aqueous sulfuric acid and glacial acetic acid for 3 h at 130 °C, as described by Büchi and Wuest,[6] gave **6** in 38% yield; under milder conditions it gave a 1:3 mixture of **6** and **7** in 92% yield.

The formation of **6**–**8** can be rationalized by assuming that, during the hydrolysis of the furan ring, **3c** and **3d** undergo an acid-catalyzed β-diketonic fission to intermediates of type **9**, which then cyclize.

The structures of **6**–**8** were confirmed by ^{13}C NMR data (Table I).

This approach provides a flexible sequence for the preparation of substituted cyclopent-2-enones by a simple procedure. The products **5**–**8**, which have not been reported in the literature, are of interest for preparative purposes; for example, **5** could be an intermediate in a synthetic route to modified prostaglandins.[7]

Experimental Section

Melting points were determined on a Kofler block and are uncorrected. ^1H NMR were recorded in CCl_4 solution, unless otherwise indicated, on a Perkin-Elmer R-32 spectrometer at 90 MHz. Chemical shifts are given in parts per million from internal Me_4Si. ^{13}C NMR spectra were recorded at 20 MHz with a Varian CFT-20 Fourier transform spectrometer in $CDCl_3$ solution. Chemical shifts are given in parts per million from internal Me_4Si. IR spectra were taken with a Perkin-Elmer 257 Infracord spectrometer. Commercial Merck silica gel was used for column chromatography. All the reactions were monitored by TLC using Carlo Erba precoated silica gel plates. The chromatograms were developed by spraying with 5 N H_2SO_4 and heating at 110 °C for 10 min. Mass spectra were obtained with an AEI MS-12 spectrometer at 70 eV, using direct insertion at a source temperature of 150 °C.

Ethyl 2-(5-Methyl-2-furyl)-3-oxobutyrate (3a). $ZnCl_2$ (7.5 mmol) was added to a stirred solution of **1** (5 mmol) and ethyl acetoacetate (7.5 mmol) in AcOH (1–1.5 mL) and H_2O (0.5 mL). After 24 h the mixture was poured into water and extracted several times with Et_2O. The combined extracts were washed with saturated aqueous $NaHCO_3$ and water and dried over Na_2SO_4, and the ether was removed in vacuo. The crude product was chromatographed on SiO_2 and eluted with *n*-hexane–Et_2O (9:1) to give 0.84 g (80%) of **3a** as an oil.

3b and **3c** were prepared under the same conditions from **2b** and **2c** in yields of 85 and 80%, respectively.

Analytical and spectroscopic data for **3a–c** agreed with those reported previously.[2]

(5-Methyl-2-furyl)dibenzoylmethane (3d). $ZnCl_2$ (1.5 g) was added to a stirred solution of **1** (13.2 mmol) and dibenzoylmethane (8.8 mmol) in dimethoxyethane (10 mL), AcOH (3 mL), and water (0.7 mL), and stirring was continued for 24 h. The mixture was worked up as for **3a**, and elution of the chromatogram with benzene–*n*-hexane (4:1) gave 2.3 g (85%) of **3d** as needles from *n*-hexane: mp 101–103 °C; ^1H NMR ($CDCl_3$) δ 18.80 (s, 1 H), 7.25 (m, 10 H), 5.62 (m, 2 H), 2.10 (s, 3 H); IR (1% $CHCl_3$) ν_{max} 3045, 1700, 1675, 1601, 1435, 1385, 1315, 1175, 1065, 1015, 895 cm^{-1}; mass spectrum, *m/e* 304 (M^+). Anal. Calcd for $C_{20}H_{16}O_3$: C, 78.93; H, 5.30. Found: C, 79.01; H, 5.26.

4-Acetyl-2-(ethoxycarbonyl)-3-methylcyclopent-2-enone (5). Acid cation-exchange resin (Merck-Ionenanstaucher I; 1 g) was added to a stirred solution of **3a** (320 mg) in 15 mL of 4:1 acetone–water at 70 °C. After 6 h 0.5 mL of HCO_2H was added and the mixture was refluxed for 24 h. The ion-exchange resin was removed by filtration through SiO_2 under anhydrous conditions, the solvent was removed in vacuo, and the product was chromatographed on SiO_2. Elution with benzene–Et_2O (1:1) gave 95 mg of **5** (50%) as an oil (130 mg of **3a** was recovered): ^1H NMR (CCl_4) δ 4.81 (dd, 1 H, J_1 = 8 Hz, J_2 = 3 Hz), 4.24 (q, 2 H), 3.00 (dd, 1 H, J_1 = 18 Hz, J_2 = 3 Hz), 2.67 (dd, 1 H, J_1 = 18 Hz, J_2 = 8 Hz), 2.54 (s, 3 H), 2.18 (s, 3 H), 1.32 (t, 3 H); IR (1% CCl_4) ν_{max} 2980, 1710, 1600, 1420, 1375, 1350, 1150, 1080, 1050 cm^{-1}; mass spectrum, *m/e* 210 (M^+). Anal. Calcd for $C_{11}H_{14}O_4$: C, 62.95; H, 6.71. Found: C, 62.93; H, 6.66.

2-Benzoyl-3-methylcyclopent-2-enone (6). A solution of **3b** (200 mg) in a mixture of AcOH (4 mL) and 10% H_2SO_4 (0.2 mL) was refluxed at 130 °C for 3 h and worked up as for **3a**. Elution of the chromatogram with benzene–Et_2O (9:1) gave 56 mg (38%) of **6** as an oil: ^1H NMR (CCl_4) δ 7.50 (m, 5 H), 2.62 (m, 2 H), 2.48 (m, 2 H), 2.10 (s, 3 H); IR (1% CCl_4) ν_{max} 3080, 2940, 1720, 1670, 1610, 1460, 1390, 1340, 1300, 1185, 1170, 1080 cm^{-1}; mass spectrum, *m/e* 200 (M^+). Anal. Calcd for $C_{13}H_{12}O_2$: C, 77.98; H, 6.04. Found: C, 77.89; H, 5.98.

4-Acetyl-3-phenylcyclopent-2-enone (7). Concentrated H_2SO_4 (1.4 mL) and $ZnCl_2$ (600 mg) were added to a stirred solution of **3b** (400 mg) in 25 mL of 4:1 acetone–water. After the mixture was refluxed for 6 h 0.6 mL of concentrated H_2SO_4 was added and the mixture was refluxed for 17 h. The mixture was diluted with AcOEt and washed several times with saturated aqueous NaCl. The neutral organic layer was dried over Na_2SO_4, the solvent was removed in vacuo, and the product was chromatographed on SiO_2. Elution with benzene–Et_2O (9:1) gave 270 mg (92%) of a mixture of **6** and **7**. Separation of the components was achieved by fractional crystallization in CCl_4–*n*-hexane (1:1.5) solution. The pure crystalline **7** (200 mg, 69%) was obtained as needles: mp 85–87 °C; ^1H NMR (CCl_4) δ 7.51 (m, 5 H), 6.58 (m, 1 H), 4.21 (dd, 1 H, J_1 = 7 Hz, J_2 = 3 Hz), 2.70 (dd, 1 H, J_1 = 18 Hz, J_2 = 7 Hz), 2.35 (dd, 1 H, J_1 = 18 Hz, J_2 = 3 Hz), 1.90 (s, 3 H); IR (1% CCl_4) ν_{max} 1715, 1660, 1600, 1450, 1360, 1330, 1180, 920 cm^{-1}; mass spectrum, *m/e* 200 (M^+). Anal. Calcd for $C_{13}H_{12}O_2$: C, 77.98; H, 6.04. Found: C, 78.08; H, 6.12. Evaporation to dryness of the mother liquors in vacuo gave pure **6** (67 mg, 23%) as an oil.

Similar treatment of **3d** gave a 50% yield of a 1:3 mixture of **6** and **7**.

4-Acetyl-3-methylcyclopent-2-enone (8). Concentrated H_2SO_4 (0.4 mL) and $ZnCl_2$ (400 mg) were added to a stirred solution of **3c** in 25 mL of 4:1 acetone–water at 70 °C. After 18 h 0.2 mL of concentrated H_2SO_4 was added and the mixture refluxed for 18 h. The mixture was worked up as for **3a**, and elution of the chromatogram with benzene–Et_2O (2:1) gave 270 mg (70%) of **8** as an oil: ^1H NMR (CCl_4) δ 5.95 (m, 1 H), 3.61 (dd, 1 H, J_1 = 7 Hz, J_2 = 3 Hz), 2.65 (dd, 1 H, J_1 = 15 Hz, J_2 = 3 Hz), 2.38 (dd, 1 H, J_1 = 15 Hz, J_2 = 3 Hz), 2.10 (s, 3 H), 2.08 (s, 3 H); IR (CCl_4) ν_{max} 2980, 2930, 2875, 1710, 1622, 1430, 1375, 1355, 1290, 1175, 1115, 940, 910 cm^{-1}; mass spectrum, *m/e* 138 (M^+). Anal. Calcd for $C_8H_{10}O_2$: C, 69.55; H, 7.30. Found: C, 69.70; H, 7.18.

Registry No. 1, 22414-24-0; **2a**, 141-97-9; **2b**, 94-02-0; **2c**, 123-54-6; **2d**, 120-46-7; **3a**, 74684-39-2; **3b**, 74684-40-5; **3c**, 74684-41-6; **3d**, 74684-42-7; **5**, 74684-43-8; **6**, 74744-22-2; **7**, 74684-44-9; **8**, 73923-20-3.

(6) (a) G. Büchi and H. Wuest, *J. Org. Chem.*, **31**, 977 (1966); (b) Under these conditions, a complex mixture of red unidentified products (50–55%), due to decomposition of the materials, was also obtained.
(7) K. Inoue, *Chem. Lett.*, 1747 (1978).

Transaminations Using 9-Fluorenone-1-carboxylic Acid[1]

Charles A. Panetta* and Ajit S. Dixit

Department of Chemistry, University of Mississippi, University, Mississippi 38677

Received June 4, 1980

In 1978, one of us (C.A.P.) reported some unusual examples of amino acid transamination with *o*-formylbenzoic acid.[2] L-Alanine and L-glutamic acid were converted into pyruvic and α-ketoglutaric acids, respectively, in low yields while *o*-formylbenzoic acid was aminated and further

(1) Most of the work described in this note was presented at the 179th ACS National Meeting, Houston, TX, March 26, 1980.
(2) Panetta, C. A.; Miller, A. L. *J. Org. Chem.* **1978**, *43*, 2113.

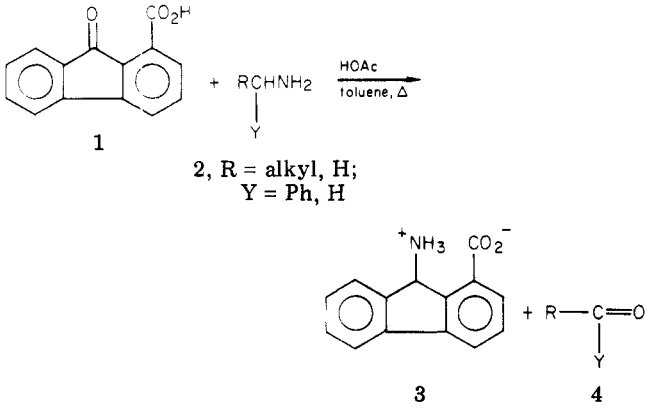
0022-3263/80/1945-4503$01.00/0

Table I. Acid-Catalyzed Transaminations Using 9-Fluorenone-1-carboxylic Acid (1): Summary of Results

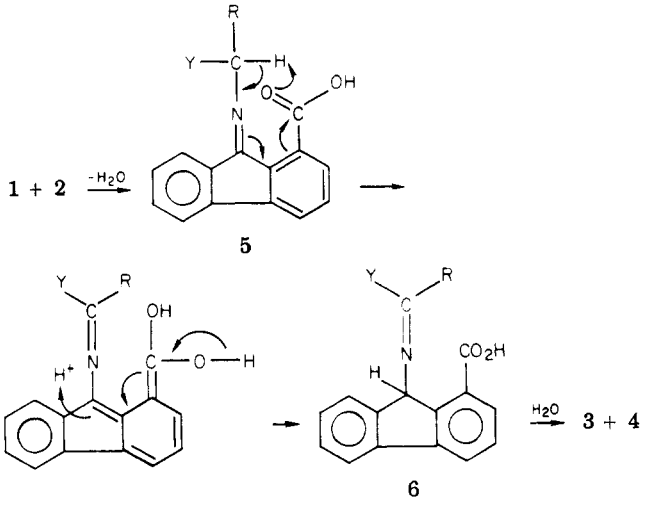
amine (2)	reflux period,[e] h	3, % yield	4,[a] % yield	recovered 1, % yield
benzylamine	6.5 (no Soxhlet)	96	benzaldehyde, 100	0
(±)-α-methylbenzylamine	23.5	54	acetophenone, 30	26
(−)-α-methylbenzylamine	23.5	72	acetophenone, 60	28
n-butylamine	22.0	100	butyraldehyde[b]	0
n-propylamine	2.0	22	propionaldehyde[c]	67
(−)-α-phenylglycine	2.3	60	benzaldehyde, 68	32
(+)-glutamic acid	8.0	78	2-oxoglutaric acid[d]	0
(+)-alanine	2.5	80	none identified	0
(±)-phenylalanine	4.0	83	phenylacetaldehyde[c]	8

[a] Identified by TLC, IR, and 2,4-dinitrophenylhydrazone derivative. [b] Isolated from the base-insoluble extract as the aldol condensation product, 2-ethyl-2-hexenal (79% yield). The latter was identified by its 2,4-dinitrophenylhydrazone and by its ^{1}H NMR spectrum[9]. [c] Yield not determined. [d] Not isolated. Detected with other products using TLC. [e] Reflux period under Soxhlet with 4-Å molecular sieves.

Scheme I



Scheme II



transformed in yields ranging from 46 to 66%. This work was a unique example of a nonenzymatic amino acid transamination by a nonpyridoxal-type structure that was not accompanied by decarboxylation.[3]

Recently, we have found that 9-fluorenone-1-carboxylic acid[4] (1), another oxo carboxylic acid, effects the transamination of benzylamines and primary alkylamines (Scheme I), as well as amino acids. The reaction conditions are similar to those used with *o*-formylbenzoic acid: the amine and 1 are dissolved in toluene and acetic acid and the resultant solution is heated at reflux temperature for several hours. During this period, a product precipitated from the reaction mixture. It was identified as 9-fluorenylamine-1-carboxylic acid (3) by comparison of spectral and physical properties with those of an authentic sample.[5] The carbonyl product, 4, was generally isolated from the neutral extracts, purified, and identified by its infrared spectrum and 2,4-dinitrophenylhydrazone derivative. In most experiments, the yields of the products 3 and 4 were greatly reduced if no provision was made to pass the reflux condensate through a Soxhlet extractor filled with molecular sieves (type 4A). This was originally employed in order to eliminate water and cause imine formation (first step, Scheme II) which is preliminary to most known transamination reactions such as those of pyridoxal phosphate.[6] However, the transaminated product, 3, is isolated in spite of the absence of hydrolysis (last step, Scheme II). We can only conclude that the chief function of the molecular sieves is something other than dehydration.

The neighboring carboxyl moiety appears to facilitate the reaction outlined in Schemes I and II since 9-fluorenone reacts with benzylamine 16 times more slowly than 1, affording 9-fluorenylamine in a yield which is about two-thirds that of product 3.

Amino acids readily react with 1 in acetic acid and toluene to form good yields of 3. Thus far, no α-keto acids have been isolated from these reactions. Instead, decarboxylated products, such as benzaldehyde (from phenylglycine), were identified in the product mixture. Transaminations of amino acids and carbonyl compounds accompanied by decarboxylation are well-known transformations.[7] Indeed, 9-fluorenone was reported to degrade several amino acids in this manner after a 3-h reflux period in aqueous pyridine.[8] We subjected 9-fluorenone-1-carboxylic acid (1) and L-alanine to these conditions. 9-Fluorenylamine-1-carboxylic acid (3) was obtained in 32% yield and 65% 1 was recovered unchanged. Neither pyruvic acid or acetaldehyde was identified or isolated.

Table I summarizes the results of transaminations using 1, acetic acid, and toluene. In general, benzylamines, primary alkylamines, and amino acids are transaminated with 9-fluorenone-1-carboxylic acid in the presence of acetic acid. Secondary alkylamines (*sec*-butylamine and cyclohexylamine) were not changed by these conditions.

(3) For similar reactions, see: Nakada, H. I.; Weinhouse, S. *J. Biol. Chem.* **1953**, *204*, 831.
(4) Forrest, J.; Tucker, S. H. *J. Chem. Soc.* **1948**, 1137. Fieser, L. F.; Seligman, A. M. *J. Am. Chem. Soc.* **1935**, *57*, 2174.
(5) Kuhn, R.; Weiser, D.; Fischer, H. *Chem. Ber.* **1961**, *94*, 2252.
(6) Bruice, T. C.; Benkovic, S. J. "Bioorganic Mechanisms"; Benjamin: New York, 1966; Vol. II, pp 226–300.
(7) Herbst, R. M. *Adv. Enzymol.* **1944**, *4*, 76.
(8) Moubasher, R. *J. Chem. Soc.* **1951**, 1928.
(9) Urry, W. H.; Nishihara, A.; Niu, J. H. Y. *J. Org. Chem.* **1967**, *32*, 347.

Experimental Section

IR spectra were recorded on a Beckman Acculab 3 spectrophotometer, ^1H NMR spectra on a Perkin-Elmer R-24B, and ^{13}C NMR spectra on a JEOL FX60Q instrument. Melting points are corrected and microanalyses were performed by the analytical Department, Bristol Laboratories, Syrause, NY.

General Acid-Catalyzed Transamination Procedure. The Transamination of α-Methylbenzylamine. To a solution of 1.12 g (5 mmol) of 9-fluorenone-1-carboxylic acid (1)[4] and 30 mL of glacial HOAc were added 0.606 g (5 mmol) of (−)-α-methylbenzylamine and 70 mL of toluene. The resultant solution was heated at reflux temperature for 23.5 h under a Soxhlet extraction apparatus filled with molecular sieves (4 Å). A precipitated solid was collected by filtration and recrystallized from either HOAc or methanol to afford essentially pure **3** in 72% yield: mp 255–280 °C dec; IR (Nujol) 3120 (NH_3^+), 1685 (CO_2H), 1590 cm^{-1} (CO_2^-); ^1H NMR (Me_2SO-d_6) δ 9.0 (br, carboxyl H), 7.4-8.3 (m, aromatic H), 5.64 (br, CHN), 1.89 (s, NH_2); ^{13}C NMR (Me_2SO-d_6) 167.71 (CO_2H), 120.64–142.46 (aromatic), 54.22 ppm ($CHNH_2$); mass spectrum (70 eV), *m/e* 225.1 (molecular ion). The acetate salt of **3** was identical with authentic[5] material with respect to its IR, melting point and mixture melting point.

The transamination of optically pure (either enantiomer) α-methylbenzylamine produced a sample of **3** whose optical rotation in several solvents was essentially zero.

After **3** was separated from the reaction mixture, the filtrate was diluted with water and the pH was adjusted from 3.0 to 8.5. The resultant aqueous solution was extracted with EtOAc and the organic extracts (EtOAc and toluene) were washed and dried. Removal of the solvent left an oily residue which contained essentially two components by TLC. The faster of these had an R_f value identical with that of acetophenone. The two components were separated by elution on a silica gel column, using 80% petroleum either (bp 30–60 °C) and 20% EtOAc. The first product eluted was identified as acetophenone (0.36 g, 60%) by its IR spectrum and 2,4-dinitrophenylhydrazone derivative.

Acidification of the above pH 8.5 aqueous solution resulted in the precipitation of 9-fluorenone-1-carboxylic acid (1) and starting material (0.32 g, 28% recovery).

Acid-Catalyzed Transamination of 9-Fluorenone and Benzylamine. To a solution of 0.9 g (5 mmol) of 9-fluorenone and 30 mL of glacial HOAc were added 50 mL of toluene and 0.54 g (5 mmol) of benzylamine. The mixture was heated at reflux temperature for 101 h, after which the volatile components were removed by distillation under reduced pressure. The residual oil crystallized from ethanol to afford 0.11 g (9.9%) of crystals of 9-acetamidofluorene: mp 267–268 °C (lit.[10,11] mp 245–246, 255, 260–261 °C); IR (Nujol) 3280 (NH), 1650 (C=O), 1550 cm^{-1} (amide II); ^1H NMR (Me_2SO-d_6) δ 8.4 (d, NH), 7.2–7.9 (m, aromatic H), 5.95 (d, CH), 1.95 (s, CH_3CO); ^{13}C NMR (Me_2SO-d_6) 170.05 (C=O), 144.86–120.05 (aromatic), 54.09 (CHN), 22.60 ppm (CH_3).

Anal. Calcd for $C_{15}H_{13}NO$: C, 80.69; H, 5.86; N, 6.27. Found: C, 80.44; H, 6.08; N, 6.31.

The filtrate from above was concentrated in vacuo to an oil (1.36 g) which was dissolved in 25 mL of EtOAc and extracted with 3 N HCl (2 × 20 mL). The organic layer was washed with water, dried, and then concentrated on a rotary evaporator at reduced pressure to a partially crystalline oil (0.64 g). According to TLC (70% hexane, 30% benzene), this oil was composed of 9-fluorenone and benzaldehyde. Oxidation with Ag_2O produced 0.05 g of benzoic acid (which indicated an 8.2% yield of benzaldehyde) and 0.3 g of 9-fluorenone (33% recovery).

The aqueous HCl extract from above was adjusted to pH 8.0 with 3 N NaOH and was then extracted with EtOAc (2 × 30 mL). The EtOAc solution was washed and dried and the solvent was removed by distillation at reduced pressure. The residual solid (0.51 g, 56.3% 9-fluorenylamine) was crystallized from hexane: mp 62–63 °C; an *N*-acetyl derivative melted at 253–254 °C (lit.[10,11] mp 64–65 and 255 °C, respectively).

Transamination of 9-Fluorenone-1-carboxylic Acid (1) and L-Alanine in Aqueous Pyridine. A solution of 0.34 g (1.5 mmol) of 1, 0.13 g (1.5 mmol) of L-alanine in 7.5 mL of water, and 22.5 mL of pyridine was heated at reflux temperature for 3 h. A tan precipitate was collected by filtration (0.11 g), mp 217–221 °C. The spectral properties of this product and its *N*-acetate salt and the melting point of the latter (210–211 °C) were identical with those of authentic **3**. The yield was 32%.

The filtrate was diluted with 25 mL of water and the pH was adjusted to 9.0 with 3 N NaOH. This was extracted with EtOAc (2 × 25 mL) and the organic layer was washed with water, dried, and distilled under reduced pressure. The residual oil weighed 0.03 g but could not be identified. It showed only a zone at the origin after TLC using 80% C_6H_6, 15% EtOAc, and 5% HOAc.

The aqueous layer from the above extraction was adjusted to pH 2.0 with 6 N HCl and was then extracted with EtOAc (2 × 30 mL). The EtOAc solution was washed and dried, and the solvent was removed in vacuo to afford a red solid which weighed 0.22 g: mp 193–194 °C [(lit.[4] mp 191–193 °C (for 1)]; TLC, R_f identical with that of 1 (80% C_6H_6, 15% EtOAc, and 5% HOAc). The yield of recovered 9-fluorenone-1-carboxylic acid was 64.7%.

Acknowledgment is made for the generous assistance of Dr. N. E. Heimer, who obtained and helped interpret many of the NMR spectra. Gratitude is also extended to Bristol Laboratories, Syracuse, NY, for all of the microanalyses.

Registry No. 1, 1573-92-8; 2 (R = Ph; Y = H), 100-46-9; (±)-2 (R = Ph; Y = CH_3), 618-36-0; (−)-2 (R = Ph; Y = CH_3), 2627-86-3; 2 (R = $(CH_2)_2CH_3$; Y = H), 109-73-9; 2 (R = CH_2CH_3; Y = H), 107-10-8; 2 (R = Ph; Y = CO_2H), 875-74-1; 2 (R = $(CH_2)_2CO_2H$; Y = CO_2H), 56-86-0; 2 (R = CH_3; Y = CO_2H), 56-41-7; 2 (R = CH_2Ph; Y = CO_2H), 150-30-1; 3, 75031-63-9; 3-acetate salt, 75031-64-0; 4 (R = Ph; Y = H), 100-52-7; 4 (R = CH_3; Y = H), 98-86-2; 2-ethyl-2-hexenal, 645-62-5; 9-fluorenone, 486-25-9; 9-acetamidofluorene, 75031-65-1.

(10) Schmidt, J.; Stutzel, H. *Chem. Ber.* **1908**, *41*, 1243.
(11) Kuhn, R.; Jacob, P. *Chem. Ber.* **1925**, *58*, 1432.

Reaction of Ethyl Cyclopropanecarboxylate with Base[1a]

Harold W. Pinnick,* Yeong-Ho Chang, Scott C. Foster,[1b] and Meledath Govindan

Department of Chemistry, University of Georgia, Athens, Georgia 30602

Received October 26, 1979

Ethyl cyclopropanecarboxylate reacts with strong base to give a self-condensation product involving 3 mol of the ester. Some chemistry of this new compound is described.

The carbonyl group renders α hydrogens acidic.[2] Deprotonation with a strong base gives a carbanion which allows the formation of bonds by alkylation, acylation, and condensation.[2] We here report our results in attempting to functionalize the α position of ethyl cyclopropanecarboxylate as the initial step in a convergent synthesis of pyrrolizidine alkaloids.[3]

Methyl cyclopropanecarboxylate reportedly is deprotonated by lithium diisopropylamide (LDA) in tetrahydrofuran (THF) at −78 °C to give a mixture of *O*- and *C*-trimethylsilyl derivatives in 10% and 40% yields, respectively, after quenching with chlorotrimethylsilane (Me_3SiCl).[4] Nevertheless, sequential mixing of LDA, ethyl

(1) (a) Presented at the 31st Southeastern Regional Meeting of the American Chemical Society, Oct 24–26, 1979, Roanoke, VA. (b) Undergraduate research participant.
(2) House, H. O. "Modern Synthetic Reactions", 2nd ed.; W. A. Benjamin: Menlo Park, CA; 1972, Chapter 9.
(3) Pinnick, H. W.; Chang, Y.-H. *Tetrahedron Lett.* **1979**, 837.
(4) (a) Ainsworth, C.; Chen, F.; Kuo, Y.-N. *J. Organomet. Chem.* **1972**, *46*, 59. (b) Ainsworth reported a 50% yield of distilled Me_3Si derivatives of the ester. Under their conditions, we obtained a nearly 1:1 mixture of Me_3Si compounds and the new material. The latter will not distill, so its presence could have been overlooked very easily.

0022-3263/80/1945-4505$01.00/0