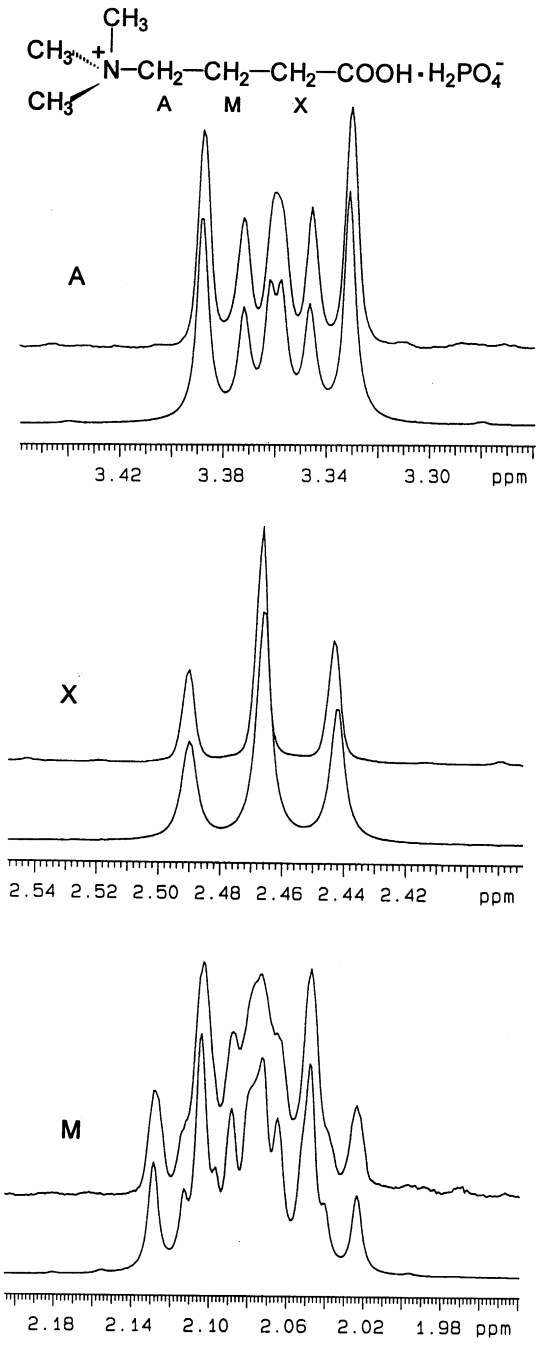


Fig. 3. Observed (upper) and calculated (lower) spectrum of **9-3H_2PO_4**.

angles for *gauche* and *trans* conformations are exactly 60 and 180°, respectively. Another problem is the size of the cavity radius (a_0) (see Ref. [18]). Finally, zwitterions form micelles [25,26] and our calculations were carried out for simple (individual) molecules. In conclusion, we can state that the values obtained are probably overestimated, however the predicted changes on going from the gas-phase to the aqueous solution seems reasonable. Because of the electrostatic attraction between the two charged end groups, the conformation and dipole moment in solution, depend strongly on tether flexibility (energy barriers between rotational *trans* and *gauche* isomers), bulkiness and solvation of charged groups preventing their close approach. If the charged groups are hydrated, their volume is enlarged by hydration water volume, and their closer approach would require water removal from them, which is allowed if the electrostatic energy is larger than the dehydration energy. Consequently, electrostatic attractions are controlled by dielectric properties of the medium; if water is the solvent, its high dielectric constant should reduce intramolecular electrostatic attractions in zwitterions. Lastly, zwitterionic molecules cause solvent polarisation and disturb dielectric discontinuity, which is a source of an additional electric field called reaction field [27]. In consequence an extended conformer (close to *trans*) in D_2O solution should be more stable than a folded one (close to *gauche*). Formation of dimers or larger aggregates favour intermolecular electrostatic interactions and extended conformations [6,8,28,29].

The question arising now is why a triplet is observed in the spectra of all compounds with two CH_2 groups in the tether or with three in the case of non-substituted pyridine ring. Electrostatic attractions in zwitterions between the two charged groups varies with the length of the tether, its bulkiness and phase, and consequently, their rotation barriers are ca. three times higher relative to those for the uncharged compounds. In betaine, $Me_3N^+CH_2COO^-$, the calculated rotational barrier of the carboxylic group is 9.7 kcal/mol [30]. In micelles tumbling of individual molecules is probably slowed down, which can cause an acceleration of proton relaxation with diversified relaxation behaviour of individual lines because the dominating relaxation mechanism is via dipole–dipole

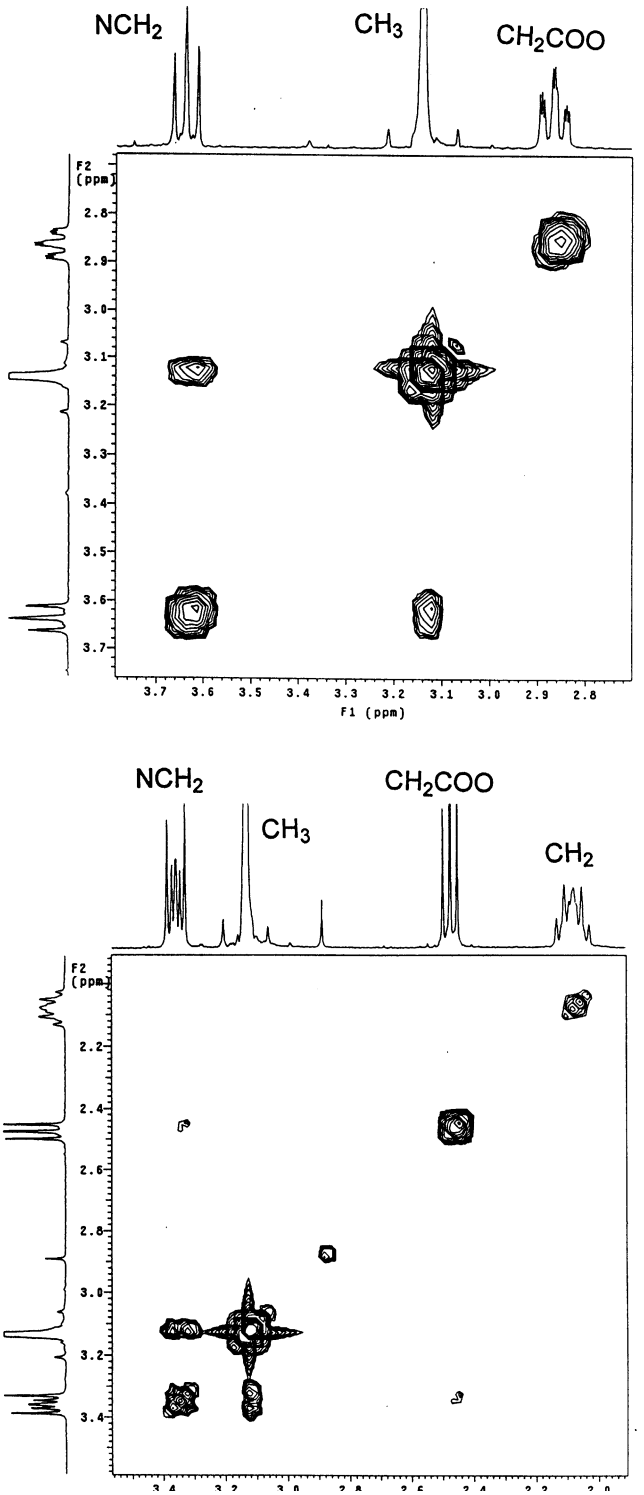
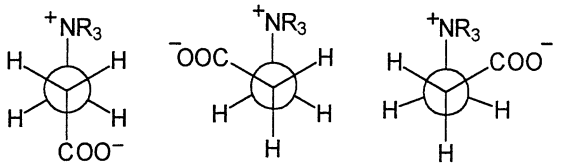


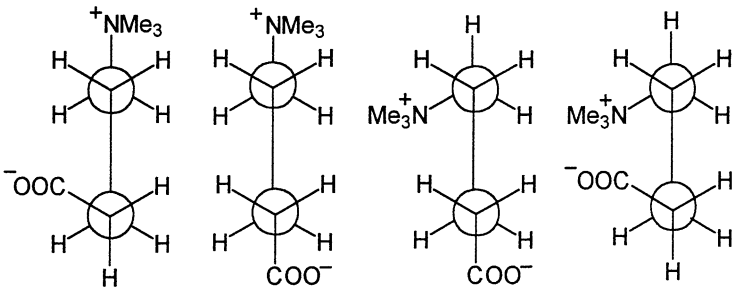
Fig. 4. Long-range COSY spectra of **9-2Br** (upper) and **9-3H_2PO_4** (lower).

Table 3
PM3 relative energies and N···O distances for $R_3N^+CH_2CH_2COO^-$



	trans		*gauche*	
	R = Me	R = H	R = Me	R = H
Gas phase				
E_{rel} (kcal/mol)	16.5	28.64	0	0
ΔH_f (kcal/mol)	−37.08	−38.61	−53.57	−67.25
N···O (Å)	4.431	4.193	2.948	2.560
N···O (Å)	4.647	4.889	4.247	4.146
Aqueous phase				
E_{rel} (kcal/mol)	0	0	20.4	14.0
ΔH_f (kcal/mol)	−123.51	−112.10	−103.09	−98.09
N···O (Å)	4.623	4.511	3.509	3.062
N···O (Å)	5.130	5.039	4.773	4.510
r_o(Å)	3.5	3.5	3.5	3.5

Table 4
PM3 relative energies and N···O distances for $Me_3N^+CH_2CH_2CH_2COO^-$



	trans–gauche	*trans–trans*	*gauche–trans*	*gauche–gauche*
Gas phase				
E_{rel} (kcal/mol)	1.8	17.2	14.6	0
ΔH_f (kcal/mol)	−46.21	−30.75	−33.43	−47.99
N···O (Å)	3.876	5.182	4.866	3.370
N···O (Å)	5.591	5.972	5.488	4.879
Aqueous phase				
E_{rel} (kcal/mol)	14.3	0	9.5	23.9
ΔH_f (kcal/mol)	−89.70	−104.04	−94.54	−80.12
N···O (Å)	4.086	5.182	5.277	3.504
N···O (Å)	5.760	5.972	5.829	5.055
r_o (Å)	4.2	4.2	4.2	4.2

interactions. Although replacement of pyridine α-protons by CH_3 group should not affect much the free rotation of the tether [24], but it change its solvation, some bonds, torsion angles and distances [31]. All these effects can be responsible for the observed multiplicity differences.

The overall conclusion that we draw is that there is still a need for more experiments for better understanding of why compounds studied here with $n = 3$ differ from those with $n = 2$ in spin multiplicity and hence in conformational preferences.

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