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NMR spectroscopic study of some novel *N,N*-disubstituted phosphonoacetamides

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ABSTRACT

The complete ^{1}H and ^{13}C NMR assignments of a series of tertiary phosphonoacetamides (**1**) with different *N*-substitution patterns are reported. *N,N*-Dialkyl derivatives show two sets of signals due to hindered rotation of the (O)C—N bond, which originate *E/Z* diastereoisomers for unsymmetrically *N,N*-disubstituted derivatives. Complete assignment of the ^{1}H NMR resonances of both rotamers was made on the basis of the magnitude of the diamagnetic shifts ($\Delta\delta$) experienced by the signals on changing the solvent from $CDCl_3$ to C_6D_6, and confirmed in the corresponding NOESY spectra. Differential assignment of the ^{13}C NMR signals was performed by HSQC experiments. *N*-Alkyl-*N*-phenyl phosphonoacetamides show a unique set of signals corresponding to the *E* isomers as disclosed by NOESY experiments.

1. Introduction

Structural features of amides have been widely studied by NMR spectroscopy and molecular modeling methods, as they represent model compounds for the peptide bond [1]. The planar arrangement of the substituents in amides, due to partial (O)C—N double bond character, has a strong influence on the superstructure of peptides and proteins [2], while *E/Z* isomerization is a key process involved in protein folding and biocatalysis [3]. NMR spectroscopy has been widely used for the study of restricted rotation in amides from early [4] up to recent years [5]. Some very recent reports focus their interest on acetamides [6] and their α-substituted derivatives [1,7,8]. Phosphonoacetamides are interesting due to their capability of complexing different metals, and have found application in the diagnosis and treatment of several diseases [9], as extracting agents for alkaline, alkaline earth and transition metals [10], and as reaction catalysts [11]. In organic synthesis, such compounds represent key synthetic precursors of α,β-unsaturated amides [12]. As part of ongoing research on the LiOH promoted Horner–Wadsworth–Emmons olefination of carbonyl compounds [13], we recently needed to prepare some *N,N*-disubstituted phosphonoacetamides. To our knowledge, no systematic study on the spectral features of such compounds is available in the literature.

The partial double bond character of the (O)C—N bond in amides causes a substantial rotational barrier, which ranges between 15 and 23 kcal/mol [14,15]. In the NMR spectra, this leads to nonequivalence of both *N*-substituents. For unsymmetrically *N*-substituted amides, hindered rotation entails the existence of non isolable *E/Z* diastereomers, which can be observed as two different sets of signals unless the equilibrium is highly biased towards one of them.

We present here the spectral characterization of a series novel tertiary phosphonoacetamides with different substitution patterns. Although all of the compounds evidence hindered rotation around the (O)C—N bond, different situations arise depending on the nature of the *N*-substituents. *N,N*-Dialkyl derivatives **1a–d** display two sets of signals corresponding to both rotamers. Differential assignment of the ^{1}H signals was performed by means of their monodimensional spectra run in $CDCl_3$ and C_6D_6, and confirmed by NOESY. Differential assignment of the ^{13}C NMR signals was performed by HSQC experiments. At variance with compounds **1a–d**, in *N*-phenyl-*N*-alkyl derivatives **1e** and **1f** only one diastereoisomer is detected. For unsymmetrically *N,N*-disubstituted phosphonoacetamides **1b–f**, the influence of the *N*-substituents on the *E/Z* equilibrium is also analyzed.

2. Experimental

2.1. Chemistry

Compounds **1a–f** were prepared by reaction of the corresponding chloroacetamides (1 mmole) and triethyl phosphite (2 mmole). The mixtures were refluxed at 120 C for 12 h. The compounds were obtained as oils in 64–73% yields. Analytical data of compound **1e** were reported in the literature [16]. Analytical data of new compounds are as follows. Compound **1a**, anal. calcd. for $C_{10}H_{20}NO_4P$: C, 48.19; H, 8.09; N, 5.62. Found: C, 48.27; H, 8.13; N, 5.51. Compound **1b**, anal. calcd. for $C_{14}H_{22}NO_4P$: C, 56.18; H, 7.41; N, 4.68. Found: C, 56.03; H, 7.46; N, 7.49. Compound **1c**, anal.

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calcd. for $C_{15}H_{24}NO_4P$: C, 57.50; H, 7.72; N, 4.47. Found: C, 57.39; H, 7.81; N, 4.48. Compound **1d**, anal. calcd. for $C_{10}H_{20}NO_4P$: C, 58.70; H, 8.01; N, 4.28. Found: C, 58.78; H, 8.09, N, 4.07. Compound **1f**, anal. calcd. for $C_{14}H_{22}NO_4P$: C, 56.18; H, 7.41; N, 4.68. Found: C, 56.1; H, 7.51; N, 4.74.

2.2. Spectra

^1H and ^{13}C NMR spectra were recorded on a Bruker Avance II 500 spectrometer operating at 500.13 and 125.77 MHz, respectively. Spectra were acquired from samples as solutions at room temperature in 5 mm tubes. Unless otherwise indicated, deuterochloroform was used as the solvent. Chemical shifts (δ) are reported in ppm, referenced to TMS as an internal standard. Coupling constants (J) are reported in Hz. Multiplicities are quoted as singlet (s), doublet (d), double doublet (dd), triplet (t), quartet (q), heptet (h) and multiplet (m). ^1H-detected, one-bond HSQC spectra were recorded on a Bruker Avance II 500 spectrometer. Phase-sensitive NOESY spectra were recorded on the same spectrometer. Reagents, solvents and starting materials were purchased from standard sources.

3. Results and discussion

The compounds described in this work are shown in Fig. 1. ^1H NMR chemical shifts, multiplicities and relative populations of *E/Z* diastereomers of compounds **1a–f** ($CDCl_3$) are given in Table 1. ^1H NMR chemical shifts, multiplicities and relative populations of both diastereomers of compounds **1a–d** (C_6D_6) are given in Table 2. ^{13}C NMR chemical shifts of compounds **1a–f** ($CDCl_3$) are given in Table 3. For compounds **1a–d**, differential assignment of the ^{13}C resonances of both rotamers was unambiguously made on the basis of the correlations observed in their HSQC spectra.

Due to hindered (O)C—N rotation, compound **1a** displays separate ^1H signals for methylenes *c–f*, both in $CDCl_3$ and C_6D_6 (Tables 1 and 2). Previous data in the literature [14] indicate that signals of *N*-alkyl groups *trans* to the carbonyl oxygen experience a stronger diamagnetic shift ($\Delta\delta$) on changing the solvent from $CDCl_3$ to C_6D_6. Following this criterion, the triplet in **1a** was attributed to the methylene group *trans* to the carbonyl oxygen (*f*), and the double triplet to the *cis* methylene (*c*) ($\Delta\delta$ = −0.42 and −0.07 ppm, respectively). The additional splitting of the second signal may arise from long-range coupling with the ^{31}P nucleus. This tentative assignment was confirmed by the correlations observed in the corresponding NOESY spectrum, which also allowed for the assignment of methylenes *d* and *e* (Fig. 2). The ^{13}C NMR spectrum of **1a** (Table 3) also displays separate signals for both rotamers around the (O)C—N bond. Unambiguous differential assignment of the resonances (Table 3) was performed on the basis of the correlations observed in the corresponding HSQC spectrum.

^1H spectra of compounds **1b–d** also show two sets of signals with different populations, corresponding to *E/Z* diastereomers. Differential assignment of the ^1H resonances of both stereoisomers of **1b–d** (Table 1) was performed on the basis of the criterion previously applied for **1a**. Thus, for **1b**, singlets at 2.96 and 3.02 ppm ($\Delta\delta$ = −0.25 and −0.48 ppm) were attributed, respectively, to the *cis* and *trans* *N*-CH_3 groups. Singlets at 4.68 and 4.60 ppm were assigned, respectively, to the *trans* and *cis* benzyl methylenes ($\Delta\delta$ = −0.30 and −0.18 ppm). For compound **1c**, the *N*-ethyl quartets at 3.42 and 3.45 ppm were attributed, respectively, to the *trans* and *cis* methylene groups ($\Delta\delta$ = −0.22 and −0.05 ppm), and singlets at 4.62 and 4.69 ppm ($\Delta\delta$ = −0.02 and −0.11 ppm) to the *cis* and *trans* methylenes in the benzyl moieties. For compound **1d**, the methyne signals at 4.88 and 4.38 ppm ($\Delta\delta$ = 0.10 and −0.17 ppm) were assigned, respectively, as *cis* and *trans*, while singlets at 4.66 and 4.57 ppm ($\Delta\delta$ = −0.06 and −0.05 ppm) were

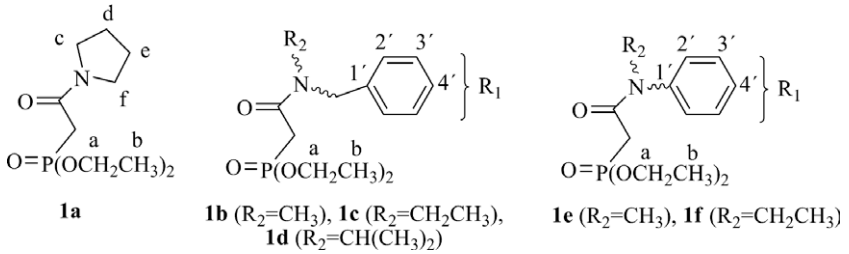

Fig. 1. *N,N*-disubstituted phosphonoacetamides **1a–f**.

Table 1
^1H NMR signals and relative populations of phosphonoacetamides **1a–f** ($CDCl_3$).

Compd.	CH_2P	OCH_2CH_3	R_1	R_2	Rel. (%)
1a	2.91 (d, J = 22.2)	a: 4.06–4.13 (m), b: 1.26 (m)	c: 3.40 (dt, J_1 = 6.9, J_2 = 1.9), d: 1.77–1.82 (m), e: 1.86–1.92 (m), f: 3.52 (t, J = 6.8)		–
1b (*Z*)	3.11 (d, J = 22.1)	a: 4.06–4.21 (m), b: 1.23 (m)	CH_2: 4.60 (s), 2′–4′: 7.21–7.37 (m)	3.02 (s)	63
1b (*E*)	3.04 (d, J = 22.1)	a: 4.06–4.21 (m), b: 1.23 (m)	CH_2: 4.68 (s), 2′–4′: 7.21–7.37 (m)	2.96 (d, J = 1.3)	37
1c (*Z*)	3.12 (d, J = 22.2)	a: 4.11–4.22 (m), b: 1.33 (m)	CH_2: 4.62 (s), 2′–4′: 7.17–7.35 (m)	CH_2:3.42 (q, J = 7.3)*, CH_3: 1.16 (t, J = 7.3)	55
1c (*E*)	3.01 (d, J = 22.0)	a: 4.11–4.22 (m), b: 1.33 (m)	CH_2: 4.69 (s), 2′–4′: 7.17–7.35 (m)	CH_2: 3.45 (q, J = 7.1)*, CH_3: 1.13 (t, J = 7.1)	45
1d (*E*)	2.92 (d, J = 22.1)	a: 4.13–4.18 (m), b: 1.33 (m)	CH_2: 4.66 (s), 2́–4́: 7.25–7.34 (m)	CH: 4.88 (h, J = 6.7), CH_3: 1.13 (d, J = 6.7)	58
1d (*Z*)	3.22 (d, J = 22.1)	a: 4.19–4.25 (m), b: 1.36 (m)	CH_2: 4.57 (s), 2́–4́: 7.25–7.34 (m)	CH: 4.38 (h, J = 6.5), CH_3: 1.19 (d, J = 6.5)	42
1e (*E*)	2.81 (d, J = 21.7)	a: 4.08–4.18 (m), b: 1.30 (t, J = 7.0)	2́: 7.29 (d, J = 7.3), 3́: 7.43 (m), 4́: 7.35 (m)	3.29 (d, J = 1.2)	100
1f (*E*)	2.72 (d, J = 21.7)	a: 4.05–4.11 (m), b: 1.26 (t, J = 7.1)	2́: 7.20 (d, J = 7.3), 3́: 7.39 (t, J = 7.3), 4́: 7.32 (t, J = 7.3)	CH_2: 3.72 (q, J = 7.2), CH_3: 1.08 (t, J = 7.2)	100

* Partially overlapping signals.

Table 2
^{1}H NMR signals and relative populations of phosphonoacetamides **1a–f** (C_6D_6).

Compd.	CH_2P	OCH_2CH_3	R_1	R_2	Rel. (%)
1a	2.81 (d, J = 21.8)	g: 4.02–4.16 (m), h: 1.09 (m)*	c: 3.33 (dt, J_1 = 6.7, J_2 = 1.8), d,e: 1.13–1.26 (m)*, f: 3.10 (t, J = 6.7)		–
1b (*Z*)	2.86 (d, J = 22.1)	a: 3.97–4.09 (m), b: 1.05 (m)	CH_2: 4.42 (s), 2́–4́: 6.90–7.22 (m)	2.54 (s)	67
1b (*E*)	2.95 (d, J = 21.8)	a: 3.97–4.09 (m), b: 1.05 (m)	CH_2: 4.38 (s), 2́–4́: 6.90–7.22 (m)	2.71 (d, J = 1.3)	33
1c (*Z*)	3.07 (d, J = 22.0)	a: 4.08–4.24 (m), b: 1.19 (t, J = 7.1)	CH_2: 4.60 (s), 2́–4́: 7.08–7.38 (m)	CH_2: 3.20 (c, J = 7.2), CH_3: 0.78 (t, J = 7.2)	55
1c (*E*)	3.04 (d, J = 22.0)	a: 4.08–4.24 (m), b: 1.17 (t, J = 7.1)	CH_2: 4.58 (s), 2́–4́: 7.08–7.38 (m)	CH_2: 3.40 (c, J = 7.1), CH_3: 1.07 (t, J = 7.1)	45
1d (*E*)	2.99 (d, J = 21.8)	a: 4.06–4.21 (m), b: 1.12 (dt, J_1 = 7.1, J_2 = 0.4)	CH_2: 4.60 (s), 2́–4́: 7.05–7.41 (m)	CH: 4.98 (h, J = 6.8), CH_3: 1.00 (d, J = 6.8)	58
1d (*Z*)	3.12 (s) (d, J = 21.8)	a: 4.06–4.21 (m), b: 1.16 (dt, J_1 = 7.1, J_2 = 0.4)	CH_2: 4.52 (s), 2́–4́: 7.05–7.41 (m)	CH: 4.21 (h, J = 6.6), CH_3: 0.89 (d, J = 6.6)	42

* Partially overlapping signals.

Table 3
^{13}C NMR signals of phosphonoacetamides **1a–f** ($CDCl_3$).

Compd.	CH_2P	C=O	OCH_2CH_3 a b	R_1	R_2
1a	34.65 (d, J = 133.5)	163.08 (d, J = 5.5)	a: 62.50 (d, J = 6.4), b: 16.27 (d, J = 6.4)	c: 46.03, d: 24.44, e: 25.98, f: 47.54	
1b (*Z*)	33.39 (d, J = 131.7)	164.98* (d, J = 5.6)	a: 62.53 (d, J = 6.8), b: 16.22 (d, J = 6.8)	CH_2: 51.00, 1́: 136.71, 2́–4́*: 127.29, 127.77, 128.48	30.23
1b (*E*)	34.29 (d, J = 131.1)	165.27* (d, 5.6)	a: 62.56 (d, J = 5.7), b: 16.22 (d, J = 6.8)	CH_2: 54.13, 1́: 136.11, 2́–4́*: 126.18, 127.60, 128.87:	28.81
1c (*Z*)	33.17 (d, J = 134.5)	164.74* (d, J = 5.6)	a: 62.58 (d, J = 5.6), b: 16.26 (d, J = 5.6)	CH_2: 47.90, 1́: 137.24, 2́–4́*: 127.20, 127.68, 128.45	*N*-CH_2CH_3: 42.57, *N*-CH_2CH_3: 13.56
1c (*E*)	33.79 (d, J = 133.4)	165.00* (d, J = 5.6)	a: 62.58(d, J = 5.6), b: 16.26 (d, J = 5.6)	CH_2: 51.42, 1́: 136.62 2́–4́*: 126.23, 127.55, 128.87	*N*-CH_2CH_3: 41.48, *N*-CH_2CH_3: 12.45
1d (*E*)	34.50 (d, J = 132.6)	165.78* (d, J = 5.5)	a: 62.51 (d, J = 6.4), b: 16.28 (d, J = 6.4)	CH_2: 46.32, 1́: 138.04, 2́–4́*: 125.71, 127.21, 128.80	*N*-$CH(CH_3)_2$: 46.16, *N*-$CH(CH_3)_2$: 20.09
1d (*Z*)	33.67 (d, J = 132.6)	164.84* (d, J = 5.5)	a: 62.62 (d, J = 6.4), b: 16.28 (d, J = 6.4)	CH_2: 43.98, 1́: 138.90, 2́–4́*: 126.54, 126.70, 128.20	*N*-$CH(CH_3)_2$: 50.18, *N*-$CH(CH_3)_2$: 21.31
1e (*E*)	33.13 (d, J = 137.9)	164.74 (d, J = 5.6)	a: 62.35 (d, J = 6.8), b: 16.26 (d, J = 6.8)	1́: 143.77, 2́,3́: 129.81,127.43, 4́: 128.10	37.57
1f (*E*)	33.50 (d, J = 138.1)	165.00 (d, J = 5.6)	a: 62.38 (d, J = 6.4), b: 16.29 (d, J = 6.4)	1́: 142.03, 2́,3́: 129.77, 128.49, 4́: 128.28	*N*-CH_2CH_3:44.45, *N*-CH_2CH_3:12.84

* Assignment corresponding to both rotamers are exchangeable.

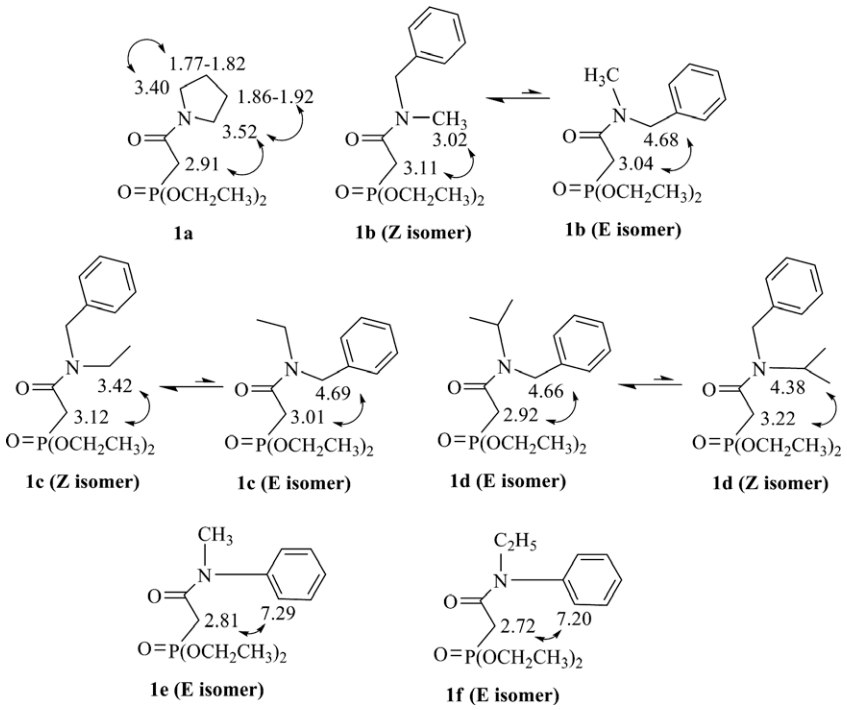

Fig. 2. Relevant correlations observed in the NOESY spectra ($CDCl_3$) of compounds **1a–f**.

attributed, respectively, to the *trans* and *cis* *N*-benzyl methylene groups. In all cases, the remaining signals were attributed to the major and minor diastereoisomers on the basis of their relative integration. Assignments of the ^{1}H NMR signals of compounds **1b–d** were confirmed by the correlations observed in the corresponding NOESY spectra (Fig. 2). The ^{13}C NMR spectra of com-

pounds **1b–d** also show separate signals for each diastereoisomer. In each case, differential assignment of the resonances (Table 3) was performed on the basis of the correlations observed in the corresponding HSQC spectra. For *N*-benzyl-*N*-alkyl derivatives **1b–d** both diastereoisomers have different populations, showing approximately the same ratio in $CDCl_3$ and C_6D_6 (Tables 1 and 2). From the differential assignments, it can be seen that the *Z* isomers (*N*-benzyl group *cis* to the carbonyl oxygen) are the predominant species in the case of **1b and c**. According to the relative volumes of both *N*-alkyl moieties, the equilibrium is more shifted towards the *Z* isomer for the methyl derivative **1b**. This preference is reversed in *N*-benzyl-*N*-isopropyl phosphonoacetamide **1d**, in which the more sterically hindering isopropyl group is *cis* to the carbonyl oxygen in the major diastereoisomer (*E*). In some of the compounds under study, it is noteworthy that the ^{1}H NMR chemical shifts of the *cis* and *trans* methylene groups are reversed with respect to the tendencies previously reported for model compounds [14], which indicate that *cis* methylene hydrogens are more deshielded than their *trans* counterparts. In fact, in **1a** this behaviour is reversed, *i.e.* the *cis* and *trans* methylenes appear respectively at 3.40 and 3.52 ppm, while in *N*-benzyl-*N*-alkyl phosphonoacetamides **1b–d** the *N*-C*H*$_2$Ph signals are more shielded in the *cis* than in the *trans* disposition. As regards the ^{13}C chemical shifts, in all cases the *N*-alkyl carbons *cis* to the carbonyl oxygen are more shielded than their *trans* counterparts. For compounds **1b–d**, the CH_2P signals *cis* to the more sterically hindering *N*-alkyl moieties are comparatively more deshielded.

At variance with *N,N*-dialkyl phosphonoacetamides, ^{1}H (Table 1) and ^{13}C NMR (Table 3) spectra of *N*-alkyl-*N*-phenyl derivatives **1e** and **1f** show a unique set of signals for each compound. This indicates that the *E/Z* equilibrium due to hindered (O)C—N rotation is shifted towards one of the diastereoisomers. The NOESY spectra of **1e** and **1f** show correlations between the *ortho* aryl hydrogens and the C*H*$_2$P signals (Fig. 2), indicating a preference for the *E* diastereoisomers. No correlations between these signals and the *N*-alkyl groups were observed.

4. Conclusions

We performed the NMR spectroscopic study of a series of tertiary phosphonoacetamides. The compounds under study evidence hindered rotation around the (O)C—N bond. *N,N*-Dialkyl derivatives show two sets of signals corresponding to both rotamers, with different populations for unsymmetrically *N*-substituted compounds. In *N*-phenyl-*N*-alkyl derivatives, only one diastereoisomer is detected. For compounds **1a–d**, assignment of the ^{1}H NMR resonances of both rotamers was based upon the differential $\Delta\delta$ experienced by the signals in $CDCl_3$ and C_6D_6 and confirmed by NOESY experiments. Differential assignment based on the $\Delta\delta$ criterion was more reliable than the one derived from chemical shifts of model compounds, which was reversed in some cases. The corresponding bidimensional heteronuclear HSQC spectra were used to assign the ^{13}C NMR signals, by correlating them with the previously assigned proton resonances.

For compounds **1b–e**, the relative stabilities of *E*/*Z* diastereomers depend on the relative steric hindrance of both *N*-substituents. In *N*-alkyl-*N*-phenyl derivatives **1e** and **1f**, the *E/Z* equilibrium is strongly biased towards the *E* diastereoisomers, which are the only detectable species. This preference cannot be entirely attributed to steric effects and may be due, at least in part, to additional interactions of the *N*-phenyl group.

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