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Synthesis of a functionalized chromane derivative via a TiCl₄-mediated cyclization reaction

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Abstract The reaction of 1-methoxy-1,3-bis(silyloxy)-1,3-butadiene with *N*-salicylideneaniline results in the formation of a functionalized chromane derivative. Diffuse reflectance UV–Vis, ATR-IR, and Raman spectroscopic investigations showed that the reaction proceeds via formation of a titanium complex consisting of TiCl₄ and two molecules of the *N*-salicylideneaniline. In situ ATR-IR spectroscopy reveals that this 2:1 complex is responsible for the subsequent mode of interaction with the 1,3bis(silyloxy)-1,3-butadiene and formation of the product.

Keywords Cycloadditions · Reaction mechanism · In situ spectroscopy · IR spectroscopy · UV–Vis spectroscopy

Introduction

Recently, we reported mechanistic studies of the Lewis acid mediated cyclization of 1,3-bis(silyloxy)-1,3-butadiene 1

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S. Reimann · P. Langer Institut für Chemie, Universität Rostock, Albert-Einstein-Str. 3a, 18059 Rostock, Germany with ketene acetals **2a**, **2b** (Scheme 1) [1]. On the basis of our studies different intermediates of the reaction could be identified. The use of $TiCl_4$ as Lewis acid leads to the formation of a bis-chelate complex of **2** and $TiCl_4$ which plays a role during the formation of salicylates **3**. In contrast, in the presence of trimethylsilyl-trifluoromethanesulfonate (TMSOTf) pyran-4-ones **4** (mostly cyclohexenones) were formed because only one site of **2** is attacked by the TMSOTf.

Herein, we report, for the first time, the reaction of 1-methoxy-1,3-bis(silyloxy)-1,3-butadiene **1** with *N*-salicylideneaniline (**5**) which provides a convenient access to a functionalized chromane derivative. The mechanism of the reaction was studied by in situ spectroscopic experiments.

Results and discussion

Reaction of 1,3-bis(silyloxy)-1,3-butadiene with N-salicylideneaniline and $TiCl_4$

The TiCl₄-mediated reaction of 1-methoxy-1,3-bis(silyloxy)-1,3-butadiene **1** with *N*-salicylideneaniline (**5**) afforded the functionalized chromane methyl 2-[2-hydroxy-4-(phenylamino)chroman-2-yl]acetate (**7**) in 82 % yield as a mixture of two diastereomers (Scheme 2). The formation of the product can be explained by Lewis acid mediated attack of the terminal carbon atom of the diene onto the imine and cyclization by attack of the phenol oxygen atom onto the carbonyl group of the β -ketoester moiety. The theoretically possible formation of naphthalene derivative **6** by cyclization via an aromatic carbon atom was not observed. The structure of **7** was independently confirmed by X-ray crystal structure analysis (Fig. 1, Supplementary Material).

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Scheme 1

Interaction between N-salicylideneaniline and TiCl₄: ex situ analysis

To study the mechanism of the reaction of 1 with 5 comprehensive spectroscopic studies comprising ATR-IR, diffuse reflectance UV–Vis, and Raman spectroscopic investigations were performed. It is known that *N*-salicylideneaniline can form bidentate complexes with transition metals [2–6]. The coordination takes place via both the nitrogen and the oxygen atom.

To elucidate the interaction between **5** and TiCl₄ a mixture of both compounds was prepared in a ratio of 2:1 in CH₂Cl₂. The concentration of 0.5 mol/dm³ was similar to the concentration of the reactants during the reaction. After mixing both components, a dark red precipitate is immediately formed which was isolated and structurally studied.

Scheme 2

In a second experiment, **5** and TiCl₄ were mixed in a ratio of 1:1 in CH₂Cl₂ in a concentration of 0.5 mol/dm³. Similar to the previous experiment, a dark red precipitate is formed which was also isolated and analyzed. The ATR-IR, diffuse reflectance UV–Vis, Raman spectra, and the obtained XRD patterns of both precipitates were very similar. Obviously, two molecules of **5** always interact with one TiCl₄ molecule.

The diffuse reflectance UV-Vis spectrum of the solid formed from the 2:1 mixture of 5 and TiCl₄ (Fig. 2a) shows a broad band containing two maxima at 286 and 484 nm with a shoulder at 533 nm. The diffuse reflectance UV-Vis spectrum of neat (solid) 5 (Fig. 2b) has a different shape as compared to the spectra recorded in diluted solutions. This is caused by bulk effects like intermolecular hydrogen bonding. The spectrum of 5 shows three maxima at 257, 300/315, and 395 nm. The band at 257 nm can be assigned to $\sigma \rightarrow \pi^*$ transitions and the band at 300 and 315 nm to $\pi \to \pi^*$ transitions of the phenyl ring of salicylideneaniline. The band at 395 nm can be assigned to a $\pi \to \pi^*$ transition as well. A proper assignment seems to be difficult in this case because the band of the $\pi \to \pi^*$ transition of the benzyl ring occurs in the liquid phase at 339 nm, whereas the long-wave band of the quinoid tautomer occurs at 427 nm [7, 8].

According to our previous studies, the shift to longer wavelengths indicates the formation of a bis-chelate complex like structure **8** (Scheme 3). This type of compound was previously described by Birdar and Kulkarni [9], but its color was yellow. This is contrary to the dark red color of the precipitate observed in the present study. Hence, a different structure than **8** has to be assumed. However, an examination of the preparation methods of **8** and related compounds [2, 3, 5, 6, 9] revealed that to obtain the desired







Fig. 2 Ex situ diffuse reflectance UV–Vis spectra of the precipitate derived from the 2:1 mixture of **5** and TiCl₄ (*a*) and of the neat solid **5** (*b*). The UV–Vis transmission spectrum of TiCl₄ dissolved in CH₂Cl₂ ($c = 0.5 \text{ mol/dm}^3$) is shown for comparison (*c*)



Scheme 3

chelate compound a final treatment of the reaction mixture with bases like NEt_3 or heating is necessary to remove HCl.

In Table 1 the calculated elemental percentages for different assumed compositions of the complex formed by reaction of 5 with $TiCl_4$ are compared with the experimental values. The obtained values clearly show that the composition of the dark red precipitate is not comparable

with **8** and instead seems to have the empirical formula $C_{26}H_{22}Cl_4N_2O_2Ti$. The differences between calculated and experimental mass percentages indicate that the new compound is not pure. However, the N/Cl ratio is 1:2 which points to the formation of $C_{26}H_{22}Cl_4N_2O_2Ti$.

The Raman and ATR-IR spectra of the dark red precipitate and the ATR-IR spectrum of **5** in different spectral ranges are shown in Fig. 3. As a result of the strong fluorescence of pure **5**, no Raman spectrum could be obtained.

The spectrum of neat solid **5** (Fig. 3A, spectrum a) shows characteristic bands at 1613, 1587, 1568, and 1273 cm⁻¹ which correspond to v(C=N), v(Ph–N), v(Ph–C), and v(C–O) vibrations, respectively [2, 10, 11]. In the spectrum of the dark red precipitate (Fig. 3A, spectrum b) a band at 1638 cm⁻¹ is observed (at 1641 cm⁻¹ in the Raman spectrum c), which can be assigned to the shifted v(C=N) vibration mode caused by the protonation of the C=N double bond in **5**. Such an effect has already been observed by studying the interaction of imines with Lewis acidic heterogeneous catalysts [12]. Obviously, the interaction between the Lewis acid TiCl₄ and **5** takes place in a similar way.

The spectrum of the precipitate shows more strong bands located at 1600 and 1588 cm⁻¹ in the ATR-IR spectrum and at 1602 and 1589 cm⁻¹ in the Raman spectrum (Fig. 3a, spectra b and c). They can be assigned to v(Ph–N) and v(Ph–C) modes. These bands are shifted due to protonation as well, because the charge stabilization takes place via the complete azomethine moiety [13]. A further new band occurs at 1544 cm⁻¹ in the ATR-IR and 1549 cm⁻¹ in the Raman spectrum, respectively. This band

Table 1 Calculated and obtained element percentages for differentassumed compositions of the complex formed between 5 and TiCl₄

Composition	Ti	Cl	Ν	Ratio Ti/Cl/N
C ₂₆ H ₂₀ Cl ₂ N ₂ O ₂ Ti [9]	8.82	13.08	5.16	1.0:2.0:2.0
C ₂₆ H ₂₂ Cl ₄ N ₂ O ₂ Ti (calc.)	8.21	24.29	4.80	1.0:4.0:2.0
Complex $[5 + TiCl_4]$ (exp.)	9.50	23.80	4.66	1.0:3.4:1.7

was also observed when imines adsorb on the surface of solid Al_2O_3 and can be assigned to a v(C=C) vibration induced by formation of a protonated azomethine moiety [12, 14].

The ATR-IR spectrum of neat solid **5** (Fig. 3A, spectrum a) shows another strong band at 1273 cm⁻¹ which is observed in the spectrum of the precipitate (Fig. 3A, spectrum b), too. This band can be assigned to δ (OH) [15] or to the v(C–O) vibration [2]. However, this band was not observed in the Raman spectrum (Fig. 3A, spectrum c). Instead, a very strong band is detected at 1302 cm⁻¹ which is not present in the corresponding ATR-IR spectrum. However, reference spectra of **5** dissolved in CCl₄ revealed a strong band at 1286 cm⁻¹ and a very weak band at 1322 cm⁻¹ in the ATR-IR spectrum but a weak band at 1286 cm⁻¹ and a strong band at 1322 cm⁻¹ in the

Fig. 3 ATR-IR spectra of the neat solid 5 (*a*), the dark red precipitate derived from the 2:1 mixture (molar ratio) of 5 and TiCl₄ (*b*), and Raman spectrum of the dark red precipitate derived from the 2:1 mixture of 5 and TiCl₄ (*c*) in the spectral range ranges $1650-1250 \text{ cm}^{-1}$ (A) and $625-400/250 \text{ cm}^{-1}$ (B)



The bands at 330/321 cm⁻¹ in the Raman spectrum (Fig. 3B, spectrum c) are related to v(Ti–Cl) vibrations [5, 16]. Alternatively, this band could be also be assigned to the v₁ vibration of TiCl₆^{2–} and the band at 272 cm⁻¹ to the respective v₂ vibration [17]. But the formation of TiCl₆^{2–} in significant amounts should be expected only if **5** and TiCl₄ are mixed in 1:1 ratio. Characteristic bands of TiCl₄ or TiCl₃ [17–20] do not appear in the spectra, and



there was no evidence for the presence of $TiCl_3$ from XRD analysis. The absence of Ti(III) species has been proved also by EPR spectroscopy.

In summary, the vibrational spectra indicate that the C=N double bond of **5** is involved in the interaction with TiCl₄. The formation of Ti–O bonds obviously proceeds with participation of the OH group of **5**. Otherwise the elementary analysis reveals the formation of a complex of TiCl₄ with two imine molecules.

Strauch et al. [6] reported the synthesis and structural properties of salicylaldiminato complexes of titanium. They described the structure of the red 1:2 complex **9** (Scheme 4) which was formed by reaction of TiCl₄ with two molar equivalents of N-(2,6-diisopropylphenyl)salicylimine in CH₂Cl₂.

The molecular structure of this compound indicates that two imines are bonded via their oxygen atoms to the titanium atom, while the four chlorine atoms remain coordinated, leading to an octahedral coordination sphere of titanium. The proton of the hydroxyl group migrated to



Scheme 4

Table 2Selected IR frequencies/ cm^{-1} of the solid complexes 9 and10

9 ^a	10 ^b	Assignment
1635 (vs)	1638 (s)	v(C=N)
1549 (s)	1544 (s)	$\nu(Ph-NH^+)$
1469 (vs)	1472 (vs)	
1283 (vs)	1275 (vs)	v(C–O)
1144 (m)	1146 (vs)	
916 (m)	912 (m)	
798 (s)	801 (s)	
753 (m)	756 (vs)	
613 (s)	602 (vs)	v(Ti–O)

^a Ref. [6]

^b This work

the nitrogen atom and is bonded via the electron lone pair of the nitrogen atom. A comparison of the IR spectroscopic data described for **9** with the data obtained for the dark red precipitate derived from the mixture **5** and TiCl₄ (Table 2) reveals that both substances are structurally very similar.

As a result of the very poor solubility of **10** in various solvents, a further characterization by NMR spectroscopy or X-ray crystal structure analysis was not possible. On the basis of the comparison with **9**, we believe that the reaction of TiCl₄ with imine **5** results in the formation of complex **10** (Scheme 4).

Reaction monitoring using in situ ATR-IR spectroscopy

To get an insight into the overall reaction of $TiCl_4$ with 1,3bis(silyloxy)-1,3-butadiene **1** and *N*-salicylideneaniline, in situ ATR-IR spectroscopic investigations were performed. These were carried out under the same reaction conditions which were used in our previous study [1] (cf. Scheme 1). The application of in situ UV–Vis transmission spectroscopy failed because the formation of the red precipitate **9** at the beginning of the reaction perturbed the measurements.

The in situ ATR-IR spectra obtained during the reaction of **5** with **1** are displayed in Fig. 4. The spectra of **5** in CH₂Cl₂ at 0 °C (spectrum a), of the mixture of **5** and TiCl₄ at -60 °C (spectrum b), and of **1** + TiCl₄ (1:1 molar ratio) in CH₂Cl₂ at 25 °C (spectrum n) were included for comparison.

The spectrum of **5** dissolved in CH_2Cl_2 (Fig. 4a) is comparable with that measured in the solid state (Fig. 3a). Merely the band of the v(C=N) vibration is shifted from 1613 to 1617 cm⁻¹ because the intermolecular interactions occurring in solution are weaker than in the solid state [10].

The addition of TiCl₄ to **5** effects characteristic shifts (Fig. 4, spectrum b) which were discussed above. This interaction occurs in CH₂Cl₂ independently from temperature. For this reason only the spectrum of the 1:1 mixture of **5** with TiCl₄ at -60 °C is shown in Fig. 4.

By addition of 1 at -50 °C the resulting spectrum of the reaction mixture (Fig. 4, spectrum c) can be perceived as a summation of spectrum b (5 + TiCl₄) with spectrum n (1 + TiCl₄). With increasing temperature the band at 1004 cm⁻¹ decreases, and a new band at 985 cm⁻¹ appears. Additionally, a band at 1526 cm⁻¹ arises at -15 °C the assignment of which is difficult. However, from the bands in the range 1060–950 cm⁻¹ some conclusions can be made concerning the changes of the v(C–O) vibrations. The band at 1004 cm⁻¹, observed in spectrum b (Fig. 4), results from v(C–O) vibrations where the oxygen is coordinated to Ti (cf. 10 in Scheme 4). The bands at 1062 and 954 cm⁻¹ in spectrum n (Fig. 3) can be assigned to v_{as} (C–O) and v_s (C–O) of the complex which is formed

Fig. 4 In situ ATR-IR spectra of the TiCl₄-mediated reaction between 5 and 1 in CH₂Cl₂: 5 at 0 °C (a), 5 + TiCl₄ (1:1 molar ratio) at -60 °C (b), 5 + TiCl₄ + 1 (1:1:2 molar ratio) at -50, -40, -35, -30, -25, -20, -15, -10, -5, -3, and -2 °C (*c*-*m*). The spectrum of 1 + TiCl₄ (1:1 molar ratio) is given for comparison (*n*)



between 1 and $TiCl_4$ [1]. A possible structure of the complex 11 is given in Scheme 5.

Absorbance

During heating of the complete reaction mixture (Fig. 4, spectra c–m) bands at 1052, 1004, and 985 cm⁻¹ can be observed where only the intensity of the first band does not change. At the end of the reaction at -2 °C, only the bands at 1052 and 985 cm⁻¹ remain which can be related to the v_{as} (C–O) and v_{s} (C–O) modes of a titanium complex similar to **10** and **11**. Obviously, TiCl₄ interacts with both reactants without preferring one of them.

It should be mentioned at this point that the dark red precipitate **10**, which is formed at the beginning of the reaction, is slowly dissolved after addition of **1** and warming of the reaction mixture. This suggests that reactant **1** interacts with complex **10** and undergoes partial elimination of TMSCl to form a new intermediate complex in which **1** additionally coordinates via the released oxygen atoms to titanium. As a result, two v(C-O) vibrations at 1052 and 985 cm⁻¹ are observed. Their band positions differ from those observed for complexes **10** and **11**.

According to the synthetic protocol [1] the reaction is quenched with aqueous HCl. Thus, the reaction was stopped after 4 h at 0 °C, and aqueous HCl was added. The respective ATR-IR spectra of the reaction mixture before and after quenching are shown in Fig. 5.

The most interesting features are the new bands at 1745 and 1719 cm⁻¹ which indicate the formation of different carbonyl groups in this reaction step (Fig. 5b). After the purification with flash chromatography, only one intensive carbonyl band at 1707 cm⁻¹ is observed, whereas the carbonyl bands at 1745 and 1719 cm⁻¹ vanish (Fig. 5c).



Scheme 5

These results clearly demonstrate, as already observed in the reaction of ketene acetal **2b** with diene **1** [1], that the work-up procedure is part of the reaction and influences the formation and nature of the final product.

Mechanistic considerations

The formation of the chromane derivative 7 can be explained by the nature of intermediate 10 (cf. Scheme 4) which obviously directs the addition of 1. A possible transition state for the reaction of the titanium complex with diene 1 is schematically shown in Scheme 6.

The formed 2:1 complex controls the formal addition of **1** in a special way preferring a concerted interaction of two molecules of **1** with the protonated nitrogens of **5**. The favored elimination of TMSCl by HCl addition causes a rearrangement of the complex **12** leading consequently to the splitting of the complex into two molecules of **7** and TiCl₄.

Conclusion

The presented ex situ and in situ spectroscopic investigations of the $TiCl_4$ -mediated reaction of *N*-salicylideneaniline (5)

ATR-IR spectrum of the isolated solid product 7 after work-up with flash chromatography (c)





Scheme 6

and 1,3-bis(silyloxy)-1,3-butadiene 1 have demonstrated, as already shown in a former study using fluorinated ketene acetals and 1 as substrates [1], that the nature of the intermediately formed titanium complex markedly influences the subsequent reaction with 1. The possible formation of bischelate complexes like TiCl₂(OOR)₂ and TiCl₂(ONR)₂ on the one hand, or $TiCl_4(OR)_2$ on the other hand, offers different interaction possibilities for the second reactant. Although 5 is known to form bis-chelate complexes with TiCl₄, the investigations show that, under the applied conditions, a solid dark red complex $TiCl_4(OR)_2$ is formed in which two molecules of 5 are bonded via their oxygen atoms only to the titanium atom. This type of intermediate titanium complex is responsible for the subsequent mode of interaction with 1 leading finally to chromane 7 instead of the naphthalene derivative 6 (Scheme 2).

As indicated by in situ ATR-IR measurements, the formed 2:1 complex manages the formal addition of **1** preferring a concerted interaction of two molecules of **1** with the protonated nitrogens of **5**. This is a slow process indicated by the continuous decrease of a distinct v(C-O-Ti) band at 1004 cm⁻¹ and the simultaneous rising of a new one at 985 cm⁻¹ over 4 h. Obviously, the proceeding dissolution of the solid 2:1 complex in the presence of **1** is the ratedetermining step. The obtained yield of the chromane derivative **7**, isolated after flash chromatography, was high (82 %). The ATR-IR measurements showed that the workup procedure is part of the reaction and significantly influences the formation and nature of the final product as already observed in the reaction of ketene acetals with **1**.

Experimental

Titanium(IV) chloride was obtained from Aldrich (99.995 %) and was used without further purification as was the used CH_2Cl_2 which was obtained by ACROS Organics (99.8 % extra dry over molecular sieves, stabilized). 1,3-Bis(silyloxy)-1,3-butadiene **1** was prepared according to the literature method [21]. *N*-Salicylideneaniline (**5**) was used as received from Aldrich (97 %).

The reaction was carried out in manner analogous to the procedure described elsewhere [1]. Cooling of the homemade reaction cell was done by a Julabo cryostat. The reaction cell was flushed with argon before reaction and during addition of the reactants. Compound **5** (788 mg, 4 mmol) was dissolved in CH_2Cl_2 at room temperature. The solution (0.44 cm³, 4 mmol) was cooled and TiCl₄ was added when a temperature of 0 °C was reached. The cooling was continued, and 2,080 mg 1,3-bis(silyloxy)-1,3-butadiene **1** (8 mmol) was added at -60 °C. Afterwards, the mixture was warmed to 0 °C over 4 h. Finally, the reaction was quenched by addition of 60 cm³ 10 % HCl solution and worked up by flash chromatography. Details concerning the preparation procedure and characterization of the chromane derivate **7** are available in the Supplementary Material.

ATR-IR spectra were measured with a Bruker Alpha FT-IR spectrometer which was equipped with a Platinum ATR single reflection diamond ATR module. All spectra were recorded with 32 scans at 4 cm⁻¹ resolution. For the in situ ATR-IR studies a fiber optic diamond ATR probe (infrared fiber sensors, Aachen, Germany) was connected to a Nicolet Avatar 370 (Thermo Electron) FT-IR spectrometer. The probe was immersed in the reaction mixture. All spectra were recorded with 128 scans at 4 cm⁻¹ resolution.

The UV–Vis spectra were sampled with an AvaSpec 2048 fiber optic spectrometer (Avantes B.V., Eerbeek, the Netherlands) equipped with an AvaLight-DHS light source and an FCR-7xx200-2-45 reflection probe. Raman spectra were recorded using an RXN1-785 Raman system (Kaiser Optical Systems, KOSI Inc., Michigan, USA) equipped with a 70 mW diode laser for excitation at a wavelength of 785 nm. The Raman spectra were recorded with three accumulations and 3 s exposure time.

Data for X-ray crystal structure analysis of 7 were collected on a STOE IPDS II diffractometer using graphitemonochromated Mo K α radiation. The structure was solved by direct methods and refined by full-matrix leastsquares procedures on F^2 with the SHELXTL software package [22]. XP (Bruker AXS) was used to generate graphical representations.

Crystal data of 7: C₁₈H₁₉NO₄, M = 313.34, monoclinic, space group $P2_1/n$, a = 5.6524(2) Å, b = 19.6510(6) Å, c = 13.9584(6) Å, $\beta = 91.285(3)^\circ$, V = 1550.04(10) Å³, T = 150 K, Z = 4, $\rho_{calcd} = 1.343$ g cm⁻³, $\mu = 0.095$ mm⁻¹, 29640 reflections measured, 4,180 independent reflections ($R_{int} = 0.0425$), final R values ($I > 2\sigma(I)$): $R_1 = 0.0449$, $wR_2 = 0.1040$, final R values (all data): $R_1 = 0.0840$, $wR_2 = 0.1126$, 252 parameters. CCDC 865825 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif.

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