CsF in Organic Synthesis. Malonic Ester Synthesis Revisited for Stereoselective Carbon–Carbon Bond Formation

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Connection of an asymmetric carbon center with a carbon nucleophile with inversion of configuration is an important C–C bond forming process. It is most conveniently achieved by substitution of a nucleofuge by a carbanion, yet this method often suffers from serious drawbacks such as elimination, lack of chemoselectivity, racemization, and so on. Consequently, various improvements have been put forth. Although the Mitsunobu reaction[1] and the use of organocopper reagents[2] serve to a considerable extent, there still exists a strong need for more practical processes, particularly in terms of versatility and feasibility under mild conditions.

In this study, we revisit malonic ester synthesis.[3] This method has been highly appreciated for a long time but needs to be developed to serve the sophisticated purposes of modern organic synthesis. We earlier disclosed that the alkylation of carboxylic acids or organotin carboxylates was effected by CsF in DMF quite smoothly to arrive at carboxylic acid esters without destruction of a wide spectrum of functional groups.[4] In our continuing investigation, we have now found that secondary mesylates can undergo nucleophilic attack by malonic ester derivatives with complete inversion at the asymmetric carbon center under almost neutral conditions.[5]

Mesylate **1** (1 mmol) was treated with malonic ester derivative **2** (3 mmol) in the presence of CsF (3 mmol) in DMF (5 mL) at 45–90 °C for a required period. The virtually stereospecific reaction proceeded to give reasonable yields of the C–C bond formation products with inverted configuration of the asymmetric carbon (eq 1;

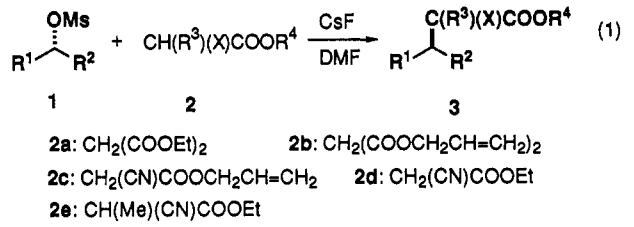
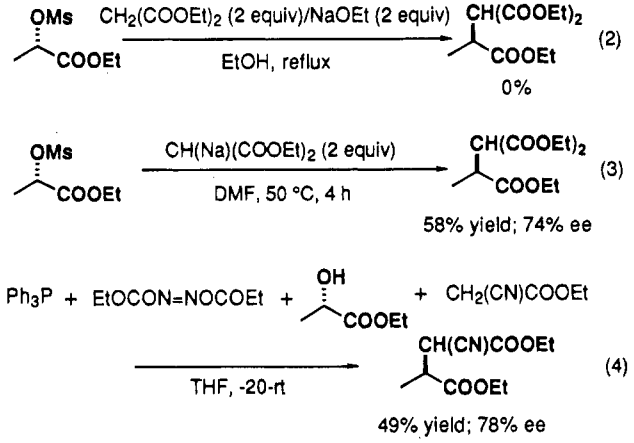


Table 1).[6] Tolerance of both base- and acid-sensitive groups under the present reaction conditions is evident from the reaction of ω-acetoxy or tetrahydropyranyloxy mesylates (Scheme 1). In neither case was the loss of these protecting groups observed.

More importantly, the superiority of our method over the conventional malonic ester synthesis and Mitsunobu reaction is exemplified by equations 2–4. The typical malonic ester synthesis failed to give the desired product (eq 2) and even the use of sodium malonate in DMF gave rise to an unsatisfactory ee (eq 3). The Mitsunobu reaction also resulted in only 78% ee (eq 4).[7]



Application of the ammonium formate reduction to the allyl ester products is particularly noteworthy. Palladium-catalyzed reduction of the allyl esters[8] results in exclusive removal of this group, leaving the others intact (Scheme 2). Accordingly, differentiation from other carboxyl groups or a cyano group is readily achievable, without a decrease of optical purity.

In conclusion, the classical malonic ester synthesis protocol has been extended and applied to higher synthetic purposes. The new version disclosed here offers a highly efficient, practical route to synthetically useful compounds in enantiomerically pure form.

Experimental Section

Commercially available CsF and malonic ester derivatives were used as received. (*S*)-Ethyl 2-[(methanesulfonyl)oxy]-propanoate,[6a] *trans*- and cis-4-*tert*-butyl-1-[(methanesulfonyl)-

(1) (a) Wada, M.; Mitsunobu, O. *Tetrahedron Lett.* **1972**, 1279. (b) Kurihara, T.; Sugizaki, M.; Kime, I.; Wada, M.; Mitsunobu, O. *Bull. Chem. Soc. Jpn.* **1981**, *54*, 2107. (c) Cabaret, D.; Maigrot, N.; Welvart, Z. *Tetrahedron Lett.* **1981**, *22*, 5279. (d) Cabaret, D.; Maigrot, N.; Welvart, Z. *Tetrahedron Lett.* **1984**, *25*, 547. (e) Liu, H.; Wynn, H. *Can. J. Chem.* **1986**, *64*, 658. (f) Macor, J. E.; Wehner, J. M. *Tetrahedron Lett.* **1991**, *32*, 7159. (g) Yu, J.; Falck, J. R.; Mioskowski, C. *J. Org. Chem.* **1992**, *57*, 3757. (h) Yu, J.; Cho, H.; Falck, J. R. *J. Org. Chem.* **1993**, *58*, 5892. For reviews: (i) Mitsunobu, O. *Synthesis* **1981**, 1. (j) Hughes, D. L. *Org. React.* **1992**, *42*, 335.

(2) (a) Johnson, C. R.; Dutra, G. A. *J. Am. Chem. Soc.* **1973**, *95*, 7783. (b) Terashima, S.; Tseng, C. C. Koga, K. *Chem. Pharm. Bull.* **1979**, *27*, 747. (c) Mori, K.; Sugai, T. *Synthesis* **1982**, 752. (d) Thottathil, J. K.; Moiot, J. L. *Tetrahedron Lett.* **1986**, *27*, 5405. (f) Hanessian, S.; Thavonekham, B.; DeHoff, B. *J. Org. Chem.* **1989**, *54*, 5831. (b) Petit, Y.; Sanner, C.; Larchevêque, M. *Tetrahedron Lett.* **1990**, *31*, 2149. For reviews: (h) Posner, G. H. *Org. React.* **1975**, *22*, 253. (i) Lipshutz, B. H.; Sengupta, S. *Org. React.* **1992**, *41*, 135.

(3) House, H. O. *Modern Synthetic Reactions*, 2nd ed.; Benjamin: Menlo Park, CA, 1972; Chapter 9. Carruthers, W. Some *Modern Methods of Organic Synthesis*, 3rd ed.; Cambridge University Press: Cambridge, 1986; p 1.

(4) Sato, T.; Otera, J.; Nozaki, H. *Tetrahedron Lett.* **1989**, *30*, 2959; *J. Org. Chem.* **1992**, *57*, 2166.

(5) Malonic ester synthesis with inversion at the asymmetric carbon center under basic conditions: (a) Shoppee, C. W.; Stephenson, R. J. *J. Chem. Soc.* **1954**, 2230. (b) Marshall, J. A.; Carroll, R. D. *J. Org. Chem.* **1965**, *30*, 2748. (c) Marshall, J. A.; Pile, M. T.; Carroll, R. D. *J. Org. Chem.* **1966**, *31*, 2933. (d) Germain, F.; Chapleur, Y.; Castro, B. *Tetrahedron* **1982**, *38*, 3593. (e) Berens, U.; Scharf, H. *Synthesis* **1991**, 832. (f) Hoffman, R. V.; Kim, H. *Tetrahedron Lett.* **1993**, *34*, 2051. For a related work: Torisawa, Y.; Okabe, H.; Ikegami, S. *J. Chem. Soc. Chem. Commun.* **1984**, 1602.

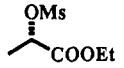
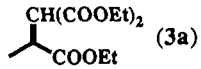
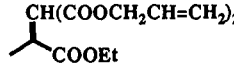
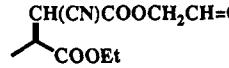
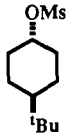
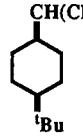
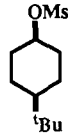
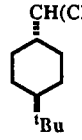
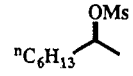
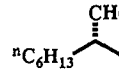
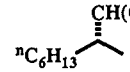
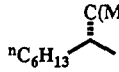
(6) Cesium carboxylate-promoted conversion of secondary mesylates to the corresponding alcohols with inversion of configuration: (a) Kruizinga, W. H.; Strijtveen, B.; Kellog, R. M. *J. Org. Chem.* **1981**, *46*, 4321. (b) Kruizinga, W. H.; Kellog, R. M. *J. Am. Chem. Soc.* **1981**, *103*, 5183. (c) Huffman, J. W.; Desai, R. C. *Synth. Commun.* **1983**, *13*, 553. (d) Torisawa, Y.; Okabe, H.; Ikegami, S. *Chem. Lett.* **1984**, 1555. (e) Burkard, U.; Effenberger, F. *Chem. Ber.* **1986**, *119*, 1594.

(7) Mitsunobu described[1b,i] that hydrolysis of diethyl 2-cyano-3-methylsuccinate obtained in this reaction gave optically pure (*S*)-(−)-methylsuccinic acid in 29% yield after recrystallization from hexane–dichloromethane.

(8) Mandai, T.; Imaji, M.; Takada, H.; Kawada, M.; Nokami, J.; Tsuji, J. *J. Org. Chem.* **1989**, *54*, 5395.

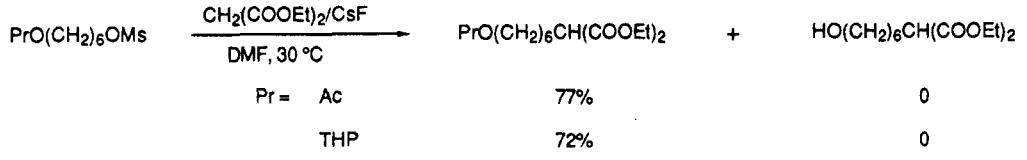
0022-3263/95/1960-2627$09.00/0

Table 1. CsF-Promoted Malonic Ester Synthesis

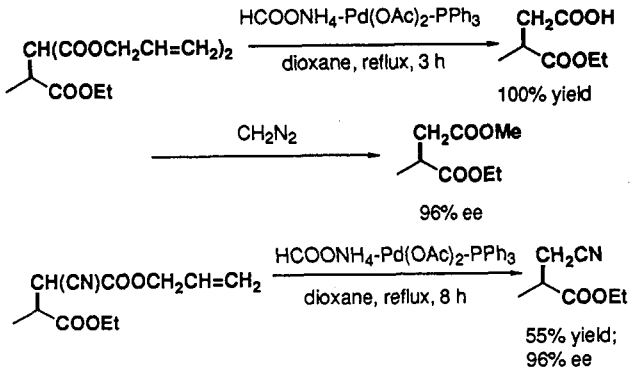
1	2	reaction temp (°C)	reaction time (h)	3	yield (%) GLC	yield (%) isolated	%ee
	2a	45	5	 (3a)	73	68	99[a]
	2b	45	12	 (3b)	70	66	97[a]
	2c	45	6	 (3c)	92	66	96[a]
	2d	90	24	 (3d)	63	57	100:0[b]
	2d	90	24	 (3e)	71	60	0:100[b]
	2a	60	7	 (3f)	60	53	100[a]
	2d	60	5	 (3g)	79	66	96[a]
	2e	60	22	 (3h)	72	65	97[a]

[a] Determined by ^{1}H NMR in the presence of Eu(hfc)$_3$. [b] Cis/trans ratio determined by capillary GLC.

Scheme 1



Scheme 2



oxy]cyclohexane,[9] (*S*)-2-[(methanesulfonyl)oxy]octane,[6a] tetrahydro-2-[[6-[(methanesulfonyl)oxy]hexyl]oxy]-2*H*-pyran,[10] diallyl malonate,[8] and allyl cyanoacetate[8] were prepared according to the literature methods. 1-Acetoxy-6-[(methanesulfonyl)oxy]hexane [R_f = 0.65 (50:50 hexane–ethyl acetate); ^{1}H NMR ($CDCl_3$) δ 1.34–1.85 (m, 8H), 2.04 (s, 3H), 3.02 (s, 3H), 4.05 (t, J = 6.59, 2H), 4.23 (t, J = 6.34, 2H); ^{13}C NMR ($CDCl_3$) δ 20.6, 24.7, 25.0, 28.0, 28.6, 36.9, 63.9, 69.7, 170.8] was prepared from 1,6-hexanediol in two steps [(i) Ac_2O, (ii) MsCl–Et_3N]. DMF was distilled from CaH_2. The %ee values were determined by either ^{1}H NMR spectra (300 or 400 MHz) in the presence of [Eu(hfc)$_3$] or capillary GLC.

CsF-Promoted Reaction of Mesylates with Malonic Ester Derivatives. General Procedure. A mixture of diethyl malonate (0.5 mL, 3 mmol) and CsF (456 mg, 3 mmol) in DMF (4 mL) was stirred for 1 h under a nitrogen atmosphere. To this mixture was added a DMF solution (1 mL) of (*S*)-ethyl 2-[(methanesulfonyl)oxy]propanoate (196 mg, 1 mmol). The mixture was stirred at 45 °C for 5 h. The reaction mixture was extracted with EtOAc (60 mL). The solution was washed with aqueous $NaHCO_3$ (20 mL) followed by brine (20 mL) and dried (Na_2SO_4). After addition of n-$C_{19}H_{40}$ as an internal standard, the solution was subjected to GLC analysis (73% yield). Furthermore, the solution was evaporated and the unreacted diethyl malonate was distilled off (130–140 °C/25 mmHg). The residue was chromatographed on silica gel to furnish (*S*)-diethyl 2-(ethoxycarbonyl)-3-methylbutanedioate (**3a**) (177 mg, 68%): $[\alpha]^{17}_D$ −41.0° (*c* 1.2, $CHCl_3$). This compound was confirmed by comparison with a (±) authentic sample.[11] The absolute configuration of this compound was determined by conversion to (*S*)-dimethyl 2-methylbutanedioate by treatment with concd HCl–CH_3COOH under reflux and subsequently with CH_2N_2. The *S* configuration was determined by $[\alpha]^{17}_D$: −4.0° (*c* 1.0, $CHCl_3$); the (*R*)-authentic sample: $[\alpha]^{29}_D$ 4.1° (*c* 4.1, $CHCl_3$).[12]

(9) Noyce, D. S.; Johnston, B. E.; Weinstein, B. *J. Org. Chem.* **1969**, *34*, 463.
(10) Sato, T.; Otera, J.; Nozaki, H. *J. Org. Chem.* **1990**, *55*, 4770.
(11) Bischoff, C. A. *Chem. Ber.* **1880**, *13*, 2161.
(12) Cohen, S. G.; Milovanovic A. *J. Am. Chem. Soc.* **1968**, *90*, 3495.

(*S*)-1-Allyl 4-ethyl 2-[(allyloxy)carbonyl]-3-methylbutanedioate (3b): $[\alpha]^{24}_D$ −40.4° (*c* 1.1, $CHCl_3$); IR (CCl_4) 1738 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.15 Hz, 3H), 1.25 (d, *J* = 7.14 Hz, 3H), 3.20 (m, 1H), 3.79 (d, *J* = 9.58 Hz, 1H), 4.16 (q, *J* = 7.15 Hz, 2H), 4.61−4.66 (m, 4H), 5.21−5.39 (m, 4H), 5.81−5.98 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 13.9, 14.9, 39.0, 54.4, 60.8, 65.9, 118.4, 118.7, 131.24, 131.28, 167.3, 167.4, 173.7; MS (*m*/*z*) 284 (M^+); HRMS calcd for $C_{12}H_{15}O_5$ ($M^+ - C_2H_5O$) 239.0919, found 239.0907. The absolute configuration of this compound was determined by conversion to (*S*)-dimethyl 2-methylbutanedioate as described above.

(*S*)-1-Allyl 4-ethyl 2-cyano-3-methylbutanedioate (3c): 1:1 diastereomer mixture; $[\alpha]^{17}_D$ −26.5° (*c* 1.3, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 1.27 (t, *J* = 7.08 Hz, 3H × 1/2), 1.28 (t, *J* = 7.21 Hz, 3H × 1/2), 1.41 (d, *J* = 7.20 Hz, 3H × 1/2), 1.42 (d, *J* = 7.39 Hz, 3H × 1/2), 3.1−3.3 (m, 1H), 3.81 (d, *J* = 4.89 Hz, 1H × 1/2), 4.06 (d, *J* = 7.26 Hz, 1H × 1/2), 4.20 (q-like, *J* = 7.15 Hz, 2H), 4.71 (m, 2H), 5.2−5.5 (m, 2H), 5.8−6.0 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 13.7, 13.8, 14.3, 39.1, 39.3, 39.8, 40.3, 61.4, 61.5, 67.0, 67.1, 114.5, 114.7, 119.2, 119.4, 130.4, 130.5, 164.3, 164.4, 171.1, 171.8; MS (*m*/*z*) 284 (M^+); HRMS calcd for $C_{12}H_{15}O_5$ ($M^+ - C_2H_5O$) 239.0919, found 239.0907. The absolute configuration of this compound was determined by conversion to (*S*)-dimethyl 2-methylbutanedioate as described above.

Ethyl *cis*- and *trans*-(4-*tert*-butylcyclohexyl)-2-cyanoacetates (3d and 3e). These compounds were confirmed by comparison with authentic samples.[13]

(*R*)-Ethyl 2-(ethoxycarbonyl)-3-methylnonanoate (3f): $[\alpha]^{25}_D$ 5.48° (*c* 0.97, $CHCl_3$); the (*R*)-authentic sample: $[\alpha]^{25}_D$ 5.0° (*c* 0.77, $CHCl_3$).[14]

(*R*)-Ethyl 2-cyano-3-methylnonanoate (3g): 1:1 diastereomer mixture; $[\alpha]^{22}_D$ 2.4° (*c* 1.15, $CHCl_3$); NMR spectra of this compound were identical with those of the authentic (±)-sample.[15] This compound was converted to (*R*)-(+)-methyl 2-(methoxycarbonyl)-3-methylnonanoate: $[\alpha]^{19}_D$ 8.0° (*c*, 1.0, $CHCl_3$). The authentic specimen of this compound was obtained from **3f**: $[\alpha]^{20}_D$ 8.5° (*c* 1.0, $CHCl_3$); IR (CCl_4) 1738 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.87 (t, *J* = 6.97 Hz, 3H), 0.97 (d, *J* = 6.72 Hz, 3H), 1.10−1.45 (m, 10H), 2.19−2.30 (m, 1H), 3.26 (d, *J* = 8.11 Hz, 1H), 3.726 (s, 3H), 3.728 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 13.9, 16.8, 22.5, 26.6, 29.1, 31.6, 33.4, 34.2, 52.0, 52.1, 57.4, 169.1, 169.3; MS (*m*/*z*) 244 (M^+); HRMS calcd for $C_{13}H_{24}O_4$ (M^+) 244.1675, found 244.1703.

(*R*)-Ethyl 2-cyano-2,3-dimethylnonanoate (3h): 1:1 diastereomer mixture; $[\alpha]^{18}_D$ 17.8° (*c* 1.0, $CHCl_3$); IR (CCl_4) 2240, 1744 m^{-1}; ^1H NMR ($CDCl_3$) δ 0.88 (m, 3H), 1.03 (d, *J* = 6.90 Hz, 3H), 1.14−1.65 (m, 10H), 1.33 (t, *J* = 7.15 Hz, 3H), 1.53 (s, 3H × 1/2), 1.55 (s, 3H × 1/2), 1.99 (m, 1H), 4.27 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 13.6, 13.8, 15.3, 20.3, 20.8, 22.4, 27.0, 28.9, 29.0, 30.8, 31.50, 31.54, 32.8, 39.5, 39.6, 49.1, 49.3, 62.3, 119.1, 119.3, 169.3, 169.5; MS (*m*/*z*) 239 (M^+); HRMS calcd for $C_{14}H_{25}NO_2$ (M^+) 239.1886, found 239.1902. The absolute configuration of this compounds was confirmed by comparison with an authentic sample obtained from **3g** and NaH−MeI: $[\alpha]^{18}_D$ 17.8° (*c*, 1.0, $CHCl_3$).

Ethyl 8-acetoxy-2-(ethoxycarbonyl)octanoate: IR (CCl_4) 1738 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.26 (t, *J* = 7.08 Hz, 6H), 1.3−1.9 (m, 10H), 2.04 (s, 3H), 3.31 (t, *J* = 7.57 Hz, 1H), 4.04 (t, *J* = 6.66 Hz, 2H), 4.20 (q, *J* = 7.08 Hz, 4H); ^{13}C NMR ($CDCl_3$) δ 13.9, 20.8, 25.5, 27.0, 28.3, 28.4, 28.7, 51.8, 61.1, 64.3, 169.4, 171.0; MS (*m*/*z*) 302 (M^+); HRMS calcd for $C_{13}H_{20}O_5$ ($M^+ - C_2H_5OH$) 256.1311, found 256.1314.

Ethyl 2-(ethoxycarbonyl)-8-(2-tetrahydro-2*H*-pyranyloxy)octanoate: IR (CCl_4) 1736 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.26 (t, *J* = 7.14 Hz, 6H), 1.30−1.95 (m, 14H), 3.31 (t, *J* = 7.64 Hz, 1H), 3.35−3.41 (m, 1H), 3.44−3.54 (m, 1H), 3.67−3.77 (m, 1H), 3.81−3.91 (m, 1H), 4.19 (q, *J* = 7.14 Hz, 4H), 4.54−4.59 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.5, 25.3, 25.8, 27.1, 28.5, 28.9, 29.4, 30.6, 51.9, 61.1, 62.1, 67.3, 98.7, 169.4; MS (*m*/*z*) 298 (M^+ − C_2H_5OH); HRMS calcd for $C_{13}H_{23}O_5$ (M^+ − THP) 259.1546, found 259.1523.

Ethyl 2-(ethoxycarbonyl)-8-hydroxyoctanoate: IR (CCl_4) 3644, 1736 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.14 Hz, 6H), 1.30−1.93 (m, 11H), 3.29 (t, *J* = 7.51 Hz, 1H), 3.61 (t, *J* = 6.54 Hz, 2H), 4.18 (q, *J* = 7.14 Hz, 4H); ^{13}C NMR ($CDCl_3$) δ 13.9, 25.2, 27.0, 28.4, 28.8, 32.3, 51.8, 61.1, 62.5, 169.4; MS (*m*/*z*) 260 (M^+); HRMS calcd for $C_{13}H_{25}O_5$ (M^+ + H) 261.1702, found 261.1614.

Palladium-Catalyzed Reduction of Allyl Ester. General Procedure. A mixture of (*S*)-1-allyl-4-ethyl 2-[(allyloxy)carbonyl]-3-methylbutanedioate (853 mg, 3 mmol), $Pd(OAc)_2$ (67 mg, 0.3 mmol), Ph_3P (157 mg, 0.6 mmol), $HCOONH_4$ (946 mg, 15 mmol), and dioxane (20 mL) was heated at reflux for 3 h. The mixture was diluted with ether (30 mL) and washed with saturated $NaHCO_3$ (10 mL × 4). The aqueous layer was acidified with 6 M HCl and then extracted with ethyl acetate (20 mL × 5). The combined organic layer was washed with brine, dried over Na_2SO_4, and evaporated to give an oil. Column chromatography of this oil on silica gel (50:50 hexane−ethyl acetate) furnished (*S*)-1-ethyl 4-hydrogen 2-methylbutanedioate (480 mg, 100%): $[\alpha]^{17}_D$ −5.1° (*c* 1.1, $CHCl_3$); IR (CCl_4) 3020, 1738, 1714 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.24 (d, *J* = 7.21 Hz, 3H), 1.25 (t, *J* = 7.20 Hz, 3H), 2.45 (dd, *J* = 5.50, 16.4 Hz, 1H), 2.79 (dd, *J* = 8.31, 16.4 Hz, 1H), 2.83−2.96 (m, 1H), 4.15 (q, *J* = 7.20 Hz, 2H), 9.50 (br, 1H); ^{13}C NMR ($CDCl_3$) δ 14.0, 16.9, 35.5, 37.2, 60.7, 175.0, 177.9. Physical data were identical with those of the authentic sample.[16] The enantiomeric excess of this compound (96% ee) was determined by ^1H NMR in the presence of Eu(hfc)$_3$ after conversion to (*S*)-1-ethyl-4-methyl 2-methylbutanedioate (CH_2N_2, 68%): $[\alpha]^{17}_D$ −4.3° (*c* 0.75, $CHCl_3$); IR (CCl_4) 1740 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.21 (d, *J* = 7.51 Hz, 3H), 1.25 (t, *J* = 7.14 Hz, 3H), 2.39 (dd, *J* = 6.04, 16.4 Hz, 1H), 2.73 (dd, *J* = 8.18, 16.4 Hz, 1H), 2.83−2.97 (m, 1H), 3.67 (s, 3H), 4.14 (q, *J* = 7.14 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 13.9, 16.7, 35.6, 37.1, 51.4, 60.4, 172.1, 175.0; MS (*m*/*z*) 174 (M^+); HRMS calcd for $C_8H_{14}O_4$ (M^+) 174.0892, found 174.0863.

(*S*)-Ethyl 3-cyano-2-methylpropanoate: $[\alpha]^{17}_D$ −11.0° (*c* 0.9, $CHCl_3$); IR (CCl_4) 2252, 1738 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.29 (t, *J* = 7.09 Hz, 3H), 1.37 (d, *J* = 7.14 Hz, 3H), 2.55 (dd, *J* = 7.69, 17.4 Hz, 1H), 2.68 (dd, *J* = 6.11, 17.4 Hz, 1H), 2.75−2.88 (m, 1H), 4.20 (q, *J* = 7.09 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 13.7, 16.2, 20.5, 35.8, 60.9, 117.6, 172.6; MS (*m*/*z*) 141 (M^+); HRMS calcd for $C_7H_{11}NO_2$ (M^+) 141.0790, found 141.0821.

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Supplementary Material Available: ^1H and ^{13}C NMR charts of new compounds (18 pages). This material is contained in libraries on michrofiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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