Dedicated to Full Member of the Russian Academy of Sciences I.P. Beletskaya on her jubilee

Efficient Halogenation of Unsaturated Organoaluminum Compounds with Sulfonyl Halides

I. R. Ramazanov, R. N. Kadikova, and U. M. Dzhemilev

Institute of Petrochemistry and Catalysis, Russian Academy of Sciences, pr. Oktyabrya 141, Ufa, 450075 Bashkortostan, Russia e-mail: iramazan@inbox.ru

Received January 11, 2013

Abstract—Alkenylalumanes prepared by carbo- or cycloalumination of substituted acetylenes reacted with an equivalent amount of sulfonyl halide (MsCl, TsCl, PhSO₂Cl, MsBr) in methylene chloride or hexane at room temperature to produce alkenyl halides in high yields. Electron-donor solvents such as diethyl ether or tetra-hydrofuran inhibited the halogenation process. β -Substituted vinylalumanes generated by hydroalumination of substituted acetylenes failed to react with sulfonyl halides.

DOI: 10.1134/S1070428013030020

Halogenation with the aid of electrophilic halogen sources is one of the most widely used methods for functionalization of organometallic compounds. Although the reaction of diethylzinc with benzenesulfonyl chloride, leading to ethyl chloride and zinc benzenesulfinate, has been known since XIXth century [1], only a few published data are available on the use of sulfonyl halides as halogenating agents for functionalization of organometallic compounds. Triethylaluminum and ethylaluminum sesquichloride reacted with sulfonyl chlorides to form sulfinic acids and ethyl chloride [2]. Benzenesulfonyl chloride was also used for monochlorination of alkylidenebiszinc compounds. Zinc atom can be replaced by chlorine both at sp^2 - and sp^{3} -hybridized carbon atoms [3–5]. However, allylzinc compounds reacted with alkane- and arenesulfonyl chlorides to give exclusively β_{γ} -unsaturated sulfones [6]. Reactions of Grignard compounds with sulfonyl chlorides also produced the corresponding sulfones [7]. Alkenyl sulfones were obtained by reaction of alkenylzirconocene derivatives with alkane- and arenesulfonyl chlorides under mild conditions [8]. Crosscoupling of arenesulfonyl chlorides with trialkylarylstannanes at 130°C [9] and with alkenylstannanes in the presence of a catalytic amount of palladium complexes also afforded aryl sulfones [10]. Diarylcadmiums reacted with arenesulfonyl chlorides to give

mixtures of aryl sulfones, aryl chlorides, and sulfinic acids [11–13], whereas no sulfones were formed from diethylcadmium. Thus, the above cross-coupling reactions of organometallic compounds with sulfonyl halides are governed by both metal nature and substituent attached thereto.

Alkyl sulfonates are efficient reagents for the alkylation of cyclic alkenylalumanes (aluminacyclopent-2-enes) [14, 15]. The high yield of the resulting cyclopropanes and selectivity of these reactions make sulfonic acid derivatives promising as reagents for transformations of organoaluminum compounds. First of all, we planned to examine reactions with sulfonyl halides which were assumed to be efficient sources of electrophilic halogen. Previously, alkenylalumanes were converted into halogen derivatives with the use of halogens (Br₂, I₂) [16], N-halosuccinimides (NCS, NBS, NIS) [17], and pseudohalogens (BrCN, ICl) [18]. Alkylaluminum compounds reacted with the above halogen derivatives in a similar way, i.e., via cleavage of the Al-C bond. In view of the aforesaid, in the present work we studied reactions of sulfonyl halides with both acyclic and cyclic alkenylalumanes with the goal of developing an efficient procedure for the synthesis of various vinyl halides.

Pure alkenylalumanes are difficult to isolate as individual substances, for they readily undergo decom-





 $\mathbf{I}, \text{Hlg} = \text{Cl}; \ \mathbf{R}^{1} = \text{C}_{6}\text{H}_{13}, \ \mathbf{R}^{2} = \text{H} (\mathbf{a}); \ \mathbf{R}^{1} = \text{Ph}, \ \mathbf{R}^{2} = \text{H} (\mathbf{b}); \ \mathbf{R}^{1} = \mathbf{R}^{2} = \text{Bu} (\mathbf{c}); \ \mathbf{II}, \ \text{Hlg} = \text{Br}; \ \mathbf{R}^{1} = \text{C}_{6}\text{H}_{13}, \ \mathbf{R}^{2} = \text{H} (\mathbf{a}); \ \mathbf{R}^{1} = \text{Ph}, \ \mathbf{R}^{2} = \text{H} (\mathbf{b}).$

position on attempted distillation. Therefore, alkenylalumanes were generated in situ by hydro-, carbo-, and cycloalumination of alkynes. Negishi carboalumination is one of the most useful transformations of alkynes [19]. Alkenylalumanes obtained by Zr-catalyzed methylalumination of alkynes (oct-1-yne, phenylacetylene, dec-5-yne) [20] reacted with methanesulfonyl chloride or bromide in 15 min at room temperature in methylene chloride to give the corresponding halogen derivatives Ia-Ic or IIa and IIb in high yield (Scheme 1). The configuration of the double bond did not change in this transformation, which was confirmed by NOE experiments. In the NOESY spectrum of Ia we observed a cross peak between proton at the double bond (singlet) and protons in the α -methylene group of the hexyl radical.

The reaction of phenylacetylene with trimethylaluminum, followed by treatment with MsBr, gave a mixture of compound **IIb** and its *Z* isomer **III** (15%). The latter was identified by NMR spectroscopy. The ¹H NMR spectra of the *Z* and *E* isomers are fairly similar, but signals from the methyl group and hydrogen at the double bond in the spectrum of *Z* isomer **III** appeared in a stronger field (by 0.11 and 0.19 ppm, respectively), relative to the corresponding signals of *E* isomer **IIb** [21].

Alkenylalumane generated by zirconium-catalyzed methylalumination of oct-1-yne was brought into reactions with different sulfonyl halides, in particular *p*-toluenesulfonyl chloride and fluoride, benzenesulfonyl chloride, and methanesulfonyl bromide. The yield of compound **Ia** in the reaction with TsCl and PhSO₂Cl was 88 and 90%, respectively; analogous result was obtained with MsCl. Methanesulfonyl fluoride failed to react with the above alkenylalumanes under the given conditions. Electrophilic fluorinating agents such as *N*-fluoro-*o*-benzenedisulfonimide, *N*-fluorobenzenesulfonimide, and Selectfluor [22] contain stronger electron-withdrawing groups on the fluorine atom as compared to sulfonyl group. Reactions of alkenylalumanes with sulfonyl iodides were

not studied due to thermal instability of the latter and high efficiency of iodine as iodinating agent.

Taking into account simplicity of the experimental procedure and accessibility of sulfonyl halides, halogenation of alkenylalumanes with various structures was studied. B-Substituted vinylalumanes obtained by hydroalumination of terminal alkynes (oct-1-yne and phenylacetylene) with diisobutylaluminum hydride (Scheme 2) [23] turned out to be inactive toward sulfonyl halides (MsCl, TsCl, MsBr). We presumed that the presence of Cp_2ZrCl_2 in the reaction mixture could favor the reaction; however, addition of the zirconocene catalyst (0.1 equiv with respect to alkenylalumane) had no effect. This may be due to lower nucleophilicity of β-substituted vinylalumanes compared to $\beta_{\beta}\beta_{\beta}$ -disubstituted. Treatment with mesyl chloride of α,β -substituted vinylalumane generated by hydroalumination of dec-5-yne with triethylaluminum in the presence of titanium catalyst [24] gave a mixture of stereoisomeric alkenyl chlorides IV and V at a ratio of $\sim 4:3$. The Z/E isomer ratio was estimated from the ¹H NMR spectrum of the reaction mixture, and the vields of IV and V were determined from the amount of the isomer mixture isolated by distillation with account taken of the isomer ratio. We failed to obtain (E)- or (Z)-1-chloroalk-1-envlsilanes from the corresponding alkenylalumanes [17] and mesyl chloride. Obviously, this procedure is hardly applicable to α , β -substituted alkenvlalumanes.

Thus, the best results were obtained in the halogenation of α,β,β - and β,β -substituted vinylalumanes synthesized by Negishi carboalumination of alkynes. Zirconium-catalyzed cycloalumination of disubstituted acetylenes with triethylaluminum, which is also known as Dzhemilev reaction, yields aluminacyclopent-2enes; the latter may be regarded as α,β,β -substituted vinylalumanes [25]. Insofar as the nucleophilicities of α,β,β - and β,β -substituted vinyl anions in the reaction under study are comparable, we expected successful reaction of aluminacyclopent-2-enes with sulfonyl halides. In fact, aluminacyclopent-2-enes obtained by cycloalumination of dec-5-yne, oct-4-yne, 1-phenyl-



 $VI, Hlg = Cl: R^{1} = R^{2} = Bu, R^{3} = D (a); R^{1} = R^{2} = Pr, R^{3} = H (b); R^{1} = Ph, R^{2} = Bu, R^{3} = H (c); R^{1} = Me_{2}NCH_{2}, R^{2} = Bu, R^{3} = H (d); VII, Hlg = Br, R^{1} = R^{2} = Bu, R^{3} = H.$

Atom numbering in the ¹H and ¹³C NMR spectra.



RUSSIAN JOURNAL OF ORGANIC CHEMISTRY Vol. 49 No. 3 2013

hex-1-yne, and N,N-dimethylhept-2-yn-1-amine [26] reacted with 1 equiv of MsCl in a regioselective fashion at the double-bonded carbon atom, and the subsequent hydrolysis or deuterolysis afforded in high vield the corresponding alkenyl chlorides VIa-VId (Scheme 3) with the same configuration of the double bond as in the initial compounds (according to the NOESY data). The second metal-carbon bond in the metallacycle was not involved even when the amount of mesyl chloride was increased to 5 equiv. Analogous regioselectivity was observed previously in the alkylation of aluminacyclopent-2-enes with dialkyl sulfates [14]. The use of mesyl bromide instead of mesyl chloride resulted in the formation of monobromo derivative VII, but the yield was only 63%. In the bromination of dibutyl-substituted aluminacyclopent-2-ene with NBS (Et₂O, -20° C) alkenyl bromide VII was formed in 55% yield, whereas the yield of alkenyl chloride VIa in the reaction of the same substrate with NCS (Et₂O, -20° C) was similar to that obtained with the use of MsCl, TsCl, or PhSO₂Cl.

It should be noted that sulfonyl halides did not react with alkenylalumanes in electron-donor solvents such as diethyl ether and tetrahydrofuran. On the other hand, the halogenation with NCS and NBS requires ether solvents and low temperature (-20° C) [17]. The bromination of alkenylalumanes with Br₂ is carried out at -70 to -50° C [16]. Elemental halogens (Br₂, I₂) and *N*-halosuccinimides (NCS, NBS) ensured successful halogenation of β -substituted vinylalumanes which were inactive toward sulfonyl halides.

EXPERIMENTAL

The ¹H and ¹³C NMR spectra were recorded on a Bruker Avance 400 spectrometer at 400.13 and 100.62 MHz, respectively, using tetramethylsilane (¹H) or CDCl₃ (¹³C) as internal reference. For atom numbering in the description of the NMR spectra of **Ia–Ic**, **IIa**, **IIb**, **III**, **VIa–VId**, and **VII**, see Scheme 3.

Most reagents used were commercial products. Reactions with organoaluminum compounds were carried out under dry argon. Hexane was distilled over (i-Bu)₃Al, methylene chloride was dried over P₂O₅, *N*,*N*-dimethylhept-2-yn-1-amine was synthesized by aminomethylation of hex-1-yne [27], 1-phenylhex-1yne was prepared according to [28], *p*-toluenesulfonyl fluoride was obtained from *p*-toluenesulfonyl chloride and potassium fluoride dihydrate [29], and methanesulfonyl bromide was prepared from methanesulfonyl chloride [30]. The products were analyzed by GLC on a Carlo Erba chromatograph (HP Ultra-1 glass capillary column, 25 m×0.2 mm; flame ionization detector; oven temperature 50–170°C; carrier gas helium). The elemental compositions were determined on a Carlo Erba 1106 analyzer. The yields were determined by the internal standard quantitation method (GLC). The boiling points were measured according to [31]. The concentrations of chlorine and bromine were determined by Schöniger oxidation [32].

Halogenation of alkenylalumanes (general procedure). Alkenylalumanes were generated from 1 mmol of the corresponding substituted acetylene according to the procedures indicated in text. Methanesulfonyl chloride, 1 mmol, was added to the reaction mixture through a rubber septum with a syringe, and the mixture was stirred for 15 min at room temperature. The mixture was then diluted with 5 ml of hexane, 3 ml of water was added dropwise on cooling in an ice bath, and the precipitate was filtered off through a filter paper. The aqueous phase was extracted with diethyl ether, and the extract was combined with the organic phase, dried over anhydrous CaCl₂, and concentrated under reduced pressure.

(*E*)-1-Chloro-2-methyloct-1-ene (Ia). Yield 81%, bp 61–63°C (5 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 0.91 t (3H, 8-H, J = 7 Hz), 1.20–1.38 m (4H, 6-H, 7-H), 1.38–1.50 m (4H, 4-H, 5-H), 1.78 s (3H, 9-H), 2.07 t (2H, 3-H, J = 8 Hz), 5.80 s (1H, 1-H). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 14.06 (C⁸), 16.34 (C⁹), 22.58 (C⁷), 27.46 (C⁵), 28.77 (C⁴), 31.63 (C⁶), 37.09 (C³), 111.61 (C¹), 138.97 (C²). Found, %: C 67.21; H 10.55; Cl 23.2. C₉H₁₇Cl. Calculated, %: C 67.29; H 10.59; Cl 22.12.

[(*E*)-1-Chloroprop-1-en-2-yl]benzene (Ib). Yield 84%, bp 80–83°C (5 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 2.24 s (3H, 9-H), 6.36 s (1H, 1-H), 7.28–7.45 m (5H, Ph). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 16.89 (C⁹), 115.8 (C¹), 125.94 and 128.54 (C⁴, C⁵, C⁷, C⁸), 127.79 (C⁶), 138.56 and 140.33 (C², C³). Found, %: C 70.32; H 5.94; Cl 26.1. C₉H₉Cl. Calculated, %: C 70.82; H 5.90; Cl 23.28.

(*E*)-6-Chloro-5-methyldec-5-ene (Ic). Yield 79%, bp 78–80°C (3 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 0.87–1.00 m (6H, 1-H, 10-H), 1.26–1.46 m (2H, 2-H), 1.46–1.60 m (6H, 2-H, 8-H, 9-H), 1.82 s (3H, 11-H), 2.11 t (4H, 4-H, 7-H, *J* = 7.2 Hz). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 13.97 (C¹, C¹⁰), 19.81 (C², C⁹), 22.06 and 22.57 (C³, C¹¹), 30.25 (C⁸), 34.04 and 34.75 (C⁴, C⁷), 129.31 (C¹), 131.25 (C²). Found, %: C 70.49; H 11.01; Cl 19.9. $C_{11}H_{21}Cl$. Calculated, %: C 70.03; H 11.14; Cl 18.83.

(*E*)-1-Bromo-2-methyloct-1-ene (IIa). Yield 77%, bp 64–66°C (5 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 0.91 t (3H, 8-H, J = 8 Hz), 1.20–1.38 m (2H, 6-H, 7-H), 1.38–1.50 m (2H, 4-H, 5-H), 1.80 s (3H, 9-H), 2.12 t (2H, 3-H, J = 4 Hz), 5.90 s (1H, 1-H). ¹³C NMR spectrum, δ_{C} , ppm: 14.07 (C⁸), 19.00 (C⁹), 22.59 (C⁷), 27.47 (C⁵), 28.75 (C⁴), 31.62 (C⁶), 38.34 (C³), 100.84 (C¹), 142.01 (C²). Found, %: C 52.81; H 8.15; Br 39.6. C₉H₁₇Br. Calculated, %: C 52.70; H 8.29; Br 39.01.

[(*E*)-1-Bromoprop-1-en-2-yl]benzene (IIb). Yield 68%, bp 81–83°C (5 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 2.26 s (3H, 9-H), 6.48 s (1H, 1-H), 7.20–7.46 m (5H, Ph). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 19.66 (C⁹), 105.39 (C¹), 126.00 and 128.54 (C⁴, C⁵, C⁷, C⁸), 127.84 (C⁶), 140.96 and 141.53 (C², C³). Found, %: C 57.70; H 4.16; Br 35.5. C₉H₉Br. Calculated, %: C 57.75; H 4.81; Br 37.44.

[(Z)-1-Bromoprop-1-en-2-yl]benzene (III). Yield 15%, bp 87–90°C (5 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 2.15 s (3H, 9-H), 6.29 s (1H, 1-H), 7.20–7.45 m (5H, Ph). ¹³C NMR spectrum, δ_{C} , ppm: 17.34 (C⁹), 125.30 (C¹), 125.08, 128.32 (C⁴, C⁵, C⁷, C⁸), 126.62 (C⁶), 133.03 and 141.94 (C², C³).

(*E*)-5-Chlorodec-5-ene (IV) and (*Z*)-5-chlorodec-5-ene (V) (isomer mixture). Overall yield 75%; $IV/V \sim 4:3$ (GLC); bp 71–73°C (5 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 0.70–1.05 m (6H, CH₃), 1.10–1.75 m (8H, CH₂CH₂CH₃), 1.90–2.20 m (2H, CH₂CH=), 2.20–2.35 m (2H, CH₂CCl=), 5.40–5.55 m (1H, =CH–).

(*E*)-5-Chloro-6-[(2-²H₁)ethyl]dec-5-ene (VIa). Yield 83%, bp 80–83°C (5 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 0.88–1.10 m (6H, 8-H, 12-H), 1.28–1.41 m (2H, 4-H), 1.50–1.60 m (2H, 10-H), 2.05–2.15 m (4H, 7-H, 11-H), 2.15–2.28 m (2H, 6-H), 2.15–2.45 m (6H, 3-H, 5-H, 9-H). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 11.79 t (2H, C⁴, *J* = 19 Hz), 13.95 and 13.98 (C⁸, C¹²), 22.01 and 22.76 (C⁷, C¹¹), 26.52 (C¹⁰), 30.26 (C³), 30.89 (C⁵), 31.72 (C⁶), 34.83 (C⁹), 129.17 (C¹), 136.94 (C²). Found, %: C 70.53; Cl 11.8. C₁₂H₂₂ClD. Calculated, %: C 70.67; Cl 10.80.

(*E*)-4-Chloro-5-ethyloct-4-ene (VIb). Yield 80%, bp 73–75°C (5 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 0.88–0.98 m (6H, 7-H, 10-H), 1.02 t (3H, 4-H, J = 7.6 Hz), 1.39–1.65 m (4H, 6-H, 9-H), 2.05–2.45 m (6H, 3-H, 5-H, 8-H). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 12.08 (C⁴), 13.23 and 14.1 (C⁷, C¹⁰), 21.18 and 21.85 (C⁶, C⁹), 26.59 (C³), 34.04 (C⁵), 36.89 (C⁸), 129.19 (C¹), 137.08 (C²). Found, %: C 68.31; H 10.84; Cl 21.2. $C_{10}H_{19}Cl$. Calculated, %: C 68.72; H 10.92; Cl 20.36.

[(*E*)-1-Chloro-2-ethylhex-1-en-1-yl]benzene (VIc). Yield 66%, bp 104–106°C (1 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 0.80 t (3H, 6-H, *J* = 7.2 Hz), 1.16 t (3H, 8-H, *J* = 7.6 Hz), 1.18–1.26 m (4H, 4-H, 5-H), 1.36–1.44 m (4H, 3-H, 7-H), 7.28– 7.38 m (5H, Ph). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 11.94 (C⁸), 13.81 (C⁶), 22.53 (C⁵), 25.93 (C⁷), 30.59 (C³), 32.34 (C⁴), 127.77 (C¹²), 128.15 (C¹⁰, C¹⁴), 129.09 (C¹¹, C¹³), 139.68 (C⁹), 140.31 (C²). Found, %: C 74.53; H 8.53; Cl 16.9. C₁₄H₁₉Cl. Calculated, %: C 74.82; H 8.15; Cl 17.03.

(*E*)-2-Chloro-3-ethyl-*N*,*N*-dimethylhept-2-en-1-amine (VId). Yield 84%, bp 84–85°C (1 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 0.93 t (3H, 7-H, *J* = 6.8 Hz), 1.05 t (3H, 9-H), 1.25–1.48 m (4H, 5-H, 6-H), 2.18 t (2H, 4-H, *J* = 7.2 Hz), 2.20–2.34 m (2H, 8-H), 2.27 s (6H, 10-H, 11-H), 3.13 s (2H, 1-H). ¹³C NMR spectrum, δ_{C} , ppm: 11.91 (C⁹), 13.93 (C⁷), 22.76 (C⁶), 26.86 (C⁸), 30.76, 31.92 (C⁴, C⁵), 44.84 (C¹⁰, C¹¹), 61.74 (C¹), 125.72 (C²), 141.8 (C³). Found, %: C 64.53; H 10.51; Cl 16.4; N 6.35. C₁₁H₂₂ClN. Calculated, %: C 64.71; H 10.78; Cl 17.65; N 6.86.

(*E*)-5-Bromo-6-ethyldec-5-ene (VII). Yield 63%, bp 80–82°C (5 mm). ¹H NMR spectrum (CDCl₃), δ , ppm: 0.90–0.98 m (6H, 8-H, 12-H), 1.02 t (3H, 4-H, J = 7.6 Hz), 1.28–1.63 m (8H, 6-H, 7-H, 10-H, 11-H), 2.08–2.18 m (2H, 3-H), 2.20–2.28 m (2H, 5-H), 2.44– 2.50 m (2H, 9-H). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 12.05 (C⁴), 13.95 and 14.02 (C⁸, C¹²), 21.88, 22.75 (C⁷, C¹¹), 29.87 (C³), 30.87 and 31.01 (C⁶, C¹⁰), 31.83 (C⁵), 37.09 (C⁹), 123.17 (C¹), 143.96 (C²). Found, %: C 58.11; H 9.23; Br 31.2. C₁₂H₂₃Br. Calculated, %: C 58.30; H 9.31; Br 32.39.

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