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Highly Diastereo- and Enantioselective Mannich Reaction of Lactones with *N*-Boc-Aldimines Catalyzed by Bifunctional Rosin-Derived Amine Thiourea Catalysts

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1.0 General Methods: All reactions were carried out under an argon atmosphere condition unless otherwise noted and solvents were dried according to established procedures. Reactions were monitored by thin layer chromatography (TLC), column chromatography purifications were carried out using silica gel GF254. Proton nuclear magnetic resonance (^{1}HNMR) spectra were recorded on Brucker 300 MHz spectrometer in $CDCl_3$ unless otherwise noted and carbon nuclear magnetic resonance (^{13}CNMR) spectra were recorded on Brucker 300 MHz spectrometer in $CDCl_3$ using tetramethylsilane (TMS) as internal standard unless otherwise noted. Data are presented as follows: chemical shift, integration, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, cm = complex multiplet) and coupling constant in Hertz (Hz). Infrared (IR) spectra were recorded on a FT-IR spectrometer. HR-MS was measured with an APEX II 47e mass spectrometer. Melting points were measured on an XT-4 melting point apparatus and were uncorrected. The ee values determination was carried out using chiral high-performance liquid chromatography (HPLC) with Daicel Chiracel AD-H and OJ-H column on Waters with a 2996 UV-detector and the dr values determined by 300 Hz ^{1}H NMR and HPLC.

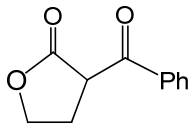
Abbreviations used: EtOAc- ethyl acetate, THF- tetrahydrofuran, *i*PrOH- isopropanol, CH_2Cl_2- dichloromethane, LDA- Lithium diisopropylamide.

Materials: Dehydroabietic amine, 3,5-bis(trifluoromethyl)phenyl isothiocyanate, *β*-ketoesters, LDA and α-acetyl-*γ*-tutyrolactone were commercially available from Acros and Aldrich. Thiourea catalysts were synthesized according to the literature procedures.[1, 2]

2. General procedure for synthesis of lactones 4b-4d.

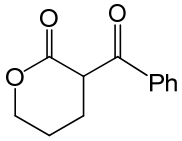
To a solution of LDA (25.0 mmol) in THF (30 mL) was added *γ*-butyrolactone (0.77 mL, 10.0 mmol) at -78 °C under an argon atmosphere and the mixture was stirred for 1 h at the same temperature. Then, benzoyl chloride (1.28 mL, 11.0 mmol) was added over a period of 30 min at -78 °C and stirring was continued for 15 min. The reaction mixture was diluted with 1 N HCl and extracted with ethyl acetate. The combined organic extracts were washed with brine and dried over Na_2SO_4. Evaporation of solvents and purification of the residual crude products by column chromatography on silica gel (hexane/ ethyl acetate=6:1 as eluent) gave α-benzoyl-*γ*-butyrolactone **4b** in 80% yield: 1.52 g.

4b: 3-benzoyldihydrofuran-2(3H)-one



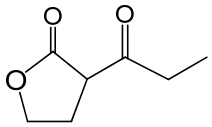
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.07-8.10(m, 2 H), 7.49-7.66(m, 3 H), 4.39-4.61(m, 3 H), 2.81-2.92(m, 1 H), 2.46-2.58(m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 193.1, 172.9, 135.2, 134.1, 129.5, 128.8, 67.8, 48.0, 26.0. ESI-MS**:** *m*/*z* 191 [M^+].

4c: 3-benzoyltetrahydro-2H-pyran-2-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.53-7.56(m, 2 H), 7.43-7.46(m, 3 H), 4.37-4.41(m, 2 H), 2.54-2.59(m, 2 H), 1.85-1.88(m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 173.2, 134.3, 130.2, 128.2, 94.3, 69.5, 24.4, 23.1. ESI-MS**:** *m*/*z* 205 [M^+].

4d: 3-propionyldihydrofuran-2(3H)-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 4.29-4.44(m, 2 H), 3.67-3.73(dd, *J* = 6.6 Hz, 9.3 Hz, 1 H), 2.99-3.13(m, 1 H), 2.73-2.85(m, 1 H), 2.38-2.66(m, 1 H), 2.25-2.35(m, 1 H), 1.11(t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 203.0, 172.9, 67.6, 52.0, 35.6, 24.0, 7.4. ESI-MS**:** *m*/*z* 143 [M^+].

3.0 General procedure for the catalytic asymmetric Mannich reaction.

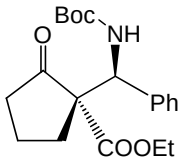
Method A: To a stirred solution of **L2** (0.015 mmol, 15 mol %), *N*-Boc-aldimine (0.1 mmol) in dry toluene (1.0 mL) under an argon atmosphere, ethyl 2-oxocyclopentanecarboxylate (0.12 mmol) was added over a period of 15 min at -60 °C. The solution was stirred at -60 °C for 12 h. After the reaction was completed (monitored by TLC), the resulting mixture was concentrated under reduced pressure and the residue was purified through column chromatography on silica gel (eluent, ethyl acetate / hexane 1:8) to give the optical pure product. The enantiomeric purity of the product was determined by using HPLC and the dr values determined by ^{1}H NMR and HPLC.

Method B: To a stirred solution of **L2** (0.015 mmol, 15 mol %), *N*-Boc-aldimine (0.1 mmol) in dry toluene (1.0 mL) under an argon atmosphere, the lactone (0.12 mmol) was added over a period

of 15 min at -60 °C. The solution was stirred at -60 °C for 12 h. After the reaction was completed (monitored by TLC), the resulting mixture was concentrated under reduced pressure and the residue was purified through column chromatography on silica gel (eluent, ethyl acetate / hexane 1:10), and affording the products were dissolved in diethyl ether. After filteration and the solvent was removed at reduced pressure to give the pure products. The enantiomeric purity of the product was determined by using HPLC and the dr values determined by ^{1}H NMR and HPLC.

4.0 Characterization data

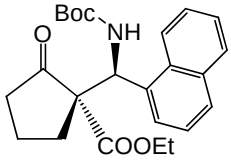
3a: (*S*)-ethyl 1-((*S*)-(*tert*-butoxycarbonylamino)(phenyl)methyl)-2-oxocyclopentane carboxylate



White solid, mp 109 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.25-7.32(m, 5 H), 5.92(br, 1 H), 5.21(d, *J* = 9.6 Hz, 1 H), 4.11-4.20(m, 2 H), 2.48-2.57(m, 1 H), 2.32-2.40(m, 2 H), 1.83-2.05(m, 3 H), 1.39(s, 9 H), 1.19(t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 211.2, 155.2, 138.5, 128.3, 128.2, 127.7, 79.7, 64.9, 61.8, 55.8, 37.6, 30.6, 28.3, 18.9, 13.9. IR: 3363, 2977, 2932, 1751, 1721, 1700, 1497, 1366, 1230, 1168, 1024, 706 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{20}H_{27}NO_5$ +NH_4^+: 379.2227; found: 379.2231, 1.1ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 2/98, 0.8 mL/min, 212 nm.) Retention time: t_{major} =31.01 min, t_{minor} =77.09 min, ee=99 %.

3b: (*S*)-ethyl 1-((*S*)-(*tert*-butoxycarbonylamino)(naphthalen-1-yl)methyl)-2-oxocyclopentane carboxylate

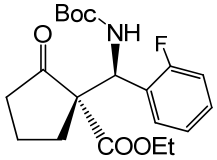


White solid, mp 136 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.28(d, *J* = 8.4 Hz, 1 H), 7.85(d, *J* = 7.8 Hz, 1 H), 7.79(d, *J* = 10.5 Hz, 1 H), 7.63(d, *J* = 7.2 Hz, 1 H), 7.37-7.58(m, 3 H), 6.02(d, *J* = 10.2 Hz, 1 H), 5.85(d, *J* = 9.9 Hz, 1 H), 4.03-4.11(dd, *J* = 7.2 Hz, 14.1 Hz, 2 H), 2.34-2.79(m, 3 H), 1.91-2.21(m, 3 H), 1.41(s, 9 H), 1.03(t, *J* = 6.9 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 211.9, 155.6, 135.0, 133.9, 128.8, 128.6, 126.6, 125.7, 125.6, 125.0, 123.6, 79.8, 64.7, 61.6, 50.5, 37.8,

33.1, 28.3, 19.1, 13.7. IR: 3421, 2976, 2932, 1750, 1714, 1567, 1493, 1368, 1230, 1166, 1025, 782 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{24}H_{29}NO_5$ $+NH_4^+$: 429.2384; found: 429.2391, 1.6ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 2/98, 1.0 mL/min, 222 nm.) Retention time: t_{major} =14.87 min, t_{minor} =27.94 min, ee=97 %.

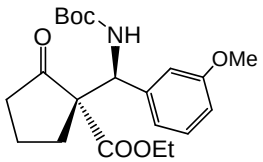
3c: (*S*)-ethyl 1-((*R*)-(*tert*-butoxycarbonylamino)(2-fluorophenyl)methyl)-2-oxocyclopentane carboxylate



Colorless needs, mp 120-121 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.49(dt, *J* = 1.8 Hz, 7.8 Hz, 1 H), 7.22-7.29(m, 1 H), 6.99-7.12(m, 2 H), 6.22(d, *J* = 9.6 Hz, 1 H), 5.56(d, *J* = 9.9 Hz, 1 H), 4.08-4.19(m, 2 H), 2.29-2.55(m, 3 H), 1.96-2.12(m, 3 H), 1.39(s, 9 H), 1.19(t, *J* = 6.9 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 210.8, 169.4, 162.1, 158.8, 155.2, 130.2, 130.1, 129.5, 129.4, 124.4, 124.3, 115.6, 115.3, 79.8, 64.8, 61.8, 49.2, 37.2, 30.6, 28.2, 18.8, 13.9. IR: 3441, 2977, 2933, 1753, 1717, 1493, 1459, 1367, 1231, 1168, 1023, 762 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{20}H_{26}FNO_5$ $+NH_4^+$: 397.2133; found: 397.2142, 2.3ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 2/98, 1.0 mL/min, 210 nm.) Retention time: t_{major} =15.24 min, t_{minor} =19.73 min, ee=92 %.

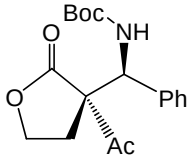
3d: (*S*)-ethyl 1-((*S*)-(*tert*-butoxycarbonylamino)(3-methoxyphenyl)methyl)-2-oxocyclopentanecarboxylate



Colorless solid, mp 128-129 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.24(t, *J* = 8.1 Hz, 1 H), 6.88(m, 2 H), 6.78-6.81(m, 1 H), 5.85(br, 1 H), 5.22(d, *J* = 9.6 Hz, 1 H), 4.09-4.17(m, 2 H), 3.78(s, 3 H), 2.47-2.56(m, 1 H), 2.29-2.32(m, 2 H), 1.91-2.05(m, 3 H), 1.39(s, 9 H), 1.20(t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 211.1, 159.5, 155.2, 129.3, 120.5, 113.9, 113.2, 79.8, 64.9, 61.9, 55.2, 37.7, 28.3, 18.9, 13.9. IR: 3363, 2975, 2929, 1751, 1720, 1700, 1494, 1385, 1231, 1167, 1044, 782 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{21}H_{29}NO_6$ $+NH_4^+$: 409.2333; found: 409.2325, 2.0ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 2/98, 1.0 mL/min, 215 nm.) Retention time: t_{major} =39.37 min, t_{minor} =101.46 min, ee=96 %.

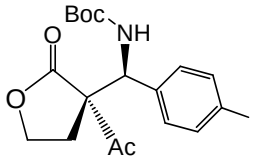
5a: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(phenyl)methylcarbamate



Colorless needs, mp 116-117 °C. ^{1}H NMR (300 MHz): δ 7.34(m, 5 H), 5.79(d, *J*=8.7 Hz, 1 H), 5.01(d, *J*=9.9 Hz, 1 H), 3.99(dd, *J*=7.8 Hz, 16.5 Hz, 1 H), 3.59(m, 1 H), 2.90 (m, 1 H), 2.49(s, 3 H), 2.03-2.13(m, 1 H), 1.39(s, 9 H); ^{13}C NMR (75 MHz): δ 173.2, 154.6, 128.8, 128.4, 127.2, 80.8, 66.9, 66.1, 56.3, 28.2, 28.1, 25.4. IR: 3341, 2977, 2926, 1761, 1716, 1522, 1456, 1368, 1246, 1165, 1025, 704 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{18}H_{23}NO_5$ +NH_4^+: 351.1914; found: 351.1918, 1.1ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 20/80, 1.0 mL/min, 215 nm.) Retention time: t_{major} =6.65 min, t_{minor} =15.82 min, ee=91 %.

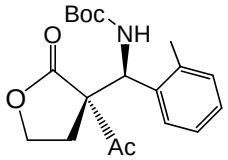
5b: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(p-tolyl)methylcarbamate



Colorless needs, mp 149 °C. ^{1}H NMR (300 MHz): δ 7.14-7.27(m, 4 H), 5.77(d, *J*=9.3 Hz, 1 H), 4.95(d, *J*=6.9 Hz, 1 H), 3.96(dd, *J*=8.1 Hz, 16.2 Hz, 1 H), 3.60-3.62(m, 1 H), 2.85-2.93(m, 1 H), 2.48(s, 3 H), 2.33(s, 3 H), 2.04-2.14(m, 1 H), 1.39(s, 9 H); ^{13}C NMR (75 MHz): δ 173.2, 154.5, 138.2, 144.0, 129.5, 127.1, 80.7, 66.9, 66.1, 56.2, 28.2, 25.4, 24.3, 20.9. IR: 3342, 2979, 2926, 1761, 1715, 1515, 1455, 1368, 1246, 1164, 1025, 733 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{19}H_{25}NO_5$ +NH_4^+: 365.2071; found: 365.2080, 2.5ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 10/90, 0.8 mL/min, 215 nm.) Retention time: t_{major} =16.05 min, t_{minor} =24.60 min, ee=99 %.

5c: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(o-tolyl)methylcarbamate

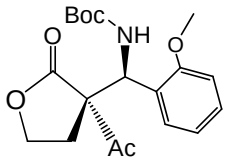


Colorless needs, mp 126. ^{1}H NMR (300 MHz): δ 7.56(s, 1 H), 7.19-7.26(m, 3 H), 5.79(d, *J*=9.9

Hz, 1 H), 5.06(d, *J*=9.0 Hz, 1 H), 4.01-4.09(m, 2 H), 2.91-2.95(m, 1 H), 2.55-2.62(m, 1 H), 2.49(s, 3 H), 2.23(s, 3 H), 1.38(s, 9 H); ^{13}C NMR (75 MHz): δ 173.8, 136.9, 136.1, 131.4, 128.4, 127.5, 126.3, 80.6, 66.9, 65.9, 51.9, 29.7, 28.2, 26.3, 20.0. IR: 3402, 2976, 2926, 1759, 1714, 1505, 1458, 1368, 1244, 1164, 1029, 754 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{19}H_{25}NO_5$+NH_4^+: 365.2071; found: 365.2076, 1.4ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 10/90, 1.0 mL/min, 212 nm.) Retention time: t_{major} =11.24 min, t_{minor} =23.72 min, ee=93 %.

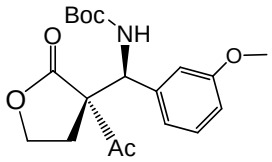
5d: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(2-methoxyphenyl)methylcarba mate



Colorless solid, mp 180-181 °C. ^{1}H NMR (300 MHz): δ 7.30-7.40(m, 2 H), 6.96(dd, *J*=7.5 Hz, 15.9 Hz, 2 H), 5.89-5.96(m, 2 H), 3.94-3.97(m, 1 H), 3.87(s, 3 H), 3.82-3.84(m, 1 H), 2.86-2.94(m, 1 H), 2.45(s, 3 H), 2.14-2.34(m, 1 H), 1.39(s, 9 H); ^{13}C NMR (75 MHz): δ 173.5, 157.3, 135.9, 131.4, 129.8, 124.4, 121.4, 111.4, 80.2, 67.2, 66.3, 55.9, 55.4, 28.3, 26.1, 25.9. IR: 3354, 2979, 2927, 2855, 1760, 1713, 1495, 1370, 1246, 1164, 1028, 735 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{19}H_{25}NO_6$+NH_4^+: 381.2020; found: 381.2012, 2.1ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 10/90, 1.0 mL/min, 224 nm.) Retention time: t_{major} =16.10 min, t_{minor} =24.84 min, ee=88 %.

5e: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(3-methoxyphenyl)methylcarba mate

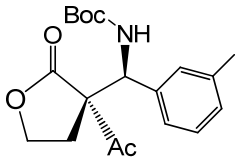


Colorless needs, mp 159 °C. ^{1}H NMR (300 MHz): δ 7.24-7.29(m, 1 H), 6.84-6.89(m, 3 H), 5.80(d, *J*=9.6 Hz, 1 H), 4.97(d, *J*=6.9 Hz, 1 H), 4.03(dd, *J*=8.1 Hz, 16.2 Hz, 1 H), 3.79(s, 3 H), 3.64-3.67(m, 1 H), 2.88(m, 1 H), 2.49(s, 3 H), 2.04-2.13(m, 1 H), 1.39(s, 9H); ^{13}C NMR (75 MHz): δ 200.8, 173.2, 159.9, 154.6, 138.6, 129.9, 119.2, 113.9, 112.9, 80.8, 66.9, 66.2, 56.2, 55.3, 28.2, 25.4, 24.3. IR: 3346, 2978, 2931, 1761, 1716, 1518, 1493, 1368, 1248, 1164, 1026, 782 cm^{-1}.

HRMS-ESI (*m/z*): calcd for $C_{19}H_{25}NO_6$+NH_4^+: 381.2020; found: 381.2025, 1.3ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 10/90, 0.8 mL/min, 215 nm.) Retention time: t_{major} =24.18 min, ee=99 %.

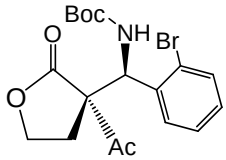
5f: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(m-tolyl)methylcarbamate



Colorless solid, mp 139-140 °C. ^{1}H NMR (300 MHz): δ 7.11-7.27(m, 4 H), 5.79(d, *J*=9.6 Hz, 1 H), 4.99(br, 1 H), 3.99(dd, *J*=8.1 Hz, 16.2 Hz, 1 H), 3.60-3.61(m, 1 H), 2.89(m, 1 H), 2.49(s, 3 H), 2.34(s, 3 H), 2.07-2.11(m, 1 H), 1.39(s, 9 H); ^{13}C NMR (75 MHz): δ 173.2, 154.6, 138.6, 129.2, 128.7, 128.0, 124.1, 80.7, 66.9, 66.1, 56.3, 28.2, 25.4, 24.2, 21.5. IR: 3350, 2923, 2853, 1757, 1714, 1518, 1456, 1369, 1245, 1163, 1027, 732 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{19}H_{25}NO_5$ +NH_4^+: 365.2071; found: 365.2065, 1.6ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 10/90, 1.0 mL/min, 224 nm.) Retention time: t_{major} =9.74 min, t_{minor} =27.86 min, ee=82 %.

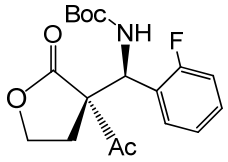
5g: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(2-bromophenyl)methylcarbamate



Colorless solid, mp 169 °C. ^{1}H NMR (300 MHz): δ 7.57-7.60(d, *J*=8.1 Hz, 1 H),7.43-7.46(d, *J*=7.8 Hz, 1H), 7.32-7.37(t, *J*=7.2 Hz, 1H), 7.17-7.22(m, 1H), 7.03(d, *J*=9.9 Hz, 1 H), 6.08(d, *J*=9.9 Hz, 1 H), 3.95-4.02(m, 2 H), 2.49(s, 3 H), 2.39-2.44(m, 1 H), 2.13-2.26(m, 1 H), 1.38(s, 9 H); ^{13}C NMR (75 MHz): δ 203.5, 175.6, 136.9, 132.9, 130.1, 129.0, 128.8, 125.5, 80.4, 65.7, 65.1, 54.2, 29.7, 28.2, 25.3. IR: 3409, 2978, 2926, 1766, 1712, 1590, 1494, 1366, 1221, 1167, 1029, 758 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{18}H_{22}BrNO_5$+NH_4^+: 429.1020; found: 429.1028, 1.9ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 10/90, 0.8 mL/min, 215 nm.) Retention time: t_{minor} =15.06 min, t_{major} =17.78 min, ee=98 %.

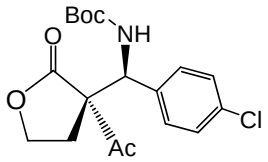
5h: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(2-fluorophenyl)methylcarbamate



Colorless solid, mp 127-128 °C. ^{1}H NMR (300 MHz): δ 7.47-7.49(m, 1 H), 7.31-7.34(m, 1 H), 7.04-7.17(m, 2 H), 5.95(d, *J*=10.5 Hz, 1 H), 5.35(d, *J*=8.4 Hz, 1 H), 3.96-4.03(m, 2 H), 2.95-3.02(m, 1 H), 2.44(s, 3 H), 2.19-2.28(m, 1 H), 1.39(s, 9 H); ^{13}C NMR (75 MHz): δ 172.9, 159.3, 154.8, 131.7, 130.5, 130.4, 124.9, 123.8, 116.5, 116.1, 80.7, 67.2, 66.5, 54.6, 28.2, 28.1, 25.7, 25.5. IR: 3365, 2979, 2929, 1767, 1716, 1493, 1457, 1368, 1246, 1164, 1023, 763 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{18}H_{22}FNO_5$ +NH_4^+: 369.1820; found: 369.1823, 0.8ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 10/90, 0.8 mL/min, 215 nm.) Retention time: t_{major} =12.68 min, t_{minor} =23.46 min, ee=81 %.

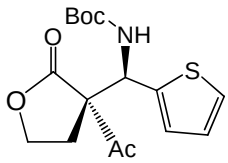
5i: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(4-chlorophenyl)methylcarbamate



White solid, mp 144-145 °C. ^{1}H NMR (300 MHz): δ 7.27-7.36(m, 4 H), 5.77(d, *J*=8.1 Hz, 1 H), 4.94(br, 1 H), 4.03(dd, *J*=8.4 Hz, 16.8 Hz, 1 H), 3.74-3.78(m, 1 H), 2.86-2.92(m, 1 H), 2.46(s, 3 H), 1.99-2.09(m, 1 H), 1.39(s, 9 H); ^{13}C NMR (75 MHz): δ 173.1, 154.6, 135.7, 134.5, 129.1, 128.8, 81.2, 66.9, 66.4, 55.9, 28.3, 25.5, 24.5. IR: 3340, 2980, 2928, 1761, 1716, 1519, 1494, 1369, 1246, 1164, 1019, 733 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{18}H_{22}ClNO_5$ +NH_4^+: 385.1525; found: 385.1518, 1.8ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel OJ-H, *i*-PrOH/ Hexane = 10/90, 1.0 mL/min, 224 nm.) Retention time: t_{minor} =11.24 min, t_{major} =15.46 min, ee=75 %.

5j: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(thiophen-2-yl)methylcarbamate

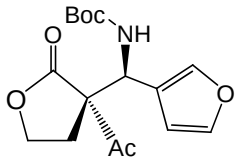


Colorless solid, mp 174-175 °C. ^{1}H NMR (300 MHz): δ 7.17-7.19(m, 1 H), 6.90-6.97(m, 2 H), 6.01(d, *J*=9.3 Hz, 1 H), 4.82(d, *J*=9.3 Hz, 1 H), 3.86-4.03(m, 2 H), 2.79-2.82(m, 1 H), 2.39(s, 3 H), 2.08-2.18(m, 1 H), 1.35(s, 9 H); ^{13}C NMR (75 MHz): δ 173.0, 154.5, 127.6, 126.8, 126.2, 125.3, 81.1, 67.5, 66.4, 53.1, 29.7, 28.2, 25.4. IR: 3306, 2922, 2852, 1749, 1715, 1682, 1526, 1367, 1248,

1162, 1024, 732 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{16}H_{21}NO_5S+NH_4^+$: 357.1479; found: 357.1471, 2.2ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel OJ-H, *i*-PrOH/ Hexane = 10/90, 1.0 mL/min, 236 nm.) Retention time: t_{major} =13.39 min, t_{minor} =14.72 min, ee=93 %.

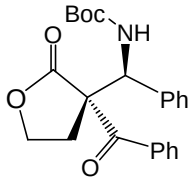
5k: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(furan-3-yl)methylcarbamate



White solid, mp 130 °C. ^{1}H NMR (300 MHz): δ 7.39(d, *J*=1.8 Hz, 2 H), 6.35(s, 1 H), 5.75(d, *J*=9.3 Hz, 1 H), 4.69(br, 1 H), 4.08(dd, *J*=5.4 Hz, 9.3 Hz, 2 H), 2.81-2.89(m, 1 H), 2.43(s, 3 H), 2.11-2.21 (m, 1 H), 1.42(s, 9 H); ^{13}C NMR (75 MHz): δ 173.4, 154.7, 143.9, 140.6, 109.4, 80.9, 66.9, 66.4, 49.9, 28.2, 25.6, 25.0. IR: 3412, 3005, 2924, 2854, 1745, 1714, 1524, 1423, 1363, 1223, 1165, 531 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{16}H_{21}NO_6+NH_4^+$: 341.1707; found: 341.1713, 1.8ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel OJ-H, *i*-PrOH/ Hexane = 10/90, 1.0 mL/min, 212 nm.) Retention time: t_{minor} =14.24 min, t_{major} =20.24 min, ee=87 %.

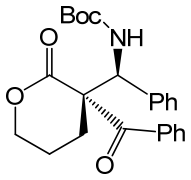
6a: *tert*-butyl (*S*)-((*R*)-3-benzoyl-2-oxotetrahydrofuran-3-yl)(phenyl)methylcarbamate



White solid, mp 152 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.86(d, *J* = 7.2 Hz, 2 H), 7.48-7.62(m, 3 H), 7.35-7.37(m, 5 H), 6.65(d, *J* = 9.0 Hz, 1 H), 5.64(d, *J* = 9.3 Hz, 1 H), 4.14(dd, *J* = 8.4 Hz, 15.9 Hz, 1 H), 3.51-3.58(m, 1 H), 2.76-2.85(m, 1 H), 2.49-2.58(m, 1 H), 1.29(s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 196.9, 176.1, 154.3, 137.2, 134.9, 133.2, 128.9, 128.8, 128.6, 128.3, 79.7, 65.6, 63.1, 57.3, 31.0, 28.1. IR: 3419, 2978, 2925, 1764, 1715, 1674, 1496, 1369, 1246, 1169, 1031, 701 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{23}H_{25}NO_5$ +NH_4^+: 413.2071; found: 413.2078, 1.7ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 20/80, 1.0 mL/min, 247 nm.) Retention time: t_{minor} =14.37 min, t_{major} =21.82 min, ee=57 %.

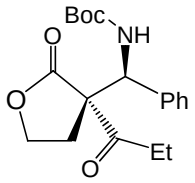
7a: *tert*-butyl (*S*)-((*R*)-3-benzoyl-2-oxotetrahydro-2H-pyran-3-yl)(phenyl)methylcarbamate



White solid, mp 146-147 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.74(d, *J* = 7.5 Hz, 2 H), 7.19-7.51(m, 8 H), 6.84(d, *J* = 9.9 Hz, 1 H), 5.28(d, *J* = 9.6 Hz, 1 H), 4.15-4.22(m, 1 H), 3.85-3.93(m, 1 H), 2.42-2.49(m, 1 H), 1.92-2.01(m, 1 H), 1.64-1.74(m, 1 H), 1.36-1.42(m, 1 H), 1.50(s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 198.6, 170.0, 153.3, 137.1, 135.7, 131.5, 127.9, 127.6, 127.2, 126.9, 78.4, 68.2, 63.4, 59.1, 28.3, 27.2, 19.0. IR: 3422, 2963, 2927, 2855，2254, 1712, 1487, 1366, 1233, 1166, 910, 733 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{24}H_{27}NO_5$ $+H^+$: 410.1962; found: 410.1968, 1.5ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 15/85, 1.0 mL/min, 245 nm.) Retention time: t_{minor} =14.78 min, t_{major} =27.84 min, ee=30 %.

8a: *tert*-butyl (*S*)-((*R*)-2-oxo-3-propionyltetrahydrofuran-3-yl)(phenyl)methylcarbamate



Colorless solid, mp 131 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.57(m, 5 H), 6.64(d, *J* = 9.3 Hz, 1 H), 5.31(d, *J* = 9.6 Hz, 1 H), 4.02(dd, *J* = 8.4 Hz, 16.8 Hz, 1 H), 3.67-3.68(m, 1 H), 2.65-2.88(m, 2 H), 2.32-2.40(m, 1 H), 2.16-2.26(m, 1 H), 1.29(s, 9 H), 1.12(t, *J* = 6.9 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 206.1, 176.1, 154.7, 137.2, 128.9, 128.6, 128.1, 80.1, 65.6, 64.1, 56.8, 31.9, 30.2, 28.2, 7.7. IR: 3425, 2986, 2915, 1773, 1695, 1500, 1457, 1371, 1216, 1168, 1026, 710 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{19}H_{25}NO_5$ $+NH_4^+$: 365.2056; found: 365.2064, 1.9ppm.

Major diastereomer: ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/ Hexane = 20/80, 1.0 mL/min, 212 nm.) Retention time: t_{major} =6.11 min, t_{minor} =11.0 min, ee=90 %.

5.0 References.

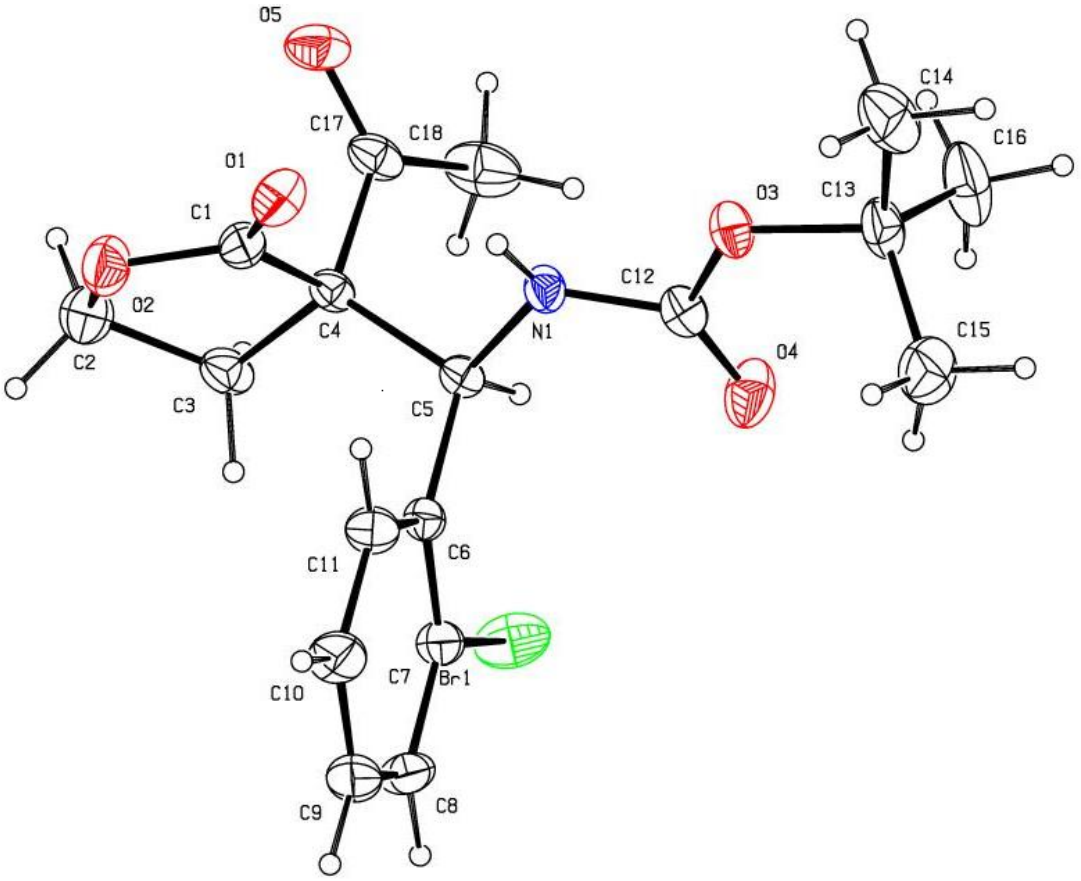
(1). T. Okino, Y. Hoashi, T. Furukawa, X. N. Xu and Y. Takemoto, *J. Am. Chem. Soc*. 2005, **127**, 119.

(2). (*a*) X. X. Jiang, Y. F. Zhang, A. S. C. Chan and R. Wang, *Org. Lett*. 2009, **11**, 153; (*b*) X. X. Jiang, Y. F. Zhang, X. Liu, G. Zhang, L. H. Lai, L. P. Wu, J. N. Zhang and R. Wang, *J. Org. Chem*. 2009, **74**, 5562.

6.0 X-Ray structure of 5g

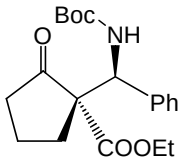
The X-ray crystal structure of 5g: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl) (2-bromophenyl)methylcarbamate

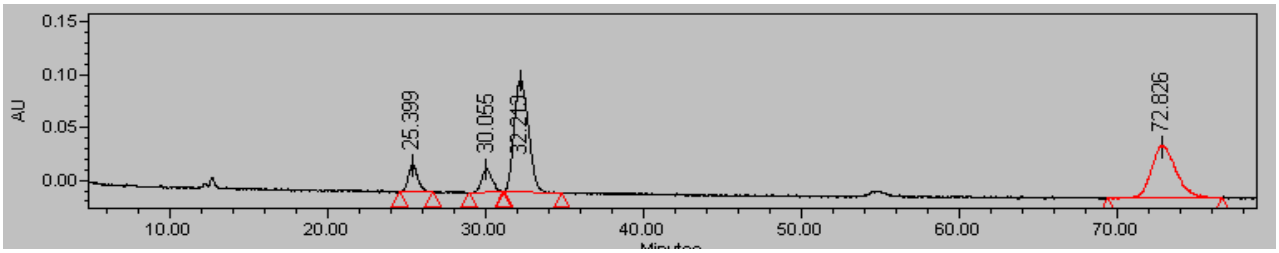
(The CCDC deposition number : CCDC 760713.)



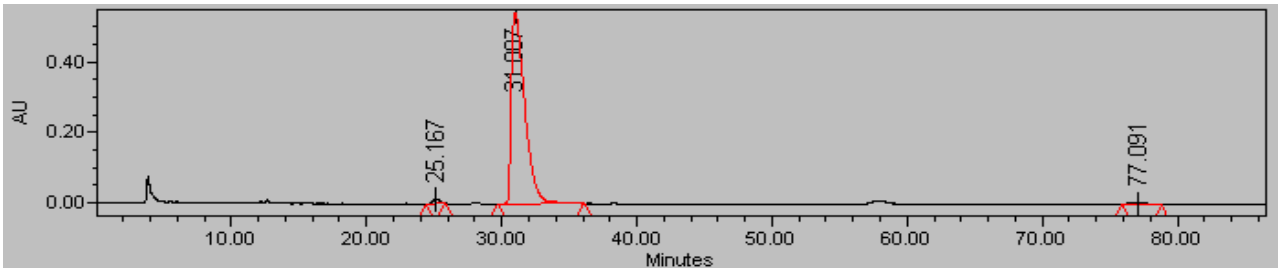
7.0 Copies of HPLC spectra of racemic /chiral products.

3a: (*S*)-ethyl 1-((*S*)-(*tert*-butoxycarbonylamino)(phenyl)methyl)-2-oxocyclopentane carboxylate



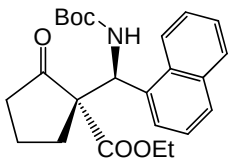


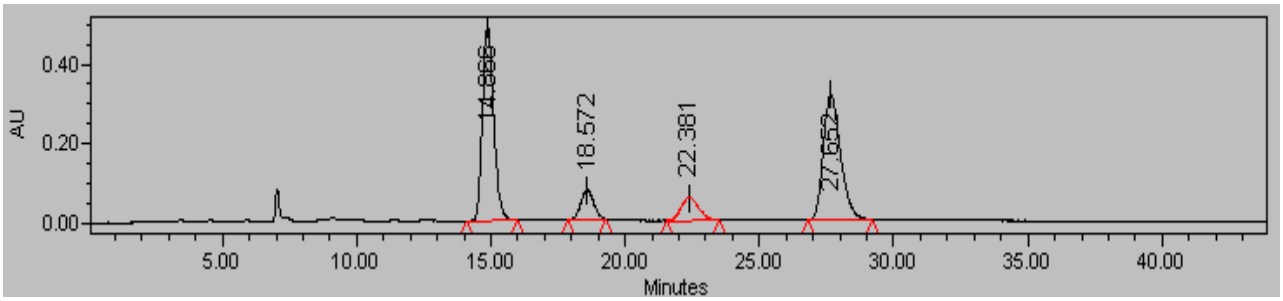
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	25.399	1061303	7.48	25980	bb	Unknown
2	30.055	1014769	7.15	22761	bb	Unknown
3	32.213	6750276	47.58	105534	bb	Unknown
4	72.826	5361809	37.79	49218	bb	Unknown



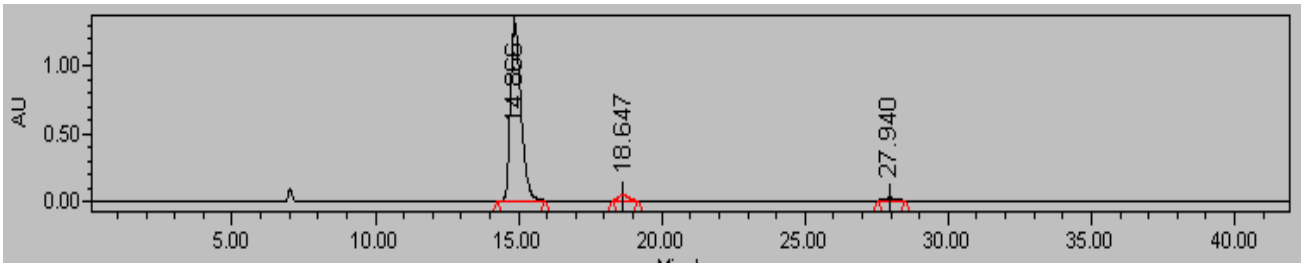
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	25.167	431142	1.13	13739	bb	Unknown
2	31.007	37227751	97.89	540569	bb	Unknown
3	77.091	372937	0.98	4206	bb	Unknown

3b: (*S*)-ethyl 1-((*S*)-(*tert*-butoxycarbonylamino)(naphthalen-1-yl)methyl)-2-oxocyclopentane carboxylate



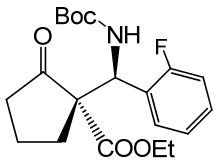


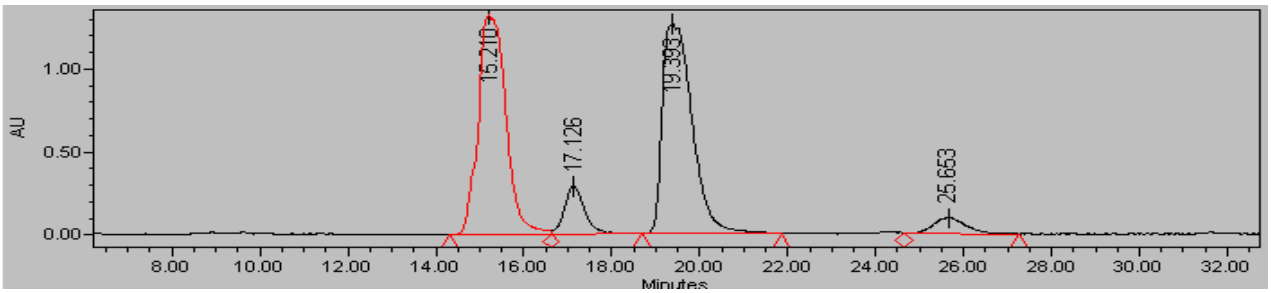
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	14.866	14109290	42.17	493075	bb	Unknown
2	18.572	2581844	7.72	75899	bb	Unknown
3	22.381	2616502	7.82	57393	bb	Unknown
4	27.652	14149777	42.29	315978	bb	Unknown



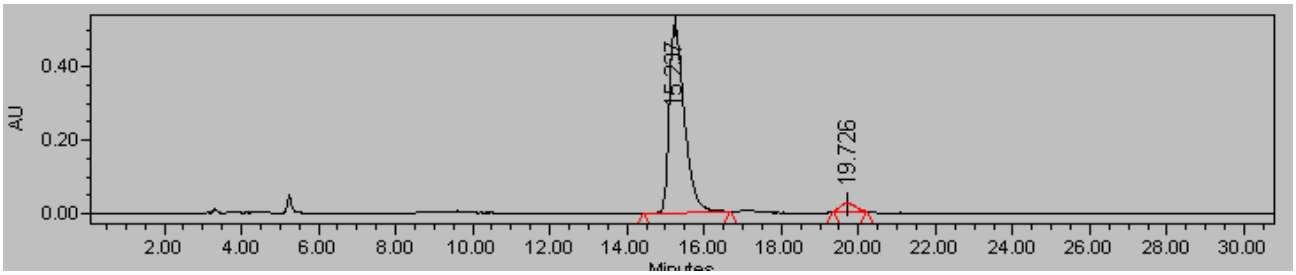
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	14.866	33703631	95.26	1315317	bb	Unknown
2	18.647	1108305	3.13	41573	bb	Unknown
3	27.940	567208	1.60	16801	bb	Unknown

3c: (*S*)-ethyl 1-((*R*)-(*tert*-butoxycarbonylamino)(2-fluorophenyl)methyl)-2-oxocyclopentane carboxylate



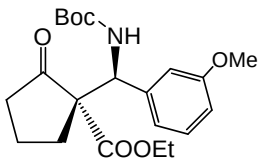


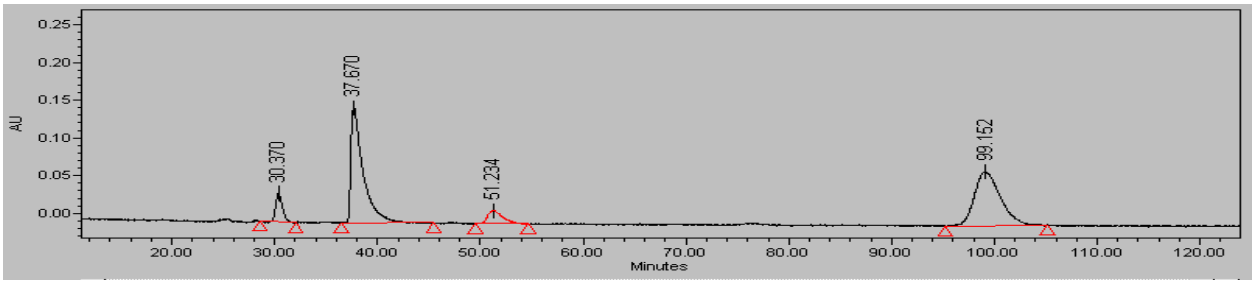
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	15.210	57570022	43.77	1321898	bb	Unknown
2	17.126	9296493	7.07	291556	bb	Unknown
3	19.393	59460051	45.21	1260556	bb	Unknown
4	25.653	5191613	3.95	97984	bb	Unknown



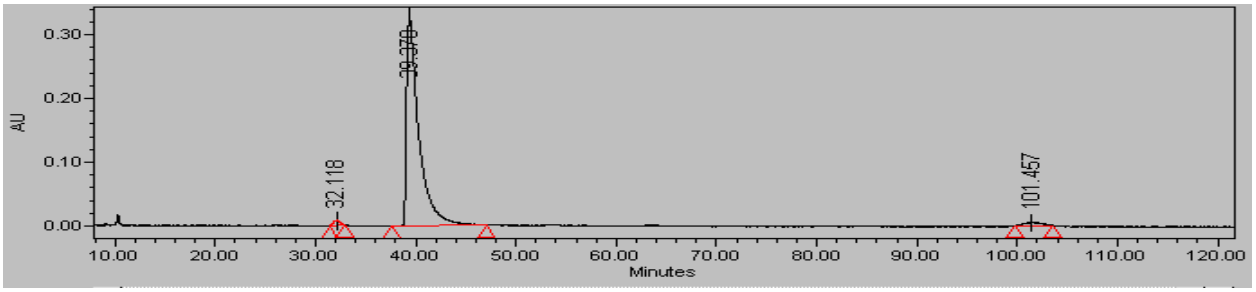
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	15.237	13924307	95.83	513347	bb	Unknown
2	19.726	605354	4.17	22257	bb	Unknown

3d: (*S*)-ethyl 1-((*S*)-(*tert*-butoxycarbonylamino)(3-methoxyphenyl)methyl)-2-oxocyclopentanecarboxylate



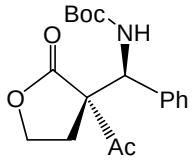


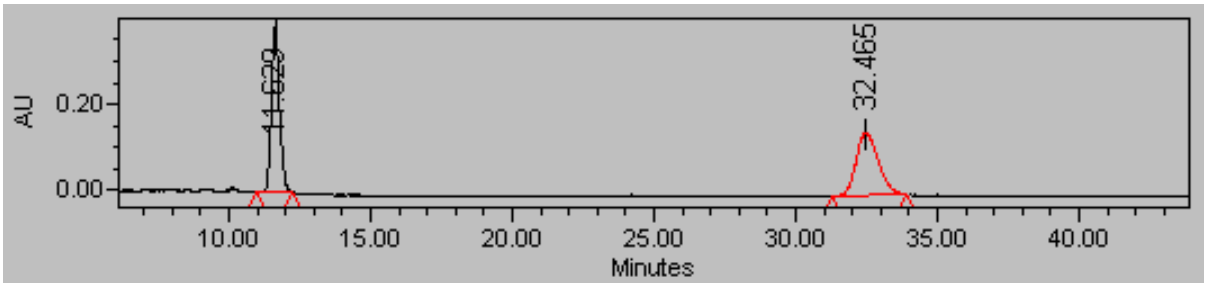
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	30.370	1788198	6.23	37998	bb	Unknown
2	37.670	12627795	43.98	153125	bb	Unknown
3	51.234	1672586	5.82	17154	bb	Unknown
4	99.152	12626779	43.97	72318	bb	Unknown



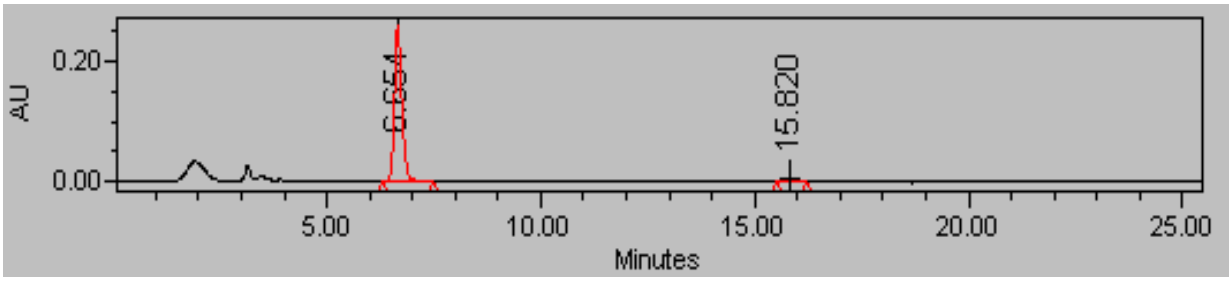
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	32.118	363551	1.26	8009	bb	Unknown
2	39.370	27864087	96.51	326258	bb	Unknown
3	101.457	643995	2.23	5425	bb	Unknown

5a: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(phenyl)methylcarbamate



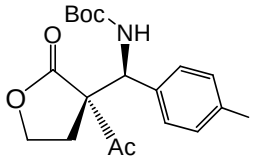


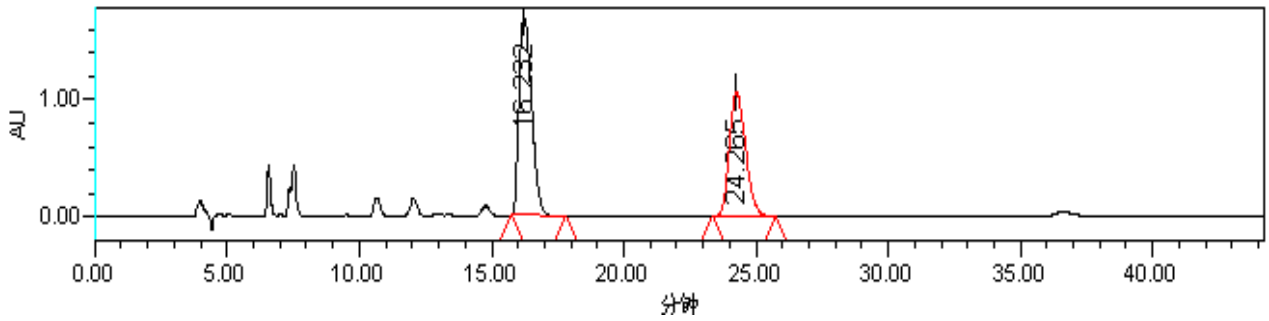
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	11.629	7714918	49.05	383352	bb	Unknown
2	32.465	8013356	50.95	145952	bb	Unknown



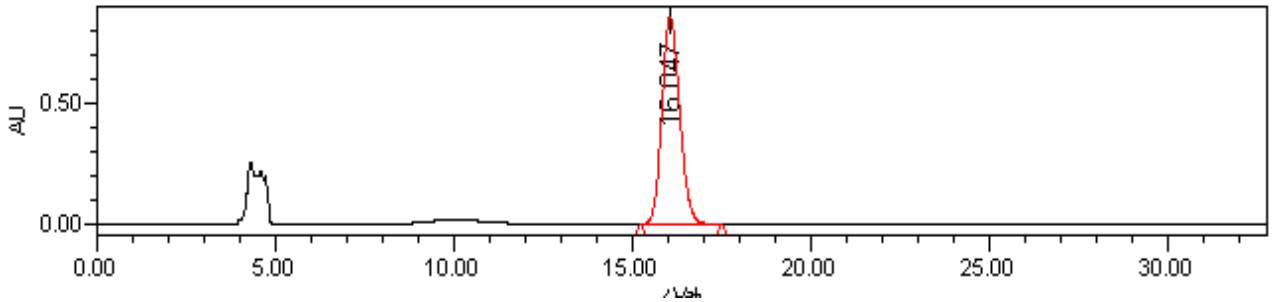
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	6.654	3104104	95.51	257631	bb	Unknown
2	15.820	146033	4.49	6332	bb	Unknown

5b: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(p-tolyl)methylcarbamate



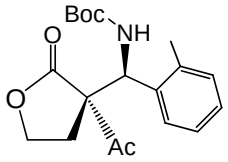


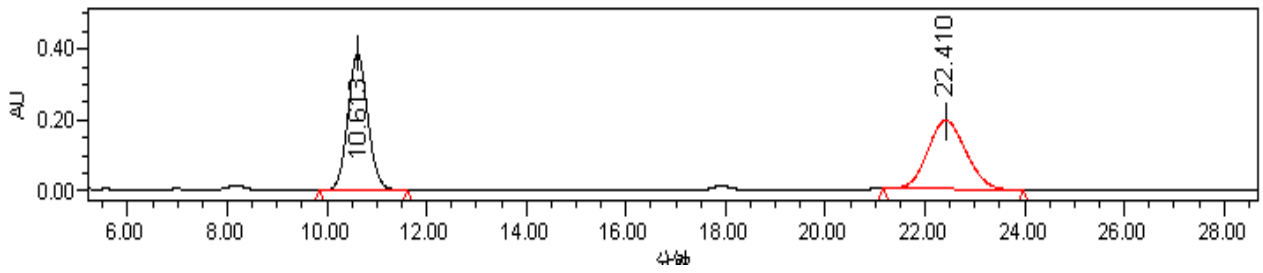
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	16.232	56140855	55.37	1677617	bb	Unknown
2	24.265	45242931	44.63	1050736	bb	Unknown



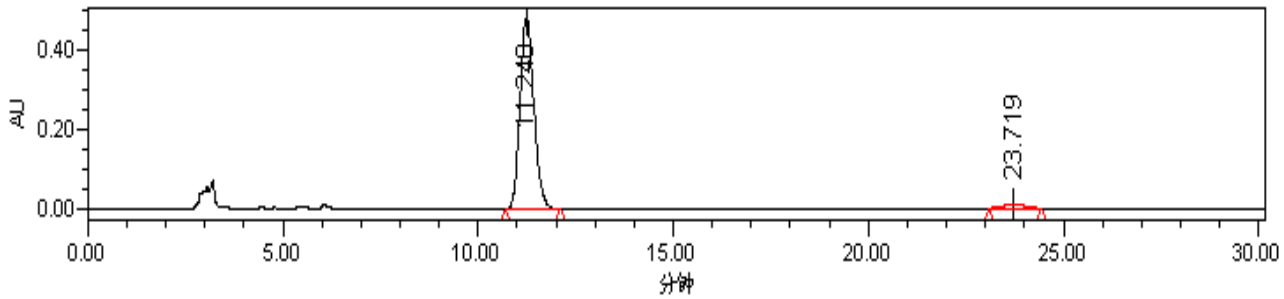
Entry	Retention Time	Area	Area(%)	Height	Int Type	Peak Type
1	16.047	19786203	100.00	579181	bb	Unknown
2	24.600	805	0.00	-43	bb	Unknown

5c: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(o-tolyl)methylcarbamate



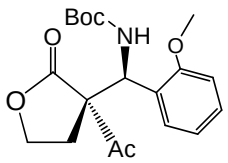


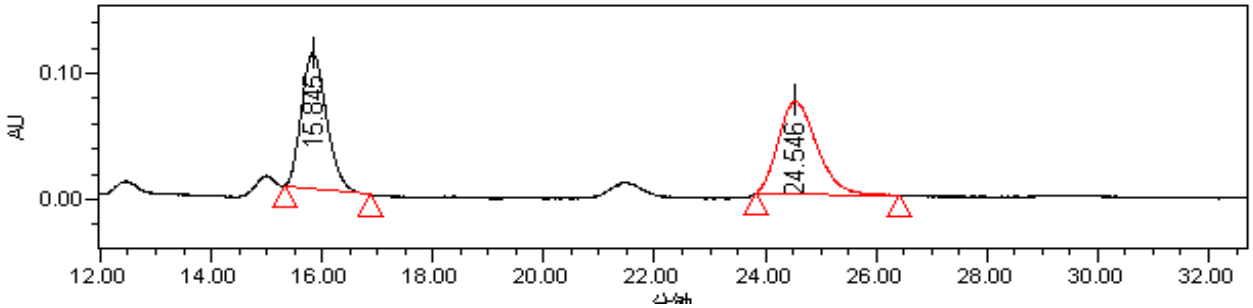
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	10.613	10578988	50.83	383516	bb	Unknown
2	22.410	10234550	49.17	193316	bb	Unknown



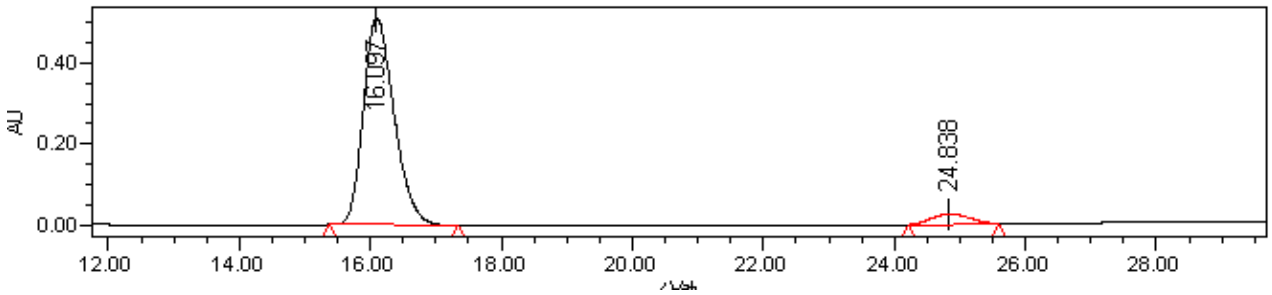
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	11.240	11901811	96.57	482065	bb	Unknown
2	23.719	422183	3.43	9442	bb	Unknown

5d: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(2-methoxyphenyl)methylcarba mate



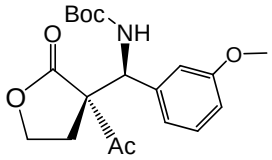


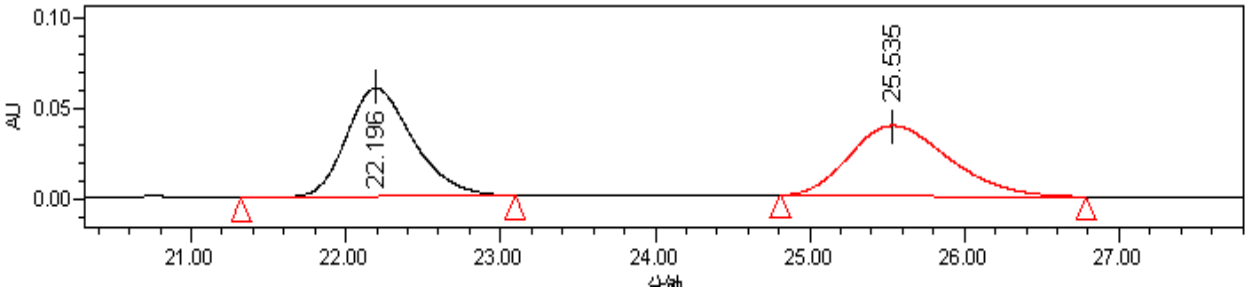
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	15.845	3305170	49.96	106684	bb	Unknown
2	24.546	3310453	50.04	72198	bb	Unknown



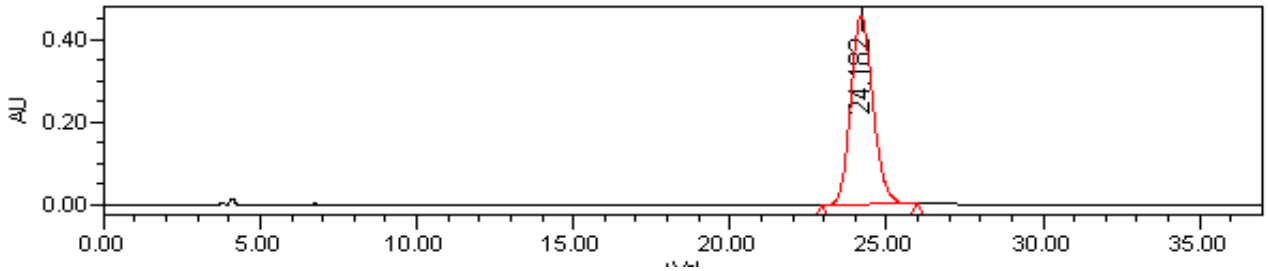
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	16.097	16704411	94.12	511686	bb	Unknown
2	24.838	1043062	5.88	25790	bb	Unknown

5e: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(3-methoxyphenyl)methylcarba mate



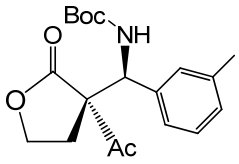


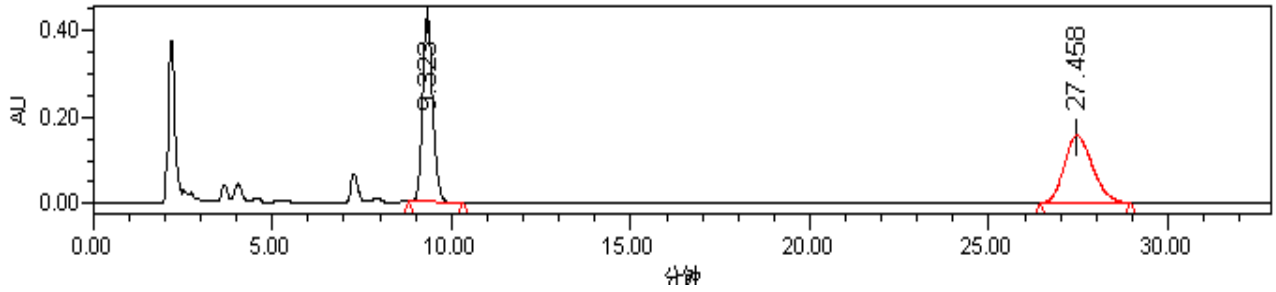
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	22.196	1804146	50.69	60266	bb	Unknown
2	25.535	1755228	49.31	39173	bb	Unknown



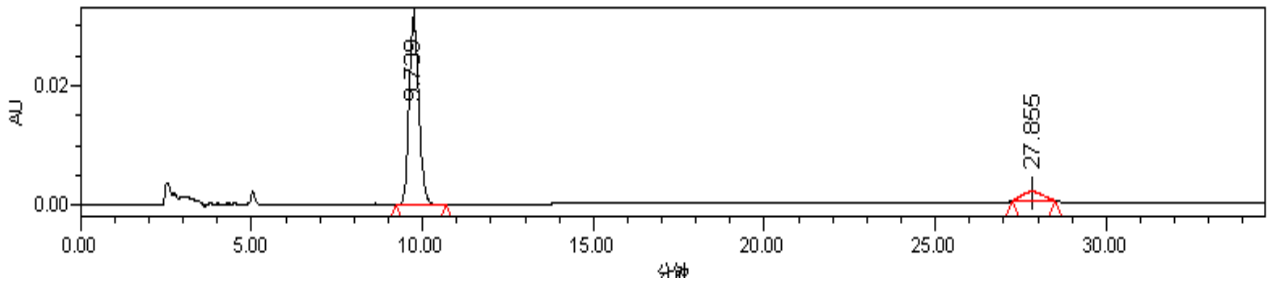
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	24.182	22300762	100.00	454724	bb	Unknown

5f: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(m-tolyl)methylcarbamate



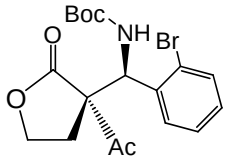


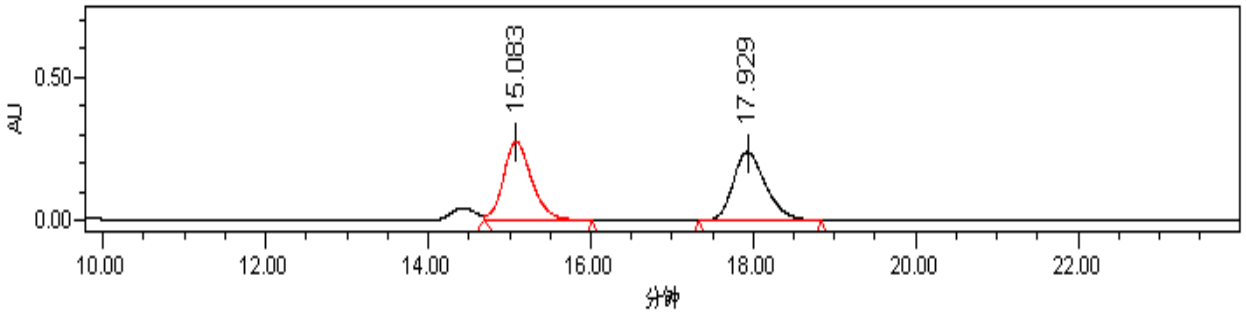
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	9.323	8579092	49.97	430607	bb	Unknown
2	27.458	8589667	50.03	156280	bb	Unknown



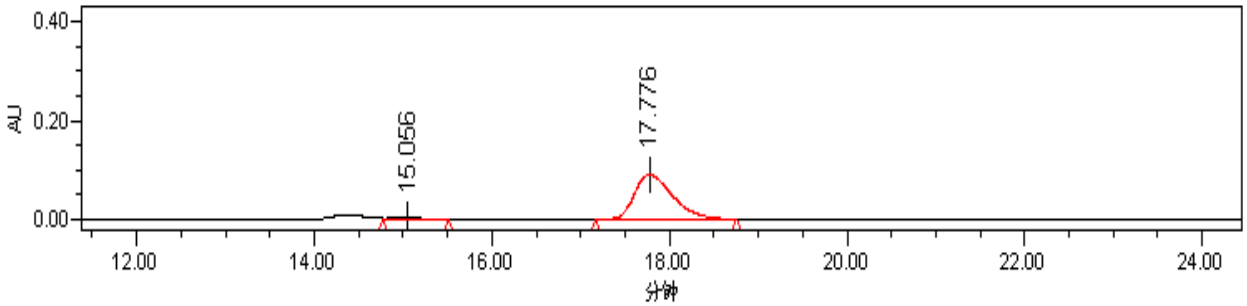
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	9.739	630727	91.00	31451	bb	Unknown
2	27.855	62404	9.00	1517	bb	Unknown

5g: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(2-bromophenyl)methylcarbamate



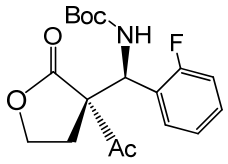


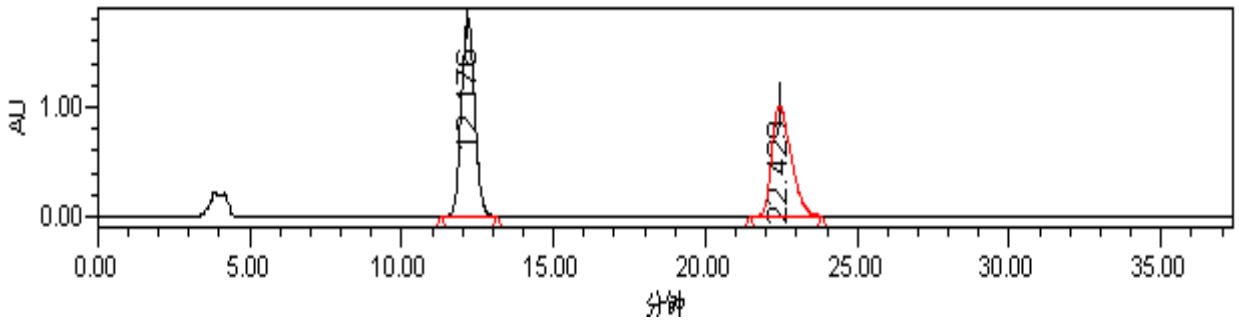
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	15.083	6705176	50.38	274903	bb	Unknown
2	17.929	6604471	49.62	238117	bb	Unknown



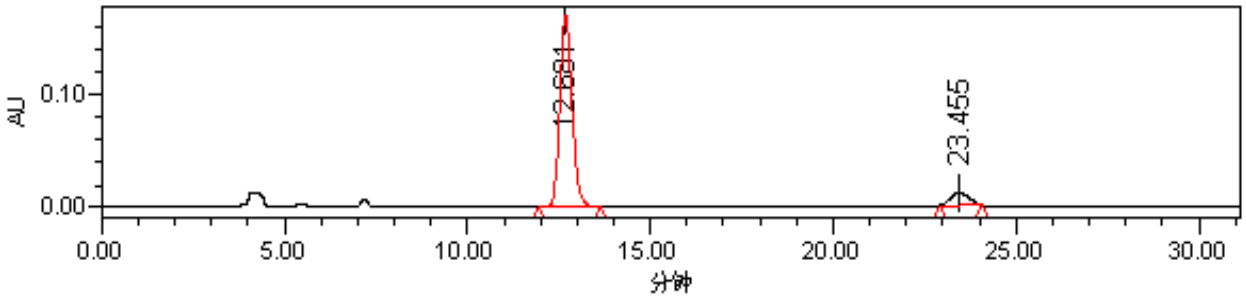
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	15.056	26764	0.99	1484	bb	Unknown
2	17.776	2667108	99.01	90460	bb	Unknown

5h: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(2-fluorophenyl)methylcarbamate



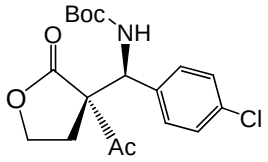


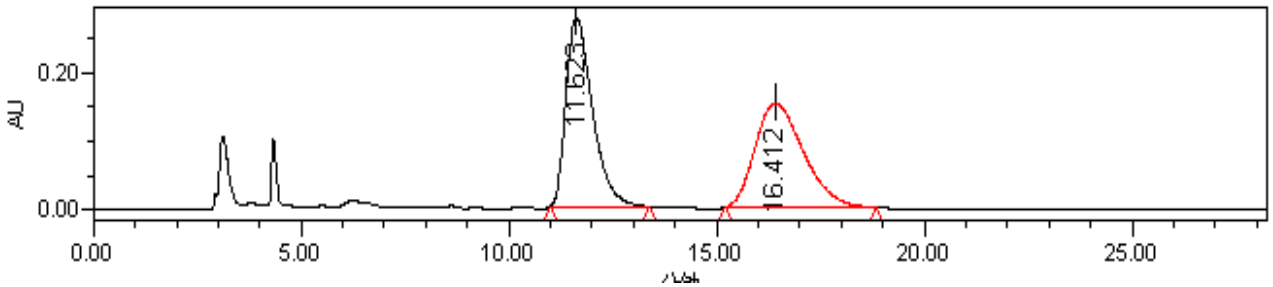
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	12.176	55543409	55.71	1806276	bb	Unknown
2	22.429	44162198	44.29	1002608	bb	Unknown



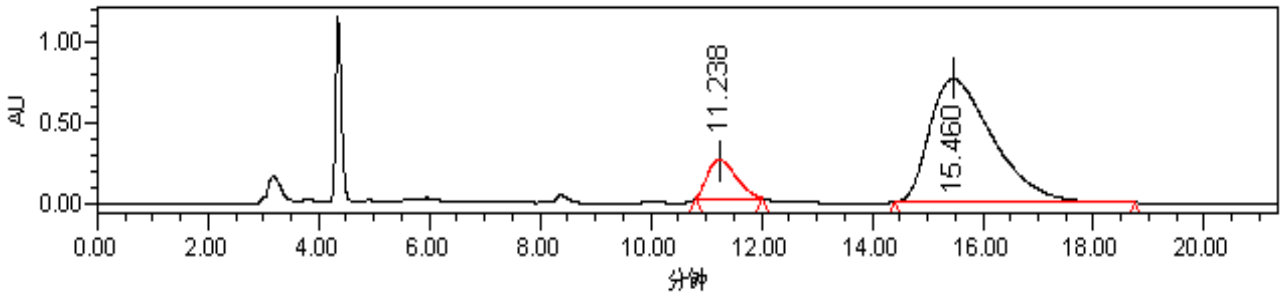
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	12.681	3935660	90.67	169615	bb	Unknown
2	23.455	404998	9.33	11397	bb	Unknown

5i: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(4-chlorophenyl)methylcarbamate



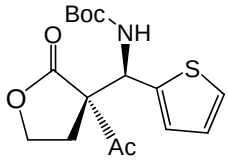


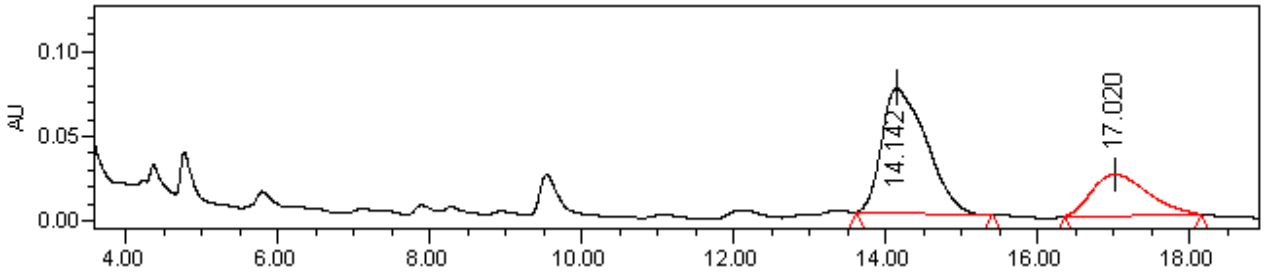
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	11.623	12329132	50.01	277111	bb	Unknown
2	16.412	12322472	49.99	152059	bb	Unknown



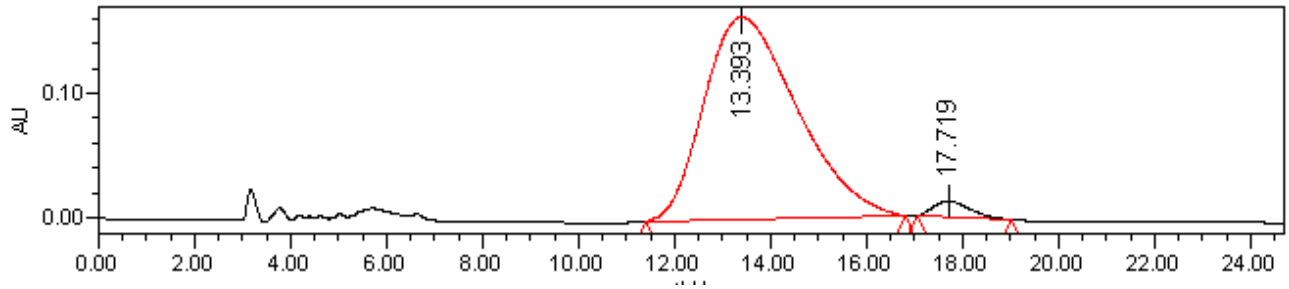
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	11.238	8583491	12.34	240183	bb	Unknown
2	15.460	60985158	87.66	765574	bb	Unknown

5j: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(thiophen-2-yl)methylcarbamate



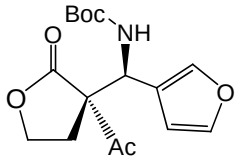


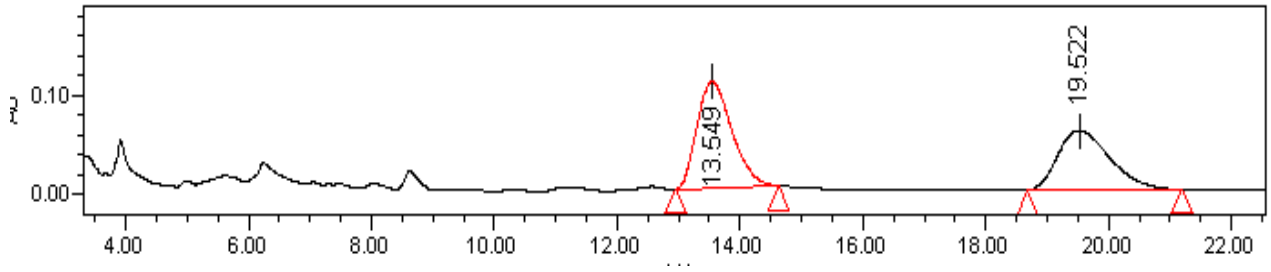
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	14.142	2993541	70.94	73410	bb	Unknown
2	17.020	1226225	29.06	24246	bb	Unknown



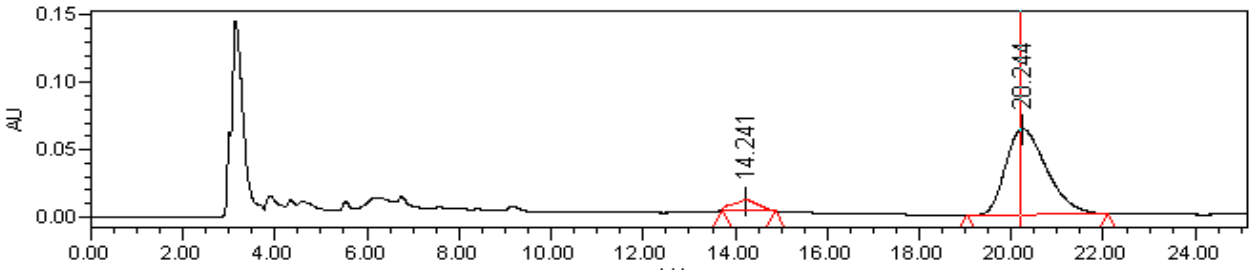
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	13.393	21830132	96.99	161961	bb	Unknown
2	17.719	677564	3.01	12688	bb	Unknown

5k: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(furan-3-yl)methylcarbamate



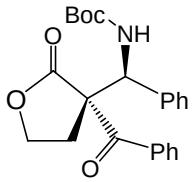


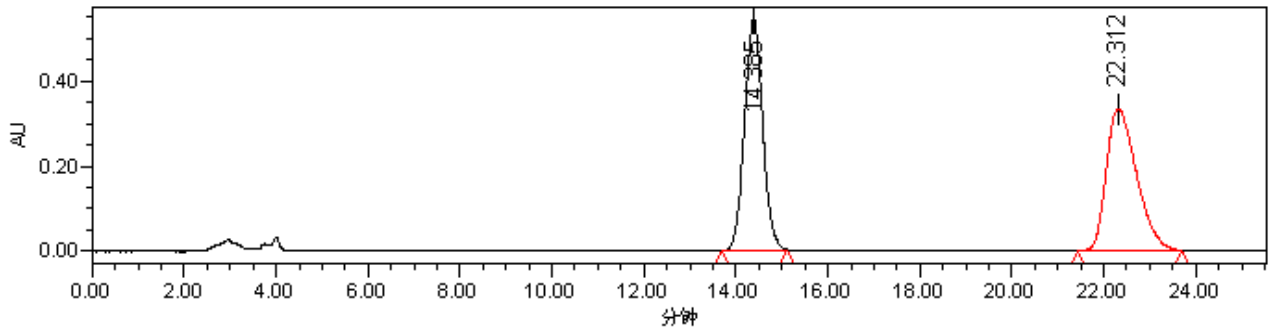
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	13.549	4236471	54.54	107222	bb	Unknown
2	19.522	3531690	45.46	60060	bb	Unknown



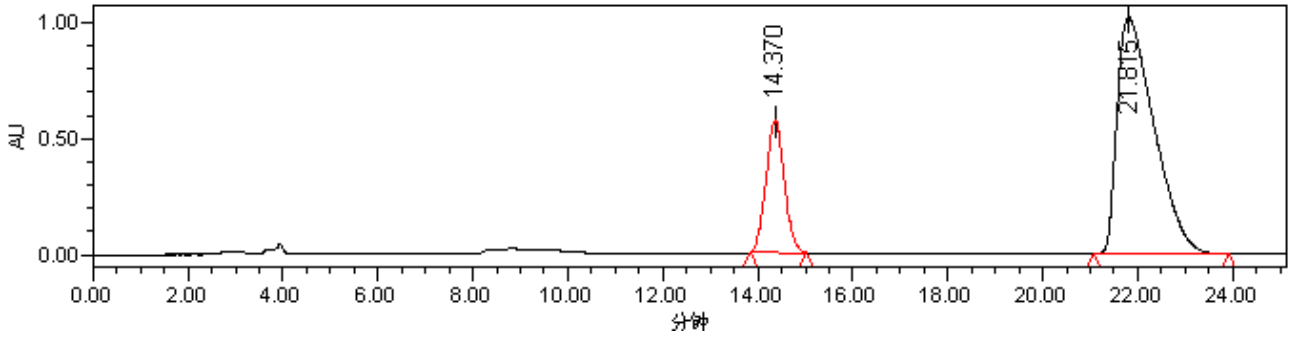
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	14.241	274567	6.52	7263	bb	Unknown
2	20.244	3937079	93.48	63735	bb	Unknown

6a: *tert*-butyl (*S*)-((*R*)-3-benzoyl-2-oxotetrahydrofuran-3-yl)(phenyl)methylcarbamate



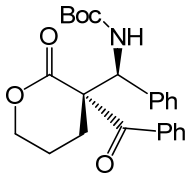


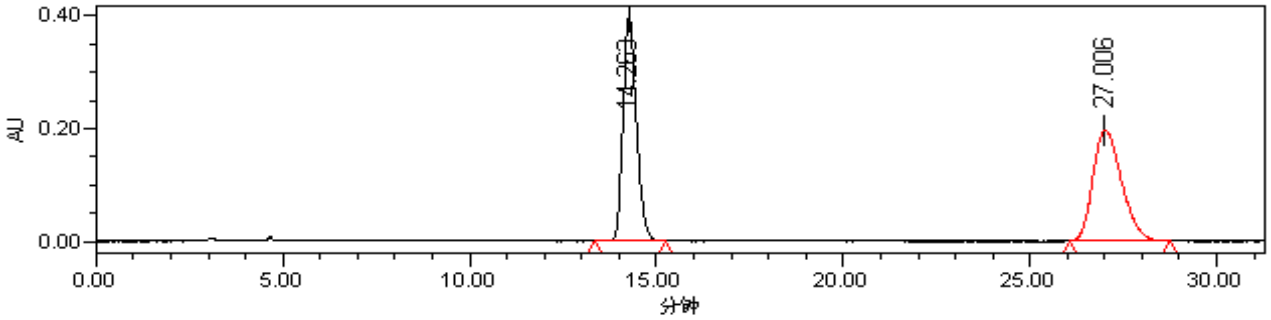
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	14.385	14905498	49.57	542229	bb	Unknown
2	22.312	15161127	50.43	332086	bb	Unknown



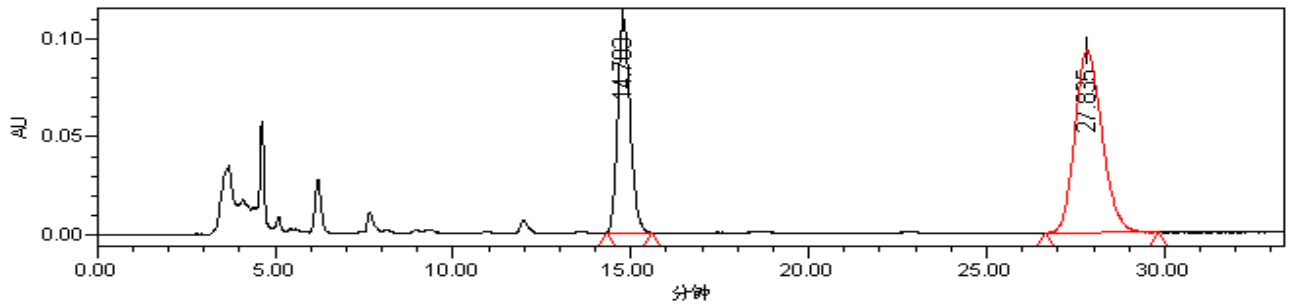
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	14.370	15371234	21.73	569969	bb	Unknown
2	21.815	55379264	78.27	1021933	bb	Unknown

7a: *tert*-butyl (*S*)-((*R*)-3-benzoyl-2-oxotetrahydro-2H-pyran-3-yl)(phenyl)methylcarbamate



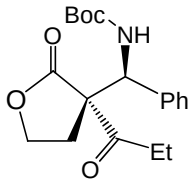


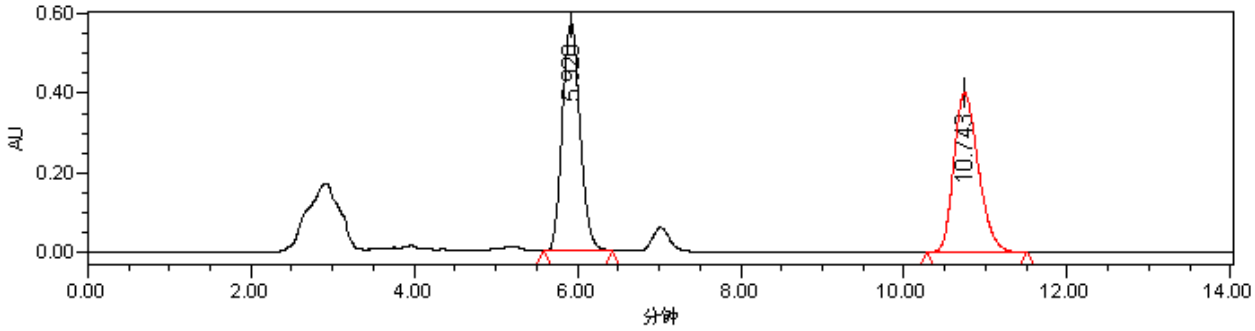
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	14.263	9862841	48.53	397239	bb	Unknown
2	27.006	10462204	51.47	197160	bb	Unknown



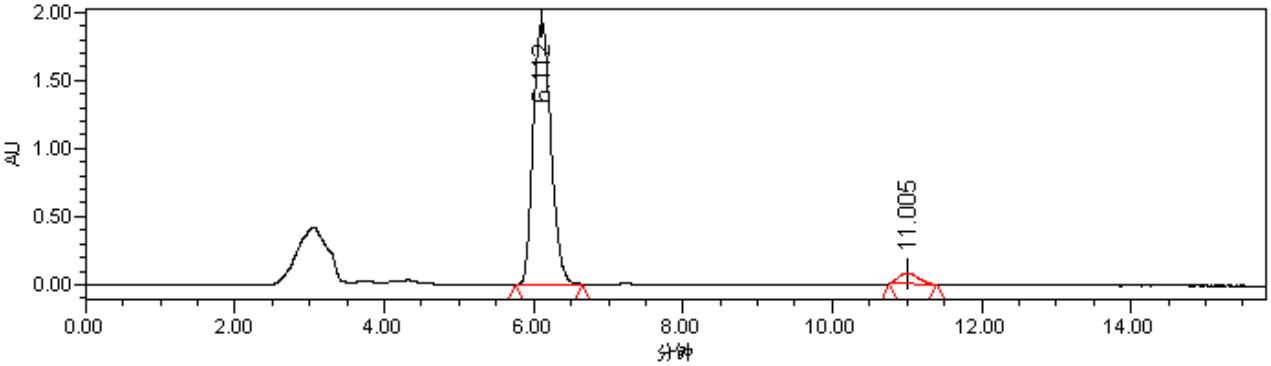
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	14.783	2685478	35.34	109010	bb	Unknown
2	27.835	4913932	64.66	92670	bb	Unknown

8a: *tert*-butyl (*S*)-((*R*)-2-oxo-3-propionyltetrahydrofuran-3-yl)(phenyl)methylcarbamate





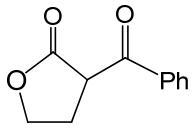
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	5.920	9305783	50.65	619077	bb	Unknown
2	10.743	9065509	49.35	433638	bb	Unknown

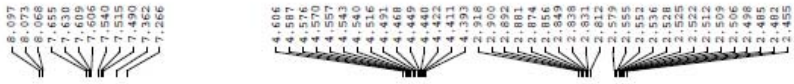


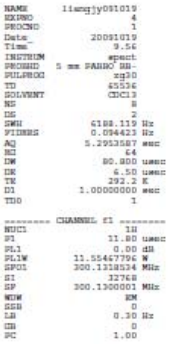
Entry	Retention Time	Area	Area (%)	Height	Int Type	Peak Type
1	6.112	31783495	95.58	1924229	bb	Unknown
2	11.005	1469640	4.42	80758	bb	Unknown

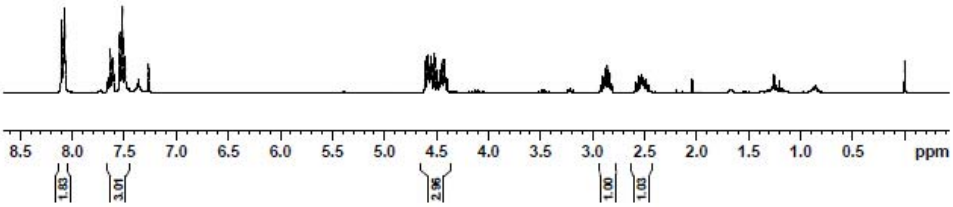
8.0 Copies of NMR spectra of products.

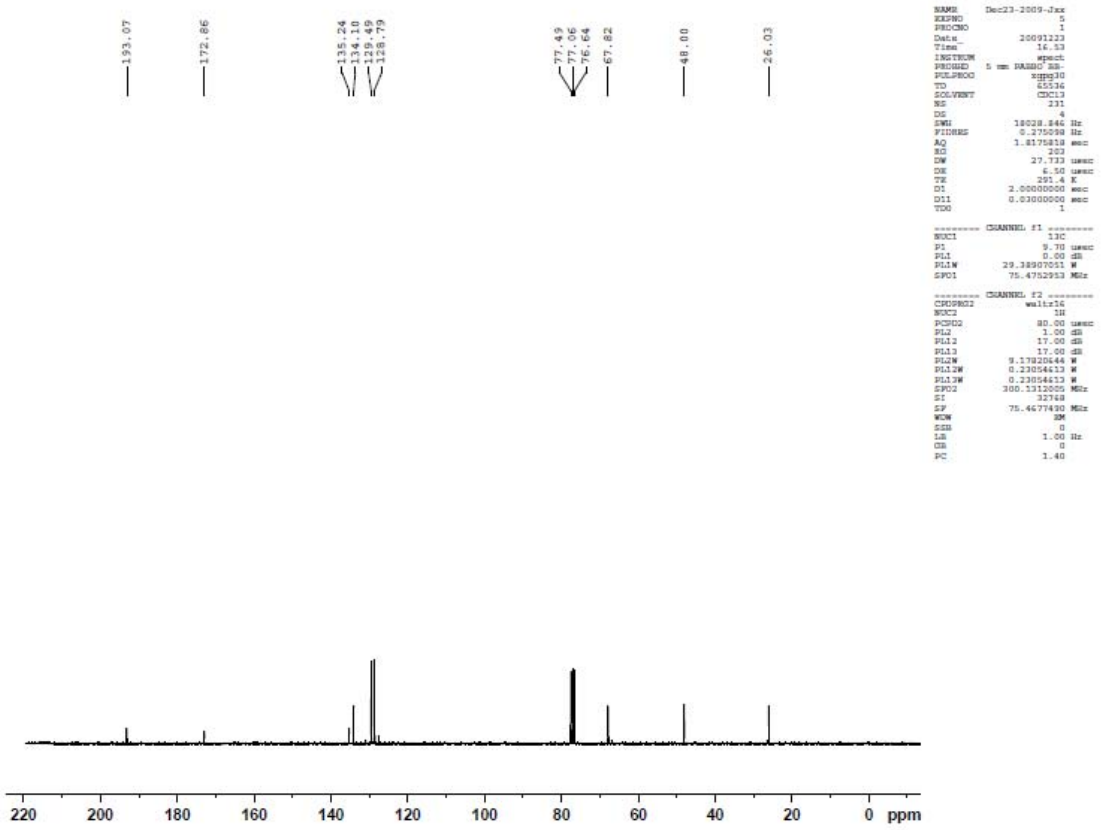
4b: 3-benzoyldihydrofuran-2(3H)-one



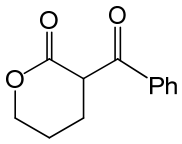


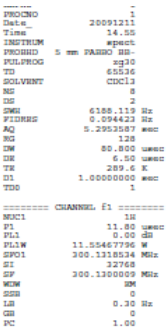


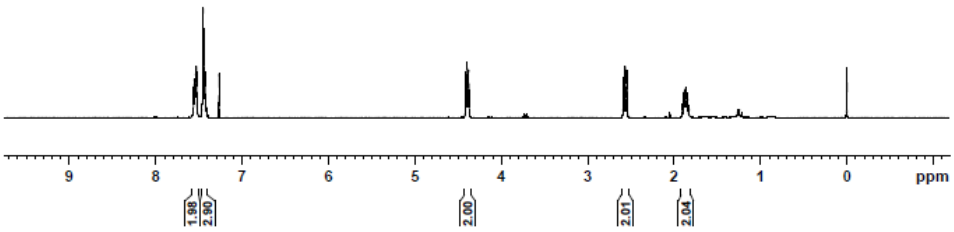


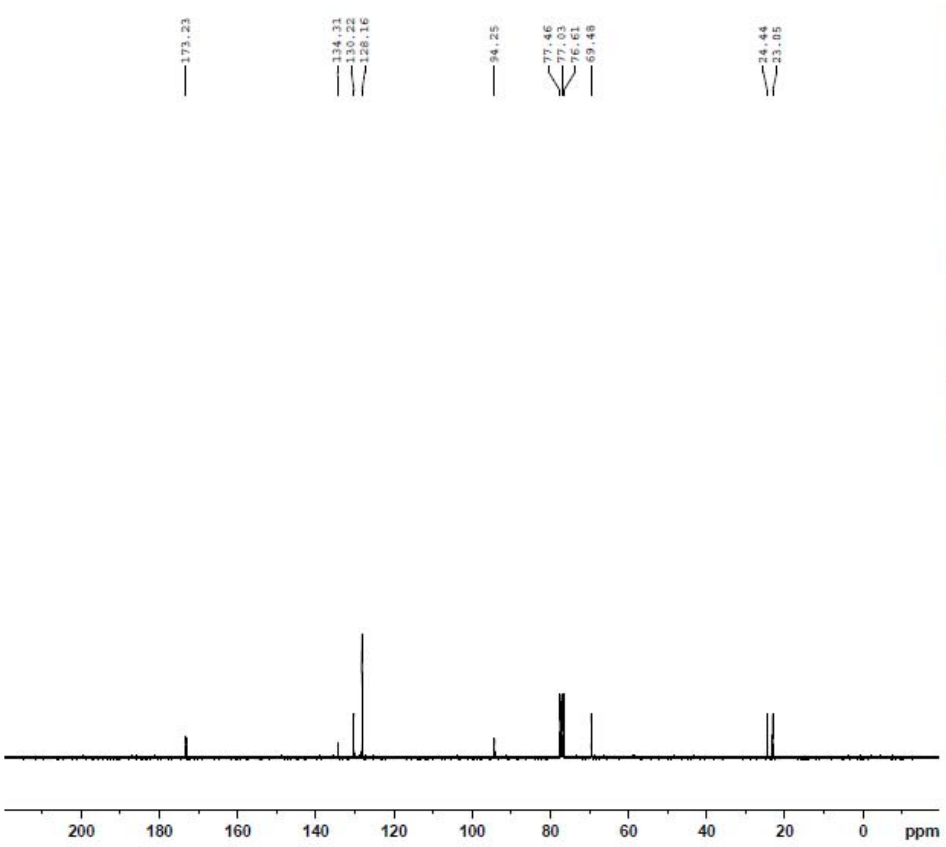

193.07
172.86
135.24
134.10
129.49
128.79
77.49
77.06
76.64
67.82
48.00
26.03
NAME Dec23-2009-Jxx
EXPNO 5
PROCNO 1
Date_ 20091223
Time 16.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 231
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 291.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
220
200
180
160
140
120
100
80
60
40
20
0 ppm

4c: 3-benzoyltetrahydro-2H-pyran-2-one



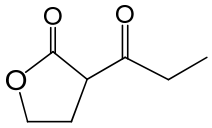


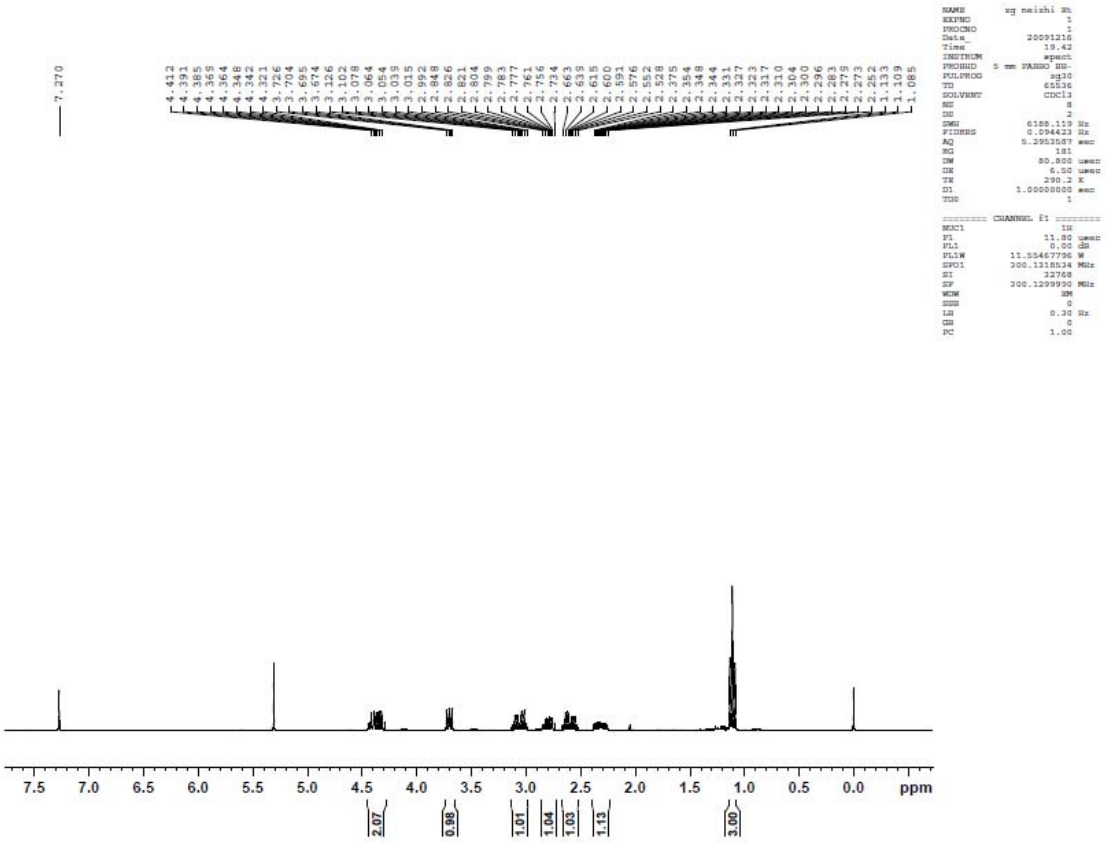


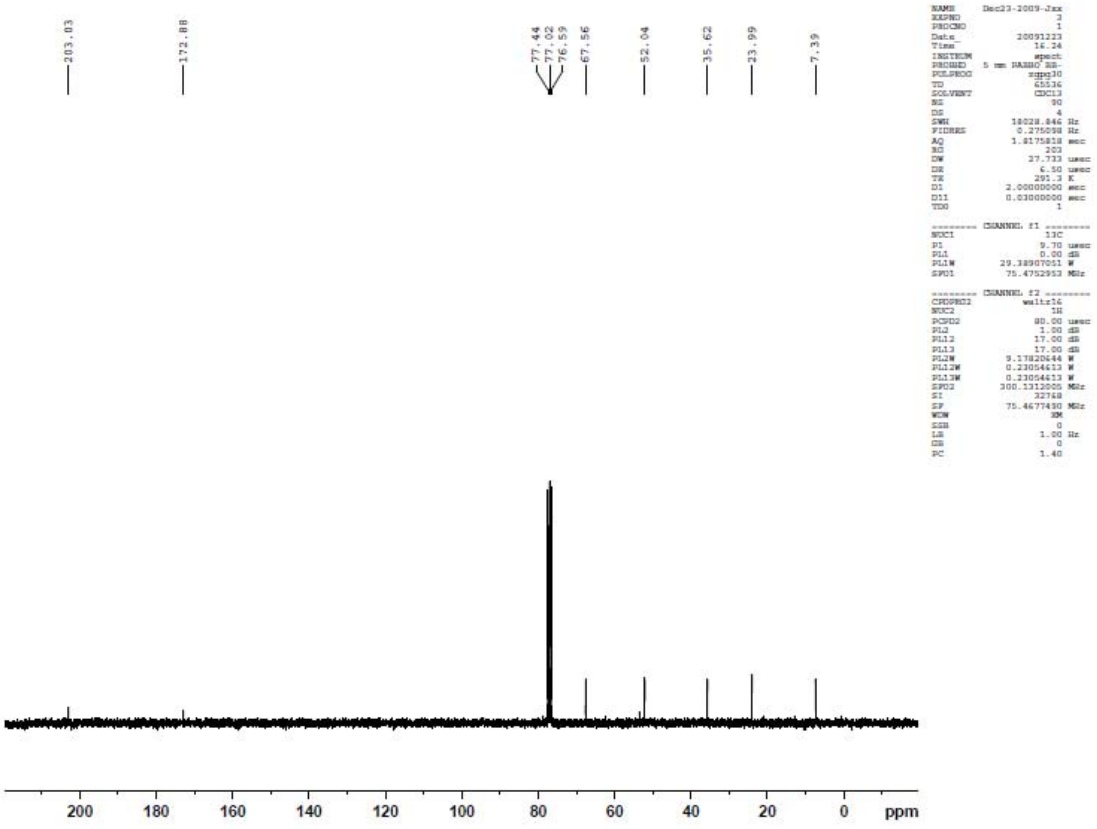



```
NAME          Dec23-2009-Jxx
EXPNO                     2
PROCNO                    1
Date_              20091223
Time                  16.52
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     242
DS                       4
SWH              18028.846 Hz
FIDRES               0.275098 Hz
AQ                1.8175616 sec
RG                     203
DW                  27.733 usec
DE                   6.50 usec
TE                  291.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.70 usec
PL1                   0.00 dB
PL1W            29.38907051 W
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
PL2W             9.17820644 W
PL12W            0.23054413 W
PL13W            0.23054413 W
SFO2            300.1312005 MHz
SI                     32768
SF              75.4677490 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                    1.40
```

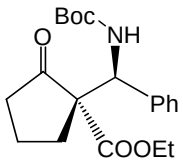
4d: 3-propionyldihydrofuran-2(3H)-one

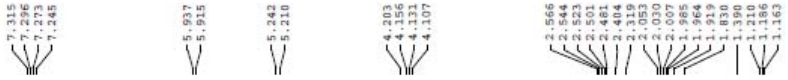


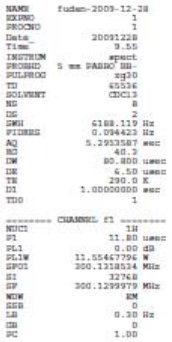


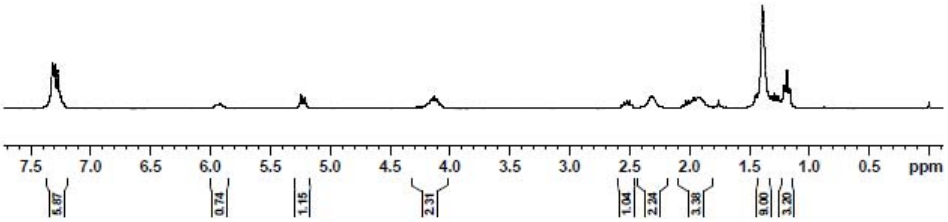

203.03
172.88
77.44
77.02
76.59
67.56
52.04
35.62
23.99
7.39
200
180
160
140
120
100
80
60
40
20
0
ppm

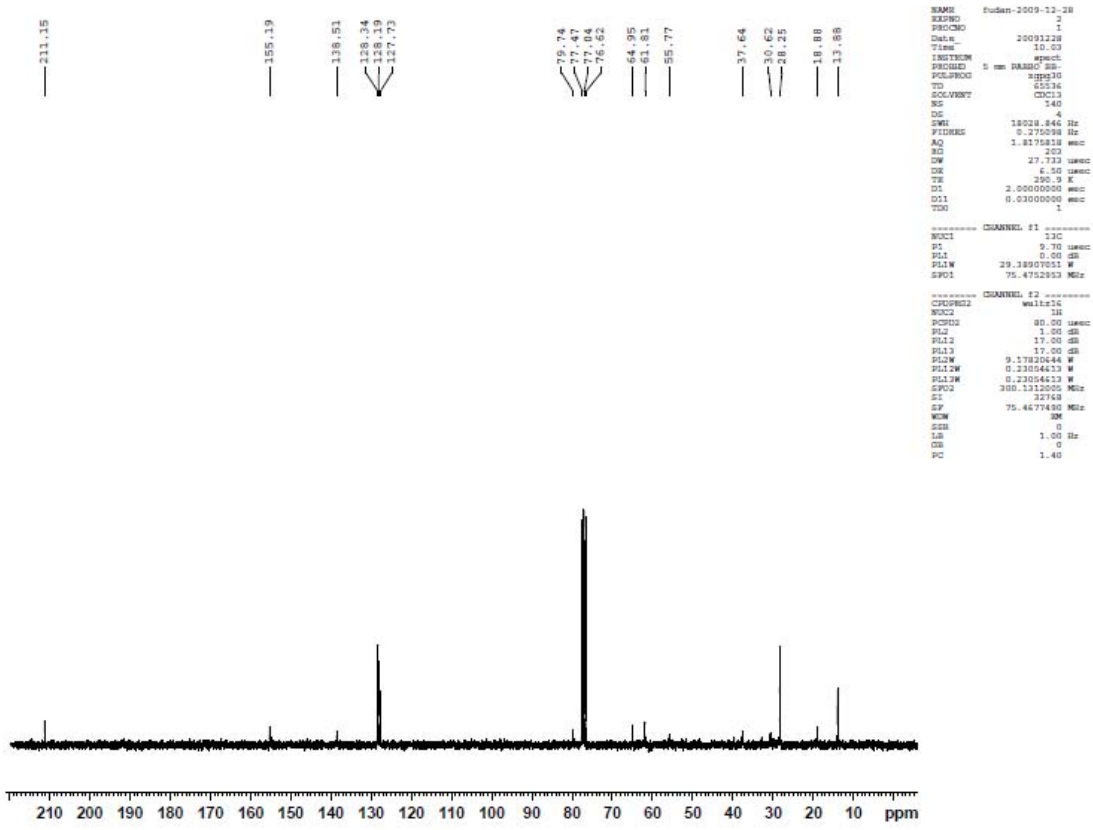
3a: (*S*)-ethyl 1-((*S*)-(*tert*-butoxycarbonylamino)(phenyl)methyl)-2-oxocyclopentane carboxylate



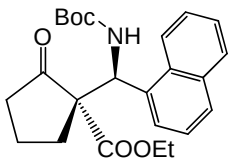


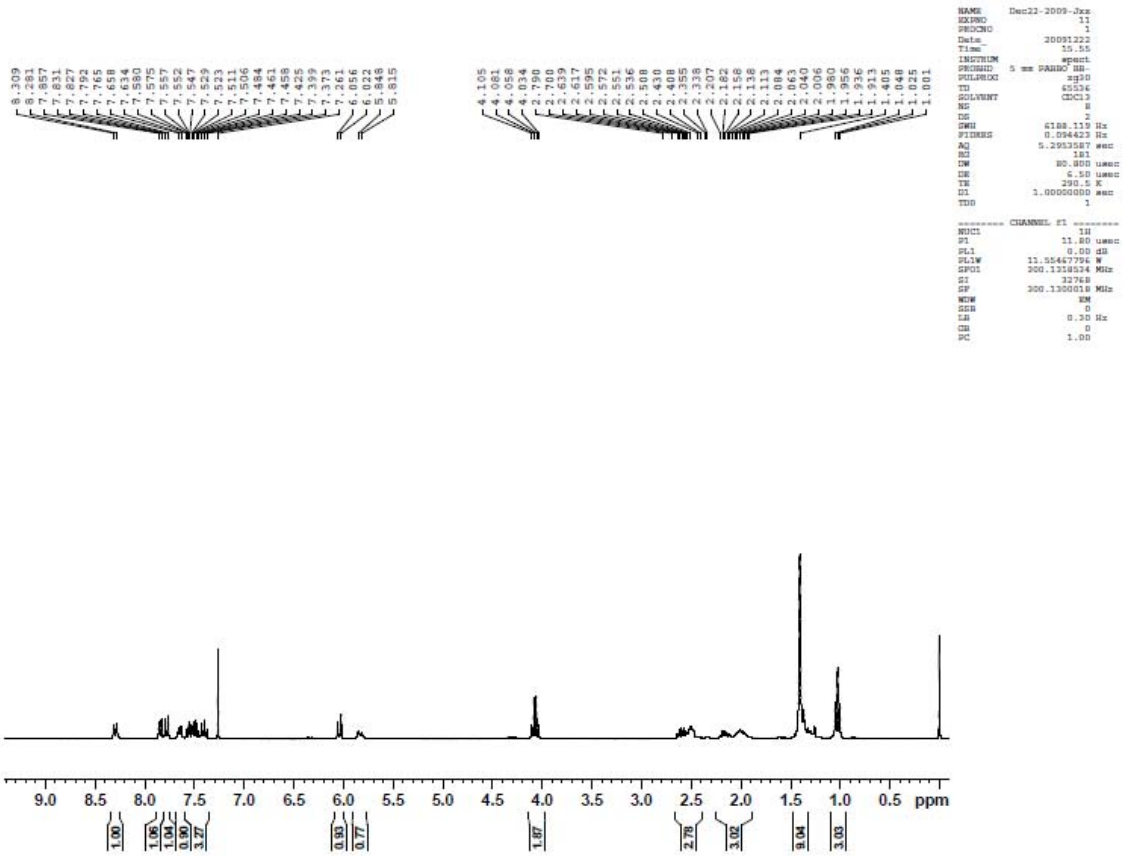


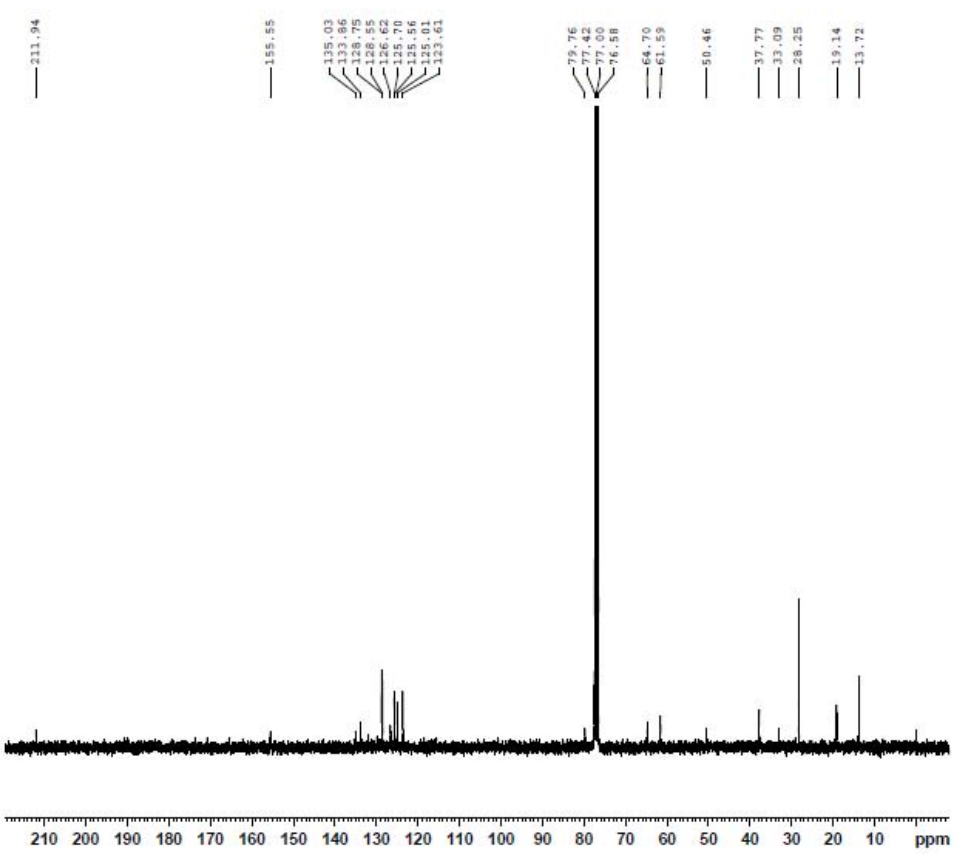



211.15
155.19
138.51
128.34
128.19
127.73
79.74
77.47
77.04
76.62
64.95
61.81
55.77
37.64
30.62
28.25
18.88
13.88
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3b: (*S*)-ethyl 1-((*S*)-(*tert*-butoxycarbonylamino)(naphthalen-1-yl)methyl)-2-oxocyclopentane carboxylate

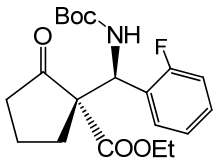


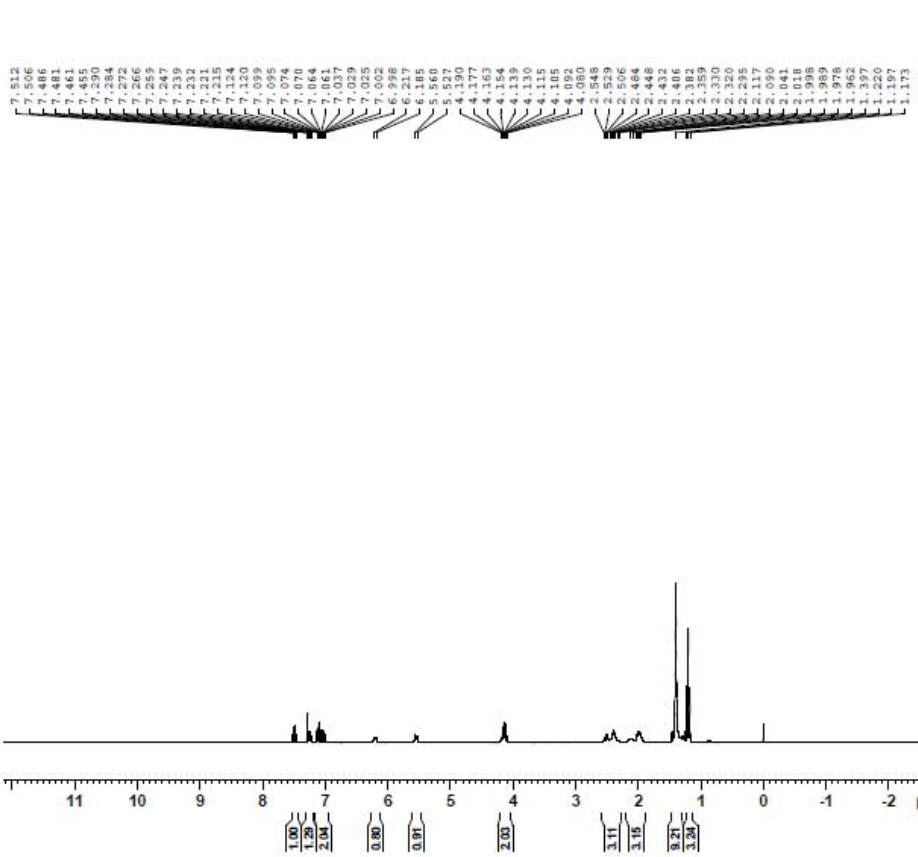


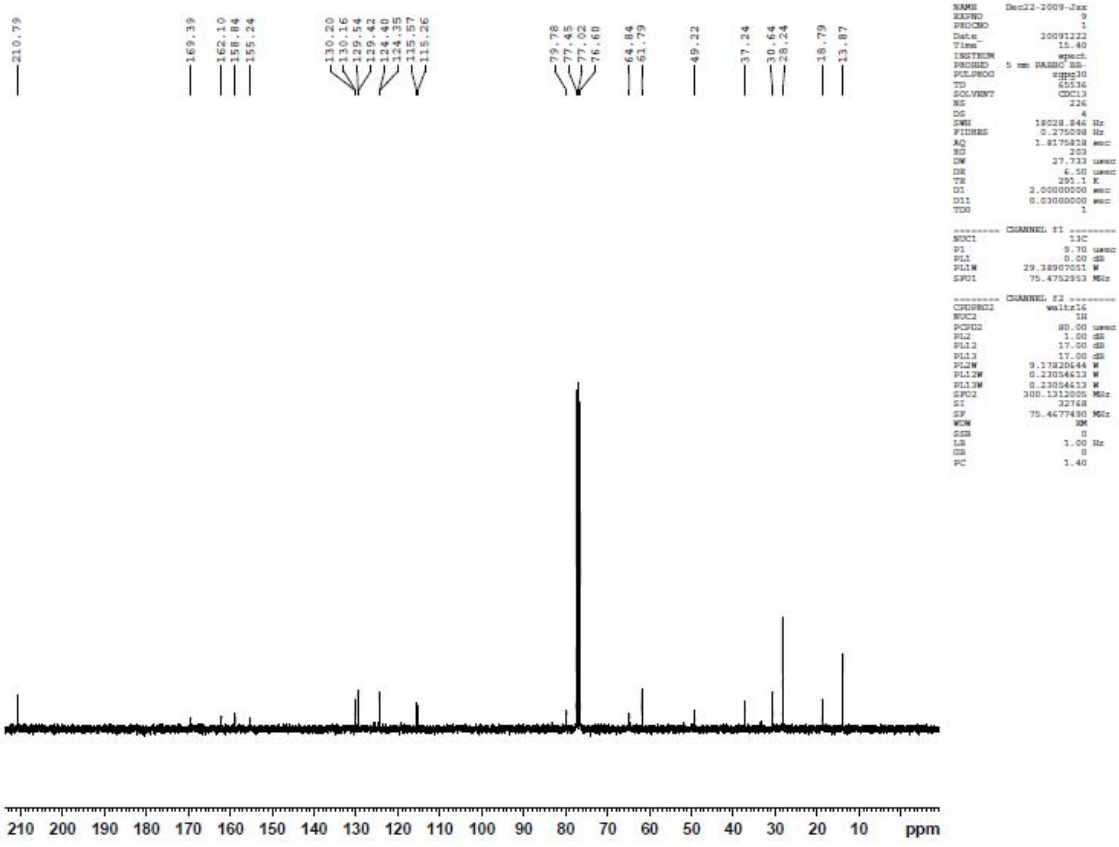



```
NAME          Dec22-2009-Jxx
EXPNO                    12
PROCNO                    1
Date_              20091222
Time                  16.03
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1051
DS                       4
SWH              18028.846 Hz
FIDRES           0.275098 Hz
AQ               1.8175818 sec
RG                     203
DW                27.733 usec
DE                 6.50 usec
TE                291.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.70 usec
PL1                   0.00 dB
PL1W            29.38907051 W
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
PL2W            9.17820644 W
PL12W           0.23054613 W
PL13W           0.23054613 W
SFO2            300.1312005 MHz
SI                    32768
SF              75.4677494 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

3c: (*S*)-ethyl 1-((*R*)-(*tert*-butoxycarbonylamino)(2-fluorophenyl)methyl)-2-oxocyclopentane carboxylate

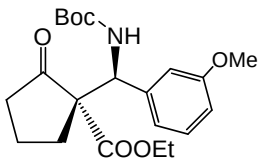


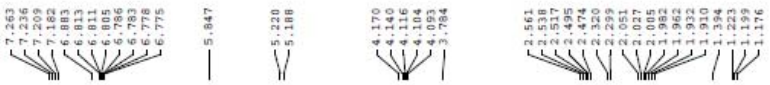


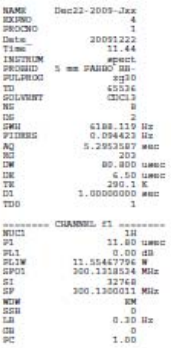


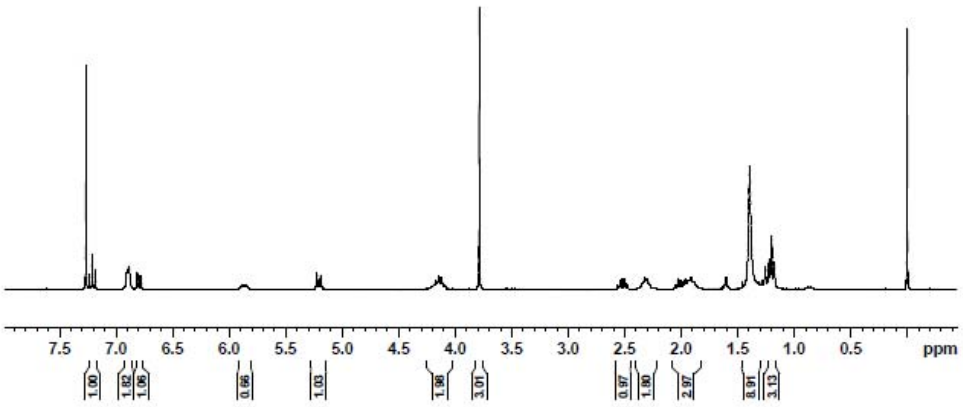

```
NAME          Dec22-2009-Jax
EXPNO                     9
PROCNO                    1
Date_          20091222
Time                  15.40
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      226
DS                        4
SWH              18028.846 Hz
FIDRES               0.275098 Hz
AQ                1.8175818 sec
RG                      203
DW                   27.733 usec
DE                    6.50 usec
TE                   291.1 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.70 usec
PL1                    0.00 dB
PL1W            29.38907051 W
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                  80.00 usec
PL2                    1.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
PL2W            9.17820644 W
PL12W           0.23054613 W
PL13W           0.23054613 W
SFO2            300.1312005 MHz
SI                     32768
SF              75.4677490 MHz
WDW                       EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

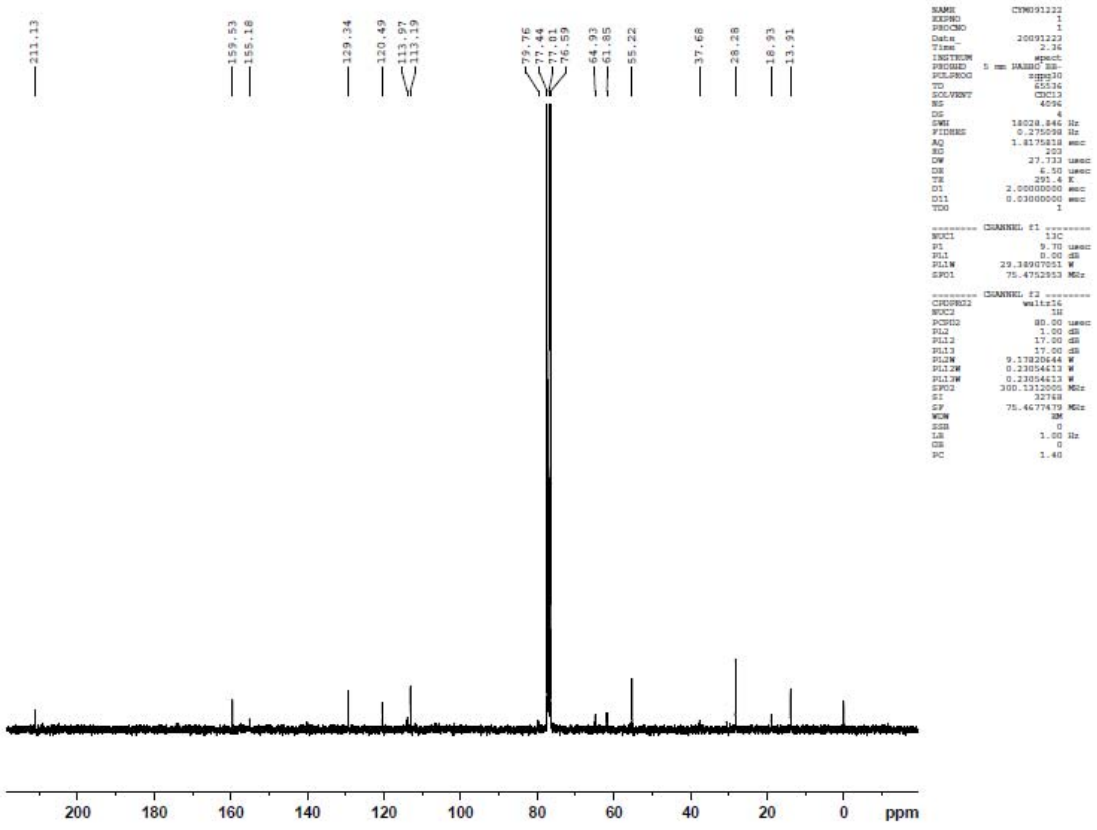
3d: (*S*)-ethyl 1-((*S*)-(*tert*-butoxycarbonylamino)(3-methoxyphenyl)methyl)-2-oxocyclopentanecarboxylate





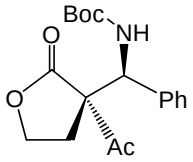


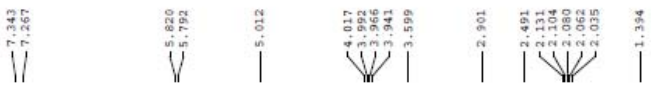


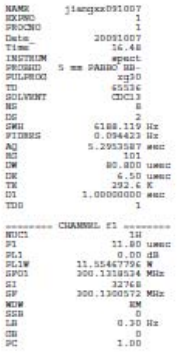


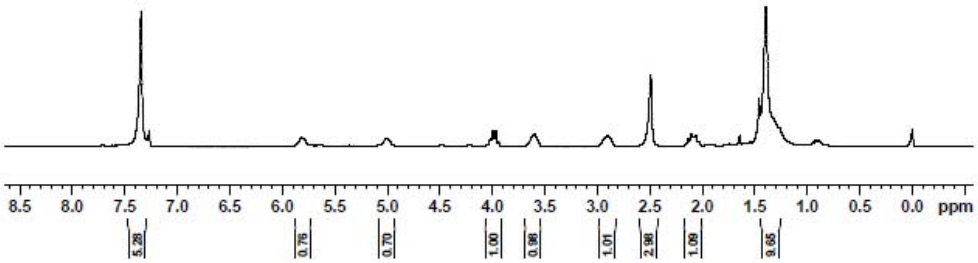

```
NAME            CEM091222
EXPNO                   1
PROCNO                  1
Date_          20091223
Time                 2.36
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                  65536
SOLVENT               CDCl3
NS                     4096
DS                        4
SWH              18028.846 Hz
FIDRES               0.275098 Hz
AQ                1.8175818 sec
RG                      203
DW                   27.733 usec
DE                    6.50 usec
TE                  291.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.70 usec
PL1                    0.00 dB
PL1W            29.38907051 W
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
PL2W            9.17820644 W
PL12W           0.23054613 W
PL13W           0.23054613 W
SFO2            300.1312005 MHz
SI                    32768
SF              75.4677479 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

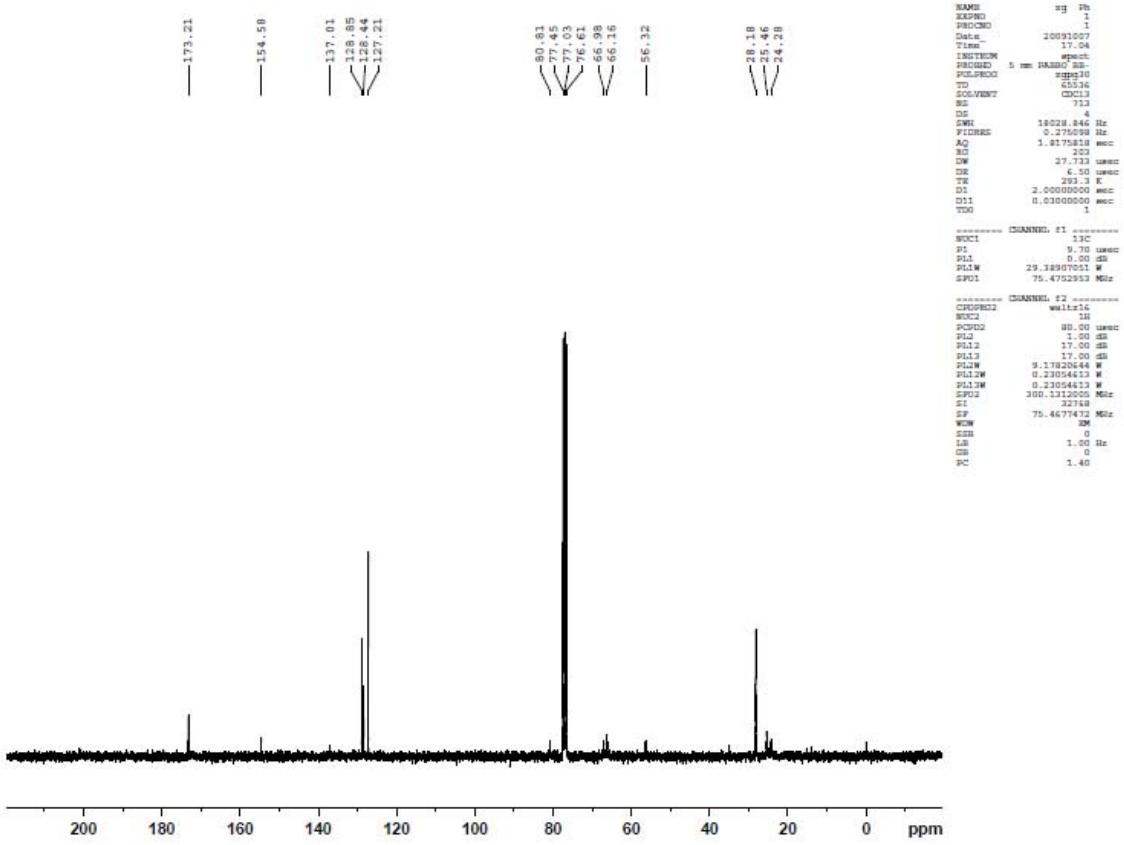
5a: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(phenyl)methylcarbamate



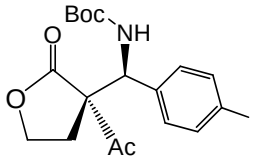


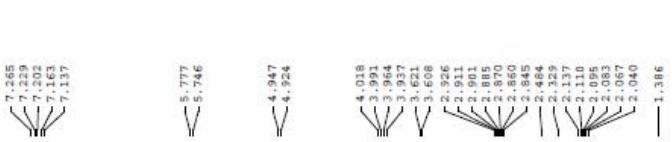


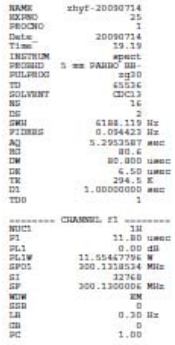


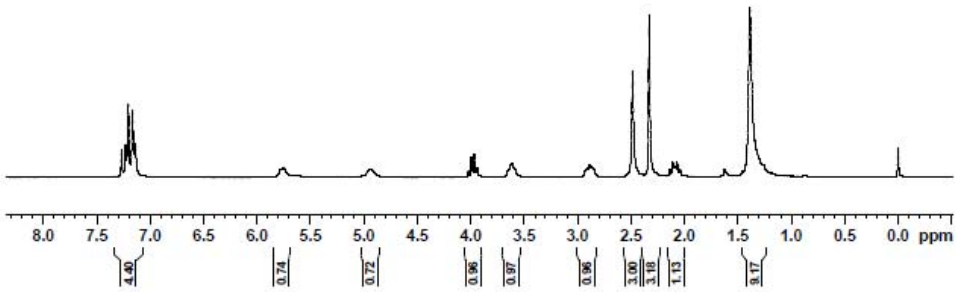

173.21
154.58
137.01
128.85
128.44
127.21
80.81
77.45
77.03
76.61
66.98
66.16
56.32
28.18
25.46
24.28
200
180
160
140
120
100
80
60
40
20
0
ppm

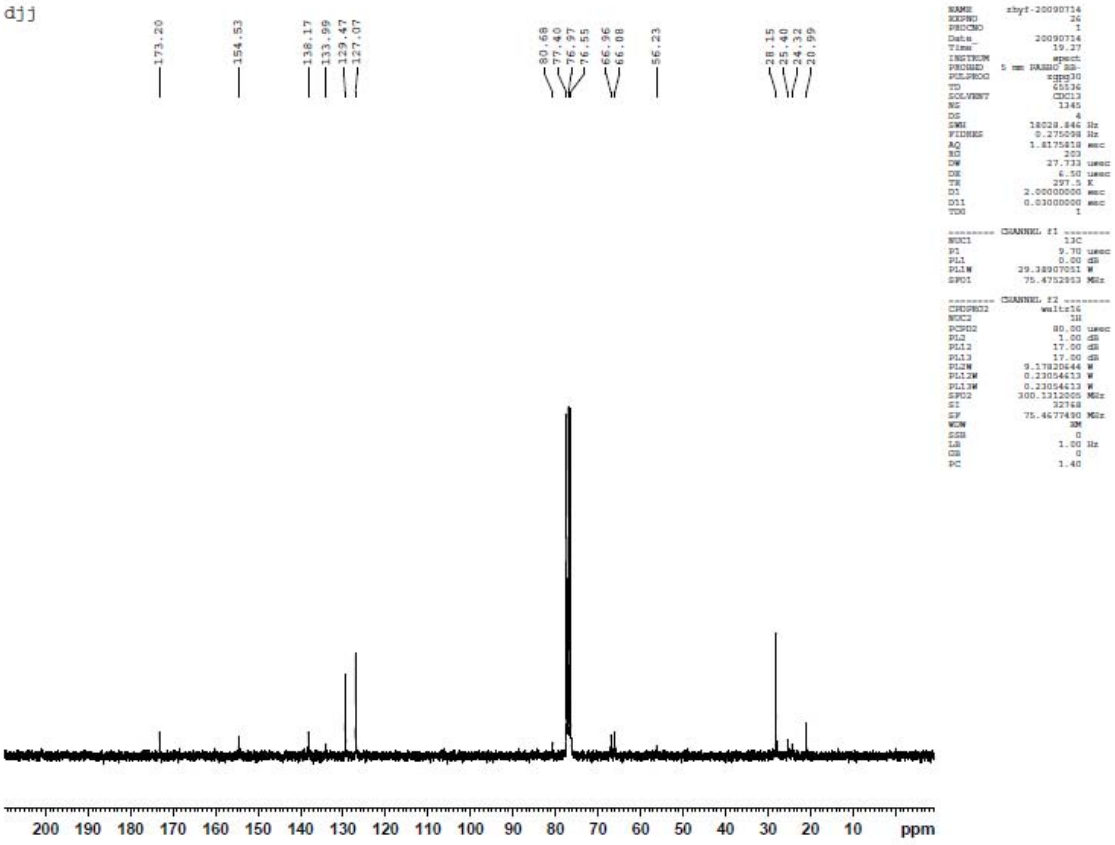
5b: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(p-tolyl)methylcarbamate





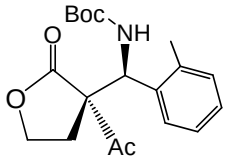


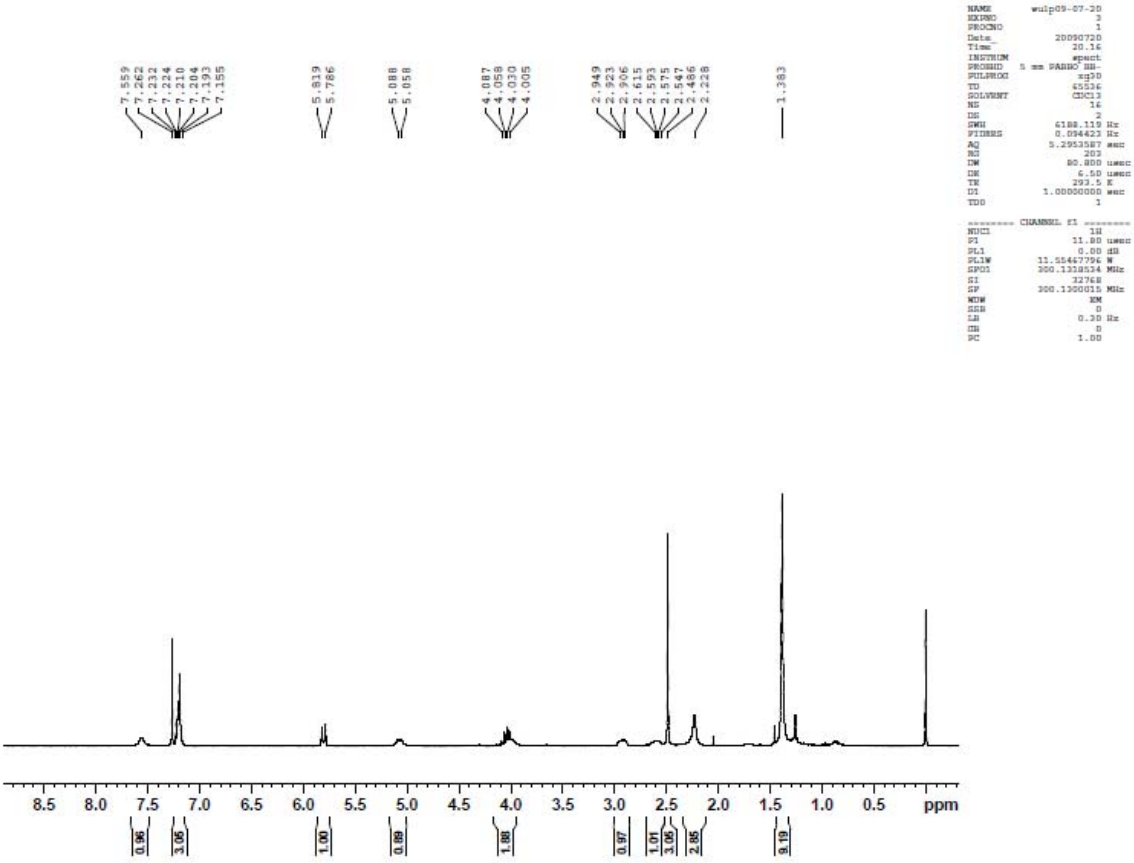


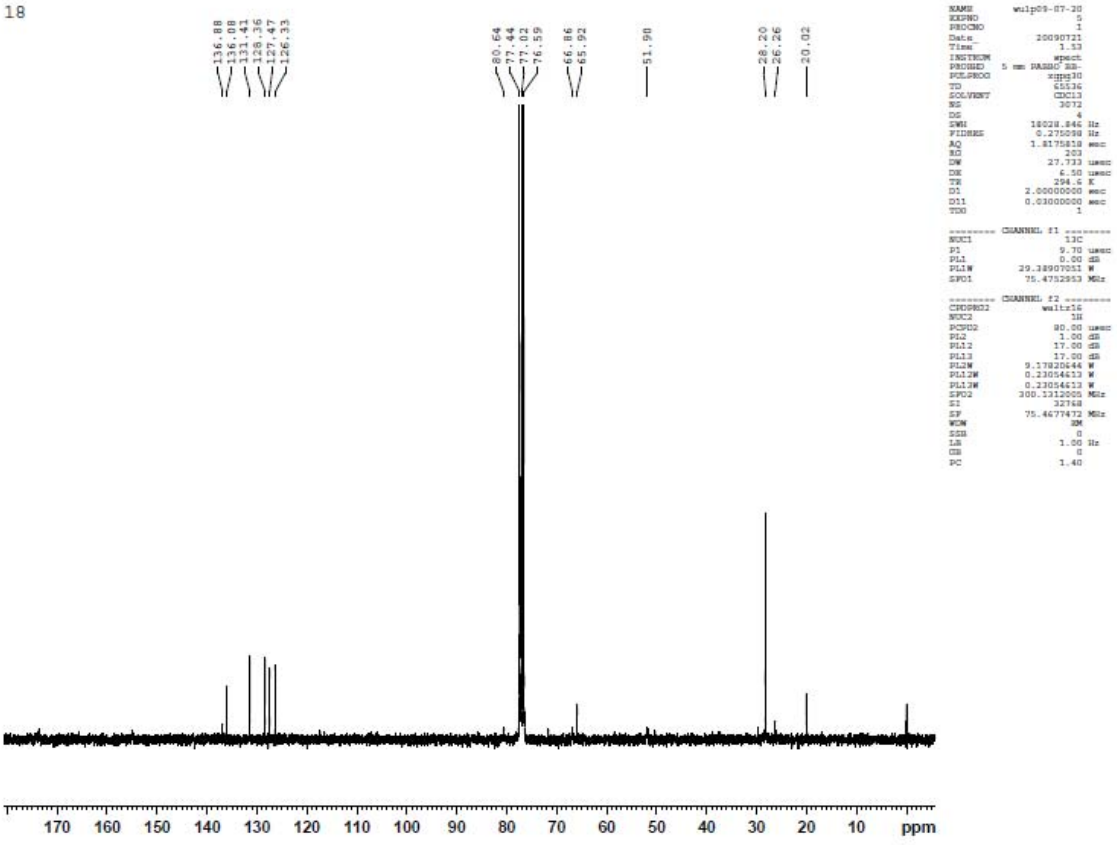


NAME	zhyf-20090714
EXPNO	26
PROCNO	1
Date_	20090714
Time	19.27
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1345
DS	4
SWH	18028.846 Hz
FIDRES	0.275098 Hz
AQ	1.8175818 sec
RG	203
DW	27.733 usec
DE	6.50 usec
TE	297.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	0.00 dB
PL1W	39.38907051 W
SFO1	75.4752853 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.00 dB
PL13	17.00 dB
PL2W	9.17820644 W
PL12W	0.23054613 W
PL13W	0.23054613 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

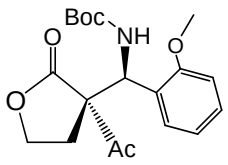
5c: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(o-tolyl)methylcarbamate

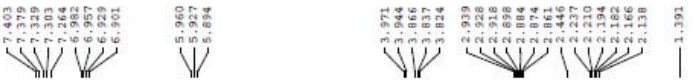


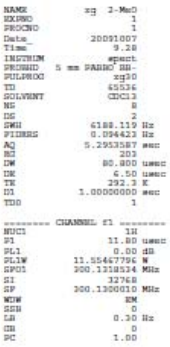


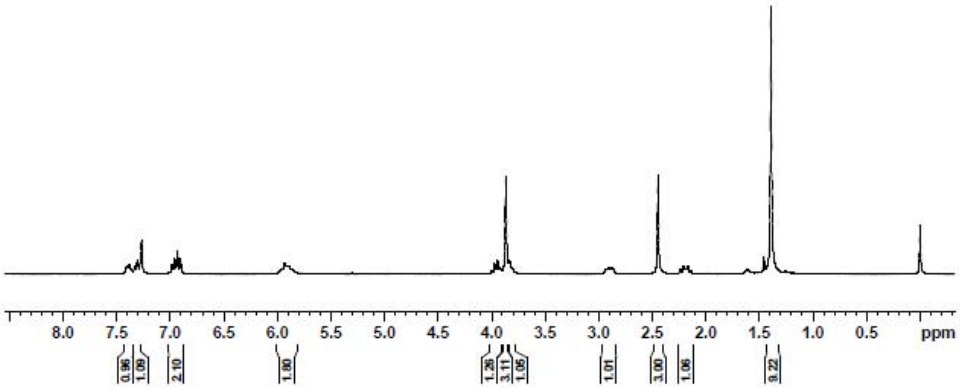

18
136.88
136.08
131.41
128.36
127.47
126.33
80.64
77.44
77.02
76.59
66.86
65.92
51.90
28.20
26.26
20.02
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

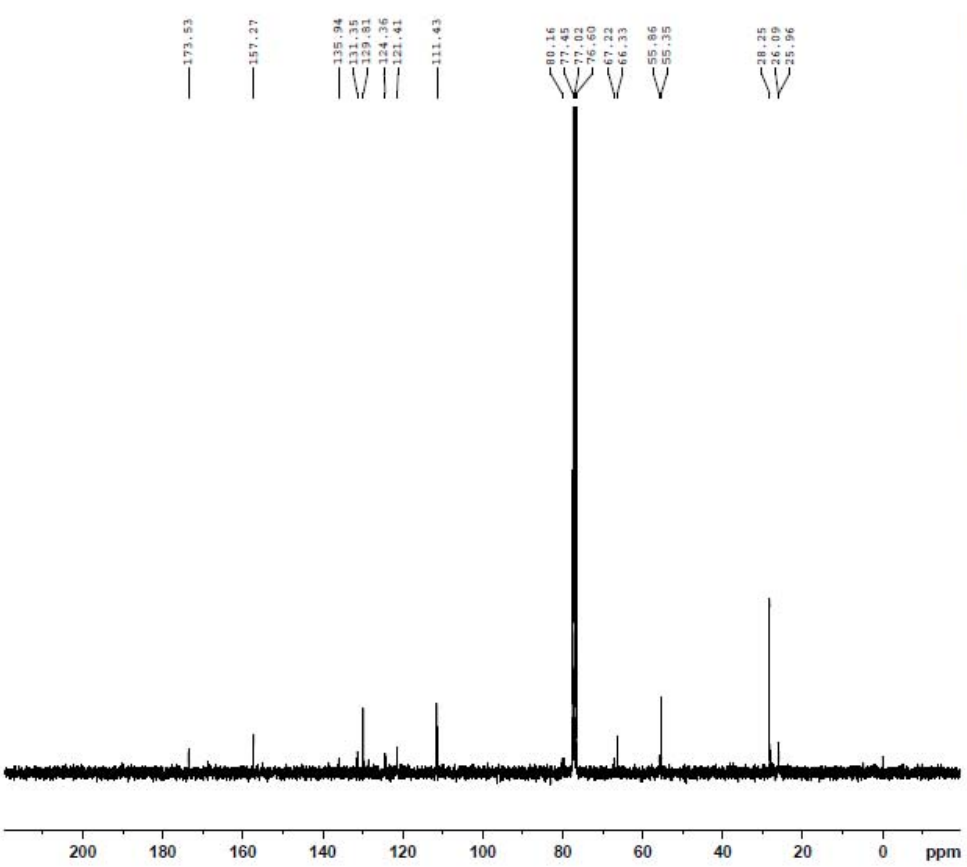
5d: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(2-methoxyphenyl)methylcarba mate





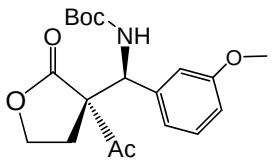


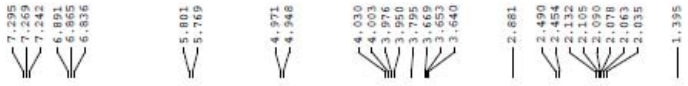


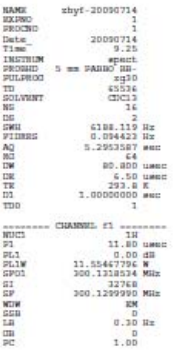


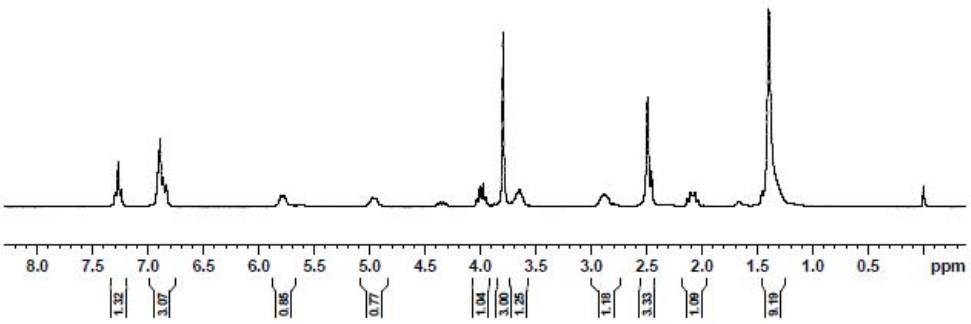

```
NAME                xg  2-MeO
EXPNO                    4
PROCNO                   1
Date_             20091007
Time                 16.04
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     542
DS                       4
SWH              18028.846 Hz
FIDRES           0.275098 Hz
AQ               1.8175818 sec
RG                     203
DW                  27.733 usec
DE                   6.50 usec
TE                  293.5 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.70 usec
PL1                   0.00 dB
PL1W            29.38907051 W
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                   1.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
PL2W            9.17820644 W
PL12W           0.23054613 W
PL13W           0.23054613 W
SFO2            300.1312005 MHz
SI                   32768
SF              75.4677475 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

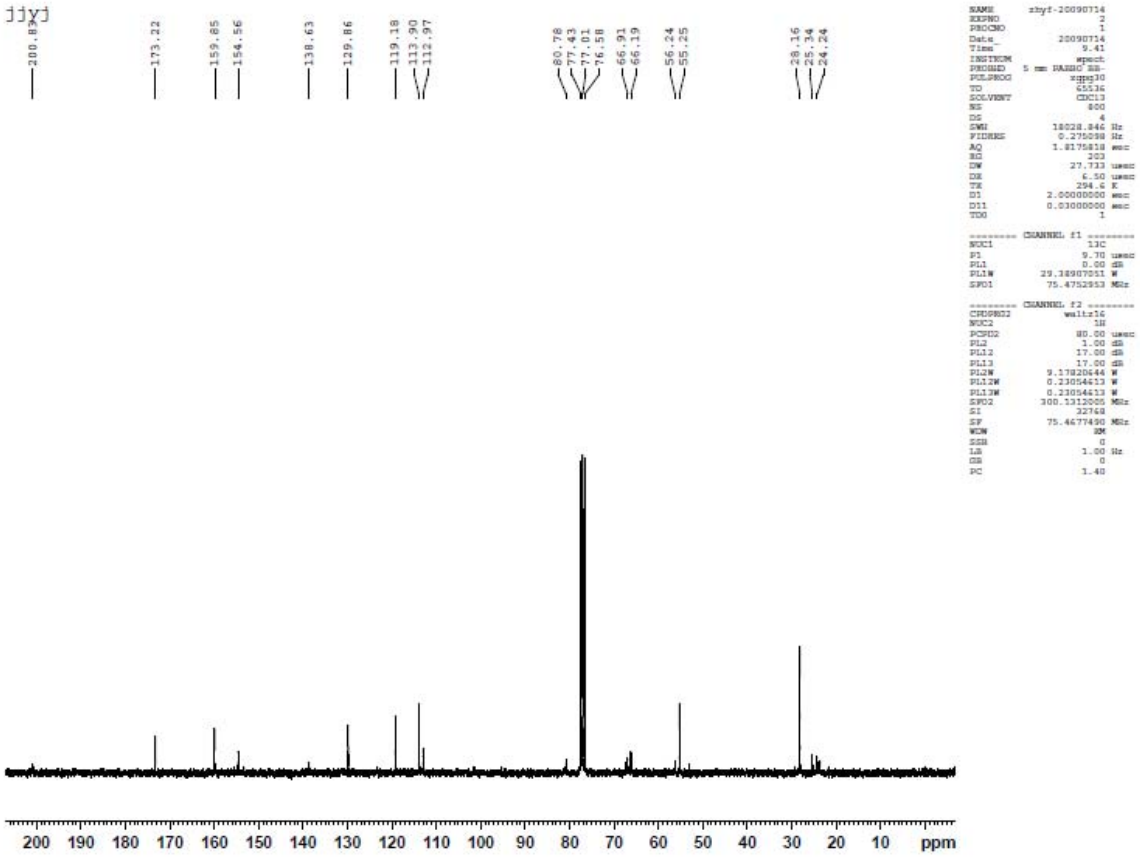
5e: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(3-methoxyphenyl)methylcarba mate



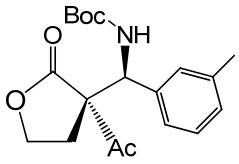


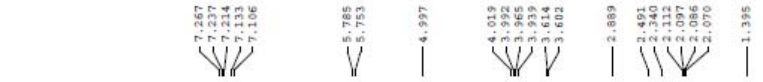


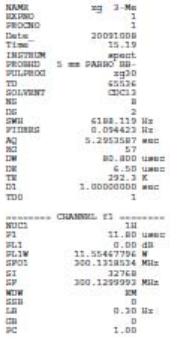


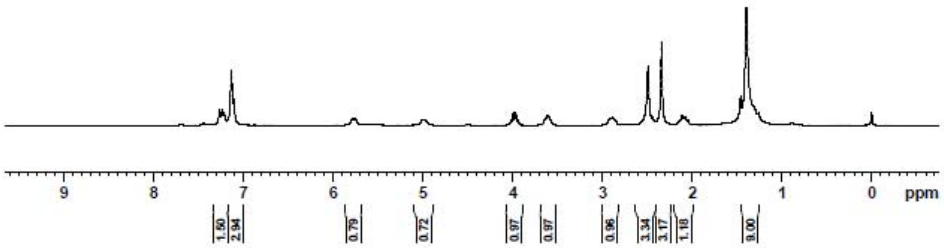

jjyj
200.83
173.22
159.85
154.56
138.63
129.86
119.18
113.90
112.97
80.78
77.43
77.01
76.58
66.91
66.19
56.24
55.25
28.16
25.94
24.24
NAME zhyf-20090714
EXPNO 2
PROCNO 1
Date_ 20090714
Time 9.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.18907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

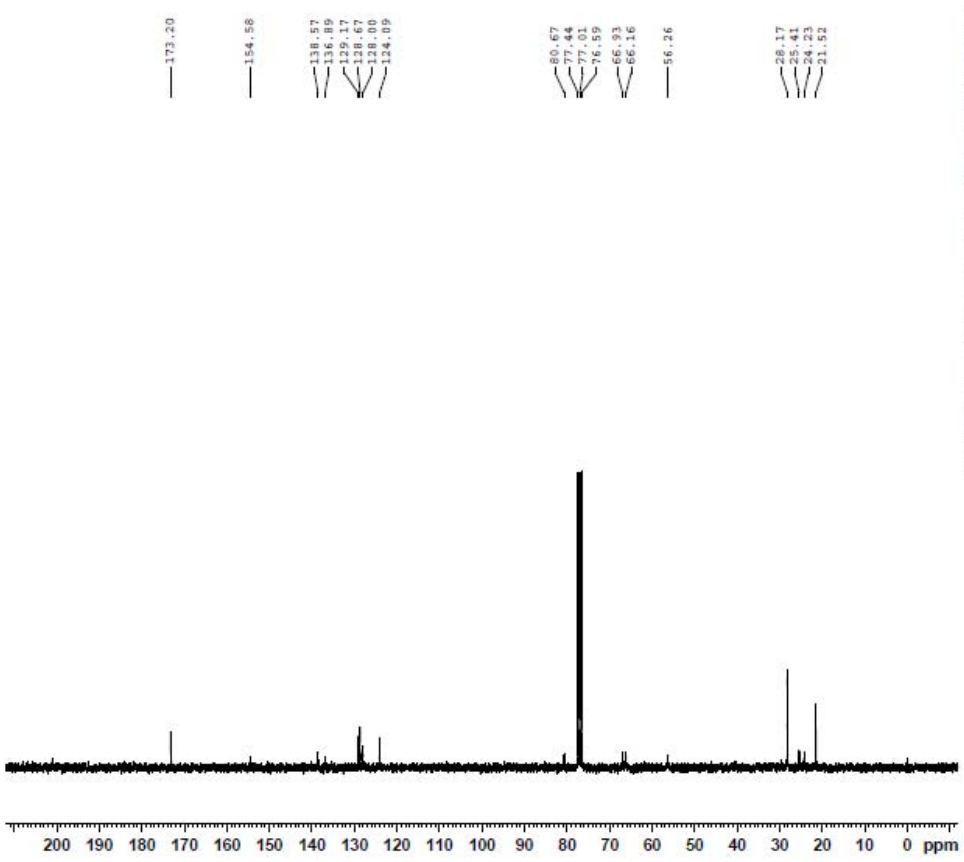
5f: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(m-tolyl)methylcarbamate



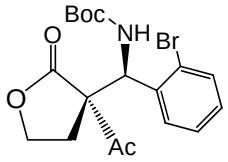


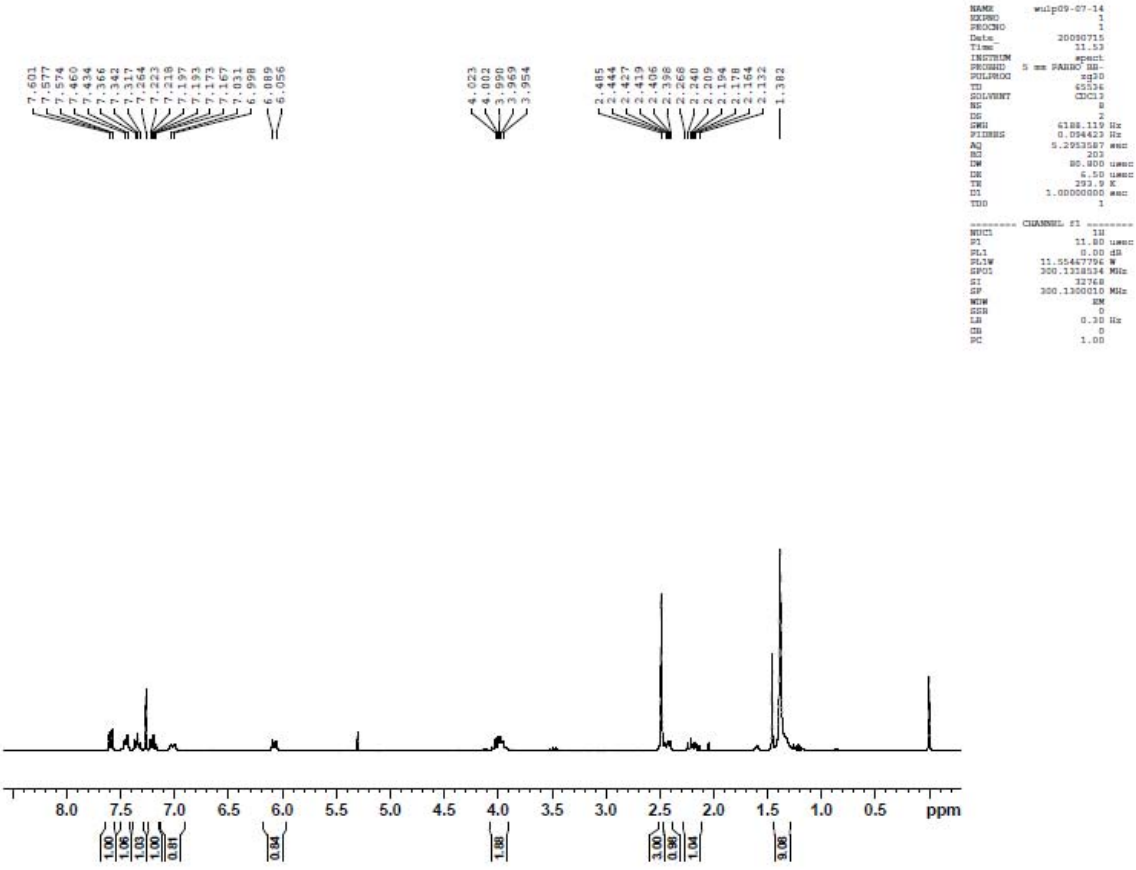


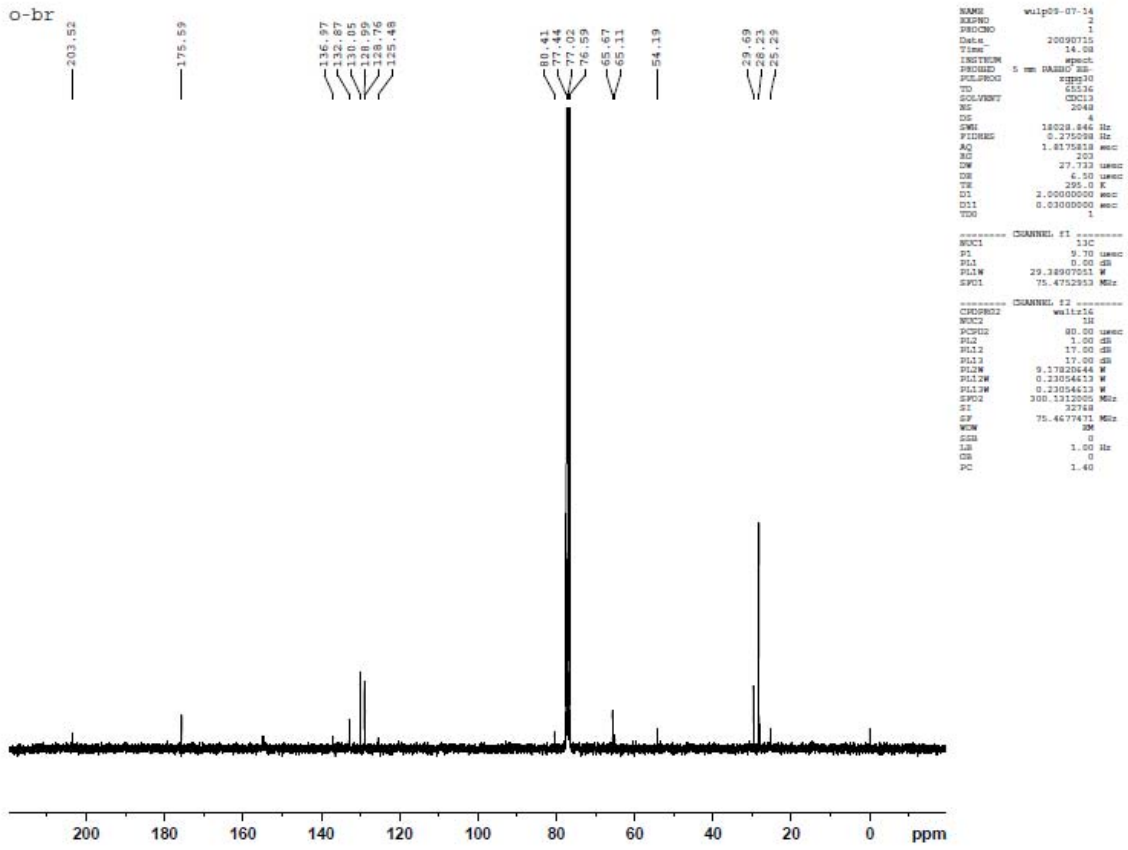



173.20
154.58
138.57
136.89
129.17
128.67
128.00
124.09
80.67
77.44
77.01
76.59
66.93
66.16
56.26
28.17
20.41
20.23
21.52
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

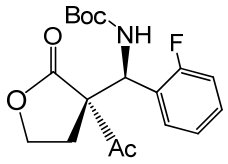
5g: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(2-bromophenyl)methylcarbamate

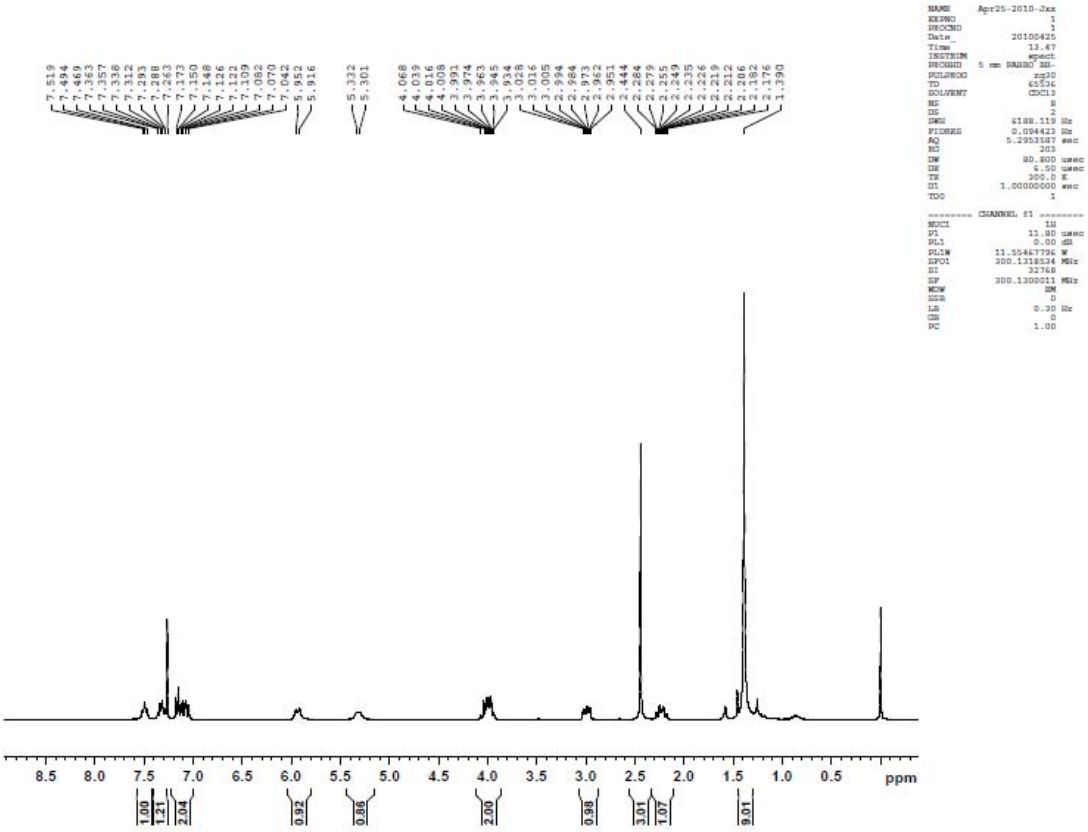


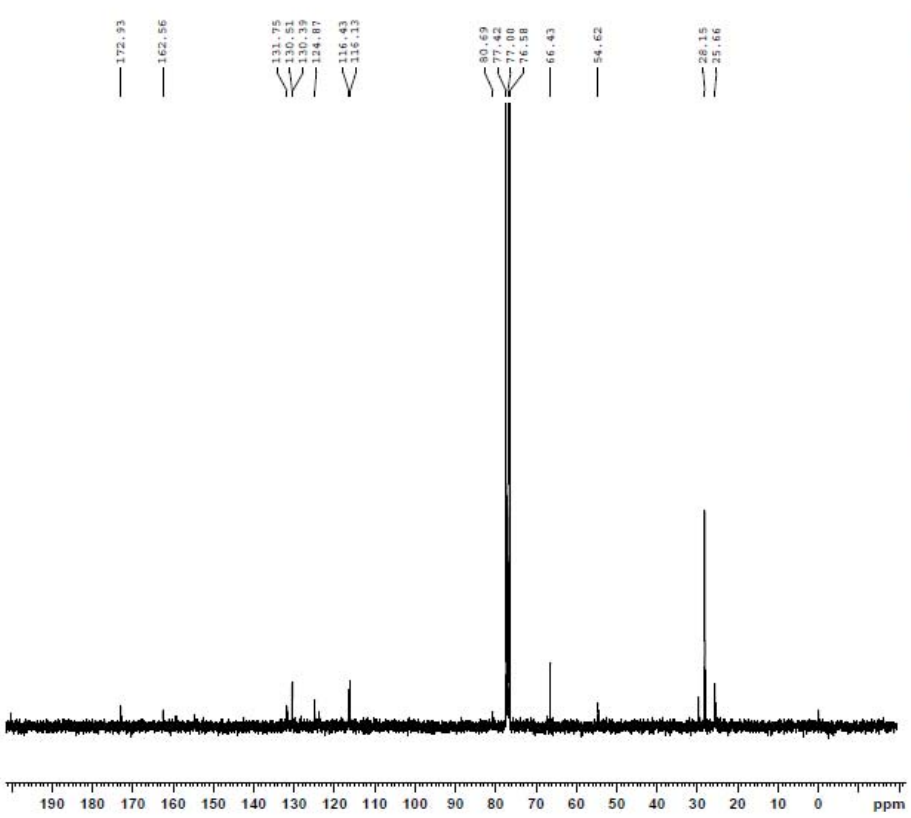



o-br
203.52
175.59
136.97
132.87
130.05
128.99
128.76
125.48
80.41
77.44
77.02
76.59
65.67
65.11
54.19
29.69
28.23
25.29
200
180
160
140
120
100
80
60
40
20
0
ppm

5h: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(2-fluorophenyl)methylcarbamate

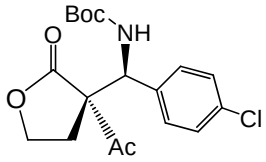


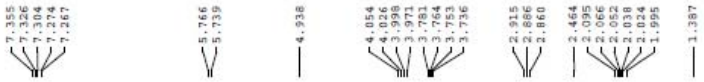


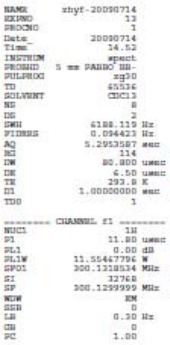


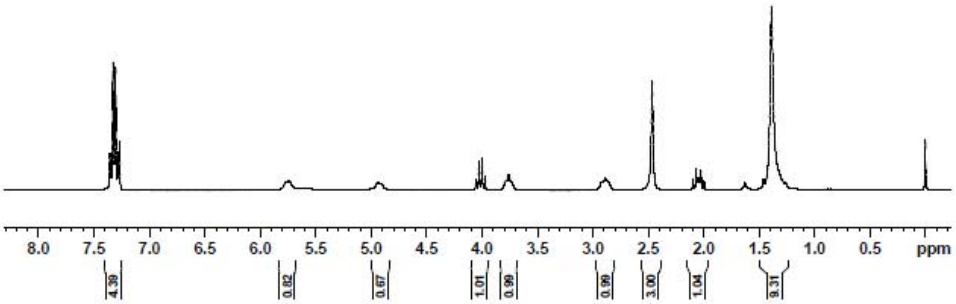

```
NAME        Apr25-2010-Jxx
EXPNO                   5
PROCNO                  1
Date_            20100425
Time                15.55
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                  65536
SOLVENT               CDCl3
NS                      672
DS                        4
SWH              18028.846 Hz
FIDRES           0.275098 Hz
AQ               1.8175818 sec
RG                      203
DW                 27.733 usec
DE                  6.50 usec
TE                 300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.70 usec
PL1                   0.00 dB
PL1W            29.38507051 W
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                   1.00 dB
PL12                 17.00 dB
PL13                 17.00 dB
PL2W            9.17820644 W
PL12W           0.23054613 W
PL13W           0.23054613 W
SFO2            300.1312005 MHz
SI                    32768
SF              75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

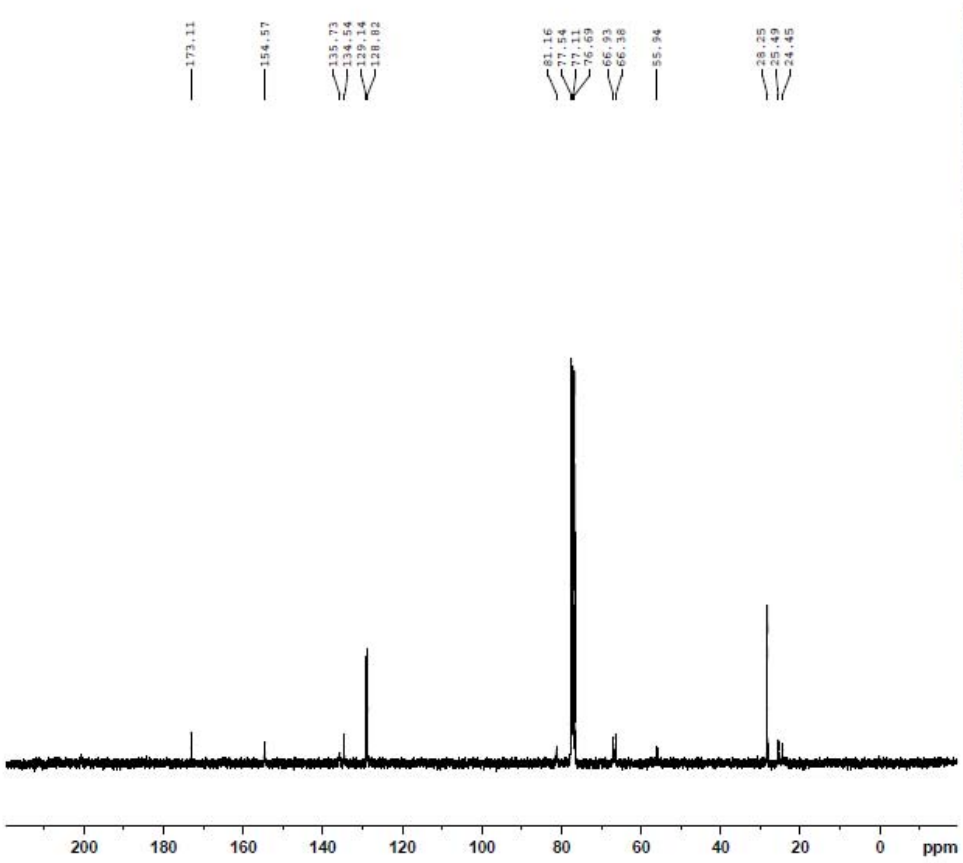
5i: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(4-chlorophenyl)methylcarbamate





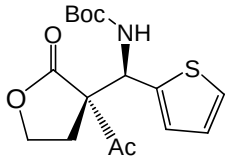


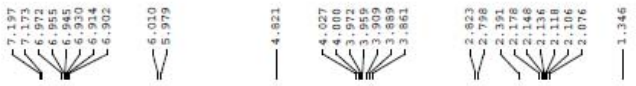


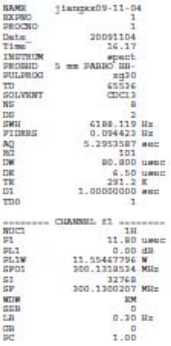


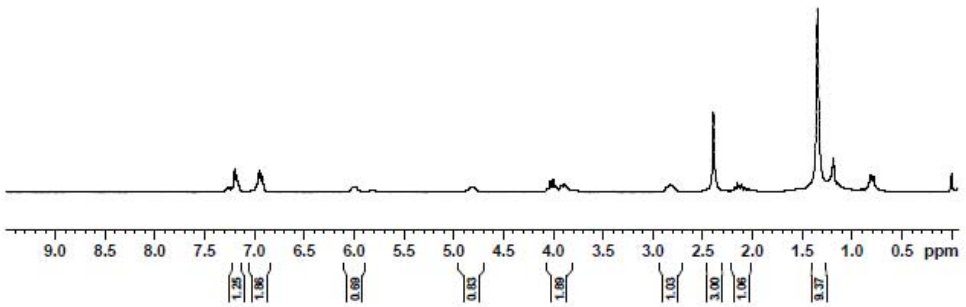

```
NAME            zhyf-20090714
EXPNO                      23
PROCNO                      1
Date_            20090714
Time                   17.50
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1921
DS                          4
SWH              18028.846 Hz
FIDRES               0.275098 Hz
AQ                1.8175818 sec
RG                        203
DW                   27.733 usec
DE                    6.50 usec
TE                   294.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                       9.70 usec
PL1                      0.00 dB
PL1W            39.38907051 W
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
PL2W             9.17820644 W
PL12W            0.23054613 W
PL13W            0.23054613 W
SFO2            300.1312005 MHz
SI                       32768
SF              75.4677480 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

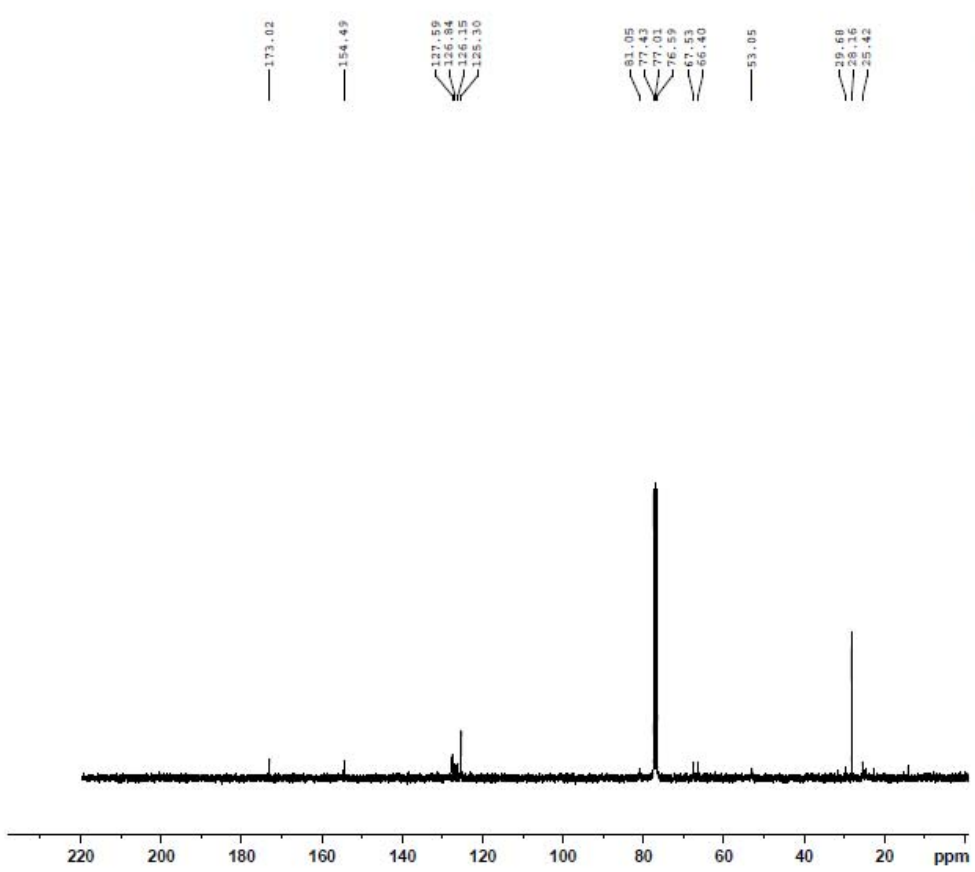
5j: *tert*-butyl (*R*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(thiophen-2-yl)methylcarbamate





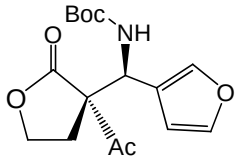


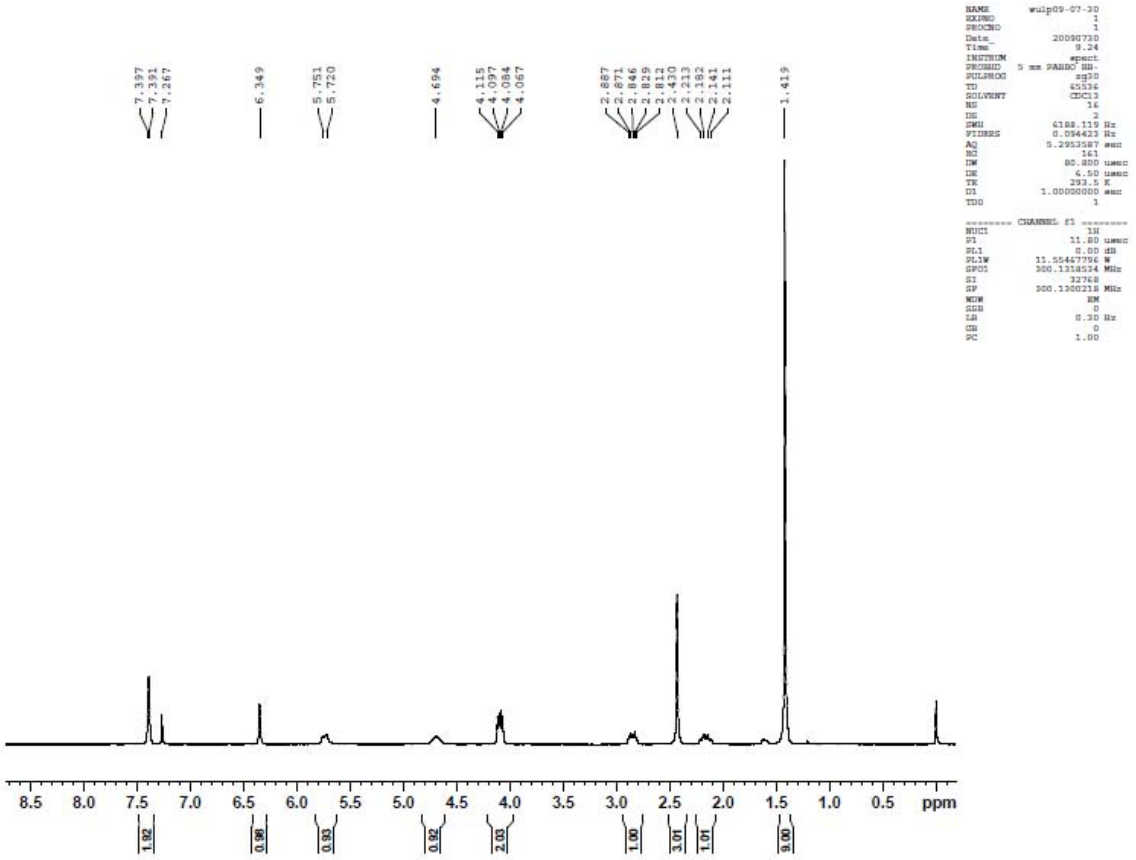


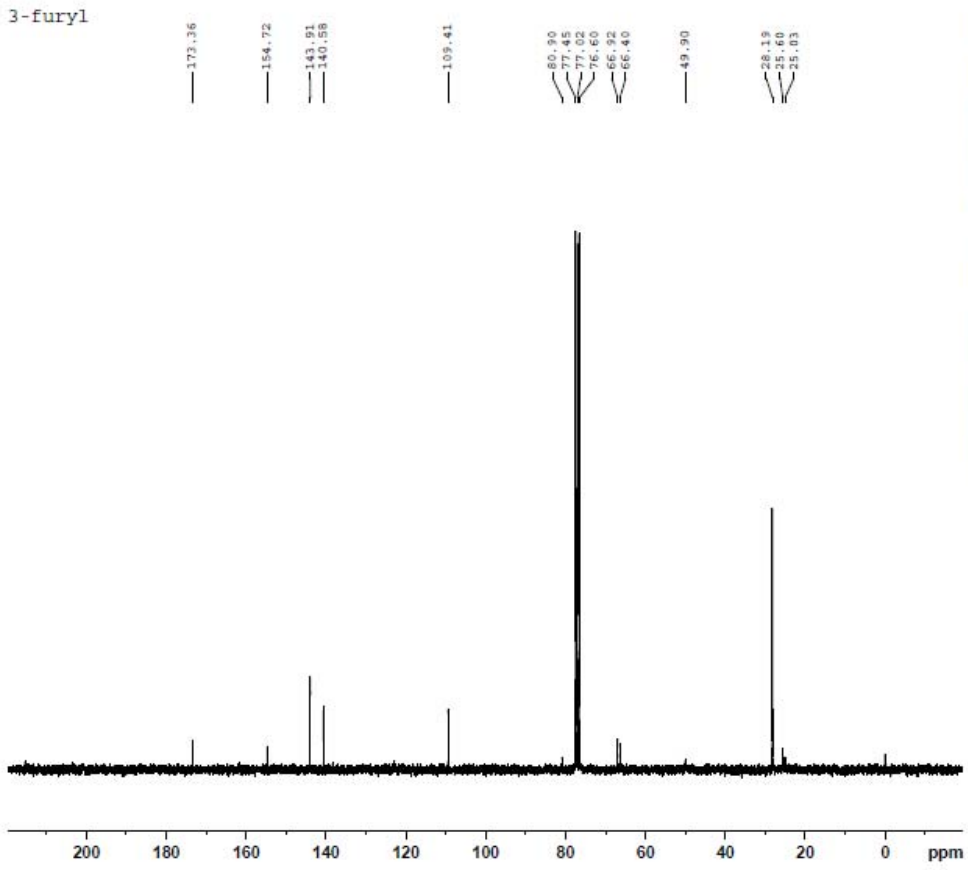


NAME	jiangxs09-11-04
EXPNO	3
PROCNO	1
Date_	20091104
Time	16.37
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	629
DS	4
SWH	18028.846 Hz
FIDRES	0.275098 Hz
AQ	1.8175818 sec
RG	203
DW	27.733 usec
DE	6.50 usec
TE	292.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.70 usec
PL1	0.00 dB
PL1W	29.38907051 W
SFO1	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	17.00 dB
PL13	17.00 dB
PL2W	9.17820644 W
PL12W	0.23054613 W
PL13W	0.23054613 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

5k: *tert*-butyl (*S*)-((*R*)-3-acetyl-2-oxotetrahydrofuran-3-yl)(furan-3-yl)methylcarbamate

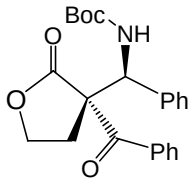


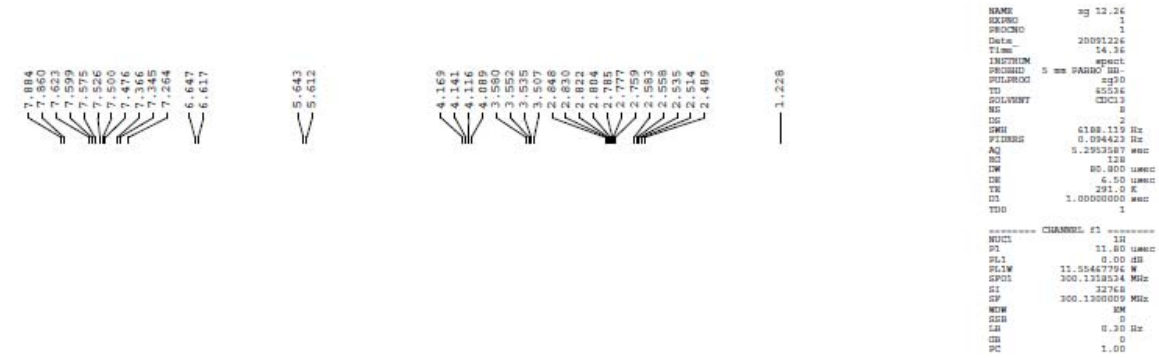


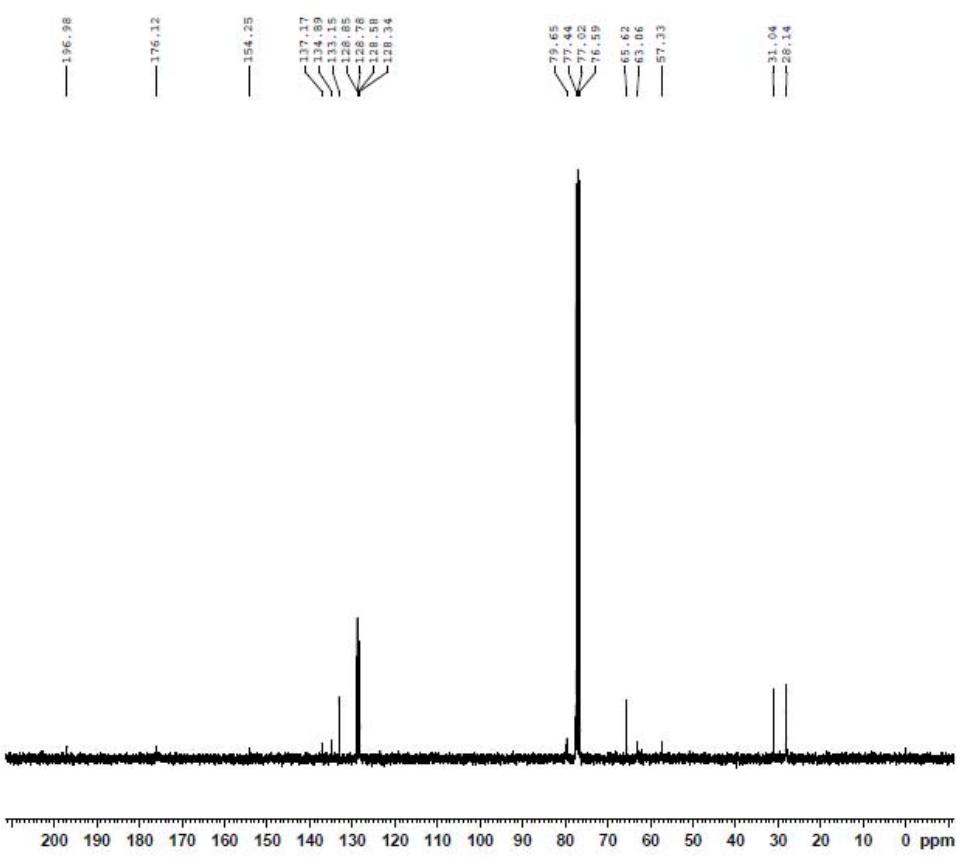


NAME wulp09-07-30
EXPNO 2
PROCNO 1
Date_ 20090730
Time 9.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175819 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 294.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38907051 W
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL1W 9.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677528 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

6a: *tert*-butyl (*S*)-((*R*)-3-benzoyl-2-oxotetrahydrofuran-3-yl)(phenyl)methylcarbamate

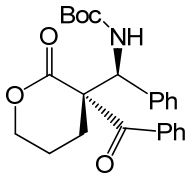


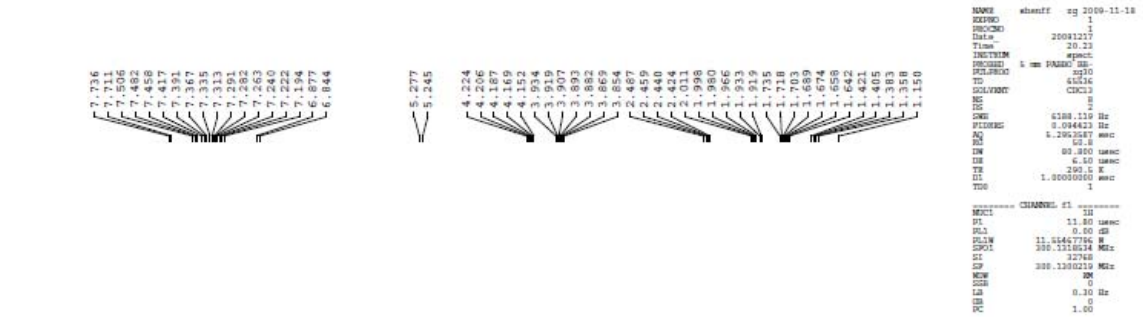


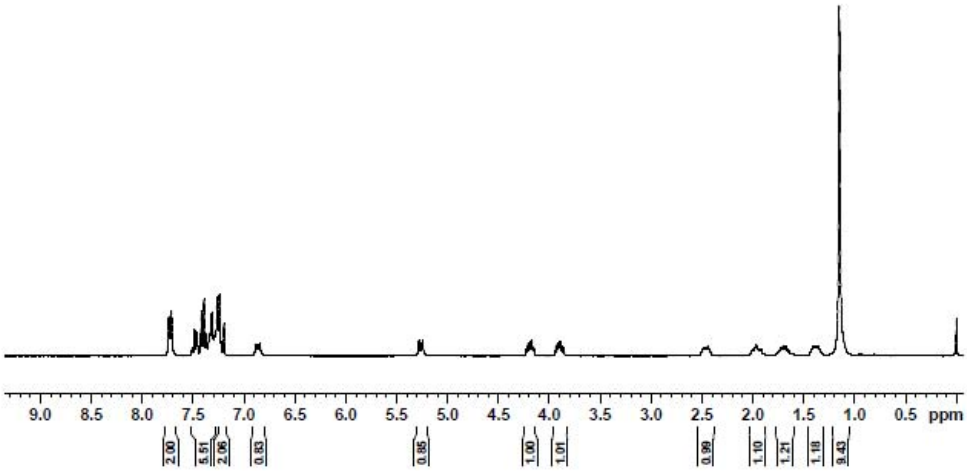


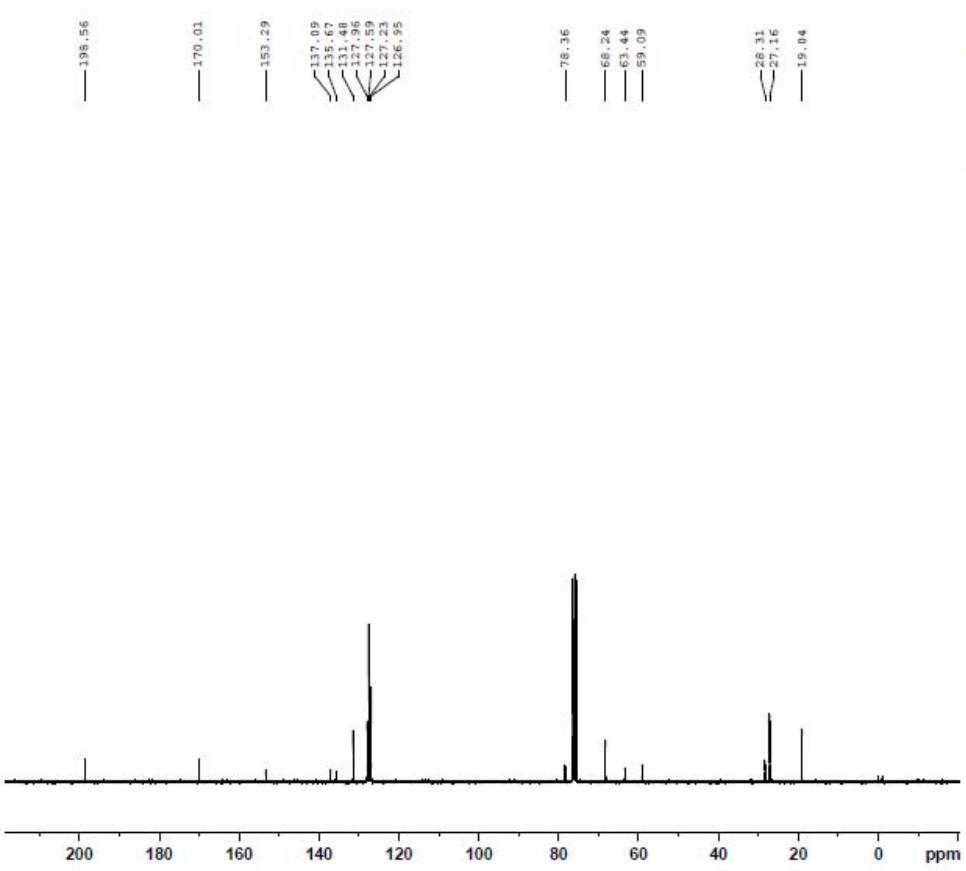

```
NAME                 zg 12.26
EXPNO                       5
PROCNO                      1
Date_              20091226
Time                   14.56
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG              zgpg30
TD                        65536
SOLVENT                 CDCl3
NS                          748
DS                            4
SWH                18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.817581 sec
RG                          203
DW                     27.733 usec
DE                      6.50 usec
TE                     295.4 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.70 usec
PL1                       0.00 dB
PL1W               29.38907051 W
SFO1               75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                       1.00 dB
PL12                     17.00 dB
PL13                     17.00 dB
PL2W               9.17820644 W
PL12W              0.23054613 W
PL13W              0.23054613 W
SFO2               300.1312005 MHz
SI                        32768
SF                 75.4677490 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

7a: *tert*-butyl (*S*)-((*R*)-3-benzoyl-2-oxotetrahydro-2H-pyran-3-yl)(phenyl)methylcarbamate



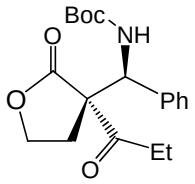


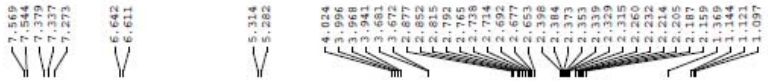


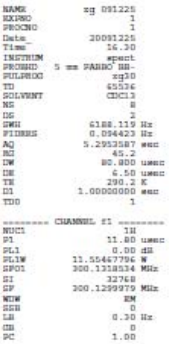


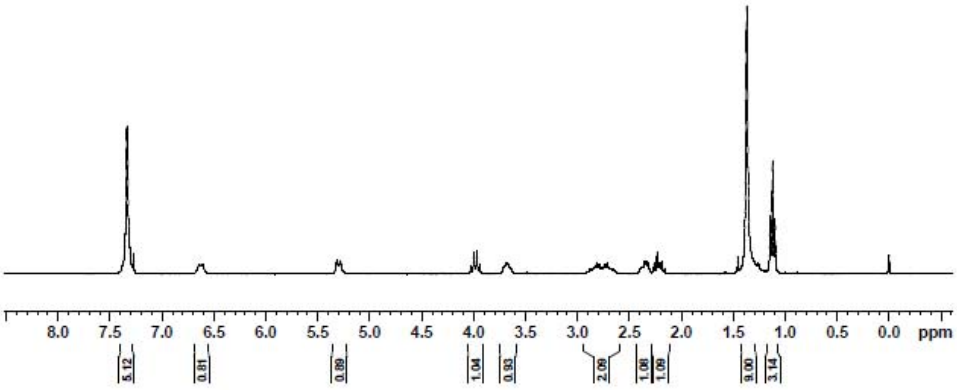

```
NAME                xg 12-17
EXPNO                      1
PROCNO                     1
Date_               20091217
Time_                  22.29
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG             xgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                 18028.846 Hz
FIDRES              0.275058 Hz
AQ                  1.8175859 sec
RG                        203
DW                     27.733 usec
DE                      6.50 usec
TE                     295.6 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                       9.70 usec
PL1                      0.00 dB
PL1W                29.38907051 W
SFO1                75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
PL2W                9.17820644 W
PL12W               0.23054613 W
PL13W               0.23054613 W
SFO2                300.1312005 MHz
SI                        32768
SW                  75.4678258 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

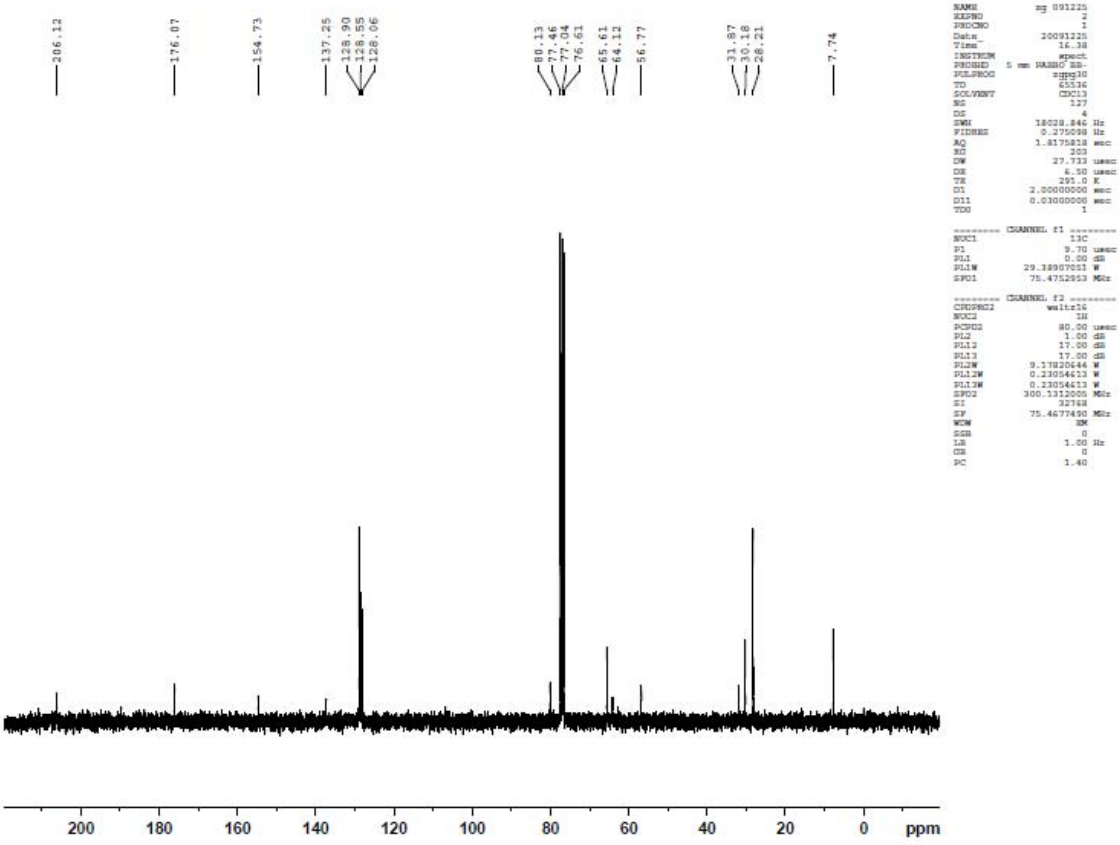
8a: *tert*-butyl (*S*)-((*R*)-2-oxo-3-propionyltetrahydrofuran-3-yl)(phenyl)methylcarbamate












```
NAME                 mg 091225
EXPNO                       2
PROCNO                      1
Date_             20091225
Time                   16.38
INSTRUM                  spect
PROBHD     5 mm HABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         127
DS                           4
SWH                18028.846 Hz
FIDRES                0.275098 Hz
AQ                    1.8175818 sec
RG                         203
DW                      27.733 usec
DE                       6.50 usec
TE                     291.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                       9.70 usec
PL1                      0.00 dB
PL1W               29.38907051 W
SFO1               75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
PL2W                9.17820644 W
PL12W               0.23054613 W
PL13W               0.23054613 W
SFO2               300.1312005 MHz
SI                        32768
SF                 75.4677490 MHz
WDW                         EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                       1.40
```