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Lewis Acid Catalyzed Ene Reactions of Ethynyl *p*-Tolyl Sulfone

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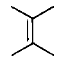
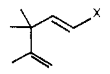
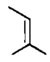
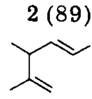
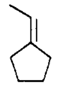
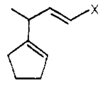
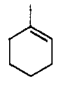
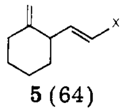
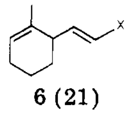
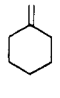
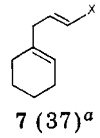
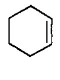
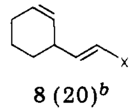
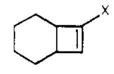
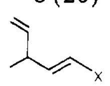
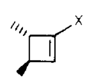
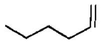
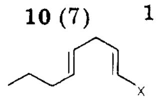
Ethynyl *p*-tolyl sulfone undergoes $EtAlCl_2$-catalyzed ene reactions with alkenes to give 1,4-dienyl *p*-tolyl sulfones in good yield.

We have recently described the Lewis acid catalyzed ene and [2 + 2] cycloaddition reactions of ethynyl esters and ketones with alkenes.[2,3] Depending on the substitution pattern of the alkene, good yields of ene adducts and/or cyclobutenes are obtained at 25 °C. The adducts which result from these reactions have been used for the syntheses of the A1 component of the female sex pheromone of the California red scale[4] and of novel inside–inside bicyclic compounds.[5]

We report here the analogous reactions of ethynyl sulfones with alkenes (Scheme I). The reaction introduces a functionalized two-carbon fragment, a vinyl sulfone, which is a versatile synthon.[6] The results of $EtAlCl_2$-catalyzed reactions of ethynyl *p*-tolyl sulfone (**1**) with alkenes are shown in Table I. These reactions proceed at 25 °C in benzene or other aromatic solvents. The reaction proceeds best for highly substituted, i.e., electron rich, alkenes. Good yields of ene adducts are obtained from all alkenes with at least one disubstituted carbon. Monosubstituted alkenes give low yields of ene adducts while 1,2-disubstituted alkenes give low yields of a mixture of ene adduct and cyclobutene. A similar propensity for 1,2-disubstituted alkenes to give cyclobutenes was observed with methyl propiolate[2] and 3-butynone.[3]

This reaction is very susceptible to changes in conditions. Use of CH_2Cl_2 as solvent or $AlCl_3$ as Lewis acid results in complex mixtures of products. The stabilization of the $1{\cdot}EtAlCl_2$ complex by aromatic solvents and the ability of $EtAlCl_2$ to act as a proton scavenger minimize the side reactions. However, even under optimal conditions, methylenecyclohexane is slowly isomerized to 1-methylcyclohexene so that mixtures of **5**, **6**, and **7** are formed. In all cases just under 1 equiv of $EtAlCl_2$ is used since the product alkenyl sulfone is more basic than **1** and complexes preferentially to $EtAlCl_2$.[7]

Table I. Adducts from the $EtAlCl_2$-Catalyzed Reactions of Ethynyl *p*-Tolyl Sulfone (1) with Alkenes

alkene	adducts[c] (% yield)
	 2 (89)
	 3 (66)
	 4 (87)
	 +  5 (64) 6 (21)
	 7 (37)[a]
	 +  8 (20)[b] 9 (11)[b]
	 +  10 (7) 11 (7)
	 12 (19)

[a] Consists of 80% **7** and 20% of a 3:1 mixture of **5** and **6**. [b] A 19% yield of **14** and cyclohexyl benzene were also obtained. [c] X = *p*-tolylsulfonyl.

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(1) Fellow of the Alfred P. Sloan Foundation, 1979–1981.

(2) Snider, B. B.; Rodini, D. J.; Conn, R. S. E.; Sealfon, S. *J. Am. Chem. Soc.* **1979**, *101*, 5283. Snider, B. B.; Roush, D. M.; Rodini, D. J.; Gonzalez, D.; Spindell, D. *J. Org. Chem.* **1980**, *45*, 2773.

(3) Snider, B. B.; Brown, L. A.; Conn, R. S. E.; Killinger, T. *Tetrahedron Lett.* **1977**, 2831.

(4) Snider, B. B.; Rodini, D. J. *Tetrahedron Lett.* **1978**, 1399.

(5) Snider, B. B.; Hrib, N. J. *Tetrahedron Lett.* **1977**, 1725.

(6) Magnus, P. *Tetrahedron* **1977**, *33*, 2019.

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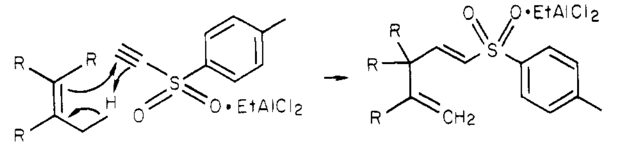
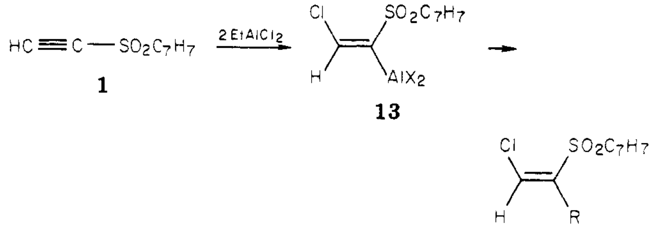
Scheme I



In some cases when more than 1 equiv of $EtAlCl_2$ is used, *cis*-2-chloroethenyl *p*-tolyl sulfone **14** is also obtained. Treatment of **1** with 2 equiv of $EtAlCl_2$ in the absence of alkene for 5 days at 25 °C followed by quenching with H_2O gives a 58% yield of **14**. Chloride **14** appears to arise from a novel chloralumination[8] of **1** since quenching with D_2O gives a 37% yield of **15**. Although HCl is known to add to **1** to give **14**,[9] this is not the origin of **15** since quenching the reaction mixture with D_2O after short reaction times gives only recovered **1**. Attempted trapping of **13** with I_2, CH_3I or CO_2 was unsuccessful.



1

13

14, R = H
15, R = D

The results described above further indicate the broad scope of the Lewis acid catalyzed reactions of electron-deficient alkynes with alkenes.

Experimental Section

Benzene was dried by distillation from sodium/benzophenone. Ethylaluminum dichloride was obtained from Texas Alkyls (25.5% in heptane, *d* = 0.772, 1.57 M).

Synthesis of Ethynyl *p*-Tolyl Sulfone (1). *cis*-1,2-Bis(*p*-tolylthio)ethylene was prepared by a modification of the procedure of Parham and Heberling.[10] *cis*-1,2-Dichloroethylene (20.0 g, 0.201 mol) was added over a period of 25 min to a solution of *p*-thiocresol (51.5 g, 0.415 mol) and 41.0 g of KOH in 600 mL of EtOH. The resulting brown solution was stirred for 30 min at 25 °C and heated under reflux for 6 h. The solvent was distilled off in vacuo and the yellow crystalline mass added to 400 mL of H_2O. This was extracted with 400 mL of ether which was concentrated to give a suspension of product in about 50 mL of solvent. Filtration gave 45.2 g (81%) of product, mp 87.5–89.5 °C, which was used for the next step.

(*p*-Tolylthio)acetylene was prepared by a modification of the procedure of Parham and Stright.[11] To a stirred solution of 250 mL of 1.6 M butyllithium in hexane was added 10 mL of a solution of 44 g (0.163 mol) of bis(*p*-tolylthio)ethylene in 400 mL of anhydrous ether. The light orange reaction mixture was cooled to −20 °C and the remaining bis(*p*-tolylthio)ethylene solution was added slowly. The reaction mixture was kept at 0 °C for 3 h, an additional 50 mL of 1.6 M butyllithium solution was added, and the mixture was then kept at 20 °C for 4 h. The reaction was carefully quenched with 200 mL of water and refluxed for 25 min. The organic layer was separated and the solvent removed by evaporation in vacuo to give 29 g of (*p*-tolylthio)acetylene which was used without further purification since it was greater than 90% pure by NMR spectroscopy: NMR ($CDCl_3$) δ 7.0–7.4 (m, 4), 3.14 (s, 1), 2.26 (s, 3).

***p*-Tolyl ethynyl sulfone (1)** was prepared by a modification of the procedure of Maioli and Modena.[9] A slurry of 70 g of *m*-chloroperbenzoic acid (85% pure, 0.43 mol) in 200 mL of chloroform was added to a solution of 29 g of crude (*p*-tolylthio)acetylene in 100 mL of chloroform at 0 °C. The addition rate was controlled to maintain the temperature below 10 °C. The milky-white reaction mixture was stirred at 20 °C for 18 h, extracted with two 200-mL portions of saturated sodium sulfite solution, 100 mL of 5% sodium bicarbonate solution, and 100 mL of 2% hydrochloric acid, dried ($MgSO_4$), and concentrated in vacuo to give 25.3 g of crude **1**. Recrystallization from cyclohexane gave 18.1 g of white needles: mp 74–74.7 °C [lit.[12] mp 74–75 °C]; NMR ($CDCl_3$) δ 7.91 (br d, 2, *J* = 8 Hz), 7.40 (br d, 2, *J* = 8 Hz), 3.51 (s, 1), 2.50 (s, 3); IR (CCl_4) 3305, 3075, 3060, 2935, 2070, 1600, 1355, 1310, 1300, 1185, 1170, 1090, 695, 640, 545 cm^{-1}.

General Procedure for Reaction of Alkenes with 1. Alkene (1–2 equiv) was added dropwise to a solution of sulfone **1** (0.360 g, 2 mmol) and $EtAlCl_2$ (1.2 mL of 1.57 M, 0.9 equiv) in 10 mL of anhydrous benzene under nitrogen at 0 °C. The solution was allowed to warm to 25 °C and was monitored by TLC.

The reaction mixture was quenched by cautious addition of 50 mL of 5% hydrochloric acid and extracted twice with 30 mL of ether. The combined organic layers were washed with 30 mL of water. The combined aqueous layers were washed with 30 mL of ether. The combined organic layers were dried (Na_2SO_4) and the solvent was removed under reduced pressure.

Reaction of 2,3-Dimethyl-2-butene with 1. Reaction with 2,3-dimethyl-2-butene (0.28 g, 1.5 equiv) for 8 days gave 0.556 g of crude product (mp 47.5–51 °C) which NMR spectroscopy showed to be ~95% **2**. Chromatography of 0.205 g on 30 g of silica gel with 1:1 pentane–ether as eluant gave 0.174 g (89%) of **2** as white needles: mp 48.5–49.5 °C; NMR ($CDCl_3$) δ 7.75 (br d, 2, *J* = 8 Hz), 7.34 (br d, 2, *J* = 8 Hz), 6.96 (d, 1, *J* = 15 Hz), 6.23 (d, 1, *J* = 15 Hz), 4.85 (br s, 1), 4.81 (br s, 1), 2.46 (s, 3), 1.69 (s, 3), 1.12 (s, 6); IR (CCl_4) 3050, 2950, 2860, 1600, 1530, 1325, 1290, 1150, 1089, 975, 900, 650 cm^{-1}. Anal. Calcd for $C_{15}H_{20}O_2S$: C, 68.14; H, 7.63; S, 12.13. Found: C, 68.48; H, 7.86; S, 11.96.

Reaction of 2-Methyl-2-butene with 1. Reaction with 2-methyl-2-butene (0.32 g, 2.3 equiv) for 6 days gave 0.528 g of crude product which NMR spectroscopy indicated to be ~80% **3**. Chromatography of 200 mg on silica gel with 1:1 pentane–ether as eluant gave 0.126 g (66%) of pure **3**: NMR ($CDCl_3$) δ 7.77 (br d, 2, *J* = 8 Hz), 7.33 (br d, 2, *J* = 8 Hz), 6.95 (dd, 1, *J* = 16, 6.5 Hz), 6.30 (dd, 1, *J* = 16, 2 Hz), 4.83 (br s, 1), 4.79 (br s, 1), 3.02 (dq, 1, *J* = 6.5, 7 Hz), 2.45 (s, 3) 1.69 (s, 3), 1.19 (d, 3, *J* = 7 Hz); IR (neat) 3040, 2950, 2850, 1600, 1320, 1305, 1280, 1145, 1085, 810, 655 cm^{-1}; mol wt calcd for $C_{14}H_{18}O_2S$ 250.1027, found 250.1015.

Reaction of Ethylidenecyclopentane with 1. Reaction with ethylidenecyclopentane (0.31 g, 1.5 equiv) for 5 days gave 0.665 g of crude product which NMR spectroscopy showed to be ~90% **4**. Chromatography of 0.415 g on 30 g of silica gel with 2:1 pentane–ether as eluant gave 0.301 g (87%) of **4**: NMR ($CDCl_3$) δ 7.75 (br d, 2, *J* = 8 Hz), 7.32 (br d, 2, *J* = 8 Hz), 6.97 (dd, 1, *J* = 15, 7 Hz), 6.28 (dd, *J* = 15, 2 Hz), 5.43 (br s, 1) 3.12 (br dq, 1, *J* = 7, 7 Hz), 2.45 (s, 3), 2.35–1.75 (m, 6), 1.20 (d, 3, *J* = 7 Hz); IR (neat) 3050, 2960, 2935, 2850, 1640, 1600, 1570, 1310, 1302, 1290, 1145, 1085, 810, 670 cm^{-1}; mol wt calcd for $C_{16}H_{20}O_2S$ 276.1184, found 276.1186.

Reaction of 1-Methylcyclohexene with 1. Reaction with 1-methylcyclohexene (0.41 g, 2 equiv) for 10 days gave 0.585 g of crude product which NMR spectroscopy showed to be ~90% ene adducts **5** and **6**. Chromatography of 0.200 g on 30 g of silica gel with 1:1 pentane–ether as eluant gave 0.160 g (85%) of an inseparable 3:1 mixture of **5** and **6**: NMR ($CDCl_3$) (**5**) δ 7.79 (br d, 2, *J* = 8 Hz), 7.32 (br d, 2, *J* = 8 Hz), 7.07 (dd, *J* = 7, 15 Hz), 6.34 (d, 1, *J* = 15 Hz), 4.75 (br s, 1), 4.53 (br s, 1), 3.92 (m, 1), 2.46 (s, 3), 2.3–1.2 (m, 8); NMR ($CDCl_3$) (**6**) δ 7.79 (br d, 2, *J* =

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[7] Puchkova, V. V.; Gur'yanova, E. N.; Laba, V. I.; Prilezhaeva, E. N. *Bull. Acad. Sci. USSR* **1973**, 111.

[8] Isolated examples of chloropalladation,[8a] chloromercuation[8b], and chloroboration[8c] of alkynes are known. (a) Maitlis, P. M. "Organic Chemistry of Palladium"; Academic Press: New York, 1971; Vol. 1, pp 79–80. (b) Larock, R. C. *J. Organometal. Chem. Libr.* **1976**, *1*, 263. (c) Onak, T. "Organoborane Chemistry"; Academic Press: New York, 1975; pp 35–36.

[9] Maioli, L; Modena, G. *Ric. Sci.* **1959**, *29*, 1931; *Chem. Abstr.* **1960**, *54*, 10928*g*.

[10] Parham, W. E.; Heberling, J. *J. Am. Chem. Soc.* **1955**, *77*, 1175.

[11] Parham, W. E.; Stright, P. L. *J. Am. Chem. Soc.* **1956**, *78*, 4783.

[12] Montanari, F. *Gazz. Chim. Ital.* **1956**, *86*, 406; *Chem. Abstr.* **1958**, *52*, 8999*h*.

8 Hz), 7.32 (br d, 2, J = 8 Hz), 6.96 (dd, 1, J = 9, 15 Hz), 6.30 (d, 1, J = 15 Hz), 5.58 (m, 1), 3.92 (m, 1), 2.46 (s, 3), 2.3–1.2 (m, 9); IR (neat) 3040, 2920, 2845, 1595, 1480, 1330, 1310, 1265, 1145, 1080, 810, 660 cm^{-1}. Anal. Calcd for $C_{16}H_{20}O_2S$: C, 69.52; H, 7.29; S, 11.60. Found: C, 69.70; H, 7.37; S, 11.35.

Reaction of Methylenecyclohexane with 1. Reaction with methylenecyclohexane (0.489 g, 2.0 equiv) for 4 days gave 0.72 g of crude product which NMR spectroscopy showed to be ~60% pure **7**. Chromatography of 0.350 g on 30 g of silica gel with 1:1 pentane–ether as eluant gave 0.102 g (37%) of an inseparable mixture consisting of 80% **7** and 20% of a 3:1 mixture of **5** and **6** as determined by NMR. The spectral data for **7** are as follows: NMR ($CDCl_3$) δ 7.78 (br d, 2, J = 8 Hz), 7.32 (br d, 2, J = 8 Hz), 6.95 (dt, 1, J = 15, 5 Hz) 6.30 (br d, 1, J = 15 Hz), 5.48 (m, 1), 2.82 (br d, 2, J = 6 Hz), 2.45 (s, 3), 2.10–1.0 (m, 8); IR (neat) 3050, 2930, 2860, 1630, 1600, 1583, 1450, 1320, 1305, 1290, 1145, 1085, 915, 810, 730, 660 cm^{-1}; mol wt calcd for $C_{16}H_{20}O_2S$ 276.1184, found 276.1188.

Reaction of Cyclohexene with 1. Reaction with cyclohexene (0.164 g, 1.1 equiv) and $EtAlCl_2$ (2.55 mL of 1.57 M, 2.0 equiv) for 9 days gave 0.695 g of crude product. Chromatography of 0.500 g on 30 g of silica gel gave 0.116 g (31%) of an inseparable 2:1 mixture of ene adduct **8** and cyclobutene **9**, 0.058 g (19%) of **14**, and 0.015 g of cyclohexylbenzene. The spectral data are as follows: NMR ($CDCl_3$) (**8**) δ 7.74 or 7.79 (br d, 2, J = 8 Hz), 7.32 (br d, 2, J = 8 Hz), 6.95 (dd, 1, J = 15, 7 Hz), 6.18 (dd, 1, J = 15, 2 Hz), 5.85 (m, 1), 5.49 (br d, 1, J = 9 Hz), 2.85 (m, 1), 2.46 (s, 3), 2.1–1.3 (m, 6); NMR ($CDCl_3$) (**9**) δ 7.74 or 7.79 (br d, 2, J = 8 Hz), 7.32 (br d, 2, J = 8 Hz), 6.72 (br s, 1), 3.15 (m, 2), 2.46 (s, 3), 2.1–1.3 (m, 6); IR (neat) 3040, 2920, 2860, 1600, 1460, 1300, 1160, 1080, 810, 675 cm^{-1}.

Reaction of *trans*-2-Butene with 1. Reaction with *trans*-2-butene (2 g, excess) for 11 days gave 2.28 g of a two-layer mixture. NMR spectroscopy showed that the bottom layer (~0.5 g) contained **10** and **11** and the top layer was mainly polymer. Chromatography of 0.200 g of the bottom layer on 35 g of silica gel with 1:1 pentane–ether as eluant gave 0.058 g (14%) of an inseparable 1:1 mixture of ene adduct **10** and cyclobutene **11**: NMR ($CDCl_3$) (**10**) δ 7.80 or 7.77 (br d, 2, J = 8 Hz), 7.35 (br d, 2, J = 8 Hz), 6.99 (dd, 1, J = 16, 7 Hz), 6.31 (dd, 1, J = 16, 2 Hz), 5.75 (ddd, 1, J = 16, 7, 11 Hz), 5.10 (br d, 1, J = 11 Hz), 5.06 (br d, 1, J = 16 Hz), 3.11 (ddq, 1, J = 7, 7, 7 Hz), 2.47 (s, 3), 1.20 (d, 3, J = 7 Hz); NMR ($CDCl_3$) (**11**) δ 7.77 or 7.80 (br d, 2, J = 8 Hz), 7.35 (br d, 2, J = 8 Hz), 6.67 (br s, 1), 2.65 (br q, 1, J = 7 Hz), 2.47 (s, 3), 2.42 (br q, 1, J = 7 Hz), ~1.2 (2 d, 6, J = 7 Hz); IR (neat) 3060, 2965, 2915, 2870, 1600, 1452, 1315, 1305, 1290, 1145, 1085, 812, 635 cm^{-1}.

Reaction of 1-Hexene with 1. Reaction with 1-hexene (0.40 g, 2.0 equiv) for 18 days gave 0.570 g of material which NMR spectroscopy showed to be ~25% **12**. Chromatography of 0.315 g on 30 g of silica gel with 1:1 pentane-ether as eluant gave 0.055 g (19%) of **12**: NMR ($CDCl_3$) δ 7.79 (br d, 2, J = 8 Hz), 7.43 (br d, 2, J = 8 Hz), 6.95 (dd, 1, J = 16, 7 Hz), 6.30 (br d, 1, J = 16 Hz), 5.4–5.7 (m, 2), 2.92 (dd, 1, J = 6, 7 Hz), 2.46 (s, 3), 2.1–0.8 (m, 7); IR (neat) 3040, 2950, 2920, 2860, 1600, 1320, 1310, 1295, 1150, 1090, 820, 660 cm^{-1}; mol wt calcd for $C_{15}H_{20}O_2S$ 264.1184, found 264.1190.

Chloroalumination of Sulfone 1. A solution of sulfone **1** (0.360 g, 2.0 mmol) and $EtAlCl_2$ (2.55 mL of 1.57 M, 4.0 mmol, 2.0 equiv) in 10 mL of benzene was stirred for 5 days. Normal workup gave 0.413 g which NMR spectroscopy showed to be a 60:40 mixture of **14** and **1**. Chromatography of 0.200 g on 25 g of silica gel with 1:2 pentane–ether as eluant gave 0.121 g (58%) of **14** which crystallized on standing and 0.035 g (20%) of recovered **1**. The data for **14** are as follows: mp 47.5–48.5 °C [lit.[12] mp 48 °C]; NMR ($CDCl_3$) δ 7.95 (br d, 2, J = 8 Hz), 7.42 (br d, 2, J = 8 Hz), 6.86 (s, 2), 2.45 (s, 3); NMR (benzene-d_6) δ 7.82 (br d, 2, J = 8 Hz), 6.85 (br d, 2, J = 8 Hz), 6.27 (d, 1, J = 7 Hz), 5.88 (d, 1, J = 7 Hz), 1.95 (s, 3); IR (CCl_4) 3060, 2930, 2880, 1920, 1595, 1580, 1555, 1340, 1305, 1175, 1150, 1085, 855, 680, 610 cm^{-1}.

Deuterium Trapping of 12. Repetition of the above reaction for 1 day followed by addition of 3 mL of D_2O and normal workup gave 0.421 g of yellow oil which NMR showed to be a 70:30 mixture of **14** and **1**. Chromatography of 0.200 g on 30 g of silica gel with 1:1 pentane–ether as eluant gave 0.074 g (37%) of pure **15** which was crystallized from pentane: mp 47.5–48.0 °C; NMR ($CDCl_3$) δ 7.95 (br d, 2, J = 8 Hz), 7.42 (br d, 2, J = 8 Hz), 6.86 (br s, 1), 2.45 (s, 3); NMR (benzene-d_6) δ 7.82 (br d, 2, J = 8 Hz), 6.85 (br d, 2, J = 8 Hz), 5.87 (br s, 1), 1.95 (s, 3); IR (neat), the spectrum is the same as that of **14** except for peaks at 2278, 1570 (shifted from 1580) and 965 (shifted from 1250) cm^{-1}; mass spectrum, m/e 217 (M^+); mol wt calcd for $C_9H_8DClO_2S$ 217.0075, found 217.0079.

Quenching the reaction mixture with D_2O after 5-min reaction time gave only traces of **15** and recovered **1**, indicating that addition of DCl does not occur during workup.

Attempted trapping of **13** with I_2, CO_2, or CH_3I was unsuccessful.

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Registry No. **1**, 13894-21-8; **2**, 75233-10-2; **3**, 75233-11-3; **4**, 75233-12-4; **5**, 75233-13-5; **6**, 75233-14-6; **7**, 75233-15-7; **8**, 75233-16-8; **9**, 75233-17-9; **10**, 75233-18-0; **11**, 75233-19-1; **12**, 75233-20-4; **14**, 773-60-4; **15**, 775-90-6; *cis*-1,2-bis(*p*-tolylthio)ethylene, 4526-53-8; *cis*-1,2-dichloroethylene, 156-59-2; *p*-thiocresol, 106-45-6; (*p*-tolylthio)acetylene, 66823-38-9; 2,3-dimethyl-2-butene, 563-79-1; 2-methyl-2-butene, 513-35-9; ethylidenecyclopentane, 2146-37-4; 1-methylcyclohexene, 591-49-1; methylenecyclohexane, 1192-37-6; cyclohexene, 110-83-8; cyclohexylbenzene, 827-52-1; *trans*-2-butene, 624-64-6; 1-hexene, 592-41-6.

Lewis Acid Catalyzed [2 + 2] Cycloaddition of Methyl 2,3-Butadienoate to Alkenes

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Methyl 2,3-butadienoate (1) undergoes $EtAlCl_2$-catalyzed stereospecific [2 + 2] cycloadditions with alkenes to give methyl cyclobutylideneacetates in good yield. The stereospecificity and ratios of E and Z isomers suggest a [$_\pi 2_s + _\pi 2_a$] cycloaddition of the 1·$EtAlCl_2$ complex analogous to that of ketenes.

We have recently shown that acetylenic esters undergo Lewis acid catalyzed ene and stereospecific [2 + 2] cycloaddition reactions with alkenes,[2] while acrylic esters undergo ene reactions exclusively.[3] The Lewis acid catalyzed reactions of methyl 2,3-butadienoate (1) with al-

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