Benzhydroximic acids — NMR study of trimethylsilyl derivatives

Jan Schraml [a],*, Magdalena Kvíčalová [a], Ludmila Soukupová [a], Vratislav Blechta [a], Otto Exner [b]

[a] *Institute of Chemical Process Fundamentals, Academy of Sciences of the Czech Republic, 16502 Prague 6, Czech Republic*
[b] *Institute of Organic Chemistry and Biochemistry, Academy of Sciences of the Czech Republic, 16610 Prague 6, Czech Republic*

Dedicated to Professor Stanisław Pasynkiewicz on the occasion of his 70th birthday in recognition of his contribution to organometallic chemistry.

Abstract

NMR spectra ([1]H ^{13}C, ^{15}N, and [29]Si) of trimethylsilylated *para* and *meta* substituted benzhydroxamic acids were studied in chloroform solutions. The silylation products have the structure of Z-O,O'-bis(trimethylsilyl) derivatives of benzhydroximic acid, independently of the ring substituent. According to aromatic proton chemical shifts, the geometry of the hydroximic group and its torsion angle with the ring plane are not affected by the *para* substituent. The chemical shifts of the nuclei in the hydroximic part of the molecule show surprisingly strong dependence on the remote ring substituent. The two [29]Si chemical shifts exhibit essentially the same sensitivity to substitution despite the fact that the $Si(O^1)$ is one bond closer to the substituent than the $Si(O^4)$ silicon. It is suggested that while electron donor substituents increase the shielding of the silicon atoms they also increase the basicity of the oxygen in the Si–O moiety, thus leading to the stronger hydrogen bonding with the solvent. Association with chloroform partially compensates the direct substituent effect on the shielding in the case of $Si(O^1)$ silicon. The influence of other factors not covered by substituent constants is demonstrated by excellent correlations with the chemical shifts in analogous *tert*-butyldimethylsilyl derivatives. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Trimethylsilyl derivatives; Substituted benzhydroximic acids; NMR chemical shifts; Hammett correlations

1. Introduction

We have recently shown that silylation (either trimethysilylation or *tert*-butyldimethylsilylation) of benzhydroxamic acid solely yields bis-silyl derivatives of the tautomeric benzhydroximic acid with the Z configuration [1]. A subsequent study [2] of 12 *para* and *meta* substituted benzhydroxamic acids has shown that the substituent, which in other respects profoundly affects the structure of the acids [3–6], in the crystal, does not influence the structure of the product of *tert*-butyldimethylsilylation in solution to the extent noticeable by NMR spectroscopy. Upon *tert*-butyldimethylsilylation all the substituted benzhydroxamic acids yielded Z-O^1,O^4-bis(*tert*-butyldimethylsilyl) derivatives (**1**, Scheme 1, R = C(CH₃)₃) of the corre-

sponding benzhydroximic acids, as proven by their [29]Si, ^{15}N, [13]C=N chemical shifts and $^1J(^{13}C-^{13}CN)$ coupling constants.

While the stable *tert*-butyldimethylsilyl (TBDMS) derivatives **1** permit time-consuming measurements necessary for their structure determination, data on the less-stable trimethylsilyl (TMS) derivatives (**2**, Scheme 1, R = CH₃) are needed for a comparison with other series of compounds. Also interesting is a quantitative comparison of the corresponding atoms in the two
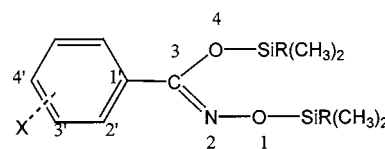
Scheme 1. Structure and atom numbering of fully silylated Z-benzhydroximic acids (in TMS derivatives, **2**, R = CH₃; in TBDMS derivatives, **1**, R = C(CH₃)₃).

* Corresponding author. Fax: +42-02-20920661.
E-mail address: schraml@icpf.cas.cz (J. Schraml)

series **1** and **2**; for series so similar one expects close correlations with the slopes near unity. In the present paper, we report on the results of a limited study of TMS derivatives **2**.

2. Experimental

The substituted benzhydroxamic acids were prepared by standard procedures as described elsewhere [3,7]. TMS derivatives were prepared from the parent acids by silylation using (trimethylsilyl)dimethylamine (TMS-DMA) in 1.2 molar excess. The reaction mixture was heated (60°C) and stirred for 2–5 h. After completion, the remaining reagent was removed by distillation under reduced pressure. The isolated compounds were identified and checked by ^{1}H- and ^{13}C-NMR spectroscopy. (^{1}H-NMR spectra were analyzed in terms of coupling constant and chemical shift values of aromatic protons only in the case of *para* substituted compounds; in the other cases the splitting pattern was used to check the position of the substituent.)

The NMR spectra were measured in dry $CDCl_3$ solutions containing 1% (v/v) of hexamethyldisilane (HMDSS) as a secondary reference. The reported ^{13}C and [29]Si chemical shifts were obtained from dilute solutions. The sample concentration was reduced until the ^{13}C chemical shift of HMDSS was $\delta = -2.48 \pm 0.02$, relative to the central line of the solvent at 76.99 ppm (see Ref. [8] for details of this standard procedure). High sample concentrations (ca. 33% v/v) were used in ^{15}N-NMR and INADEQUATE measurements. ^{15}N-NMR chemical shifts are referenced externally to CH_3NO_2 (50%, w/w) in $CDCl_3$.

^{1}H-, ^{13}C-, and ^{29}Si-NMR spectral measurements were performed on a Varian UNITY-200 spectrometer (operating at 200.04 MHz for ^{1}H, at 50.3 MHz for ^{13}C and at 39.7 MHz for ^{29}Si-NMR measurements) and ^{15}N spectra were measured on a Varian UNITY 500 spectrometer (at 50.667 MHz). In all cases the standard software (APT, INADEQUATE, and INEPT pulse sequences) was used. The spectra were recorded in the temperature range 22–24°C. The ^{29}Si-NMR spectra were measured by INEPT with the pulse sequence optimized [8] for TMS derivatives, i.e. for coupling to nine protons and the coupling constant of 6.5 Hz. Acquisition (2.0 s) was followed by a relaxation delay of 5 s. During the acquisition period, WALTZ decoupling was used and FID data (16 K) were sampled for the spectral width of 4000 Hz. Zero filling to 32 K and a mild exponential broadening were used in data processing. The [29]Si $\pi/2$ pulses were at maximum 17 μs long, whereas [1]H $\pi/2$ pulses were 12 μs in a 5 mm switchable probe. The [29]Si spectra were referenced to the line of HMDSS at $\delta = -19.79$. The ^{13}C-NMR spectra were measured using the spectral width of

16 000 Hz. WALTZ decoupling was applied both during acquisition (1 s) and relaxation delay (2–5 s). Zero filling to 64 K and 1–3 Hz line broadening were used in data processing.

Aromatic carbon chemical shifts were assigned by combining additive increments (using the values from Ref. [9]) and the shifts assigned for the parent Z-O-trimethylsilyl trimethylsilylbenzhydroximate through a 2D INADEQUATE experiment [10].

When a fluorine-containing substituent was present (4-F, 4-CF_3), the [13]C–[19]F couplings were used to check the assignment of aromatic carbon lines.

In contrast to [19]F–[15]N coupling across six bonds observed earlier in the ^{15}N-NMR spectrum of Z-O[1],O[4]-bis(*tert*-butyldimethylsilyl) 4-fluorobenzhydroximic acid [2], no such coupling was detected ($J < 0.5$ Hz) in its TMS analogue. Apparently, crowding at the hydroximic end of the molecule plays some role; such effects were noticed in other through-space [15]N–[19]F couplings [11].

Solvent accessible surface (A) [12] was calculated as described elsewhere [13].

3. Results and discussion

The assigned chemical shifts and coupling constants of the studied compounds are summarized in Tables 1 and 2. Since the chemical shifts (^{13}C, ^{15}N, and [29]Si) correlate well with substituent constants, we will briefly compare these correlations with those found in related classes of compounds also bearing TMS substituents.

In all the studied ring-substituted benzhydroxamic acids, trimethylsilylation yields only one product (using [29]Si INEPT spectra of concentrated solutions with excellent S/N it can be estimated that if any other trimethylsilylated product were formed, it would constitute only less than 2% of the main product). This is in agreement with the earlier findings on trimethylsilylation [16] or *tert*-butyldimethylsilylation of benzhydroxamic acid [2]. Using the values of one-bond [13]C–[13]C couplings ([13]C–[13]CN), we have shown [1] that the sole product of trimethylsilylation of the parent benzhydroxamic acid (R = H) is not an exchange-averaged mixture of *E*- and *Z*-isomers, as suggested by Rigaudy et al. [16], but only the *Z*-trimethylsilyl ester of *N*-(trimethylsiloxy)benzoimidic acid (**2**). Since the chemical shifts in the studied series of TMS derivatives of the ring-substituted benzhydroxamic acids follow some obvious and regular dependences on Hammett constants of the ring substituents, and the parent compound fits these dependences well, one can safely assume that all the studied compounds have the same structure **2**. Similarly, we extend the assignment of [29]Si lines in **1** (as derived from model compounds in Ref. [1]) to all compounds **2**.

Table 1

^{29}Si-, ^{15}N- and ^{13}C-NMR chemical shifts (ppm) in Z-R–C$_6$H$_4$–C(O^{4}SiMe$_3$)=N–O^1–SiMe$_3$ derivatives [a]

R	σ [b]	$\delta(^{29}$Si) Si-1	Si-4	$\delta(^{15}$N) [c]	$\delta(^{13}$C) C=N	C-1	C-2	C-3	C-4	C-5	C-6	CH$_3$Si	Other
Parasubstituted													
N(CH$_3$)$_2$	−0.83	24.48	20.3	−84.9 [d]	154.38	120.47	127.37	111.43	151.41	111.43	127.37	1.47/−0.55	40.31
NHSiMe$_3$	−0.66 [e]	24.62	20.41 [f]	−84.7 [g]	154.21	121.87	127.59	115.42	149.04	115.42	127.59	1.51/−0.56	−0.03
OCH$_3$	−0.27	25.24	21.00	−82.4	153.74	125.35	127.72	113.40	160.82	113.40	127.72	1.52/−0.57	55.28
CF$_3$	0.54	26.84	22.51	−74.80	152.76	136.32	126.45	124.97 [h]	131.06 [i]	124.97 [h]	126.45	1.55/−0.61	124.18 [j]
NO$_2$	0.78	27.66	23.26	−71.3 [k]	152.26	139.03	126.92	123.33	148.44	123.33	126.92	1.61/−0.63	
H	0.00	25.76	21.38	−78.6	153.82	132.80	126.22	128.02	129.58	128.02	126.22	1.55/−0.57	
CH$_3$	−0.17	25.44	21.10	−80.0	153.96	130.01	126.17	128.74	139.63	128.74	126.17	1.50/−0.57	21.35
F	0.06	25.95	21.72	−80.3	153.07	128.90 [l]	128.17 [m]	114.99 [n]	163.82 [c,o]	114.99 [n]	128.17 [m]	1.57/−0.59	
Cl	0.23	26.24	21.97	−78.1	153.05	131.36	127.52	128.25	135.55	128.25	127.52	1.56/−0.59	
Metasubstituted													
NO$_2$	0.71	27.39	23.33	−75.3 [p]	152.00	134.81	121.19	148.25	124.14	129.04	131.90	1.61/−0.61	
Cl	0.37	26.51	22.25	−77.0	152.73	134.05 [q]	126.26	134.70 [q]	129.57 [q]	129.31 [q]	124.34	1.57/−0.60	
OCH$_3$	0.12	25.86	21.49	−77.9	153.69	134.22	111.65	159.29	115.26	129.06	118.78	1.51/−0.58	55.20
Orthosubstituted													
OSiMe$_3$		24.73	19.94 [r]	−77.2	154.48 [q]	125.64	153.57 [q]	120.20 [q]	130.80 [s]	120.96 [q]	130.24 [s]	1.91/0.51	−0.41

[a] Unless otherwise indicated the values are from diluted solutions.
[b] Values of Hammett σ constants taken from Ref. [14].
[c] Value(s) from concentrated solution(s).
[d] N(CH$_3$)$_2$ at −330.
[e] Value for NH$_2$ group.
[f] SiNH at 3.57.
[g] NH at −308.7 (J(NH) = 76.5 Hz).
[h] J(FC) = 3.9 Hz.
[i] J(FC) = 32.7 Hz.
[j] J(FC) = 272.0 Hz.
[k] NO$_2$ at −10.1.
[l] J(FC) = 2.8 Hz.
[m] J(FC) = 8.3 Hz.
[n] J(FC) = 22.0 Hz.
[o] J(FC) = 249.1 Hz.
[p] NO$_2$ at −9.9.
[q] Assignments in the row can be interchanged.
[r] Si at 19.86, the two lines cannot be assigned.
[s] Assignments in the row can be interchanged.

Table 2
^{1}H-NMR data and substituent induced chemical shifts (ppm) of aromatic protons in *para* substituted Z-4R–C$_6$H$_4$–C(O^{4}SiMe$_3$)=N–O^1–SiMe$_3$ derivatives [a]

R	$\delta(^1$H)		$J(2,3)$ [b]	SCS [c]	
	H-2′	H-3′		Ortho	Meta
N(CH$_3$)$_2$	7.6625	6.6555	9.0	−0.66	−0.18
NHSiMe$_3$	7.577	6.597	8.8	[d]	[d]
OCH$_3$	7.7275	6.860	8.9	−0.48	−0.09
CF$_3$	7.9005	7.593	8.3	0.32	0.14
NO$_2$	8.1925	7.9485	9.0	0.95	0.26
H	7.798	7.345	8.2	0.00	0.00
CH$_3$	7.678	7.1415	8.4	−0.20	−0.12
F	7.7735	7.018	8.5	−0.26	0.00
Cl	7.721	7.304	8.7	0.03	−0.02

[a] The values are from diluted solutions, ^{1}H chemical shifts in δ scale, coupling constants in Hz units; H-2′ and H-3′ denote protons *ortho* and *meta* to the hydroximic group, respectively.

[b] Values of coupling constants between protons H-2′ and H-3′.

[c] Values of substituent induced chemical shifts, SCS, taken from Ref. [15].

[d] Not available.

These 'empirical' assignments are supported by the correlations of chemical shifts in **2** with those in the TBDMS analogues **1** in which the Z configuration was confirmed by the measurements of $^1J(^{13}C-^{13}CN)$ coupling constants in all the compounds of the series [2]. These correlations of chemical shifts in **1** against those in **2** are extremely good, having slopes (b) close to unity and the standard errors (SD) of the predicted chemical shift in **2** do not appreciably exceed the experimental errors (12 data points in all cases; Si-1: $R = 0.9999$, $b = 0.932 \pm 0.004$, SD = 0.013; Si-4: $R = 0.9996$, $b = 1.023 \pm 0.009$, SD = 0.030 ppm; ^{15}N: $R = 0.9953$, $b = 1.01 \pm 0.03$, SD = 0.4 ppm; ^{13}C(C=N): $R = 0.9988$, $b = 0.905 \pm 0.014$, SD = 0.035 ppm). The slope values in ^{29}Si correlations are similar to those found in other pairs of TBDMS and TMS derivatives with Si–O bonds (e.g. in silylated alcohols 0.990 [17] or amino acids 0.860 [18]). The quality of these correlations allowed us to identify the deviating points as those of silicon atoms in different moieties [19]; no such significant deviations have been found in the series studied here. The quality of these chemical shift–chemical shift correlations indicates that the ring substituents control the shifts in question by the same mechanisms in these two closely related series of compounds, and that if the bulky TBDMS group exercises some steric effects they are constant within the series of **1**. In contrast, the somewhat worse Hammett-type correlations discussed below indicate that Hammett constants do not cover all the factors involved.

Further support for the suggested structure and detailed information can be drawn from aromatic proton chemical shifts. The protons *ortho* to the hydroximic group (H-2′ in Table 2) exhibit a significant linear correlation with the substituent chemical shifts (SCS) induced by the substituents in monosubstituted benzenes [15], $\delta(H-2′) = 7.81 + 1.15*SCS_{meta}$, $R = 0.947$, $n = 8$; the slope does not differ significantly (on 90% significance level) from unity. Following the reasoning of Holík and Matějková [20], we can conclude that the geometry of hydroximic group and its torsion angle with a benzene ring do not change under the influence of the substituent.

The substituents exercise very 'normal' effects on the electronic structure, as manifested by Hammett-type correlations of NMR chemical shifts, and do not have such dramatic effects on the structure of Z-O,O'-bis(trimethylsilyl) hydroximic acids, as reported for the parent hydroxamic acids [6] and their alkali metal salts [4]. Thus, in *para* substituted compounds **2** the ^{13}C chemical shifts of C-1 carbons exhibit Hammett-type correlation with the same slope ($b = 11.55$, $R = 0.9684$, SE = 1.66, $n = 9$), as found in monosubstituted benzenes for the *para* carbons ($b = 11.5$ [9]). The negative slope in the correlation for the C=N carbon (*meta* and *para* derivatives combined $b = -1.46$; *para* only $b = -1.31$) was not unexpected. The negative slope was already discussed [2] in connection with compounds **1** where it was somewhat smaller in absolute value ($b = -1.30$ and 1.14, respectively). In both series **1** and **2** the slopes are smaller than in methyl benzoates (-1.907 and -2.408, respectively) [21], benzonitriles (-2.66 and -2.39, respectively) [22], and N,N-dimethylbenzamides (-2.33 *para* only) [23]. Considering the polarizability of the involved groups and the properties of O–Si bonds, the low values of the slopes in **1** and **2** may be accounted for by the proposed model in which the double C=N bond behaves as an isolated unit polarized by the distant substituent [2].

The two silicon-29 chemical shifts in **2** correlate linearly with Hammett substituent constants; surprisingly, the slopes have the same value (1.98 ± 0.11 and 1.95 ± 0.15 for Si-1 and Si-4, respectively). As follows from the above-mentioned correlations between TMS and TBDMS derivatives (with the slopes close to unity), the slopes do not differ significantly from those found in Hammett correlations for bulkier TBDMS derivatives (1.82 ± 0.09 and 1.96 ± 0.13 for Si-1 and Si-4, respectively) [2]. This sensitivity of silicon shielding to substituent effects is lower than that reported for ring-substituted phenoxytrimethylsilanes (4.93 ± 0.85 in CCl$_4$ and 4.77 ± 0.94 in neat liquids) [24]. While this might seem to be an obvious example of the role of substituent distance, a part of this reduced sensitivity is due to different experimental conditions. The compounds studied here were measured in dilute chloroform solutions where hydrogen bonding reduces the sensitivity of the silicon to substituent effects (as elec-

Table 3
Statistics of the correlations of substituent-induced $\delta(^{15}N)$ shifts in different series of compounds

	Compound series [a]	Explanatory variables [b]	Regression coefficients [c]	SD [d]	R [e]	N [f]
1	R–CO–NH–OH [g]	$\sigma_{m,p}$	3.22(26)	0.43	0.969	12
2	R–CO–NH$_2$ [h]	$\sigma_{m,p}$	3.79(39)	0.54	0.951	12
3	R–C(OT)=N–OT [i]	$\sigma_{m,p}$	7.89(63)	1.05	0.969	12
4	R–C(OT)=N–OT [j]	$\sigma_{m,p}$	7.73(65)	1.09	0.966	12
5	R–CH=N–OH [k]	$\sigma_{m,p}$	15.14(1.11)	1.34	0.987	7
6	R–C≡N [l]	$\sigma_{m,p}$	10.46(55)	0.53	0.993	7

[a] $R = X–C_6H_4$.
[b] Values of substituent constants taken from Ref. [14].
[c] Standard deviation in parentheses.
[d] Standard deviation from the regression.
[e] Correlation coefficient.
[f] Number of observations.
[g] Benzhydroxamic acids, Ref. [7].
[h] Benzamides, Ref. [25].
[i] Z-O^1,O^4-bis(*tert*-butyldimethysilyl) benzhydroximic acids, $T = Si(CH_3)_2C(CH_3)_3$, Ref. [2].
[j] Z-O^1,O^4-bis(trimethysilyl) benzhydroximic acids, $T = Si(CH_3)_3$, this work.
[k] Benzaldoximes, Ref. [26].
[l] Benzonitriles, Ref. [25].

tron donors increase the shielding of the silicon they also increase the basicity of the Si–O oxygen, and hence enhance H-bonding which in turn decreases the shielding [13]). Some combinations of these two mechanisms also contribute to the observed equal sensitivity of Si-1 and Si-4 to substitution. However, the interplay of the above mechanisms is not obvious as the calculated solvent accessible surface (A) of the O^1 oxygen is smaller than that of O^4 both in TMS and in the bulkier TBDMS derivatives (the actual numerical values strongly depend on the conformation).

The ^{15}N chemical shifts exhibit similarly good linear correlations with substituent constants. Since such correlations have not been recently reviewed, some ^{15}N correlations for closely related series of compounds are presented in Table 3. Again, correlations with Hammett constants have the same slopes in TMS and TBDMS [2] derivatives of benzhydroximic acids; their slopes are intermediate between those in benzhydroxamic acids [7] or benzamides [25] and those in benzonitriles [26]. This observation is certainly in accord with the bond order of the C=N bond in hydroximic derivatives as intermediate between those in amides and nitriles. Nevertheless, such a picture would be oversimplifying. Comparison with benzaldoximes [26] and benzamides [25] indicates that the value of the slope is not simply related to the C–N bond order; polarizability of the other substituents obviously plays a significant role here also. Attempts to analyze the results of Table 2 in terms of the dual substituent parameter (DSP) treatment using σ_F and σ_R were successful only in some cases (RCONHOH, RCONH$_2$). The correlations were better than with $\sigma_{m,p}$, but the improvement was of little significance.

4. Conclusions

Trimethylsilylation of ring-substituted benzhydroxamic acids produces solely *syn* (Z) O,O'-disilylated derivatives of benzhydroximic acid. The hydroximic structure follows directly from ^{29}Si and ^{15}N chemical shifts; the Z configuration follows from one-bond ^{13}C–^{13}C couplings of $^{13}C=N$ carbon in the parent compound and from comparison with analogous TBDMS derivatives. The geometry of the hydroximic group does not change with the ring substituent.

Chemical shifts of various nuclei in the hydroximic moiety of TMS and TBDMS derivatives exhibit excellent mutual correlations and also good Hammett-type correlations. Notably, the dependences of S(O^4) and Si(O^1) show the same sensitivity to substituent effects despite the latter being one bond closer to the substituent; the sensitivity is lower than that in phenoxytrimethylsilanes. It is suggested that hydrogen bonding with the solvent plays a certain role in this observation. In accord with the hydroximic structure, the sensitivity of ^{15}N chemical shifts falls between that in amides and nitriles.

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