Highly Efficient Asymmetric *anti*-Mannich Reactions of Carbonyl Compounds with *N*-Carbamoyl Imines Catalyzed by Amino-thiourea Organocatalysts †

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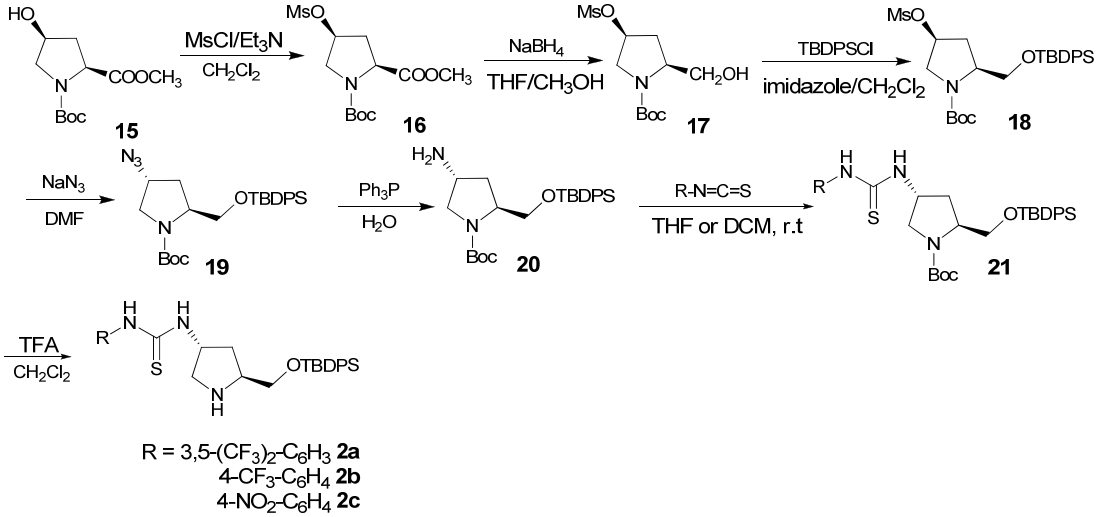
Supporting Information

1. General methods

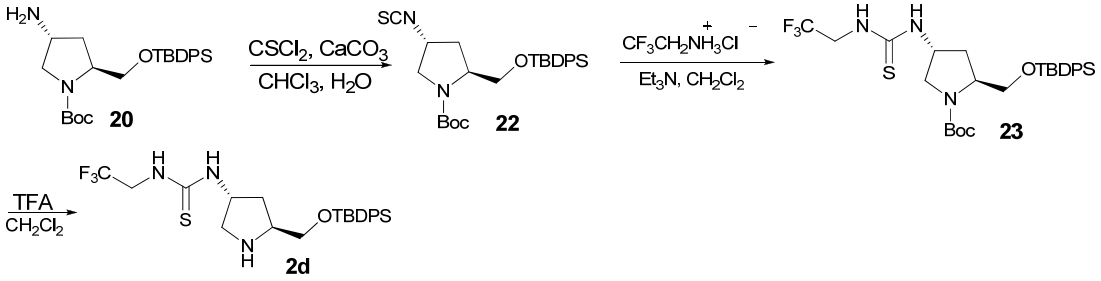
All solvents were purified by standard procedures and distilled prior to use. Reagents obtained from commercial source were used without further purification. Petroleum ether and ethyl acetate for flash column chromatography were distilled before use. All reactions were monitored by TLC with silica gel coated plates. Flash column chromatography was performed on silica gel H (10-40 μ). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance 300 MHz spectrometer. Chemical shifts are reported in ppm from tetramethyl silane (TMS) with the solvent resonance as the internal standard. Proton signal multiplicities are given as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad) or a combination of them. *J*-values are in Hz. Melting points were determined on an X-6 digital melting-point apparatus and were uncorrected. Optical rotations were measured on a Perkin Elmer 341 Polarimeter at λ = 589 nm. Analytical high performance liquid chromatography (HPLC) was carried out on WATERS 510 instrument (2487 Dual λ Absorbance Detector and 515 HPLC Pump) using chiral column. ChiralPak columns were purchased from Daicel Chemical Industries, LTD. ESI HRMS was recorded on a Bruke P-SIMS-Gly FT-ICR mass spectrometer.

2. Synthesis of chiral catalysts

The synthesis of catalysts **2a** from (L)-4-hydroxyproline has been reported, the detail of synthetic procedure see the reference,[1] the route as follows:



Synthesis of catalyst 2d



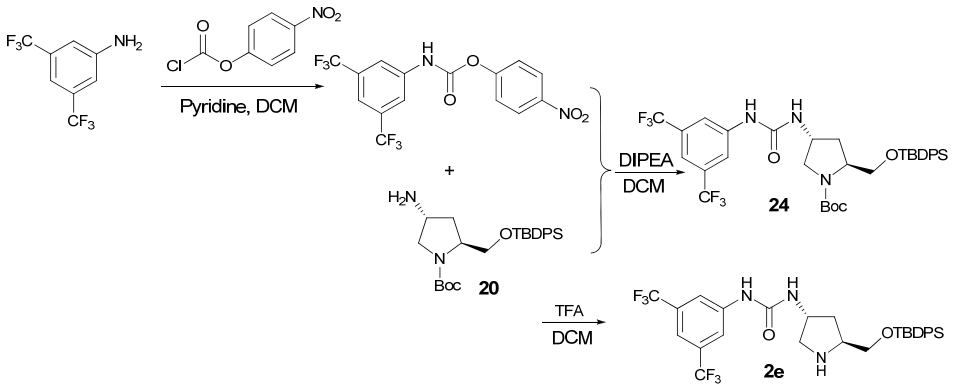
Thiophosgene (1.7 mL, 22 mmol) was added dropwise to the mixture of **20** (4.9 g, 10.8 mmol), $CaCO_3$ (1.41 g, 14.1 mmol) and H_2O (6 mL) in $CHCl_3$ (60 mL) at 0°C. The mixture was stirred overnight. After filtration, the organic phase was washed with brine and dried over anhydrous Na_2SO_4, concentrated under reduced pressure to afford crude product **21**. Purification by silica gel column chromatography gave pure compound **22** as colorless oil (4.75 g, yield 88%).

Et_3N (0.30 mL, 2.2 mmol) was added to the solution of 2,2,2-trifluoroethanaminium chloride (0.3 g, 2 mmol) in fresh distill CH_2Cl_2 (10 mL) under ice bath, then, **22** (0.9 g, 2 mmol) was added to the mixture and warmed to 30°C, stirred further for 5 h, dilute with CH_2Cl_2(30 mL), washed with saturated NH_4Cl and brine, dried over anhydrous Na_2SO_4, concentrated under reduced pressure to afford crude product **23**. Purification by silica gel column chromatography gave pure compound **23** (0.5 g, yield 51%).

23 (0.5 g, 1.0 mmol) was added to the mixture of trifluoroacetic acid and CH_2Cl_2 (6 mL, V/V = 1:5) at 0°C and the solution was stirred for 6 h, concentrated and removed the excess trifluoroacetic acid, diluted

with water and adjusted the pH of the solution to 8 with aqueous ammonia. Extracted with dichloromethane three times and combined organic layers, dried over anhydrous Na_2SO_4. Concentrated and purified by flash column chromatography to give thiourea catalyst **2d** (0.37 g, yield 70%).

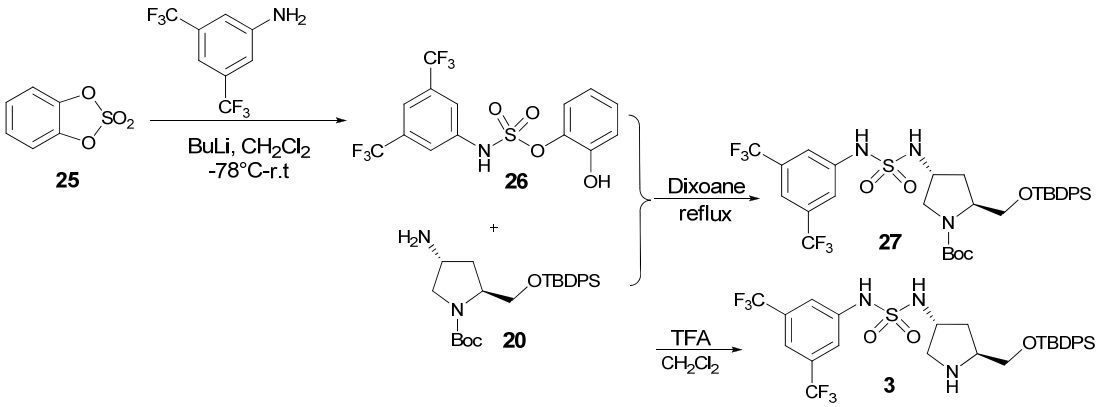
Synthesis of catalyst 2e



4-nitrophenyl carbonochloridate (0.9 g, 4.5 mmol) was added to the mixture of 3,5-bis(trifluoromethyl)aniline (0.7 mL, 4.5 mmol) and pyridine (0.4 mL, 5.0 mmol) in dry CH_2Cl_2 (20 mL) at room temperature, stirred for 5 minute, then, **20** (1.4 g, 3 mmol) in 10 mL CH_2Cl_2 and DIPEA (0.6 mL, 0.43 mmol) were added to the mixture successively. The resulting mixture was stirred for 5 hours further. Extracted with CH_2Cl_2 three times and combined organic layers, washed with saturated $NaHCO_3$ and brine, dried over anhydrous Na_2SO_4. Concentrated and purified by flash column chromatography to give the product **24** (1.9 g, yield 92%).

24 (0.62 g, 0.87 mmol) was added to the mixture of trifluoroacetic acid and CH_2Cl_2 (5mL, V/V = 1:4) at 0°C and the solution was stirred for 6 h. The mixture was concentrated to remove the excess trifluoroacetic acid, diluted with water and adjusted the pH of the solution to 8 with aqueous ammonia. Extracted with dichloromethane three times and combined organic layers, dried over anhydrous Na_2SO_4. Concentrated and purified by flash column chromatography to give urea catalyst **2e** (0.3 g, yield 71%).

Synthesis of catalyst 3



The details of synthesis of catechol sulfate **25** see the reference.[2]

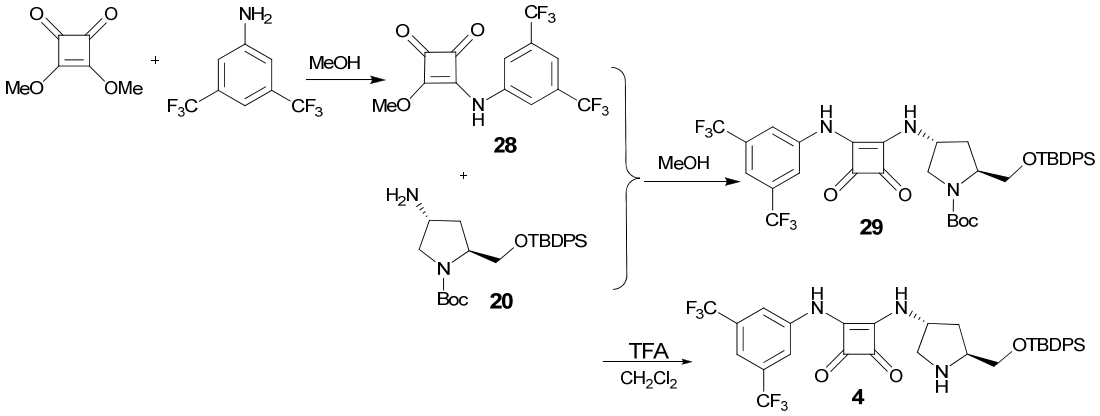
n-BuLi (1 mL, 2.4 mmol) was added dropwise to the solution of 3, 5-bis(trifluoromethyl) aniline (0.46 g, 2 mmol) in CH_2Cl_2 (10 mL) under -78 °C, stirred for 20 minute, then, the solution of catechol sulfate **25** (0.2 g, 1 mmol) in CH_2Cl_2 (5 mL) was added to this mixture, warmed to room temperature, monitored by TLC, stirred until the catechol sulfate disappeared. The reaction was quenched with saturated NH_4Cl, extracted with ethyl acetate three times and combined organic layers, washed with brine, and dried over anhydrous Na_2SO_4. Concentrated and purified by flash column chromatography to give **26** (0.2 g, yield 43%).

20 (1.6 g, 3.5 mmol) was added to the solution of **26** (1.3 g, 3.5 mmol) in dioxane (20 mL) and refluxed for 24 hours, removed the dioxane under reduce pressure, diluted with 50 mL ethyl acetate, washed with brine, dried over anhydrous Na_2SO_4. Concentrated and purified by flash column chromatography to give **27** (1.6 g, yield 70%).

27 (0.7 g, 0.97 mmol) was added to the mixture of trifluoroacetic acid and CH_2Cl_2 (5mL, V/V = 1:4) at 0°C and the solution was stirred for 6 h, concentrated to remove the excess trifluoroacetic acid, diluted with

water and adjusted the pH of the solution to 8 with aqueous ammonia. Extracted with dichloromethane three times and combined organic layers, dried over anhydrous Na_2SO_4. Concentrated and purified by flash column chromatography to give the catalyst **3** (0.33 g, yield 65%).

Synthesis of catalyst 4



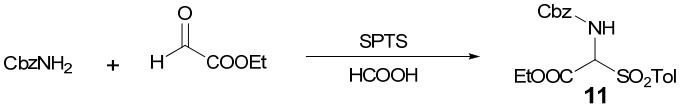
3,4-Dimethoxy-3-cyclobutene-1,2-dione (0.2 g, 1.7 mmol) was added to the solution of 3,5-bis(trifluoromethyl)aniline (0.3 mL, 1.7 mmol) in 2 mL MeOH and stirred for 2 days at room temperature. The resulting pale yellow solid was isolated by filtration to give **28** (0.4 g, 67% yield).

28 (0.4 g, 1.1 mmol) was added to the solution of **20** (0.5 g, 1.14 mmol) in MeOH (3 mL) and stirred at room temperature, monitored by TLC until the reaction finished, removed the MeOH under reduce pressure, purified by flash column chromatography to give **29** (0.4 g, 50% yield).

29 (0.3 g, 0.3 mmol) was added to the mixture of trifluoroacetic acid and CH_2Cl_2 (3mL, V/V = 1:4) at 0 °C and the solution was stirred for 6 h. The reaction mixture was concentrated to remove the excess trifluoroacetic acid, diluted with water and adjusted the pH of the solution to 8 with aqueous ammonia. Extracted with dichloromethane three times and combined organic layers, dried over anhydrous Na_2SO_4. Concentrated and purified by flash column chromatography to give the squaramide catalyst **4** (0.24 g, 85% yield).

All the *N*-Boc protected *α*-amido sulfones and *N*-Boc protected imines derived from aromatic aldehyde were prepared by the method reported by Jacobsen.[3] All the *N*-Cbz protected *α*-amido sulfones and *N*-Cbz protected imines were prepared by the method reported by Dixon.[4]

3. Synthesis of *N*-Cbz *α*-amido sulfone 11 derived from ethyl glyoxalate:



Benzyl carbamate (3.0 g, 20 mmol) was added to the mixture of sodium *p*-tolylsulfinate monohydrate (SPTS) (11.8 g, 60 mmol) and ethyl glyoxalate (4.0 mL, 40 mmol) in formic acid (99%, 20 mL). The solution was stirred for two days at room temperature, then poured the mixture into ice water, white solid was precipitated and isolated by filtration, washed with water and ether, dried under reduce pressure to obtain the *N*-Cbz *α*-amido sulfone **11** (6.6 g, 85% yield).

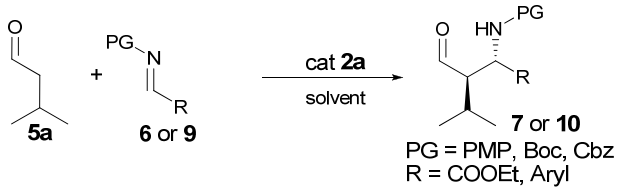
4. Determination of diastereomeric ratios and enantiomeric purity:

The diastereomeric ratios were determined by integration of one set of ^{1}H NMR signal (C<u>H</u>O) or by chiral HPLC. Chiral HPLC analysis was performed on an WATERS 510 instrument (2487 Dual λ Absorbance Detector and 515 HPLC Pump) using chiral column, ChiralPak columns purchased from Daicel Chemical Industries, Daicel ChiralPak AD-H or AS-H column with *i*-PrOH/hexane or ethanol/hexane as the eluent was used. HPLC traces were compared to the retention time of the racemic samples prepared by carrying out the reactions with pyrolidine add HOAc as the catalyst.

The absolute configuration of the obtained *anti*-Mannich products have been confirmed by Maruoka group.[5] The absolute configuration of the *anti*-isomer obtained in the reaction between isovaleraldehyde and *N*-Boc-imine **14r** was determined to be (***1S, 2R***) by comparison of the HPLC retention times with the literature data.[5] HPLC analysis: Daicel ChiralPak AS-H, hexane/*i*-PrOH = 100/1, flow rate = 1 mL/min, λ = 205 nm, major product: t_{major} = 12.15 min, t_{minor} = not fond, *anti*: *ee* >99%, *dr* 90/10. (Reference: Daicel

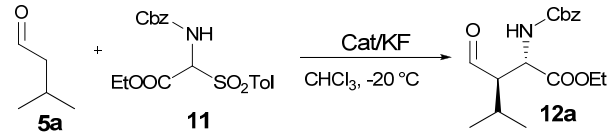
ChiralPak AS-H, hexane/i-PrOH = 100/1, flow rate = 1 mL/min, λ = 205 nm, retention time; t_{major} = 12.2 min, t_{minor} =10.4 min).[5]

5. General procedure for the *anti*-selective Mannich reaction of imine and aldehyde



N-PMP imine **6** (or *N*-Boc imine **9**) (0.2 mmol, 1 equiv.) and catalyst **2a** (0.01 mmol, 0.05 equiv.) were dissolved in anhydrous 1, 2-dichloroethane (or $CHCl_3$) (1 mL), subsequently, isovaleraldehyde (1.0 mmol, 5 equiv.) was added at designated temperature. The mixture was stirred and monitored by TLC until the imine was completely disappearance. The mixture was worked up by addition of aqueous saturated ammonium chloride solution and extracted with AcOEt (three times). The combined organic layers were washed with brine, dried over Na_2SO_4, concentrated in *vacuo* and the residue was purified by flash column chromatography (5-10% AcOEt/PE) to afford the corresponding *anti*-Mannich product **7** or **10**. The *ee* and *dr* of *anti*-Mannich product **7** were determined by a chiral phase ChiralPak AS-H column (hexane / *i*-PrOH = 90/10, 254 nm, 0.5 mL / min, *t* (major) = 15.40 min, *t* (minor) = 25.90 min. *ee* > 99%**,** *dr* = 98/2;). The *ee* and *dr* of *anti*-Mannich product **10** were determined by a chiral phase ChiralPak AS-H column (96/4 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 20.9 min, t_{minor} = 18.6 min, *anti*: *ee* >99%, *dr* = 95/5)

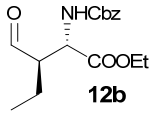
6. General procedure for the *anti*-selective Mannich reaction of *α*-amido sulfone and aldehyde



To the mixture of *α*-amido sulfone **11** (0.2 mmol, 1 equiv.), KF (1 mmol, 5 equiv.), catalyst **2a** (0.01 mmol, 0.05 equiv.) in 1 mL $CHCl_3$, isovaleraldehyde (1.0 mmol, 5 equiv.) was added under -20°C, the mixture stirred for additional 11 hours, quenched with saturated NH_4Cl, extract with CH_2Cl_2 (3×10 mL), combined organic phase and washed with brine, dried over anhydrous Na_2SO_4. Concentrated and purified by flash column chromatography to give the *anti*-selective Mannich product in 90% yield (58 mg). The *ee* and *dr* were determined by a chiral phase Daicel ChiralPak AD-H column: 90/10 hexane/*i*PrOH, flow rate 0.7 mL/min, λ = 214 nm, 254 nm, t_{major} = 18.61 min, t_{minor} = 27.35 min, *anti*: *ee* >99%, *dr* >99/1.

7. Characterization of the Mannich reaction products:

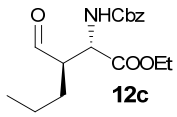
(2*S*, 3*R*)-ethyl 2-(benzyloxycarbonylamino)-3-formylpentanoate[7]



12b

The title compound was isolated as colorless oil in 85% yield. HPLC analysis on a Daicel ChiralPak AD-H column: 90/10 hexane/iPrOH, flow rate 0.75 mL/min, λ = 254 nm, t_{major} = 11.35 min, t_{minor} = 16.82 min, *anti*: *ee* = 97%, *dr* = 80/20; $[\alpha]_D^{20}$ = + 60.7 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.62 (s, 1H), 7.35 (s, 5H), 5.55 (d, *J* = 8.9 Hz, 1H), 5.13 (d, *J* = 3.6 Hz, 2H), 4.66 (d, *J* = 8.9 Hz, 1H), 4.18 (q, *J* = 6.9 Hz,2H), 3.04 (s, 1H), 1.82 (dd, *J* = 13.9, 6.9 Hz, 1H), 1.53 (dd, *J* = 14.4, 7.2 Hz, 1H), 1.24 (t, *J* = 7.0 Hz, 3H), 1.12 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.45, 170.91, 156.57, 136.21, 128.37, 128.00, 67.16, 61.92, 53.49, 52.54, 27.12, 20.64, 13.93 ppm.

(2*S*, 3*R*)-ethyl 2-(benzyloxycarbonylamino)-3-formylhexanoate

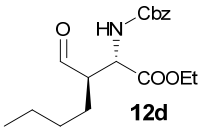


12c

The title compound was isolated as colorless oil in 83% yield. HPLC analysis on a Daicel ChiralPak AD-H column: 90/10 hexane/*i*PrOH, flow rate 0.7mL/min, λ = 254 nm, t_{major} = 19.02 min, t_{minor} = 29.91 min, *anti*: *ee* = 98%, *dr* = 85/15; $[\alpha]_D^{20}$ = + 64.8 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s,

1H), 7.43 - 7.28 (m, 5H), 5.14 (s, 2H), 4.63 (dd, *J* = 9.5, 3.5 Hz, 1H), 4.21 - 4.14 (m, 2H), 3.18 - 3.10 (m, 1H), 1.78 - 1.66 (m, 1H), 1.52 - 1.41 (m, 3H), 1.28 - 1.23 (m, 3H), 0.96 (t, *J* = 6.7 Hz, 3H)ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.55, 170.94, 156.57, 136.17, 128.33, 127.93, 67.16, 61.92, 53.45, 52.50, 27.18, 20.64, 14.05, 13.93 ppm.

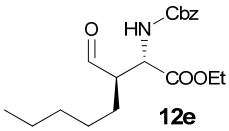
(2*S*, 3*R*)-ethyl 2-(benzyloxycarbonylamino)-3-formylheptanoate[7]



12d

The title compound was isolated as colorless oil in 76% yield. HPLC analysis on a Daicel ChiralPak AD-H column: 94/6 hexane/*i*PrOH, flow rate 0.5mL/min, λ = 254 nm, t_{major} = 29.81 min, t_{minor} = 46.82 min, *anti*: *ee* >99%, *dr* = 89/11; $[\alpha]_D^{20}$ = + 58.6 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H), 7.45 - 7.23 (m, 5H), 5.57 (d, *J* = 9.5 Hz, 1H), 5.21 - 5.07 (m, 2H), 4.62 (dt, *J* = 17.6, 8.8 Hz, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 3.11 (dd, *J* = 9.9, 6.3 Hz, 1H), 1.85 - 1.62 (m, 1H), 1.45 (tdd, *J* = 19.4, 14.2, 7.0 Hz, 6H), 1.24 (t, *J* = 7.1 Hz, 3H), 0.91 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.45, 170.91, 156.55, 136.23, 128.36, 128.00, 67.13, 61.90, 53.69, 52.59, 29.48, 24.75, 22.51, 14.05, 13.77 ppm.

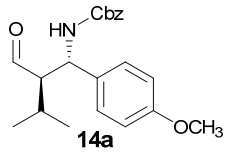
(2*S*, 3*R*)-ethyl 2-(benzyloxycarbonylamino)-3-formyloctanoate



12e

The title compound was isolated as colorless oil in 67% yield. HPLC analysis on a Daicel ChiralPak AD-H column: 95/5 hexane/*i*PrOH, flow rate 0.6mL/min, λ = 254 nm, t_{major} = 25.65 min, t_{minor} = 41.83 min, *anti*: *ee* = 97%, *dr* = 83/17; $[\alpha]_D^{20}$ = + 53.2 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (s, 1H), 7.46 - 7.25 (m, 5H), 5.56 (d, *J* = 9.4 Hz, 1H), 5.13 (s, 2H), 4.66 - 4.56 (m, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 3.21 - 2.96 (m, 1H), 1.58 - 1.39 (m, 3H), 1.26 (dd, *J* = 17.6, 10.4 Hz, 8H), 0.89 (t, *J* = 5.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.19, 170.72, 156.13, 136.17, 128.71, 128.00, 67.08, 61.86, 53.64, 52.50, 31.59, 26.77, 24.88, 22.24, 13.87, 10.73 ppm.

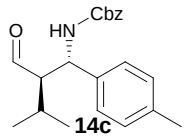
Benzyl (*1S, 2R*)-2-formyl-1-(4-methoxyphenyl)-3-methylbutylcarbamate[7b, 8]



14a

The title compound was isolated as colorless oil in 97% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 90/10 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 30.2 min, t_{minor} = 36.8 min, *anti*: *ee* >99%, *dr* = 97/3; $[\alpha]_D^{20}$ = + 5.7 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): *δ* 9.76 - 9.77 (d, *J* = 3 Hz, 1H), 7.17 - 7.20 (d, *J* = 9 Hz, 2H), 7.31 (s, 5H), 6.84 - 6.87 (d, *J* = 9 Hz, 2H), 5.66 - 5.69 (d, *J* = 9 Hz, 1H), 5.09 - 5.14 (m, 1H), 5.04 - 5.05 (m, 2H), 2.62 - 2.63(m, 1H), 3.87 (s, 3H), 1.82 - 1.87 (m, 1H), 0.99 - 1.06 (m, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): *δ* 206.14, 158.95, 155.60, 136.20, 132.44, 128.44, 128.07, 127.99, 127.69, 114.11, 66.88, 62.62, 55.22, 28.22, 21.35, 18.70 ppm.

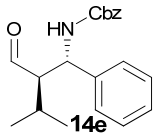
Benzyl (*1S, 2R*)-2-formyl-3-methyl-1-*p*-tolylbutylcarbamate[7b, 8]



14c

The title compound was isolated as colorless oil in 95% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 92/8 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 21.2 min, t_{minor} = not found, *anti*: *ee* >99%, *dr* = 96/4; $[\alpha]_D^{20}$ = + 14.2 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): *δ* 9.75 - 9.76 (d, *J* = 3 Hz, 1H), 7.14 - 7.30 (m, 9H), 5.72 - 5.75 (d, *J* = 9 Hz, 1H), 5.16 - 5.18 (m, 1H), 4.99 - 5.12 (m, 2H), 2.61 - 2.64 (m, 1H), 2.32 (s, 3H), 1.80 - 1.89 (m, 1H), 0.90 - 1.09 (m, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): *δ* 206.16, 155.63, 137.39, 137.33, 136.20, 129.42, 128.42, 128.04, 127.99, 126.44, 66.86, 62.55, 53.33, 28.20, 21.34, 21.00, 18.74 ppm.

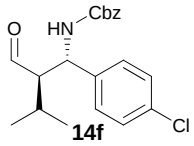
Benzyl (*1S, 2R*)-2-formyl-3-methyl-1-phenylbutylcarbamate[7b, 8]



14e

The title compound was isolated as colorless oil in 97% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 90/10 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 21.2 min, t_{minor} = not found, *anti*: *ee* >99%, *dr* = 95/5; $[\alpha]_D^{20}$ = + 24.5 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.75 - 9.76 (d, *J* = 3 Hz, 1H), 7.25 - 7.27 (m, 10H), 5.76 - 5.79 (d, *J* = 9 Hz, 1H), 5.16 - 5.21 (m, 1H), 5.01 - 5.10 (m, 2H), 2.67 - 2.68 (m, 1H), 1.84 - 1.96 (m, 1H), 0.95 - 1.08 (m, 6H) ppm ^{13}C NMR (75 MHz, $CDCl_3$): δ 206.02, 155.66, 140.53, 136.24, 136.21, 128.77, 128.46, 128.09, 127.62, 126.52, 66.94, 62.58, 53.57, 28.30, 21.33, 19.02 ppm.

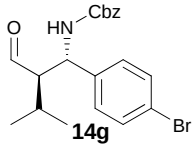
Benzyl (*1S, 2R*)-1-(4-chlorophenyl)-2-formyl-3-methylbutylcarbamate[8]



14f

The title compound was isolated as colorless oil in 98% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 94/6 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 31.2 min, t_{minor} = 34.2 min, *anti*: *ee* >99%, *dr* = 91/9; $[\alpha]_D^{20}$ = + 17.5 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.73 - 9.74 (d, *J* = 3 Hz, 1H), 7.18 - 7.32 (m, 9H), 5.88 - 5.91 (d, *J* = 9 Hz, 1H), 5.01 - 5.16 (m, 3H), 2.65(s, 1H), 1.88 - 1.95 (m, 1H), 1.02 - 1.08 (m, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 205.89, 155.63, 139.17, 136.07, 133.34, 128.86, 128.47, 128.15, 128.02, 127.91, 67.01, 62.26, 52.94, 28.38, 21.29, 19.15 ppm.

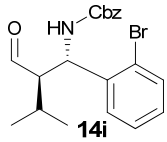
Benzyl (*1S, 2R*)-1-(4-bromophenyl)-2-formyl-3-methylbutylcarbamate[8]



14g

The title compound was isolated as colorless oil in 98% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 94/6 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm: t_{major} = 31.2 min, t_{minor} = 34.2 min, *anti*: *ee* >99%, *dr* >99:1; $[\alpha]_D^{20}$ = + 25.6 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.72 - 9.73 (d, *J* = 3 Hz, 1H), 7.43 - 7.45 (d, *J* = 6 Hz, 2H), 7.32 (s, 5H), 7.13 - 7.15 (d, *J* = 9 Hz, 2H), 5.89 - 5.92 (d, *J* = 9 Hz, 1H), 5.01 - 5.14 (m, 3H), 2.65(s, 1H), 1.86 - 1.97 (m, 1H), 1.02 - 1.10 (m, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 205.76, 155.64, 139.76, 136.11, 131.82, 128.47, 128.27, 128.15, 128.01, 121.46, 67.03, 62.23, 53.04, 28.38, 21.29, 19.17 ppm.

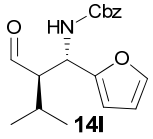
Benzyl (*1S, 2R*)-1-(2-bromophenyl)-2-formyl-3-methylbutylcarbamate



14i

The title compound was isolated as colorless oil in 85% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 94/6 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm: t_{major} = 29.27 min, t_{minor} = 26.37 min, *anti*: *ee* >99%, *dr* >99:1; $[\alpha]_D^{20}$ = + 74.7 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.70 (d, *J* = 1.1 Hz, 1H), 7.53 (d, *J* = 7.9 Hz, 1H), 7.40 - 7.18 (m, 6H), 7.16 - 7.04 (m, 1H), 6.26 (t, *J* = 17.2 Hz, 1H), 5.49 (dd, *J* = 8.9, 4.5 Hz, 1H), 5.15 - 4.98 (m, 2H), 2.96 (d, *J* = 28.0 Hz, 1H), 2.13 (ddt, *J* = 27.1, 13.4, 6.7 Hz, 1H), 1.10 (dt, *J* = 29.5, 14.8 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.56, 155.51, 139.72, 136.33, 133.40, 129.09, 128.59, 128.54, 128.19, 128.11, 127.69, 122.63, 67.02, 59.63, 53.28, 29.15, 21.12, 20.17 ppm. HRMS (ESI): calcd. For $C_{20}H_{22}BrNO_3$ $[M + Na]^+$ 426.0681; found 426.0667. IR (neat) : 3422, 3326, 3064, 3033, 2963, 2874, 2741, 1716, 1503, 1468, 1341, 1276, 1244, 1112, 1053, 1026, 754, 698, 629, 539 cm^{-1}.

Benzyl (*1S, 2R*)-2-formyl-1-(furan-2-yl)-3-methylbutylcarbamate

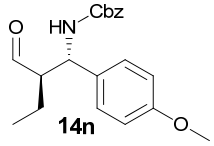


14l

The title compound was isolated as colorless oil in 95% yield. HPLC analysis on a Daicel ChiralPak IC column: 90/10 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm: t_{major} = 20.71 min, t_{minor} = not found, *anti*: *ee* >99%, *dr* = 97/3; $[\alpha]_D^{20}$ = - 6.3 (c = 1, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.83 (t, *J* = 4.7 Hz, 1H), 7.40 - 7.26 (m, 5H), 6.30 (dd, *J* = 3.0, 1.9 Hz, 1H), 6.22 (s, 1H), 5.73 (d, *J* = 9.2 Hz, 1H), 5.36 - 5.23

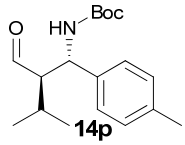
(m, 1H), 5.10 (s, 2H), 2.81 (td, J = 6.6, 3.1 Hz, 1H), 2.01 - 1.87 (m, 1H), 1.11 - 0.96 (m, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.13, 155.83, 152.90, 142.03, 136.19, 128.50, 128.16, 128.02, 110.42, 106.96, 67.06, 59.83, 47.87, 28.04, 21.17, 19.26 ppm. HRMS (ESI): calcd. For $C_{18}H_{21}NO_4$ $[M + Na]^+$ 338.1368; found 338.1369. IR (neat) : 3401, 3091, 3065, 3034, 2963, 2874, 2737, 1698, 1588, 1519, 1456, 1391, 1373, 1334, 1261, 1117, 1054, 868, 842, 811, 740, 698, 598 cm^{-1}.

Benzyl (*1S, 2R*)-2-formyl-1-(4-methoxyphenyl)butylcarbamate



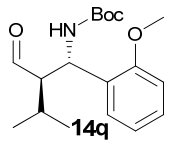
The title compound was isolated as white solid in 86% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 92/8 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm: t_{major} = 54.46 min, t_{minor} = 47.63 min, *anti*: *ee* >99%, *dr* = 91/9; $[\alpha]_D^{20}$ = - 4.7 (c = 1, $CHCl_3$). mp 81.5 - 85°C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.56 (t, J = 13.9 Hz, 1H), 7.40 - 7.02 (m, 7H), 6.83 (t, J = 7.5 Hz, 2H), 5.60 (t, J = 13.4 Hz, 1H), 5.04 (dd, J = 12.5, 2.0 Hz, 2H), 4.94 (dd, J = 15.2, 6.5 Hz, 1H), 3.76 (s, 3H), 2.58 (dd, J = 21.4, 17.5 Hz, 1H), 1.59 (dq, J = 21.9, 7.3 Hz, 1H), 1.43 - 1.28 (m, 1H), 0.89 (q, J = 7.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.05, 159.00, 155.67, 136.21, 132.18, 128.51, 128.14, 128.05, 128.01, 114.19, 66.97, 59.19, 55.29, 20.43, 11.57 ppm. HRMS (ESI): calcd. For $C_{20}H_{23}NO_4$ $[M + Na]^+$ 364.1525, found 364.1528. IR (KBr): 3330, 3063, 3035, 2963, 2934, 2874, 2842, 2742, 1719, 1687, 1612, 1530, 1514, 1426, 1293, 1246, 1214, 1179, 1090, 1028, 834, 758, 697, 617, 524 cm^{-1}.

***tert*-butyl (*1S, 2R*)-2-formyl-3-methyl-1-*p*-tolylbutylcarbamate**[7b, 8]



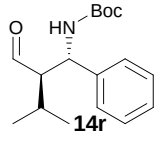
The title compound was isolated as colorless oil in 84% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 98/2 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 19.8 min, t_{minor} = 16.3 min, *anti*: *ee* = 98%, *dr* = 88/12; $[\alpha]_D^{20}$ = - 5.6 (c = 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.75 - 9.77 (d, J = 6 Hz, 1H), 0.98 - 1.00 (d, J = 6 Hz, 3H), 7.10 - 7.27 (m, 4H), 5.33 - 5.35 (d, J = 6 Hz, 1H), 5.08 - 5.13 (m, 1H), 2.56 - 2.58 (m, 1H), 2.33 (s, 3H), 1.80 - 1.87 (m, 1H), 1.43 (s, 9H),1.05 - 1.07 (d, J = 6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 206.15, 155.08, 137.76, 137.21, 129.42, 129.12, 79.74, 63.01, 52.80, 28.08, 21.33, 21.04, 21.01, 18.66 ppm.

***tert*-butyl (*1S, 2R*)-2-formyl-1-(2-methoxyphenyl)-3-methylbutylcarbamate**



The title compound was isolated as colorless oil in 87% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 96/4 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 14.42 min, t_{minor} = 12.07 min, *anti*: *ee* >99%, *dr* = 88/12; $[\alpha]_D^{20}$ = + 5.4 (c = 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.74 (d, J = 4.2 Hz, 1H), 7.29 - 7.19 (m, 2H), 6.91 (dd, J = 16.3, 8.0 Hz, 2H), 5.72 (d, J = 9.6 Hz, 1H), 5.35 (t, J = 9.0 Hz, 1H), 3.86 (s, 3H), 2.84 - 2.65 (m, 1H), 1.85 - 1.65 (m, 1H), 1.40 (s, 9H), 1.01 (dd, J = 21.4, 6.9 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.70, 156.91, 155.05, 128.99, 128.79, 128.07, 120.73, 110.91, 79.56, 61.33, 55.22, 50.79, 28.34, 21.40, 18.39 ppm. HRMS (ESI): calcd. For $C_{18}H_{27}NO_4$ $[M + Na]^+$ 344.1838; found 344.1844. IR (neat): 3445, 3352, 3069, 2965, 2875, 2839, 2728, 1715, 1699, 1602, 1492, 1460, 1367, 1244, 1163, 1050, 1026, 879, 755, 628, 563 cm^{-1}.

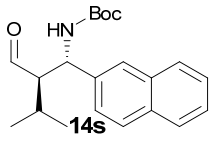
***tert*-butyl (*1S, 2R*)-2-formyl-3-methyl-1-phenylbutylcarbamate**[5b, 7b, 8]



The title compound was isolated as colorless oil in 86% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 100/1 hexane/*i*PrOH, flow rate 1 mL/min, λ = 205 nm, t_{major} = 12.15 min, t_{minor} = not found, *anti*: *ee* >99%, *dr* = 89/11; $[\alpha]_D^{20}$ = + 6.2 (c = 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.75 - 9.76 (d, J = 3 Hz, 1H), 7.26 - 7.36 (m, 5H), 5.41 - 5.44 (d, J = 9 Hz, 1H), 5.13 (m, 1H), 2.60 - 2.65 (m, 1H),

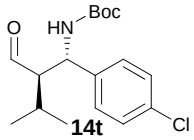
1.84 - 1.90 (m, 1H), 1.38 (s, 9H), 1.0 - 1.08 (m, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 206.15, 155.03, 140.78, 128.72, 127.49, 126.52, 79.76, 62.91, 53.02, 28.25, 28.11, 21.29, 18.80 ppm.

***tert*-butyl (*1S, 2R*)-2-formyl-3-methyl-1-(naphthalen-2-yl)butylcarbamate**[8]



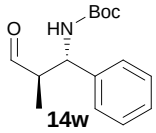
The title compound was isolated as colorless oil in 85% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 98/2 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 23.6 min, t_{minor} = 27.0 min, *anti*: *ee* >99%, *dr* = 85/15; $[\alpha]_D^{20}$ = - 3.2 (c = 0.75, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.78 - 9.79 (d, *J* = 3 Hz, 1H), 7.35 - 7.84 (m, 7H), 5.50 - 5.53 (d, *J* = 9 Hz, 1H), 5.3 (m, 1H), 2.71 - 2.76 (m, 1H), 1.84 - 1.93 (m, 1H), 1.39 (s, 9H), 1.08 - 1.10 (d, *J* = 6 Hz, 3H), 1.02 - 1.04(d, *J* = 6 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 206.04, 171.10, 155.07, 138.15, 133.27, 132.75, 128.69, 127.92, 127.59, 126.33, 126.02, 125.62, 124.32, 79.87, 62.73, 60.35, 53.26, 28.20, 21.31, 21.00, 18.87, 14.16 ppm.

***tert*-butyl (*1S, 2R*)-1-(4-chlorophenyl)-2-formyl-3-methylbutylcarbamate**[7b, 8]



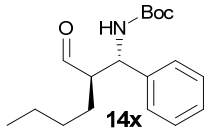
The title compound was isolated as colorless oil in 83% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 98/2 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 16.1 min, t_{minor} = 15.6 min, *anti*: *ee* = 98%, *dr* = 98/2; $[\alpha]_D^{20}$ = + 7.5 (c = 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.73 - 9.74 (d, *J* = 3Hz, 1H), 7.29 - 7.32 (d, *J* = 9 Hz, 2H), 7.19 - 7.22 (d, *J* = 9 Hz, 2H), 5.50 - 5.53 (d, *J* = 9 Hz, 1H), 5.08 (m, 1H), 2.60 (s, 1H), 1.87 - 1.93 (m, 1H), 1.39 (s, 9H), 1.02 - 1.08 (m, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 205.82, 155.01, 139.60, 133.21, 128.84, 127.92, 79.98, 62.60, 52.46, 28.25, 21.24, 19.07 ppm.

***tert*-butyl (*1S, 2R*)-2-methyl-3-oxo-1-phenylpropylcarbamate**[5b, 7b]



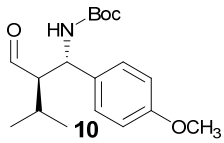
The title compound was isolated as white solid in 93% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 94/6 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm: t_{major} = 21.56 min, t_{minor} = 16.68 min, *anti*: *ee* = 99%, *dr* = 85/15; $[\alpha]_D^{20}$ = - 16.0 (c = 1, $CHCl_3$). mp 100 - 103°C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.73 - 9.63 (m, 1H), 7.39 - 7.21 (m, 4H), 5.28 (dd, *J* = 22.1, 15.2 Hz, 1H), 4.87 (s, 1H), 2.96 - 2.71 (m, 1H), 1.40 (d, *J* = 5.2 Hz, 9H), 1.04 (dd, *J* = 13.6, 7.0 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.43, 155.19, 139.93, 128.75, 127.71, 127.60, 126.81, 126.62, 79.95, 51.55, 28.25, 11.87, 9.22 ppm.

***tert*-butyl (*1S, 2R*)-2-formyl-1-phenylhexylcarbamate**[5b, 7b]



The title compound was isolated as white solid in 95% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 94/6 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm: t_{major} = 13.64 min, t_{minor} = 12.59 min, *anti*: *ee* >99%, *dr* = 85:15; $[\alpha]_D^{20}$ = + 12.0 (c = 1, $CHCl_3$). mp 79 - 83°C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.60 (d, *J* = 3.7 Hz, 1H), 7.43 - 7.10 (m, 5H), 5.28 (dd, *J* = 23.3, 8.9 Hz, 1H), 4.92 (s, 1H), 2.71 (d, *J* = 3.3 Hz, 1H), 1.71 - 1.52 (m, 1H), 1.36 (d, *J* = 18.1 Hz, 9H), 1.27 (dd, *J* = 16.9, 8.6 Hz, 5H), 0.94 - 0.72 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.94, 155.10, 140.32, 128.73, 127.62, 126.70, 79.82, 57.77, 54.46, 29.20, 28.25, 26.70, 22.46, 13.70 ppm.

***tert*-butyl (*1S, 2R*)-2-formyl-1-(4-methoxyphenyl)-3-methylbutylcarbamate**[7b,8]



The title compound was isolated as colorless oil in 90% yield. HPLC analysis on a Daicel ChiralPak AS-H column: 96/4 hexane/*i*PrOH, flow rate 0.5 mL/min, λ = 220 nm, t_{major} = 20.9 min, t_{minor} = 18.6 min, *anti*: *ee* = 99%, *dr* = 92/8; $[\alpha]_D^{20}$ = - 8.7 (c = 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ

9.76 (d, 1H, *J* = 3.9 Hz), 7.14 (d, 2H, *J* = 8.7 Hz), 6.83(d, 2H, *J* = 8.4 Hz), 5.32 (d, 1H, *J* = 9 Hz), 5.08 (br, 1H), 3.79 (s, 3H), 2.54 - 2.60 (m, 1H), 1.80 - 1.87(m, 1H), 1.40 (s, 9H), 1.04 - 1.11 (m, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 206.14, 158.87, 155.00, 132.82, 127.68, 114.08, 79.70, 63.00, 55.18, 28.06, 21.31, 18.54 ppm.

8. References:

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3 A. G. Wenzel, E. N. Jacobsen, *J. Am. Chem. Soc.* 2002, **124**, 12964-12965.

4 A. L. Tillman, J. Ye, D. J. Dixon, *Chem. Commun*. 2006, 1191-1193.

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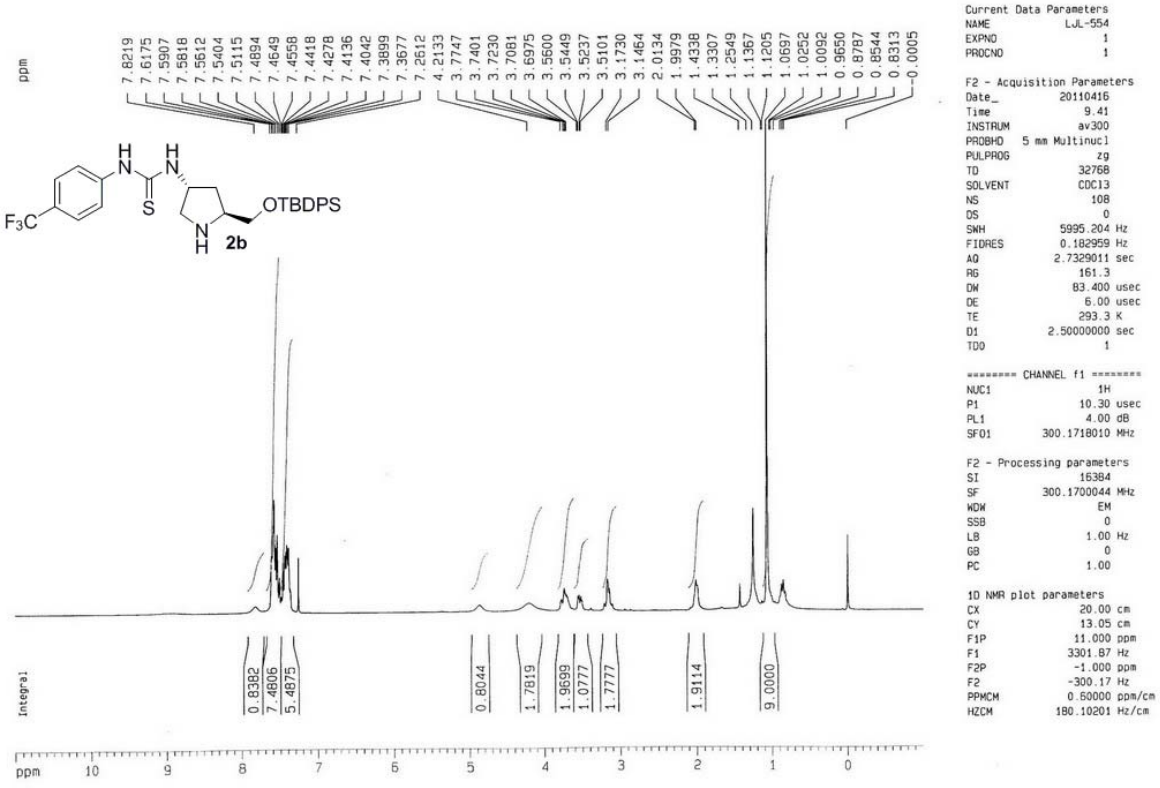
9. NMR spectra and HPLC for catalysts and part of the Mannich products

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PROCNO                      1

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NS                        108
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FIDRES              0.182959 Hz
AQ                  2.7329011 sec
RG                      161.3
DW                     83.400 usec
DE                      6.00 usec
TE                     293.3 K
D1                  2.50000000 sec
TD0                         1

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PL1                     4.00 dB
SF01              300.1718010 MHz

F2 - Processing parameters
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WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
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CY                     13.05 cm
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F1                  3301.87 Hz
F2P                  -1.000 ppm
F2                  -300.17 Hz
PPMCM              0.60000 ppm/cm
HZCM              180.10201 Hz/cm
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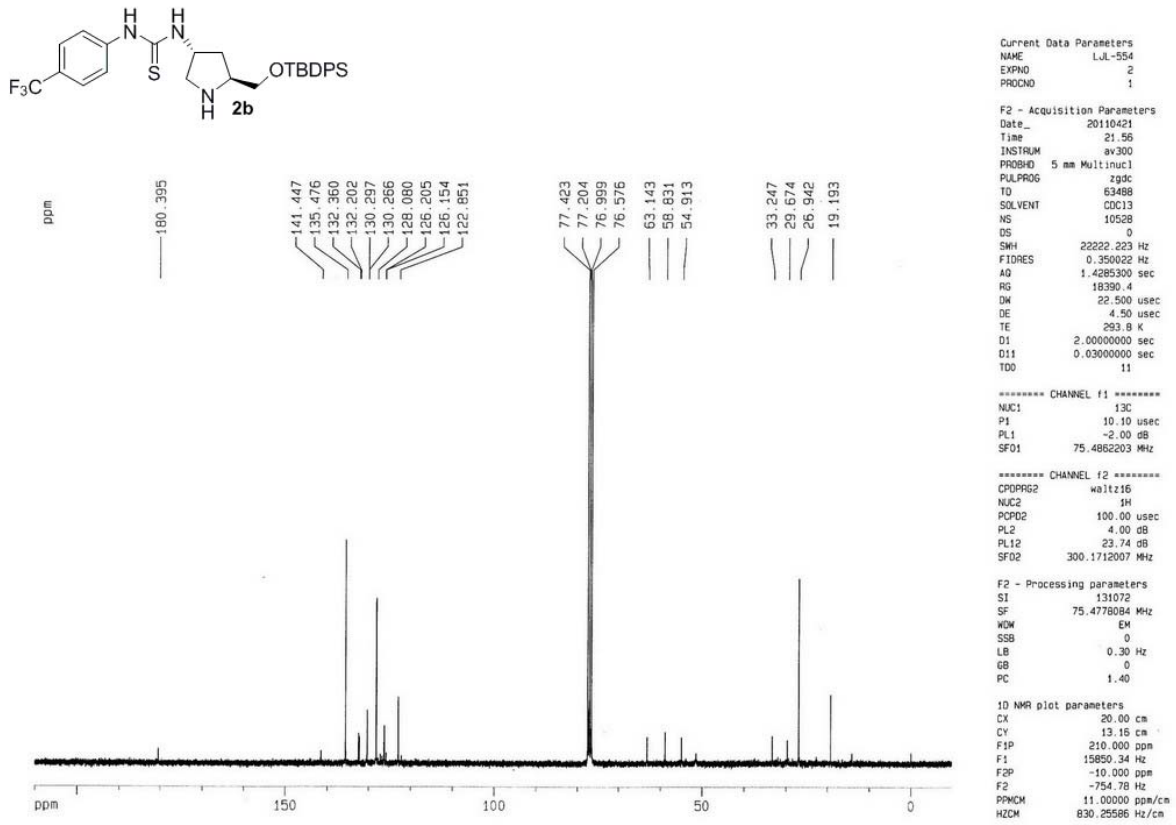


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PROCNO                      1

F2 - Acquisition Parameters
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PULPROG                 zgdc
TD                      63488
SOLVENT                CDCl3
NS                        1052
DS                          0
SWH                22222.223 Hz
FIDRES              0.350022 Hz
AQ                  1.4285300 sec
RG                     18390.4
DW                     22.500 usec
DE                      4.50 usec
TE                     293.8 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                        11

======== CHANNEL f1 ========
NUC1                       13C
P1                     10.10 usec
PL1                     -2.00 dB
SF01              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                     4.00 dB
PL12                   23.74 dB
SF02              300.1712007 MHz

F2 - Processing parameters
SI                     131072
SF                75.4778084 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                     20.00 cm
CY                     13.15 cm
F1P                  210.000 ppm
F1                  15850.34 Hz
F2P                  -10.000 ppm
F2                   -754.78 Hz
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HZCM              830.25586 Hz/cm
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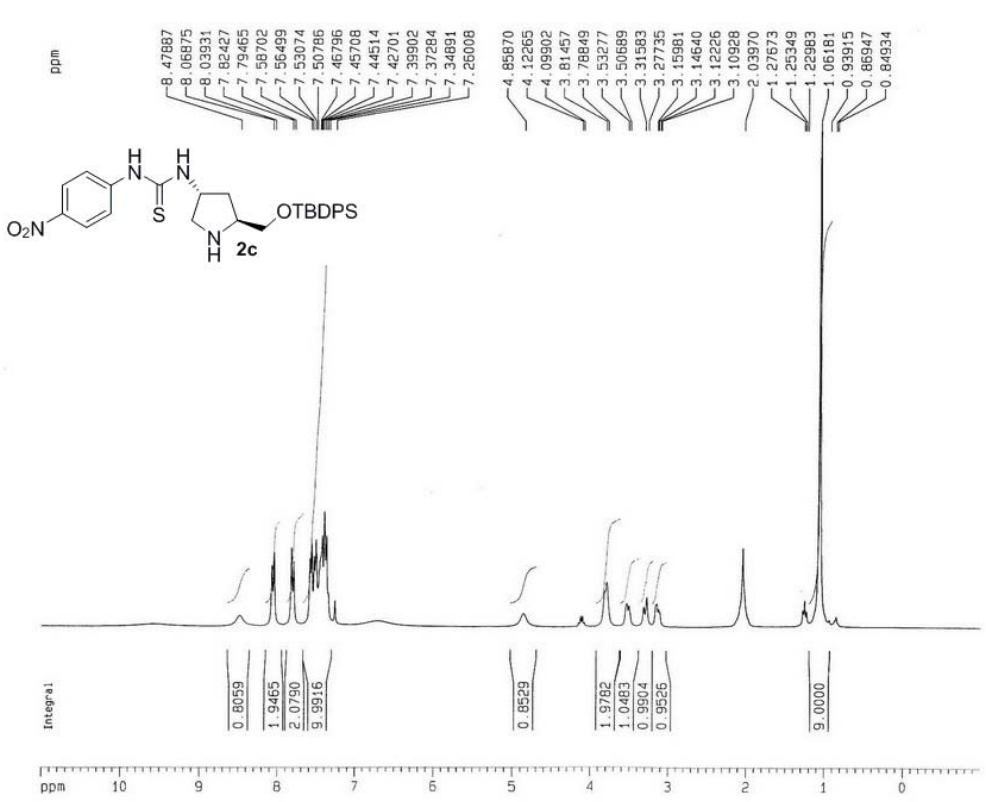


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Current Data Parameters
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EXPNO                      1
PROCNO                     1

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PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        23
DS                         0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                      71.8
DW                    83.400 usec
DE                     6.00 usec
TE                    673.2 K
D1                 2.50000000 sec
TD0                        1

======== CHANNEL f1 ========
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PL1                     4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
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SF                300.1700045 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     12.76 cm
F1P                   11.000 ppm
F1                   3301.87 Hz
F2P                  -1.000 ppm
F2                   -300.17 Hz
PPMCM                0.60000 ppm/cm
HZCM               180.10201 Hz/cm
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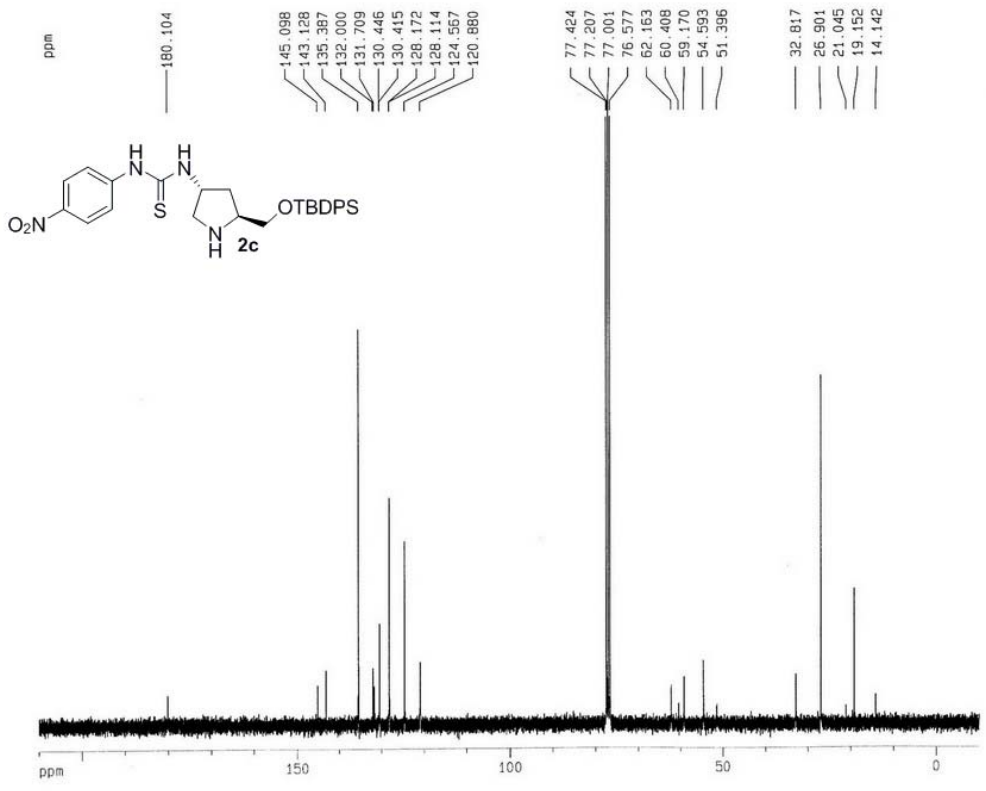


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Current Data Parameters
NAME                 LJL-555
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
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PULPROG                 zgdc
TD                     63488
SOLVENT                CDCl3
NS                       845
DS                         0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                    5160.6
DW                    22.500 usec
DE                     4.50 usec
TE                    673.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       11

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.10 usec
PL1                    -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 100.00 usec
PL2                     4.00 dB
PL12                   23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                    131072
SF                75.4778116 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                     13.22 cm
F1P                  210.000 ppm
F1                   15850.34 Hz
F2P                  -10.000 ppm
F2                   -754.78 Hz
PPMCM               11.00000 ppm/cm
HZCM               830.25592 Hz/cm
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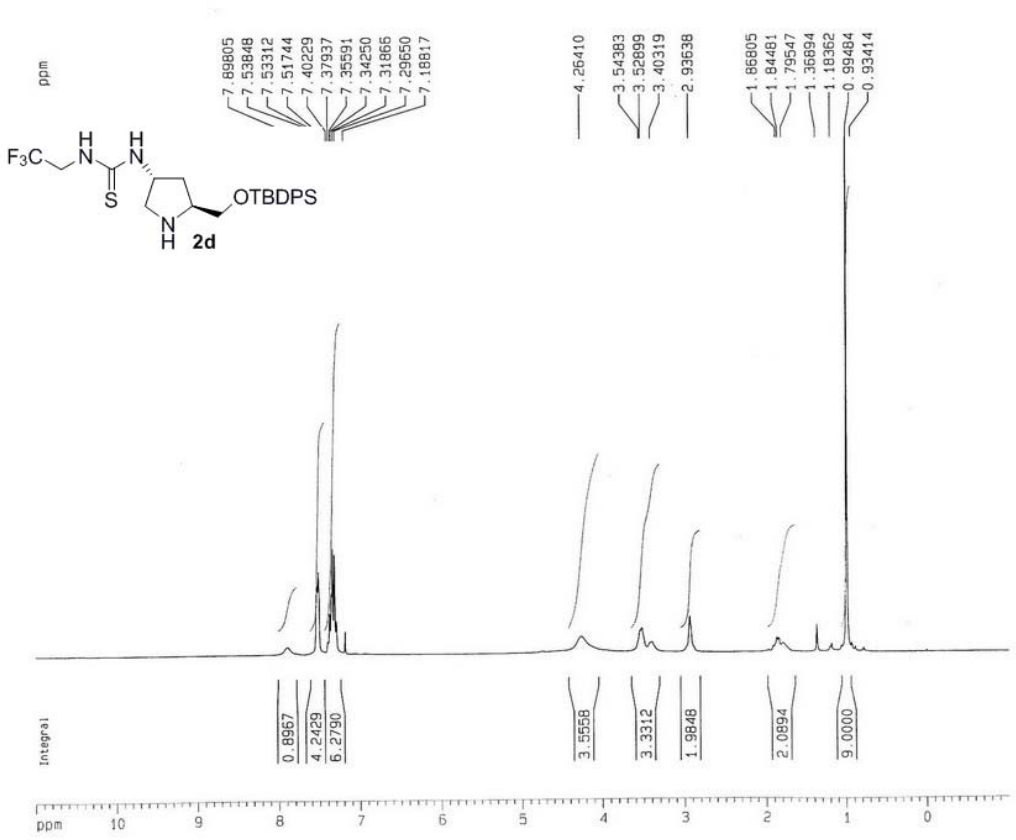


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NAME                  LJL-551
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110403
Time                  19.13
INSTRUM               av300
PROBHD   5 mm Multinucl
PULPROG                  zg
TD                    32768
SOLVENT               CDCl3
NS                       55
DS                        0
SWH                5995.204 Hz
FIDRES            0.182959 Hz
AQ                2.7329011 sec
RG                     71.8
DW                   83.400 usec
DE                    6.00 usec
TE                   673.2 K
D1                2.50000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.30 usec
PL1                    4.00 dB
SF01             300.1718010 MHz

F2 - Processing parameters
SI                    16384
SF                300.1700263 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    12.57 cm
F1P                  11.000 ppm
F1                 3301.87 Hz
F2P                 -1.000 ppm
F2                 -300.17 Hz
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HZCM             180.10201 Hz/cm
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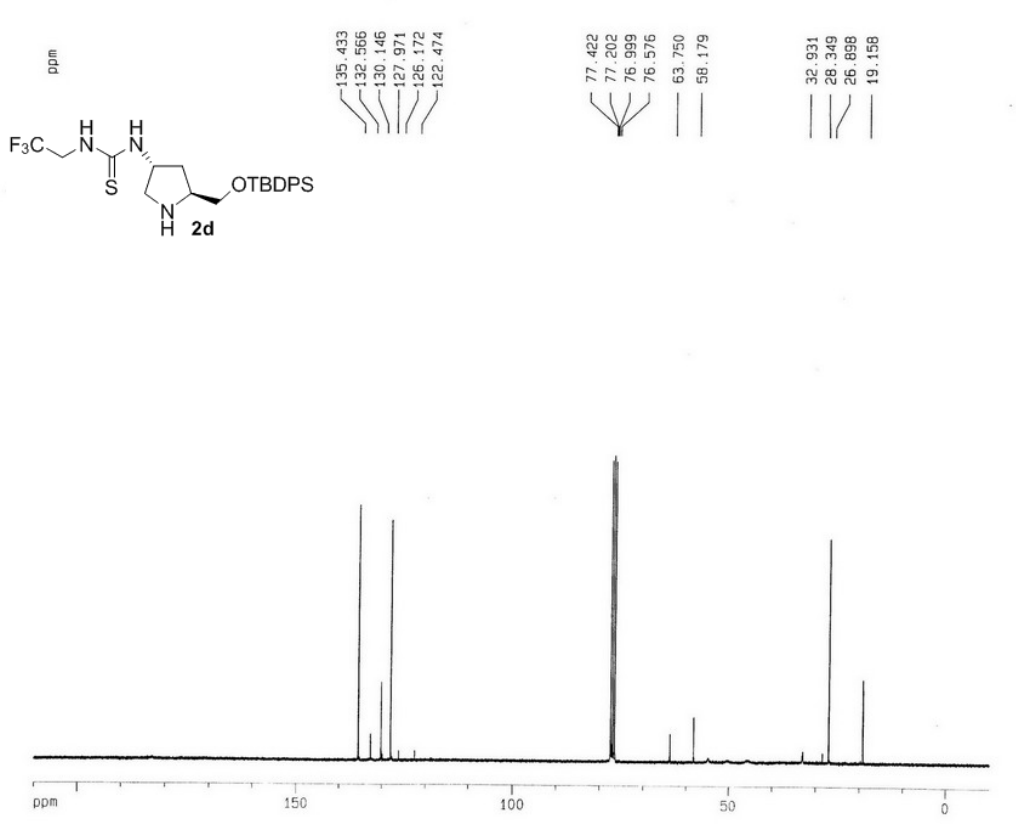


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Current Data Parameters
NAME                  LJL-551
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110403
Time                  19.20
INSTRUM               av300
PROBHD   5 mm Multinucl
PULPROG                zgdc
TD                    63488
SOLVENT               CDCl3
NS                    12214
DS                        0
SWH               22222.223 Hz
FIDRES            0.350022 Hz
AQ                1.4285300 sec
RG                   18390.4
DW                   22.500 usec
DE                    4.50 usec
TE                   673.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      11

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.10 usec
PL1                   -2.00 dB
SF01             75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                    4.00 dB
PL12                  23.74 dB
SF02             300.1712007 MHz

F2 - Processing parameters
SI                   131072
SF                75.4778103 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     6.38 cm
F1P                  210.000 ppm
F1                15850.34 Hz
F2P                 -10.000 ppm
F2                 -754.78 Hz
PPMCM              11.00000 ppm/cm
HZCM             830.25592 Hz/cm
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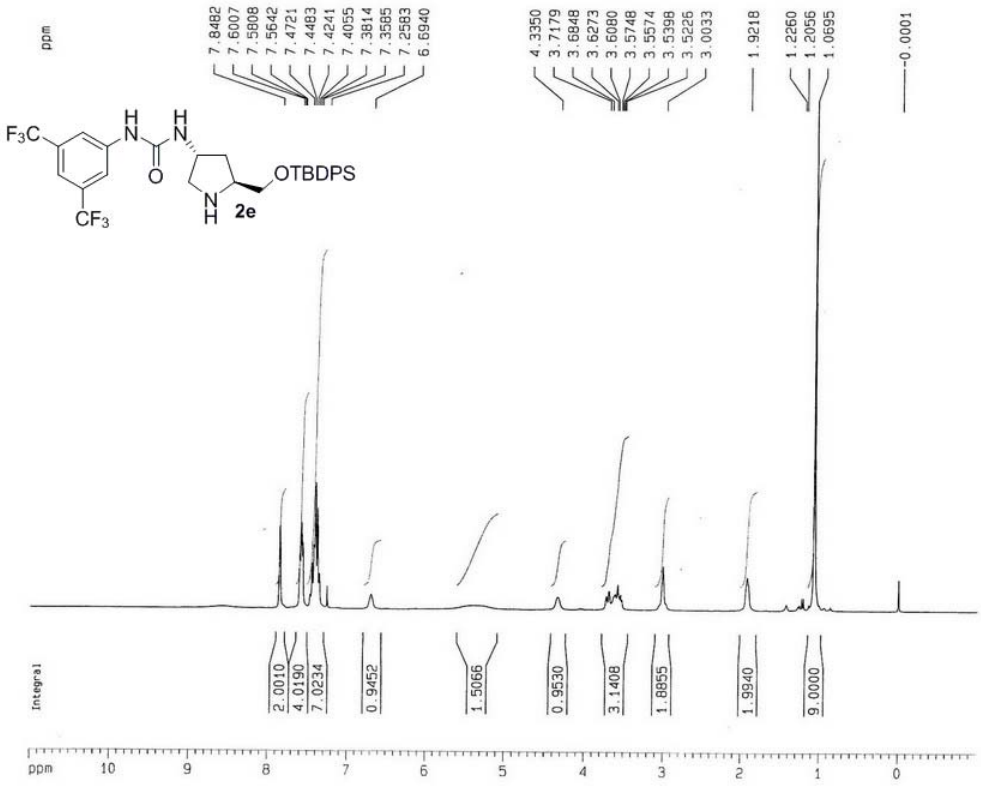

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EXPNO 1
PROCNO 1

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Time 19.43
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PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 57
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 90.5
DW 83.400 usec
DE 6.00 usec
TE 295.5 K
D1 2.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz

F2 - Processing parameters
SI 16384
SF 300.1700051 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.06 cm
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F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
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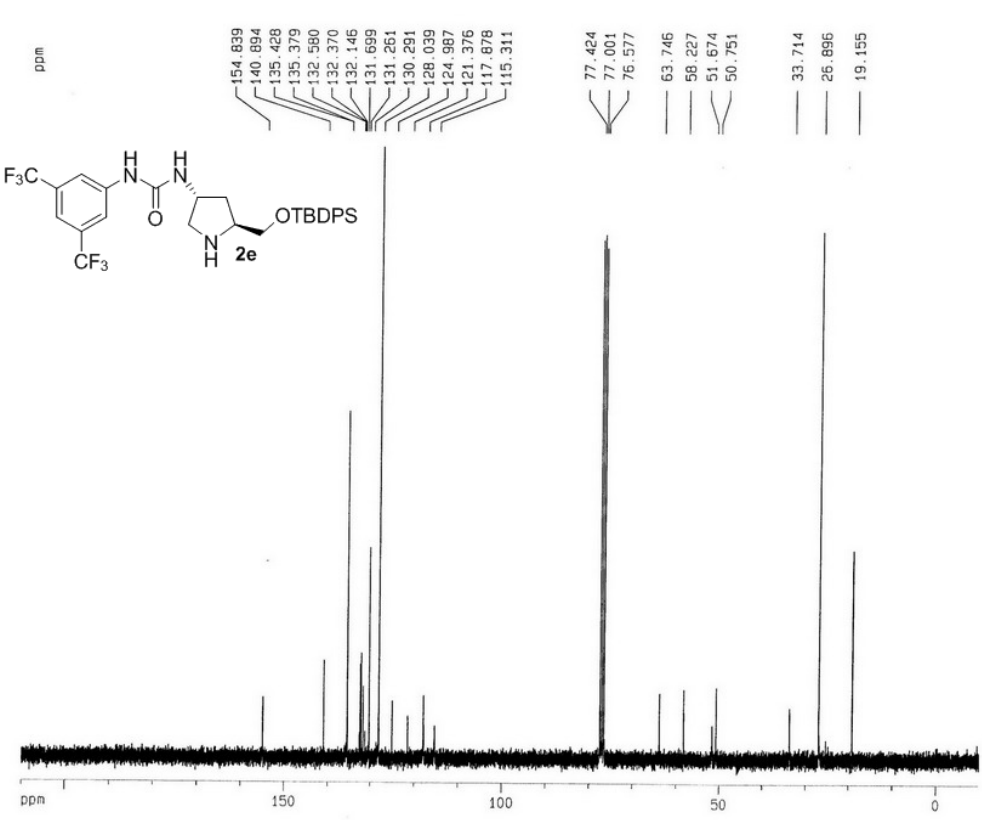
HZCM 180.10201 Hz/cm



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EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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PROBHD 5 mm Multinucl
PULPROG zgdc
TD 63488
SOLVENT CDCl3
NS 679
DS 0
SWH 22222.223 Hz
FIDRES 0.350022 Hz
AQ 1.4285300 sec
RG 16384
DW 22.500 usec
DE 4.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 11

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 -2.00 dB
SFO1 75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 23.74 dB
SFO2 300.1712007 MHz

F2 - Processing parameters
SI 131072
SF 75.4778101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.72 cm
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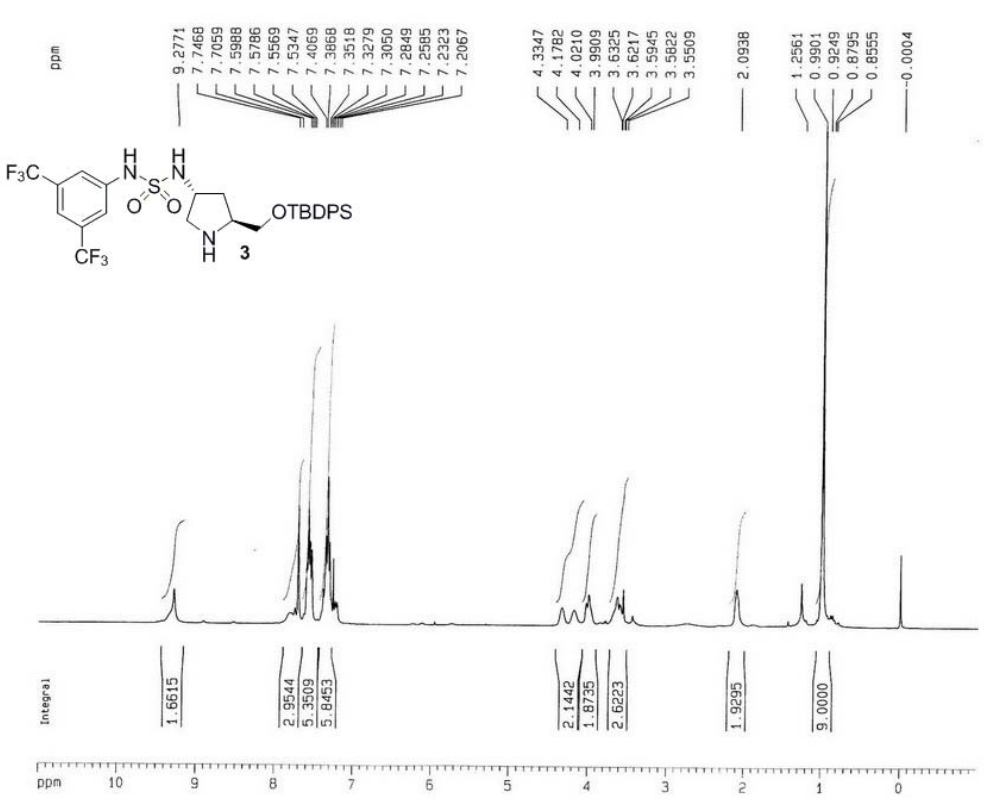
F1 15850.34 Hz
F2P -10.000 ppm
F2 -754.78 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.25592 Hz/cm

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EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
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Time                  20.11
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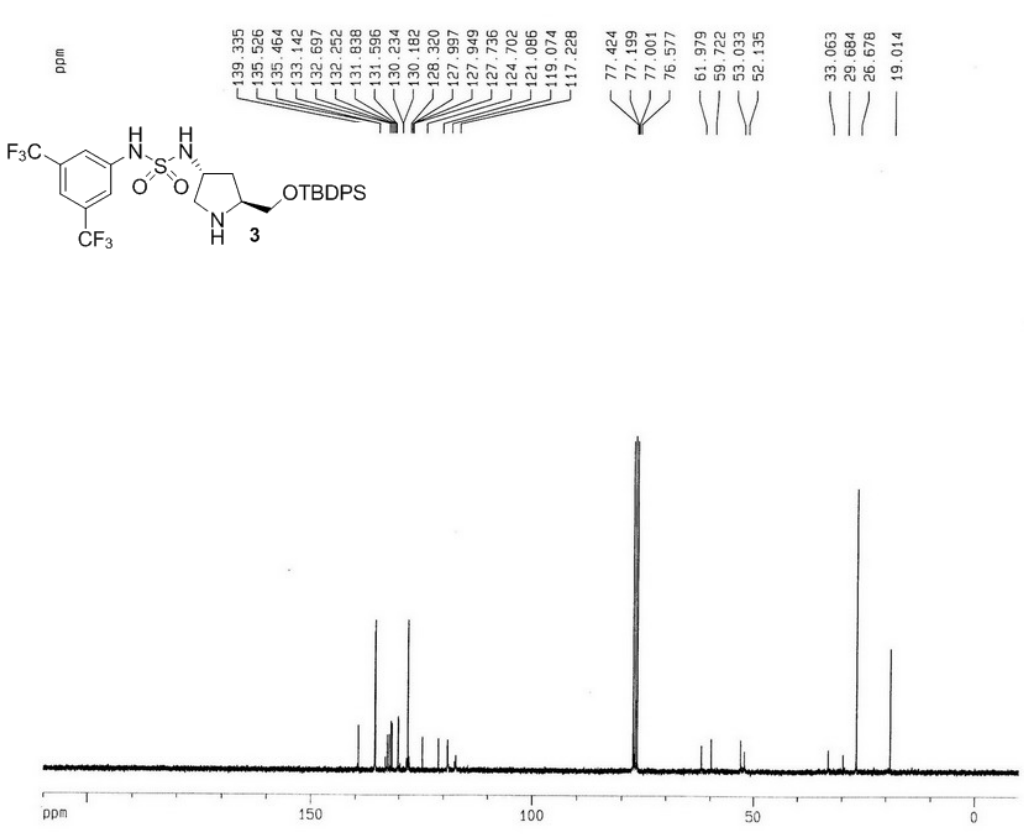
PROBHD   5 mm Multinucl
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        56
DS                         0
SWH                5995.204 Hz
FIDRES            0.182959 Hz
AQ                2.7329011 sec
RG                       128
DW                  83.400 usec
DE                   6.00 usec
TE                   293.6 K
D1                2.50000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                   10.30 usec
PL1                   4.00 dB
SFO1             300.1718010 MHz

F2 - Processing parameters
SI                        16384
SF               300.1700051 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    12.70 cm
F1P                  11.000 ppm
F1                  3301.87 Hz
F2P                 -1.000 ppm
F2                  -300.17 Hz
PPMCM             0.60000 ppm/cm
HZCM             180.10201 Hz/cm
```

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EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
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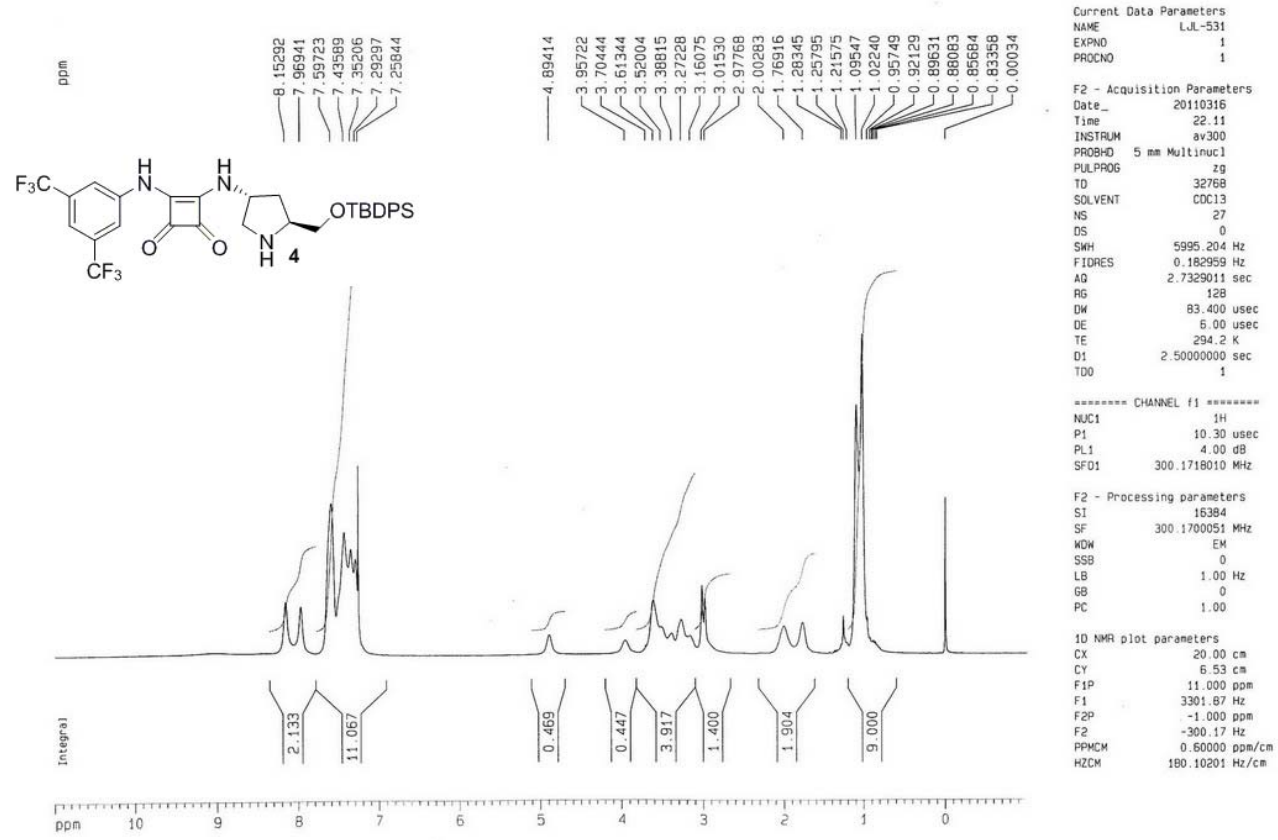
PULPROG                zgdc
TD                     63488
SOLVENT                CDCl3
NS                      2562
DS                         0
SWH               22222.223 Hz
FIDRES            0.350022 Hz
AQ                1.4285300 sec
RG                   18390.4
DW                  22.500 usec
DE                   4.50 usec
TE                   294.6 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       10

======== CHANNEL f1 ========
NUC1                      13C
P1                   10.10 usec
PL1                  -2.00 dB
SFO1             75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   4.00 dB
PL12                 23.74 dB
SFO2             300.1712007 MHz

F2 - Processing parameters
SI                       131072
SF               75.4778094 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     6.86 cm
F1P                  210.000 ppm
F1                  15850.34 Hz
F2P                 -10.000 ppm
F2                  -754.78 Hz
PPMCM             11.00000 ppm/cm
HZCM             830.25586 Hz/cm
```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3.61344
3.52004
3.38815
3.27228
3.16075
3.01530
2.97768
2.00283
1.76916
1.28345
1.25795
1.21575
1.09547
1.02240
0.95749
0.92129
0.89631
0.86083
0.85684
0.83358
0.00034
F3C
CF3
H
N
O
OTBDPS
4
Integral
2.133
11.067
0.469
0.447
3.917
1.400
1.904
9.000
Current Data Parameters
NAME LJL-531
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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Time 22.11
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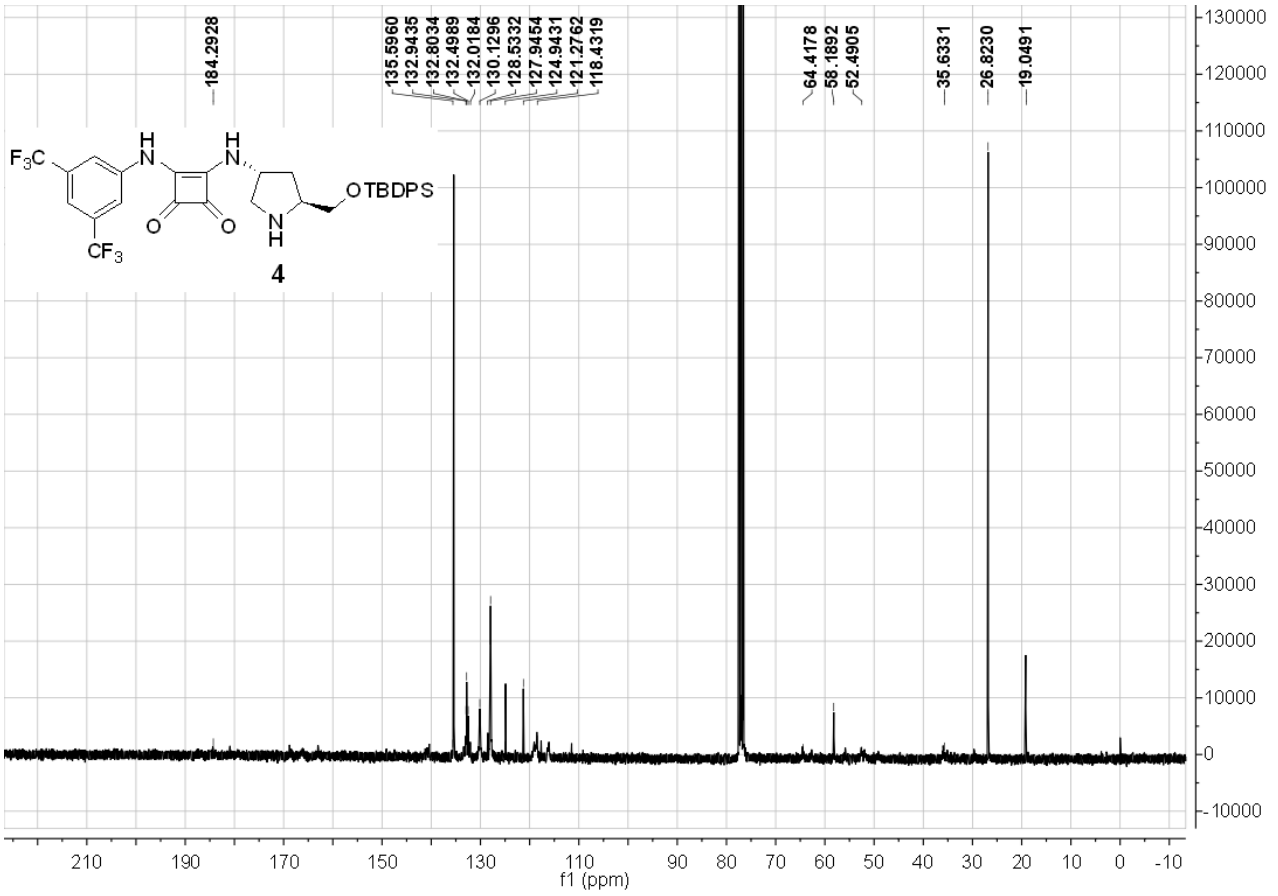
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 27
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 294.2 K
D1 2.50000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz
F2 - Processing parameters
SI 16384
SF 300.1700051 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 6.53 cm
F1P 11.000 ppm
F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.10201 Hz/cm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118.4319
64.4178
58.1892
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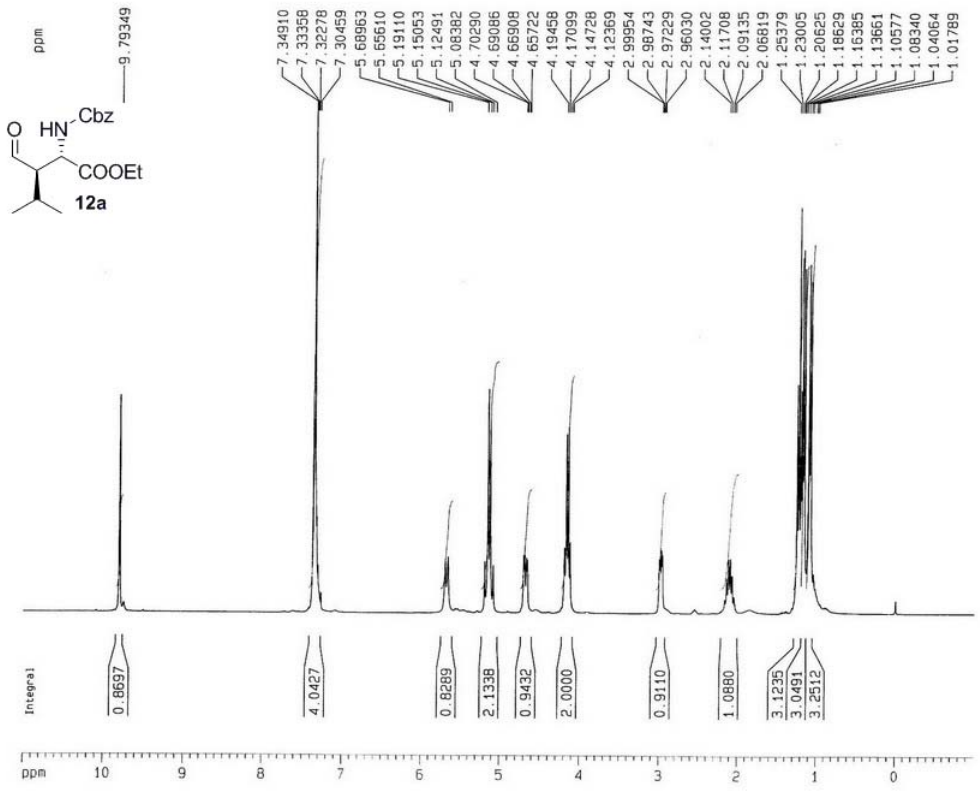
F3C
CF3
H
N
O
OTBDPS
4
f1 (ppm)

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Current Data Parameters
NAME                  LJL632
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110524
Time                  19.18
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        50
DS                         0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                      35.9
DW                   83.400 usec
DE                    6.00 usec
TE                   673.2 K
D1                 2.50000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                    10.30 usec
PL1                    4.00 dB
SFO1           300.1718010 MHz

F2 - Processing parameters
SI                      16384
SF                 300.1700025 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    12.56 cm
F1P                  11.000 ppm
F1                  3301.87 Hz
F2P                 -1.000 ppm
F2                  -300.17 Hz
PPMCM                0.60000 ppm/cm
HZCM               180.10201 Hz/cm
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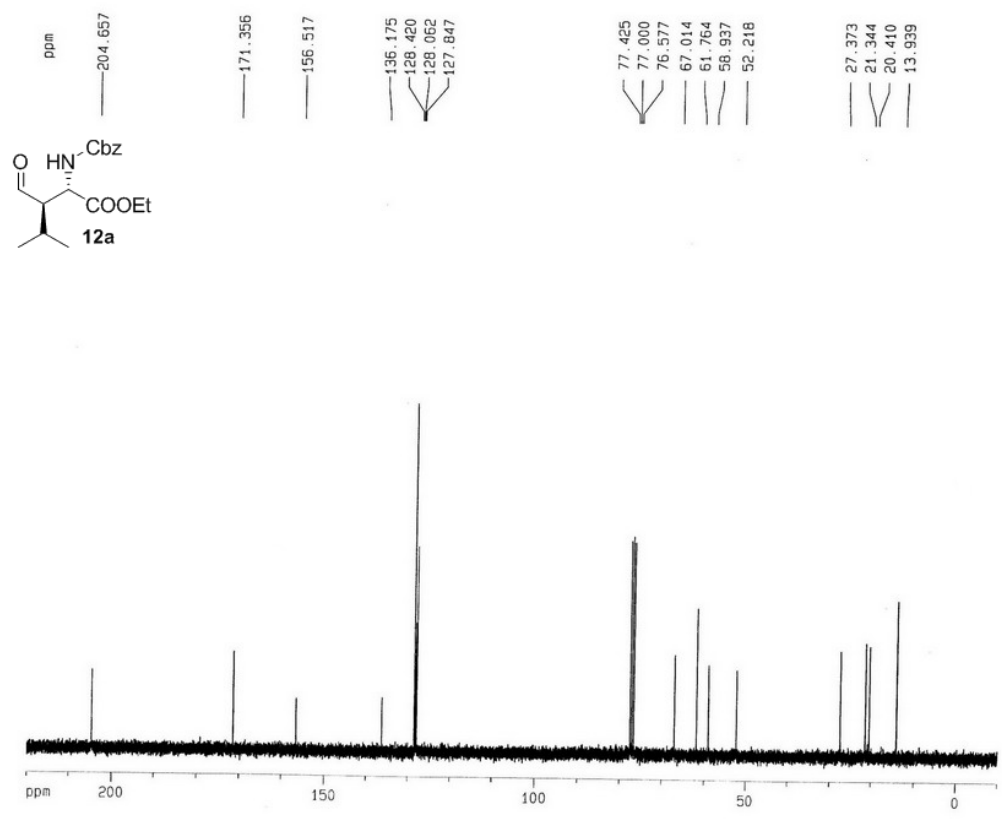


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Current Data Parameters
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EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110524
Time                  19.26
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PROBHD   5 mm Multinucl
PULPROG                zgdc
TD                     63488
SOLVENT                CDCl3
NS                       277
DS                         0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                   18390.4
DW                   22.500 usec
DE                    4.50 usec
TE                   673.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       11

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.10 usec
PL1                   -2.00 dB
SFO1           75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                    4.00 dB
PL12                  23.74 dB
SFO2           300.1712007 MHz

F2 - Processing parameters
SI                     131072
SF                 75.4778136 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     7.26 cm
F1P                  220.000 ppm
F1                  16605.12 Hz
F2P                 -10.000 ppm
F2                  -754.78 Hz
PPMCM               11.50000 ppm/cm
HZCM               867.99487 Hz/cm
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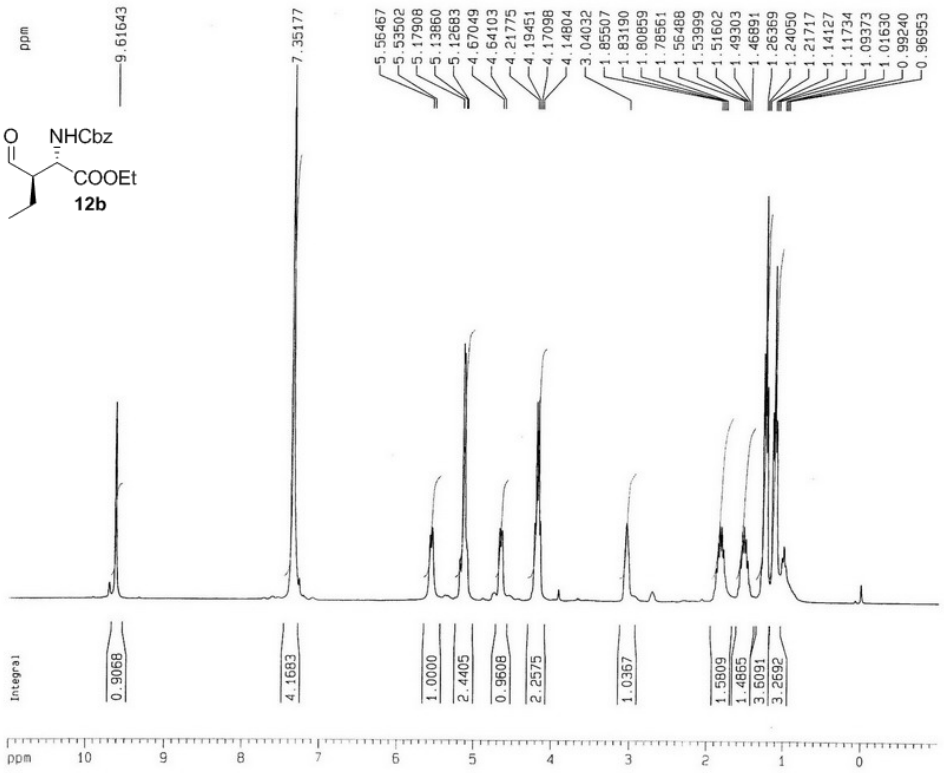


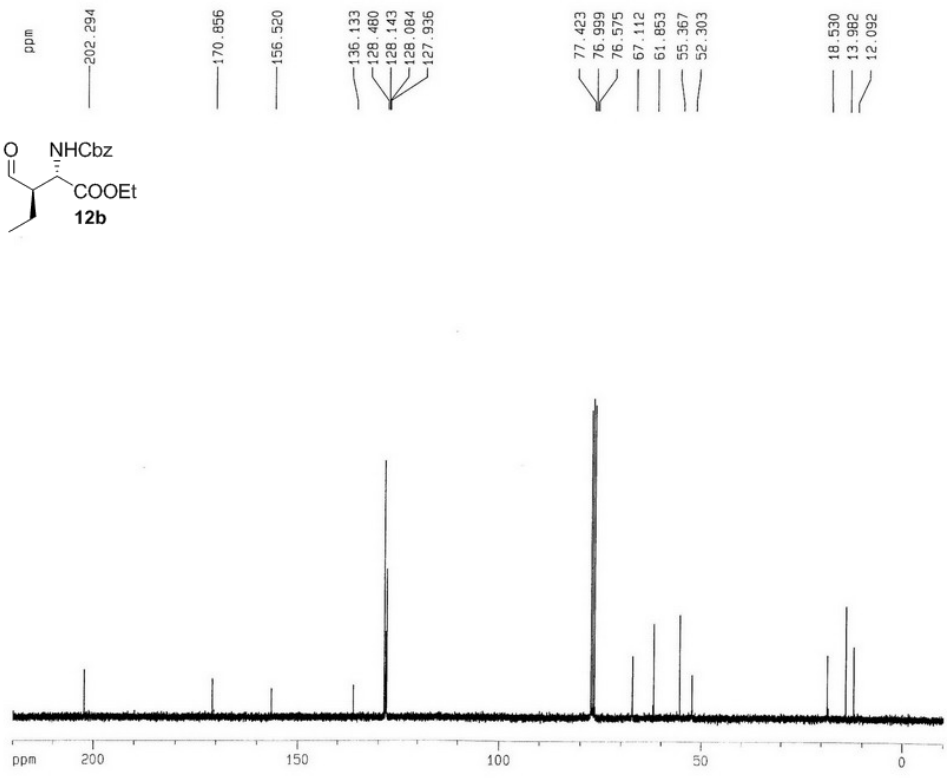
Current Data Parameters
NAME LJL-608-2-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110514
Time 20.47
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 51
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 64
DW 83.400 usec
DE 6.00 usec
TE 296.9 K
D1 2.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz

F2 - Processing parameters
SI 16384
SF 300.1700036 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 13.31 cm
F1P 11.000 ppm
F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.10201 Hz/cm



Current Data Parameters
NAME LJL-608-2-1
EXPNO 2
PROCNO 1

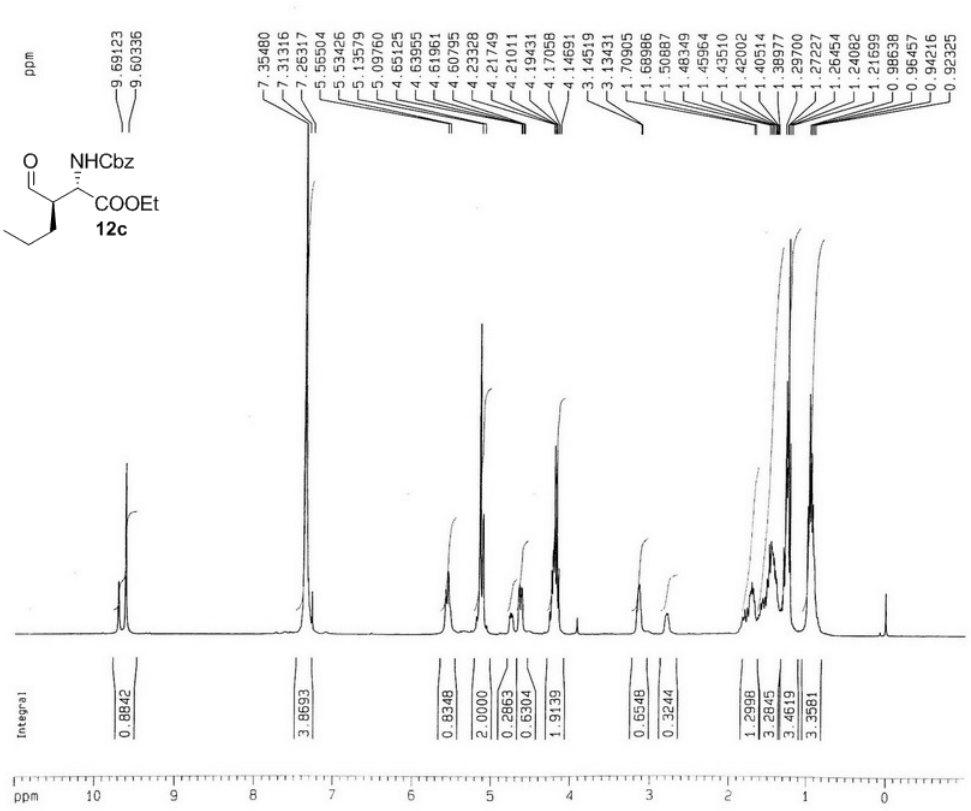
F2 - Acquisition Parameters
Date_ 20110514
Time 21.52
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 63488
SOLVENT CDCl3
NS 1174
DS 0
SWH 22222.223 Hz
FIDRES 0.350022 Hz
AQ 1.4285300 sec
RG 18390.4
DW 22.500 usec
DE 4.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 11

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 -2.00 dB
SFO1 75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 23.74 dB
SFO2 300.1712007 MHz

F2 - Processing parameters
SI 131072
SF 75.4778106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.86 cm
F1P 220.000 ppm
F1 16605.12 Hz
F2P -10.000 ppm
F2 -754.78 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.99487 Hz/cm



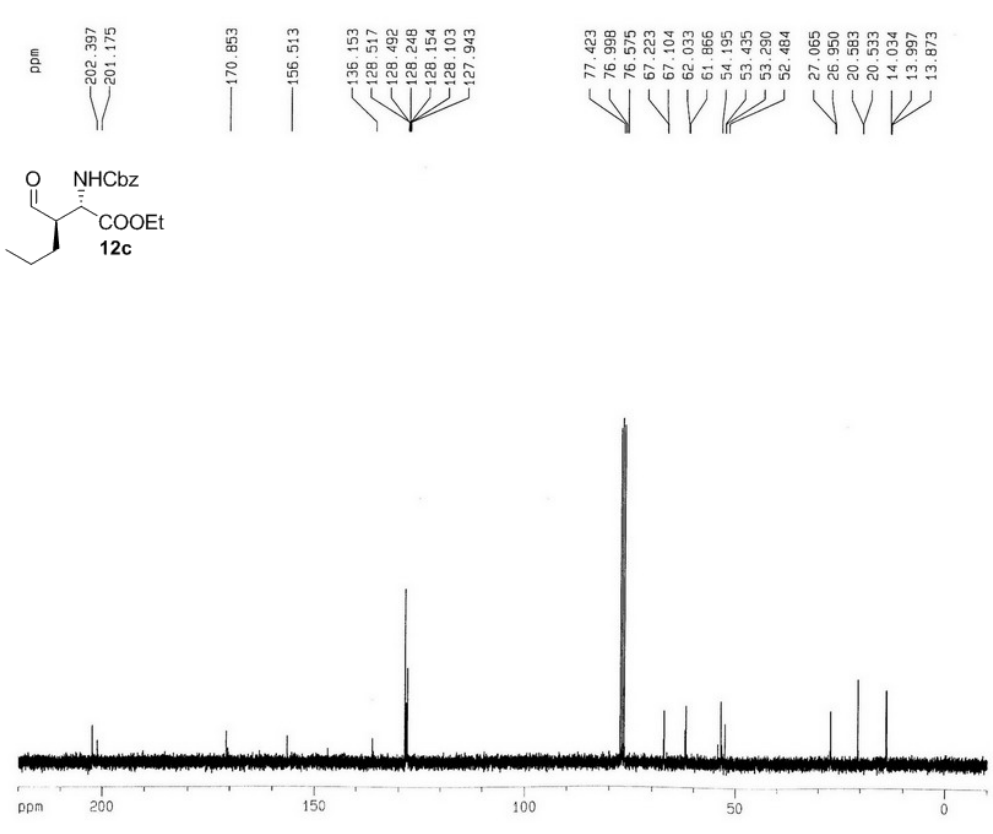

```
Current Data Parameters
NAME                  LJL-617
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110518
Time                   20.26
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        37
DS                         0
SWH               5995.204 Hz
FIDRES               0.182959 Hz
AQ                 2.7329011 sec
RG                        71.8
DW                    83.400 usec
DE                     6.00 usec
TE                    673.2 K
D1                 2.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    10.30 usec
PL1                    4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                       16384
SF                300.1700036 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     12.81 cm
F1P                   11.000 ppm
F1                  3301.87 Hz
F2P                   -1.000 ppm
F2                  -300.17 Hz
PPMCM                0.60000 ppm/cm
HZCM               180.10201 Hz/cm
```




```
Current Data Parameters
NAME                  LJL-617
EXPNO                       2
PROCNO                      1

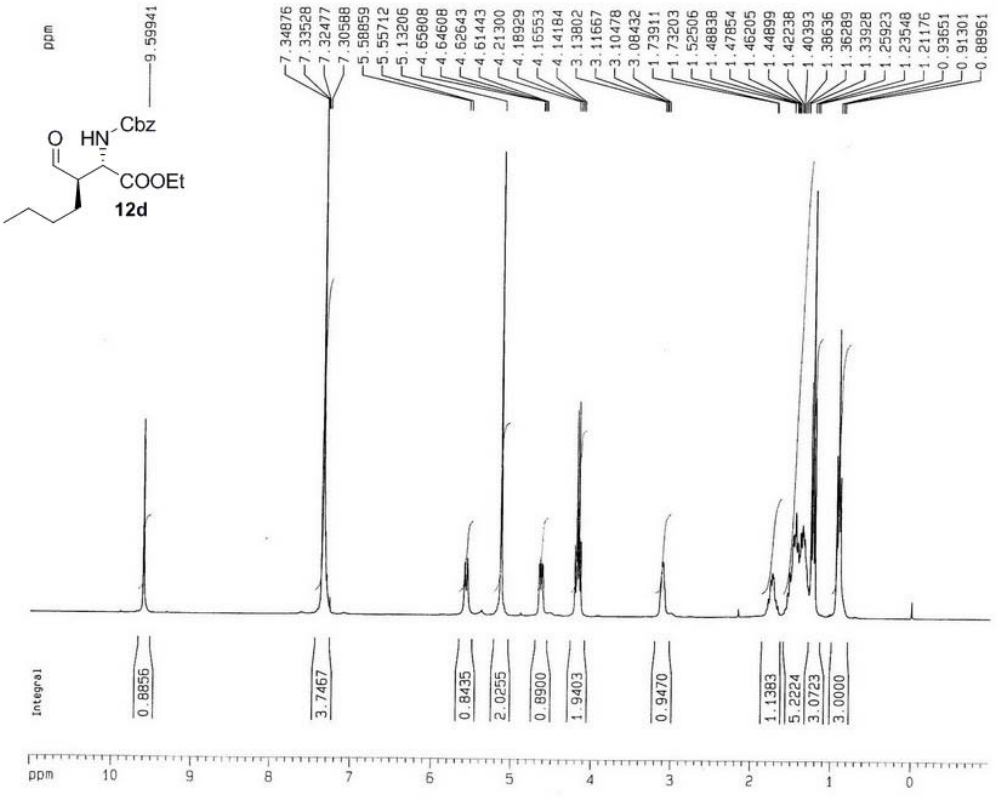
F2 - Acquisition Parameters
Date_              20110518
Time                   20.34
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                 zgdc
TD                     63488
SOLVENT                CDCl3
NS                       523
DS                         0
SWH              22222.223 Hz
FIDRES               0.350022 Hz
AQ                 1.4285300 sec
RG                      18390.4
DW                    22.500 usec
DE                     4.50 usec
TE                    673.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        11

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.10 usec
PL1                    -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                100.00 usec
PL2                    4.00 dB
PL12                  23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                      131072
SF                75.4778106 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                      7.15 cm
F1P                  220.000 ppm
F1                  16605.12 Hz
F2P                  -10.000 ppm
F2                  -754.78 Hz
PPMCM               11.50000 ppm/cm
HZCM               867.99487 Hz/cm
```



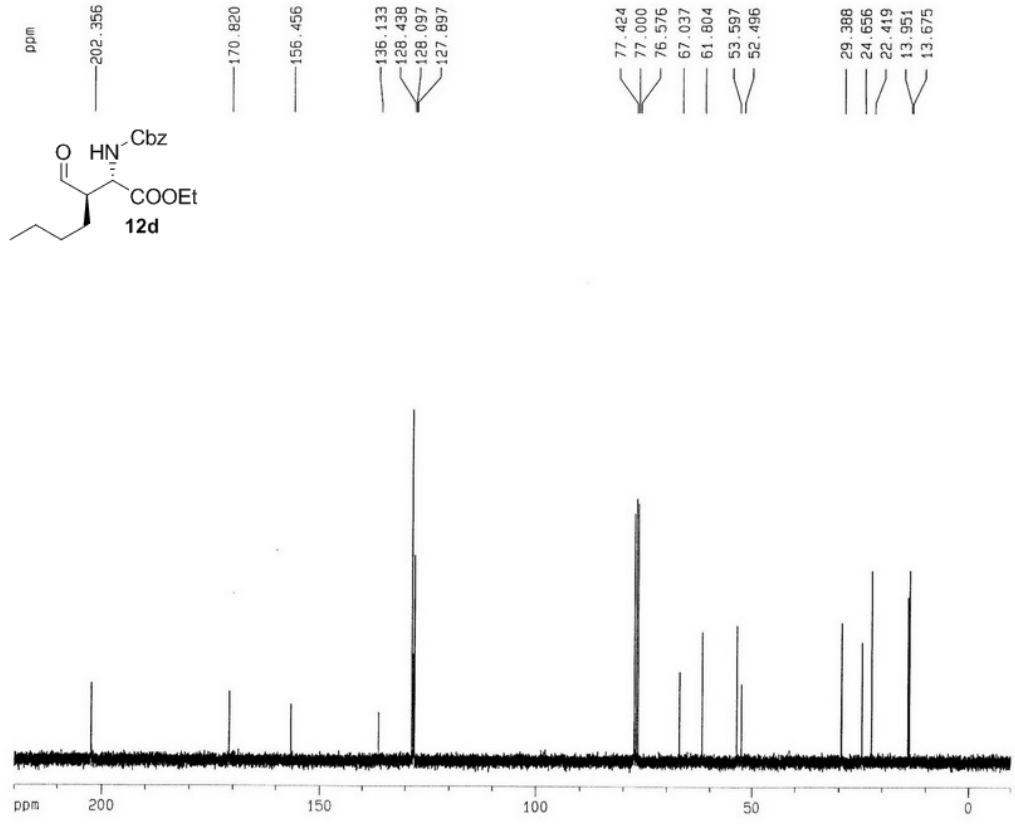

```
Current Data Parameters
NAME                  LJL-691
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110802
Time                  10.56
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        49
DS                         0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                      35.9
DW                   83.400 usec
DE                    6.00 usec
TE                   295.2 K
D1                 2.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      10.30 usec
PL1                      4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                      16384
SF                300.1700029 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      20.00 cm
CY                      12.50 cm
F1P                    11.000 ppm
F1                   3301.87 Hz
F2P                   -1.000 ppm
F2                  -300.17 Hz
PPMCM                0.60000 ppm/cm
HZCM               180.10201 Hz/cm
```




```
Current Data Parameters
NAME                  LJL-691
EXPNO                       2
PROCNO                      1

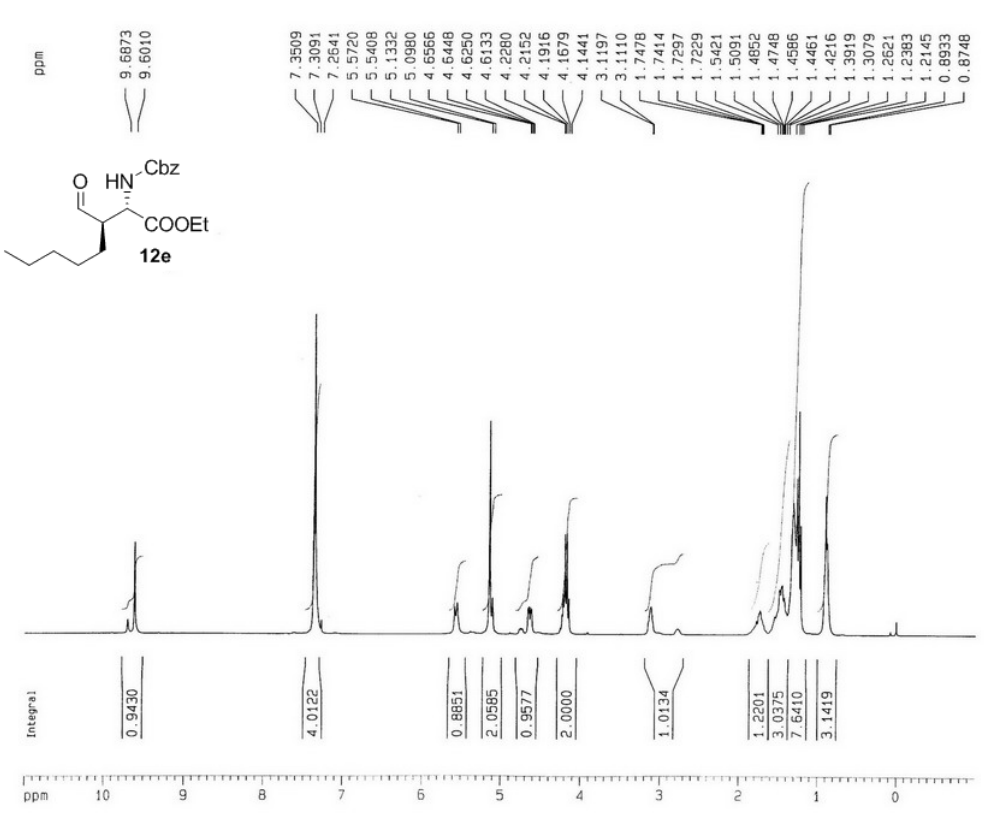
F2 - Acquisition Parameters
Date_              20110802
Time                  11.04
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                zgdc
TD                     63488
SOLVENT                CDCl3
NS                       253
DS                         0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                   18390.4
DW                   22.500 usec
DE                    4.50 usec
TE                   295.7 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        11

======== CHANNEL f1 ========
NUC1                      13C
P1                      10.10 usec
PL1                      -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                      4.00 dB
PL12                    23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                     131072
SF                75.4778136 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                      20.00 cm
CY                       7.13 cm
F1P                   220.000 ppm
F1                  16605.12 Hz
F2P                  -10.000 ppm
F2                   -754.78 Hz
PPMCM               11.50000 ppm/cm
HZCM               867.99487 Hz/cm
```



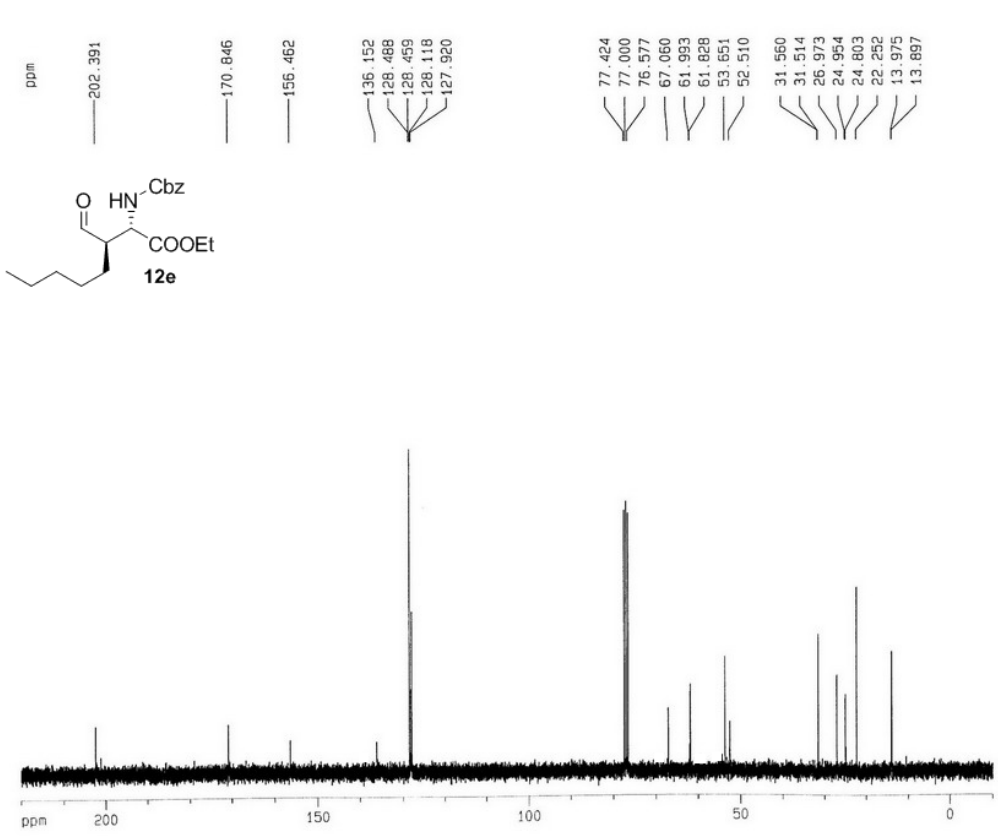
Current Data Parameters
NAME LJL-639
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110528
Time 20.17
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 49
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 35.9
DW 83.400 usec
DE 6.00 usec
TE 295.6 K
D1 2.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz

F2 - Processing parameters
SI 16384
SF 300.1700033 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 6.71 cm
F1P 11.000 ppm
F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.10201 Hz/cm



Current Data Parameters
NAME LJL-639
EXPNO 2
PROCNO 1

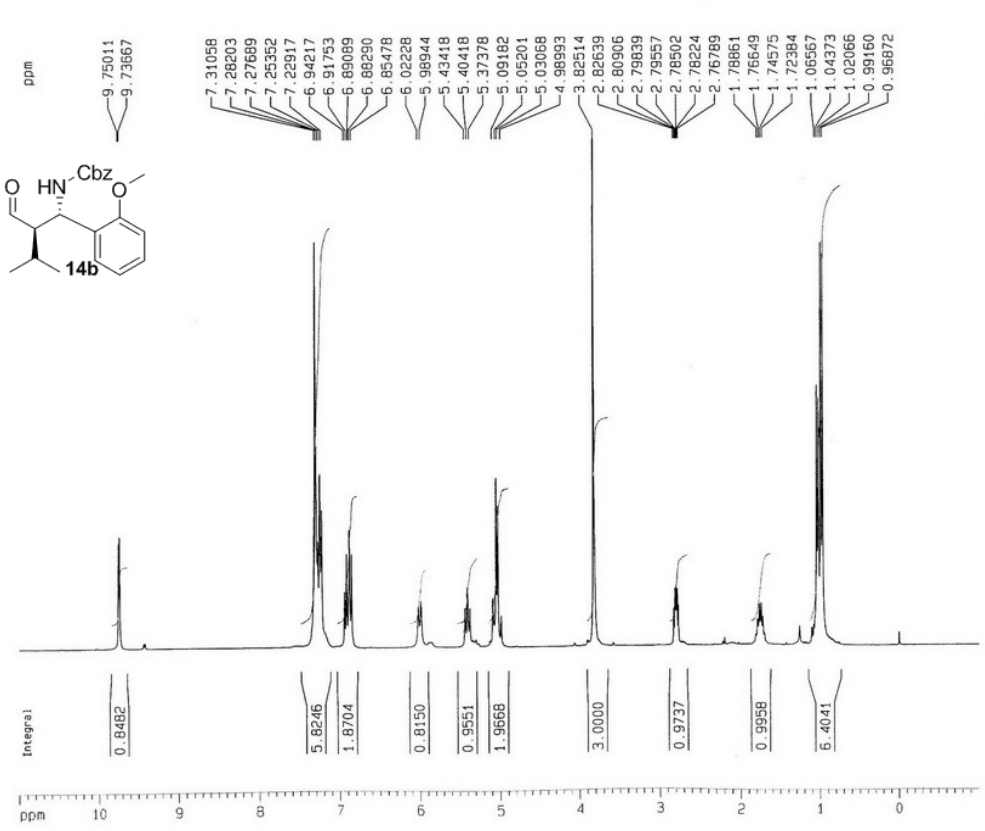
F2 - Acquisition Parameters
Date_ 20110528
Time 20.23
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 63488
SOLVENT CDCl3
NS 200
DS 0
SWH 22222.223 Hz
FIDRES 0.350022 Hz
AQ 1.4285300 sec
RG 18390.4
DW 22.500 usec
DE 4.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 11

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 -2.00 dB
SFO1 75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 23.74 dB
SFO2 300.1712007 MHz

F2 - Processing parameters
SI 131072
SF 75.4778123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.71 cm
F1P 220.000 ppm
F1 16605.12 Hz
F2P -10.000 ppm
F2 -754.78 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.99487 Hz/cm



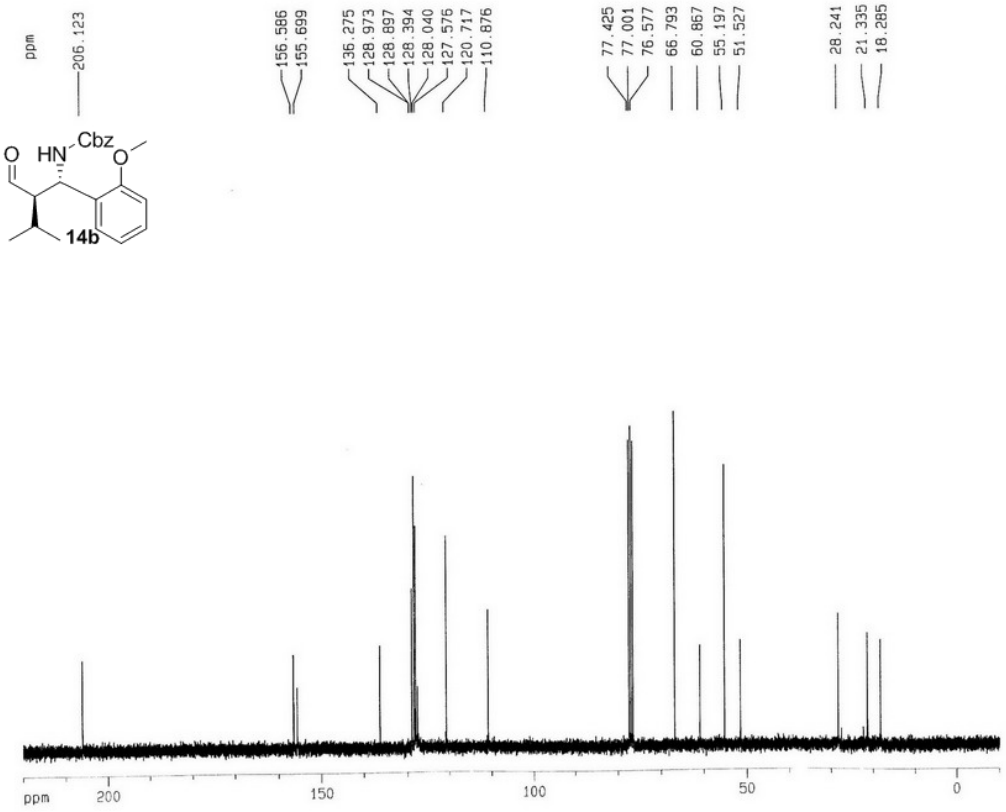
Current Data Parameters
NAME LJL-684
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110705
Time 20.21
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 50
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 35.9
DW 83.400 usec
DE 6.00 usec
TE 673.2 K
D1 2.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz

F2 - Processing parameters
SI 16384
SF 300.1700095 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.46 cm
F1P 11.000 ppm
F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.10201 Hz/cm



Current Data Parameters
NAME LJL-684
EXPNO 2
PROCNO 1

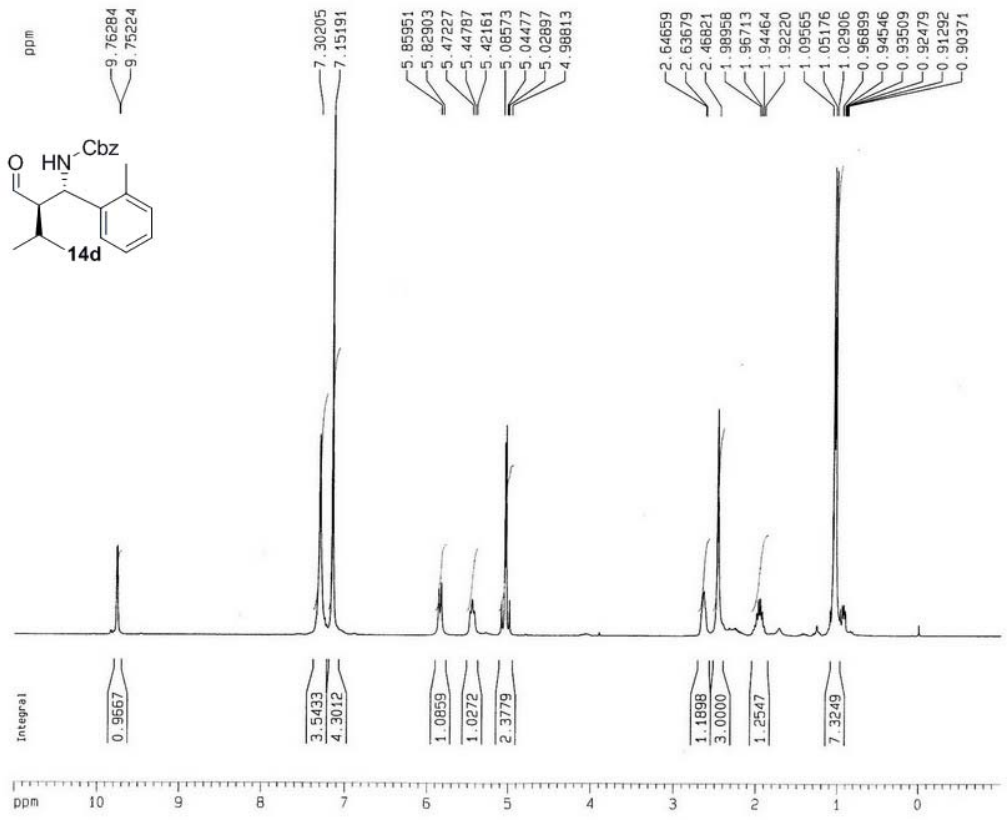
F2 - Acquisition Parameters
Date_ 20110705
Time 20.29
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 63488
SOLVENT CDCl3
NS 307
DS 0
SWH 22222.223 Hz
FIDRES 0.350022 Hz
AQ 1.4285300 sec
RG 5160.6
DW 22.500 usec
DE 4.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 11

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 -2.00 dB
SFO1 75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 23.74 dB
SFO2 300.1712007 MHz

F2 - Processing parameters
SI 131072
SF 75.4778155 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.86 cm
F1P 220.000 ppm
F1 16605.12 Hz
F2P -10.000 ppm
F2 -754.78 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.99487 Hz/cm



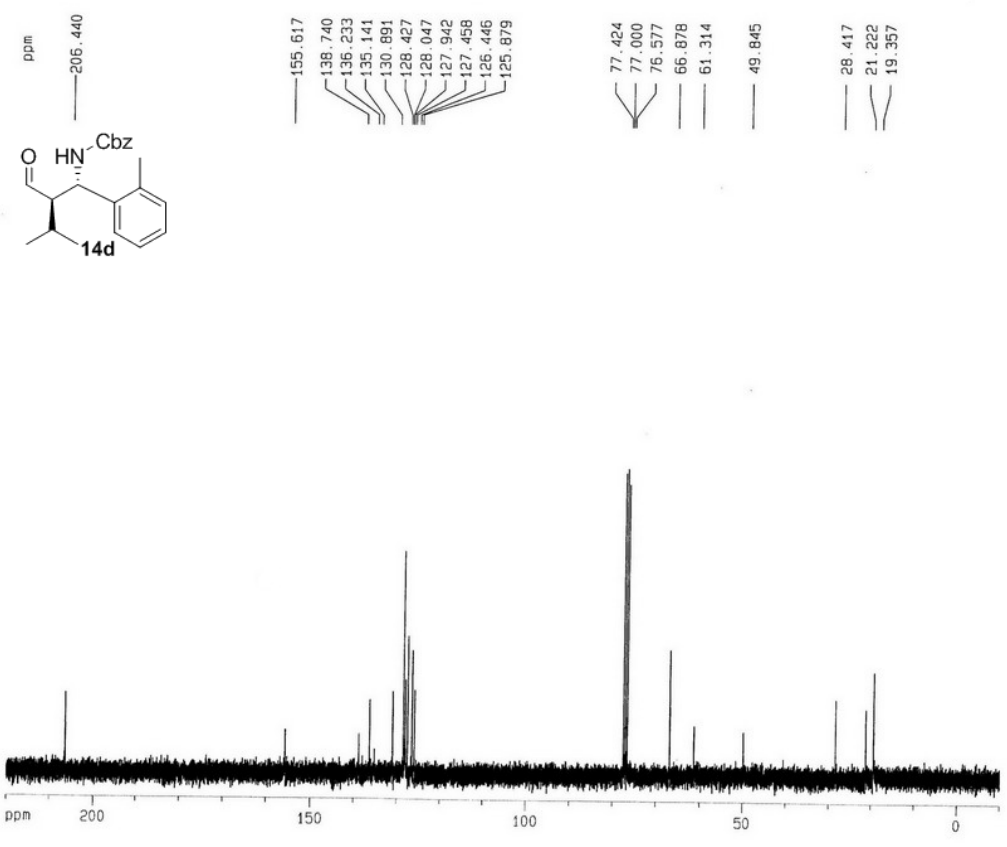
Current Data Parameters
NAME LJL680
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110630
Time 20.39
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 31
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 35.9
DW 83.400 usec
DE 6.00 usec
TE 673.2 K
D1 2.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz

F2 - Processing parameters
SI 16384
SF 300.1700098 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 13.09 cm
F1P 11.000 ppm
F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.10201 Hz/cm



Current Data Parameters
NAME LJL680
EXPNO 2
PROCNO 1

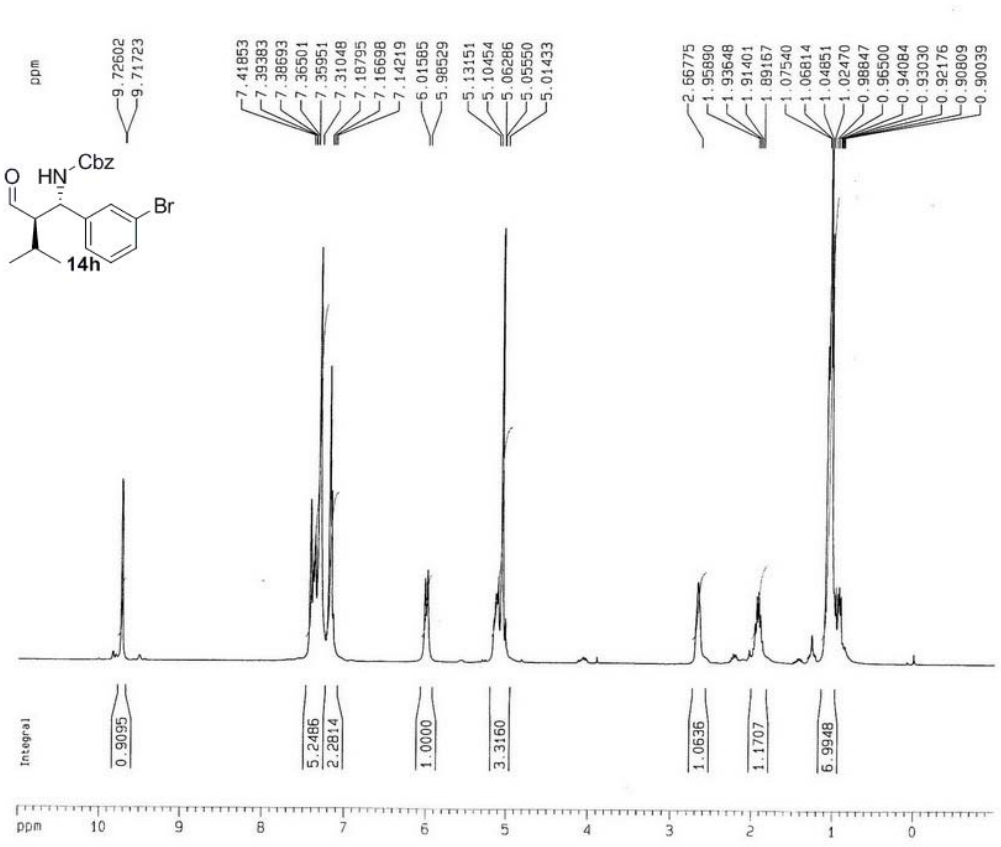
F2 - Acquisition Parameters
Date_ 20110630
Time 20.44
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 63488
SOLVENT CDCl3
NS 144
DS 0
SWH 22222.223 Hz
FIDRES 0.350022 Hz
AQ 1.4285300 sec
RG 5792.6
DW 22.500 usec
DE 4.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 11

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 -2.00 dB
SFO1 75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 23.74 dB
SFO2 300.1712007 MHz

F2 - Processing parameters
SI 131072
SF 75.4778123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.16 cm
F1P 220.000 ppm
F1 16605.12 Hz
F2P -10.000 ppm
F2 -754.78 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.99487 Hz/cm



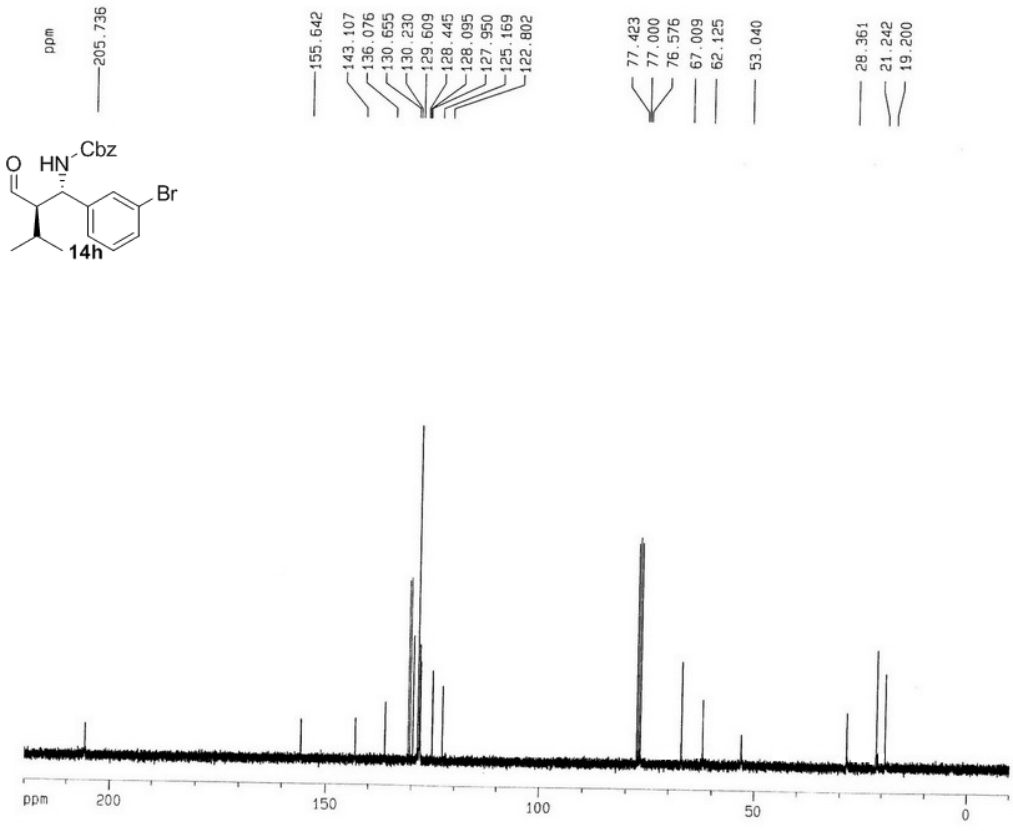
Current Data Parameters
NAME LJL679-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110628
Time 20.36
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 50
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 35.9
DW 83.400 usec
DE 6.00 usec
TE 297.4 K
D1 2.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz

F2 - Processing parameters
SI 16384
SF 300.1700080 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.08 cm
F1P 11.000 ppm
F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.10201 Hz/cm



Current Data Parameters
NAME LJL679-1
EXPNO 2
PROCNO 1

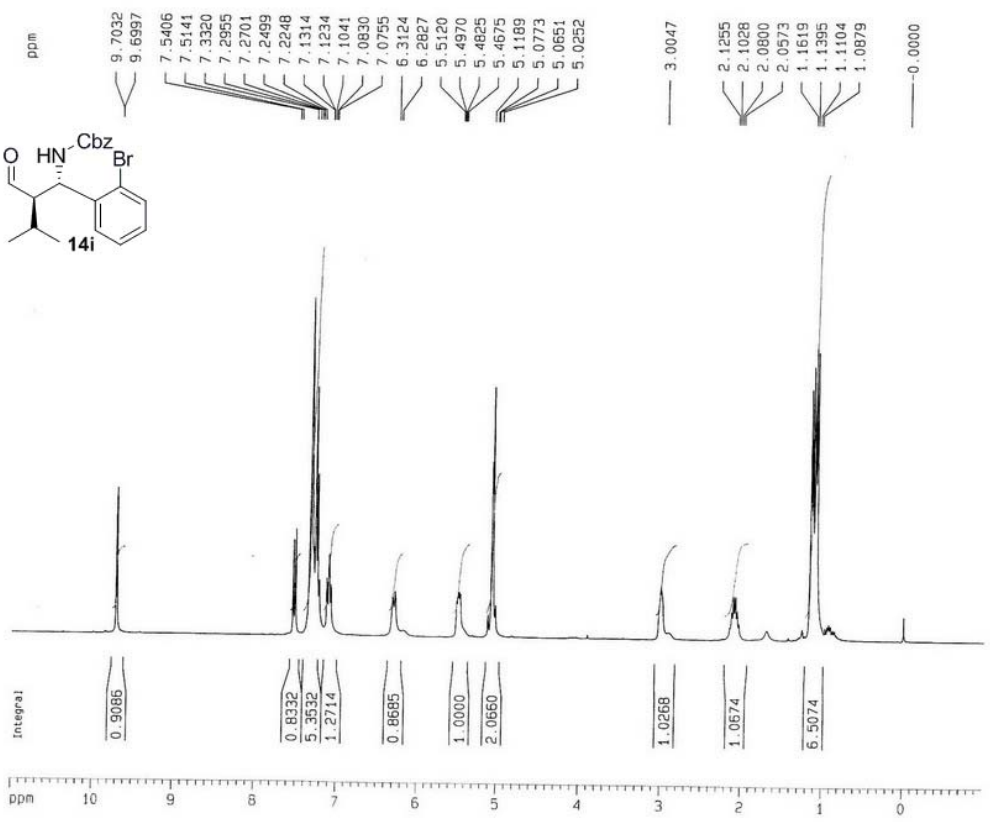
F2 - Acquisition Parameters
Date_ 20110628
Time 20.44
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 63488
SOLVENT CDCl3
NS 557
DS 0
SWH 22222.223 Hz
FIDRES 0.350022 Hz
AQ 1.4285300 sec
RG 16390.4
DW 22.500 usec
DE 4.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 11

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 -2.00 dB
SFO1 75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 23.74 dB
SFO2 300.1712007 MHz

F2 - Processing parameters
SI 131072
SF 75.4778140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.79 cm
F1P 220.000 ppm
F1 16605.12 Hz
F2P -10.000 ppm
F2 -754.78 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.99487 Hz/cm



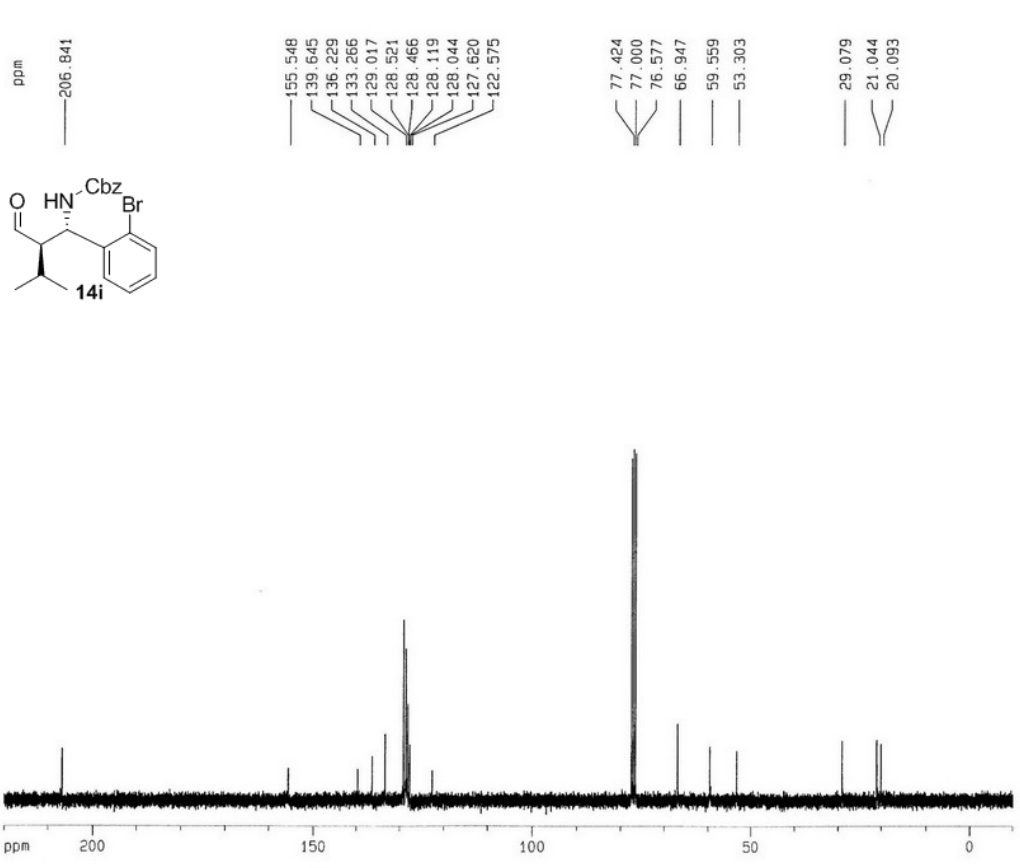

```
Current Data Parameters
NAME                  LJL-685
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110706
Time                  20.43
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        61
DS                         0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                      71.8
DW                    83.400 usec
DE                     6.00 usec
TE                    673.2 K
D1                 2.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                     10.30 usec
PL1                     4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                      16384
SF                300.1700077 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                    20.00 cm
CY                     6.91 cm
F1P                  11.000 ppm
F1                  3301.87 Hz
F2P                 -1.000 ppm
F2                  -300.17 Hz
PPMCM             0.60000 ppm/cm
HZCM              180.10201 Hz/cm
```




```
Current Data Parameters
NAME                  LJL-685
EXPNO                       2
PROCNO                      1

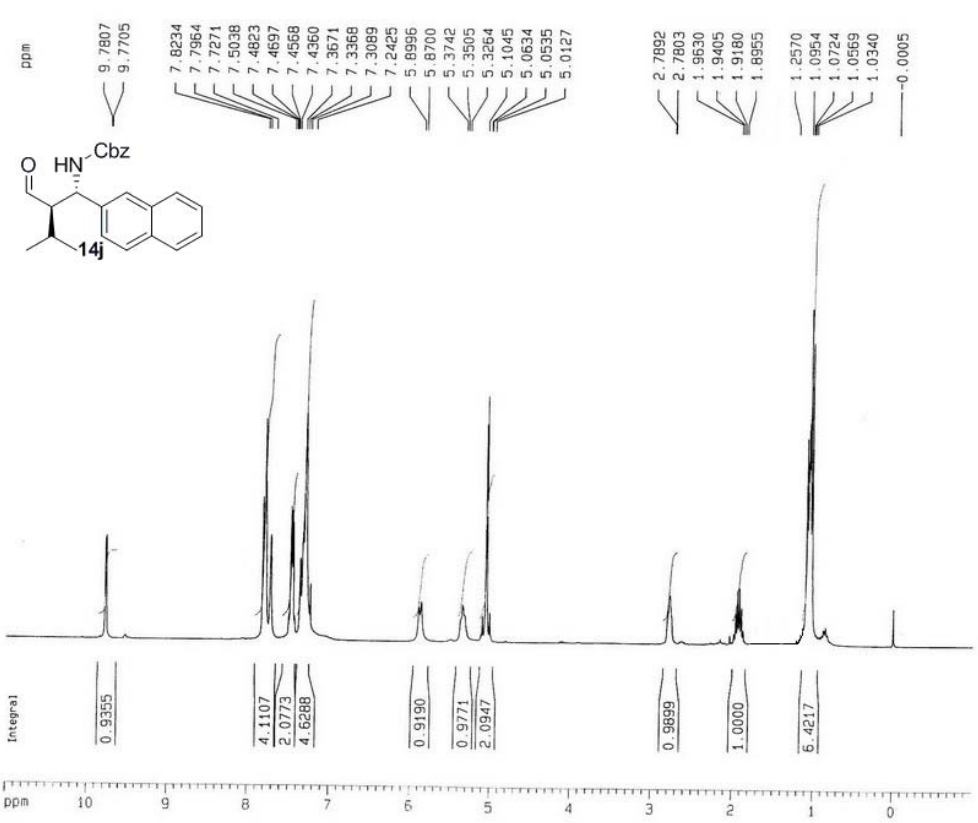
F2 - Acquisition Parameters
Date_              20110706
Time                  20.46
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                zgdc
TD                     63488
SOLVENT                CDCl3
NS                        402
DS                         0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                     9195.2
DW                    22.500 usec
DE                     4.50 usec
TE                    673.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        11

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.10 usec
PL1                     -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                     4.00 dB
PL12                   23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                     131072
SF                75.4778121 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     6.98 cm
F1P                  220.000 ppm
F1                  16605.12 Hz
F2P                 -10.000 ppm
F2                  -754.78 Hz
PPMCM             11.50000 ppm/cm
HZCM              867.99487 Hz/cm
```



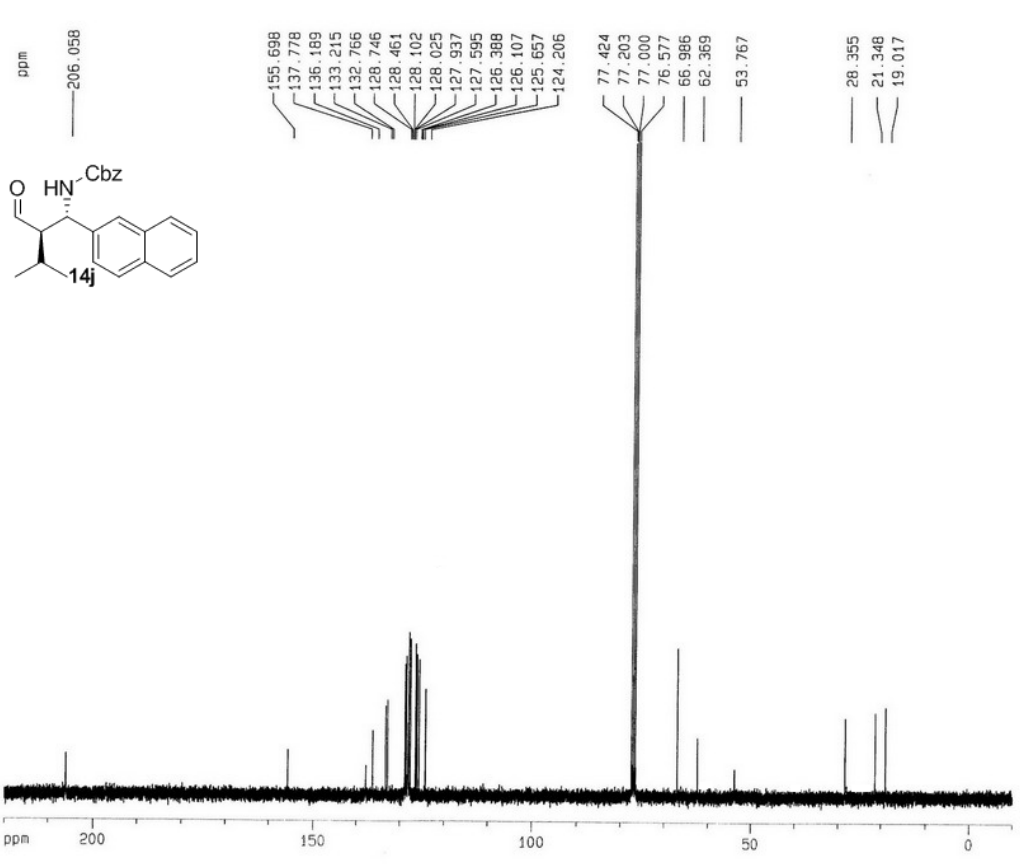

```
Current Data Parameters
NAME                  LJL-686
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110707
Time                   16.08
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                    zg
TD                      32768
SOLVENT                CDCl3
NS                         47
DS                          0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                       71.8
DW                     83.400 usec
DE                      6.00 usec
TE                     296.3 K
D1                 2.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                     10.30 usec
PL1                     4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                      16384
SF                300.1700098 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                      6.92 cm
F1P                   11.000 ppm
F1                   3301.87 Hz
F2P                   -1.000 ppm
F2                    -300.17 Hz
PPMCM                0.60000 ppm/cm
HZCM                180.10201 Hz/cm
```




```
Current Data Parameters
NAME                  LJL-686
EXPNO                       2
PROCNO                      1

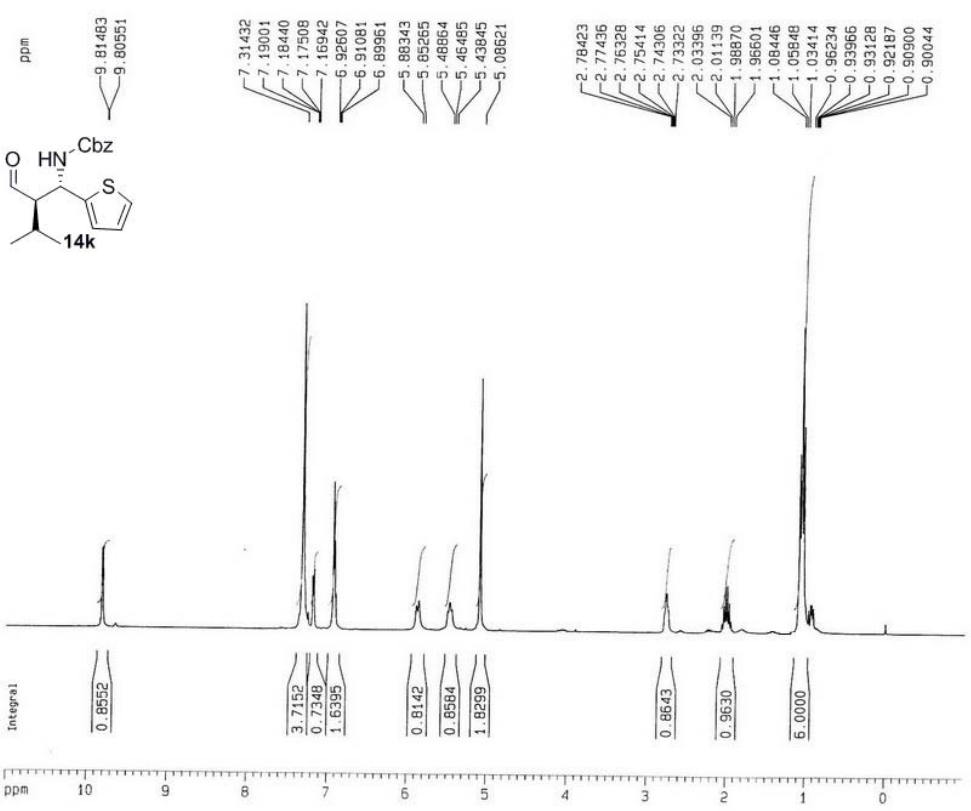
F2 - Acquisition Parameters
Date_              20110707
Time                   16.15
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                 zgdc
TD                      63488
SOLVENT                CDCl3
NS                       1025
DS                          0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                      13004
DW                     22.500 usec
DE                      4.50 usec
TE                     296.3 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        11

======== CHANNEL f1 ========
NUC1                       13C
P1                     10.10 usec
PL1                     -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                     4.00 dB
PL12                   23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                     131072
SF                75.4778111 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                     13.45 cm
F1P                  220.000 ppm
F1                   16605.12 Hz
F2P                  -10.000 ppm
F2                    -754.78 Hz
PPMCM               11.50000 ppm/cm
HZCM               867.99487 Hz/cm
```



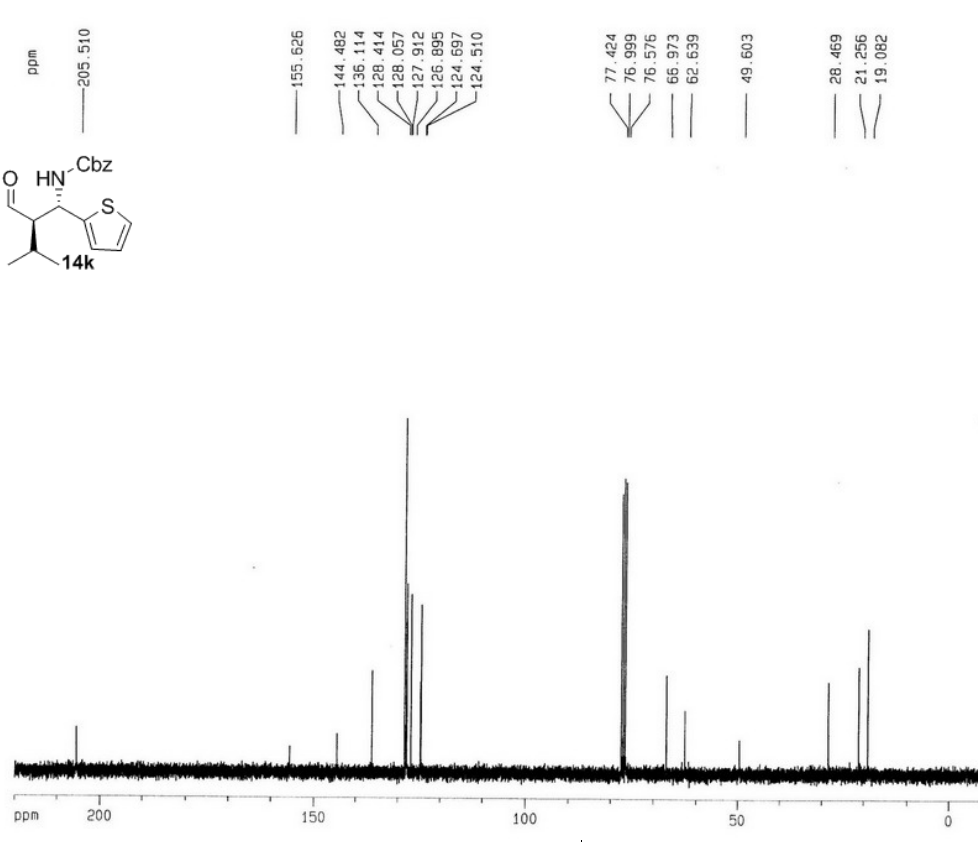

```
Current Data Parameters
NAME                  LJL-698
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110810
Time                  21.18
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        46
DS                         0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                      35.9
DW                    83.400 usec
DE                     6.00 usec
TE                    296.1 K
D1                 2.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    10.30 usec
PL1                    4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                      16384
SF                300.1700091 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                    20.00 cm
CY                     6.75 cm
F1P                  11.000 ppm
F1                  3301.87 Hz
F2P                  -1.000 ppm
F2                  -300.17 Hz
PPMCM              0.60000 ppm/cm
HZCM              180.10201 Hz/cm
```




```
Current Data Parameters
NAME                  LJL-698
EXPNO                       2
PROCNO                      1

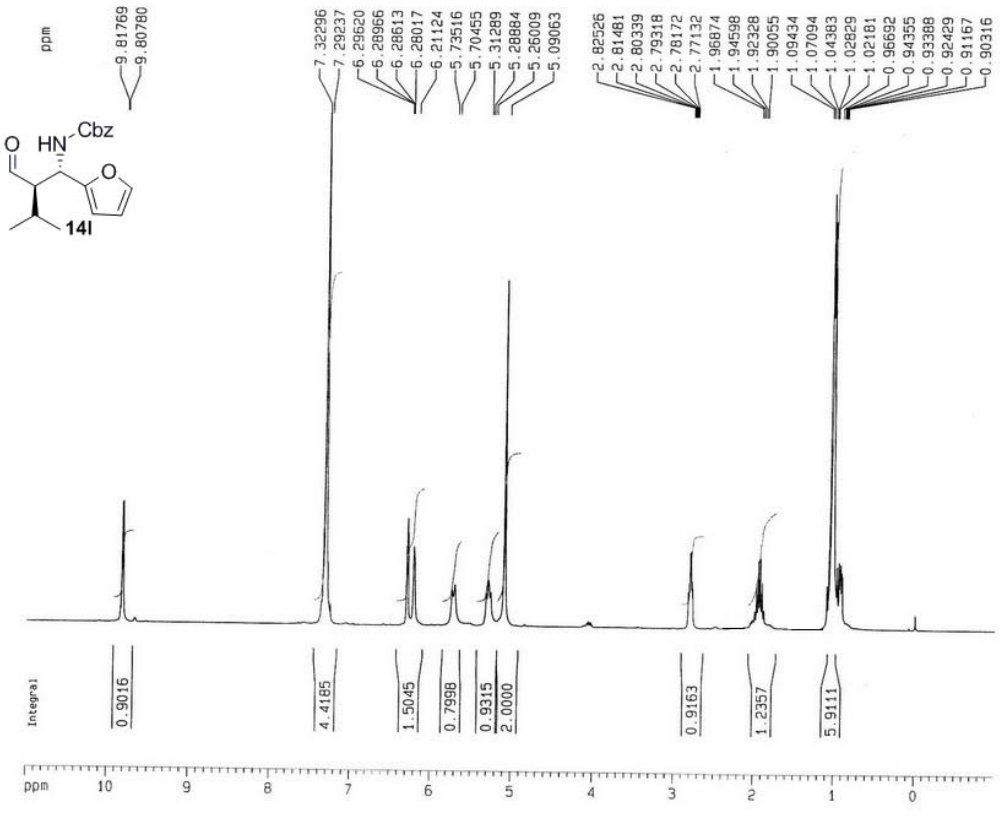
F2 - Acquisition Parameters
Date_              20110810
Time                  20.56
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                zgdc
TD                     63488
SOLVENT                CDCl3
NS                       301
DS                         0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                   18390.4
DW                    22.500 usec
DE                     4.50 usec
TE                    296.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.10 usec
PL1                   -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                100.00 usec
PL2                    4.00 dB
PL12                  23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                     131072
SF                75.4778157 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     7.29 cm
F1P                  220.000 ppm
F1                  16605.12 Hz
F2P                  -10.000 ppm
F2                  -754.78 Hz
PPMCM              11.50000 ppm/cm
HZCM              867.99487 Hz/cm
```



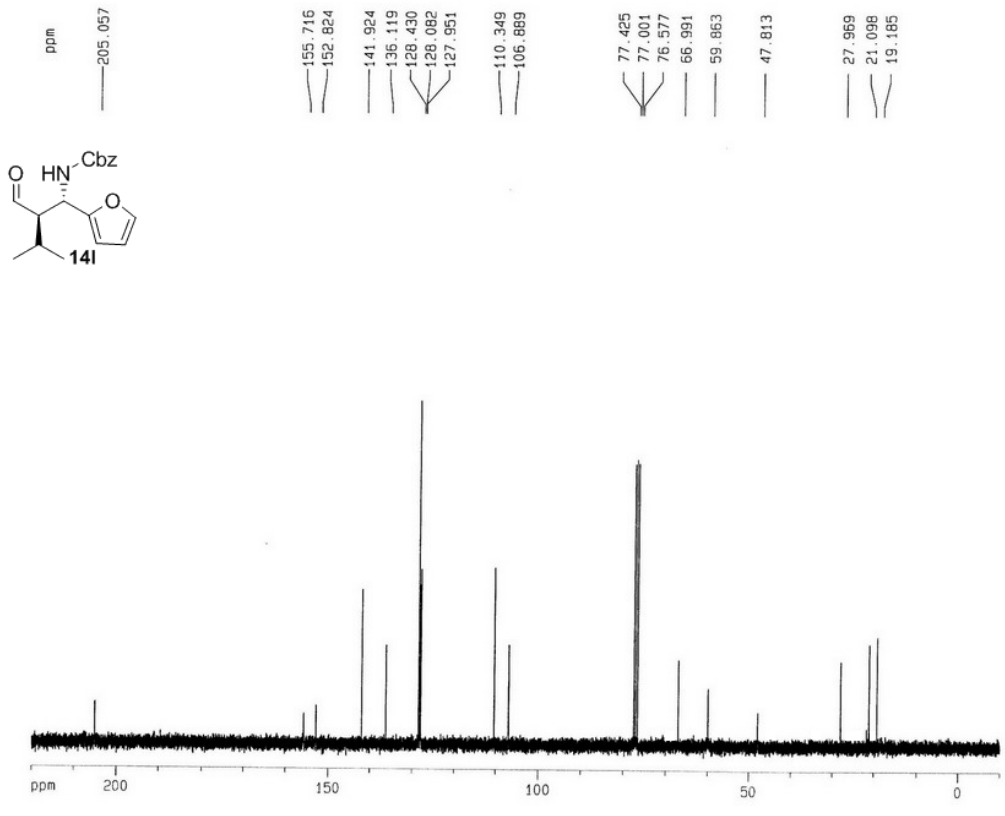

```
Current Data Parameters
NAME                  LJL-700
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110812
Time                   16.59
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                    zg
TD                      32768
SOLVENT                CDCl3
NS                         51
DS                          0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                      35.9
DW                   83.400 usec
DE                    6.00 usec
TE                    296.6 K
D1                 2.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    10.30 usec
PL1                    4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                      16384
SF                300.1700062 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    12.44 cm
F1P                  11.000 ppm
F1                  3301.87 Hz
F2P                  -1.000 ppm
F2                  -300.17 Hz
PPMCM              0.60000 ppm/cm
HZCM              180.10201 Hz/cm
```




```
Current Data Parameters
NAME                  LJL-700
EXPNO                       2
PROCNO                      1

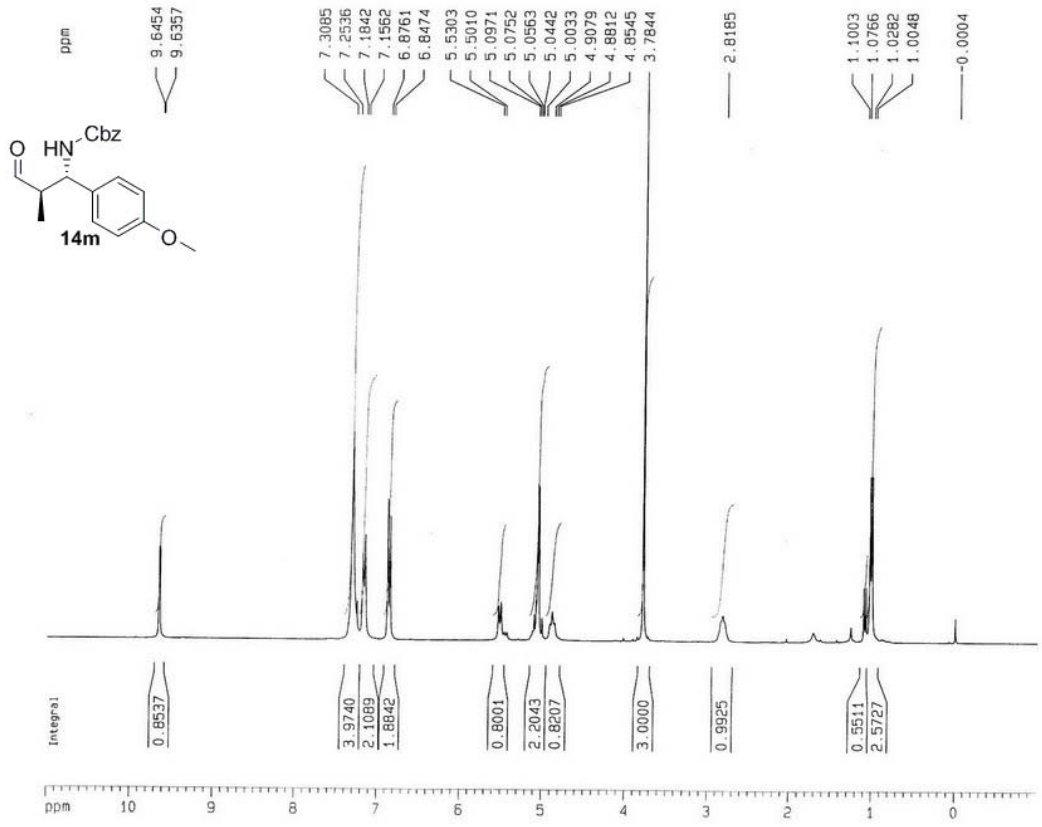
F2 - Acquisition Parameters
Date_              20110812
Time                   17.06
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                 zgdc
TD                      63488
SOLVENT                CDCl3
NS                        310
DS                          0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                    18390.4
DW                   22.500 usec
DE                    4.50 usec
TE                    296.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                    10.10 usec
PL1                   -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                100.00 usec
PL2                    4.00 dB
PL12                  23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                     131072
SF                75.4778138 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     7.09 cm
F1P                  220.000 ppm
F1                  16605.12 Hz
F2P                  -10.000 ppm
F2                  -754.78 Hz
PPMCM              11.50000 ppm/cm
HZCM              867.99487 Hz/cm
```



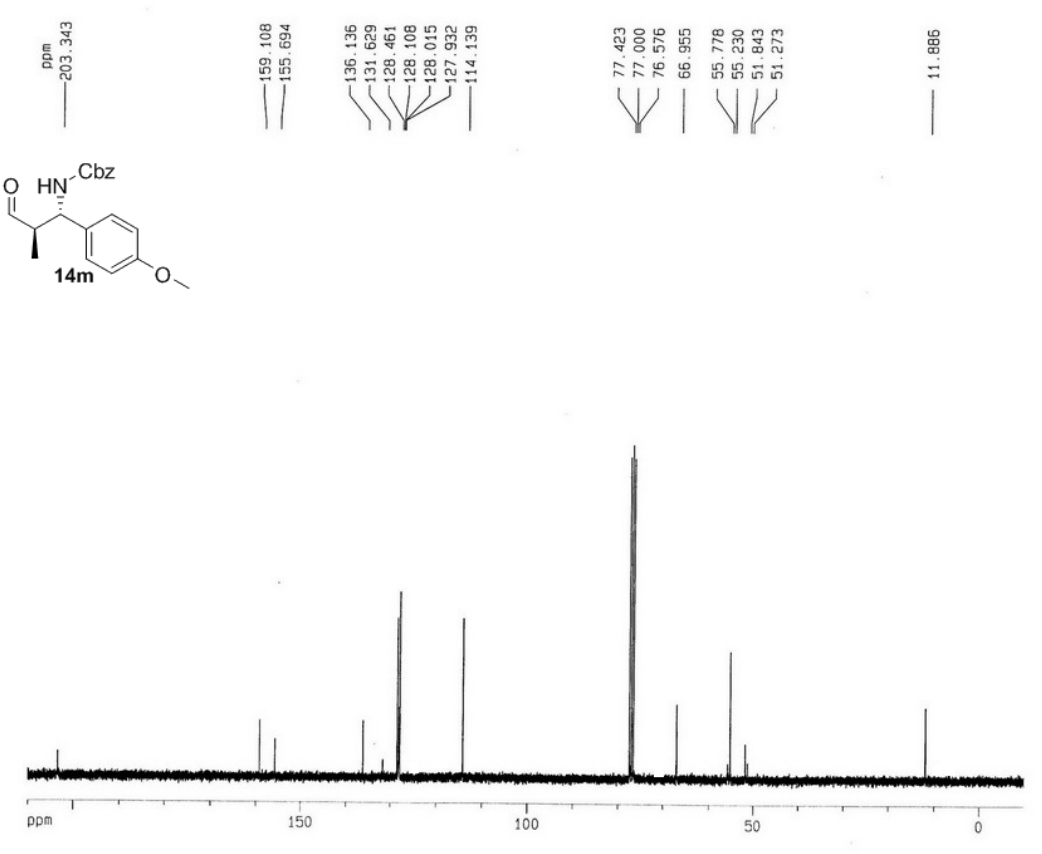

```
Current Data Parameters
NAME                  LJL-681-1
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_              20110707
Time                  15.14
INSTRUM               av300
PROBHD   5 mm Multinucl
PULPROG                  zg
TD                    32768
SOLVENT               CDCl3
NS                       48
DS                        0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                     71.8
DW                   83.400 usec
DE                    6.00 usec
TE                   296.4 K
D1                 2.50000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.30 usec
PL1                    4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                    16384
SF                300.1700066 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    12.41 cm
F1P                  11.000 ppm
F1                 3301.87 Hz
F2P                 -1.000 ppm
F2                 -300.17 Hz
PPMCM              0.60000 ppm/cm
HZCM              180.10201 Hz/cm
```




```
Current Data Parameters
NAME                  LJL-681-1
EXPNO                         2
PROCNO                        1

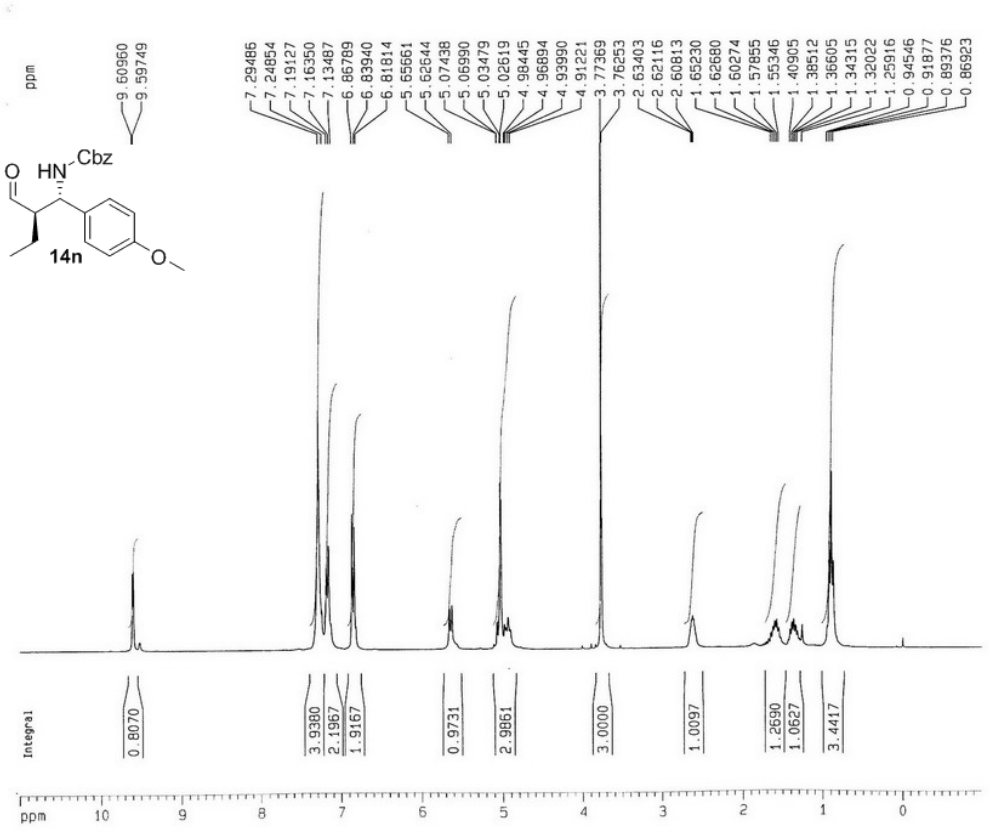
F2 - Acquisition Parameters
Date_              20110707
Time                  15.21
INSTRUM               av300
PROBHD   5 mm Multinucl
PULPROG                zgdc
TD                    63488
SOLVENT               CDCl3
NS                      722
DS                        0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                   18390.4
DW                   22.500 usec
DE                    4.50 usec
TE                   296.3 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                      11

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.10 usec
PL1                   -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                    4.00 dB
PL12                  23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                   131072
SF                75.4778114 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     6.72 cm
F1P                  210.000 ppm
F1                 15850.34 Hz
F2P                 -10.000 ppm
F2                 -754.78 Hz
PPMCM              11.00000 ppm/cm
HZCM              830.25592 Hz/cm
```



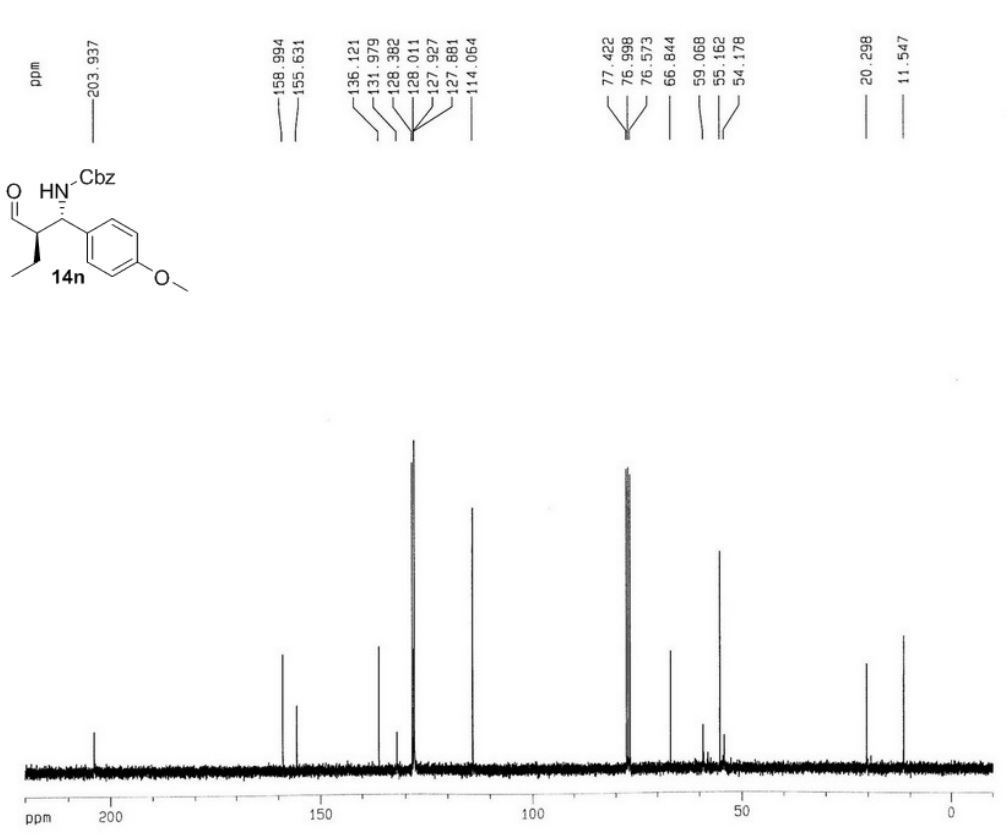

```
Current Data Parameters
NAME                  LJL673
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110625
Time                  10.27
INSTRUM                av300
PROBHD    5 mm Multinucl
PULPROG                  zg
TD                    32768
SOLVENT               CDCl3
NS                       52
DS                        0
SWH                5995.204 Hz
FIDRES            0.182959 Hz
AQ                2.7329011 sec
RG                       32
DW                  83.400 usec
DE                   6.00 usec
TE                   294.6 K
D1                2.50000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   10.30 usec
PL1                   4.00 dB
SFO1             300.1718010 MHz

F2 - Processing parameters
SI                       16384
SF                300.1700080 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   13.17 cm
F1P                 11.000 ppm
F1                 3301.87 Hz
F2P                -1.000 ppm
F2                 -300.17 Hz
PPMCM              0.60000 ppm/cm
HZCM             180.10201 Hz/cm
```




```
Current Data Parameters
NAME                  LJL673
EXPNO                      2
PROCNO                     1

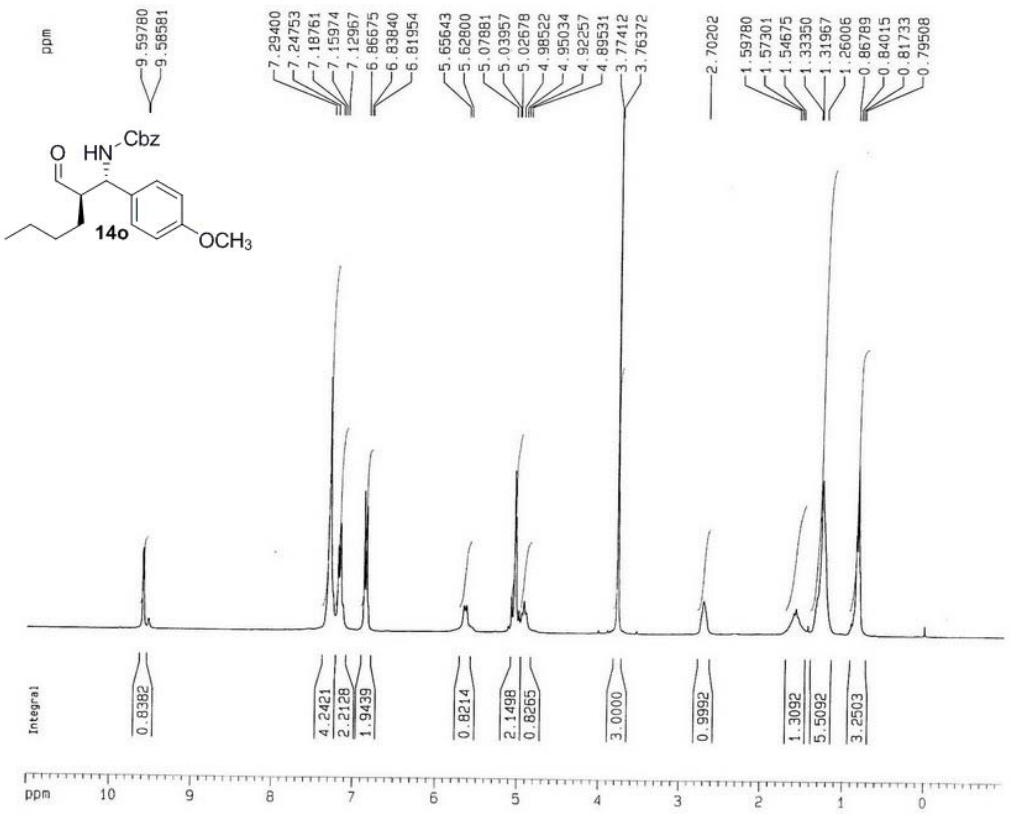
F2 - Acquisition Parameters
Date_              20110625
Time                  10.36
INSTRUM                av300
PROBHD    5 mm Multinucl
PULPROG                zgdc
TD                    63488
SOLVENT               CDCl3
NS                      407
DS                        0
SWH               22222.223 Hz
FIDRES            0.350022 Hz
AQ                1.4285300 sec
RG                   18390.4
DW                  22.500 usec
DE                   4.50 usec
TE                   294.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      11

======== CHANNEL f1 ========
NUC1                     13C
P1                   10.10 usec
PL1                   -2.00 dB
SFO1             75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2               100.00 usec
PL2                   4.00 dB
PL12                 23.74 dB
SFO2             300.1712007 MHz

F2 - Processing parameters
SI                      131072
SF                75.4778160 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    6.84 cm
F1P                 220.000 ppm
F1                16605.12 Hz
F2P                -10.000 ppm
F2                 -754.78 Hz
PPMCM              11.50000 ppm/cm
HZCM             867.99487 Hz/cm
```



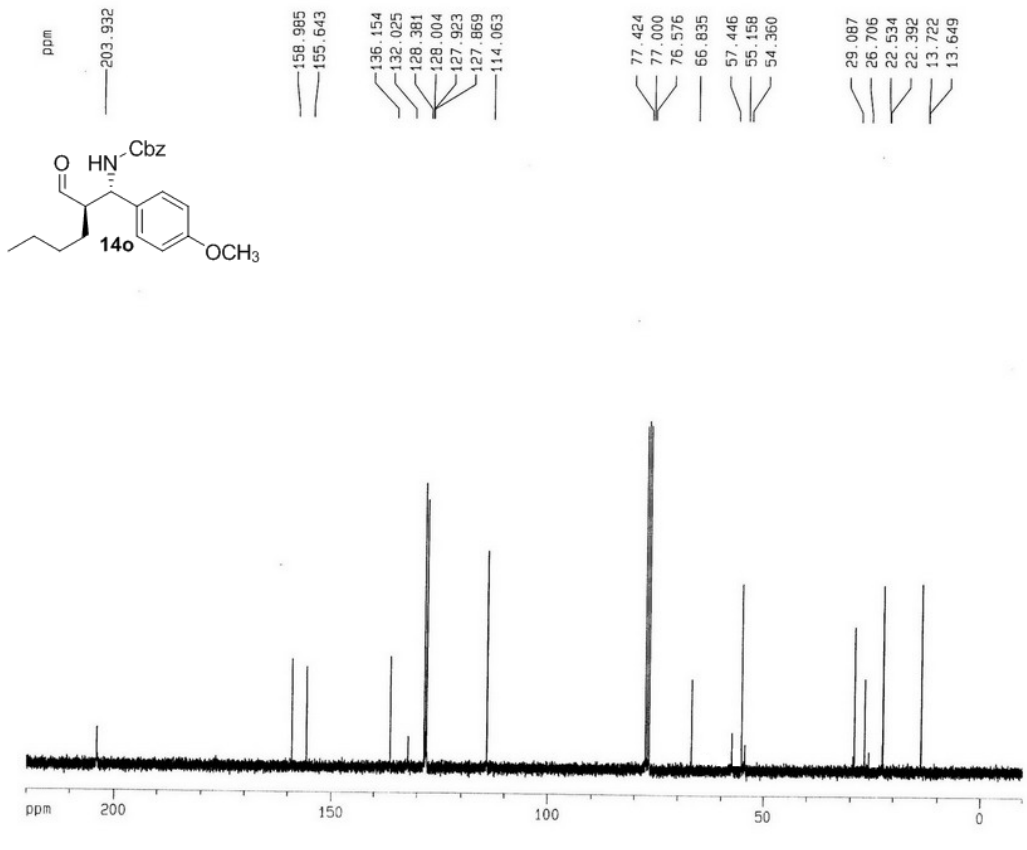

```
Current Data Parameters
NAME                  LJL-693
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110808
Time                   20.04
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        51
DS                         0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                        32
DW                   83.400 usec
DE                    6.00 usec
TE                    295.8 K
D1                 2.50000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                    10.30 usec
PL1                    4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                        16384
SF                 300.1700084 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    12.59 cm
F1P                  11.000 ppm
F1                  3301.87 Hz
F2P                 -1.000 ppm
F2                  -300.17 Hz
PPMCM              0.60000 ppm/cm
HZCM              180.10201 Hz/cm
```




```
Current Data Parameters
NAME                  LJL-693
EXPNO                       2
PROCNO                      1

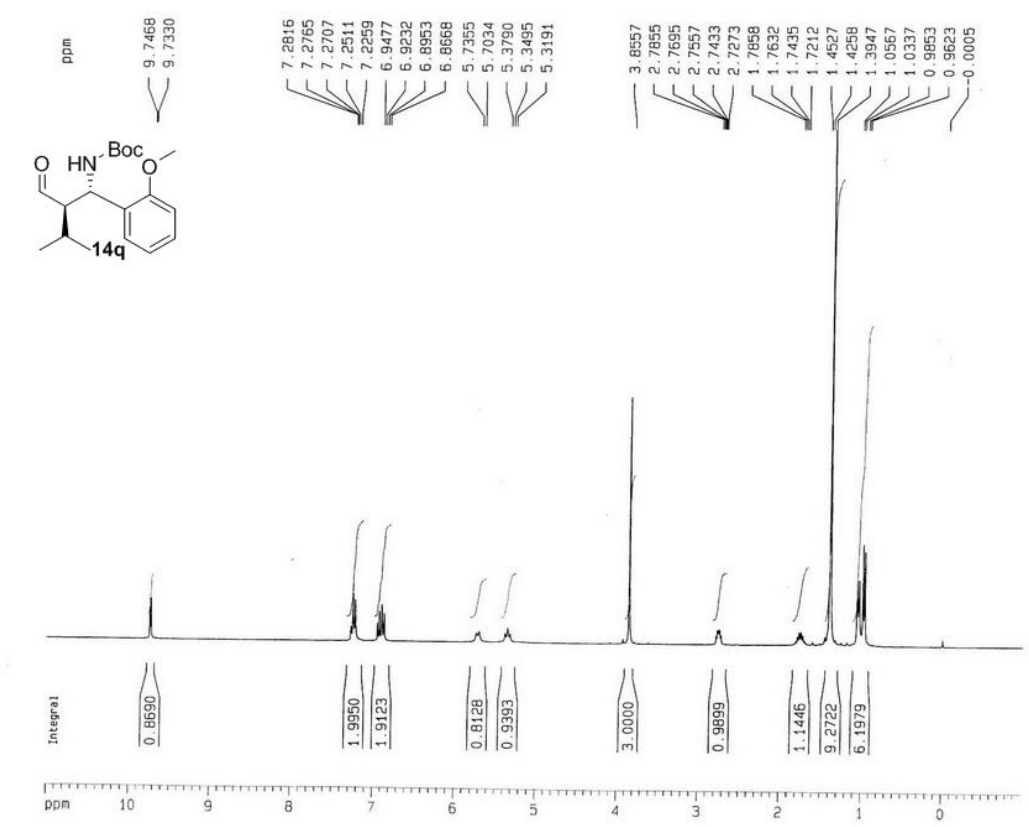
F2 - Acquisition Parameters
Date_              20110808
Time                   20.12
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                 zgdc
TD                     63488
SOLVENT                CDCl3
NS                       633
DS                         0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                     18390.4
DW                   22.500 usec
DE                    4.50 usec
TE                    296.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.10 usec
PL1                    -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                    4.00 dB
PL12                  23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                        131072
SF                 75.4778155 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     6.98 cm
F1P                 220.000 ppm
F1                  16605.12 Hz
F2P                -10.000 ppm
F2                  -754.78 Hz
PPMCM              11.50000 ppm/cm
HZCM              867.99487 Hz/cm
```



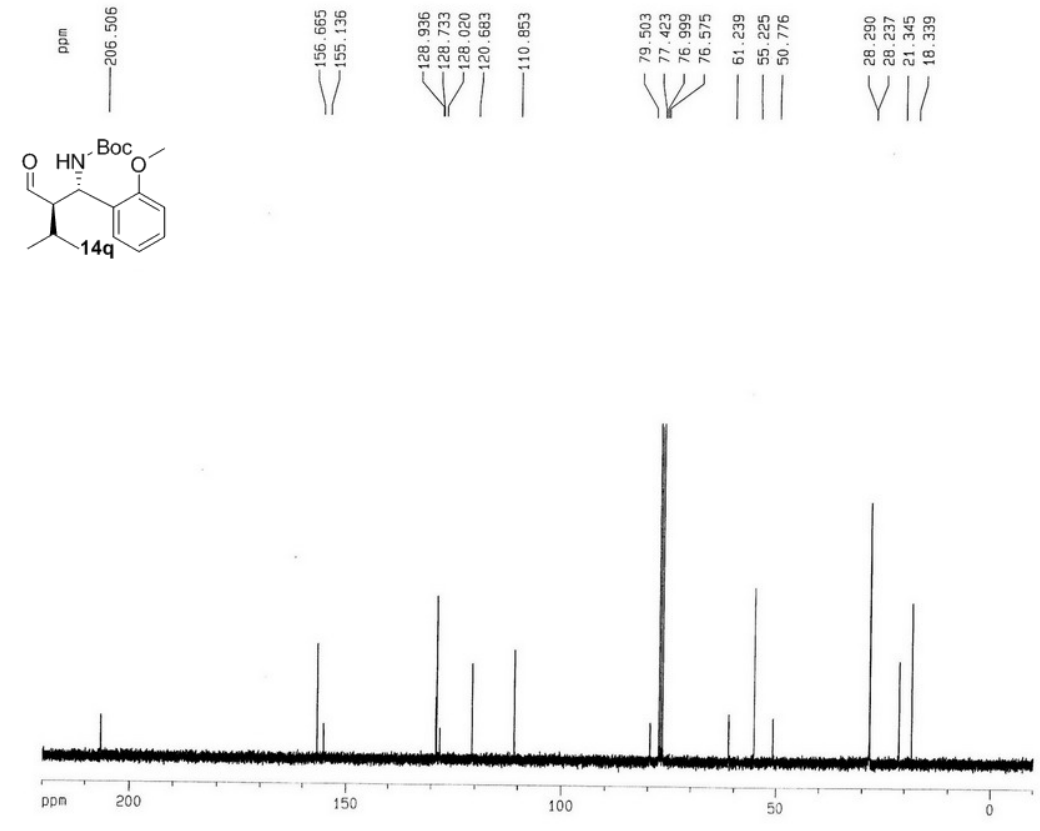
Current Data Parameters
NAME LJL-688
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110708
Time 15.19
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 51
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 35.9
DW 83.400 usec
DE 6.00 usec
TE 673.2 K
D1 2.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz

F2 - Processing parameters
SI 16384
SF 300.1700014 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.03 cm
F1P 11.000 ppm
F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.10201 Hz/cm



Current Data Parameters
NAME LJL-688
EXPNO 2
PROCNO 1

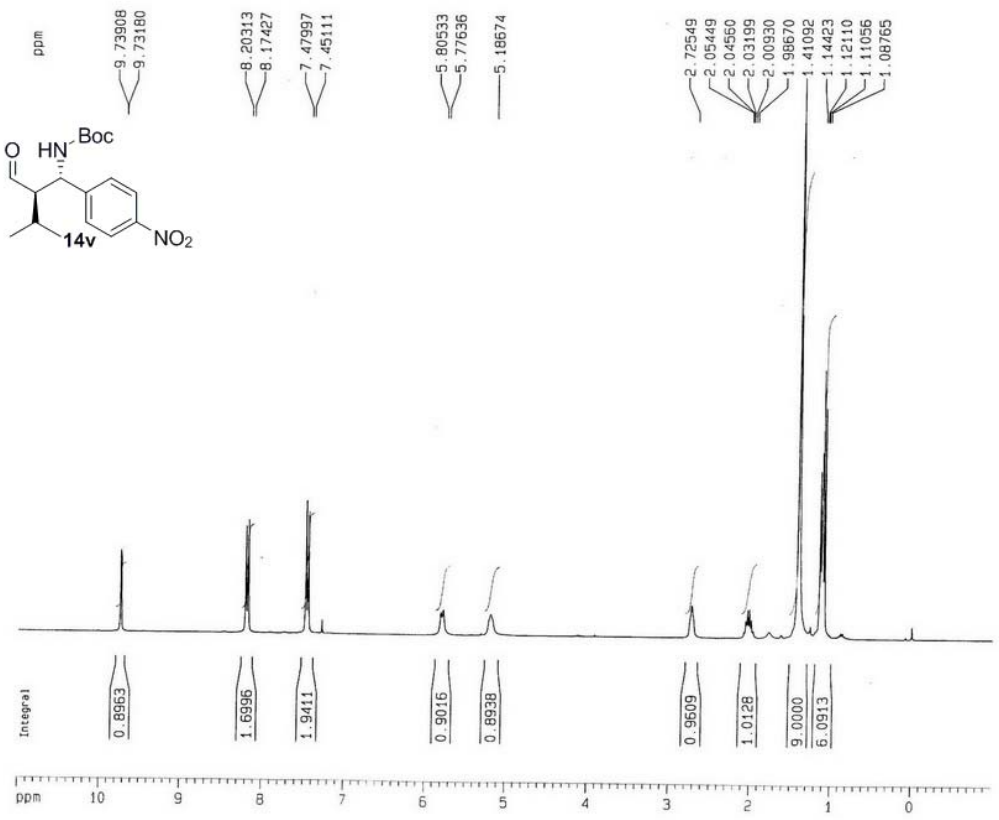
F2 - Acquisition Parameters
Date_ 20110708
Time 15.26
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 63488
SOLVENT CDCl3
NS 401
DS 0
SWH 22222.223 Hz
FIDRES 0.350022 Hz
AQ 1.4285300 sec
RG 8192
DW 22.500 usec
DE 4.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 11

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 -2.00 dB
SFO1 75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 23.74 dB
SFO2 300.1712007 MHz

F2 - Processing parameters
SI 131072
SF 75.4778106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.84 cm
F1P 220.000 ppm
F1 16605.12 Hz
F2P -10.000 ppm
F2 -754.78 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.99487 Hz/cm



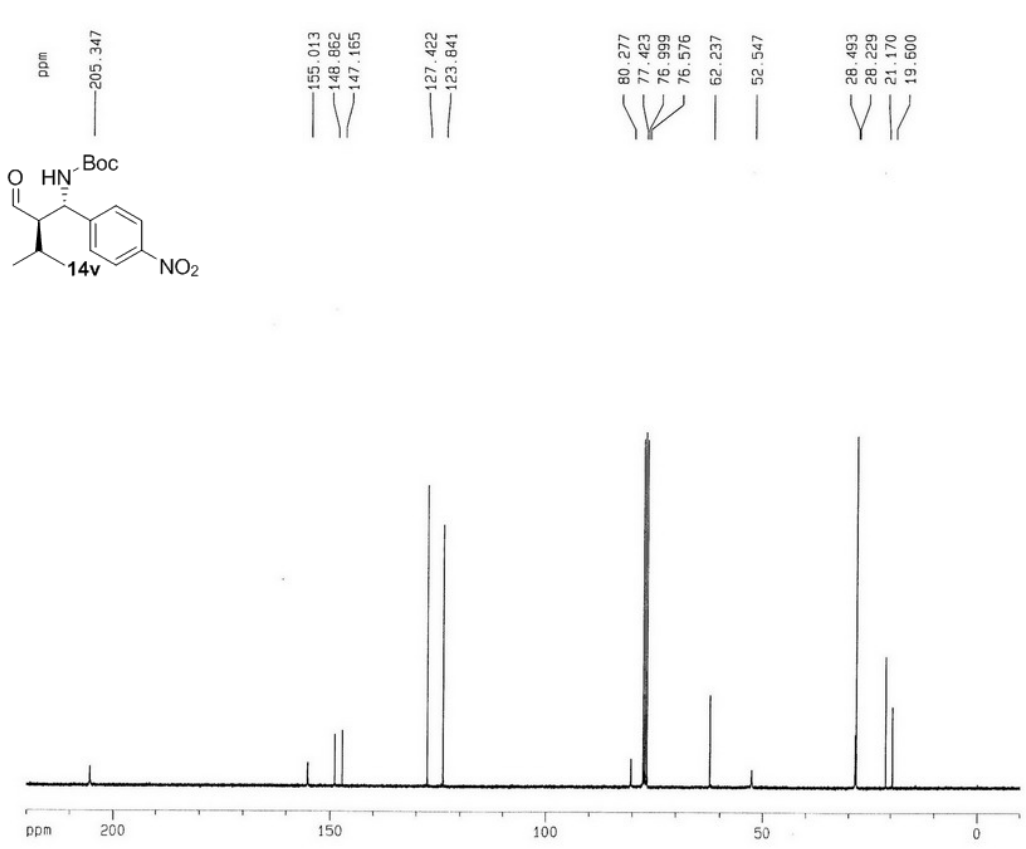
Current Data Parameters
NAME LJL675
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110627
Time 21.19
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 52
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 71.8
DW 83.400 usec
DE 6.00 usec
TE 297.6 K
D1 2.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz

F2 - Processing parameters
SI 16384
SF 300.1699985 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.62 cm
F1P 11.000 ppm
F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.10201 Hz/cm



Current Data Parameters
NAME LJL675
EXPNO 2
PROCNO 1

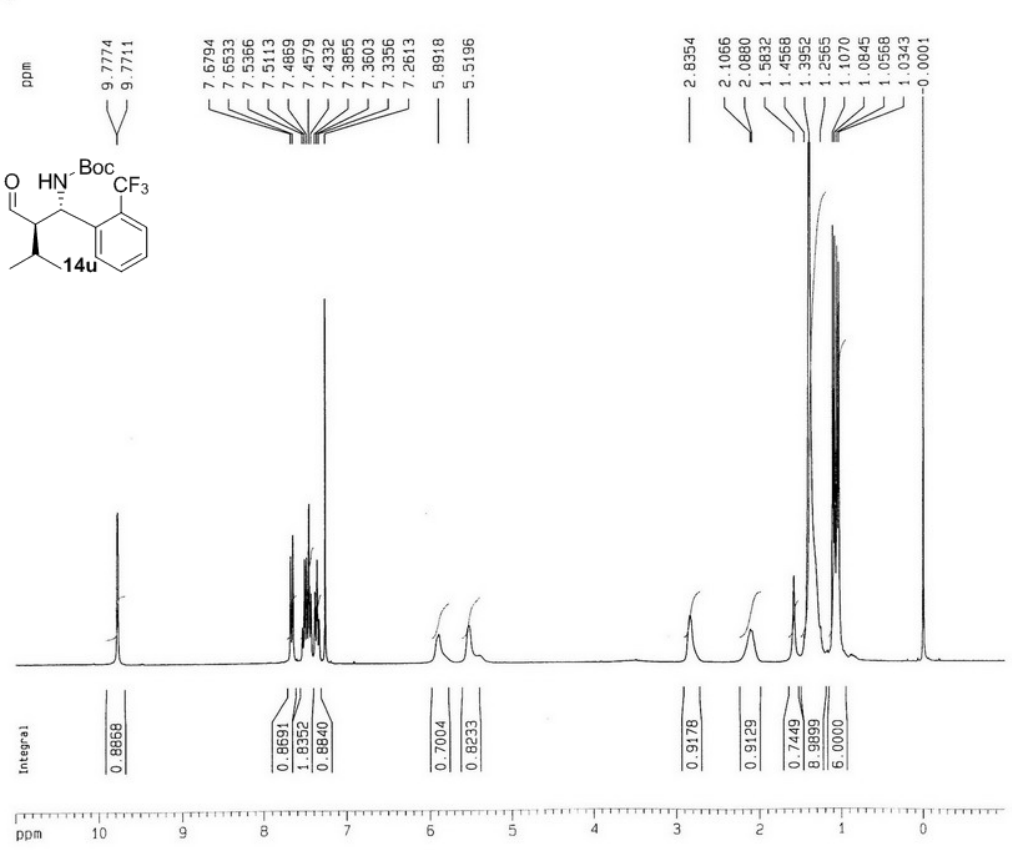
F2 - Acquisition Parameters
Date_ 20110627
Time 21.25
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 63488
SOLVENT CDCl3
NS 10693
DS 0
SWH 22222.223 Hz
FIDRES 0.350022 Hz
AQ 1.4285300 sec
RG 18390.4
DW 22.500 usec
DE 4.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 11

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 -2.00 dB
SFO1 75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 23.74 dB
SFO2 300.1712007 MHz

F2 - Processing parameters
SI 131072
SF 75.4778094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 7.12 cm
F1P 220.000 ppm
F1 16605.12 Hz
F2P -10.000 ppm
F2 -754.78 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.99475 Hz/cm




```
Current Data Parameters
NAME                  LJL-689
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110712
Time                   11.33
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        29
DS                         0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                       256
DW                  83.400 usec
DE                   6.00 usec
TE                   297.5 K
D1                 2.50000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                  10.30 usec
PL1                  4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                     16384
SF                300.1700044 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   13.09 cm
F1P                 11.000 ppm
F1                 3301.87 Hz
F2P                -1.000 ppm
F2                 -300.17 Hz
PPMCM              0.60000 ppm/cm
HZCM              180.10201 Hz/cm
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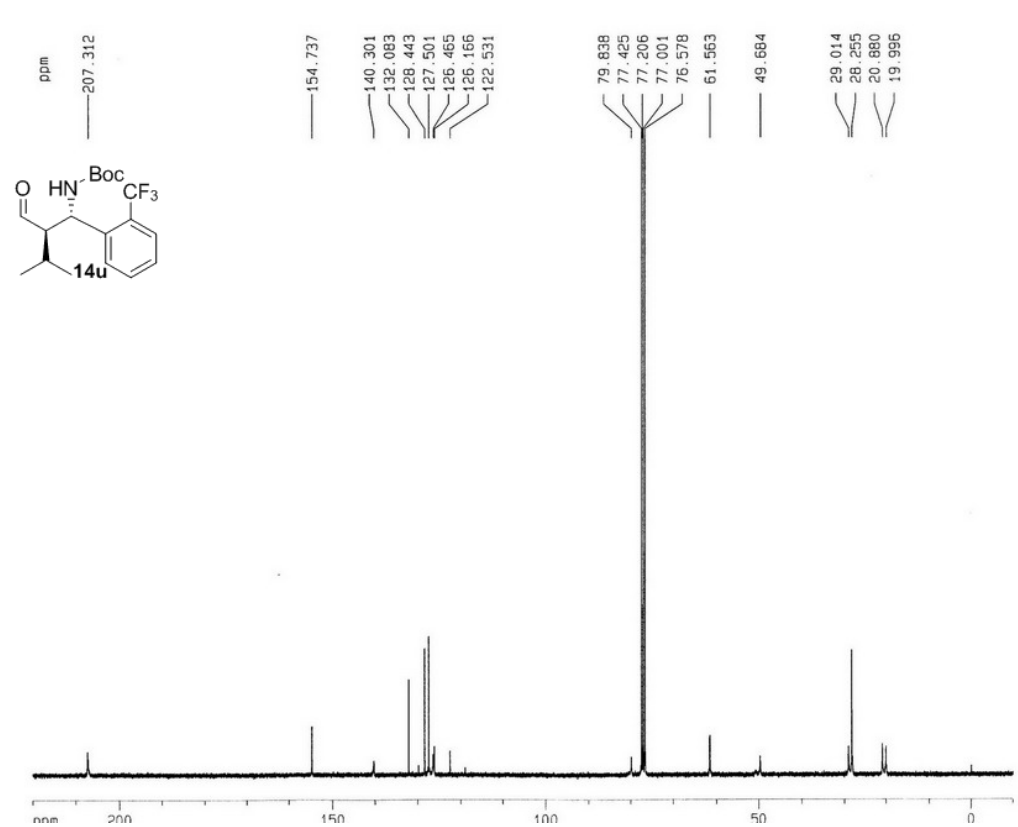


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Current Data Parameters
NAME                  LJL-689
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110711
Time                   20.08
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                zgdc
TD                     63488
SOLVENT                CDCl3
NS                      12288
DS                         0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                   18390.4
DW                  22.500 usec
DE                   4.50 usec
TE                   673.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       12

======== CHANNEL f1 ========
NUC1                      13C
P1                  10.10 usec
PL1                  -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2               100.00 usec
PL2                  4.00 dB
PL12                23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                    131072
SF                75.4778075 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   13.72 cm
F1P                 220.000 ppm
F1                 16605.12 Hz
F2P                -10.000 ppm
F2                 -754.78 Hz
PPMCM              11.50000 ppm/cm
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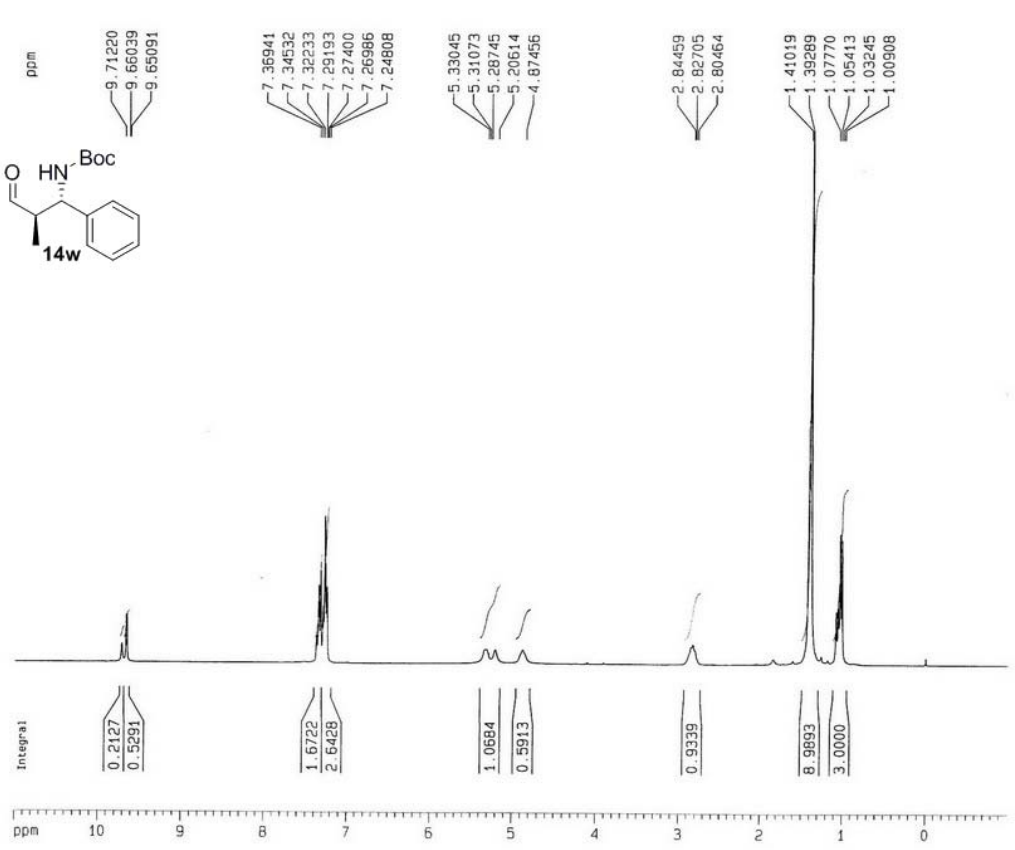


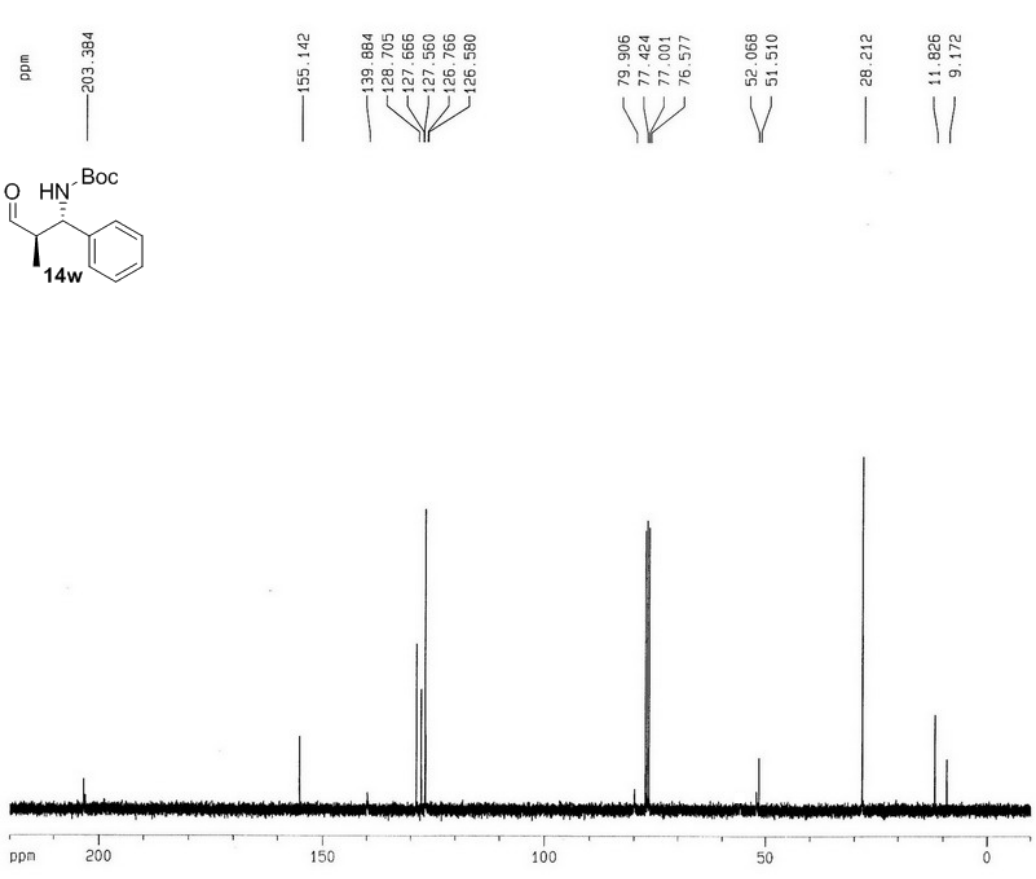
Current Data Parameters
NAME LJL-697
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110810
Time 19.58
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 55
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 35.9
DW 83.400 usec
DE 6.00 usec
TE 296.1 K
D1 2.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 4.00 dB
SFO1 300.1718010 MHz

F2 - Processing parameters
SI 16384
SF 300.1700011 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.70 cm
F1P 11.000 ppm
F1 3301.87 Hz
F2P -1.000 ppm
F2 -300.17 Hz
PPMCM 0.60000 ppm/cm
HZCM 180.10201 Hz/cm



Current Data Parameters
NAME LJL-697
EXPNO 2
PROCNO 1

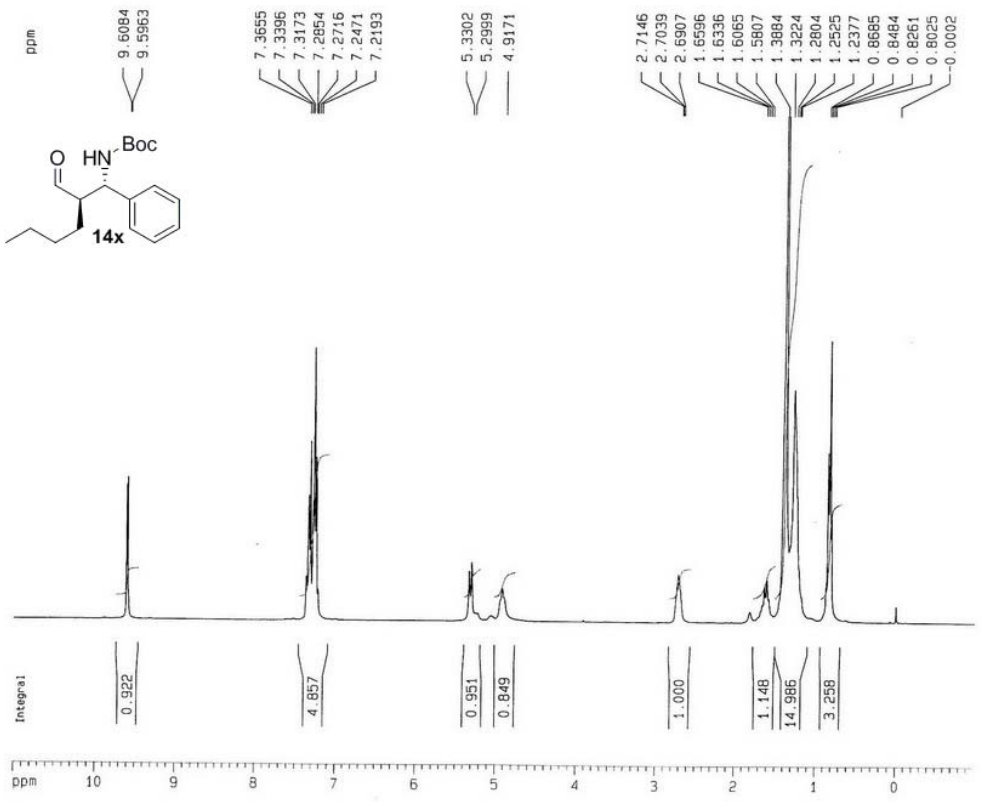
F2 - Acquisition Parameters
Date_ 20110811
Time 9.17
INSTRUM av300
PROBHD 5 mm Multinucl
PULPROG zgdc
TD 63488
SOLVENT CDCl3
NS 520
DS 0
SWH 22222.223 Hz
FIDRES 0.350022 Hz
AQ 1.4285300 sec
RG 18390.4
DW 22.500 usec
DE 4.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 -2.00 dB
SFO1 75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 4.00 dB
PL12 23.74 dB
SFO2 300.1712007 MHz

F2 - Processing parameters
SI 131072
SF 75.4778114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.89 cm
F1P 220.000 ppm
F1 16605.12 Hz
F2P -10.000 ppm
F2 -754.78 Hz
PPMCM 11.50000 ppm/cm
HZCM 867.99497 Hz/cm




```
Current Data Parameters
NAME                  LJL-694
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110809
Time                   9.17
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        78
DS                         0
SWH                5995.204 Hz
FIDRES             0.182959 Hz
AQ                 2.7329011 sec
RG                        32
DW                   83.400 usec
DE                    6.00 usec
TE                   295.7 K
D1                 2.50000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      1H
P1                    10.30 usec
PL1                    4.00 dB
SFO1              300.1718010 MHz

F2 - Processing parameters
SI                      16384
SF                300.1700014 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     25.04 cm
F1P                  11.000 ppm
F1                  3301.87 Hz
F2P                 -1.000 ppm
F2                  -300.17 Hz
PPMCM               0.60000 ppm/cm
HZCM              180.10201 Hz/cm
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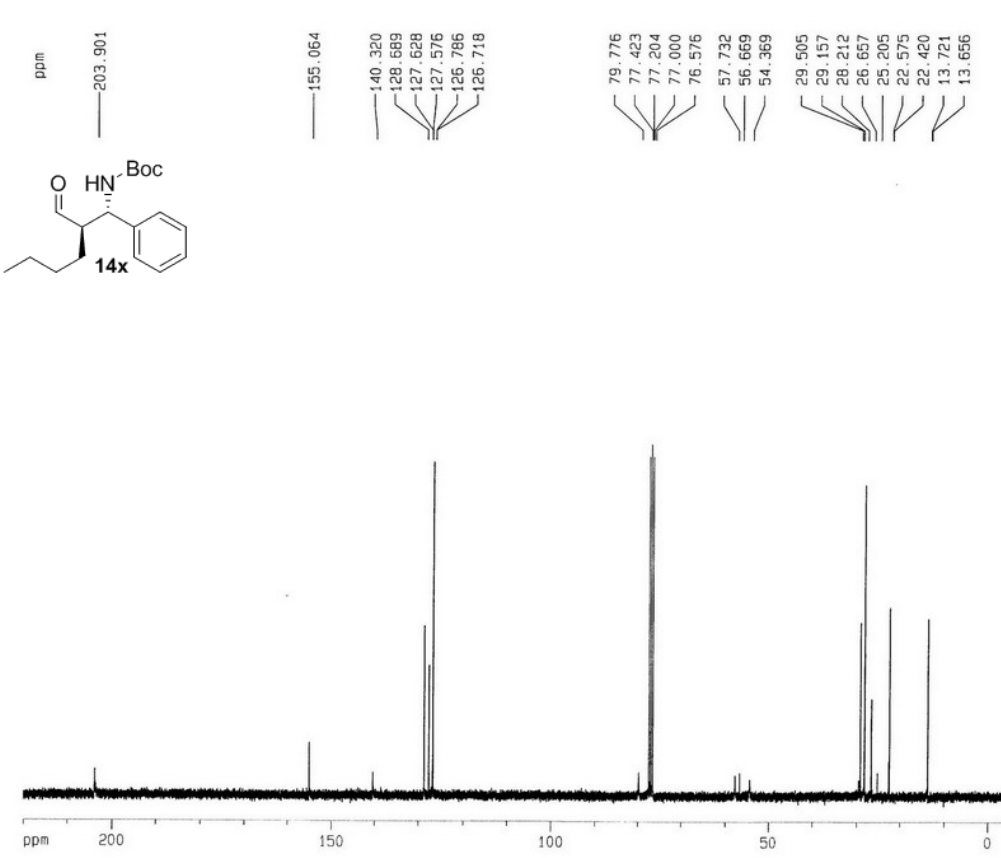


```
Current Data Parameters
NAME                  LJL-694
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110809
Time                   9.27
INSTRUM                av300
PROBHD   5 mm Multinucl
PULPROG                 zgdc
TD                     63488
SOLVENT                CDCl3
NS                      1001
DS                         0
SWH               22222.223 Hz
FIDRES             0.350022 Hz
AQ                 1.4285300 sec
RG                    18390.4
DW                   22.500 usec
DE                    4.50 usec
TE                   296.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.10 usec
PL1                   -2.00 dB
SFO1              75.4862203 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                    4.00 dB
PL12                  23.74 dB
SFO2              300.1712007 MHz

F2 - Processing parameters
SI                     131072
SF                75.4778113 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                      6.97 cm
F1P                  220.000 ppm
F1                  16605.12 Hz
F2P                 -10.000 ppm
F2                  -754.78 Hz
PPMCM               11.50000 ppm/cm
HZCM              867.99487 Hz/cm
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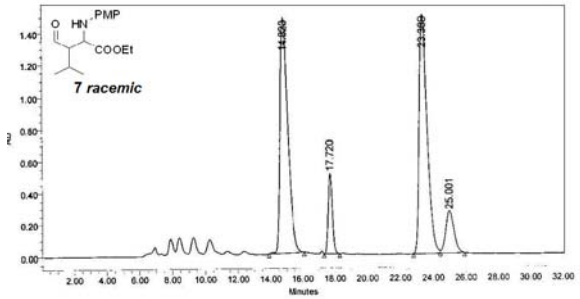
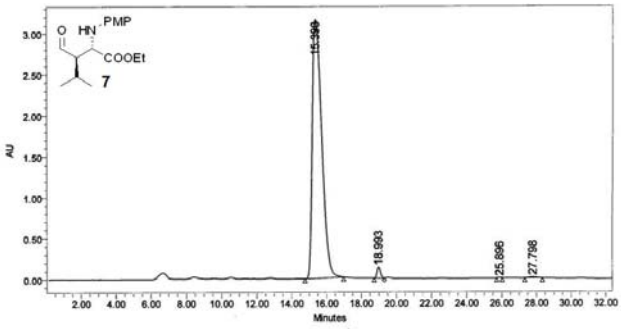
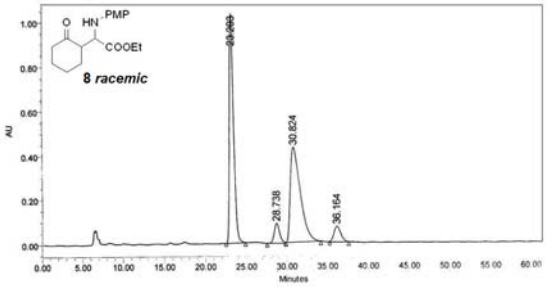


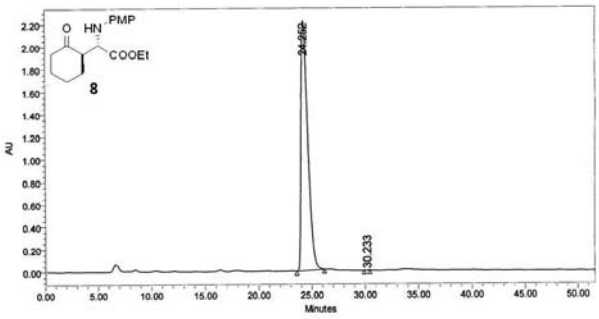
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	14.823	50114774	42.25	1477793	39.34
2	17.720	8880065	7.49	508915	13.55
3	23.389	50474890	42.56	1505118	40.07
4	25.001	9139903	7.71	264553	7.04



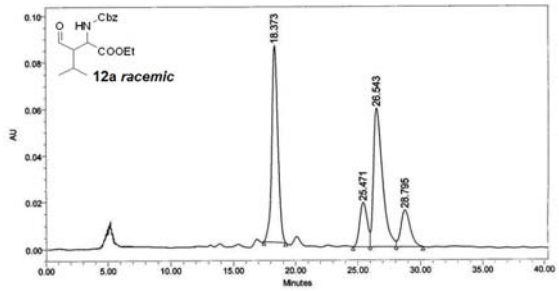
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	15.398	119016176	98.20	3155450	95.59
2	18.993	1760161	1.45	131405	3.98
3	25.896	15320	0.01	1268	0.04
4	27.798	411288	0.34	12961	0.39



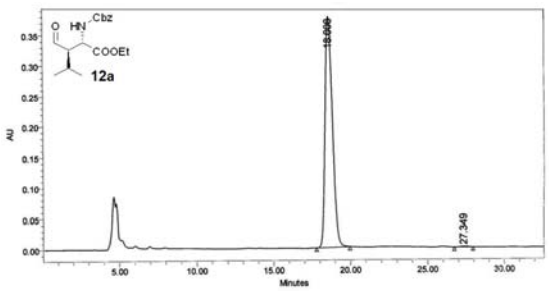
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	23.203	36423738	45.01	1031406	63.70
2	28.738	4013951	4.96	89077	5.50
3	30.824	36407931	44.99	428066	26.44
4	36.164	4082270	5.04	70486	4.35



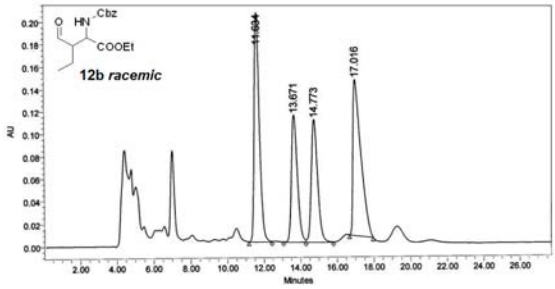
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	24.252	97900579	99.99	2220478	99.97
2	30.233	10208	0.01	568	0.03



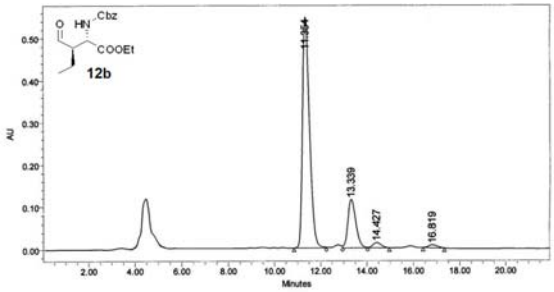
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	18.373	2853394	38.57	84157	47.41
2	25.471	778081	10.52	18614	10.49
3	26.543	2956260	39.97	59352	33.43
4	28.795	809363	10.94	15395	8.67



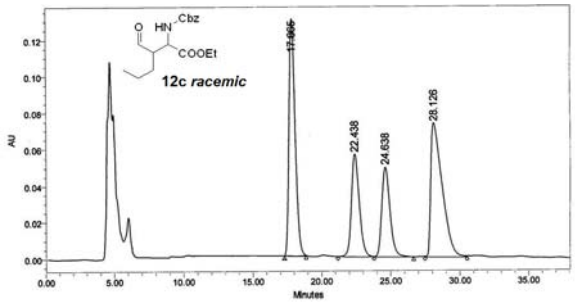
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	18.608	12430563	99.67	376394	99.70
2	27.349	41263	0.33	1141	0.30



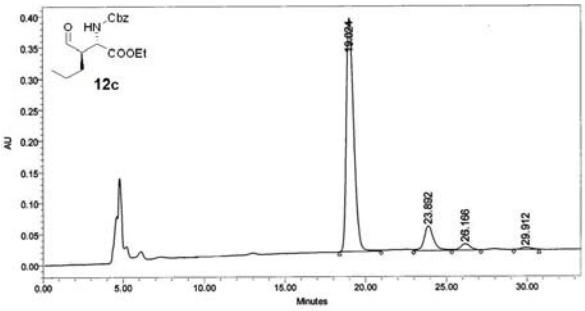
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	11.634	4000526	30.13	203706	36.14
2	13.671	2532068	19.07	112648	19.99
3	14.773	2604033	19.61	108622	19.27
4	17.016	4139690	31.18	138630	24.60



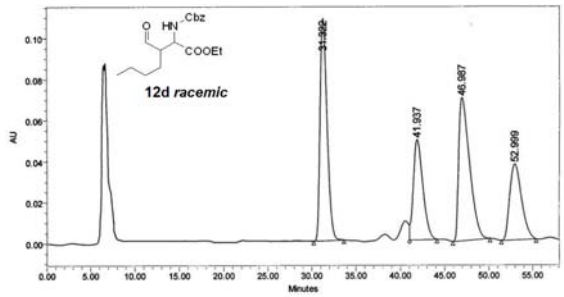
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	11.354	11582337	78.39	546773	80.46
2	13.339	2695767	18.24	112948	16.62
3	14.427	311440	2.11	12624	1.86
4	16.819	186151	1.26	7202	1.06



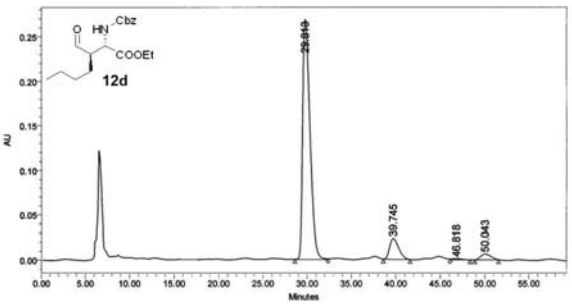
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	17.865	4046785	32.45	130157	42.15
2	22.438	2224249	17.83	56002	18.14
3	24.638	2018293	16.18	49265	15.96
4	28.126	4183047	33.54	73350	23.76



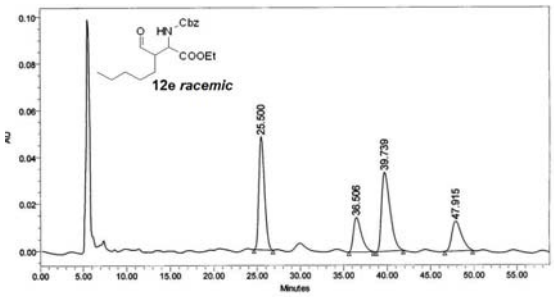
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	19.024	11998253	84.62	375264	87.65
2	23.892	1589530	11.21	38910	9.09
3	26.166	444619	3.14	10502	2.45
4	29.912	146390	1.03	3455	0.81



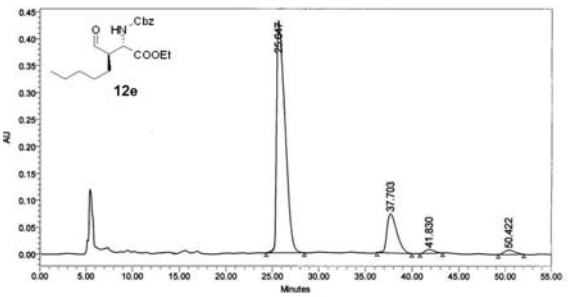
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	31.322	5985809	31.44	108419	41.06
2	41.937	3609923	18.96	48754	18.46
3	46.987	5964152	31.33	69925	26.48
4	52.999	3478974	18.27	36978	14.00



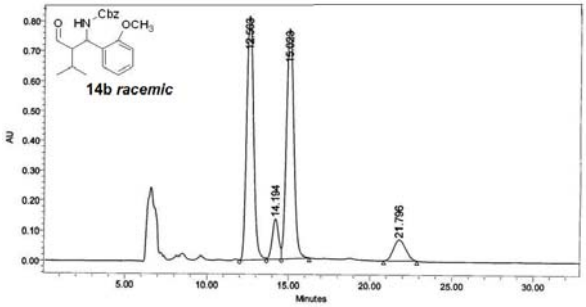
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	29.813	15427409	87.26	268289	89.68
2	39.745	1637832	9.26	23208	7.76
3	46.818	99578	0.56	1256	0.42
4	50.043	515039	2.91	6417	2.14



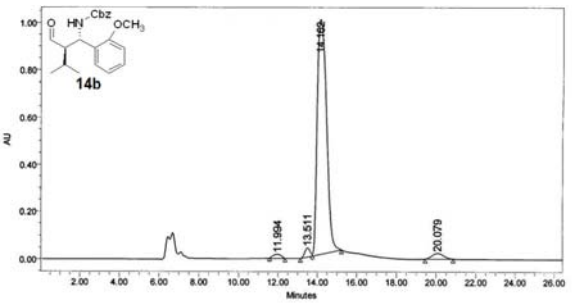
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	25.500	2172369	34.09	47980	44.20
2	36.506	931755	14.62	14384	13.25
3	39.739	2274403	35.69	33737	31.08
4	47.915	993280	15.59	12461	11.48



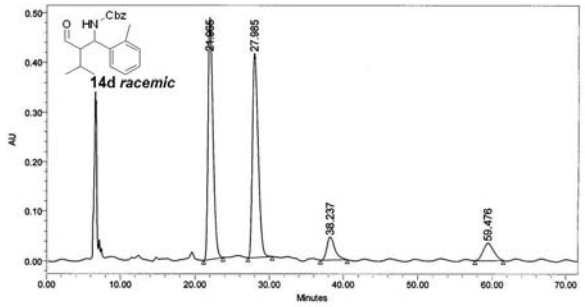
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	25.647	26978048	80.73	431383	83.11
2	37.703	5320024	15.92	72368	13.94
3	41.830	474471	1.42	7329	1.41
4	50.422	646282	1.93	7954	1.53



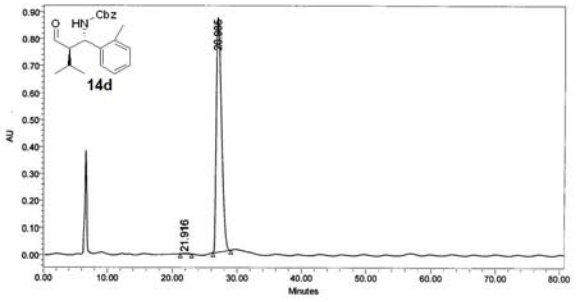
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	12.563	24198992	43.49	815648	45.71
2	14.194	3561883	6.40	131746	7.38
3	15.023	24283554	43.64	767070	42.99
4	21.796	3601905	6.47	69950	3.92



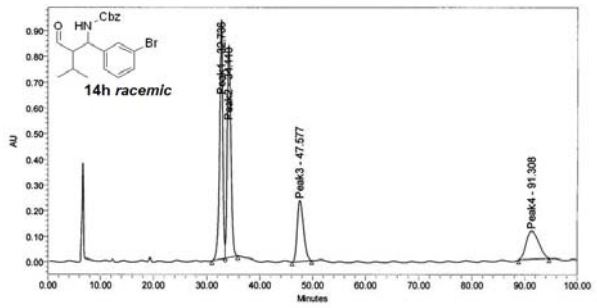
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	11.994	391111	1.13	14187	1.33
2	13.511	696815	2.01	37911	3.55
3	14.162	32542199	94.04	992344	92.84
4	20.079	974089	2.81	24418	2.28



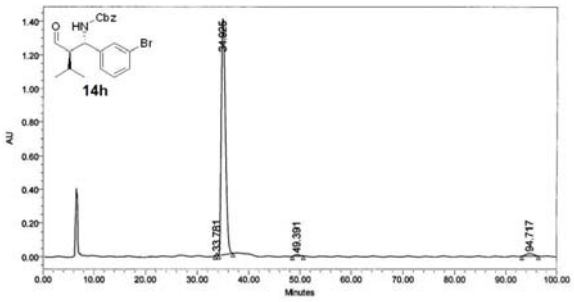
	RT (min)	Area (µV*sec)	% Area	Height (µV)	% Height
1	21.965	22414624	43.54	487552	49.75
2	27.985	22063497	42.86	410442	41.88
3	38.237	3503251	6.80	46367	4.73
4	59.476	3500962	6.80	35581	3.63



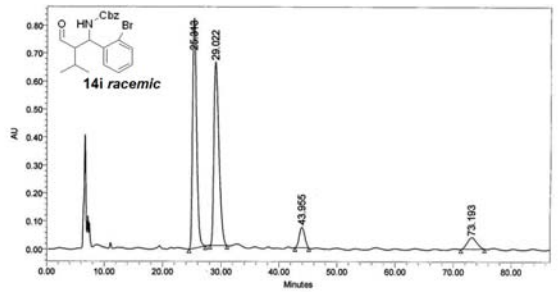
	RT (min)	Area (µV*sec)	% Area	Height (µV)	% Height
1	21.916	134718	0.28	2023	0.23
2	26.985	48552948	99.72	869451	99.77



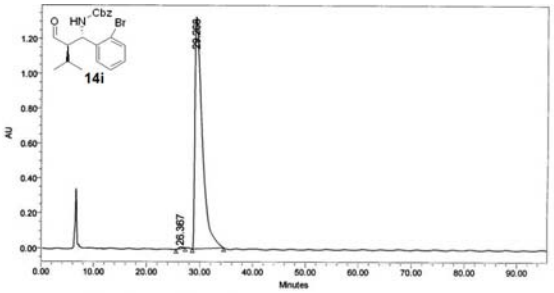
	Peak Name	RT (min)	Area (µV*sec)	% Area	Height (µV)	% Height
1	Peak1	32.736	38273755	32.94	932048	44.29
2	Peak2	34.110	40120196	34.53	826411	39.27
3	Peak3	47.577	19551102	16.83	236151	11.22
4	Peak4	91.308	18255331	15.71	110011	5.23



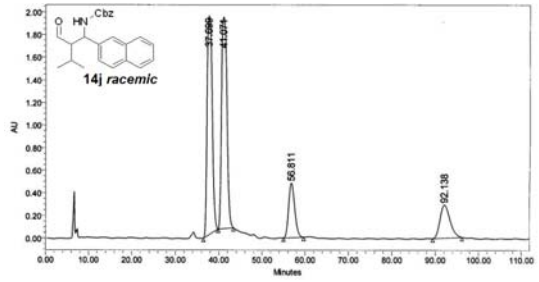
	RT (min)	Area (µV*sec)	% Area	Height (µV)	% Height
1	33.781	274243	0.34	14581	1.01
2	34.925	78896913	96.95	1402606	97.34
3	49.391	568367	0.70	8196	0.57
4	94.717	1643634	2.02	15610	1.08



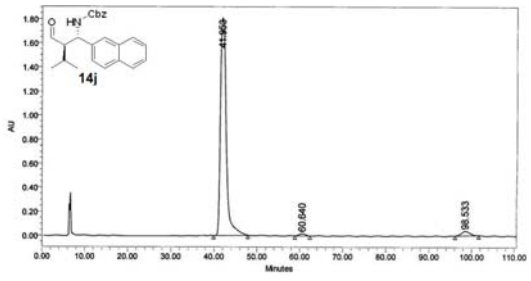
	RT (min)	Area (µV*sec)	% Area	Height (µV)	% Height
1	25.313	39280236	44.44	822235	51.65
2	29.022	39572443	44.77	654076	41.09
3	43.955	4810052	5.44	73880	4.64
4	73.193	4733307	5.35	41789	2.62



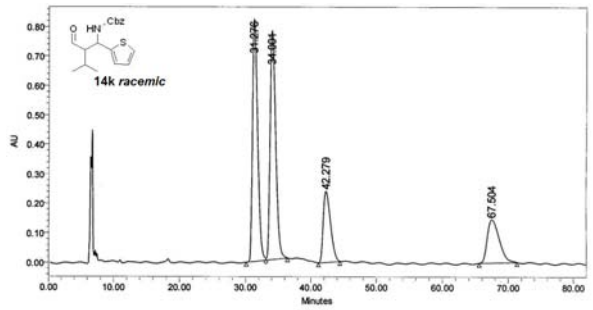
	RT (min)	Area (µV*sec)	% Area	Height (µV)	% Height
1	26.367	395517	0.31	7995	0.60
2	29.268	126962027	99.69	1326665	99.40



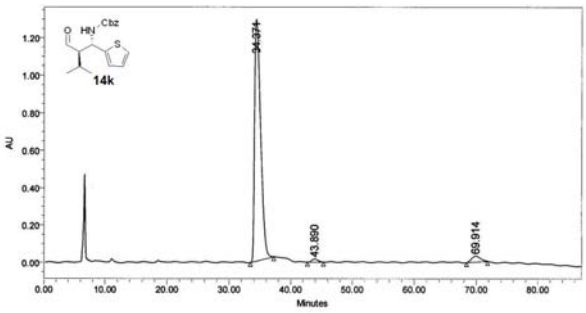
	RT (min)	Area (µV*sec)	% Area	Height (µV)	% Height
1	37.699	159068633	38.39	1933901	42.25
2	41.071	161070045	38.87	1868806	40.82
3	56.811	47311307	11.42	481778	10.52
4	92.138	46913892	11.32	293200	6.40



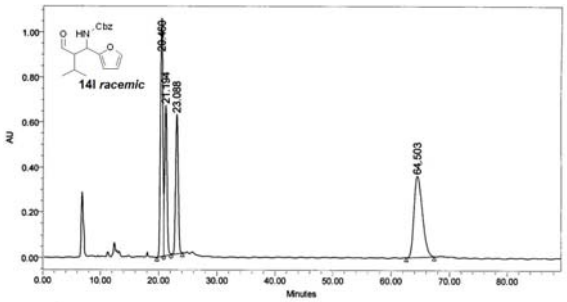
	RT (min)	Area (µV*sec)	% Area	Height (µV)	% Height
1	41.953	171970557	96.02	1810545	96.98
2	60.640	1978553	1.10	19701	1.06
3	98.533	5141681	2.87	36692	1.97



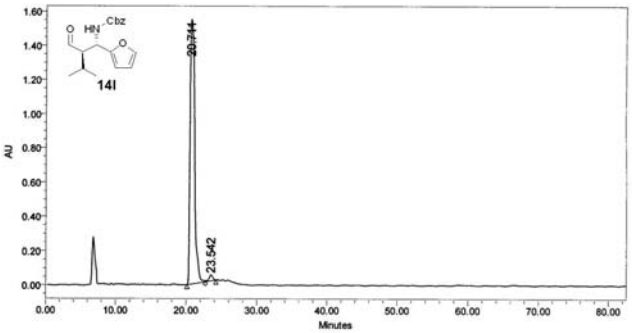
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	31.276	44610743	35.03	821979	41.41
2	34.001	45687014	35.88	776526	39.12
3	42.279	18619991	14.62	240815	12.13
4	67.504	18419489	14.47	145661	7.34



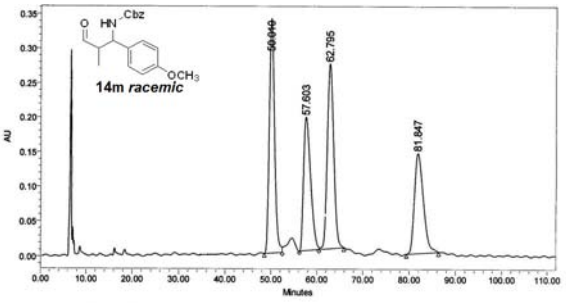
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	34.371	88062882	95.21	1291444	96.34
2	43.890	1207181	1.31	17214	1.28
3	69.914	3222724	3.48	31915	2.38



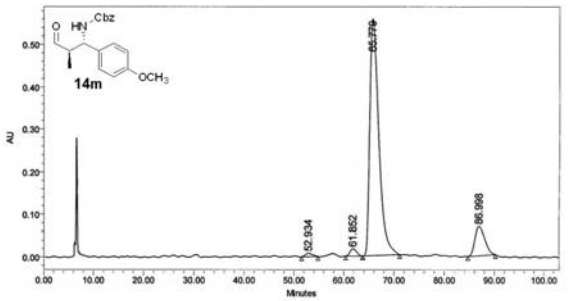
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	20.460	30730661	29.37	1057837	39.17
2	21.194	19316994	18.46	666417	24.68
3	23.088	18772067	17.94	617484	22.86
4	64.503	35809418	34.23	358977	13.29



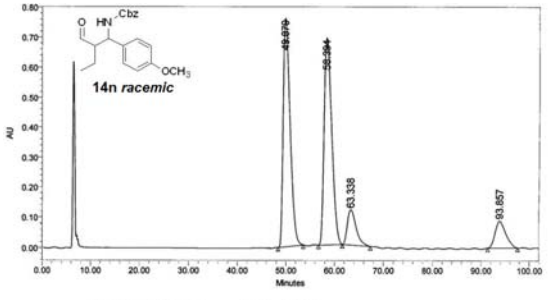
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	20.711	69211246	98.30	1547065	97.86
2	23.542	1197495	1.70	33869	2.14



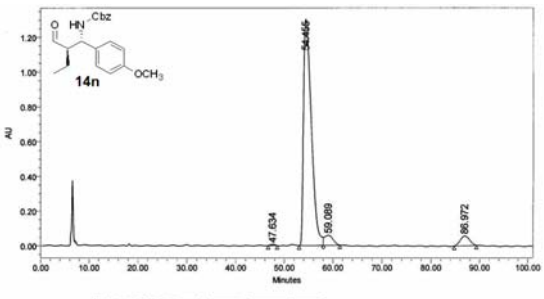
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	50.010	26569567	28.95	338410	36.04
2	57.603	20292506	22.11	191066	20.35
3	62.795	25769864	28.08	266090	28.33
4	81.847	19147482	20.86	143527	15.28



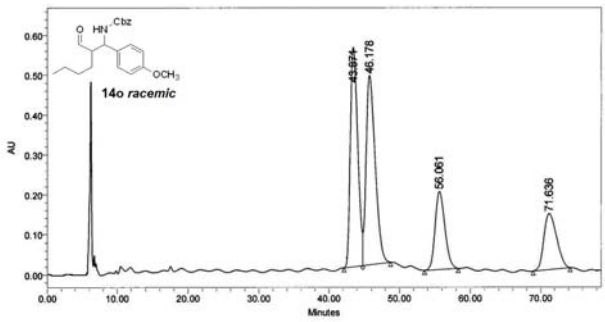
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	52.934	758206	0.94	7871	1.21
2	61.852	1528311	1.89	17092	2.63
3	65.779	69079276	85.63	557249	85.78
4	86.998	9309275	11.54	67409	10.38



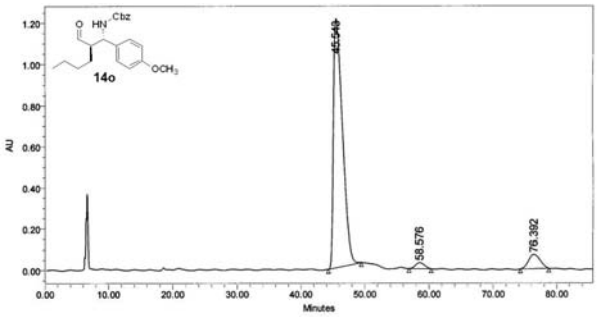
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	49.879	74669762	42.15	759572	46.10
2	58.394	74558252	42.08	687183	41.70
3	63.338	14278244	8.06	114070	6.92
4	93.857	13657898	7.71	86992	5.28



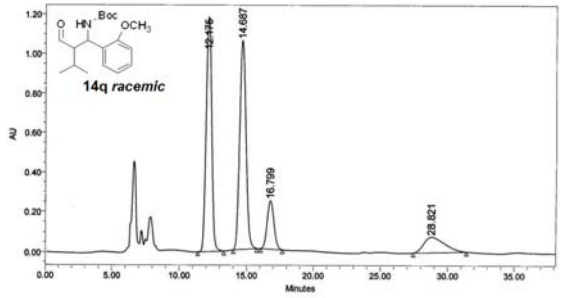
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	47.634	484484	0.30	7739	0.54
2	54.455	146178272	90.66	1305075	91.48
3	59.089	7193614	4.46	57960	4.06
4	86.972	7385612	4.58	55905	3.92



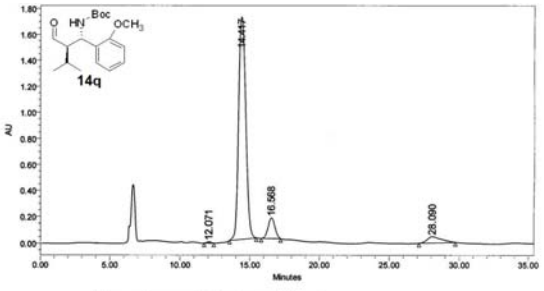
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	43.871	40223927	33.84	549452	40.49
2	46.178	42607103	35.84	473284	34.88
3	56.061	18540261	15.60	194856	14.36
4	71.636	17507326	14.73	139320	10.27



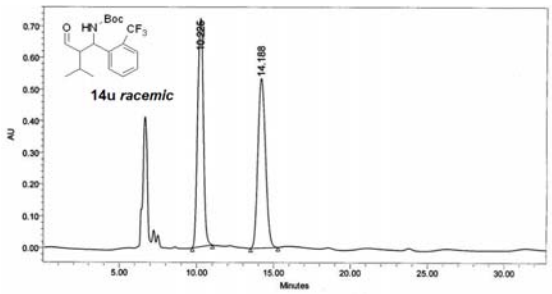
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	45.543	119199309	91.12	1208725	92.44
2	58.576	2787613	2.13	29576	2.26
3	76.392	8831388	6.75	69268	5.30



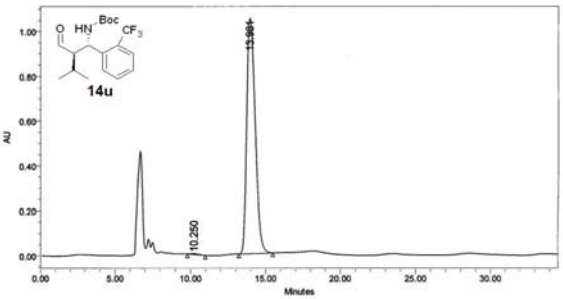
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	12.175	34075535	39.92	1184603	46.36
2	14.687	34452891	40.36	1052075	41.17
3	16.799	8456576	9.91	240256	9.40
4	28.821	8382712	9.82	78381	3.07



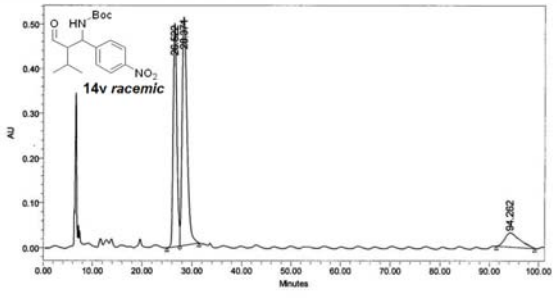
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	12.071	225623	0.33	10113	0.53
2	14.417	60466422	87.25	1703723	89.04
3	16.568	5213804	7.52	151528	7.92
4	28.090	3397576	4.90	48086	2.51



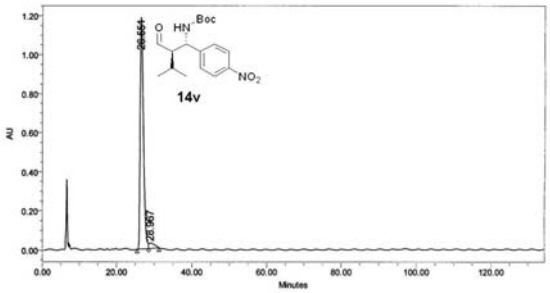
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.225	18571682	50.11	720071	57.38
2	14.188	18493034	49.89	534953	42.62



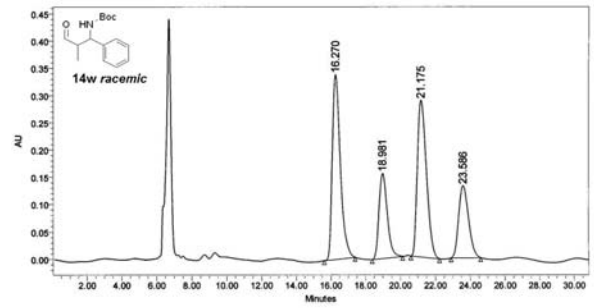
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	10.250	121705	0.29	4121	0.39
2	13.981	41266050	99.71	1048672	99.61



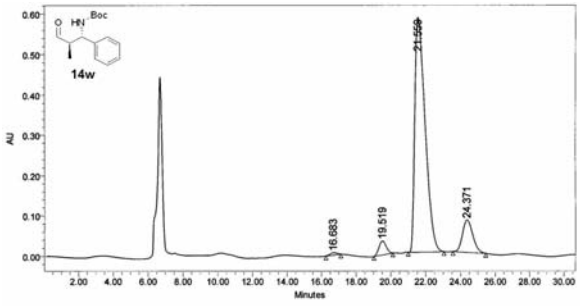
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	26.522	25432653	37.81	499243	47.96
2	28.371	34909044	51.90	509873	48.98
3	94.262	6922787	10.29	31817	3.06



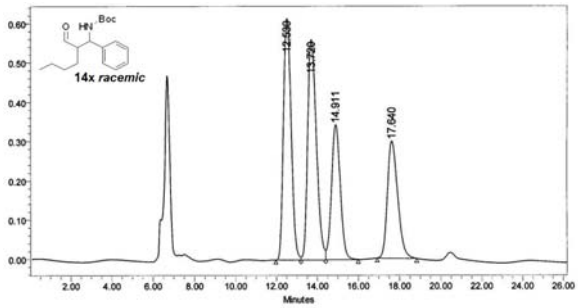
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	26.551	70754339	96.03	1184726	97.74
2	28.967	2921601	3.97	27409	2.26



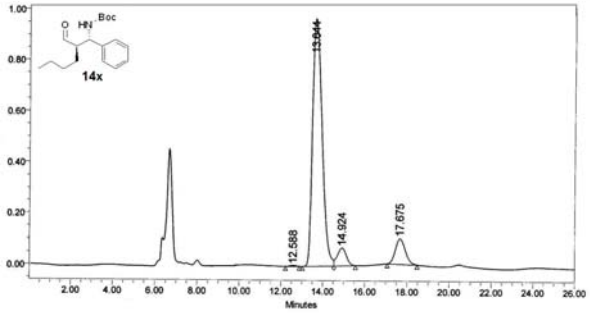
	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	16.270	10913193	34.62	338350	37.08
2	18.981	5129280	16.27	155039	16.99
3	21.175	10328368	32.76	287589	31.51
4	23.586	5154375	16.35	131582	14.42



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	16.683	160535	0.57	5917	0.84
2	19.519	989890	3.49	34453	4.91
3	21.559	23909746	84.36	580889	82.87
4	24.371	3283975	11.59	79737	11.37



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	12.530	16155849	30.27	615079	33.86
2	13.720	16714888	31.32	560363	30.84
3	14.911	10200111	19.11	343264	18.89
4	17.640	10302314	19.30	298010	16.40



	RT (min)	Area (μV*sec)	% Area	Height (μV)	% Height
1	12.588	86524	0.24	4205	0.37
2	13.644	30590195	84.92	970296	84.80
3	14.924	2097349	5.82	70137	6.13
4	17.675	3248543	9.02	99552	8.70