MAGNETIC RESONANCE IN CHEMISTRY
*Magn. Reson. Chem.* 2005; **43**: 27–30
Published online 24 September 2004 in Wiley InterScience (www.interscience.wiley.com). DOI: 10.1002/mrc.1497

# Substituent effects on $^{15}$N NMR chemical shifts in selected *N*-alkylthiohydroxamic acids. A comparative study

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Received 2 December 2003; Revised 2 August 2004; Accepted 10 August 2004

**The $^{15}$N NMR spectra of three *N*-alkyl-δ-carbomethoxyvalerothiohydroxamic acids (2) and six synthesized *N*-isopropylbenzothiohydroxamic acids (3) were measured and compared with appropriate spectra of structurally similar hydroxylamines (1), benzohydroxamic acids (4), benzamides (5) and thiobenzamides (6). The analysis of the chemical shifts of the thiohydroxamic acids under investigation indicates that the inductive effect of the hydroxyl group rather than steric hindrance is responsible for non-additivity of the effect of substituents. Additionally, *N*-hydroxyl diminishes the effect of aromatic ring substituents on the $^{15}$N chemical shifts in the thiohydroxamic acids 3 which is approximately half that in the respective thiobenzamides 6. The chemical shift values correlate best with Brown's $\sigma^+$ parameter. Copyright © 2004 John Wiley & Sons, Ltd.**

**KEYWORDS:** NMR; $^{15}$N NMR; chemical shifts; gHMQC; thiohydroxamic acids; hydroxylamine derivatives

## INTRODUCTION

Owing to their biological activity, widespread application in chemical analysis, industry, and technology and especially in organic synthesis (so called Barton esters), thiohydroxamic acids (THAs) provide a very interesting subject of studies.[1] To our knowledge, this class of compounds has not been investigated earlier by means of $^{15}$N NMR. An issue that requires consideration is that even the chemical shift of the most popular representative of this class of compounds, i.e. *N*-hydroxypyridine-2-thione, is unknown.

We synthesized selected aliphatic thiohydroxamic acids (**2**) from the corresponding *S*-thioacyl dithiophosphates while investigating the chemoselectivity of these new thioacylating agents.[2] Aromatic thiohydroxamic acids (**3**) were obtained by direct thionation of hydroxamic acids, specifically for the spectroscopic study described in this paper. Moreover, our knowledge of the chemical shift values of some of the thiohydroxamic acids proved indispensable in continuing the investigation of the mechanism of reaction of benzohydroxamic acids with Lawesson's reagent.[3] Considering all the above, we present here $^{15}$N NMR resonance data for selected aliphatic and aromatic representatives of *N*-alkyl-THAs and the determination of the susceptibility of their $^{15}$N chemical shifts to electronic effects.

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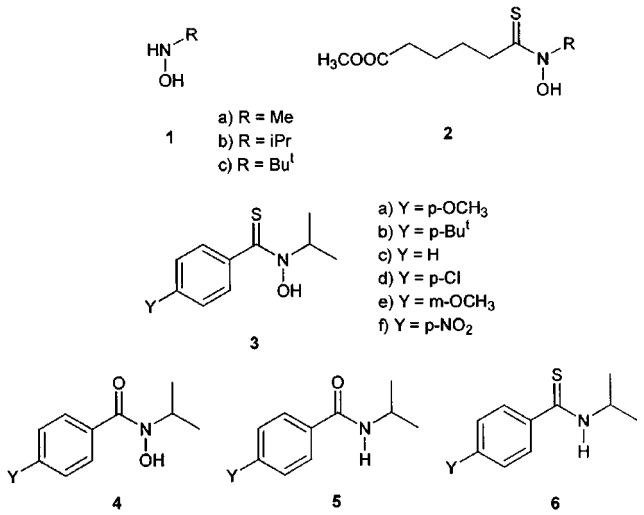
Contract/grant sponsor: Polish State Committee for Scientific Research (KBN); Contract/grant number: T09A 06116.

## RESULTS AND DISCUSSION

If we take into account both the data on chemical shifts of thioamides, whose spectra show signals at higher frequency values than those of the corresponding amides, and the data on hydroxamic acids, whose signals appear at even higher frequency values,[4] the shifts for thiohydroxamic acids can be expected at still higher $\delta$ values. It is well known that as the substituent-induced electron deficiency on the nitrogen atom increases, the $^{15}$N chemical shift becomes more positive. Let us consider the transmission of substituent-induced electronic effects and the substituent chemical shifts of (thio)amide nitrogen in the analogs of benzohydroxamic acids, i.e. benzamides and thiobenzamides. As expected, in both cases the presence of an electron-accepting group on the aromatic ring results in a higher frequency shift of the resonance signal of the nitrogen atom. The observed shift range ($\Delta\delta_{NO_2-OCH_3}$) measured in DMSO for *para*-substituted primary thiobenzamides is 7.85 ppm, which is almost twice that for the corresponding benzamides (4.59 ppm).[5] This is probably due to the fact that the lone electron pair on the N atom conjugates more effectively with a thiocarbonyl than with a carbonyl group.[6]

As we will show below, the data on $^{15}$N chemical shifts presented in this paper are in accordance with the general rule that the nitrogen nucleus is deshielded in amides and their derivatives.

The structures of all compounds investigated are shown in Scheme 1. Apart from *N*-alkyl-δ-carbomethoxyvalerothiohydroxamic acids (**2**) and ring-substituted *N*-isopropylthiobenzhydroxamic acids (**3**), for comparison we also recorded and analyzed the spectra of the hydroxylamines **1** and




**Scheme 1**

structurally similar benzhydroxamic acids **4**, benzamides **5** and thiobenzamides **6**. $^{15}$N chemical shifts are listed in Tables 1 and 2.

$^{15}$N spectra of all THAs **2** and **3**, and of benzohydroxamic acids **4**, benzamides **5** and thiobenzamides **6**, consist of single signals, which are the averaged signals of *E*- and *Z*-isomers. As we established by means of a cursory analysis of low-temperature $^1$H spectra, at room temperature the mutual *E*–*Z* isomerization for **4c** is a fast process [acetone-$d_6$, $T_c$ − 12 °C, $\Delta\nu$ = 320 Hz; $\Delta G \approx$ 11.8 kcal mol$^{-1}$ (1 kcal = 4.184 kJ)]. A typical barrier for rotation around the C—N bond in acetothiohydroxamic acids is higher (20.0 kcal mol$^{-1}$, $T_c$ 87 °C).[7] As we predicted, all substituents on the nitrogen in THAs, i.e. alkyl, hydroxyl and thiocarbonyl groups, cause $^{15}$N resonance to occur at the highest frequencies. Thus, for the compounds investigated the $^{15}$N chemical shifts decrease in the order **3** > **4** > **6** > **1** > **5**. By comparison, the respective series of $^1$H chemical shifts (for methine protons) is **6** > **3** > **5** > **4** > **1**.

**Table 1.** $^{15}$N NMR chemical shifts (ppm from external $CH_3NO_2$) for hydroxylamine derivatives **1**–**4** in $CDCl_3$

| No. | $\delta$ $^{15}$N (ppm) | $\Delta\delta$ (ppm) |
|---|---|---|
| **1a** | −252.0 | ↑ |
| **1b** | −234.0 | 21.6 |
| **1c** | −230.4[7] | ↓ |
| **2a** | −178.0 | ↑ |
| **2b** | −150.6 | 34.4 |
| **2c** | −143.6 | ↓ |
| **3a** | −149.2 | ↑ |
| **3b** | −148.0 | |
| **3c** | −147.6 | 2.8 |
| **3d** | −147.3 | |
| **3e** | −147.5 | |
| **3f** | −146.4 | ↓ |
| **4a** | −183.6 | ↑ |
| **4c** | −182.0 | 2.4 |
| **4f** | −181.2 | ↓ |

**Table 2.** $^{15}$N NMR chemical shifts (ppm from external $CH_3NO_2$) for benzamides **5** and thiobenzamides **6** in $CDCl_3$

| No. | $\delta$ $^{15}$N (ppm) | $\Delta\delta$ (ppm) |
|---|---|---|
| **5a** | −250.0 | ↑ |
| **5c** | −248.4 | 4.3 |
| **5f** | −245.7 | ↓ |
| **6a** | −208.3 | ↑ |
| **6c** | −205.1 | 5.9 |
| **6f** | −202.4 | ↓ |

A characteristic feature of the investigated hydroxylamine derivatives is the non-additivity of all substituent increments, which makes it impossible to apply additivity rules for predicting the $^{15}$N chemical shifts. Hence the deshielding effect of an *N*-alkyl substituent in hydroxylamines **1** and THAs **2** proves to be slightly different from that of the corresponding amines.[8] The shift range in the methyl–*tert*-butyl series in amines is 2.5 and 1.5 times smaller than for hydroxylamines **1** and THAs **2**, respectively. Moreover, in THAs **2** the introduction of two methyl groups on the $\alpha$-carbon atom (*i*Pr) results in a 40% increase in the deshielding effect (**2b**), whereas the third methyl group (*tert*-Bu, **2c**) caused a 30% smaller deshielding effect in comparison with other amine derivatives, which allows the application of additivity rules to substituent chemical shifts for alkyl substituents.

We have also observed similar trends for hydroxyl and thiocarbonyl group effects. The deshielding effect caused by the presence of an OH group is ca 10 ppm lower in THAs **3** than the shift difference between benzhydroxamic acids **4** and benzamides **5**. The deshielding effects of thioalkanoyl (RC═S) and thiobenzoyl (PhC═S) groups in **2** and **3** are similar: 83.4 and 86.4 ppm, respectively (Table 3). These effects are larger than that of a benzoyl group in benzohydroxamic acids **4** (PhC═O, $\delta_4 - \delta_1$ = 52 ppm). On the other hand, the difference in $^{15}$N chemical shifts between thiobenzamides **6** and benzamides **5** (43.3 ppm) is ca 10 ppm larger than the shift difference between THAs **3** and benzohydroxamic acids **4** (34.4 ppm.)

The reduction in the deshielding effects of the substituents (C═S and OH) in THAs may be associated with steric crowding around the *N*-alkyl-*N*-hydroxythioamide moiety. However, in our opinion, steric compression contributes dominantly to this reduction only in *N*-*tert*-butyl thiohydroxamic acids. To support this statement, we studied the initial results of the x-ray analysis for **4a** (where the O═CNO torsion angle is ca 5°) and literature data for other

**Table 3.** Deshielding effect ($\Delta\delta = \delta_{2/3} - \delta_1$) of thioaryoyl and thioalkanoyl groups in thiohydroxamic acids **2** and **3**

| *N*-Alkyl series | $EtO_2C(CH_2)_4C$═S in **2** (ppm) | 4-$YC_6H_4C$═S in **3** (ppm) | | |
|---|---|---|---|---|
| | | Y = $OCH_3$ | Y = H | Y = $NO_2$ |
| Methyl | 74.0 | — | — | — |
| Isopropyl | 83.4 | 84.8 | 86.4 | 87.6 |
| *tert*-Butyl | 86.8 | — | — | — |

THAs[9] (torsion angle ca 2°). Based on such evidence, we presume that for investigated *N*-isopropyl THAs **2b** and **3**, the degree of pyramidization of the nitrogen atom due to the fact that its lone electron pair is not coplanar with the thiocarbonyl moiety is relatively low. Hence the lack of additivity of OH and C═S substituent influences results most probably from the competition of both groups for electrons on the nitrogen atom.

Furthermore, characteristic of these compounds, the effects of aryl ring substituents in THAs **3** ($\Delta\delta_{NO_2-OCH_3}$ = 2.8 ppm) and benzohydroxamic acids **4** ($\Delta\delta_{NO_2-OCH_3}$ = 2.4 ppm) are approximately half those for the corresponding thiobenzamides **6** and benzamides **5** (see Table 2). This may also be due to a competitive deshielding inductive effect of the OH group in THAs **3** and benzohydroxamic acids **4** or to a significant reduction in the conjugation of ring substituents with the (thio)carbonyl group (the lack of coplanarity of both moieties) as compared with derivatives **5** and **6**. It should be stated that even for benzamides the twisting of the carbonyl group out of the plane of the aromatic ring is a typical phenomenon. The torsion angles, measured by x-ray analysis, are 28 and 45.8° for benzamide[10] and *N*,*N*-dimethylbenzamide,[11] respectively. The torsion angles Θ that we determined experimentally for benzohydroxamic acids **4** range from 42 to 48°, so they are closer to those of the *N*,*N*-dimethylbenzamides. As can be expected in the presence of sulfur, the actual torsion angle in THAs **3** may undoubtedly be slightly larger, hence the resonance conjugation of substituents with the thiocarbonyl moiety should be smaller. To determine the reason why the chemical shifts of THAs **3** are less sensitive to ring substituents, we plotted the changes in chemical shifts versus the parameters of the Hammett equation. It is known that for systems exhibiting steric inhibition of resonance, the Yukawa–Tsuno equation is recommended:[12]

$$\delta_x = \rho(\sigma^0 + r\Delta\sigma_R^+) \quad (1)$$

where $r = r_{max}\cos^2\Theta$ and $\Delta\sigma_R^+ = \sigma^+ - \sigma^0$.

For the lack of appropriate data, we assumed the torsion angle in THAs **3** to be equal to that in benzohydroxamic acids **4** (Θ = 45°). After applying the Yukawa–Tsuno equation, we obtained a rather poor linear correlation, which was distorted decisively by an electron-donating substituent (**3a**, Y = $OCH_3$):

$$\delta_{^{15}N} = (2.12 \pm 0.20)(\sigma^0 + 0.5\Delta\sigma_R^+) - (147.9 \pm 0.2), \quad R = 0.956 \quad (2)$$

Using Brown $\sigma^+$ parameters, on the other hand, gives a definitely better correlation (Fig. 1):

$$\delta_{^{15}N} = (1.78 \pm 0.15)\sigma^+ - (147.6 \pm 0.2), R = 0.989 \quad (3)$$

At first sight, obtaining good correlation for Brown parameters seems surprising, as they are assumed to reflect full conjugation (Θ = 0°) between the two above-mentioned moieties. Nevertheless, Spychala and Boykin arrived at similar results on analyzing the relationship between $^{17}O$ chemical shift data in *para*-substituted *N*,*N*-dimethylbenzamides and the substituent effects.[13] By finding a linear relationship between $^{15}N$ chemical shifts in THAs **3** and values of the $\sigma^+$ parameter, we have confirmed a theory for which for certain systems the transmission of resonance effects is effective in excited states and not necessarily distorted even by a significant ground-state torsion angle.[14] Hence the observed reduction in ring substituent effects is a result of the inductive effect of the OH group.

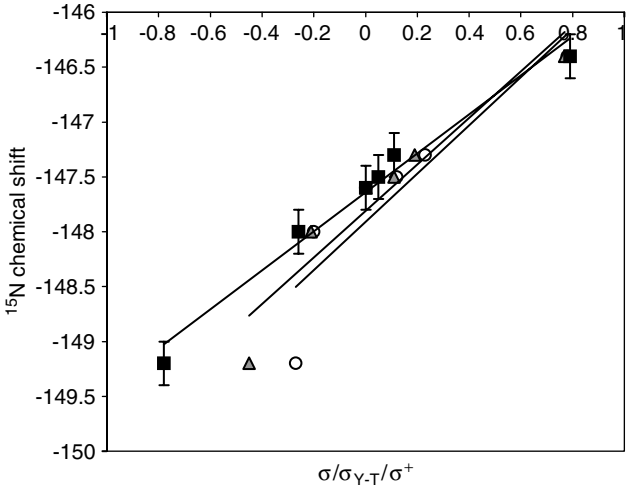



**Figure 1.** Plot of $\delta_{^{15}N}$ for thiohydroxamic acids **3** vs Hammett $\sigma$ (○, $r = 0.903$), Yukawa–Tsuno (▲, $r = 0.956$) and Brown $\sigma^+$ (■, $r = 0.989$) parameters.

As a result of this study, we conclude that (i) $^{15}N$ chemical shift values are similar for aliphatic and aromatic *N*-isopropyl THAs (−148 ± 2 ppm); (ii) a significant deshielding effect of methyl groups on the β-carbon of *N*-alkyl substituent (34.4 ppm) was observed in THAs **2**; (iii) the effect of ring substituents in THAs **3**, similarly to the case of benzohydroxamic acids **4**, is half that in the respective thiobenzamides **6** and benzamides **5**, which results from the inductive effect of the OH group rather than from structure distortion within the $C_{Ar}$—(C═S) bond; (iv) the δ $^{15}N$ correlation with $\sigma^+$ indicates that the conjugation between electron-donating substituents and the thiocarbonyl group is significantly affected by the excited state of THAs **3**.

## EXPERIMENTAL

### Compounds

*N*-Methylhydroxylamine (**1a**) was distilled from a mixture of its hydrochloride and KOH. *N*-Isopropylhydroxylamine (**1b**) was liberated from its hydrogen oxalate salt with saturated $NaHCO_3$ solution and extracted into chloroform-$d_1$. THAs **2** and **3** were obtained by thioacylation of the corresponding hydroxylamines **1** with δ-carbomethoxythiovalericdithiophosphoric anhydride[2] and by thionation of the parent benzohydroxamic acids **4** with Lawesson's reagent,[15] respectively.

*N*-Isopropylbenzohydroxamic acids (**4**) were prepared from the corresponding aroyl chlorides. *N*-Isopropylbenzamides (**5a**,[16] **5c**[15] and **5f**[16]) were obtained by the Schotten–Baumann method using the corresponding aryloyl chlorides. *N*-Isopropylthiobenzamides (**6a**,[17] **6c**[15] and **6f**[18])

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were prepared by thionation of the respective amides **5** using Lawesson reagent.

Ring-substituted THAs **3** and benzohydroxamic acids **4** are new compounds.

*N*-Isopropyl-4-methoxybenzothiohydroxamic acid (**3a**): yield 10%; $\nu_{max}$ ($cm^{-1}$) 3439 (OH), 1222, 1180, 1167; $\delta_H$ 1.37 (6H, d, $CHCH_3$), 3.85 (3H, s, $OCH_3$), 4.53 (1H, spt, $CHCH_3$), 6.92 (2H, d, $J = 9$, H-3/5), 7.34 (2H, d, $J = 9$, H-2/6), 10.7 (1H, br s, OH); $\delta_C$ 19.9 ($CH_3CH$), 55.4 ($CH_3CH$), 55.4 ($OCH_3$), 114.0 (C-3/5), 130.5 (C-1), 128.2 (C-2/6), 160.6 (C-4), 180.8 (C=S); HRMS: calcd for $C_{11}H_{15}NO_2S$, 225.08235; found, 225.08170.

*N*-Isopropyl-4-*tert*-butylbenzothiohydroxamic acid (**3b**): yield 15%; $\delta_H$ 1.34 (9H, s, $CCH_3$), 1.38 (6H, d, $CHCH_3$), 4.51 (1H, spt, $CHCH_3$), 7.30 (2H, d, $J = 9$, H-2/6), 7.42 (2H, d, $J = 9$, H-3/5), 9.0 (1H, br s, OH); $\delta_C$ 20.2 ($CH_3CH$), 31.4 ($CCH_3$), 35.0 ($CCH_3$), 55.6 ($CH_3CH$), 125.9 and 126.5 (C-2/6 and C-3/5), 135.5 (C-1), 153.2 (C-4), 181.0 (C=S). HRMS: calcd for $C_{14}H_{21}NOS$, 251.13439; found, 251.13371.

*N*-Isopropyl-4-chlorobenzothiohydroxamic acid (**3d**):- yield 37%; m.p. 104–109 °C; $\delta_H$ 1.37 (6H, d, $CHCH_3$), 4.41 (1H, spt, $CHCH_3$), 7.26–7.47 (4H, 2 × d, $J = 9$, Ar-H), 9.8 (1H, br s, OH); $\delta_C$ 19.9 ($CH_3CH$), 55.6 ($CH_3CH$), 127.8 and 129.0 (C-2/6 and C-3/5), 131.4 (C-1), 136.3 (C-4), 179.2 (C=S). HRMS: calcd for $C_{10}H_{12}NOS^{35}Cl$, 229.03281; found, 229.03224.

*N*-Isopropyl-3-methoxybenzothiohydroxamic acid (**3e**): yield 28%; $\delta_H$ 1.36 (6H, d, $CHCH_3$), 4.45 (1H, spt, $CHCH_3$), 6.89 (1H, s, H-2), 6.90 (2H, d, $J = 9$, H-4 and H-6), 7.30 (1H, t, $J = 9$, H-5), 10.8 (1H, br s, OH); $\delta_C$ 20.2 ($CH_3CH$), 55.6 ($CH_3CH$), 55.8 ($OCH_3$), 112.3 (C-2), 115.6 (C-6), 118.7 (C-6), 130.1 (C-5), 139.6 (C-1), 159.9 (C-3) 180.3 (C=S). HRMS: calcd for $C_{11}H_{15}NO_2S$, 225.08235; found, 225.08219.

*N*-Isopropyl-4-nitrobenzothiohydroxamic acid (**3f**): yield 38%; m.p. 153–155 °C; $\delta_H$ 1.40 (6H, d, $CHCH_3$), 4.29 (1H, spt, $CHCH_3$), 7.54 (2H, d, $J = 9$, H-2/6), 8.28 (2H, d, $J = 9$, H-3/5), 10.8 (1H, br s, OH); $\delta_C$ 19.9 ($CH_3CH$), 56.0 ($CH_3CH$), 124.1 (C-3/5), 127.5 (C-2/6), 144.0 (C-1), 148.1 (C-4), 177.7 (C=S). HRMS: calcd for $C_{10}H_{12}N_2O_3S$, 240.05686; found, 240.05717.

*N*-Isopropyl-4-methoxybenzohydroxamic acid (**4a**): yield 70%; m.p. 121–122 °C (ethyl acetate); $\delta_H$ 1.29 (6H, d, $CHCH_3$), 3.84 (3H, s, $OCH_3$), 4.26 (1H, spt, $CHCH_3$), 6.93 (2H, d, $J = 9$, H-3/5), 7.48 (2H, d, J = 9, H-2/6), 8.32 (1H, br s, OH); $\delta_C$ 19.0 ($CH_3CH$), 52.0 ($CH_3CH$), 54.7 ($OCH_3$), 113.2 (C-3/5), 124.3 (C-1), 128.8 (C-2/6), 160.9 (C-4), 166.6 (C=O). Elemental analysis: calcd for $C_{11}H_{15}NO_3$, C 63.14, H 7.23, N 6.69; found, C 63.41, H 7.48, N 6.69%.

*N*-Isopropyl-4-nitrobenzohydroxamic acid (**4f**): yield 60%; m.p. 107–110 °C (benzene–cyclohexane); $\delta_H$ 1.32 (6H, d, $CHCH_3$), 4.12 (1H, spt, $CHCH_3$), 7.67 (2H, d, $J = 9$, H-2/6), 8.30 (2H, d, $J = 9$, H-3/5), 8.25 (1H, br s, OH); $\delta_C$ 19.6 ($CH_3CH$), 52.4 ($CH_3CH$), 123.8 (C-3/5), 128.6 (C-2/6), 138.9 (C-1), 148.9 (C-4), 164.4 (C=O). Elemental analysis: calcd for $C_{10}H_{12}N_2O_4$, C 53.57, H 5.39, N 12.49; found, C 53.55, H 5.34, N 12.30%.

## NMR spectra

All spectra were recorded on a Varian Unity 500 Plus spectrometer operating at 500 MHz ($^1H$), 125.7 MHz ($^{13}C$) and 50.7 MHz ($^{15}N$) in deuterochloroform. $^{15}N$ spectra were measured in $CDCl_3$ solutions at 0.14 M concentration in standard NMR tubes (5 mm o.d.). Chemical shifts are referenced to external $CH_3NO_2$.

$^{15}N$ long-range gHMQC spectra were acquired with pulse field gradients in absolute value mode. The spectral windows for $^1H$ and $^{15}N$ domains were 6643 and 14 690 Hz, respectively. The multiple-bond delay was set to 90 ms. The data were collected in a 2048 × 150 matrix with 24 transients per $t_1$ increment. The recycle period was 1.4 s. Sine-bell window functions were applied before Fourier transformation in a 2K × 1K matrix.

Chosen 2-D spectra slices were subjected to inverse Fourier transformation (FT), zero-filling to 4K real points and repeated FT of the resulting 1-D spectra in a digital resolution of 2.5–3.5 Hz per point.

Standard procedures of the spectrometer software (VNMR 5.1) were used. A comparison was made between the results of the analysis for a central slice cutting the center of each correlation peak and two additional neighboring slices which gave the accuracy of ±0.1 ppm. The reproducibility of the $^{15}N$ chemical shifts values for three spectra recorded at different times of each compound was 0.3–0.4 ppm.

## Acknowledgements

Financial support was provided by the Polish State Committee for Scientific Research (KBN) (grant No. T09A 06116). We thank Dr P. Sowiński for his help in measuring the NMR spectra.

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