

Catalytic activity was evaluated in a Chemical Data Systems isothermal tubular reactor. The reactor consists of a 316 stainless steel tube (22.9 cm \times 0.64 cm) mounted inside a close-fitting metal block, which is instrumented for temperature control. A thermowell extending axially next to the reactor measured reactor temperature. Reagents were metered to the reactor as liquids by using Isco Model 314 high-pressure syringe pumps. Prior to introduction to the reactor, the feeds were vaporized and mixed in a counter-current mixer maintained at 150 °C. Flow rates of ammonia and isobutene were adjusted to obtain the desired mole ratio of reactants and total flow rate (GHSV at STP).

Product analyses were carried out by on-line gas-liquid chromatography with a Varian Model 6000 Gas Chromatograph equipped with a 6 ft \times 1/4 in. 20% Carbowax 20M on Chromosorb T column and a VISTA 402 Chromatography Data System. Quantitation was based on comparison of peaks with external standards. Identities of major products were confirmed by GC-MS.

Catalyst samples (12-18 mesh) were heated (90 °C) in the reactor under nitrogen (10 sccm at 1 atmosphere) for 16-18 h. The temperature was then raised to the desired level over 3 h. Nitrogen was shut off. Ammonia was introduced, and the desired pressure was set with the back-pressure regulator. Isobutene was then introduced to obtain the desired feed ratio. Performance of each catalyst was evaluated over 24 h.

Catalyst acidity was determined by heating a sample of the powdered catalyst at 400 °C under nitrogen in a Du Pont Model 951 Thermogravimetric Analyzer. The sample was cooled to 20 °C, and ammonia was adsorbed onto it. Temperature-programmed desorption of ammonia indicated the number of strong acid sites (millimole of ammonia chemisorbed at 200 °C/gram of catalyst; see Table I).

Registry No. SiO₂, 7631-86-9; Al₂O₃, 1344-28-1; isobutene, 115-11-7; NH₃, 7664-41-7; *tert*-butylamine, 75-64-9.

Lithium Enolate Additions to the Tropone Nucleus

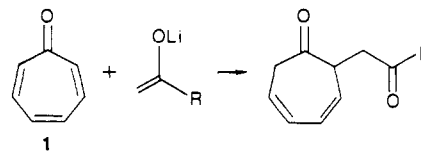
James H. Rigby,* Chrisantha H. Senanayake, and Sushil Rege

Department of Chemistry, Wayne State University, Detroit, Michigan 48202

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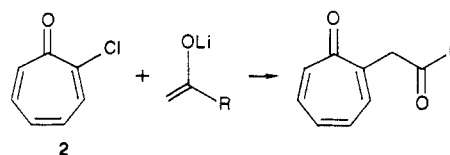
Nature is replete with examples of natural products that display a seven-membered carbocycle as a prominent structural feature. Although considerable progress has been made on the synthesis of many of these compounds,¹ only recently have strategies emerged that attempt to assemble the target molecules by starting from a pre-existing seven-membered ring system.² This situation is due, in part, to a paucity of appropriately functionalized candidates for starting materials. We have found 2,4,6-cycloheptatrien-1-one (tropone, 1) to be an excellent building block from which to construct complex carbon skeletons.^{2a,b} In conjunction with our continuing efforts to exploit the unique reactivity of this compound in natural product synthesis, we herein describe an efficient protocol for appending *functionalized* substituents onto the tropone nucleus via the addition of enolate nucleophiles.

To date direct substitution of tropones has, for the most part, been restricted to the addition of relatively unfunctionalized nucleophiles,³ and it is surprising that, prior to



our current work, enolates had not been examined in this context.⁴ This methodology was developed in response to the more general inaccessibility of substituted tropones and dihydrotropone species and was designed to provide products amenable to further manipulation in a synthetic sequence.⁵ To this end, the procedure exploits the unique proclivity of the tropenoids for incorporating nucleophiles through additions to the conjugated trienone system in a 1,8-fashion.⁶ This mode of addition is particularly attractive from a synthetic perspective since it results in the formation of products that retain a maximum level of functionalization.

The chemistry described herein is equally efficient in producing 2-substituted tropones and dihydrotropenes. Typically, substituted dihydrotropenes can be prepared by the low-temperature addition of tropone (1) to a solution of the appropriate lithium enolate in THF. Table I displays the results of several enolate additions to the parent trienone. Of particular note is the clean regioselective formation of the 1,6-disubstituted adducts in entries 3 and 4.



A similar procedure using 2-chlorotropone (2) yields the corresponding substituted tropone adducts. A presumed addition-elimination process ensues under these conditions to provide the tropone products directly.^{5c,d} Attempts to isolate and identify any intermediates formed during these reactions have been unsuccessful to date. Inspection of these observations reveals that a wide range of functionalized substituents can be efficiently incorporated onto the tropone nucleus through the use of this technology.

Experimental Section

Proton and ¹³C NMR spectra were recorded on a Nicolet QE-300 spectrometer. Chemical shifts are reported in parts per million downfield from tetramethylsilane (Me₄Si). Infrared spectra were obtained on a Nicolet 20-Dx spectrophotometer. THF was distilled from sodium benzophenone ketyl prior to use, and flash chromatography was done according to the method of Still.⁷ Tropone was prepared by the procedure developed by Radlick⁸ and 2-chlorotropone was prepared by Terborg's procedure.^{5c}

General Procedure for the Addition of Lithium Enolates to Tropone or 2-Chlorotropone. To a solution of diisopropylamine (0.14 mL, 1.1 mmol) in 1 mL of dry THF at -78 °C was added *n*-BuLi (1.6 M in hexanes) (0.53 mL, 1.1 mmol). After the mixture was stirred for 1 h, 1 mmol of the appropriate carbonyl

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Table I. Addition Products of Enolates to Troponone (1)

entry	substrate	enolate source	product	yield, ^a %
1	1	CH ₃ CO ₂ - <i>t</i> -Bu		93
2	1	CH ₃ COCH ₃		68
3 ^b		CH ₃ CO ₂ - <i>t</i> -Bu		90
4 ^b		CH ₃ CO ₂ - <i>t</i> -Bu		89
5	1	(CH ₃) ₂ CHCO ₂ Et		95
6	1			84

^a Isolated yield of pure product. ^b Prepared by the addition of the corresponding Grignard reagent to 2-chlorotropone, see ref 5c,d.

Table II. Addition Products of Enolates to 2-Chlorotropone

entry	substrate	enolate source	product	yield, ^a %
1	2			70
2	2			75
3	2			83
4	2			84
5	2			48

^a Yield of isolated, pure product.

compound in THF was added, and the reaction mixture was stirred for an additional hour at -78°C . At this time tropone^b or 2-chlorotropone^{bc} (1 mmol) in 0.25 mL of dry THF was added to the enolate solution, and the resultant mixture was stirred for 15–25 min at -78°C . The reaction mixture was then poured into 20 mL of cold (0–10 $^{\circ}\text{C}$) 5% aqueous hydrochloric acid solution, and the mixture was extracted with three 25-mL portions of ether. The organic phase was washed consecutively with saturated aqueous sodium bicarbonate solution and brine and then dried over anhydrous sodium sulfate. The solvent was removed in vacuo to give the crude product, which was purified by flash chromatography on silica gel 60 obtained from EM Science.

2-(2-*tert*-Butoxy-2-oxoethyl)cyclohepta-3,5-dien-1-one: IR (CCl₄) 3030, 2984, 2932, 1737, 1714, 1369, 1244, 1235, 1151 cm⁻¹; ¹H NMR (CDCl₃) δ 1.42 (s, 9 H), 2.35 (dd, $J = 5.7, 16$ Hz, 1 H), 2.86–3.04 (m, 2 H), 3.24–3.40 (m, 2 H), 5.50 (dd, $J = 5.2, 10.2$ Hz,

1 H), 5.90 (m, 1 H), 6.23–6.32 (m, 2 H); ¹³C NMR (CDCl₃) δ 27.96, 34.76, 44.53, 49.78, 76.57, 125.75, 128.29, 128.57, 129.12, 171.23, 208.07; mass spectrum, m/e (%) 222 (3), 180 (5), 149 (16); high-resolution mass spectrum calcd for C₁₃H₁₈O₃ 222.1558, found 222.1556.

2-(2-Oxoprop-1-yl)cyclohepta-3,5-dien-1-one: IR (CCl₄) 3032, 2958, 2906, 1718, 1405, 1363, 1015 cm⁻¹; ¹H NMR (CDCl₃) δ 2.18 (s, 3 H), 2.70 (dd, $J = 6, 17$ Hz, 1 H), 2.99–3.35 (m, 3 H), 3.46 (m, 1 H), 5.42 (dd, $J = 6, 11$ Hz, 1 H), 5.85 (m, 1 H), 6.17–6.29 (m, 2 H); ¹³C NMR (CDCl₃) δ 29.85, 42.69, 44.42, 48.85, 125.76, 128.10, 128.51, 128.93, 206.27, 207.91; mass spectrum, m/e (%) 164 (5), 122 (21), 107 (17), 91 (18); high-resolution mass spectrum calcd for C₁₀H₁₂O₂ 164.0837, found 164.0842.

2-(2-*tert*-Butoxy-2-oxoethyl)-7-methylcyclohepta-3,5-dien-1-one: IR (CCl₄) 3023, 2984, 2884, 1730, 1715, 1653, 1361, 1246, 1150 cm⁻¹; ¹H NMR (CDCl₃) δ 1.33 (d, $J = 7$ Hz, 3 H), 1.44 (s, 9 H), 2.40–3.20 (m, 4 H), 5.62 (m, 2 H), 6.25 (m, 2 H); mass spectrum, m/e (%) no M⁺, 163 (25), 57 (100).

2-(2-*tert*-Butoxy-2-oxoethyl)-7-[2-(1,3-dioxolan-2-yl)ethyl]cyclohepta-3,5-dien-1-one: IR (CCl₄) 3020, 2950, 1735, 1715, 1350, 1120 cm⁻¹; ¹H NMR (CDCl₃) δ 1.39 (s, 9 H), 1.60–2.0 (m, 4 H), 2.60–3.00 (m, 4 H), 3.75–4.00 (m, 4 H), 4.85 (m, 1 H), 5.60 (m, 2 H), 6.25 (m, 2 H); mass spectrum, m/e (%) no M⁺, 249 (10), 73 (100), 57 (97).

2-(1,1-Dimethyl-2-ethoxy-2-oxoethyl)cyclohepta-3,5-dien-1-one: IR (CCl₄) 3036, 2979, 1738, 1715, 1408, 1244, 1146 cm⁻¹; ¹H NMR (CDCl₃) δ 1.17 (s, 3 H), 1.20 (t, $J = 6$ Hz, 3 H), 1.28 (s, 3 H), 2.87 (m, 1 H), 3.22 (m, 1 H), 3.44 (m, 1 H), 4.07 (q, $J = 6$ Hz, 2 H), 5.81 (m, 2 H), 6.30 (m, 2 H); ¹³C NMR (CDCl₃) δ 13.87, 20.28, 25.71, 42.22, 44.84, 60.36, 125.46, 125.82, 129.07, 129.28, 177.17, 207.80; mass spectrum, m/e (%) 222 (4), 177 (31), 149 (33), 121 (95), 107 (100); high-resolution mass spectrum calcd for C₁₃H₁₈O₃ 222.1558, found 222.1249.

2-(2-Oxocyclohex-1-yl)cyclohepta-3,5-dien-1-one: IR (CCl₄) 3034, 2942, 2860, 1719, 1712 cm⁻¹; ¹H NMR (CDCl₃) δ 1.20–2.50 (m, 8 H), 2.85–3.61 (m, 4 H), 5.50 (m, 1 H), 5.80 (m, 1 H), 6.35 (m, 2 H); mass spectrum, m/e (%) 204 (18), 107 (100), 98 (73), 79 (60), 55 (76); high-resolution mass spectrum calcd for C₁₃H₁₆O₂ 204.1150, found 204.1144.

Preparation of 2-(4-Ethoxy-2,4-dioxobut-1-yl)-2,4,6-cycloheptatrien-1-one. To a suspension of NaH (420 mg, 4.4 mmol) of a 50% oil suspension, which was washed three times with pentane prior to use) in 3 mL of THF at 0 $^{\circ}\text{C}$ was added ethyl acetoacetate (0.25 mL, 2 mmol) in 1 mL of THF. This mixture was stirred at this temperature for 15 min at which time *n*-bu-

tyllithium (2.5 M in hexanes) (0.88 mL, 2.2 mmol) was added, and stirring was continued for an additional 20 min. The temperature was lowered to -50°C , and 2-chlorotropone^{5c} (281 mg, 2 mmol) in 1 mL of THF was added. Stirring was continued at this temperature until all of the chlorotropone was consumed as judged by TLC analysis (1:1 petroleum ether-ether). The reaction mixture was quenched with saturated aqueous ammonium chloride solution, diluted with ether, washed with brine, and dried over anhydrous sodium sulfate. Removal of solvent in vacuo and purification of the crude product by flash chromatography (6 g of silica gel; 80% petroleum ether, 20% ether as eluent) gave 141 mg (65%) of product: IR (CCl₄) 1739, 1722, 1635, 1587, 1326 cm⁻¹; ¹H NMR (CDCl₃) δ 1.26 (t, $J = 7$ Hz, 3 H), 3.67 (s, 2 H), 3.76 (s, 2 H), 4.19 (q, $J = 7$ Hz, 2 H), 7.00-7.34 (m, 5 H); ¹³C NMR (CDCl₃) δ 13.95, 48.97, 49.55, 61.14, 133.54, 133.88, 136.09, 137.54, 140.45, 148.74, 167.28, 186.06, 199.51; mass spectrum, m/e (%) 234 (5), 188 (16), 147 (59), 120 (100), 92 (65), 65 (31); high-resolution mass spectrum calcd for C₁₃H₁₄O₄ 234.0891, found 234.0895.

2-[1-(2-Oxopropyl)]-2,4,6-cycloheptatrien-1-one was prepared as described in the general procedure for the addition of lithium enolates to 2-chlorotropone: IR (CCl₄) 1719, 1639 cm⁻¹; ¹H NMR (CDCl₃) δ 2.27 (s, 3 H), 3.62 (s, 2 H), 6.93-7.25 (m, 5 H); ¹³C NMR (CDCl₃) δ 30.21, 49.62, 133.48, 133.52, 135.83, 137.12, 140.40, 149.00, 186.14, 204.90; high-resolution mass spectrum calcd for C₁₀H₁₀O₂ 162.0878, found 162.0915.

2-(1-Carbomethoxy-4,6-heptadien-1-yl)-2,4,6-cycloheptatrien-1-one: IR (CCl₄) 1736, 1635, 1592 cm⁻¹; ¹H NMR (CDCl₃) δ 1.80 (q, $J = 8$ Hz, 2 H), 2.20 (q, $J = 8$ Hz, 2 H), 3.68 (s, 3 H), 3.95 (t, $J = 9.5$ Hz, 1 H), 5.17 (m, 2 H), 5.68 (m, 1 H), 6.10 (m, 1 H), 6.32 (m, 1 H), 7.01-7.45 (m, 5 H); ¹³C NMR (CDCl₃) δ 25.80, 30.53, 48.07, 51.96, 117.34, 130.17, 131.87, 132.01, 133.46, 133.72, 135.25, 135.55, 140.93, 151.99, 173.71, 185.77; mass spectrum, m/e (%) 258 (4), 198 (9), 178 (27), 159 (18), 146 (100), 131 (16), 115 (11), 91 (15), 77 (17); high-resolution mass spectrum calcd for C₁₆H₁₈O₃ 258.1255, found 258.1261.

2-(1-Carbomethoxycyclobutan-1-yl)-2,4,6-cycloheptatrien-1-one: IR (CCl₄) 1734, 1638, 1588 cm⁻¹; ¹H NMR (CDCl₃) δ 1.76 (m, 1 H), 2.18 (m, 3 H), 2.69 (m, 2 H), 3.58 (s, 3 H), 6.91-7.29 (m, 5 H); ¹³C NMR (CDCl₃) δ 15.98, 30.61, 51.99, 53.08, 132.77, 132.97, 133.38, 135.41, 140.45, 155.84, 174.93, 185.79; mass spectrum, m/e (%) 218 (5), 186 (30), 159 (71), 141 (19), 131 (100), 115 (23), 103 (49), 91 (25), 77 (60); high-resolution mass spectrum calcd for C₁₃H₁₄O₃ 218.0942, found 218.0946.

2-(2,3,4,5-Tetrahydro-2-oxofuran-3-yl)-2,4,6-cycloheptatrien-1-one: IR (CCl₄) 1768, 1679, 1588 cm⁻¹; ¹H NMR (CDCl₃) δ 2.30-4.41 (m, 1 H), 2.47-2.60 (m, 1 H), 3.77 (t, $J = 10$ Hz, 1 H), 4.36 (q, $J = 8.5$ Hz, 1 H), 4.56 (dt, $J = 8.5, 3.6$ Hz, 1 H), 6.96-7.37 (m, 5 H); ¹³C NMR (CDCl₃) δ 27.99, 46.96, 66.95, 133.50, 134.54, 136.17, 137.47, 141.12, 150.66, 172.24, 185.26; mass spectrum, m/e (%) 190 (5), 146 (59), 118 (100), 91 (16), 77 (15); high-resolution mass spectrum calcd for C₁₁H₁₀O₃ 190.1982, found 190.1979.

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Registry No. 1, 539-80-0; 2, 3839-48-3; MeCOCH₂CO₂Et, 141-97-9; H₂C=CHCH=CH(CH₂)₃CO₂Me, 73501-25-4; MeCOMe, 67-64-1; MeCO₂Bu-*t*, 540-88-5; Me₂CHCO₂Et, 97-62-1; cyclohexanone, 108-94-1; methyl cyclobutanecarboxylate, 765-85-5; 2,3,4,5-tetrahydro-2-furanone, 96-48-0; 2-methyltropone, 29639-53-0; 2-(1,3-dioxolan-2-yl)ethyltropone, 115912-59-9; 2-(2-*tert*-butoxy-2-oxoethyl-yl)cyclohepta-3,5-dien-1-one, 115912-60-2; 2-(2-oxoprop-1-yl)cyclohepta-3,5-dien-1-one, 115912-61-3; 2-(2-*tert*-butoxy-2-oxoethyl-yl)-7-methylcyclohepta-3,5-dien-1-one, 115912-62-4; 2-(2-*tert*-butoxy-2-oxoethyl-yl)-7-[2-(1,3-dioxolan-2-yl)ethyl]cyclohepta-3,5-dien-1-one, 115912-63-5; 2-(1,1-dimethyl-2-ethoxy-2-oxoethyl-yl)cyclohepta-3,5-dien-1-one, 115912-64-6; 2-(2-oxocyclohex-1-yl)cyclohepta-3,5-dien-1-one, 115912-65-7; 2-(4-ethoxy-2,4-dioxobut-1-yl)-2,4,6-cycloheptatrien-1-one, 115912-66-8; 2-[1-(2-oxopropyl)]-2,4,6-cycloheptatrien-1-one, 39479-44-2; 2-(1-carbomethoxy-4,6-heptadien-1-yl)-2,4,6-cycloheptatrien-1-one, 115912-67-9; 2-(1-carbomethoxycyclobutan-1-yl)-2,4,6-cycloheptatrien-1-one, 115912-68-0; 2-(4,5-dihydro-2-oxofuran-3-yl)-2,4,6-cycloheptatrien-1-one, 115912-69-1; *tert*-butyl bromomagnesium acetate, 115912-70-4.

Electrochemical 1,2-Addition of Trifluoromethyl and Acetamide Groups to Methyl Methacrylate

Kenji Uneyama* and Hiromi Nanbu

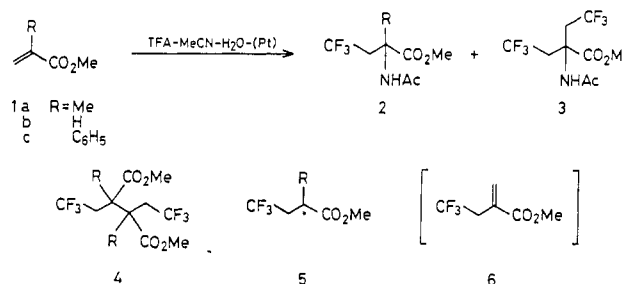
Department of Applied Chemistry, Faculty of Engineering, Okayama University, Okayama 700, Japan

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Organofluorine compounds have attracted increasing attention for medicinal and agricultural usage and for material science.¹ Biologically active trifluoromethylated amino acid derivatives² have also been prepared.³

Trifluoromethylation by the use of metal complexes of trifluoromethyl iodide,⁴ *N*-(trifluoromethyl)-*N*-nitroso-trifluoromethanesulfonamide,⁵ and perfluoroacyl peroxide⁶ has been known, but no reagent can realize 1,2-addition of the trifluoromethyl group and a nucleophile to the carbon-carbon double bond. The desired 1,2-addition requires an oxidation step during the reaction course. An electrochemical trifluoromethylation of olefins would be promising for the purpose. Here we describe a novel electrochemical trifluoromethyl-acetamidation in which trifluoromethyl and acetamide groups are incorporated at the β - and α -carbons of an α,β -unsaturated carboxylate, respectively.

Electrolysis of a mixture of trifluoroacetic acid (TFA, 6 mmol) and methyl methacrylate (MMA, 2 mmol) in acetonitrile (MeCN, 20 mL)-water (3 mL) containing sodium hydroxide (0.6 mmol) was conducted in an undivided cell by using platinum foil electrodes. Constant current (1 mA/cm², 4 F/mol) was applied at 0-5 $^{\circ}\text{C}$. Trifluoromethylated acetamides **2a** and **3** were obtained in 20% and 5% yield, respectively, after chromatographic isolation.



The acetamide **2a** is easily separable by column chromatography and thus could be prepared on a large scale. This compound exhibited the following spectral characteristics consistent with the assigned structure. The ¹³C NMR spectrum shows two quartets at 125.8 ($J = 278.5$ Hz) and 38.0 ppm ($J = 27.5$ Hz), revealing a CF₃CH₂ moiety. The ¹⁹F NMR spectrum gives a triplet at 99.2 ppm ($J = 10.3$ Hz), suggesting a CF₃CH₂ group. The IR spectrum shows strong bands at 3258 and 1652 cm⁻¹ characteristic for an

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