Supporting Information for:

Aldol Reaction under Solvent-free conditions: Highly Stereoselective Synthesis of 1,3-Amino alcohols

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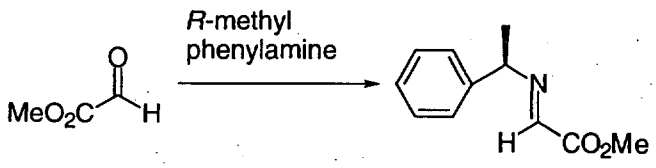
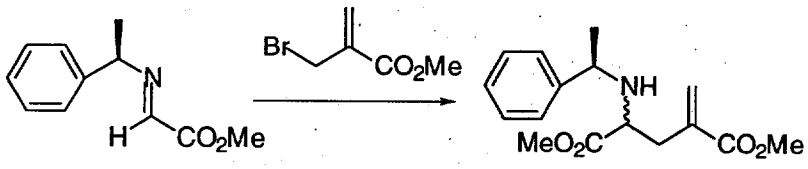
Experimental

Material. Commercial solvents and reagents were used without further purification with the following exceptions: hexane, dichloromethane, ethyl acetate were fractionally distilled; dry dichloromethane, dimethylsulfoxide and triethylamine were distilled from Calcium hydride under nitrogen; diethyl ether and tetrahydrofuran were distilled from Sodium benzophenone ketyl under nitrogen; Methyl glyoxylate and 1-ethoxyl-trimethylsioxyl-ethylene were prepared according to the corresponding literature.

General. Melting points were taken in open capillaries and are uncorrected; Infrared (IR) spectra were recorded on a Perkin-Elmer 1600 FTIR spectrophotometer. Optical rotations were determined using a Jasco DIP-1000 digital polarimeter. ^{1}H NMR and ^{13}C spectra were recorded on a ACF 300, Bruker DPX 300 and Bruker AMX 500 spectrophotometer. Chemical shifts values are expressed in ppm relative to internal trimethylsilane. Multiplicities are given as: s (singlet); d (doublet); t (triplet); q (quartet); dd (double of doublets); or m (multiplets). Mass spectral analyses were carried out on a VG7035 Micromass mass spectrometer and were reported in unit of mass to charge (*m/z*). Electron impact (EI) at an ion current of 70 eV was used for fragmentation of molecules.



Methyl (2*E*)(4*R*)-3-aza-4-phenylpent-2-enoate: A solution of methyl glyoxylate (3.0 g, 34 mmol) in CH Cl_2 (30 mL) was stirred together with anhydrous Na_2SO_4 (8.0 g). After cooling to 0 °C, a solution of *R*-methyl benzylamine (4.4 mL, 34 mmol) in CH_2Cl_2 (10 mL) was added dropwise to the suspension. The reaction mixture was allowed to warm to room temperature and the stirring was continued for another 12 h. The reaction mixture was then filtered through a pad of celite and the filtrate was concentrated under reduced pressure to give the crude product. Purification by vccum silica gel chromatography (5% ethyl acetate in hexane) afforded the imine quantitatively as a light yellow oil in 90% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 (s, 1H), 7.50-7.10 (m, 5H), 4.82 (q, *J* = 6.8 Hz, 1H), 3.82 (s, 3H), 1.70 (d, *J* = 6.8, 3H); ^{13}C NMR (75.4 MHz, $CDCl_3$) d 163.6, 151.8, 142.4, 128.6, 127.4, 125.3, 69.6, 52.5, 23.6.

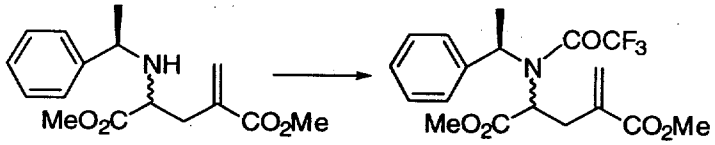


Dimethyl 4-[((*R*)-1-phenylethyl)amino]-2-methylenepentane-1,5-dioate: The indium complex was preformed by stirring allylic bromide (9.3 g, 56 mmol) with the indium metal (6.4 g, 56 mmol) in dry DMF for 2 h. The indium metal disappeared and a green solution of the indium complex was formed. In another vessel, $EtAlCl_2$ (17 ml, 1.8 M solution in toluene, 31 mmol) was added slowly to the imine (5.0 g, 26 mmol) in CH_2Cl_2 (20 mL) at -78 °C. The reaction mixture was stirred at this temperature for 40 min followed by adding the indium complex. The resulted mixture was allowed to warm up to room temperature slowly and stirring was continued for another 3 h. Saturated sodium bicarbonate was added to quench the reaction. The resulted suspension was filtered with a pad of celite followed by repeated

washing with ether (3x100 mL). The organic phase of the combined filtrate was separated and washed with water (4x50 mL), brine (20 mL). The organic phase was dried over magnesium sulfate and concentrated under reduced pressure to provide the crude prodouct. Purification by flash silica gel chromatography (10% ethyl acetate in hexane) afforded the product as a colorless oil (5.3 g, 18 mmol, 70%). The product was obtained as a mixture of two diastereomers in the ratio of 88:12 as determined by ^{1}H NMR analysis. IR (KBr) ν 3333, 1744, 1734, 1631, 1439 cm^{-1}; HRMS Calcd for $C_{16}H_{21}O_4N$: 291.1471. Found: 291.1456.

Dimethyl (4S)-4-[((1*R*)-1-phenylethyl)amino]-2-methyl enepentane-1,5-dioate: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35-7.20 (m, 5H), 6.21 (s, 1H), 5.61 (s, 1H), 3.80-3.70 (m, 2H), 3.78 (s, 3 H), 3.50 (s, 3 H), 2.65 (dd, *J* = 14.0 Hz, *J* =6.5 Hz, 2H), 1.35 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 174.7, 167.1, 145.1, 136.6, 128.2, 127.2, 127.0, 126.6, 58.3, 56.2, 51.7, 51.3, 35.8, 22.9.

Dimethyl (4*R*)-4-[((1*R*)-1-phenylethyl)amino]-2-methyl enepentane-1,5-dioate: IR (KBr) ν 3333, 1744, 1734, 1631, 1439 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35–7.20 (m, 5H), 6.21 (s, 1H), 5.51 (s, 1H), 3.80-3.70 (m, 2H), 3.69 (s, 3H), 3.65 (s, 3H), 2.55 (d, *J* = 7.0 Hz, 2H), 1.35 (d, *J* = 6.8 Hz, 3H).

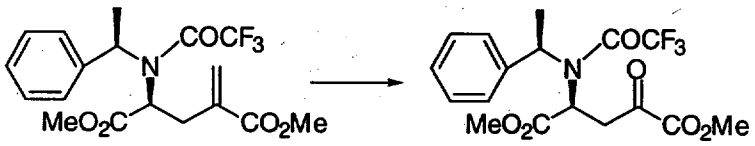


A mixture of pyridine (3.3 mL, 41.1 mmol) and the amino ester (4.0 g, 13.7 mmol) in CH_2Cl_2 (20mL) was treated with trifluoroacetic acid anhydride (2.1 mL, 27.4 mmol) at 0 °C. The reaction mixture was allowed to warm to room temperature and stirring was continued for another 12 h. The reaction mixture was then slowly poured into ice-water (30 mL), then ether (100 mL) was added to the mixture. The organic phase was separated and washed with 1M HCl solution (2x20 mL), water (50 mL) and brine (10 mL). The organic phase was dried over magnesum sulfate and concentrated under reduced pressure to provide the crude product. The diastereomers can be separated by silica gel chromatography (hexane, 5% ether in hexane, 10%

ether in hexane then 20% ether in hexane). The 5% ether in hexane solvent eluted out the minor *(R,R)* -isomer (2.1g, 40%) as a solid and the 10% ether in hexane solvent eluted out the major *(R,S)*-isomer (3.2g, 60%) as a solid.

Dimethyl (4*S*)-4-[N-((1*R*)-1-phenylethyl)-2,2,2-trifluoro acetyl amino]-2-methylenepentane-1,5-dioate: $[\alpha]^{31}_D$ = - 32 (c 1.13, EtOAc); mp 100-101 °C ; IR (KBr) ν 1743, 1705, 1691, 1634, 1450 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.30 (m, 5H), 5.78 (s, 1H), 5.29 (q, *J* = 6.8 Hz, 1H), 5.08 (s, 1 H), 4.05 (dd, *J* = 5.9 Hz, *J* = 7.8 Hz, 1H), 3.70 (s, 3H), 3.55 (s, 3H), 3.10 (dd, *J* = 5.9 Hz, *J* = 14.4 Hz, 1H), 2.40 (dd, *J* = 7.8 Hz, *J* = 14.4 Hz, 1H), 1.55 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 169.5, 166.3, 156.4 (q, *J* = 35.7 Hz), 136.7, 135.7, 128.6, 128.5, 127.7, 127.6, 116.50(q, *J* = 287.5 Hz), 57.2, 55.9, 52.4, 52.4, 30.8, 17.1; HRMS Calcd for $C_{18}H_{20}O_5NF_3$: 387.1294. Found: 387.1293.

Dimethyl (4*R*)-4-[N-((1*R*)-1-phenylethyl)-2,2,2-trifluoro acetylamino]-2-methylenepentane-1,5-dioate: $[\alpha]^{31}_D$ = + 55 (c 1.29, EtOAc); mp 67-69 °C ; IR (KBr) ν 1743, 1705, 1691, 1634, 1450 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.45-7.25 (m, 5H), 6.22 (s, 1H), 5.85 (s, 1H), 5.35 (q, *J* = 6.8 Hz, 1H), 4.05 (dd, *J* = 5.9 Hz, *J* = 7.8 Hz, 1H), 3.80 (s, 3 H), 3.40 (s, 3 H), 3.39-3.20 (m, 1 H), 2.70 (dd, *J* = 7.8 Hz, *J* = 14.4 Hz, 1H), 1.55 (d, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 168.7, 167.3, 156.4 (q, *J* = 35.7 Hz), 136.2, 135.9, 130.1, 128.7, 128.5, 128.1, 116.50(q, *J* = 287.5 Hz), 57.2, 56.1, 55.6, 51.7, 32.6, 17.6.

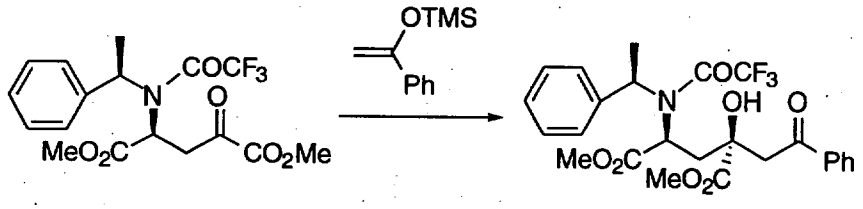


Ozone was bubbled into a stirred solution of alkene (1.80 g, 4.6 mmol) in CH_2Cl_2 at -78 °C for 10 min until the blue color of the saturated ozone solution persisted. The solution was then purged with nitrogen for 5 min. Excess dimethyl sulfide was added to quench the ozonalide. The reaction was allowed to warm to room temperature over a period of 2 h. The

CH_2Cl_2 was removed under reduced pressure. The crude product was purified by flash silica gel chromatography (10% ethyl acetate in hexane) affording a solid product (1.63 g, 4.18 mmol, 90%).

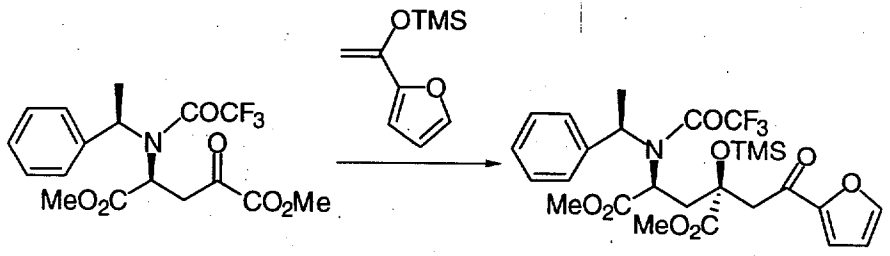
(*R,S*)-**1a**: **Dimethyl (2*S*)-2-[N-((1*R*)-1-phenylethyl)-2,2,2-trifluoro acetyl amino]-4-oxopentane-1,5-dioate:** $[\alpha]^{33}_D$ = - 70 (c 1.97, EtOAc); mp 81-82 °C ; IR (KBr) ν 1744 , 1731, 1686, 1450 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.36-7.28 (m, 5H), 5.41 (q, *J* = 6.6 Hz, 3H), 4.39 (dd, *J* = 2.2 Hz, *J* = 9.8 Hz, 1H), 3.89 (dd, *J* = 9.8 Hz, *J* = 18.5 Hz, 1H), 3.77 (s, 3H), 3.68 (s, 3H), 1.78 (dd, *J* = 2.2 Hz, *J* = 18.5 Hz, 1H), 1.76 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 189.1, 169.0, 160.0, 156.3 (q, *J* = 35.9 Hz), 136.3, 129.1, 128.9, 127.7, 116.3(q, *J* = 285.9 Hz), 56.0 (q, *J* = 3.2 Hz), 53.0, 52.9, 52.6, 39.2, 16.6; HRMS Calcd for $C_{17}H_{18}NO_6F_3$: 389.1086. Found: 389.1092.

General procedure for aldol reaction: The ketone (0.05 mmol) was first activated by using Lewis acid (0.01 mmol) and then adding the silyl enol ether or ketene silyl acetal (0.25 mmol). The reaction mixture was then stirred at room temperature for 16 h. THF (2 mL) and hydrochloric acid (1M, 1mL) was then added followed by stirring for 2 h. Ether (25 mL) was then added to the reaction mixture. The organic phase was washed with saturated $NaHCO_3$ (5 mL), water (10 mL) and brine (5 mL). The organic phase was dried over magnesium sulfate and concentrated to give the crude product. Purification by flash silica gel chromatography (10% ethyl acetate in hexane) afforded the product.

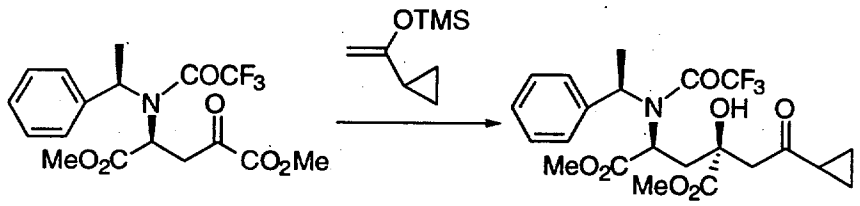


Dimethyl 4-[N-((1R)-1-phenylethyl)-2,2,2-trifluoroacetylamino] (4S)-2-hydroxy-2-(2-oxo-2-phenylethyl)pentane-1,5-dioate (18.6 mg, 73%): colorless oil; IR (KBr) ν 3491, 1792, 1749, 1685, 1599, 1451 cm^{-1}; ^1H NMR (300 MHz, $CDCL_3$) δ 7.78-7.26 (m, 10H), 5.42 (q, *J* = 6.9 Hz, 1H), 4.25 (d, *J* = 9.0 Hz, 1H), 3.70 (s,

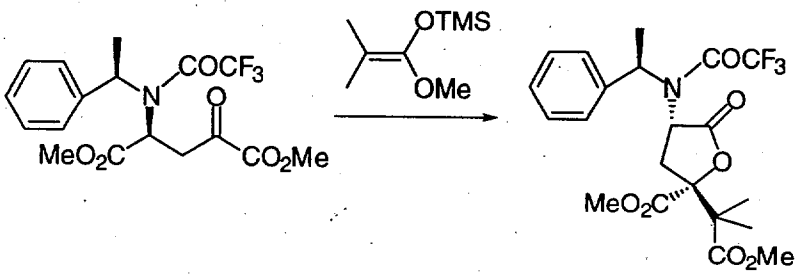
3H), 3.58 (s, 3H), 3.18 (d, J = 17.1 Hz, 1H), 3.04 (dd, J = 9.0 Hz, J = 14.4 Hz, 1H), 2.54 (d, J = 17.1 Hz, 1H), major: 1.76 (d, J = 6.9 Hz, 3H); minor: 1.86 (d, J = 6.9 Hz, 3H), 1.26 (d, J = 14.4, 1H); ^{13}C NMR (75.4 MHz, $CDCL_3$) δ 197.8, 175.0, 170.2, 156.7 (q, J = 35.7 Hz), 136.7, 136.3, 133.5, 128.8, 128.6, 128.5, 128.3, 128.1, 116.5 (q, J = 286.0 Hz), 72.5, 56.0 (q, J = 3.3 Hz), 52.6, 52.0, 47.7, 40.3, 29.6, 16.9; HRMS Calcd for $C_{25}H_{26}O_7NF_3$: 509.1661. Found: 509.1635.



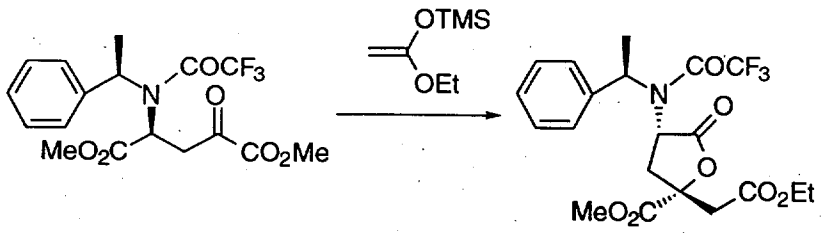
Dimethyl 4-[N-((*1R*)-1-phenylethyl)-2,2,2-trifluoroacetylamino] (*4S,2R*)-2-(1,1-dimethyl-1-silaethoxy)-2-(2-(2-furyl)-2-oxoethyl)pentane-1,5-dioate (16.0 mg, 56%): colorless oil; $[\alpha]^{32}_D$ = - 65 (c 0.13, EtOAC); IR (KBr) ν 1753, 1687, 1562, 1457 cm^{-1}; ^{1}H NMR (300 MHz, $CDCL_3$) δ 7.57 (s, 1H), 7.41-7.36 (m, 5H), 6.98 (d, J = 3.5 Hz, 1H), 6.54-6.52 (m, 1H), 5.39 (q, J = 6.8 Hz, 1H), 4.11 (d, J = 10.1 Hz, 1H), 3.67 (s, 3 H), 3.63 (s, 3 H), 3.14 (dd, J = 10.1 Hz, J = 14.0 Hz, 1 H), 2.62 (d, J = 14.0 Hz, 1H), 2.47 (d, J = 14.0 Hz, 1 H), 1.78 (d, J = 6.8 Hz, 3 H), 1.30 (d, J = 14.5 Hz, 1H), -0.05 (s, 9H); ^{13}C NMR (75.4 MHz, $CDCL_3$) δ 176.0, 170.5, 169.5, 156.7 (q, J = 35.6 Hz), 153.2,146.4, 140.7, 128.9, 128.3, 128.0, 117.7, 116.5 (q, J = 284.9 Hz), 112.2, 76.8, 55.7 (q, J = 3.3 Hz), 53.1, 52.2, 52.1, 49.3, 39.4, 17.0, 2.3; HRMS Calcd for $C_{26}H_{32}O_8SiNF_3$: 571.1849. Found: 571.1860.



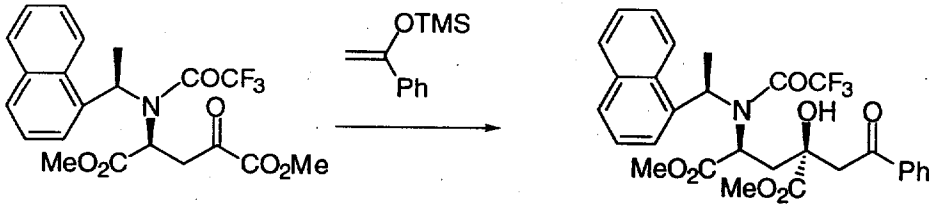
Dimethyl 4-[N-((*1R*)-1-phenylethyl)-2,2,2-trifluoroacetylamino] (*4S*)-2-(2-cyclopropyl-2-oxoethyl)-2-hydroxypentane-1,5-dioate (17.0 mg, 72%): colorless oil;: IR (KBr) ν 3487, 1748, 1688, 1452, 1392 cm^{-1}; ^1H NMR (300 MHz, $CDCL_3$) δ 7.43–7.35 (m, 5H), 5.40 (q, *J* = 6.9 Hz, 1H), 4.12 (d, *J* = 9.1 Hz, 1H), 3.68 (s, 3H), 3.67 (s, 3H), 2.94 (dd, *J* = 9.1 Hz, *J* = 14.3 Hz, 1H), 2.65 (d, *J* = 17.0 Hz, 1H), 1.97 (d, *J* = 17.0 Hz, 1H), major: 1.74 (d, *J* = 6.8 Hz, 3H); minor: 1.80 (d, *J* = 6.8 Hz, 3H), 1.71-1.63 (m, 1H), 1.20 (d, *J* = 14.3, 1H), 0.96-0.81 (m, 4H); ^{13}C NMR (75.4 MHz, $CDCL_3$) δ 208.4, 174.9, 170.4, 156.7 (q, *J* = 35.8 Hz), 136.8, 128.9, 128.6, 128.3, 116.5 (q, *J* = 285.9 Hz), 72.3, 55.9 (q, *J* = 3.2 Hz), 52.7, 52.5, 52.1, 52.0, 40.0, 21.0, 16.9, 11.0, 10.9; HRMS Calcd for $C_{22}H_{26}O_7NF_3$: 473.1661. Found: 473.1659.



Methyl 2-{4-[N-((1R)-1-phenylethyl)-2,2,2-trifluoroacetylamino] (*4S,2R*)-2-(methoxycarbonyl)-5-oxo(2-2,3,4-trihydrofuryl)}-2-methyl propanoate: (16.5 mg, 72%): colorless oil; $[\alpha]^{25}_D$ = - 4 (c 1.15, EtOAC); IR (KBr) ν 1794, 1735, 1684, 1457 cm^{-1}; ^1H NMR (300 MHz, $CDCL_3$) δ 7.48-7.32 (m, 5H), 5.36 (q, *J* = 6.8 Hz, 1H), 3.94 (t, *J* = 9.0 Hz, 1H), 3.82 (s, 3H), 3.58 (s, 3 H), 2.22 (d, *J* = 9.0 Hz, 2H), 1.79 (d, *J* = 6.8 Hz, 3H), 1.23 (s, 3H), 1.21 (s, 3H); ^{13}C NMR (75.4 MHz, $CDCL_3$) δ 174.2, 171.6, 171.1, 155.9 (q, *J* = 36.8 Hz), 137.4, 129.1, 128.7, 127.3, 116.2 (q, *J* = 284.9 Hz), 87.6, 55.8 (q, *J* = 3.3 Hz), 53.7, 52.8, 52.2, 48.2, 32.6, 21.4, 22.9, 17.3; HRMS Calcd for $C_{21}H_{24}O_7NF_3$: 459.1505. Found: 459.1514.

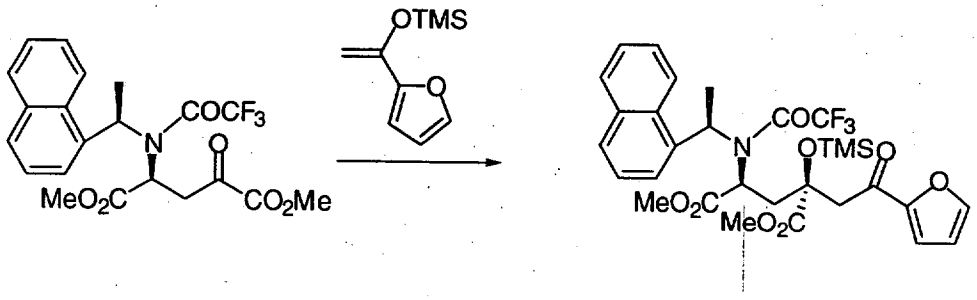


Ethyl 2-{4-[N-((*1R*)-1-phenylethyl)-2,2,2-trifluoroacetylamino] (*4S*)-2-(methoxycarbonyl)-5-oxo-2-2,3,4-trihydrofuryl}acetate (12.9 mg, 58%): colorless oil; IR (KBr) ν 1798, 1744, 1728, 1682, 1498, 1457 cm^{-1}; ^1H NMR (300 MHz, $CDCL_3$) δ 7.45-7.32 (m, 5H), 5.36 (q, *J* = 6.7 Hz, 1H), major:4.16 (dd, *J*=7.5 Hz, *J* = 11.0 Hz, 1 H); minor: 4.30 (dd, *J* = 6.2 Hz, *J* = 7.0 Hz, 1H), 4.11-3.98 (m, 2H), 3.82 (s, 3H), 2.88 (d, *J* = 17.2 Hz, 1H), 2.64 (d, *J* = 17.2 Hz, 1H), 2.41 (dd, *J* = 7.5 Hz, *J* = 13.8 Hz, 1H), 1.85 (dd, *J* = 11.0 Hz, *J* = 13.8 Hz, 1H), 1.77 (d, *J* = 6.7 Hz, 3H), 1.18 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75.4 MHz, $CDCL_3$) δ 171.3, 171.0, 168.3, 155.9 (q, *J* = 36.8 Hz), 137.4, 129.1, 128.7, 127.3, 116.2 (q, *J* = 284.9 Hz), 80.6, 61.1, 55.8, (q, *J* = 3.3 Hz), 53.5, 53.3, 41.0, 33.7, 17.1, 13.9; HRMS Calcd for $C_{20}H_{22}O_7NF_3$: 445.1348. Found: 445.1345.

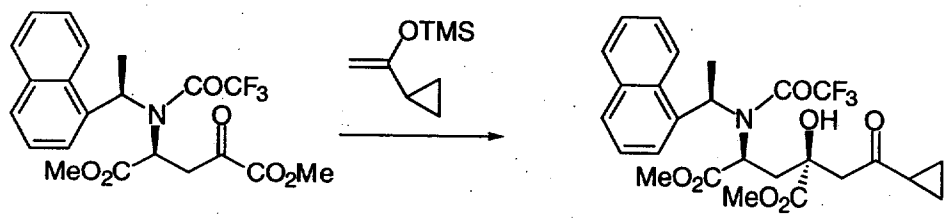


Dimethyl 4-[N-((*1R*)-1-naphthylethyl)-2,2,2-trifluoroacetylamino] (*4S*)-2-hydroxy-2-(2-oxo-2-phenylethyl)pentane-1,5-dioate (20.4 mg, 73%): colorless oil; IR (KBr) ν 3476, 1747, 1681, 1599, 1449 cm^{-1}; ^1H NMR (300 MHz, $CDCL_3$) δ 8.09-7.40 (m, 12H), 6.03 (q, *J* = 6.8 Hz, 1H), 4.23 (d, *J* = 9.2 Hz, 1H), 3.82 (s, 3H), 3.67 (s, 3 H), 2.86 (dd, *J* = 9.2 Hz, *J* = 14.7 Hz, 1H), 2.79 (d, *J* = 17.1 Hz, 1H), major: 2.03 (d, *J* = 6.8 Hz, 3H); minor: 1.96 (d, *J* = 6.8 Hz, 3H), 1.60 (d, *J* = 17.1, 1H), 0.83 (d, *J* = 14.7 Hz, 1H); ^{13}C NMR (125.7 MHz, $CDCL_3$) δ 197.8, 175.2, 170.2, 157.4 (q, *J* = 36.2 Hz), 136.3, 134.2, 133.5, 132.2, 130.3, 129.3, 128.5, 128.1, 127.8, 127.3, 126.8, 125.7, 125.6, 122.4

(q, J = 2.2 Hz), 115.9 (q, J = 284.4 Hz), 71.8, 54.2, 53.0, 52.6, 47.9, 40.2, 29.7, 19.3; HRMS Calcd for $C_{29}H_{28}O_7NF_3$: 559.1818. Found: 559.1830.

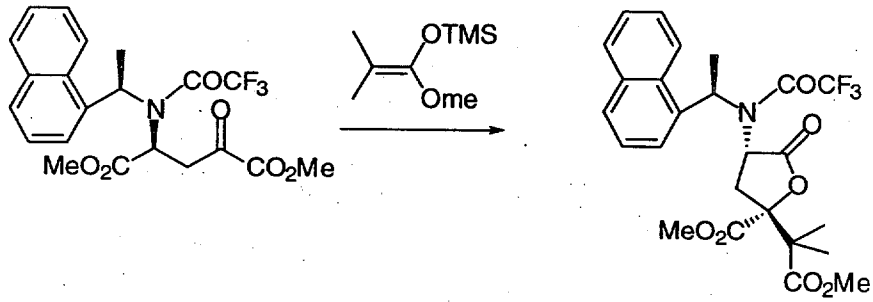


Dimethyl 4-[N-((*1R*)-1-naphthylethyl)-2,2,2-trifluoroacetylamino] (*4S,2R*)-2-(1,1-dimethyl-1-silaethoxy)-2-(2-(2-furyl)-2-oxoethyl)pentane-1,5-dioate(17.1 mg, 55%): colorless oil;: $[\alpha]^{25}_D$ = - 89 (c 1.03, EtOAC); IR (KBr) ν 1754, 1682, 1567, 1467, 1446 cm^{-1}; ^1H NMR (300 MHz, $CDCL_3$) δ 8.09-7.54 (m, 7H), 6.86 (d, J = 3.5 Hz, 1H), 6.54-6.52 (m, 1H), 6.05 (q, J = 6.8 Hz, 1H), 4.23 (d, J = 10.0 Hz, 1H), 3.65 (s, 3H), 3.62 (s, 3H), 3.04 (dd, J = 10.0 Hz, J = 14.5 Hz, 1H), 2.15 (d, J = 14.0 Hz, 1H), 2.04 (d, J = 14.0 Hz, 1H), 1.97 (d, J = 6.8 Hz, 3H), 1.09 (d, J = 14.5 Hz, 1H), - 0.10 (s, 9H); ^{13}C NMR (75.4 MHz, $CDCL_3$) δ 184.5, 173.4, 169.6, 157.5 (q, J = 35.8 Hz), 153.0, 146.4, 133.6, 133.4, 131.4, 129.4, 129.0, 127.2, 126.2, 126.1, 125.8, 122.3, 117.7, 116.4 (q, J = 286.0 Hz), 112.1, 77.1, 54.2, 53.9 (q, J = 2.2 Hz), 52.4, 52.1, 48.9, 39.4, 19.5, 2.3; HRMS Calcd for $C_{30}H_{34}O_8SiNF_3$: 621.2006. Found: 621.2002.

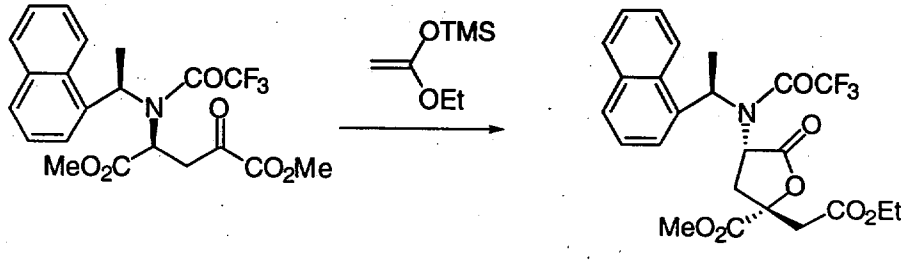


Dimethyl 4-[N-((*1R*)-1-naphthylethyl)-2,2,2-trifluoroacetylamino] (*4S*)-2-(2-cyclopropyl-2-oxoethyl)-2-hydroxypentane-1,5-dioate (18.9 mg, 72%): colorless oil; IR (KBr) ν 3483, 1747, 1680, 1638, 1440cm^{-1}; ^1H NMR (300 MHz, $CDCL_3$) δ 8.05–7.50 (m, 7H), 6.00 (q, J = 6.7 Hz, 1H), 4.13 (d, J = 9.1 Hz, 1H), 3.66 (s, 3H), 3.59 (s, 3H), 2.76 (dd, J = 9.1 Hz, J = 14.7 Hz, 1 H), 2.16 (d, J = 16.9 Hz, 1H), 1.94 (d, J = 6.7

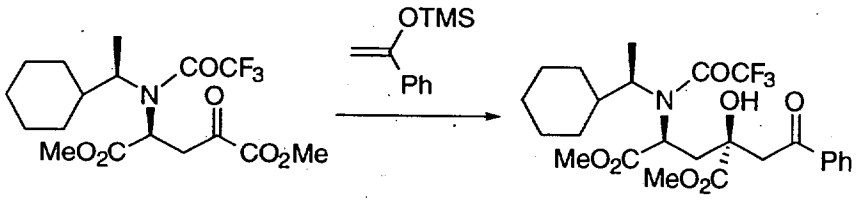
Hz, 3 H), 1.54-1.45 (m, 1H), 1.04 (d, J = 16.9 Hz, 1H), 0.82-0.75 (m, 4H), 0.70 (d, J = 14.7 Hz, 1H); ^{13}C NMR (75.4 MHz, $CDCL_3$) δ 208.4, 174.9, 170.3, 157.2 (q, J = 36.8 Hz), 134.2, 132.1, 132.0, 130.2, 129.1, 127.2, 126.8, 125.8, 125.5, 122.3 (q, J = 3.2 Hz), 116.5 (q, J = 285.9 Hz), 71.5, 54.1 (q, J = 3.0 Hz), 52.9, 52.6, 52.4, 51.0, 39.7, 20.8, 19.3, 10.7, 10.6; HRMS Calcd for $C_{26}H_{28}O_7NF_3$: 524.1896. Found: 524.1874.



Methyl 2-{4-[N-((*1R*)-1-naphthylethyl)-2,2,2-trifluoroacetyl amino](*4S*)-2-(methoxycarbonyl)-5-oxo(2-2,3,4-trihydrofuryl)}-2-methylpropanoate (18.8 mg, 74%): colorless oil; $[\alpha]^{25}_D$ = - 64 (c 0.20, EtOAC); mp 139-141 °C IR (KBr) ν 1796, 1741, 1722, 1676, 1474, 1459 cm^{-1}; ^{1}H NMR (300 MHz, $CDCL_3$) δ 7.95-7.53 (m, 7H), 5.96 (q, J = 6.6 Hz, 1H), 3.92 (dd, J = 7.0 Hz, J = 11.4 Hz, 1H), 3.77 (s, 3H), 3.47 (s, 3H), 1.97 (d, J = 7.0 Hz, 3H), 1.82 (dd, J = 7.0 Hz, J = 14.5 Hz, 1H), 1.45 (dd, J = 11.4 Hz, J = 14.5 Hz, 1H), 1.07 (s, 3 H), 0.94 (s, 3H); ^{13}C NMR (75.4 MHz, $CDCL_3$) δ 173.9, 171.8, 170.9, 156.4 (q, J = 37.1 Hz), 133.9, 132.8, 131.1, 130.4, 129.3, 127.5, 126.5, 126.0, 125.0, 122.0 (q, J = 2.8 Hz), 116.4 (q, J = 285.9 Hz), 87.5, 54.1, 53.9 (q, J = 2.8 Hz), 52.9, 52.1, 48.2, 32.0, 21.3, 20.7, 19.4; HRMS Calcd for $C_{25}H_{26}O_7NF_3$: 509.1661. Found: 509.1667.



Ethyl 2-{4-[N-((*1R*)-1-naphthylethyl)-2,2,2-trifluoroacetylamino] (*4S*)-2-(methoxycarbonyl)-5-oxo-2-2,3,4-trihydrofuryl}acetate (13.4 mg, 54%): colorless oil; IR (KBr) ν 1797, 1737, 1679, 1447 cm^{-1}; ^1H NMR (300 MHz, $CDCL_3$) δ 8.01-7.47 (m, 7H), 5.93 (q, *J* = 6.6 Hz, 1H), major: 4.17 (dd, *J* =7.9 Hz, *J* = 10.1 Hz, 1 H); minor: 4.27 (dd, *J* = 5.7 Hz, *J* = 7.4 Hz, 1H), 4.03-3.93 (m, 2H), major: 3.74 (s, 3H); minor: 3.65 (s, 3H), 2.73 (d, *J* = 17.1 Hz, 1H), 2.43 (d, *J* = 17.1 Hz, 1H), 2.01 (dd, *J* = 7.9 Hz, *J* = 13.8 Hz, 1H), 1.95 (d, *J* = 6.6 Hz, 3H), 1.06 (dd, *J* = 10.1 Hz, *J* = 13.8 Hz, 1H), 1.15 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (75.4 MHz, $CDCL_3$) δ 171.4, 171.8, 168.0, 156.3 (q, *J* = 36.8 Hz), 137.7, 132.3, 131.0, 130.3, 129.2, 127.3, 126.4, 126.1, 125.1, 121.8 (q, *J* = 3.3 Hz), 116.3 (q, *J* = 286.0 Hz), 80.5, 61.0, 54.0, 53.9 (q, *J* = 2.2 Hz), 53.2, 40.9, 33.2, 19.1, 13.8; HRMS Calcd for $C_{24}H_{24}O_7NF_3$: 495.1505. Found: 495.1481.



Dimethyl 4-[N-((*1R*)-1-cyclohexylethyl)-2,2,2-trifluoroacetylamino] (*4S*)-2-hydroxy-2-(2-oxo-2-phenylethyl)pentane-1,5-dioate (18.0 mg, 70%): colorless oil; $[\alpha]^{25}_D$ = + 63 (c 0.17, EtOAc); IR (KBr) ν 3507, 3063, 2922, 2853, 1761, 1746, 1699, 1664, 1470 cm^{-1}; ^1H NMR (300 MHz, $CDCL_3$) δ 7.92-7.44 (m, 5H), 4.46 (d, *J* = 8.4 Hz, 1H), 3.82 (d, *J* = 18.0 Hz, 1H), 3.80-3.69 (m, 1H), 3.75 (s, 3H), 3.65 (s, 3H), 3.33 (dd, *J* = 8.4 Hz, *J* = 14.4 Hz, 1H), 3.20 (d, *J* = 18.0 Hz, 1H), 2.18-0.82 (m, 11H), 1.74 (d, *J* = 14.4 Hz, 1H), 1.36 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 198.4, 174.9, 170.0, 157.4 (q = 35.6 Hz), 136.1, 133.8, 128.7, 128.1, 116.4 (q, *J* = 285.8 Hz), 74.0, 60.4, 52.8, 52.4, 51.5, 48.7, 41.9, 40.2, 31.2, 29.6, 26.3, 26.1, 25.9, 17.1; HRMS Calcd for $C_{25}H_{32}O_7NF_3$: 515.2131. Found: 515.2111.

Crystallographic Data of (*R*,*S*)-7

Table 1. Crystal data and structure refinement.

Crystal growing solvent	ether and hexane	
Empirical formula	C22 H22 F3 N O5	
Formula weight	437.41	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 7.5376(1) Å	α= 90°.
	b = 13.9892(1) Å	β= 90°.
	c = 20.1185(2) Å	γ = 90°.
Volume	2121.40(4) $Å^3$	
Z	4	
Density (calculated)	1.370 Mg/m^3	
Absorption coefficient	0.114 mm^{-1}	
F(000)	912	
Crystal size	0.35 x 0.30 x 0.20 mm^3	
Theta range for data collection	1.77 to 29.35°.	
Index ranges	-10<=h<=8, -19<=k<=18, -26<=l<=24	
Reflections collected	13869	
Independent reflections	5336 [R(int) = 0.0178]	
Absorption correction	Sadabs (Sheldrick, 1996)	
Max. and min. transmission	0.9655 and 0.8343	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5335 / 6 / 281	
Goodness-of-fit on F^2	1.037	
Final R indices [I>2sigma(I)]	R1 = 0.0398, wR2 = 0.0948	
R indices (all data)	R1 = 0.0539, wR2 = 0.1042	
Absolute structure parameter	-0.13(67)	
Extinction coefficient	0.0062(10)	
Largest diff. peak and hole	0.170 and -0.170 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
F(1)	3015(2)	-1558(1)	759(1)	86(1)
F(2)	3407(2)	-485(1)	1477(1)	81(1)
F(3)	2119(2)	-1775(1)	1746(1)	96(1)
O(1)	-804(2)	-970(1)	1475(1)	62(1)
O(2)	-4035(2)	1325(1)	-469(1)	70(1)
O(3)	-3955(2)	386(1)	-1360(1)	72(1)
O(4)	-3472(2)	1013(1)	1517(1)	78(1)
O(5)	-571(2)	1094(1)	1702(1)	62(1)
N(1)	227(2)	-10(1)	658(1)	38(1)
C(1)	1208(2)	588(1)	-451(1)	42(1)
C(2)	747(2)	1481(1)	-673(1)	52(1)
C(3)	273(3)	1644(2)	-1341(1)	66(1)
C(4)	267(3)	920(2)	-1781(1)	67(1)
C(5)	750(2)	-14(2)	-1587(1)	56(1)
C(6)	1246(2)	-188(1)	-915(1)	45(1)
C(7)	1748(3)	-1126(1)	-747(1)	60(1)
C(8)	1749(4)	-1847(2)	-1202(1)	81(1)
C(9)	1244(4)	-1671(2)	-1857(1)	84(1)
C(10)	754(3)	-784(2)	-2046(1)	75(1)
C(11)	1724(2)	438(1)	276(1)	39(1)
C(12)	2375(2)	1337(1)	620(1)	51(1)
C(13)	408(2)	-692(1)	1129(1)	47(1)
C(14)	2249(3)	-1132(1)	1272(1)	57(1)
C(15)	-1505(2)	475(1)	656(1)	42(1)
C(16)	-3049(2)	-141(1)	404(1)	53(1)
C(17)	-3154(2)	-281(1)	-333(1)	49(1)
C(18)	-2823(4)	-1104(2)	-616(1)	82(1)
C(19)	-3744(2)	563(1)	-718(1)	50(1)
C(20)	-4571(4)	1181(2)	-1756(1)	95(1)
C(21)	-1984(3)	877(1)	1342(1)	51(1)
C(22)	-934(4)	1458(2)	2363(1)	85(1)

Table 3. Bond lengths [Å] and angles.

F(1)-C(14)	1.324(2)	C(2)-C(3)	1.408(3)
F(2)-C(14)	1.324(2)	C(3)-C(4)	1.347(3)
F(3)-C(14)	1.313(2)	C(4)-C(5)	1.411(3)
O(1)-C(13)	1.213(2)	C(5)-C(10)	1.418(3)
O(2)-C(19)	1.199(2)	C(5)-C(6)	1.424(2)
O(3)-C(19)	1.325(2)	C(6)-C(7)	1.406(3)
O(3)-C(20)	1.444(3)	C(7)-C(8)	1.363(3)
O(4)-C(21)	1.191(2)	C(8)-C(9)	1.394(4)
O(5)-C(21)	1.323(2)	C(9)-C(10)	1.348(4)
O(5)-C(22)	1.450(2)	C(11)-C(12)	1.518(2)
N(1)-C(13)	1.351(2)	C(13)-C(14)	1.546(3)
N(1)-C(15)	1.472(2)	C(15)-C(21)	1.532(2)
N(1)-C(11)	1.502(2)	C(15)-C(16)	1.534(2)
C(1)-C(2)	1.371(2)	C(16)-C(17)	1.497(2)
C(1)-C(6)	1.433(2)	C(17)-C(18)	1.309(3)
C(1)-C(11)	1.528(2)	C(17)-C(19)	1.481(3)

C(19)-O(3)-C(20)	115.6(2)	C(8)-C(7)-C(6)	121.9(2)
C(21)-O(5)-C(22)	115.5(2)	C(7)-C(8)-C(9)	120.3(2)
C(13)-N(1)-C(15)	114.56(13)	C(10)-C(9)-C(8)	120.2(2)
C(13)-N(1)-C(11)	125.30(14)	C(9)-C(10)-C(5)	121.1(2)
C(15)-N(1)-C(11)	118.25(12)	N(1)-C(11)-C(12)	110.81(12)
C(2)-C(1)-C(6)	118.89(15)	N(1)-C(11)-C(1)	110.85(12)
C(2)-C(1)-C(11)	120.15(15)	C(12)-C(11)-C(1)	113.87(13)
C(6)-C(1)-C(11)	120.94(14)	O(1)-C(13)-N(1)	123.6(2)
C(1)-C(2)-C(3)	121.6(2)	O(1)-C(13)-C(14)	116.19(15)
C(4)-C(3)-C(2)	120.4(2)	N(1)-C(13)-C(14)	120.15(15)
C(3)-C(4)-C(5)	120.9(2)	F(3)-C(14)-F(1)	106.9(2)
C(4)-C(5)-C(10)	121.6(2)	F(3)-C(14)-F(2)	106.9(2)
C(4)-C(5)-C(6)	119.3(2)	F(1)-C(14)-F(2)	105.3(2)
C(10)-C(5)-C(6)	119.2(2)	F(3)-C(14)-C(13)	109.9(2)
C(7)-C(6)-C(5)	117.3(2)	F(1)-C(14)-C(13)	115.18(15)
C(7)-C(6)-C(1)	123.7(2)	F(2)-C(14)-C(13)	112.16(15)
C(5)-C(6)-C(1)	118.9(2)	N(1)-C(15)-C(21)	112.13(13)

N(1)-C(15)-C(16)	114.52(14)	O(2)-C(19)-O(3)	123.5(2)
C(21)-C(15)-C(16)	108.99(13)	O(2)-C(19)-C(17)	123.0(2)
C(17)-C(16)-C(15)	116.23(14)	O(3)-C(19)-C(17)	113.4(2)
C(18)-C(17)-C(19)	122.1(2)	O(4)-C(21)-O(5)	124.0(2)
C(18)-C(17)-C(16)	122.5(2)	O(4)-C(21)-C(15)	123.1(2)
C(19)-C(17)-C(16)	115.4(2)	O(5)-C(21)-C(15)	112.78(14)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
F(1)	109(1)	80(1)	69(1)	-3(1)	-5(1)	43(1)
F(2)	70(1)	86(1)	87(1)	2(1)	-29(1)	-2(1)
F(3)	99(1)	98(1)	91(1)	56(1)	-4(1)	13(1)
O(1)	64(1)	71(1)	51(1)	19(1)	2(1)	-14(1)
O(2)	80(1)	65(1)	64(1)	-2(1)	3(1)	10(1)
O(3)	79(1)	91(1)	46(1)	-2(1)	-11(1)	5(1)
O(4)	58(1)	115(1)	62(1)	-13(1)	15(1)	12(1)
O(5)	62(1)	79(1)	45(1)	-20(1)	8(1)	-8(1)
N(1)	40(1)	40(1)	33(1)	0(1)	1(1)	-3(1)
C(1)	40(1)	47(1)	39(1)	3(1)	7(1)	-1(1)
C(2)	52(1)	50(1)	53(1)	10(1)	11(1)	4(1)
C(3)	66(1)	73(1)	60(1)	25(1)	9(1)	11(1)
C(4)	58(1)	99(2)	43(1)	21(1)	3(1)	4(1)
C(5)	47(1)	83(1)	39(1)	-1(1)	6(1)	-8(1)
C(6)	42(1)	53(1)	40(1)	-1(1)	6(1)	-4(1)
C(7)	78(1)	53(1)	49(1)	-8(1)	-2(1)	4(1)
C(8)	112(2)	60(1)	72(1)	-19(1)	5(1)	0(1)
C(9)	104(2)	88(2)	61(1)	-30(1)	10(1)	-18(2)
C(10)	74(2)	109(2)	42(1)	-12(1)	5(1)	-21(1)
C(11)	39(1)	40(1)	39(1)	0(1)	4(1)	-4(1)
C(12)	51(1)	48(1)	54(1)	-5(1)	4(1)	-11(1)
C(13)	59(1)	43(1)	38(1)	3(1)	-3(1)	-8(1)
C(14)	75(1)	52(1)	45(1)	6(1)	-7(1)	5(1)
C(15)	40(1)	48(1)	37(1)	3(1)	3(1)	-3(1)
C(16)	45(1)	66(1)	48(1)	7(1)	0(1)	-11(1)
C(17)	42(1)	57(1)	50(1)	-1(1)	-3(1)	-8(1)
C(18)	106(2)	61(1)	80(2)	-14(1)	-7(1)	4(1)
C(19)	40(1)	65(1)	45(1)	-2(1)	-1(1)	-2(1)
C(20)	89(2)	133(2)	62(1)	26(2)	-15(1)	15(2)
C(21)	52(1)	56(1)	44(1)	-1(1)	9(1)	0(1)
C(22)	99(2)	107(2)	50(1)	-30(1)	14(1)	-15(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3).

	x	y	z	U(eq)
H(2)	747(2)	1990(1)	-376(1)	62
H(3)	-38(3)	2257(2)	-1478(1)	79
H(4)	-60(3)	1036(2)	-2220(1)	80
H(7)	2088(3)	-1257(1)	-312(1)	72
H(8)	2089(4)	-2459(2)	-1076(1)	98
H(9)	1245(4)	-2166(2)	-2166(1)	101
H(10)	412(3)	-676(2)	-2484(1)	90
H(11)	2709(2)	-20(1)	280(1)	47
H(12A)	2675(2)	1192(1)	1073(1)	76
H(12B)	1457(2)	1812(1)	612(1)	76
H(12C)	3405(2)	1576(1)	394(1)	76
H(15)	-1403(2)	1021(1)	354(1)	50
H(16A)	-2966(2)	-764(1)	612(1)	64
H(16B)	-4149(2)	149(1)	552(1)	64
H(18A)	-2957(4)	-1172(2)	-1073(1)	99
H(18B)	-2454(4)	-1621(2)	-360(1)	99
H(20A)	-4683(4)	984(2)	-2211(1)	142
H(20B)	-3735(4)	1697(2)	-1725(1)	142
H(20C)	-5704(4)	1390(2)	-1593(1)	142
H(22A)	165(4)	1595(2)	2584(1)	128
H(22B)	-1582(4)	988(2)	2612(1)	128
H(22C)	-1624(4)	2033(2)	2330(1)	128

Crystallographic Data of Aldol Product 2

Table 1. Crystal data and structure refinement.

Crystal growing data	ether and hexane	
Empirical formula	C25 H26 F3 N O7	
Formula weight	509.47	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 9.0772(3) Å	α= 90°.
	b = 14.2140(3) Å	β= 113.786(1)°.
	c = 10.4104(3) Å	γ = 90°.
Volume	1229.09(6) $Å^3$	
Z	2	
Density (calculated)	1.377 Mg/m^3	
Absorption coefficient	0.115 mm^{-1}	
F(000)	532	
Crystal size	0.55 x 0.30 x 0.28 mm^3	
Theta range for data collection	2.14 to 29.44°.	
Index ranges	-12<=h<=5, -18<=k<=19, -12<=l<=14	
Reflections collected	7923	
Independent reflections	5443 [R(int) = 0.0157]	
Completeness to theta = 29.44°	88.9 %	
Absorption correction	SADABS (Sheldrick, 1996)	
Max. and min. transmission	0.9766 and 0.7731	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5443 / 1 / 326	
Goodness-of-fit on F^2	1.029	
Final R indices [I>2sigma(I)]	R1 = 0.0434, wR2 = 0.1047	
R indices (all data)	R1 = 0.0589, wR2 = 0.1136	
Absolute structure parameter	0.3(7)	
Extinction coefficient	0.019(2)	
Largest diff. peak and hole	0.294 and -0.190 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
F(1)	12123(3)	817(2)	-987(2)	120(1)
F(2)	10545(2)	-351(2)	-1318(3)	135(1)
F(3)	12709(2)	-262(1)	497(2)	88(1)
O(1)	4854(2)	2934(2)	1377(2)	85(1)
O(2)	7390(2)	3314(1)	1979(2)	76(1)
O(3)	9358(2)	337(1)	4520(2)	60(1)
O(4)	7632(3)	-334(1)	2561(2)	80(1)
O(5)	9597(2)	1818(1)	3158(1)	49(1)
O(6)	11359(2)	2512(1)	2485(2)	66(1)
O(7)	10838(2)	403(1)	1575(2)	70(1)
N(1)	9599(2)	1442(1)	-164(2)	46(1)
C(1)	4395(4)	3796(3)	548(4)	111(1)
C(2)	6401(3)	2788(2)	2039(2)	51(1)
C(3)	5316(3)	1270(2)	2631(3)	71(1)
C(4)	7510(4)	2204(2)	4467(2)	64(1)
C(5)	6800(3)	1887(2)	2924(2)	48(1)
C(6)	9674(5)	-583(2)	5196(3)	86(1)
C(7)	8323(3)	342(2)	3196(2)	52(1)
C(8)	8073(2)	1335(1)	2558(2)	42(1)
C(9)	7633(2)	1268(2)	962(2)	47(1)
C(10)	8875(2)	1872(2)	717(2)	44(1)
C(11)	10126(3)	2105(2)	2179(2)	46(1)
C(12)	11513(3)	239(2)	-361(3)	73(1)
C(13)	10607(3)	723(2)	419(2)	54(1)
C(14)	9176(3)	1870(2)	-1583(2)	52(1)
C(15)	10213(4)	2731(2)	-1439(3)	82(1)
C(16)	7375(3)	2026(2)	-2306(2)	52(1)
C(17)	6306(3)	1270(2)	-2971(2)	53(1)
C(18)	6821(3)	332(2)	-2988(3)	61(1)
C(19)	5768(4)	-364(2)	-3714(4)	83(1)
C(20)	4152(4)	-168(3)	-4460(4)	94(1)

C(21)	3594(4)	717(3)	-4460(4)	89(1)
C(22)	4641(3)	1452(2)	-3718(3)	67(1)
C(23)	4077(5)	2378(3)	-3727(4)	93(1)
C(24)	5086(5)	3081(3)	-3084(3)	93(1)
C(25)	6750(4)	2906(2)	-2393(3)	71(1)

Table 3. Bond lengths [Å] and angles [°].

F(1)-C(12)	1.302(4)	C(5)-C(8)	1.565(3)
F(2)-C(12)	1.328(3)	C(7)-C(8)	1.537(3)
F(3)-C(12)	1.304(3)	C(8)-C(9)	1.547(3)
O(1)-C(2)	1.307(3)	C(9)-C(10)	1.518(3)
O(1)-C(1)	1.460(4)	C(10)-C(11)	1.523(3)
O(2)-C(2)	1.189(3)	C(12)-C(13)	1.533(3)
O(3)-C(7)	1.318(3)	C(14)-C(15)	1.514(4)
O(3)-C(6)	1.458(3)	C(14)-C(16)	1.515(3)
O(4)-C(7)	1.192(3)	C(16)-C(25)	1.362(3)
O(5)-C(11)	1.352(2)	C(16)-C(17)	1.425(3)
O(5)-C(8)	1.441(2)	C(17)-C(18)	1.415(4)
O(6)-C(11)	1.184(3)	C(17)-C(22)	1.417(3)
O(7)-C(13)	1.223(3)	C(18)-C(19)	1.371(4)
N(1)-C(13)	1.342(3)	C(19)-C(20)	1.383(5)
N(1)-C(10)	1.461(2)	C(20)-C(21)	1.356(5)
N(1)-C(14)	1.498(3)	C(21)-C(22)	1.413(4)
C(2)-C(5)	1.533(3)	C(22)-C(23)	1.411(5)
C(3)-C(5)	1.531(3)	C(23)-C(24)	1.338(5)
C(4)-C(5)	1.538(3)	C(24)-C(25)	1.409(4)

C(2)-O(1)-C(1)	115.8(2)	C(4)-C(5)-C(8)	111.23(19)
C(7)-O(3)-C(6)	115.3(2)	O(4)-C(7)-O(3)	124.7(2)
C(11)-O(5)-C(8)	112.76(14)	O(4)-C(7)-C(8)	123.2(2)
C(13)-N(1)-C(10)	115.56(16)	O(3)-C(7)-C(8)	112.09(18)
C(13)-N(1)-C(14)	127.78(17)	O(5)-C(8)-C(7)	107.55(16)
C(10)-N(1)-C(14)	116.62(17)	O(5)-C(8)-C(9)	106.38(14)
O(2)-C(2)-O(1)	123.1(2)	C(7)-C(8)-C(9)	109.71(17)
O(2)-C(2)-C(5)	123.8(2)	O(5)-C(8)-C(5)	109.23(15)
O(1)-C(2)-C(5)	113.13(19)	C(7)-C(8)-C(5)	110.42(16)
C(3)-C(5)-C(2)	112.48(19)	C(9)-C(8)-C(5)	113.32(17)
C(3)-C(5)-C(4)	109.85(19)	C(10)-C(9)-C(8)	104.92(15)
C(2)-C(5)-C(4)	106.28(18)	N(1)-C(10)-C(9)	114.93(18)
C(3)-C(5)-C(8)	109.99(18)	N(1)-C(10)-C(11)	112.05(16)
C(2)-C(5)-C(8)	106.93(16)	C(9)-C(10)-C(11)	105.08(15)

O(6)-C(11)-O(5)	121.95(18)	C(25)-C(16)-C(14)	120.2(2)
O(6)-C(11)-C(10)	128.13(18)	C(17)-C(16)-C(14)	121.2(2)
O(5)-C(11)-C(10)	109.83(16)	C(18)-C(17)-C(22)	116.7(2)
F(1)-C(12)-F(3)	106.4(2)	C(18)-C(17)-C(16)	123.5(2)
F(1)-C(12)-F(2)	108.0(3)	C(22)-C(17)-C(16)	119.7(2)
F(3)-C(12)-F(2)	106.6(3)	C(19)-C(18)-C(17)	121.7(3)
F(1)-C(12)-C(13)	114.1(3)	C(18)-C(19)-C(20)	120.7(3)
F(3)-C(12)-C(13)	111.0(2)	C(21)-C(20)-C(19)	119.9(3)
F(2)-C(12)-C(13)	110.3(2)	C(20)-C(21)-C(22)	121.1(3)
O(7)-C(13)-N(1)	123.0(2)	C(23)-C(22)-C(21)	121.6(3)
O(7)-C(13)-C(12)	116.0(2)	C(23)-C(22)-C(17)	118.5(3)
N(1)-C(13)-C(12)	121.0(2)	C(21)-C(22)-C(17)	119.9(3)
N(1)-C(14)-C(15)	109.22(19)	C(24)-C(23)-C(22)	121.4(3)
N(1)-C(14)-C(16)	110.20(16)	C(23)-C(24)-C(25)	120.0(3)
C(15)-C(14)-C(16)	115.8(2)	C(16)-C(25)-C(24)	121.9(3)
C(25)-C(16)-C(17)	118.5(2)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
F(1)	121(2)	157(2)	120(2)	54(2)	86(1)	63(2)
F(2)	78(1)	169(2)	128(2)	-89(2)	9(1)	32(1)
F(3)	67(1)	89(1)	97(1)	-1(1)	23(1)	25(1)
O(1)	60(1)	83(1)	101(2)	39(1)	22(1)	3(1)
O(2)	71(1)	59(1)	101(2)	23(1)	37(1)	-5(1)
O(3)	70(1)	54(1)	48(1)	7(1)	16(1)	6(1)
O(4)	107(2)	51(1)	70(1)	-5(1)	22(1)	-23(1)
O(5)	50(1)	61(1)	32(1)	-6(1)	14(1)	-16(1)
O(6)	56(1)	82(1)	57(1)	-8(1)	21(1)	-29(1)
O(7)	83(1)	71(1)	52(1)	12(1)	22(1)	16(1)
N(1)	54(1)	50(1)	38(1)	1(1)	22(1)	4(1)
C(1)	81(2)	105(3)	130(3)	60(2)	25(2)	15(2)
C(2)	58(1)	53(1)	46(1)	-1(1)	26(1)	-3(1)
C(3)	66(2)	67(2)	95(2)	9(2)	46(2)	-11(1)
C(4)	98(2)	56(1)	47(1)	0(1)	40(1)	2(1)
C(5)	57(1)	46(1)	47(1)	0(1)	28(1)	-7(1)
C(6)	108(2)	69(2)	79(2)	25(2)	34(2)	29(2)
C(7)	60(1)	51(1)	48(1)	-3(1)	25(1)	-4(1)
C(8)	47(1)	46(1)	32(1)	-3(1)	15(1)	-11(1)
C(9)	47(1)	56(1)	35(1)	-9(1)	13(1)	-10(1)
C(10)	49(1)	48(1)	35(1)	-2(1)	17(1)	-1(1)
C(11)	51(1)	50(1)	39(1)	-3(1)	19(1)	-5(1)
C(12)	65(2)	90(2)	58(1)	-9(2)	19(1)	18(2)
C(13)	54(1)	58(1)	50(1)	-6(1)	19(1)	2(1)
C(14)	65(1)	59(1)	39(1)	0(1)	27(1)	-4(1)
C(15)	96(2)	74(2)	86(2)	7(2)	46(2)	-21(2)
C(16)	68(1)	57(1)	33(1)	6(1)	23(1)	9(1)
C(17)	58(1)	63(1)	42(1)	13(1)	25(1)	7(1)
C(18)	57(1)	57(1)	71(2)	9(1)	27(1)	0(1)
C(19)	78(2)	66(2)	104(2)	8(2)	34(2)	-11(2)
C(20)	75(2)	88(2)	108(3)	2(2)	24(2)	-28(2)

C(21)	56(2)	107(3)	95(2)	17(2)	20(2)	-2(2)
C(22)	59(1)	81(2)	60(2)	12(1)	25(1)	11(1)
C(23)	80(2)	110(3)	76(2)	17(2)	19(2)	43(2)
C(24)	109(3)	83(2)	70(2)	-2(2)	19(2)	45(2)
C(25)	93(2)	62(2)	49(1)	3(1)	21(1)	19(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3).

	x	y	z	U(eq)
H(1A)	3242	3839	105	166
H(1B)	4825	3788	-157	166
H(1C)	4815	4330	1151	166
H(3A)	5624	716	3206	107
H(3B)	4858	1091	1659	107
H(3C)	4535	1617	2844	107
H(4A)	8440	2590	4644	95
H(4B)	7814	1662	5067	95
H(4C)	6717	2559	4653	95
H(6A)	10437	-519	6151	129
H(6B)	10103	-996	4702	129
H(6C)	8688	-841	5179	129
H(9A)	7690	622	686	56
H(9B)	6555	1506	431	56
H(10)	8353	2459	270	52
H(14)	9463	1411	-2145	63
H(15A)	11328	2562	-970	124
H(15B)	9955	3202	-901	124
H(15C)	10013	2976	-2354	124
H(18)	7902	184	-2495	74
H(19)	6143	-973	-3704	100
H(20)	3450	-642	-4961	113
H(21A)	2505	843	-4957	107
H(23)	2982	2502	-4190	111
H(24)	4688	3685	-3093	111
H(25)	7442	3406	-1984	85