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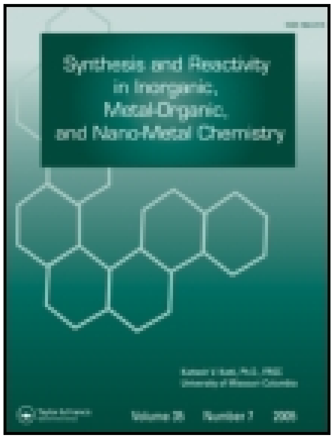

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SYNTHESIS, REACTIONS, AND SPECTRAL [NMR (^{1}H, ^{13}C, ^{29}Si), IR] STUDIES OF TRIMETHYLSILYL-SUBSTITUTED N-ARYLSALICYLALDIMINATES

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SYNTHESIS, REACTIONS, AND SPECTRAL [NMR (^1H, ^{13}C, ^{29}Si), IR] STUDIES OF TRIMETHYLSILYL-SUBSTITUTED N-ARYLSALICYLALDIMINATES

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ABSTRACT

Trimethylsilyl-substituted N-arylsalicylaldiminates $Me_3Si(OC_6H_4CH{=}NAr)$ [$Ar{=}C_6H_5$ (**1**), $C_6H_3Me_2$-2,6 (**2**), $C_6H_2Me_3$-2,4,6 (**3**), $C_6H_3Et_2$-2,6 (**4**), and C_6H_3(Pr-*i*)$_2$-2,6 (**5**)] have been prepared by the equimolar reactions of Me_3SiCl with N-arylsalicylaldimines (LH) in benzene using Et_3N as a base. Reactions in the desired molar ratios (1:1 or 2:1) of $Me_3Si(OC_6H_4CH{=}NAr)$ with $TiCl_4$ and $ZrCl_4$ afford a convenient and clean synthetic procedure for the preparation of chloride-salicylaldiminate derivatives of early transition metals. Characterisations of all these new derivatives have been carried out by elemental analyses, molecular weight determinations and spectroscopic [IR, NMR (^1H, ^{13}C and ^{29}Si)] studies.

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INTRODUCTION

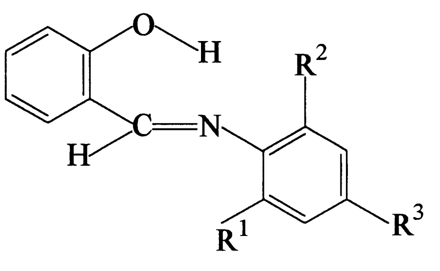
The chemistry of metal-organic compounds containing alkoxo and/or phenoxo functionalities[1,2] has grown considerably in the last thirty years. These compounds are of great interest due to their potential applications as precursors for advanced materials in different areas of science and technology such as ceramics and glasses, as well as catalysis. We have previously described the preparation and spectral properties of some silicon and organosilicon aryloxides[3,4] with the aim to examine the utility of (i) NMR data in explaining the variation in the electron density around the silicon atom with the number of methyl and/or aryloxide groups attached to silicon, and (ii) trimethylsilyl aryloxides in providing neat and high yield synthesis of mixed chloride-aryloxide derivatives of titanium(IV) and niobium(V). With similar objectives in mind, we report herein the synthesis, chemistry and spectroscopy of trimethylsilyl-substituted N-arylsalicylaldiminates **(1)**–**(5)**.

The present study assumes further importance due to the fact that although there have been a number of reports[5,6,7] during the past 13 years of compounds that are similar to those described herein, a systematic and detailed study of trimethylsilicon derivatives of monofunctional bidentate salicylaldimines of the type shown in Fig. 1 does not appear to have carried out so far.

RESULTS AND DISCUSSION

Equimolar reactions of Me_3SiCl with N-arylsalicylaldimines (LH) in benzene in the presence of Et_3N as a base afford trimethylsilyl-substituted N-aryl-salicylaldiminates, Me_3SiL [L = L^1 **(1)**, L^2 **(2)**, L^3 **(3)**, L^4 **(4)**, and L^5 **(5)**], by the replacement of the phenolic proton of ligands shown in Fig. 1 by Me_3Si group.

The derivatives **(1)**–**(5)** (Table I) are yellow or orange volatile liquids, soluble in common organic solvents (benzene, carbon tetrachloride,



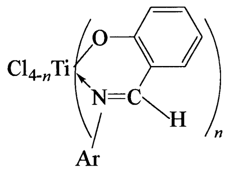
	R^1	R^2	R^3
(L^1 H) :	H	H	H
(L^2 H) :	Me	Me	H
(L^3 H) :	Me	Me	Me
(L^4 H) :	Et	Et	H
(L^5 H) :	Pr-*i*	Pr-*i*	H

Figure 1. Structure of the ligands (LH).

dichloromethane, toluene, *n*-hexane), and show monomeric behaviour (ebullioscopically) in benzene.

The reactions of **(3)** and **(5)** with $TiCl_4$ in 2:1 and 1:1 molar ratios in *n*-hexane provide a clean and convenient procedure for the synthesis of mono- and bis-aldiminate derivatives of titanium(IV) Eq. (1).

$$TiCl_4 + n\ Me_3SiOC_6H_4CH{=}NAr \xrightarrow{n\text{-hexane}}$$



$$+\ n\ Me_3SiCl\uparrow$$

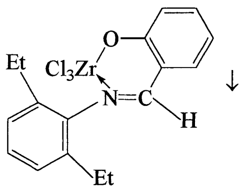
(6): Ar = $C_6H_2Me_3$-2,4,6 ($n = 2$)

(7): Ar = $C_6H_3(Pr\text{-}i)_2$-2,6 ($n = 1$)

(1)

In contrast, similar reactions with $ZrCl_4$ in benzene produced only the mono-substituted product according to the reaction (eq. 2) illustrated below, wherein unreacted $Me_3SiOC_6H_4CH{=}NC_6H_3Et_2$-2,6 (b.p. 175/0.2 mm) was removed by distillation.

$$ZrCl_4 + 2Me_3SiOC_6H_4CH{=}NC_6H_3Et_2\text{-}2,6 \xrightarrow{\text{benzene}}$$



(8)

$$\downarrow +\ Me_3SiOC_6H_4CH{=}NC_6H_3Et_2\text{-}2,6 + Me_3SiCl\uparrow$$

(2)

The derivatives **(6)** and **(7)** are red, moisture-sensitive solids, soluble in organic solvents (C_6H_6, $C_6H_5CH_3$, $CHCl_3$), and show monomeric behaviour (ebullioscopically) in benzene. The derivative **(8)** is a yellow solid which is insoluble in benzene but moderately soluble in chloroform and dichloromethane.

Table I. Preparative and Analytical Data for Trimethylsilyl-Substituted N-arylsalicylaldiminates

Reactants (g, mmol)			Product Empirical Formula Yield[a] (g , %) Colour, State and Boiling Point (°C/mm)	Analysis (%) Found (Calcd.)				M. wt. Found (Calcd.)
Me_3SiCl	Schiff Base	Et_3N		C	H	N	Si	
(2.29, 21.24)	L^1 H (4.17, 21.16)	(2.20, 21.75)	$Me_3SiOC_6H_4CH{=}NC_6H_5$ (**1**) $C_{16}H_{19}NOSi$ (5.25 g , 92%) Yellow liquid (110/0.05)	71.3 (71.4)	7.05 (7.11)	5.11 (5.20)	10.3 (10.4)	275 (269)
(3.31, 30.53)	L^2 H (6.37, 30.76)	(3.11, 30.76)	$Me_3SiOC_6H_4CH{=}NC_6H_3$ Me_2-2,6 (**2**) $C_{18}H_{23}NOSi$ (8.60 g , 94%) Orange liquid (160/0.2)	72.7 (72.8)	7.85 (7.80)	4.68 (4.70)	9.40 (9.43)	303 (297)
(1.33, 12.32)	L^3 H (2.90, 12.15)	(1.25, 12.41)	$Me_3SiOC_6H_4CH{=}NC_6H_2$ Me_3-2,4,6 (**3**) $C_{19}H_{25}NOSi$ (3.41 g , 90%) Yellow liquid (130/0.05)	73.2 (73.4)	8.00 (8.10)	4.46 (4.50)	8.88 (9.02)	310 (311)
(2.77, 25.49)	L^4 H (6.47, 25.25)	(2.65, 25.23)	$Me_3SiOC_6H_4CH{=}NC_6H_3$-$Et_2$-2,6 (**4**) $C_{20}H_{27}NOSi$ (7.78 g , 93%) Orange liquid (175/0.2)	73.8 (73.9)	8.20 (8.37)	4.25 (4.30)	8.59 (8.62)	329 (325)
(1.09, 10.08)	L^5 H (2.83, 10.09)	(1.04, 10.35)	$Me_3SiOC_6H_4CH{=}NC_6H_3$ $(Pr\text{-}i)_2$-2,6 (**5**) $C_{22}H_{31}NOSi$ (3.37 g, 94%) Light yellow liquid (135/0.05)	74.7 (74.9)	8.78 (8.85)	3.89 (3.96)	7.77 (7.95)	350 (353)

[a] Corresponds to distilled product.

IR Spectra

The IR spectra (Table II) of **(1)**–**(5)** exhibit (i) the absence of -OH absorption in the range 3500–3150 cm^{-1}, (ii) the presence of ν(C=N) at 1616–1632 cm^{-1}, (iii) ν(C-O) at 1280–1300 cm^{-1}, which shows a shift to higher frequency of about $\sim 25 \pm 10\,\text{cm}^{-1}$ from those observed for the parent ligands, (iv) the appearance of a new band at 904–929 cm^{-1} due to ν(Si-O), and (v) strong absorptions at 1237–1260 and 750–851 cm^{-1} due to Si-CH_3 symmetric deformation and Si-C stretching, respectively. These assignments are based on the published data in the literature[8–12].

^{1}H NMR Spectra

^{1}H NMR spectra (Table III) of **(1)**–**(5)** show a singlet due to the azomethine proton in the range δ 8.40–8.83, exhibiting a downfield shift of 0.15 ± 0.05 ppm with respect to the parent ligands. The aromatic protons appear as multiplets in the range δ 6.79–7.80. Signals due to substituents (Me, Et, Pr-*i*) present on the aniline moiety appear in the region δ 1.25–2.94 with characteristic multiplicity. The methyl groups attached to silicon appear as singlets at δ 0.16–0.29.

^{13}C NMR Spectra

^{13}C NMR spectra (Table III) of **(1)**–**(4)** exhibit signals due to C-O and CH=N carbons at δ 155.47–157.89 and 152.82–155.52, respectively. An upfield shift of ~ 9 and ~ 6–9 ppm for the C-O and CH=N group carbons, respectively, has been observed. Aromatic carbons show signals in the range δ 116.90–133.90. The ^{13}C signals due to Me, Et or Pr-*i* groups on the aniline moiety appear in the region δ 14.89–24.75. The trimethyl silicon moiety shows a singlet at δ 0.21–1.69

The observed ^{1}J(Si-C) coupling constants, 70.30 Hz for **(1)** and 68.54 Hz for **(4)**, are consistent with tetrahedral organosilicon compounds[13].

^{29}Si NMR Spectra

The derivatives **(1)**–**(5)** show ^{29}Si NMR signals (Table III) in the range δ +16.08 to 23.04, which is consistent with tetrahedral organosilicon compounds[14].

Table II. IR Data (cm^{-1}) for the New Compounds

Compounds	ν(C=N)	ν(C-O)	ν(Si-O)	ν(Si-CH$_3$) Deformation	ν(Si-C) Stretching	Other Important Absorptions
(**1**)	1620 s	1280 s	904 m	1254 s	851 s, 750 s	–
(**2**)	1632 s	1295 s	929 m	1237 s	836 s, 751 s	–
(**3**)	1616 s	1280 s	904 m	1263 s	851 s, 756 s	–
(**4**)	1632 s	1300 s	927 m	1244 s	844 s, 760 s	–
(**5**)	1624 s	1285 s	906 m	1260 s	851 s, 808 s	–
(**6**)	1630 s	1300 s	–	–	–	607 m [ν(Ti-O)], 537 w [ν(Ti-N)], 350 m [ν(Ti-Cl)]
(**7**)	1632 s	1307 s	–	–	–	595 m [ν(Ti-O)], 540 w [ν(Ti-N)], 355 m [ν(Ti-Cl)]
(**8**)	1629 s	1274 s	–	–	–	551 m [ν(Zr-O)], 451 w [ν(Zr-N)], 340 m [ν(Zr-Cl)]

Abbreviations: m = medium, s = strong, and w = weak.

Table III. NMR Spectral Data (δ, ppm) for the New Compounds

Compound	^{1}H	^{13}C	^{29}Si
(1)	8.80 (s, 1H, C**H**=N), 6.79–7.68 (m, 9H, Ar-**H**), 0.18 (s, 9H, **Me**$_3$Si)	155.47 (**C**-O), 152.82 (**C**H=N), 132.81 (=N-**C**), 128.87–117.66 (Ar-**C**), 0.26 (**Me**$_3$Si)	+21.67
(2)	8.83 (s, 1H, C**H**=N), 6.90–7.42 (m, 7H, Ar-**H**), 2.25 (s, 6H, **Me**$_2$-2,6), 0.29 (s, 9H, **Me**$_3$Si)	157.89 (**C**-O), 155.45 (**C**H=N), 151.75 (=N-**C**), 132.80-119.57 (Ar-**C**), 18.33 (**Me**$_2$-2,6), 0.21 (**Me**$_3$Si)	+23.04
(3)	8.44 (s, 1H, C**H**=N), 6.83–7.64 (m, 6H, Ar-**H**), 2.21 (s, 6H, **Me**$_2$-2,6), 2.34 (s, 3H, **Me**-4), 0.20 (s, 3H, **Me**$_3$Si)	157.69 (**C**-O), 155.48 (**C**H=N), 151.69 (=N-**C**), 133.90-116.90 (Ar-**C**), 20.43 (**Me**$_2$-2,6), 18.05 (**Me**-4), 1.69 (**Me**$_3$Si)	+19.20
(4)	8.64 (s, 1H, C**H**=N), 7.11–7.80 (m, 7H, Ar-**H**), 2.63 (q, J = 6.27 Hz, 4H, CH$_2$C**H**$_3$), 1.17 (t, J = 6.27 Hz, 6H, C**H**$_2$CH$_3$), 0.16 (s, 9H, **Me**$_3$Si)	157.42 (**C**-O), 155.52 (**C**H=N), 151.03 (=N-**C**), 132.61-119.39 (Ar-**C**), 24.75 (**C**H$_2$CH$_3$), 14.89 (CH$_2$**C**H$_3$), 0.26 (**Me**$_3$Si)	+22.21
(5)	8.40 (s, 1H, C**H**=N), 6.80–7.64 (m, 7H, Ar-**H**), 2.94 (sept, J = 6.27 Hz, 2H, C**H**Me$_2$), 1.21 (d, J = 6.27 Hz, 12H, CH**Me**$_2$), 0.20 (s, 9H, **Me**$_3$Si)	–	+16.08
(6)	8.45 (s, 2H, C**H**=N), 6.84–7.64 (m, 12H, Ar-**H**), 2.32 (s, 6H, **Me**-4), 2.23(s, 12H, **Me**$_2$-2,6)	–	–
(7)	8.40 (s, 1H, C**H**=N), 6.88–7.60 (m, 7H, Ar-**H**) 3.02 (sept, J = 6.27 Hz, 1H, C**H**Me$_2$), 1.20 (d, J = 6.27 Hz,12H, CH**Me**$_2$)	–	–
(8)	8.64 (s, 1H, C**H**=N), 6.96–7.83 (m, 7H, Ar-**H**) 2.62 (q, J = 6.27 Hz, 4H, C**H**$_2$CH$_3$), 1.17 (t, J = 6.27 Hz, 6H, CH$_2$C**H**$_3$)	–	–

EXPERIMENTAL

All experimental work was performed under a moisture-free atmosphere using glass apparatus fitted with interchangeable quick-fit joints. The (BDH) solvents were dried by refluxing over suitable drying agents given in parentheses: benzene, toluene, and *n*-hexane (Na/benzophenone), CCl_4 (P_2O_5) and distilled under anhydrous conditions prior to use. Triethylamine was dried by keeping over KOH pellets for ~48 h and then refluxing for a period of ~7 h, followed by distillation (b.p. 88.8 °C). Trimethylsilicon chloride (Fluka) was distilled prior to use (b.p. 57 °C). The new substituted N-arylsalicylaldimines were prepared by equimolar reactions of salicylaldehyde with substituted anilines in the presence of isopropyl alcohol in benzene under refluxing conditions with continuous removal of the liberated H_2O as a ternary H_2O-*i*-PrOH-C_6H_6 azeotrope. After completion of the reaction volatile components were removed from the solution under reduced pressure. The analytically pure ligands were obtained by distillation.

Nitrogen and chloride were determined by Kjeldahl's or Volhard's methods, respectively[15]. Silicon and titanium were determined as SiO_2 and TiO_2, respectively. Zirconium was precipitated as zirconium mandelate and determined as ZrO_2 after ignition[15].

^1H (89.55 MHz) and ^{13}C (22.49 MHz) NMR spectra were recorded in $CDCl_3$ and CCl_4 solutions, respectively, on a JEOL FX-90Q FT spectrometer using TMS as an internal reference. ^{29}Si (17.75 MHz) NMR spectra were recorded in CCl_4 solutions using TMS as an external reference. IR spectra (4000-200 cm^{-1}) were recorded as Nujol mulls on a Nicolet Magna 550 spectrophotometer using CsI optics. Microanalyses (C, H, N) were performed using a Perkin Elmer 2400 CHNS/O analyzer. Molecular weights were measured in benzene with a Gallenkamp ebulliometer using a thermister sensing device.

Synthesis of $Me_3SiOC_6H_4CH{=}NC_6H_3Me_2$-2,6 (2)

To a benzene solution (40 mL) of $HOC_6H_4CH{=}NC_6H_3Me_2$-2,6 (6.87 g, 30.76 mmol) and Et_3N (3.11 g, 30.76 mmol) was added Me_3SiCl (3.31 g, 30.53 mmol) dissolved in benzene (50 mL). The reaction mixture was allowed to stir at room temperature for ~12 h, followed by refluxing for ~2 h. The precipitated $Et_3N{\cdot}HCl$ (4.21 g, 30.58 mmol) was filtered and the volatile components of the filtrate were removed under reduced pressure to yield an orange liquid (8.98 g, 99%). The product was distilled at 160 °C/0.2 mm. Yield, 8.16 g (90%).

The derivatives **(1)**, **(3)**, **(4)**, and **(5)** were prepared by a method similar to that used for **(2)**. Preparative and analytical data for **(1)**–**(5)** are listed in Table I.

Reactions of $Me_3SiOC_6H_4CH=NAr$ with $TiCl_4$ in *n*-Hexane

To a solution of $TiCl_4$ (0.31 g, 1.6 mmol) in *n*-hexane (25 mL), $Me_3SiOC_6H_4CH=NC_6H_2Me_3$-2,4,6 (1.00 g, 3.21 mmol) was added, and the reaction mixture was stirred at room temperature for ~12 h, followed by refluxing for 4 h. During the above mentioned period, the colour of the reaction mixture changed from yellow to orange-red and finally a red solid precipitated, which was removed by filtration. Recrystallization from hot *n*-hexane afforded $[TiCl_2(OC_6H_4CH=NC_6H_2Me_3\text{-}2,4,6)_2]$ **(6)**. Yield, 0.81 g (85%). Anal. Found: Ti, 8.21; Cl, 11.76; N, 4.52. Calcd. for $C_{32}H_{32}TiCl_2O_2N_2$ (595.38): Ti, 8.04; Cl, 11.91; N, 4.70%. M.wt., 600.

Adopting a similar procedure as in the previous description, equimolar reaction of $TiCl_4$ (0.45 g, 2.37 mmol) with $Me_3SiOC_6H_4CH=NC_6H_3(Pr\text{-}i)_2$-2,6 (0.85 g, 2.40 mmol) afforded red solid $[TiCl_3(OC_6H_4CH=NC_6H_3(Pr\text{-}i)_2\text{-}2,6)]$ **(7)**. Yield, 0.80 g (78%). Anal. Found: Ti, 11.00; Cl, 24.35; N, 3.26. Calcd. for $C_{19}H_{22}TiCl_3ON$ (434.61): Ti, 11.02; Cl, 24.47; N, 3.22%. M.wt., 444.

Reactions of $Me_3SiOC_6H_4CH=NC_6H_3Et_2$-2,6 with $ZrCl_4$ in Benzene

Reaction of equimolar amounts of $Me_3SiOC_6H_4CH=NC_6H_3Et_2$-2,6 (1.88 g, 5.78 mmol) with $ZrCl_4$ (1.34 g, 5.72 mmol) in benzene (40 mL) yielded an insoluble (moderately soluble in $CHCl_3$) yellow, powdery solid **(8)**, which was washed twice with benzene to obtain an analytically pure product. Yield, 2.39 g (92%). Anal. Found: Zr, 20.04; Cl, 23.32; N, 3.01. Calcd. for $C_{17}H_{18}Cl_3ONZr$ (449.91): Zr, 20.27; Cl, 23.63; N, 3.11%.

Interestingly, the 1:2 reaction of $ZrCl_4$ (0.73 g, 3.13 mmol) with $Me_3SiOC_6H_4CH=NC_6H_3Et_2$-2,6 (2.03 g, 6.03 mmol) in benzene, even after refluxing for a period of 14 h, produced only the insoluble derivative **(8)**. From the reaction mixture the unreacted $Me_3SiOC_6H_4CH=NC_6H_3Et_2$-2,6 was recovered by distillation (b.p. 175 °/0.2 mm). Anal. Found: Zr, 20.15; Cl, 23.54; N, 3.06. Calcd. for $C_{17}H_{18}Cl_3ONZr$ (449.91): Zr, 20.27; Cl, 23.63; N, 3.11%.

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