Reactions of *p*-toluenesulfenyl chloride with enol acetates. The synthetic potential of the resulting adducts

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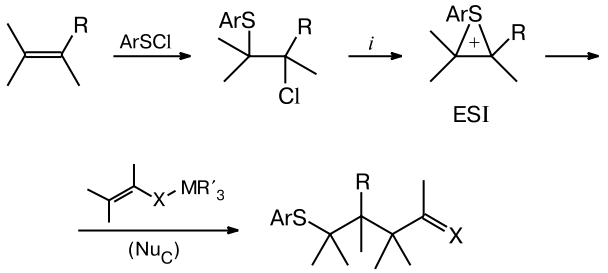
Reactions of vinyl and propen-2-yl acetates with *p*-toluenesulfenyl chloride afforded the corresponding α-chloro-β-(*p*-tolyl)thioalkyl acetates in nearly quantitative yields. These adducts reacted with some C-nucleophiles in the presence of Lewis acids to give the corresponding alkylation products.

Key words: enol acetates, arenesulfenyl chlorides, electrophilic addition, C-nucleophiles, β-arylthioalkylation.

Electrophilic addition of arenesulfenyl halides to a carbon—carbon multiple bond belongs to the well-studied reactions of alkenes of various types.[1] As a rule, this reaction occurs under mild conditions, giving rise to aryl β-haloalkyl sulfides in nearly quantitative yields. Apart from being suitable for functionalization of a double bond, this reaction is preparatively valuable because the resulting adducts can also be used as electrophilic reagents, which react in the presence of Lewis acids with a broad range of C-nucleophiles (Nu_C) of the π-donor type to form a new carbon—carbon bond.[2] Presumably, this reaction proceeds through the formation of a cationoid intermediate of the episulfonium ion (ESI) type (Scheme 1).

Scheme 1



i. Lewis acid.

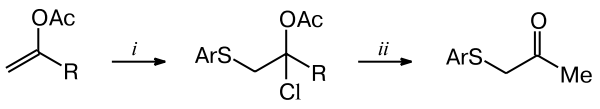
R = Alk, Ar, OAlk; X = O, CH_2; M = Si, Sn; R′ = Alk

This type of a transformation has been studied in most detail for adducts obtained from alkoxyalkenes (R = OAlk) since, in particular, the resulting coupling products are promising for sequential intra- and intermolecular transformations leading to polyfunctional derivatives, as has been shown by us earlier.[2,3]

To extend the area of synthetic application of these transformations, it was expedient to study the possible use of acetoxy alkenes (R = OAc) as the starting unsaturated substrates. The literature data on reactions of arenesulfenyl halides with such alkenes are virtually lacking. Indeed, the reaction of benzenesulfenyl chloride with vinyl acetate was mentioned in only one study:[4] its rate constant was reported to be two to three orders of magnitude lower than the rate constants for common alkenes. However, no properties of the adduct obtained were reported, except for its ^{1}H NMR spectrum.

Vinyl ethers are known[5] to virtually instantaneously react with ArSCl even at −70 °C. We found that vinyl acetate **1** does not react with *p*-TolSCl under these conditions (the characteristic color of sulfenyl halide persisted for 2 h). However, the room-temperature reaction was completed over 5 min. The resulting adduct **2** was isolated in 95% yield (Scheme 2). Its structure was established from analytical and spectroscopic data. Interestingly, adduct **2** is hydrolytically stable and withstands column chromatography on silica gel, while analogous products ob-

Scheme 2



1, **3** **2**, **4** **5**

i. ArSCl; *ii*. H_2O for R = Me.

R = H (**1**, **2**), Me (**3**, **4**), Ar = *p*-Tol.

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tained from vinyl ethers usually cannot be isolated in the individual state.[5] As expected, the reaction of propen-2-yl acetate **3** with *p*-TolSCl occurs sufficiently easily even at −30 °C. However, on attempted isolation and purification, the corresponding adduct **4** readily hydrolyzed to give (*p*-tolylthio)acetone (**5**) in 95% yield. The *in situ* obtained product **4** was characterized by ^{1}H NMR data; the absence of signals of impurities in its spectrum allowed subsequent use of this adduct without purification.

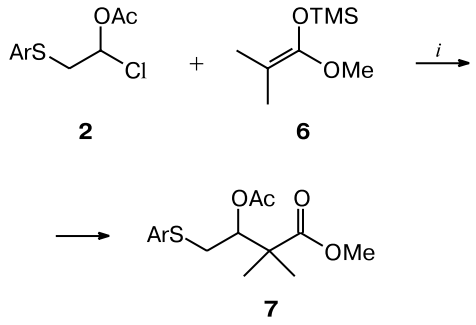
As already noted, β-arylthio-α-chloroalkyl ethers (obtained from arenesulfenyl chlorides and alkoxyalkenes) form reactive cationoid ESI intermediates in the presence of Lewis acids even at −70 °C (see Scheme 1); these ions alkylate various C-nucleophiles (Nu_C).[2,3] As expected, because of the deactivating effect of the acetoxy substituent, β-arylthio-α-chloroalkyl acetates **2** and **4** proved to be substantially less reactive in analogous transformations. For instance, adduct **2** remained intact on treatment with such Lewis acids as $TiCl_4$, TMSOTf, and $LiClO_4/MeNO_2$ in CH_2Cl_2 over the temperature range from −70 to 0 °C (TLC data). In an attempt to use adduct **2** for the *in situ* generation of a cationoid intermediate under the action of $TiCl_4$ at room temperature in the presence of such a reactive C-nucleophile as dimethylketene methyl trimethylsilyl acetal **6**, no formation of the expected alkylation product **7** was observed (see below); after several hours, complete resinification of the reaction mixture occurred. Encouraging results were obtained with the use of stronger Al-containing Lewis acids. Indeed, the reaction with Et_2AlCl or $EtAlCl_2$ afforded adduct **7**, though in low yield (5—10%). The mixed systems Et_2AlCl—TMSOTf or methylaluminum bis(4-bromo-2,6-di-*tert*-butylphenolate) (MABR)—TMSOTf are known[6] to be especially strong Lewis acids. Use of these systems in the reaction of compound **2** with acetal **6** increased the yield of product **7** to 50—60%.

However, in these cases as well, the reaction occurred only at room temperature, which reduces its overall efficiency and the possibility of its application to less reactive Nu_C. A more successful alternative procedure involves silver salts as specific Lewis acids which irreversibly bind the leaving chloride anion. When adduct **2** was treated with silver triflate in CH_2Cl_2, a precipitate formed even at −15 °C. The direct reaction product could be expected to be a cationoid acetoxy-ESI intermediate (Scheme 4). However, the latter seems to be kinetically unstable, because subsequent treatment of the reaction mixture with nucleophiles (MeOH or Nu_C) gave no expected "quenching" products from the suggested intermediate; instead, β-arylthio acylal **8** was isolated as the major product instead. We did not investigate the reaction mechanism involved; most likely, this product is formed in the reaction of the generated intermediate with the starting substrate **2** (Scheme 4).

The reaction of adduct **2** with AgOTf was found to be synthetically useful in the presence of such C-nucleophiles as ketene silyl acetals (**6** or **9**), enol silyl ethers (*e.g.*, **10**), or allylstannanes (**11** or **12**). These nucleophiles trap the *in situ* generated cationoid to give the corresponding alkylation products **7** and **13**—**16** in good or satisfactory yields (Scheme 5). The preparation of adduct **7** in acetonitrile and nitromethane demonstrated that these solvents are also suitable for this reaction.

The presence of an additional methyl group in adduct **4** substantially alters its properties compared to product **2**. Our attempts to carry out the reaction of adduct **4** with acetal **6** in the presence of such Lewis acids as TMSOTf, $TiCl_4$, Et_2AlCl, and $LiClO_4/MeNO_2$ in the temperature range from −78 to 20 °C failed: in all cases, the major product was (*p*-tolylthio)acetone **5** isolated by conventional work-up of the reaction mixture (ether—aqueous $NaHCO_3$). Since compound **4** is easily hydrolyzed in aqueous media to compound **5** (see above), these results allowed, by themselves, no conclusions to be drawn either about the assumed generation of a cationoid intermediate from compound **4** or about its reactivity. For this reason, we studied the reaction of compound **4** with TMSOTf (1 : 1 ratio) in CD_2Cl_2 by ^{1}H NMR spectroscopy. It was found that the spectrum of adduct **4** at −70 °C remains unchanged for 1 h. However, signals for ketone **5** appeared at −30 °C (15 min, **4** : **5** = 8 : 1) and became dominant at higher temperatures (0 °C, 15 min, **4** : **5** = 1 : 2; 10 °C, 5 min, **4** : **5** = 20 : 1). Simultaneously, TMSCl and AcOTf accumulated in the reaction mixture (signals at δ 0.4 and 2.6, respectively). Hence, the chloride anion and the acetyl cation are eliminated concertedly under these conditions (Scheme 6).

Scheme 3

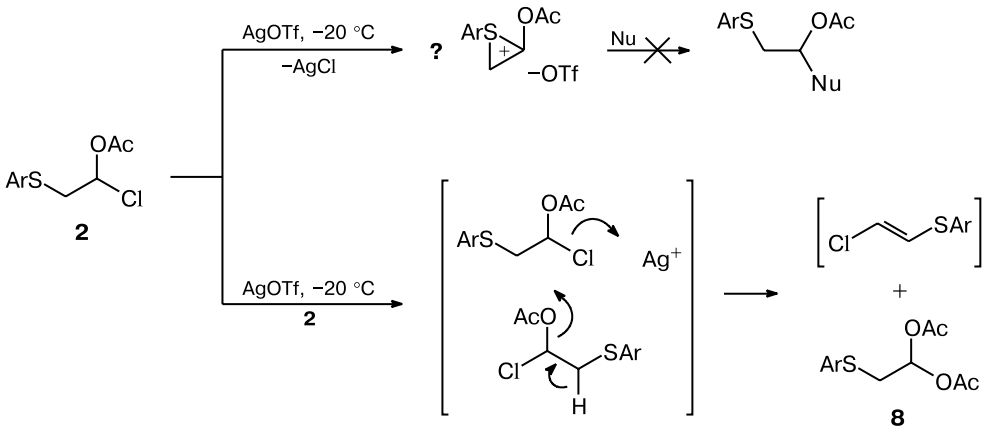


Ar = *p*-Tol

Reagents and conditions: Lewis acid, 20 °C, CH_2Cl_2.

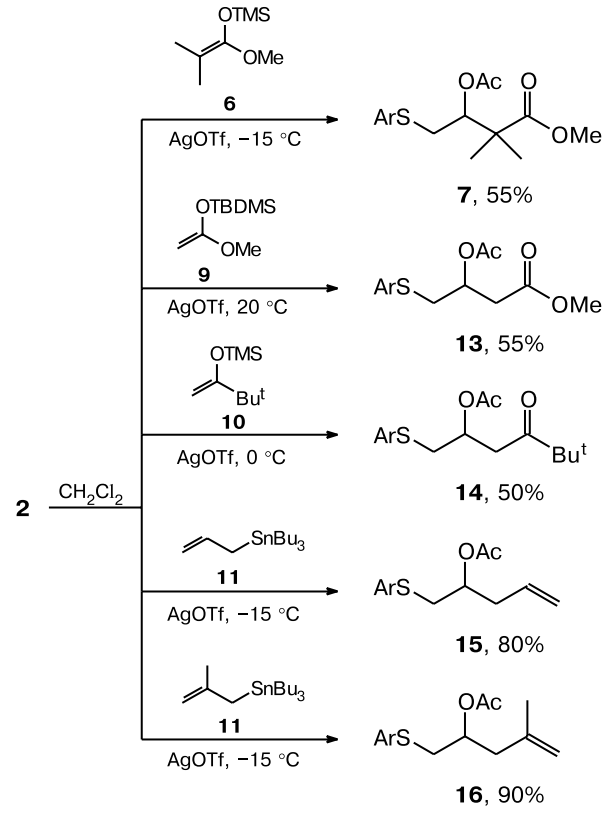
Lewis acid	Yield (%)
Et_2AlCl	10
$EtAlCl_2$	5
Et_2AlCl—TMSOTf	50
MABR—TMSOTf	60

Scheme 4

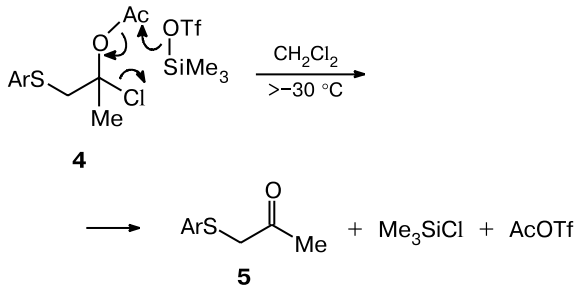


Ar = *p*-Tol

Scheme 5



Scheme 6

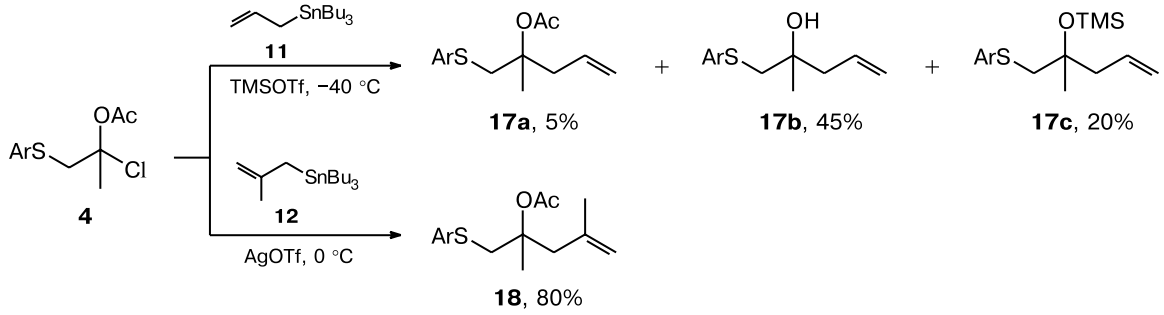


Ar = *p*-Tol

It also follows from the results obtained that a search for the possibility of using the adduct **4**—Lewis acid system as an electrophile in reactions with Nu_C is stringently limited: the reaction temperature should be as low as possible and silyl-containing Nu_C cannot be used (in reactions with the latter, TMSOTf is regenerated). Indeed, the reaction of adduct **4** with allylstannane **11** in the presence of TMSOTf at −40 °C gave a mixture of allylation products **17a**—**c** in 70% total yield. However, compound **4** did not react with dimethylketene trimethylsilyl acetal **6** under the same conditions; ketone **5** was isolated as the only product. In contrast, the reaction of compound **4** with methallylstannane **12** as Nu_C in the presence of AgOTf at 0 °C smoothly gave product **18** in good yield.

A comparison of the results obtained with the known data on the properties of ArSCl—alkene adducts[2,7] suggests that the presence of an acetoxy substituent in adducts of the type **2** and **4** substantially reduces their abilities to generate cationoid intermediates under the action of Lewis acids. Moreover, it is incorrect to regard these intermediates in Lewis acid—catalyzed reactions of adducts **2** and **4** with C-nucleophiles as kinetically stable species, as was the case in previous examples (see above). Apparently, the transformations of adducts **2** and **4** into alkylation products of the aforementioned type (see Schemes 5, 7) would be more correctly described in terms of the S_N2 mechanism. Note that according to the literature data,[8] nucleophilic substitution reactions in the series of related compounds such as α-chloroalkyl acetates

Scheme 7



Ar = *p*-Tol

can follow both the S_N1 and S_N2 mechanisms depending on the nature of substrates and reagents.[8]

Thus, easily accessible adducts from acetoxy alkenes and arenesulfenyl chlorides can be used as electrophilic reagents for the formation of a new carbon—carbon bond. This makes it promising to further develop both preparative conditions for these reactions and the synthetic potential of the resulting polyfunctional adducts.

Experimental

All experiments were carried out in an atmosphere of dry argon in solvents dried according to standard procedures. TLC analysis was performed on Merck chromatographic plates (SiO_2, 220—440 mesh, ASTM). ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker WM-250 instrument. Commercial $EtAlCl_2$, Et_2AlCl, $AlMe_3$, AgOTf, TMSOTf (Aldrich), allyl(tri-*n*-butyl)stannane (Lancaster Co.), 4-bromo-2,6-di-*tert*-butylphenol, vinyl acetate, and propen-2-yl acetate (Acros Co.) were used. Tri-*n*-butyl(methallyl)stannane was prepared as described earlier.[9] Siloxyalkenes employed as C-nucleophiles were prepared according to a known procedure.[10] *p*-Toluenesulfenyl chloride was synthesized by the reaction of 4-methylbenzenethiol with sulfuryl chloride.[11]

1-Chloro-2-(4-tolylthio)ethyl acetate (2). *p*-Toluenesulfenyl chloride (135 mg, 0.84 mmol) was added at 20 °C to a solution of vinyl acetate (**1**) (73 mg, 0.84 mmol) in CH_2Cl_2 (5 mL). Stirring for 5 min resulted in complete decoloration of the solution. The solvent was removed *in vacuo* and the residue was chromatographed on SiO_2 with hexane—ethyl acetate (10 : 1) as the eluent to give adduct **2** (200 mg, 95%) as a colorless oil, R_f 0.7 (hexane—ethyl acetate, 5 : 1). ^1H NMR, δ: 7.35, 7.15 (both d, 4 H each, Ar); 6.45 (dd, X part of the ABX system, 1 H, CH, J_1 = 3.6 Hz, J_2 = 8.5 Hz); 3.40 (m, AB part of the ABX system, 2 H, CH_2, J_{AB} = 14 Hz, J_{AX} = 3.6 Hz, J_{BX} = 8.5 Hz); 2.35 (s, 3 H, Me—Ar); 2.05 (s, 3 H, MeCO). ^{13}C NMR, δ: 169.8, 135.3, 131.6, 130.6, 129.6, 81.5, 42.2, 20.67, 20.1. Found (%): C, 53.91; H, 5.47; Cl, 14.52; S, 13.05. $C_{11}H_{13}Cl_2S$. Calculated (%): C, 53.98; H, 5.35; Cl, 14.49; S, 13.10.

2-Chloro-1-(4-tolylthio)propan-2-yl acetate (4). Adduct **4** was obtained analogously from propen-2-yl acetate (**3**) (200 mg). The adduct was not purified by chromatography because of its instability. The solvent was removed to give adduct **4** (500 mg, 96%) as a slightly colored oil, R_f 0.4 (hexane—ethyl acetate, 5 : 1). Elemental analysis data were irreproducible because of the instability of the adduct. Its purity was confirmed by the data from the ^1H NMR spectrum which was free from signals of impurities. ^1H NMR, δ: 7.35, 7.15 (both d, 4 H each, Ar); 4.00, 3.40 (both d, AB system, 2 H, CH_2, J_{AB} = 14 Hz); 2.25 (s, 3 H, Me—Ar); 1.95 (s, 3 H, MeCOO); 1.75 (s, 3 H, Me).

1-(4-Tolylthio)propan-2-one (5). When chromatographed on silica gel, adduct **4** obtained from acetate **3** as described above completely converted into ketone **5**. The yield of propanone **5** as a colorless oil from acetate **3** (100 mg) was 170 mg (95%), R_f 0.4 (hexane—ethyl acetate, 5 : 1). ^1H NMR, δ: 7.35, 7.15 (dd, 4 H, Ar); 3.60 (s, 2 H, CH_2); 2.30 (s, 3 H, Me—Ar); 2.25 (s, 3 H, MeCO).

Methyl 3-acetoxy-2,2-dimethyl-4-(4-tolylthio)butanoate (7). ***A.*** Ketene silyl acetal **6** (320 mg, 2.2 mmol) and a solution of Et_2AlCl—TMSOTf (2.52 mmol, 1 : 1) in hexane (2.5 mL) were added at −15 °C to a stirred solution of adduct **2** (0.84 mmol) in CH_2Cl_2 (5 mL) obtained *in situ* as described above. After 30 min, the temperature was elevated to 20 °C and the reaction mixture was left for 16 h and then poured into a stirred mixture of saturated $NaHCO_3$ and ether. The product from the aqueous layer was additionally extracted with hexane (2×25 mL) and the combined extracts were dried with $CaCl_2$. The solvent was evaporated and the residue was chromatographed on SiO_2 with hexane—ethyl acetate (10 : 1) as the eluent. The yield of compound **7** as a colorless oil was 122 mg (50%), R_f 0.3 (hexane—ethyl acetate, 5 : 1). ^1H NMR, δ: 7.30, 7.10 (both d, 4 H each, Ar); 5.40 (dd, X part of the ABX system, 1 H, CH, J_1 = 3.6 Hz, J_2 = 7.5 Hz); 3.60 (s, 3 H, MeOCO); 2.95 (m, AB part of the ABX system, 2 H, CH_2, J_{AB} = 14 Hz, J_{AX} = 3.6 Hz, J_{BX} = 7.5 Hz); 2.30 (s, 3 H, Me—Ar); 2.00 (s, 3 H, MeCOO); 1.25 (s, 6 H, Me_2). Found (%): C, 61.83; H, 7.33; S, 10.23. $C_{16}H_{22}O_4S$. Calculated (%): C, 61.91; H, 7.14; S, 10.33.

B. Ketene silyl acetal **6** (160 mg, 1.1 mmol) was added to a stirred solution of adduct **2** (0.84 mmol) in CH_2Cl_2 (5 mL) obtained *in situ* as described above. The mixture was cooled to −20 °C and AgOTf (260 mg, 1 mmol) was added in portions for 40 min. The precipitate of AgCl was formed. After 1 h, the reaction mixture contained no starting adduct **2** (TLC data). Conventional workup followed by column chromatography gave adduct **7** in 55% yield. Products **7** from procedures ***A*** and ***B*** were completely identical.

1,1-Diacetoxy-2-(4-tolylthio)ethane (8). Silver triflate (260 mg, 1 mmol) was added at 20 °C to a stirred solution of

adduct **2** (0.84 mmol) in CH_2Cl_2 (5 mL) obtained *in situ* as described above. The reaction mixture was stirred for 30 min and treated with a mixture of saturated $NaHCO_3$ and ether. The product from the aqueous layer was additionally extracted with hexane (2×25 mL) and the combined organic extracts were dried over $CaCl_2$. The solvent was evaporated and the residue was subjected to column chromatography with hexane—ethyl acetate (10 : 1) as the eluent to give product **8** (70 mg, 62%) as a colorless oil, R_f 0.4 (hexane—ethyl acetate, 5 : 1). ^{1}H NMR, δ: 7.30, 7.10 (both d, 4 H each, Ar); 6.85 (t, 1 H, CH, J = 5.5 Hz); 3.20 (d, 2 H, J = 5.5 Hz); 2.30 (s, 3 H, MeAr); 2.00 (s, 6 H, MeCOO). ^{13}C NMR, δ: 137.1, 131.1, 129.7, 89.1; 37.4, 20.9, 20.5. Found (%): C, 59.01; H, 6.3; S, 11.24. $C_{13}H_{16}O_4S$. Calculated (%): C, 58.19; H, 6.01; S, 11.95.

Methyl 3-acetoxy-4-(4-tolylthio)butanoate (13) was obtained from ketene silyl acetal **9** as a C-nucleophile at 20 °C as described in procedure ***B*** for ester **7**. The yield of adduct **13** was 55%. ^{1}H NMR, δ: 7.35, 7.10 (both d, 4 H each, Ar); 5.30 (m, 1 H, CH); 3.70 (s, 3 H, MeOCO); 3.15 (m, SCH_2, AB part of the ABX system, 2 H, CH_2, J_{AB} = 14 Hz, J_{AX} = 6.7 Hz, J_{BX} = 6.1 Hz); 2.75 (m, $COCH_2$, AB part of the ABX system, 2 H, CH_2, J_{AB} = 16 Hz, J_{AX} = 7.9 Hz, J_{BX} = 5.6 Hz); 2.30 (s, 3 H, Me—Ar); 1.95 (s, 3 H, MeCOO). Found (%): C, 60.05; H, 6.63; S, 11.23. $C_{14}H_{18}O_4S$. Calculated (%): C, 59.55; H, 6.43; S, 11.36.

5,5-Dimethyl-4-oxo-1-(4-tolylthio)hexan-2-yl acetate (14) was obtained analogously at 0 °C from silyl vinyl ether **10** as a C-nucleophile. The yield of adduct **14** was 50%. ^{1}H NMR, δ: 7.30, 7.10 (both d, 4 H each, Ar); 5.40 (quint, 1 H, CH, J = 6.1 Hz); 3.18 (d, SCH_2, 2 H, CH_2, J = 6.1 Hz); 2.92 (d, $COCH_2$, 2 H, CH_2, J = 6.1 Hz); 2.30 (s, 3 H, Me—Ar); 1.90 (s, 3 H, MeCOO); 1.10 (s, 9 H, Bu^t). Found (%): C, 66.50; H, 7.93; S, 10.23. $C_{17}H_{24}O_3S$. Calculated (%): C, 66.20; H, 7.84; S, 10.39.

1-(4-Tolylthio)pent-4-en-2-yl acetate (15) was obtained analogously at −15 °C from allyl(tri-*n*-butyl)stannane **11** as a C-nucleophile. The yield of adduct **15** was 80%. ^{1}H NMR, δ: 7.30, 7.10 (both d, 4 H each, Ar); 5.85 (m, 1 H, CH=); 5.10 (m, 2 H, $=CH_2$); 4.50 (m, 1 H, CH); 3.0 (m, 2 H, CH_2); 2.35 (m, 2 H, CH_2); 2.30 (s, 3 H, Me—Ar); 2.05 (s, 3 H, MeCOO). Found (%): C, 67.01; H, 7.03; S, 12.45. $C_{14}H_{18}O_2S$. Calculated (%): C, 67.16; H, 7.25; S, 12.81.

4-Methyl-1-(4-tolylthio)pent-4-en-2-yl acetate (16) was obtained analogously at −15 °C from tri-*n*-butyl(methallyl)stannane **12** as a C-nucleophile. The yield of adduct **16** was 90%. ^{1}H NMR, δ: 7.30, 7.10 (both d, 4 H each, Ar); 5.15 (m, 1 H, CHOAc); 4.82, 4.72 (both s, 2 H each, $=CH_2$); 3.03 (m, 2 H, CH_2); 2.35 (m, 2 H, CH_2); 2.30 (s, 3 H, Me—Ar); 1.95 (s, 3 H, MeCOO); 1.70 (s, 3 H, Me). Calculated (%): C, 68.14, H, 7.62, S, 12.13. $C_{14}H_{20}O_2S$. Found (%): C, 69.01, H, 7.7; S, 12.10.

2,4-Dimethyl-1-(4-tolylthio)pent-4-en-2-yl acetate (18). Methallylstannane **12** and AgOTf (358 mg, 1.5 mmol) were added at 0 °C to a solution of adduct **4** obtained *in situ* from propen-2-yl acetate **3** (100 mg, 1 mmol) and *p*-TolSCl (158 mg, 1 mmol) in CH_2Cl_2 (5 mL). The reaction mixture was kept at this temperature for 12 h and then treated according to a standard procedure. Purification by column chromatography gave adduct **18** (220 mg, 80%) as a colorless oil. ^{1}H NMR, δ: 7.30, 7.10 (both d, 4 H each, Ar); 4.78, 4.92 (both m, 1 H each, CHOAc); 4.82, 4.72 (both s, $=CH_2$); 3.53, 3.35 (both m, AB, 2 H, CH_2, J_{AB} = 1 Hz, J_{AB} = 16 Hz); 2.80, 2.50 (both m, AB, 2 H, CH_2, J_{AB} = 16 Hz); 2.30 (s, 3 H, Me—Ar); 1.95 (s, 3 H, MeCOO); 1.80 (s, 3 H, Me); 1.50 (s, 3 H, Me). Found (%): C, 68.73; H, 8.21; S, 11.30. $C_{16}H_{22}O_2S$. Calculated (%): C, 69.02; H, 7.96; S, 11.52.

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