Catalytic Asymmetric γ-Alkylation of Carbonyl Compounds via Stereoconvergent Suzuki Cross-Couplings

Susan L. Zultanski and Gregory C. Fu*

Department of Chemistry, Massachusetts Institute of Technology, Cambridge, MA 02139

Supporting Information

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I. General Information

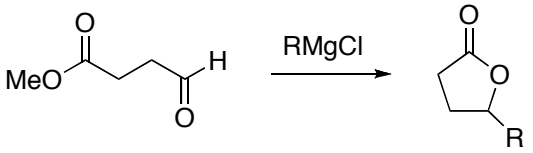
The following reagents were purchased and used as received: 9-BBN dimer (Aldrich), $NiBr_2$•diglyme (Aldrich; note: hygroscopic), ligands *(R,R)*-**1** and *(S,S)*-**1** (Acros, Aldrich), KO*t*-Bu (Aldrich), *n*-hexanol (anhydrous; Aldrich), Et_2O (anhydrous; Aldrich), and hexanes (anhydrous; Aldrich). The 1-alkenes (precursors to the nucleophiles) were purchased (Aldrich or Alfa Aesar) and purified by flash chromatography prior to use, or they were prepared according to literature procedures.

All reactions were carried out in oven-dried glassware under an atmosphere of nitrogen.

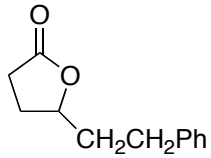
^{1}H NMR and ^{13}C NMR data were collected on a Bruker Avance 400 spectrometer at ambient temperature. HPLC analyses were carried out on an Agilent 1100 series system with Daicel CHIRALCEL® columns (internal diameter 4.6 mm, column length 250 mm, particle size 5 μ). SFC analyses were carried out on an SFC ProNTo system with Daicel CHIRALCEL® columns (internal diameter 4.6 mm, column length 250 mm, particle size 5 μ).

II. Preparation of Electrophiles

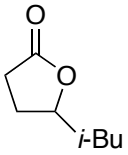
The procedures and yields have not been optimized.



General Procedure A: Preparation of lactones. Anhydrous THF (170 mL) and then methyl 4-oxobutanoate (3.0 g, 24 mmol; Aldrich) were added to an oven-dried round-bottom flask. The reaction mixture was cooled to –78 °C, and the alkyl Grignard reagent (1.0 equiv) was added dropwise to the stirred solution. The reaction mixture was allowed to warm to room temperature and then stirred for 1 hour. Next, the reaction was quenched by the addition of water (5 mL). A saturated aqueous solution of NH_4Cl (60 mL) was then added to the reaction mixture, which was stirred until it was homogeneous. The mixture was transferred to a separatory funnel, and the product was extracted with Et_2O (100 mL x2) and CH_2Cl_2 (100 mL). The combined organic extracts were washed with brine (100 mL x2), dried over magnesium sulfate, filtered, and concentrated to yield the γ-lactone, which was purified by flash chromatography with 10→60% Et_2O/hexanes.

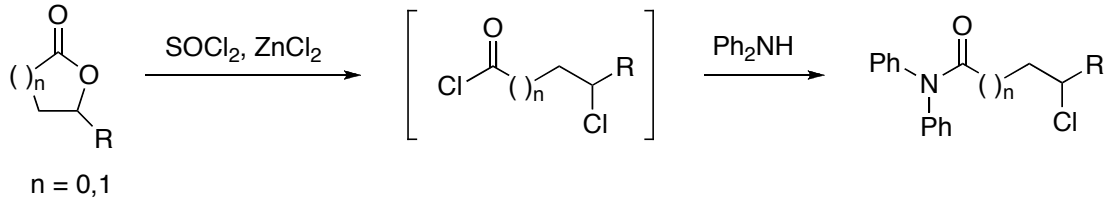


5-Phenethyldihydrofuran-2(3*H*)-one. General Procedure A was followed using phenylethylmagnesium chloride (24 mL; 1.0 M in THF; Aldrich), which furnished the lactone as a colorless oil (3.76 g, 83%). The spectral data match those described in the literature.[1]



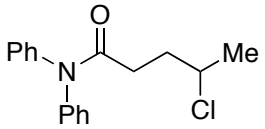
5-Isobutyldihydrofuran-2(3*H*)-one. General Procedure A was followed using isobutylmagnesium chloride (12 mL; 2.0 M in THF; Aldrich), which furnished the lactone as a colorless oil (2.15 g, 66%). The spectral data match those described in the literature.[2]

(1) Cossy, J.; Bargiggia, F.; Bouzbouz, S. *Org. Lett.* **2003**, *5*, 459–462.
(2) Pollack, J. A.; Clark, K. M.; Martynowicz, B. J.; Pridgeon, M. G.; Rycenga, M. J.; Stolle, K. E.; Taylor, S. K. *Tetrahedron: Asymmetry* **2007**, *18*, 1888–1892.



General Procedure B: Preparation of 3- and 4-chloro-*N,N*-diphenylamides.[3,4] Anhydrous $ZnCl_2$ (180 mg, 1.3 mmol note: hygroscopic) was added to an oven-dried two-neck round-bottom flask, which was then capped with a septum and purged with nitrogen. Thionyl chloride (2.4 mL, 33 mmol) was added to the flask, followed by the lactone (30 mmol). The reaction mixture was stirred at 55 °C for 24 h, during which time it turned dark-brown and became viscous. The excess thionyl chloride was removed under reduced pressure, and the acid chloride was used in the next step without further purification.

The two-neck flask containing the acid chloride was equipped with a reflux condenser and purged with nitrogen. Next, anhydrous benzene (100 mL) and then the diarylamine (33 mmol) were added. The reaction mixture was refluxed for 6 h, and then it was allowed to cool to room temperature. Brine (100 mL) was added, and the mixture was transferred to a separatory funnel. The layers were separated, and the aqueous layer was extracted with Et_2O (100 mL). The combined organic layers were washed with brine (100 mL), dried over magnesium sulfate, filtered, and concentrated to yield the γ- or δ-chloro-*N,N*-diarylamide. The product was purified by reverse-phase flash chromatography on C-18 silica gel with 10→100% acetonitrile/water, followed by normal-phase flash chromatography on silica gel with 10→70% Et_2O/hexanes, which furnished pure γ- or δ-chloro-*N,N*-diarylamide (alternatively, if the acid chloride is distilled prior to its use in the second step, purification by reverse-phase column chromatography is unnecessary). The products are stable for at least 6 months when stored under an inert atmosphere at 0 °C.



4-Chloro-*N,N*-diphenylpentanamide. The amide was prepared according to General Procedure B, using γ-valerolactone and diphenylamine. White solid (5.14 g, 60%).

^{1}H NMR ($CDCl_3$) δ 7.33–7.22 (m, 10H), 4.13–4.05 (m, 1H), 2.45–2.42 (m, 2H), 2.22–2.14 (m, 1H), 1.93–1.83 (m, 1H), 1.47 (d, 3H, J = 6.4 Hz).

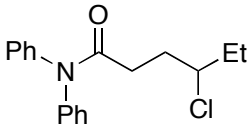
^{13}C NMR ($CDCl_3$) δ 172.1, 142.7, 130.0–125.0 (broad), 58.3, 35.7, 32.4, 25.6.

FT-IR (film) 3062, 2973, 2926, 1672, 1593, 1492, 1380, 1351, 1291, 756, 702 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{17}H_{19}ClNO$: 288, found: 288.

(3) Reppe, W. et al. *Annalen der Chemie, Justus Liebigs* **1955**, *596*, 158–224.

(4) Wise, L. D.; Pattison, I. C.; Butler, D. E.; DeWald, H. A.; Lewis, E. P.; Lobbestael, S. J.; Nordin, I. C.; Poschel, B. P. H.; Coughenour, L. L. *J. Med. Chem.* **1985**, *28*, 606–612.



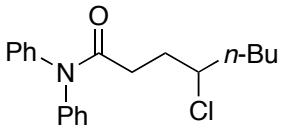
4-Chloro-*N*,*N*-diphenylhexanamide. The amide was prepared according to General Procedure B, using γ-caprolactone and diphenylamine. White solid (6.02 g, 67%).

^{1}H NMR ($CDCl_3$) δ 7.33–7.23 (m, 10H), 3.91–3.88 (m, 1H), 2.46–2.43 (m, 2H), 2.30–2.23 (m, 1H), 2.01–1.94 (m, 1H), 1.87–1.76 (m, 2H), 0.98 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 172.2, 142.7, 129.7–126.4 (broad), 65.2, 33.6, 32.4, 31.9, 11.0.

FT-IR (film) 3063, 2969, 2936, 2878, 1673, 1594, 1492, 1452, 1381, 1272, 1162 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{18}H_{21}ClNO$: 302, found: 302.



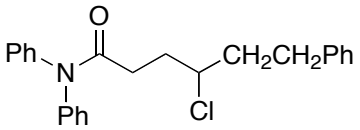
4-Chloro-*N*,*N*-diphenyloctanamide. The amide was prepared according to General Procedure B, using γ-octanoic lactone and diphenylamine. White solid (5.50 g, 56%).

^{1}H NMR ($CDCl_3$) δ 7.33–7.23 (m, 10H), 3.95–3.93 (m, 1H), 2.46–2.43 (m, 2H), 2.23–2.18 (m, 1H), 1.86–1.82 (m, 1H), 1.69–1.64 (m, 2H), 1.46–1.44 (m, 1H), 1.36–1.24 (m, 3H), 0.86 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 172.2, 142.7, 128.7–126.4 (broad), 63.7, 38.5, 33.9, 32.3, 28.6, 22.3, 14.0.

FT-IR (film) 2957, 2871, 2360, 1674, 1594, 1492, 1379, 1280, 756, 701 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{20}H_{25}ClNO$: 330, found: 330.



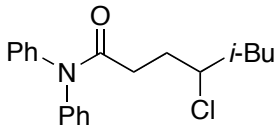
4-Chloro-*N*,*N*-6-triphenylhexanamide. The amide was prepared according to General Procedure B, using 5-phenethyldihydrofuran-2(3*H*)-one and diphenylamine. White solid (3.89 g, 52%).

^{1}H NMR ($CDCl_3$) δ 7.34–7.14 (m, 15H), 3.94–3.90 (m, 1H), 2.84–2.80 (m, 1H), 2.72–2.69 (m, 1H), 2.46–2.43 (m, 2H), 2.22–2.20 (m, 1H), 2.02–1.92 (m, 3H).

^{13}C NMR ($CDCl_3$) δ 172.1, 142.7, 141.1, 130.0–125.0 (broad), 128.53, 128.50, 126.1, 62.7, 40.5, 34.0, 32.7, 32.2.

FT-IR (film) 3027, 2921, 2360, 2340, 1670, 1593, 1492, 1381, 1293, 1158, 700 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{24}H_{25}ClNO$: 378, found: 378.



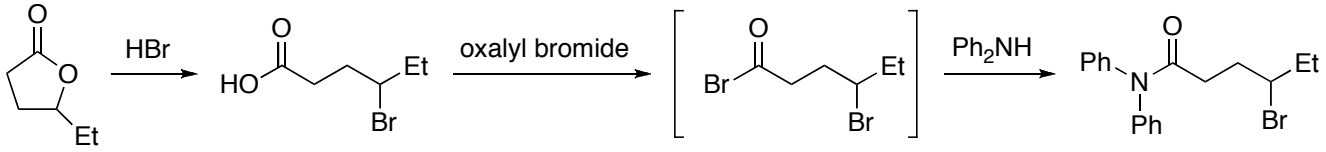
4-Chloro-6-methyl-*N*,*N*-diphenylheptanamide. The amide was prepared according to General Procedure B, using 5-isobutyldihydrofuran-2(3*H*)-one and diphenylamine. White solid (2.40 g, 48%).

^{1}H NMR ($CDCl_3$) δ 7.34–7.23 (m, 10H), 4.02–4.00 (m, 1H), 2.48–2.44 (m, 2H), 2.21–2.18 (m, 1H), 1.88–1.81 (m, 2H), 1.67–1.60 (m, 1H), 1.46–1.41 (m, 1H), 0.88 (d, 3H, J = 6.4 Hz), 0.85 (d, 3H, J = 6.4 Hz).

^{13}C NMR ($CDCl_3$) δ 172.2, 142.7, 130.0–125.0 (broad), 61.8, 47.9, 34.2, 32.3, 25.3, 23.0, 21.4.

FT-IR (film) 2957, 2360, 1674, 1491, 1381, 1270, 756, 701 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{20}H_{25}ClNO$: 330, found: 330.



4-Bromo-*N*,*N*-diphenylhexanamide.[5] In accordance with a literature procedure,[6] γ-caprolactone (8.0 g, 70 mmol) was added to a round-bottom flask that contained HBr in AcOH (70 mL; 30% in AcOH). The flask was equipped with a reflux condenser, and the reaction mixture was stirred at room temperature for 2 hours and then at 70 °C for 5 hours. Next, the mixture was allowed to cool to room temperature, and then the AcOH was removed by rotary evaporation. CH_2Cl_2 (50 mL) and a solution of saturated sodium thiosulfate (50 mL) were then added, and the mixture was transferred to a separatory funnel, where the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (50 mL x2), and the combined organic layers were washed with brine (50 mL), dried over magnesium sulfate, filtered, and concentrated to furnish 4-bromohexanoic acid (light-red oil). The product was used in the following step without further purification.

Anhydrous CH_2Cl_2 (240 mL) and then oxalyl bromide (20.4 g, 94.5 mmol; Aldrich) were added to an oven-dried round-bottom flask under nitrogen. The solution was cooled to 0 °C, and the unpurified 4-bromohexanoic acid (13.7 g, 70.1 mmol) was added. Next, DMF (1.1 mL, 14 mmol) was added dropwise, and the reaction was monitored at 0 °C for 2 h, at which time gas evolution ended. The reaction mixture was concentrated to remove the excess oxalyl bromide and CH_2Cl_2, affording 4-bromohexanoyl bromide, which was used without purification in the next step.

The flask was equipped with a reflux condenser and purged with nitrogen. Anhydrous benzene (240 mL) was added, followed by diphenylamine (11.8 g, 69.7 mmol). The reaction mixture was refluxed for 6 h, and then it was allowed to cool to room temperature. The mixture

(5) Wise, L. D.; Pattison, I. C.; Butler, D. E.; DeWald, H. A.; Lewis, E. P.; Lobbestael, S. J.; Nordin, I. C.; Poschel, B. P. H.; Coughenour, L. L. *J. Med. Chem.* **1985**, *28*, 606–612.

(6) Sashida, H.; Nakayama, A.; Kaname, M. *Synthesis* **2008**, 3229–3236.

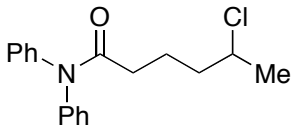
was transferred to a separatory funnel, and brine (100 mL) was added. The layers were separated, and the aqueous layer was extracted with Et_2O (100 mL). The combined organic layers were washed with brine (50 mL x2), dried over magnesium sulfate, filtered, and concentrated. The product was purified by flash chromatography (10→70% Et_2O/hexanes), which furnished 4-bromo-*N*,*N*-diphenylhexanamide as a white solid (15.0 g, 62% over three steps). This compound is stable for at least 3 months when stored under an inert atmosphere at 0 °C.

^{1}H NMR ($CDCl_3$) δ 7.33–7.23 (m, 10H), 4.06–4.02 (m, 1H), 2.48–2.44 (m, 2H), 2.23–2.18 (m, 1H), 1.88–1.82 (m, 1H), 1.77–1.54 (m, 2H), 0.99 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 172.0, 142.6, 130.6–125.5 (broad), 60.0, 34.2, 34.5, 32.6, 12.1.

FT-IR (film) 3061, 2969, 1672, 1593, 1492, 1452, 1381, 1271, 756, 702 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{18}H_{21}BrNO$: 346, 348, found: 346, 348.



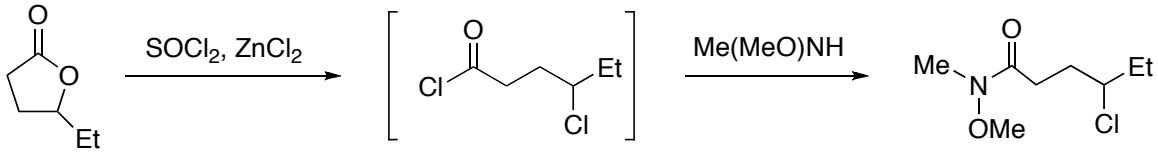
5-Chloro-*N*,*N*-diphenylhexanamide. The amide was prepared according to General Procedure B, using δ-hexalactone and diphenylamine. White solid (3.70 g, 41%).

^{1}H NMR ($CDCl_3$) δ 7.34–7.23 (m, 10H), 3.91–3.88 (m, 1H), 2.46–2.43 (m, 2H), 2.23–2.18 (m, 1H), 1.88–1.82 (m, 1H), 1.77–1.54 (m, 2H), 1.00–0.96 (m, 3H).

^{13}C NMR ($CDCl_3$) δ 172.2, 142.7, 129.3–126.4 (broad), 65.2, 33.6, 32.4, 31.9, 11.0.

FT-IR (film) 3062, 3038, 2969, 2936, 1673, 1594, 1492, 1452, 1381, 1272, 1162 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{18}H_{21}ClNO$: 302, found: 302.



4-Chloro-*N*-methoxy-*N*-methylhexanamide. The first step was performed as described in General Procedure B.

Next, *N*,*O*-dimethylhydroxylamine hydrochloride (1.9 g, 19 mmol) and Et_2O (30 mL) were added to a stirred 0 °C solution of potassium carbonate (6.6 g, 48 mmol) in water (30 mL). Then, 4-chlorohexanoyl chloride (4.0 g, 24 mmol) was added dropwise over 5 minutes. The reaction mixture was stirred at 0 °C for 30 minutes, and then it was diluted with Et_2O (50 mL) and brine (50 mL). The layers were separated, and the aqueous layer was extracted with Et_2O (50 mL). The combined organic layers were washed with 1N HCl (30 mL), dried over magnesium sulfate, filtered, and concentrated. The product was purified by flash chromatography with 20→90% Et_2O/hexanes, which furnished the amide as a yellow oil (2.9 g, 79% yield for step 2). This compound is stable for at least 6 months when stored under an inert atmosphere at 0 °C.

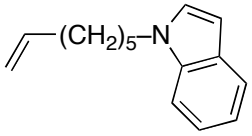
^{1}H NMR ($CDCl_3$) δ 3.91–3.88 (m, 1H), 3.67 (s, 3H), 3.15 (s, 3H), 2.63 (t, 2H, J = 6.8 Hz), 2.16–2.12 (m, 1H), 1.90–1.70 (m, 3H), 1.01 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 173.3, 65.0, 61.0, 32.5, 31.9, 31.6, 28.6, 10.7.
FT-IR (film) 2969, 1775, 1666, 1417, 1386, 1178, 1120, 994, 848, 815 cm^{-1}.
MS (ESI/APCI) m/z ($M+H^+$) calcd for $C_8H_{17}ClNO_2$: 194, found: 194.

III. Preparation of Nucleophiles

General procedure for the preparation of *B*-alkyl-(9-BBN) reagents. In a nitrogen-filled glovebox, the olefin (6.0 mmol; purified) was added to 9-BBN dimer (3.0 mmol) in a 20-mL vial equipped with a stir bar. Et_2O was then added to provide a concentration of 1.5 M of the organoborane, and the vial was sealed with a teflon-lined septum cap. The mixture was heated at 40 °C for 1.5 hours (outside of the glovebox), during which time it became homogenous. The solution was allowed to cool to room temperature. This solution could be stored in a glovebox at ambient temperature for 3 months without noticeable degradation.

Procedure for the preparation of *B*-phenyl- and *B*-cyclopropyl-(9-BBN) reagents. These reagents were prepared according to a literature procedure[7] by reacting phenylmagnesium bromide (3.0 M in Et_2O; Aldrich) or cyclopropylmagnesium bromide (0.5 M in Et_2O; Aldrich) with *B*-MeO-(9-BBN). The resulting products were purified by distillation.



(Hept-6-en-1-yl)-1*H*-indole. The title compound was synthesized via a modification of a literature method.[8] Anhydrous DMF (7 mL) and indole (1.1 g, 9.4 mmol) were added to an oven-dried two-neck round-bottom flask under nitrogen. The reaction mixture was cooled to 0 °C, and then NaH (0.21 g, 8.7 mmol) was added, followed by the dropwise addition of 7-bromo-hept-1-ene (2.0 g, 11.3 mmol). The reaction was warmed to room temperature and stirred for 5 hours. Next, water was added (10 mL), and the mixture was transferred to a separatory funnel. Brine (20 mL) was added, and the aqueous layer was extracted with Et_2O (50 mL x3). The combined organic layers were dried over magnesium sulfate, filtered, and concentrated. The product was purified by flash chromatography with 5→40% Et_2O/hexanes, which furnished the 1-(hept-6-en-1-yl)-1*H*-indole as a red oil (1.0 g, 54%).

^{1}H NMR ($CDCl_3$) δ 8.05–8.04 (m, 1H), 7.67–7.65 (m, 1H), 7.59–7.53 (m, 1H), 7.51–7.50 (m, 1H), 7.34 (d, 1H, J = 2.2 Hz), 6.88–6.83 (m, 1H), 6.12–6.10 (m, 1H), 5.41–5.35 (m, 2H), 4.29 (t, 2H, J = 7.1 Hz), 2.38–2.36 (m, 2H), 2.11–2.04 (m, 2H), 1.75–1.68 (m, 2H), 1.64–1.53 (m, 2H).

^{13}C NMR ($CDCl_3$) δ 139.0, 136.4, 129.1, 128.1, 121.7, 121.4, 119.6, 115.0, 109.8, 101.3, 46.6, 34.0, 30.5, 28.9, 26.8.

FT-IR (film) 3074, 2930, 2856, 1640, 1612, 1511, 1484, 1353, 1316, 910, 740 cm^{-1}.

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(7) Fang, G. Y.; Wallner, O. A.; Di Blasio, N.; Ginesta, X.; Harvey, J. N.; Aggarwal, V. K. *J. Am. Chem. Soc.* **2007**, *129*, 14632–14639.

(8) Xenon Pharmaceuticals, Inc. Spiro-Oxindole Compounds and Their Uses as Therapeutic Agents. WO2006/110917 A2, October 19, 2006; pp 74–75.

MS (EI) m/z ($M+H^+$) calcd for $C_{15}H_{20}N$: 214, found: 214.

IV. Catalytic Asymmetric γ-Alkylation of Carbonyl Compounds

General procedure for catalytic asymmetric γ-alkylations. In a nitrogen-filled glovebox, a solution of the organoboron reagent (670 μL, 1.0 mmol; 1.5 M) was added to a slurry of potassium *tert*-butoxide (78.5 mg, 0.70 mmol) and 1-hexanol (113 μL, 92 mg, 0.90 mmol) in a 4-mL vial equipped with a stir bar. The vial was sealed with a teflon-lined septum cap, and the mixture was stirred vigorously for 30 minutes and then used in the next step.

In a glovebox, $NiBr_2$·diglyme (17.6 mg, 0.050 mmol), (*R*,*R*)-**1** (14.5 mg, 0.060 mmol), hexanes (3.1 mL), and Et_2O (1.4 mL) were added in turn to a 20-mL vial equipped with a stir bar. The vial sealed with a teflon-lined septum cap, and the mixture was stirred vigorously for 45 minutes (a light-blue slurry forms). The solution of the activated organoboron reagent was then added to the slurry, and the vial was sealed with a teflon-lined cap and stirred for 30 minutes (the reaction mixture turns brown). The electrophile (0.50 mmol in 0.5 mL of Et_2O; purified) was added to the slurry via syringe, and the vial that contained the electrophile was then rinsed with additional Et_2O (0.5 mL), and this solution was added to the slurry. The mixture was sealed with a teflon-lined cap and stirred vigorously at room temperature for 24 hours (outside of the glovebox). Next, the reaction mixture was passed through a short plug of silica gel, eluting with Et_2O. The solution was concentrated to furnish an oil, which was purified by reverse-phase flash chromatography on C-18 silica gel with 10→100% acetonitrile/water.

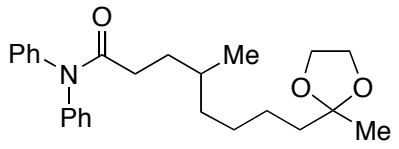
A second run was conducted with (*S*,*S*)-**1**.

Glovebox-free procedure for catalytic asymmetric γ-alkylation (Table 1, entry 4). A 25-mL two-neck round-bottom flask equipped with a stir bar was capped with a septum and with a hose adapter, which was connected to a Schlenk line. The flask was placed under vacuum and flame-dried. The flask was then filled with nitrogen, and, under a positive pressure of nitrogen, 9-BBN dimer (732 mg, 3.0 mmol) was added. The flask was purged with nitrogen for 3 minutes, and then 1-allyl-4-methoxybenzene (890 mg, 6.0 mmol) was added via syringe. Next, anhydrous Et_2O was added by syringe to bring the concentration to 1.5 M, and the mixture was heated at 40 °C for 1.5 hours, during which time it became homogenous. The solution was allowed to cool to room temperature and then used in the next step.

A 50-mL two-neck round-bottom flask equipped with a stir bar was capped with a septum and with a hose adapter, which was connected to a Schlenk line. The flask was placed under vacuum and flame-dried. The flask was then filled with nitrogen, and, under a positive pressure of nitrogen, potassium *tert*-butoxide (78.5 mg, 0.70 mmol) was added. The flask was purged with nitrogen for 3 minutes, and then anhydrous 1-hexanol (92 mg, 113 μL, 0.90 mmol) and a solution of the *B*-alkyl-(9-BBN) reagent (670 μL, 1.0 mmol; 1.5 M) were added in turn via syringe. The resulting mixture was stirred vigorously for 30 minutes and then used in the next step.

A 50-mL two-neck round-bottom flask equipped with a stir bar was capped with a septum and with a hose adapter, which was connected to a Schlenk line. The flask was placed under vacuum and flame-dried. The flask was then filled with nitrogen, and, under a positive pressure of nitrogen, $NiBr_2$·diglyme (17.6 mg, 0.050 mmol) and (*R*,*R*)-**1** (14.5 mg, 0.060 mmol)

were added. The flask was purged with nitrogen for 3 minutes, and then anhydrous hexanes (3.1 mL) and Et_2O (1.4 mL) were added via syringe. The mixture was stirred vigorously for 45 minutes (a light-blue slurry forms). The solution of the activated *B*-alkyl-(9-BBN) reagent was then transferred to the slurry via cannula, and the reaction mixture was stirred for 30 minutes (the reaction mixture turns brown). The electrophile (151 mg, 0.50 mmol in 0.5 mL of Et_2O; in a flame-dried flask under nitrogen) was added to this reaction mixture via syringe, and the flask that contained the electrophile was rinsed (under nitrogen) with an additional 0.5 mL of Et_2O, which was also added to the slurry via syringe. The reaction mixture was stirred vigorously under nitrogen for 24 hours at room temperature. Next, the mixture was passed through a short plug of silica gel, eluting with Et_2O. The solution was concentrated to furnish an oil, which was purified by reverse-phase flash chromatography on C-18 silica gel with 10→100% acetonitrile/water. Colorless oil (147 mg, 71%; 88% ee).



4-Methyl-8-(2-methyl-1,3-dioxolan-2-yl)-*N*,*N*-diphenyloctanamide (Table 1, Entry 1). The title compound was prepared according to the general procedure, using 4-chloro-*N*,*N*-diphenylpentanamide (144 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 2-(but-3-en-1-yl)-2-methyl-1,3-dioxolane[9] with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 125 mg (63%, 84% ee). Second run: 123 mg (62%, 85% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 32.9 min (minor), 37.9 min (major)).

^{1}H NMR ($CDCl_3$) δ 7.33–7.21 (m, 10H), 3.92–3.87 (m, 4H), 2.25–2.19 (m, 2H), 1.71–1.61 (m, 1H), 1.58–1.54 (m, 2H), 1.49–1.40 (m, 1H), 1.30–1.15 (m, 9H), 1.08–0.99 (m, 1H), 0.71 (d, 3H, J = 6.8 Hz).

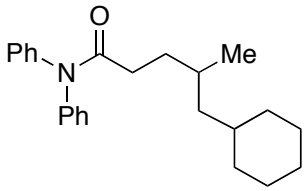
^{13}C NMR ($CDCl_3$) δ 173.5, 143.0, 129.2–126.8 (broad), 110.1, 64.6 (2C), 39.2, 36.6, 33.0, 32.6, 32.3, 27.1, 24.3, 23.8, 19.4.

FT-IR (film) 3438, 2941, 2870, 1673, 1595, 1492, 1375, 1273, 1051, 757, 701 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{25}H_{34}NO_3$: 396, found: 396.

$[\alpha]^{25}_D$ = 0.61 (c = 1.06, $CHCl_3$; obtained with (*S*,*S*)-**1**).

(9) Collins, P. W.; Gasiecki, A. F.; Perkins, W. E.; Gullikson, G. W.; Jones, P. H.; Bauer, R. F. *J. Med. Chem.* **1989**, *32*, 1001–1006.



5-Cyclohexyl-4-methyl-*N*,*N*-diphenylpentanamide (Table 1, Entry 2). The title compound was prepared according to the general procedure, except that the catalyst loading was doubled: $NiBr_2$·diglyme (35.2 mg, 0.10 mmol) and **1** (29 mg, 0.12 mmol). 4-Chloro-*N*,*N*-diphenylpentanamide (144 mg, 0.50 mmol) was used, along with a solution of the alkylborane prepared by hydroboration of methylenecyclohexane with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). White solid.

First run: 96 mg (55%, 90% ee). Second run: 91 mg (52%, 90% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 2% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 19.3 min (minor), 25.0 min (major)).

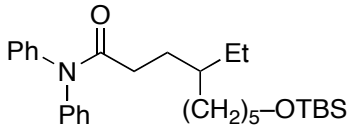
^{1}H NMR ($CDCl_3$) δ 7.34–7.25 (m, 10H), 2.31–2.17 (m, 2H), 1.65–1.62 (m, 6H), 1.44–1.36 (m, 2H), 1.20–1.15 (m, 4H), 1.05–0.95 (m, 1H), 0.95–0.88 (m, 1H), 0.88–0.70 (m, 5H).

^{13}C NMR ($CDCl_3$) δ 173.7, 143.1, 129.2–125.0 (broad), 44.9, 34.8, 34.0, 33.3, 33.0, 32.9, 29.2, 26.7, 26.4, 26.3, 19.6.

FT-IR (film) 2921, 2850, 2360, 1675, 1593, 1491, 1449, 1375, 1272, 755, 701 cm^{-1}.

MS (EI) *m*/*z* (M+H$^+$) calcd for $C_{24}H_{32}NO$: 350, found: 350.

$[\alpha]^{24}_D$ = –1.2 (*c* = 1.26, $CHCl_3$; obtained with (*S*,*S*)-**1**).



9-((*tert*-Butyldimethylsilyl)oxy)-4-ethyl-*N*,*N*-diphenylnonanamide (Table 1, Entry 3; eq 6). The title compound was prepared according to the general procedure, using 4-chloro-*N*,*N*-diphenylhexanamide (151 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of *tert*-butyldimethyl(pent-4-en-1-yloxy)silane[10] with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 178 mg (76%, 90% ee). Second run: 168 mg (72%, 92% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 1% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 12.6 min (minor), 13.2 min (major)).

^{1}H NMR ($CDCl_3$) δ 7.33–7.22 (m, 10H), 3.55 (t, 2H, *J* = 6.8 Hz), 2.22–2.18 (m, 2H), 1.61–1.56 (m, 2H), 1.45–1.42 (m, 2H), 1.19–1.10 (m, 9H), 0.86 (s, 9H), 1.06 (t, 3H, *J* = 7.2 Hz), 0.01 (s, 6H).

^{13}C NMR ($CDCl_3$) δ 173.7, 143.1, 129.3–126.6 (broad), 63.3, 38.5, 32.9, 32.8, 29.0, 26.3, 26.2, 26.0, 25.6, 18.4, 10.7, –4.9, –5.2.

FT-IR (film) 2929, 2857, 1676, 1594, 1492, 1462, 1360, 1255, 1098, 835, 775, 755, 701 cm^{-1}.

MS (EI) *m*/*z* (M+H–*t*-Bu$^+$) calcd for $C_{29}H_{46}NO_2Si$: 468, found: 411.

$[\alpha]^{24}_D$ = –0.84 (*c* = 1.1, $CHCl_3$; obtained with (*S*,*S*)-**1**).

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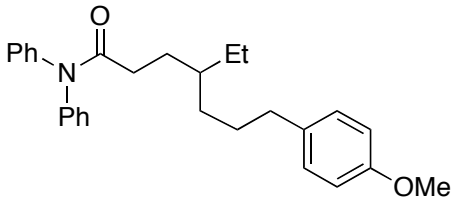
(10) Liang, B.; Negishi, E.-i. *Org. Lett.* **2008**, *10*, 193–195.

When 4-bromo-*N,N*-diphenylhexanamide (173 mg, 0.50 mmol) was employed as the electrophile (eq 6): First run: 164 mg (60%, 86% ee). Second run: 166 mg (62%, 86% ee).



4-Ethyl-7-(4-methoxyphenyl)-*N,N*-diphenylheptanamide (Table 1, Entry 4). The title compound was prepared according to the general procedure, using 4-chloro-*N,N*-diphenylhexanamide (151 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-4-methoxybenzene with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 164 mg (79%, 89% ee). Second run: 166 mg (80%, 88% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 17.2 min (minor), 18.4 min (major)).

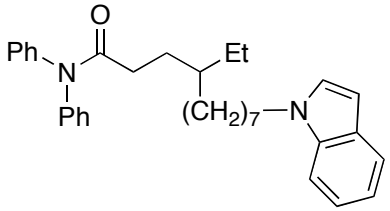
^{1}H NMR ($CDCl_3$) δ 7.33–7.20 (m, 10H), 7.02 (d, 2H, J = 8.4 Hz), 6.78 (d, 2H, J = 4.0 Hz), 3.76 (s, 3H), 2.43 (t, 2H, J = 6.8 Hz), 2.25–2.16 (m, 2H, J = 8.0 Hz), 1.61–1.54 (m, 2H), 1.49–1.41 (m, 2H), 1.30–1.18 (m, 5H), 0.77 (t, 3H, J = 7.6 Hz).

^{13}C NMR ($CDCl_3$) δ 173.7, 157.7, 143.1, 134.8, 129.3, 127.4–125.6 (broad), 113.7, 55.3, 38.4, 35.3, 32.8, 32.4, 29.0, 28.6, 25.6, 10.7.

FT-IR (film) 2931, 1674, 1594, 1558, 1540, 1512, 1491, 1456, 1245, 1177, 1035, 756, 702 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{28}H_{34}NO_2$: 416, found: 416.

$[\alpha]^{24}_D$ = 1.8 (c = 1.26, $CHCl_3$; obtained with (*R,R*)-**1**).



4-Ethyl-11-(1*H*-indol-1-yl)-*N,N*-diphenylundecanamide (Table 1, Entry 5). The title compound was prepared according to the general procedure, using 4-chloro-*N,N*-diphenylhexanamide (151 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-(hept-6-en-1-yl)-1*H*-indole with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Yellow oil.

First run: 147 mg (61%, 89% ee). Second run: 154 mg (64%, 90% ee).

The ee was determined by SFC analysis (CHIRALPAK AD-H, 10% MeOH; 3.0 mL/min; retention times (when *(R,R)*-**1** is employed): 43.0 min (major), 47.6 min (minor)).

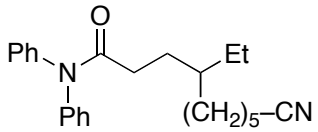
^{1}H NMR ($CDCl_3$) δ 7.66 (d, 1H, J = 8.0 Hz), 7.28–7.26 (m, 5H), 7.25–7.21 (m, 7H), 7.14–7.10 (m, 2H), 6.52–6.51 (m, 1H), 4.12 (t, 2H, J = 6.8 Hz), 2.26 (t, 2H, J = 7.6 Hz), 1.85–1.82 (m, 2H), 1.67–1.62 (m, 2H), 1.30–1.11 (m, 13H), 0.81 (t, 3H, J = 6.6 Hz).

^{13}C NMR ($CDCl_3$) δ 173.9, 143.3, 136.2, 129.4–127.2 (broad), 128.8, 128.0, 121.5, 121.2, 119.4, 109.6, 101.0, 46.6, 38.7, 33.0, 32.9, 30.5, 30.1, 29.5, 29.2, 27.2, 26.6, 25.8, 10.9.

FT-IR (film) 2927, 2855, 1673, 1592, 1491, 1464, 1315, 740, 702 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{33}H_{41}N_2O$: 481, found: 481.

$[\alpha]^{24}_D$ = 0.33 (c = 1.82, $CHCl_3$; obtained with (*S*,*S*)-**1**).



9-Cyano-4-ethyl-*N*,*N*-diphenylnonanamide (Table 1, Entry 6). The title compound was prepared according to the general procedure, except that the reaction was heated to 60 °C in *i*-Pr_2O, using 4-chloro-*N*,*N*-diphenylhexanamide (151 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of hex-5-enenitrile with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 94 mg (52%, 68% ee). Second run: 91 mg (50%, 70% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 5% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 55.8 min (minor), 59.2 min (major)).

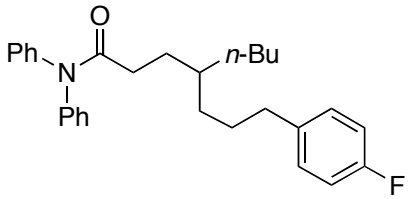
^{1}H NMR ($CDCl_3$) δ 7.28–7.18 (m, 10H), 2.28 (t, 2H, J = 7.2 Hz), 2.24–2.18 (m, 2H), 1.61–1.55 (m, 5H), 1.40–1.29 (m, 2H), 1.25–1.00 (m, 6H), 0.74 (t, 3H, J = 7.0 Hz).

^{13}C NMR ($CDCl_3$) δ 173.6, 143.0, 130.2–126.8 (broad), 119.9, 38.4, 32.7, 32.4, 29.0, 28.9, 25.7, 25.5, 25.3, 17.1, 10.7.

FT-IR (film) 2931, 2859, 1671, 1595, 1491, 1452, 1357, 1273, 757, 703 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{24}H_{31}N_2O$: 363, found: 363.

$[\alpha]^{24}_D$ = –1.3 (c = 1.45, $CHCl_3$; obtained with (*R*,*R*)-**1**).



4-(3-(4-Fluorophenyl)propyl)-*N*,*N*-diphenyloctanamide (Table 1, Entry 7). The title compound was prepared according to the general procedure, using 4-chloro-*N*,*N*-diphenyloctanamide (165 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-4-fluorobenzene with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 134 mg (62%, 89% ee). Second run: 140 mg (65%, 90% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 3% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 19.2 min (minor), 20.8 min (major)).

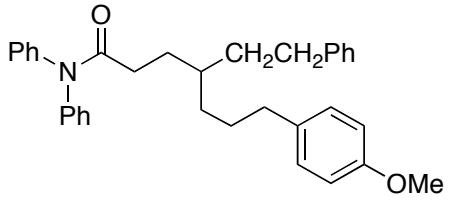
^{1}H NMR ($CDCl_3$) δ 7.32–7.20 (m, 10H), 7.07–7.03 (m, 2H), 6.93–6.89 (m, 2H), 2.46 (t, 2H, J = 7.6 Hz), 2.20–2.16 (m, 2H), 1.61–1.56 (m, 2H), 1.48–1.44 (m, 2H), 1.24–1.07 (m, 9H), 0.80 (t, 3H, J = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 173.6, 161.2 (d, J = 964 Hz), 143.0, 138.2, 129.7, 129.6–126.6 (broad), 115.0, 114.8, 36.9, 35.5, 35.4, 33.1, 32.9, 29.4, 28.7, 28.4, 23.0, 14.1.

FT-IR (film) 2928, 2858, 2361, 2340, 1675, 1598, 1509, 1491, 1362, 1273, 1220, 1157, 832, 756, 702, 668 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{29}H_{35}FNO$: 432, found: 432.

$[\alpha]^{24}_D$ = 1.9 (c = 1.21, $CHCl_3$; obtained with (*S*,*S*)-**1**).



7-(4-Methoxyphenyl)-4-phenethyl-*N*,*N*-diphenylheptanamide (Table 1, Entry 8). The title compound was prepared according to the general procedure, using 4-chloro-*N*,*N*-6-triphenylhexanamide (189 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-4-methoxybenzene with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 202 mg (82%, 88% ee). Second run: 206 mg (84%, 88% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 3% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 56.7 min (minor), 65.3 min (major)).

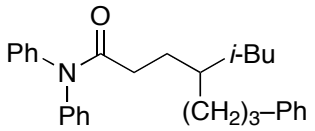
^{1}H NMR ($CDCl_3$) δ 7.33–7.14 (m, 13H), 7.07–7.02 (m, 4H), 6.81–6.79 (m, 2H), 3.77 (s, 3H), 2.48–2.43 (m, 4H), 2.25–2.20 (m, 2H), 1.70–1.68 (m, 2H), 1.54–1.35 (m, 5H), 1.22–1.19 (m, 2H).

^{13}C NMR ($CDCl_3$) δ 173.5, 157.7, 143.0, 142.9, 134.7, 130.0–125.0 (broad), 129.3, 128.4, 128.3, 125.7, 113.7, 55.3, 53.5, 36.6, 35.3, 32.9, 32.71, 32.66, 29.3, 28.5.

FT-IR (film) 2930, 2857, 1672, 1594, 1511, 1491, 1452, 1245, 1177, 1033, 756, 701 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{34}H_{38}NO_2$: 492, found: 492.

$[\alpha]^{24}_D$ = 0.84 (c = 1.12, $CHCl_3$; obtained with (*S*,*S*)-**1**).



4-Isobutyl-*N*,*N*,7-triphenylheptanamide (Table 1, Entry 9). The title compound was prepared according to the general procedure, using 4-chloro-6-methyl-*N*,*N*-diphenylheptanamide (165 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 124 mg (60%, 82% ee). Second run: 128 mg (62%, 82% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 3% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 15.8 min (minor), 17.9 min (major)).

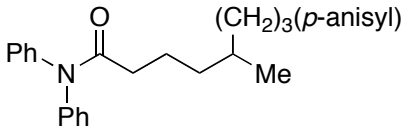
^{1}H NMR ($CDCl_3$) δ 7.32–7.10 (m, 15H), 2.48 (t, 2H, J = 7.6 Hz), 2.21–2.16 (m, 2H), 1.60–1.56 (m, 2H), 1.53–1.40 (m, 3H), 1.39–1.30 (m, 1H), 1.14–1.10 (m, 2H), 0.96–0.95 (m, 1H), 0.88–0.86 (m, 1H), 0.77–0.74 (m, 6H).

^{13}C NMR ($CDCl_3$) δ 173.7, 143.1, 142.7, 129.2–125.0 (broad), 128.5, 128.3, 125.7, 43.4, 36.4, 34.6, 33.3, 32.6, 29.6, 28.2, 25.2, 23.1, 22.9.

FT-IR (film) 2952, 2929, 1675, 1594, 1492, 1453, 1364, 1271, 755, 700 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{29}H_{36}NO$: 414, found: 414.

$[\alpha]^{24}_D$ = 0.79 (c = 1.07, $CHCl_3$; obtained with (*R,R*)-**1**).



8-(4-Methoxyphenyl)-5-methyl-*N,N*-diphenyloctanamide (eq 7). The title compound was prepared according to the general procedure, using 5-chloro-*N,N*-diphenylhexanamide (144 mg, 0.50 mmol) and a solution of the alkylborane prepared by hydroboration of 1-allyl-4-methoxybenzene with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 131 mg (63%, 83% ee). Second run: 135 mg (65%, 84% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 10% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 18.3 min (minor), 19.8 min (major)).

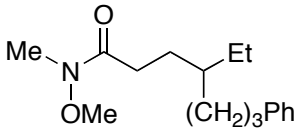
^{1}H NMR ($CDCl_3$) δ 7.33–7.21 (m, 10H), 7.04–7.02 (m, 2H), 6.80–6.78 (m, 2H), 3.76 (s, 3H), 2.44 (t, 2H, J = 7.6 Hz), 2.21–2.17 (m, 2H), 1.62–1.57 (m, 2H), 1.48–1.44 (m, 2H), 1.20–1.11 (m, 5H), 0.75–0.71 (m, 3H).

^{13}C NMR ($CDCl_3$) δ 173.6, 157.6, 143.0, 134.8, 129.2, 129.2–125.2 (broad), 113.7, 55.3, 38.4, 35.3, 32.8, 32.4, 29.0, 28.6, 25.5, 10.7.

FT-IR (film) 2931, 2858, 1674, 1594, 1512, 1491, 1457, 1374, 1246, 1035, 756, 702 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{28}H_{34}NO_2$: 416, found: 416.

$[\alpha]^{24}_D$ = 1.3 (c = 1.30, $CHCl_3$; obtained with (*R,R*)-**1**).



4-Ethyl-*N*-methoxy-*N*-methyl-7-phenylheptanamide (eq 8). The title compound was prepared according to the general procedure, except that potassium iodide (21 mg, 0.13 mmol; water content: 180 ppm) was added to the vial containing $NiBr_2$·diglyme and **1**, before the solvent was added. 4-Chloro-*N*-methoxy-*N*-methylhexanamide (97 mg, 0.50 mmol) was used, along with a solution of the alkylborane prepared by hydroboration of allylbenzene with 9-BBN dimer (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 87 mg (63%, 86% ee). Second run: 86 mg (62%, 86% ee).

The ee was determined by HPLC analysis (CHIRALPAK OJ-H, 1% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 14.2 min (minor), 15.0 min (major)).

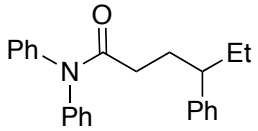
^{1}H NMR ($CDCl_3$) δ 7.24–7.22 (m, 2H), 7.16–7.12 (m, 3H), 3.62 (s, 3H), 3.14 (s, 3H), 2.56 (t, 2H, *J* = 7.6 Hz), 2.33 (t, 2H, *J* = 7.6 Hz), 1.62–1.54 (m, 4H), 1.33–1.24 (m, 5H), 0.86 (t, 3H, *J* = 7.2 Hz).

^{13}C NMR ($CDCl_3$) δ 175.0, 142.8, 128.4, 128.2, 125.6, 61.2, 38.5, 36.3, 32.5, 32.2, 29.4, 28.5, 27.9, 25.6, 10.8.

FT-IR (film) 2930, 1670, 1457, 1382, 1003, 747, 699 cm^{-1}.

MS (EI) *m*/*z* (M+H$^+$) calcd for $C_{17}H_{28}NO_2$: 278, found: 278.

$[\alpha]^{24}_D$ = 0.42 (*c* = 4.40, $CHCl_3$; obtained with (*R*,*R*)-**1**).



***N*,*N*,4-Triphenylhexanamide (eq 10 and eq 9).** The title compound was prepared according to the general procedure, using 4-chloro-*N*,*N*-diphenylhexanamide (151 mg, 0.50 mmol), along with a solution of 4,4,5,5-tetramethyl-2-phenyl-1,3,2-dioxaborolane (Aldrich; diluted to 1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

First run: 81 mg (47%, 83% ee). Second run: 81 mg (47%, 81% ee).

The ee was determined by HPLC analysis (CHIRALPAK OD-H, 3% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when ligand *(R,R)*-**1** is employed): 16.0 min (minor), 17.5 min (major)).

^{1}H NMR ($CDCl_3$) δ 7.28–7.00 (m, 15H), 2.41–2.40 (m, 1H), 2.09–2.03 (m, 3H), 1.85–1.75 (m, 1H), 1.62–1.48 (m, 2H), 0.72 (t, 3H, *J* = 7.2 Hz).

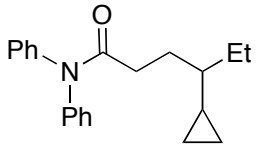
^{13}C NMR ($CDCl_3$) δ 173.2, 144.7, 142.8, 128.2, 127.8, 126.0, 130.0–125.0 (broad), 47.0, 33.3, 31.8, 29.8, 12.1.

FT-IR (film) 3060, 2827, 1680, 1593, 1492, 1375, 1270, 756, 700 cm^{-1}.

MS (ESI/APCI) *m*/*z* (M+H$^+$) calcd for $C_{24}H_{26}NO$: 344, found: 344.

$[\alpha]^{25}_D$ = 11 (*c* = 1.80, $CHCl_3$; obtained with (*R*,*R*)-**1**).

When *B*-phenyl-(9-BBN)[11] was employed instead of 4,4,5,5-tetramethyl-2-phenyl-1,3,2-dioxaborolane (eq 9): First run: 75 mg (44%, 80% ee). Second run: 77 mg (45%, 78% ee).



4-Cyclopropyl-*N*,*N*-diphenylhexanamide (eq 11). The title compound was prepared according to the general procedure, except that Et_2O was the only solvent (0.08 M) and the catalyst loading was doubled: $NiBr_2$·diglyme (35.2 mg, 0.10 mmol) and **1** (29 mg, 0.12 mmol). 4-Chloro-6-methyl-*N*,*N*-diphenylheptanamide (165 mg, 0.50 mmol) was used, along with a solution of *B*-cyclopropyl-(9-BBN) (1.5 M in Et_2O; 670 μL, 1.0 mmol). Colorless oil.

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(11) Fang, G. Y.; Wallner, O. A.; Di Blasio, N.; Ginesta, X.; Harvey, J. N.; Aggarwal, V. K. *J. Am. Chem. Soc.* **2007**, *129*, 14632–14639.

First run: 108 mg (68%, 84% ee). Second run: 109 mg (71%, 83% ee).

The ee was determined by HPLC analysis (CHIRALPAK OJ-H, 1% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when *(R,R)*-**1** is employed): 14.2 min (minor), 15.0 min (major)).

^{1}H NMR ($CDCl_3$) δ 7.33–7.00 (m, 10H), 2.42–2.27 (m, 2H), 1.82–1.67 (m, 2H), 1.32–1.25 (m, 2H), 0.84 (t, 3H, J = 7.2 Hz), 0.44–0.39 (m, 1H), 0.32–0.29 (m, 3H), –0.03– –0.10 (m, 2H).

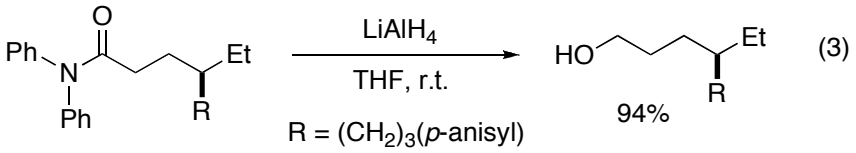
^{13}C NMR ($CDCl_3$) δ 173.8, 143.1, 129.3–126.5 (broad), 44.4, 33.2, 30.3, 27.5, 15.5, 11.3, 4.0, 3.6.

FT-IR (film) 2961, 2924, 1675, 1491, 1373, 1271, 756, 702, 693 cm^{-1}.

MS (EI) m/z (M+H$^+$) calcd for $C_{21}H_{26}NO$: 308, found: 308.

$[\alpha]^{24}_D$ = –0.80 (c = 1.54, $CHCl_3$; obtained with (*S*,*S*)-**1**).

V. Transformation of the Cross-Coupling Products



(3)

(*S*)-4-Ethyl-7-(4-methoxyphenyl)heptan-1-ol (eq 3). (*S*)-7-(4-Methoxyphenyl)-4-ethyl-*N*,*N*-diphenylheptanamide (100 mg, 0.24 mmol; 89% ee) and THF (13 mL) were added to an oven-dried two-neck round-bottom flask under nitrogen. This solution was cooled to 0 °C, and a solution of lithium aluminum hydride (1.45 mL, 2.9 mmol; 2.0 M in THF) was added dropwise with stirring. The mixture was allowed to warm to room temperature, and it was stirred for 12 h. The reaction mixture was then cooled to 0 °C, and the reaction was quenched by the addition of water (1 mL). The mixture was filtered through Celite, which was washed with THF. The filtrate was concentrated, and the residue was purified by flash chromatography with 10→70% Et_2O/hexanes, which furnished the product as a clear oil. First run: 55 mg (92%, 88% ee). Second run: 57 mg (95%, 88% ee).

The ee was determined by HPLC analysis (CHIRALPAK IC, 2% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 41.9 min (minor), 44.0 min (major)).

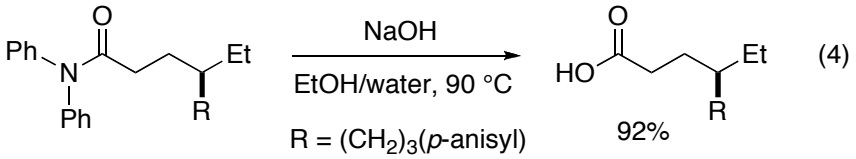
^{1}H NMR ($CDCl_3$) δ 7.07 (d, 2H, J = 8.0 Hz), 6.81–6.79 (m, 2H), 3.76 (s, 3H), 3.58 (t, 2H, J = 6.7 Hz), 2.50 (t, 2H, J = 8.0 Hz), 1.54 (s, 1H), 1.54–1.50 (m, 4H), 1.28–1.26 (m, 7H), 0.81 (t, 3H, J = 6.8 Hz).

^{13}C NMR ($CDCl_3$) δ 157.6, 134.9, 129.2, 113.7, 63.5, 55.3, 38.6, 35.5, 32.7, 30.0, 29.1, 28.9, 25.8, 10.8.

FT-IR (film) 3336 (broad), 2932, 2858, 2360, 2340, 1512, 1457, 1419, 1245, 1039, 829.

MS (EI) m/z (M+H$^+$) calcd for $C_{16}H_{27}O_2$: 251, found: 251.

$[\alpha]^{24}_D$ = –2.0 (c = 2.67, $CHCl_3$).



(*S*)-4-Ethyl-7-(4-methoxyphenyl)heptanoic acid (eq 4). (*S*)-7-(4-Methoxyphenyl)-4-ethyl-*N*,*N*-diphenylheptanamide (100 mg, 0.24 mmol; 89% ee), EtOH (7 mL), water (0.5 mL), and then sodium hydroxide (0.93 mg of a 30% w/w solution) were added to a 20-mL vial, which was then sealed with a septum cap and heated to 90 °C for 8 h. The reaction mixture was allowed to cool to room temperature, and then 2 N HCl (2 mL) was added. The mixture was transferred to a separatory funnel, to which Et_2O (50 mL) and brine (50 mL) were added. The layers were separated, and the aqueous layer was washed with Et_2O (50 mL x2). The combined organic layers were dried over magnesium sulfate, filtered, and concentrated. The product was purified by flash chromatography with 10→70% Et_2O/hexanes, which furnished the product as a clear oil. First run: 57 mg (90%, 88% ee). Second run: 59 mg (93%, 88% ee).

The ee was determined by HPLC analysis (CHIRALPAK IC, 1% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 24.7 min (major), 27.9 min (minor)).

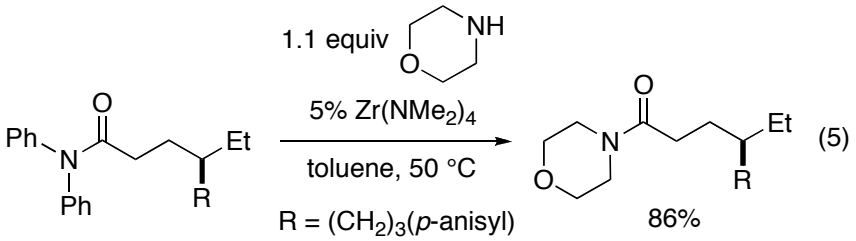
^{1}H NMR ($CDCl_3$) δ 7.04 (d, 2H, *J* = 8.4 Hz), 6.86 (d, 2H, *J* = 8.4 Hz), 3.77 (s, 3H), 2.51 (t, 2H, *J* = 7.6 Hz), 2.29 (t, 2H, *J* = 7.6 Hz), 1.61–1.51 (m, 4H), 1.29–1.26 (m, 5H), 0.81 (t, 3H, *J* = 6.8 Hz).

^{13}C NMR ($CDCl_3$) δ 180.1, 157.8, 134.9, 129.4, 113.9, 55.4, 38.4, 35.5, 32.5, 31.7, 28.8, 28.1, 25.6, 10.9.

FT-IR (film) 2932 (broad), 2859, 1708, 1512, 1457, 1300, 1245, 1177, 1039, 829.

MS (EI) *m*/*z* (M+H$^+$) calcd for $C_{16}H_{25}O_3$: 265, found: 265.

$[\alpha]^{24}_D = -0.62$ (*c* = 2.22, $CHCl_3$).



***(S)*-4-Ethyl-7-(4-methoxyphenyl)-1-morpholinoheptan-1-one (eq 5)** (modified from a literature method[12]). In a nitrogen-filled glovebox, activated molecular sieves (20 mg; 4 Å), (*S*)-4-ethyl-7-(4-methoxyphenyl)-*N*,*N*-diphenylheptanamide (50 mg, 0.12 mmol; 89% ee), and toluene (0.36 mL; anhydrous, Aldrich) were added to a flame-dried 4-mL vial equipped with a stir bar. The mixture was stirred for 20 minutes, and then it was filtered through a 2 μm acrodisc filter into another flame-dried 4-mL vial equipped with a stir bar (the original vial was rinsed with toluene (0.1 mL x2), and the washings were filtered through the acrodisc into the second vial). Freshly distilled morpholine (23 μL, 0.27 mmol) was added to the vial by syringe. In another flame-dried 4-mL vial, a stock solution of $Zr(NMe_2)_4$ in anhydrous toluene was

(12) Stephenson, N. A.; Zhu, J.; Gellman, S. H.; Stahl, S. S. *J. Am. Chem. Soc.* **2009**, *131*, 10003–10008.

prepared (10.8 mg per 1.0 mL of toluene). This solution (143 μL, 1.6 mg, 0.0060 mmol) was added to the solution of amine and amide, immediately resulting in a light-yellow solution. The vial was sealed with a teflon-lined septum cap, and the mixture was stirred at 50 °C for 10 hours. The reaction mixture was then allowed to cool to room temperature, the solvent was removed, and the product was purified by reverse-phase flash chromatography on C-18 silica gel with 10→100% acetonitrile/water.

A second run was conducted using (*R*)-4-ethyl-7-(4-methoxyphenyl)-*N*,*N*-diphenylheptanamide and afforded *(R)*-4-ethyl-7-(4-methoxyphenyl)-1-morpholinoheptan-1-one.

First run: 35 mg (87%, 89% ee). Second run: 34 mg (85%, 89% ee).

The ee was determined by HPLC analysis (CHIRALPAK AD-H, 3% *i*-PrOH in hexanes; 1.0 mL/min; retention times (when (*S*)-4-ethyl-7-(4-methoxyphenyl)-*N*,*N*-diphenylheptanamide is employed): 28.6 min (major), 30.7 min (minor)).

^{1}H NMR ($CDCl_3$) δ 7.05 (d, 2H, *J* = 5.2 Hz), 6.79 (d, 2H, *J* = 4.8 Hz), 3.75 (s, 3H), 3.64–3.57 (m, 5H), 3.38 (t, 2H, *J* = 5.2 Hz), 2.92 (d, 1H, *J* = 16.8 Hz), 2.50 (t, 2H, *J* = 7.6 Hz), 2.20 (t, 2H), 1.58–1.50 (m, 4H), 1.29–1.28 (m, 5H), 0.81 (t, 3H, *J* = 7.1 Hz).

^{13}C NMR ($CDCl_3$) δ 172.1, 157.7, 134.7, 129.3, 113.7, 67.0, 66.7, 55.3, 46.0, 41.9, 38.6, 35.3, 32.3, 30.5, 28.6, 28.5, 25.7, 10.8.

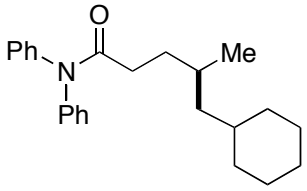
FT-IR (film) 2930, 2856, 2361, 2339, 1653, 1512, 1457, 1300, 1245, 1116, 1035, 830 cm^{-1}.

MS (EI) *m*/*z* ($M+H^+$) calcd for $C_{20}H_{32}NO_3$: 334, found: 334.

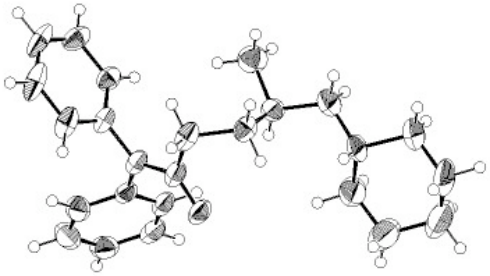
$[\alpha]^{24}_D$ = 0.97 (*c* = 0.95, $CHCl_3$; *(S)*-4-ethyl-7-(4-methoxyphenyl)-1-morpholinoheptan-1-one).

VI. Determination of Absolute Configuration

Assignment of absolute configuration of the γ-alkylated products. The absolute configuration of the product of entry 2 of Table 1 (using ligand (*S*,*S*)-**1**) was determined by X-ray crystallography. The absolute configurations of the other γ-alkylation products were assigned by analogy.



The cross-coupling product was purified to >99% ee by chiral HPLC (CHIRALPAK AD-H). Crystals suitable for X-ray structural analysis were obtained by solvent evaporation of a pentane solution.



Half of the molecule (C2 - C12) was modeled as a two-part disorder (68:32). Pictured above is one of the two modeled structures.

Absolute configuration: The Flack test is inconclusive due to quality of the data. However, the method by Spek and Hooft, which is based on Bayesian statistics, results in the following probabilities: The probability P2 of the model to be correct assuming that the sample is KNOWN to be enantiomerically pure is 1.0. The probability P3 of the model to be correct assuming that the structure is either right or wrong or a 50:50 racemic twin is 0.90. The probability of the model to be a 50:50 racemic twin is 0.10. The inverted model gives rise to opposite results in the Bayesian statistics, further improving the confidence in the absolute configuration as determined by X-ray diffraction.

Table 1. Crystal data and structure refinement for x10032_t4.

Identification code	x10032_t4	
Empirical formula	C24 H31 N O	
Formula weight	349.50	
Temperature	100(2) K	
Wavelength	1.54178 ≈	
Crystal system	Monoclinic	
Space group	C2	
Unit cell dimensions	a = 17.5701(5) ≈	α= 90∞.
	b = 5.6668(2) ≈	β= 96.082(2)∞.
	c = 20.4694(7) ≈	γ = 90∞.
Volume	2026.59(12) ≈^{3}	
Z	4	
Density (calculated)	1.145 Mg/m^3	
Absorption coefficient	0.524 mm^{-1}	
F(000)	760	
Crystal size	0.20 x 0.02 x 0.02 mm^3	
Theta range for data collection	2.17 to 67.77∞.	
Index ranges	-20<=h<=20, -6<=k<=5, -24<=l<=24	
Reflections collected	3273	
Independent reflections	3273 [R(int) = 0.0000]	
Completeness to theta = 67.77∞	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9922 and 0.9024	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3273 / 383 / 326	
Goodness-of-fit on F^2	1.054	
Final R indices [I>2sigma(I)]	R1 = 0.0476, wR2 = 0.1306	
R indices (all data)	R1 = 0.0506, wR2 = 0.1328	
Absolute structure parameter	-0.1(4)	
Largest diff. peak and hole	0.329 and -0.159 e.≈$^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($\approx^2$x 10^3) for x10032_t4. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	7176(1)	2053(3)	3198(1)	48(1)
C(1)	6488(1)	2303(4)	3075(1)	43(1)
C(2)	6151(1)	3704(5)	2478(1)	51(1)
C(3)	6590(2)	3275(8)	1842(2)	43(1)
C(4)	6577(3)	764(9)	1616(2)	43(1)
C(5)	5760(2)	-186(9)	1445(2)	61(1)
C(6)	7008(2)	374(9)	1005(2)	60(1)
C(7)	7831(3)	1169(12)	1064(2)	56(1)
C(8)	8309(3)	-92(13)	1632(2)	75(2)
C(9)	9135(3)	511(18)	1645(2)	111(2)
C(10)	9450(3)	-196(18)	999(3)	111(2)
C(11)	8997(3)	1136(19)	439(3)	104(2)
C(12)	8173(3)	544(15)	409(2)	90(2)
C(3A)	6735(5)	4233(17)	2105(5)	51(2)
C(4A)	7045(4)	2140(17)	1744(4)	54(2)
C(5A)	6405(7)	710(20)	1351(7)	70(3)
C(6A)	7653(5)	3111(17)	1357(4)	60(2)
C(7A)	8044(7)	1400(20)	943(6)	62(2)
C(8A)	8406(6)	-686(18)	1358(4)	59(2)
C(9A)	8843(5)	-2260(20)	967(5)	74(2)
C(10A)	9431(6)	-1030(20)	583(5)	80(3)
C(11A)	9095(8)	1110(30)	193(6)	85(3)
C(12A)	8672(6)	2730(20)	614(5)	75(2)
N(1)	5976(1)	1306(3)	3457(1)	40(1)
C(21)	6230(1)	-247(4)	3992(1)	39(1)
C(22)	5988(1)	123(5)	4602(1)	54(1)
C(23)	6207(2)	-1440(6)	5108(1)	64(1)
C(24)	6666(1)	-3361(5)	5012(1)	54(1)
C(25)	6911(1)	-3700(5)	4395(1)	48(1)
C(26)	6690(1)	-2173(4)	3891(1)	42(1)
C(31)	5162(1)	1771(4)	3363(1)	39(1)
C(32)	4873(1)	3876(5)	3574(1)	55(1)
C(33)	4088(2)	4251(6)	3485(2)	66(1)

C(34)	3604(1)	2565(6)	3209(1)	62(1)
C(35)	3889(1)	464(6)	3008(1)	54(1)
C(36)	4676(1)	64(4)	3084(1)	41(1)

Table 3. Bond lengths [≈] and angles [∞] for x10032_t4.

O(1)-C(1)	1.216(3)
C(1)-N(1)	1.374(3)
C(1)-C(2)	1.524(4)
C(2)-C(3A)	1.374(8)
C(2)-C(3)	1.600(4)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(2)-H(2C)	0.9900
C(2)-H(2D)	0.9900
C(3)-C(4)	1.496(7)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-C(5)	1.539(6)
C(4)-C(6)	1.544(5)
C(4)-H(4)	1.0000
C(5)-H(5A)	0.9800
C(5)-H(5B)	0.9800
C(5)-H(5C)	0.9800
C(6)-C(7)	1.508(7)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.537(7)
C(7)-C(12)	1.566(5)
C(7)-H(7)	1.0000
C(8)-C(9)	1.489(7)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(10)	1.540(8)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(10)-C(11)	1.525(9)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.480(8)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900

C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(3A)-C(4A)	1.529(12)
C(3A)-H(3A1)	0.9900
C(3A)-H(3A2)	0.9900
C(4A)-C(6A)	1.500(10)
C(4A)-C(5A)	1.543(13)
C(4A)-H(4A)	1.0000
C(5A)-H(5A1)	0.9800
C(5A)-H(5A2)	0.9800
C(5A)-H(5A3)	0.9800
C(6A)-C(7A)	1.500(13)
C(6A)-H(6A1)	0.9900
C(6A)-H(6A2)	0.9900
C(7A)-C(12A)	1.547(11)
C(7A)-C(8A)	1.554(11)
C(7A)-H(7A)	1.0000
C(8A)-C(9A)	1.468(11)
C(8A)-H(8A1)	0.9900
C(8A)-H(8A2)	0.9900
C(9A)-C(10A)	1.530(11)
C(9A)-H(9A1)	0.9900
C(9A)-H(9A2)	0.9900
C(10A)-C(11A)	1.533(12)
C(10A)-H(10C)	0.9900
C(10A)-H(10D)	0.9900
C(11A)-C(12A)	1.508(13)
C(11A)-H(11C)	0.9900
C(11A)-H(11D)	0.9900
C(12A)-H(12C)	0.9900
C(12A)-H(12D)	0.9900
N(1)-C(21)	1.438(3)
N(1)-C(31)	1.446(2)
C(21)-C(22)	1.377(3)
C(21)-C(26)	1.387(3)
C(22)-C(23)	1.385(4)
C(22)-H(22)	0.9500
C(23)-C(24)	1.382(4)

C(23)-H(23)	0.9500
C(24)-C(25)	1.389(4)
C(24)-H(24)	0.9500
C(25)-C(26)	1.371(3)
C(25)-H(25)	0.9500
C(26)-H(26)	0.9500
C(31)-C(36)	1.374(3)
C(31)-C(32)	1.384(3)
C(32)-C(33)	1.388(4)
C(32)-H(32)	0.9500
C(33)-C(34)	1.361(5)
C(33)-H(33)	0.9500
C(34)-C(35)	1.370(4)
C(34)-H(34)	0.9500
C(35)-C(36)	1.394(3)
C(35)-H(35)	0.9500
C(36)-H(36)	0.9500

O(1)-C(1)-N(1)	122.1(2)
O(1)-C(1)-C(2)	121.3(2)
N(1)-C(1)-C(2)	116.62(18)
C(3A)-C(2)-C(1)	108.0(4)
C(3A)-C(2)-C(3)	29.5(4)
C(1)-C(2)-C(3)	113.2(2)
C(3A)-C(2)-H(2A)	84.5
C(1)-C(2)-H(2A)	108.9
C(3)-C(2)-H(2A)	108.9
C(3A)-C(2)-H(2B)	134.2
C(1)-C(2)-H(2B)	108.9
C(3)-C(2)-H(2B)	108.9
H(2A)-C(2)-H(2B)	107.8
C(3A)-C(2)-H(2C)	110.1
C(1)-C(2)-H(2C)	110.1
C(3)-C(2)-H(2C)	81.5
H(2A)-C(2)-H(2C)	130.8
H(2B)-C(2)-H(2C)	30.3
C(3A)-C(2)-H(2D)	110.1
C(1)-C(2)-H(2D)	110.1

C(3)-C(2)-H(2D)	128.5
H(2A)-C(2)-H(2D)	28.2
H(2B)-C(2)-H(2D)	81.2
H(2C)-C(2)-H(2D)	108.4
C(4)-C(3)-C(2)	113.8(3)
C(4)-C(3)-H(3A)	108.8
C(2)-C(3)-H(3A)	108.8
C(4)-C(3)-H(3B)	108.8
C(2)-C(3)-H(3B)	108.8
H(3A)-C(3)-H(3B)	107.7
C(3)-C(4)-C(5)	112.7(4)
C(3)-C(4)-C(6)	113.2(4)
C(5)-C(4)-C(6)	106.9(3)
C(3)-C(4)-H(4)	107.9
C(5)-C(4)-H(4)	107.9
C(6)-C(4)-H(4)	107.9
C(4)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
C(4)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(7)-C(6)-C(4)	116.2(3)
C(7)-C(6)-H(6A)	108.2
C(4)-C(6)-H(6A)	108.2
C(7)-C(6)-H(6B)	108.2
C(4)-C(6)-H(6B)	108.2
H(6A)-C(6)-H(6B)	107.4
C(6)-C(7)-C(8)	111.3(4)
C(6)-C(7)-C(12)	108.4(4)
C(8)-C(7)-C(12)	108.4(4)
C(6)-C(7)-H(7)	109.6
C(8)-C(7)-H(7)	109.6
C(12)-C(7)-H(7)	109.6
C(9)-C(8)-C(7)	111.1(5)
C(9)-C(8)-H(8A)	109.4
C(7)-C(8)-H(8A)	109.4
C(9)-C(8)-H(8B)	109.4

C(7)-C(8)-H(8B)	109.4
H(8A)-C(8)-H(8B)	108.0
C(8)-C(9)-C(10)	111.4(5)
C(8)-C(9)-H(9A)	109.4
C(10)-C(9)-H(9A)	109.4
C(8)-C(9)-H(9B)	109.4
C(10)-C(9)-H(9B)	109.4
H(9A)-C(9)-H(9B)	108.0
C(11)-C(10)-C(9)	108.1(5)
C(11)-C(10)-H(10A)	110.1
C(9)-C(10)-H(10A)	110.1
C(11)-C(10)-H(10B)	110.1
C(9)-C(10)-H(10B)	110.1
H(10A)-C(10)-H(10B)	108.4
C(12)-C(11)-C(10)	110.5(6)
C(12)-C(11)-H(11A)	109.6
C(10)-C(11)-H(11A)	109.6
C(12)-C(11)-H(11B)	109.6
C(10)-C(11)-H(11B)	109.6
H(11A)-C(11)-H(11B)	108.1
C(11)-C(12)-C(7)	112.0(5)
C(11)-C(12)-H(12A)	109.2
C(7)-C(12)-H(12A)	109.2
C(11)-C(12)-H(12B)	109.2
C(7)-C(12)-H(12B)	109.2
H(12A)-C(12)-H(12B)	107.9
C(2)-C(3A)-C(4A)	115.1(7)
C(2)-C(3A)-H(3A1)	108.5
C(4A)-C(3A)-H(3A1)	108.5
C(2)-C(3A)-H(3A2)	108.5
C(4A)-C(3A)-H(3A2)	108.5
H(3A1)-C(3A)-H(3A2)	107.5
C(6A)-C(4A)-C(3A)	106.4(7)
C(6A)-C(4A)-C(5A)	116.0(8)
C(3A)-C(4A)-C(5A)	112.5(8)
C(6A)-C(4A)-H(4A)	107.2
C(3A)-C(4A)-H(4A)	107.2
C(5A)-C(4A)-H(4A)	107.2

C(4A)-C(5A)-H(5A1)	109.5
C(4A)-C(5A)-H(5A2)	109.5
H(5A1)-C(5A)-H(5A2)	109.5
C(4A)-C(5A)-H(5A3)	109.5
H(5A1)-C(5A)-H(5A3)	109.5
H(5A2)-C(5A)-H(5A3)	109.5
C(7A)-C(6A)-C(4A)	117.2(8)
C(7A)-C(6A)-H(6A1)	108.0
C(4A)-C(6A)-H(6A1)	108.0
C(7A)-C(6A)-H(6A2)	108.0
C(4A)-C(6A)-H(6A2)	108.0
H(6A1)-C(6A)-H(6A2)	107.3
C(6A)-C(7A)-C(12A)	108.9(9)
C(6A)-C(7A)-C(8A)	111.5(9)
C(12A)-C(7A)-C(8A)	109.8(9)
C(6A)-C(7A)-H(7A)	108.9
C(12A)-C(7A)-H(7A)	108.9
C(8A)-C(7A)-H(7A)	108.9
C(9A)-C(8A)-C(7A)	111.8(8)
C(9A)-C(8A)-H(8A1)	109.2
C(7A)-C(8A)-H(8A1)	109.2
C(9A)-C(8A)-H(8A2)	109.2
C(7A)-C(8A)-H(8A2)	109.2
H(8A1)-C(8A)-H(8A2)	107.9
C(8A)-C(9A)-C(10A)	115.1(9)
C(8A)-C(9A)-H(9A1)	108.5
C(10A)-C(9A)-H(9A1)	108.5
C(8A)-C(9A)-H(9A2)	108.5
C(10A)-C(9A)-H(9A2)	108.5
H(9A1)-C(9A)-H(9A2)	107.5
C(9A)-C(10A)-C(11A)	112.6(9)
C(9A)-C(10A)-H(10C)	109.1
C(11A)-C(10A)-H(10C)	109.1
C(9A)-C(10A)-H(10D)	109.1
C(11A)-C(10A)-H(10D)	109.1
H(10C)-C(10A)-H(10D)	107.8
C(12A)-C(11A)-C(10A)	111.7(9)
C(12A)-C(11A)-H(11C)	109.3

C(10A)-C(11A)-H(11C)	109.3
C(12A)-C(11A)-H(11D)	109.3
C(10A)-C(11A)-H(11D)	109.3
H(11C)-C(11A)-H(11D)	107.9
C(11A)-C(12A)-C(7A)	111.7(10)
C(11A)-C(12A)-H(12C)	109.3
C(7A)-C(12A)-H(12C)	109.3
C(11A)-C(12A)-H(12D)	109.3
C(7A)-C(12A)-H(12D)	109.3
H(12C)-C(12A)-H(12D)	107.9
C(1)-N(1)-C(21)	121.02(17)
C(1)-N(1)-C(31)	123.14(19)
C(21)-N(1)-C(31)	115.82(16)
C(22)-C(21)-C(26)	119.7(2)
C(22)-C(21)-N(1)	119.7(2)
C(26)-C(21)-N(1)	120.47(19)
C(21)-C(22)-C(23)	119.5(2)
C(21)-C(22)-H(22)	120.3
C(23)-C(22)-H(22)	120.3
C(24)-C(23)-C(22)	121.2(2)
C(24)-C(23)-H(23)	119.4
C(22)-C(23)-H(23)	119.4
C(23)-C(24)-C(25)	118.7(3)
C(23)-C(24)-H(24)	120.7
C(25)-C(24)-H(24)	120.7
C(26)-C(25)-C(24)	120.4(2)
C(26)-C(25)-H(25)	119.8
C(24)-C(25)-H(25)	119.8
C(25)-C(26)-C(21)	120.5(2)
C(25)-C(26)-H(26)	119.7
C(21)-C(26)-H(26)	119.7
C(36)-C(31)-C(32)	120.2(2)
C(36)-C(31)-N(1)	119.3(2)
C(32)-C(31)-N(1)	120.4(2)
C(31)-C(32)-C(33)	118.9(3)
C(31)-C(32)-H(32)	120.5
C(33)-C(32)-H(32)	120.5
C(34)-C(33)-C(32)	121.0(3)

C(34)-C(33)-H(33)	119.5
C(32)-C(33)-H(33)	119.5
C(33)-C(34)-C(35)	120.2(2)
C(33)-C(34)-H(34)	119.9
C(35)-C(34)-H(34)	119.9
C(34)-C(35)-C(36)	119.8(3)
C(34)-C(35)-H(35)	120.1
C(36)-C(35)-H(35)	120.1
C(31)-C(36)-C(35)	119.9(2)
C(31)-C(36)-H(36)	120.1
C(35)-C(36)-H(36)	120.1

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\approx^2$x 10^3) for x10032_t4. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + \ldots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	29(1)	52(1)	64(1)	-8(1)	13(1)	-7(1)
C(1)	34(1)	36(1)	61(1)	-10(1)	16(1)	-4(1)
C(2)	38(1)	43(1)	75(2)	1(1)	18(1)	4(1)
C(3)	40(2)	44(2)	47(2)	13(2)	12(2)	-1(2)
C(4)	40(2)	53(2)	34(2)	6(2)	3(2)	0(2)
C(5)	59(2)	62(3)	62(2)	2(2)	4(2)	-10(2)
C(6)	61(2)	83(3)	35(2)	-2(2)	8(1)	7(2)
C(7)	58(2)	86(3)	27(2)	3(2)	14(2)	8(2)
C(8)	64(2)	110(4)	52(2)	8(3)	7(2)	23(3)
C(9)	55(2)	204(7)	75(3)	19(4)	8(2)	18(4)
C(10)	57(2)	170(7)	112(4)	11(4)	37(3)	26(4)
C(11)	72(3)	181(6)	68(3)	-1(4)	43(3)	18(3)
C(12)	69(2)	158(5)	48(2)	-20(3)	22(2)	9(3)
C(3A)	48(4)	39(5)	68(5)	15(3)	11(3)	-2(3)
C(4A)	47(4)	57(5)	59(4)	4(3)	10(3)	18(3)
C(5A)	63(6)	63(6)	85(9)	-11(6)	12(6)	16(4)
C(6A)	74(4)	59(5)	47(4)	13(3)	8(3)	11(3)
C(7A)	62(5)	82(5)	44(5)	12(4)	12(3)	15(4)
C(8A)	78(5)	63(5)	36(4)	5(3)	11(4)	6(4)
C(9A)	61(5)	76(6)	88(6)	19(5)	21(4)	17(4)
C(10A)	72(5)	107(7)	64(5)	17(5)	22(4)	12(5)
C(11A)	74(6)	123(7)	61(6)	26(5)	24(4)	12(5)
C(12A)	85(5)	87(6)	55(4)	25(4)	16(4)	2(4)
N(1)	28(1)	38(1)	57(1)	-5(1)	11(1)	0(1)
C(21)	24(1)	39(1)	54(1)	-10(1)	4(1)	-4(1)
C(22)	53(1)	54(2)	53(1)	-15(1)	-1(1)	17(1)
C(23)	69(2)	77(2)	44(1)	-13(1)	-5(1)	23(2)
C(24)	46(1)	58(2)	56(1)	-5(1)	-10(1)	8(1)
C(25)	29(1)	45(2)	71(2)	-7(1)	4(1)	0(1)
C(26)	27(1)	40(1)	61(1)	-9(1)	11(1)	-3(1)
C(31)	31(1)	36(1)	52(1)	6(1)	14(1)	4(1)
C(32)	45(1)	42(2)	82(2)	0(1)	28(1)	6(1)
C(33)	55(2)	50(2)	99(2)	22(2)	45(2)	21(1)

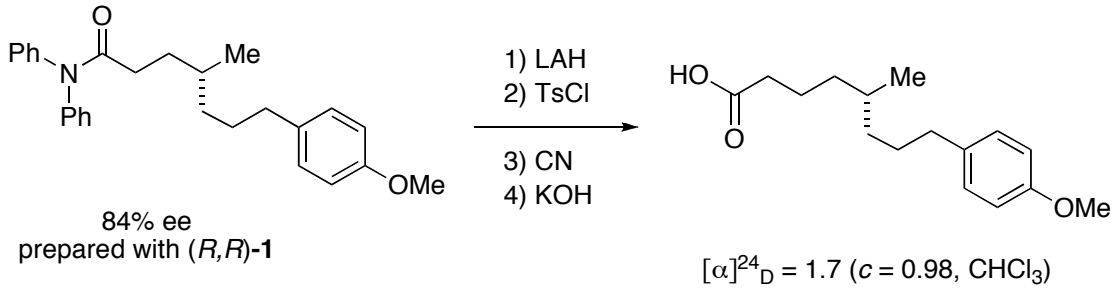
C(34)	33(1)	79(2)	78(2)	41(2)	27(1)	17(1)
C(35)	32(1)	74(2)	54(1)	23(1)	5(1)	-6(1)
C(36)	34(1)	43(1)	46(1)	9(1)	7(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3}) for x10032_t4.

	x	y	z	U(eq)
H(2A)	6170	5406	2588	61
H(2B)	5607	3260	2374	61
H(2C)	5753	2760	2217	61
H(2D)	5912	5173	2620	61
H(3A)	6353	4277	1480	52
H(3B)	7129	3782	1941	52
H(4)	6832	-220	1981	51
H(5A)	5783	-1829	1298	92
H(5B)	5494	772	1093	92
H(5C)	5481	-108	1835	92
H(6A)	6992	-1331	899	72
H(6B)	6727	1214	630	72
H(7)	7857	2913	1136	67
H(8A)	8124	377	2054	90
H(8B)	8242	-1820	1582	90
H(9A)	9428	-317	2016	133
H(9B)	9203	2229	1715	133
H(10A)	10000	214	1018	133
H(10B)	9396	-1918	928	133
H(11A)	9066	2855	506	125
H(11B)	9191	714	17	125
H(12A)	8104	-1164	319	109
H(12B)	7890	1419	42	109
H(3A1)	7161	4939	2396	62
H(3A2)	6550	5442	1777	62
H(4A)	7304	1062	2085	65
H(5A1)	6630	-632	1138	105
H(5A2)	6131	1721	1016	105
H(5A3)	6046	126	1649	105
H(6A1)	7420	4370	1067	72
H(6A2)	8050	3861	1669	72
H(7A)	7658	767	592	74

H(8A1)	8749	-57	1732	71
H(8A2)	7995	-1598	1538	71
H(9A1)	8478	-3126	652	89
H(9A2)	9113	-3435	1265	89
H(10C)	9627	-2173	276	96
H(10D)	9868	-505	894	96
H(11C)	9514	1992	17	101
H(11D)	8740	553	-183	101
H(12C)	8434	4024	338	90
H(12D)	9040	3442	958	90
H(22)	5674	1440	4675	64
H(23)	6038	-1186	5528	77
H(24)	6812	-4429	5360	65
H(25)	7234	-4997	4322	58
H(26)	6853	-2438	3469	51
H(32)	5206	5045	3777	66
H(33)	3886	5706	3619	79
H(34)	3069	2847	3154	74
H(35)	3550	-716	2819	64
H(36)	4876	-1385	2943	49

Assignment of absolute configuration of the δ-alkylated product. γ-Alkylated product (*R*)-7-(4-methoxyphenyl)-4-methyl-*N*,*N*-diphenylheptanamide, synthesized using *(R,R)*-**1,** was homologated. The specific rotation of the final product, 8-(4-methoxyphenyl)-5-methyloctanoic acid, was determined.



(*R*)-8-(4-Methoxyphenyl)-5-methyloctanoic acid.

^{1}H NMR ($CDCl_3$) δ 7.07 (d, 2H, *J* = 8.4 Hz), 6.81 (d, 2H, *J* = 8.5 Hz), 3.77 (s, 3H), 2.53–2.48 (m, 2H), 2.32–2.29 (m, 2H), 1.62–1.57 (m, 5H), 1.55–1.41 (m, 3H), 1.31–1.22 (m, 2H), 0.86 (d, 3H, *J* = 6.5 Hz).

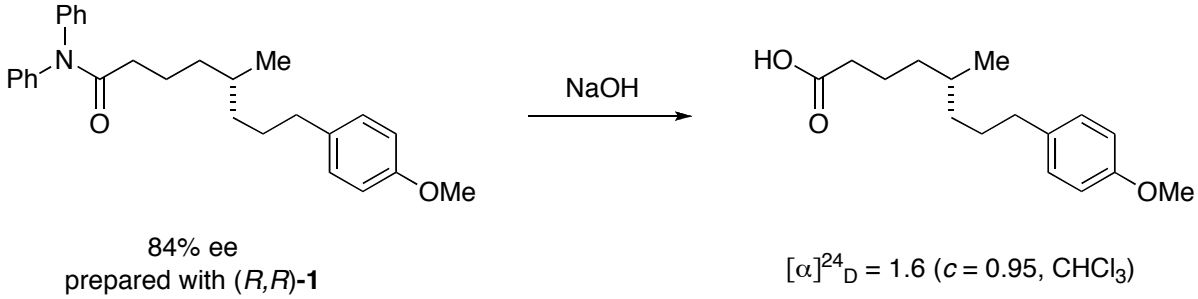
^{13}C NMR ($CDCl_3$) δ 180.3, 157.6, 134.9, 129.2, 113.7, 55.3, 36.4, 36.3, 35.3, 34.4, 32.5, 29.2, 22.2, 19.5.

FT-IR (film) 2931 (broad), 1708, 1612, 1512, 1463, 1300, 1245, 1177, 1038, 829.

MS (EI) *m*/*z* ($M+H^+$) calcd for $C_{16}H_{25}O_3$: 265, found: 265.

$[\alpha]^{24}_D$ = 1.7 (*c* = 0.98, $CHCl_3$).

This specific rotation was compared to the product that was generated through the δ-alkylation illustrated in eq 7 (with *(R,R)*-**1**), followed by hydrolysis of the amide. The specific rotations had the same sign.



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Acq. Operator	: jtm	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence !	Actual Inj Volume	: 1 µl	
Acq. Method	: C:\HPCHEM\1\METHODS\ADH-0560.M		
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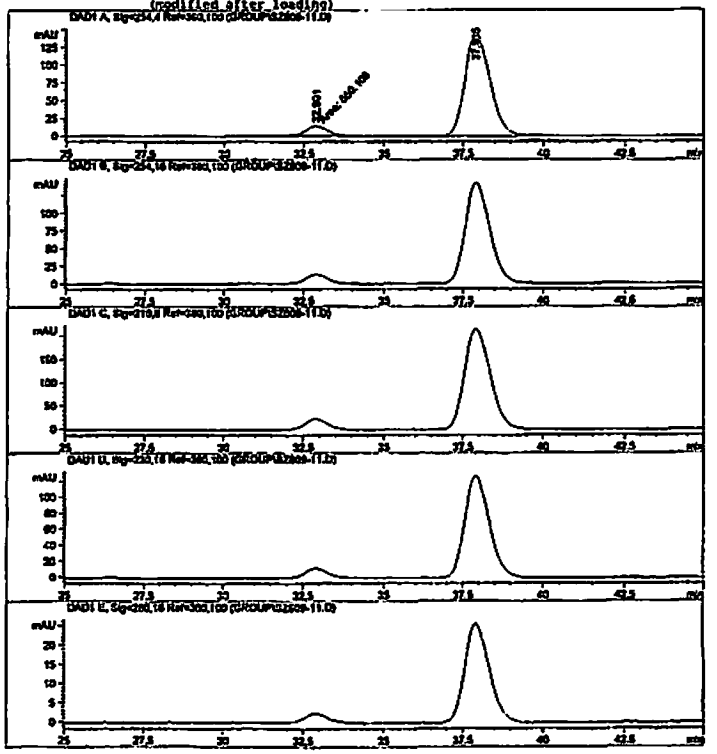

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Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.901	MM	0.7750	650.10822	13.98044	7.6338
2	37.915	VV	0.6609	7866.04443	145.00969	92.3662
Totals :				8516.15265	158.99013	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

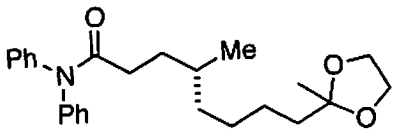
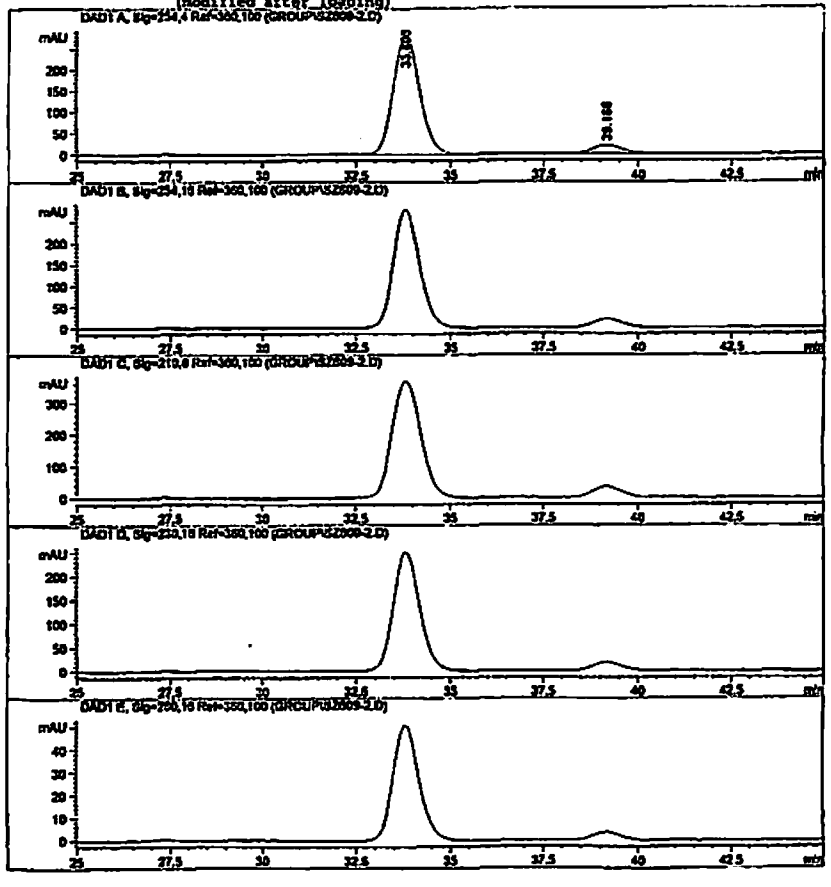


Table 1, entry 1

with (*R*,*R*)-**1**

Data File C:\HPCHEM\1\DATA\GROUP\SZ609-2.D

Injection Date	: 9/7/2010 7:57:26 PM	Seq. Line	: 8
Sample Name	:	Location	: Vial 22
Acq. Operator	: jtm	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ADH-0560.M		
Last changed	: 5/8/2009 8:38:42 AM by NN		
Analysis Method	: C:\HPCHEM\1\METHODS\OJB-0060.M		
Last changed	: 8/21/2011 6:34:45 PM by SN		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ609-2.D

Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	33.808	VB	0.6789	1.37349e4	282.65738	92.0135
2	39.186	VV	0.6556	1192.14404	21.54304	7.9865
Totals :				1.49271e4	304.20041	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

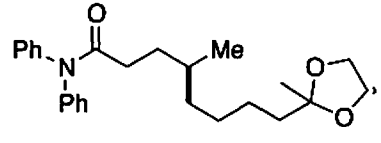
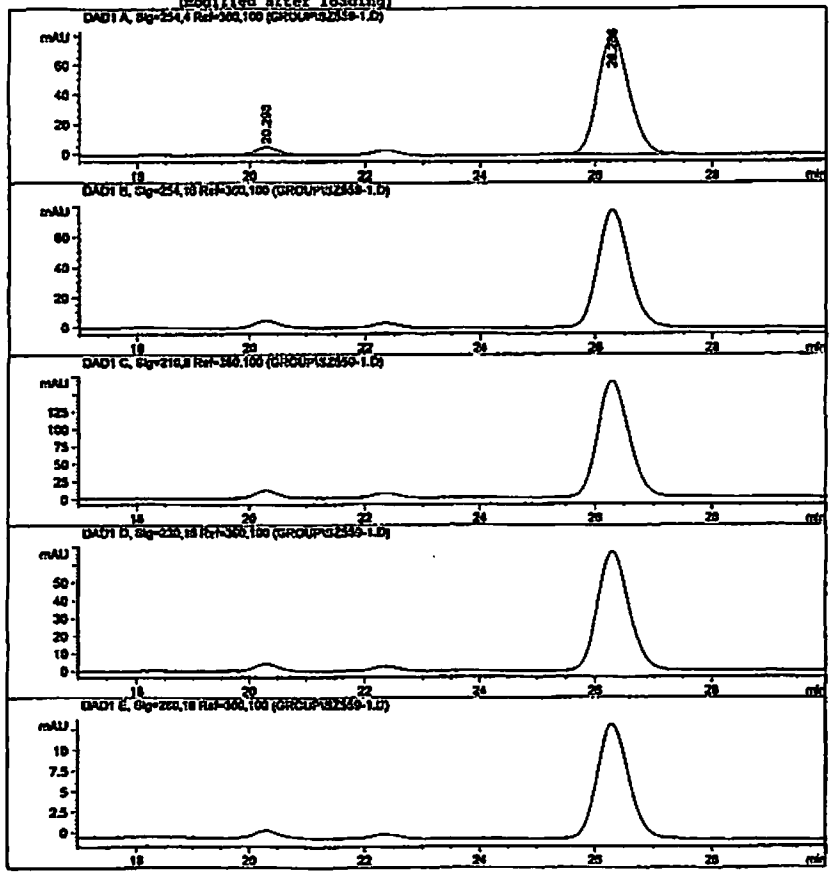


Table 1, entry 1

with (*S*,*S*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ559-1.D

Injection Date : 7/26/2010 1:39:57 PM Seq. Line : 2
Sample Name : Location : Vial 1
Acq. Operator : jtm Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\ADH-0240.M
Last changed : 8/11/2009 4:43:29 PM by SZ
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 6:37:45 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ559-1.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.298	BB	0.3400	142.86464	5.08223	4.6211
2	26.286	BB	0.5600	2948.72095	80.37132	95.3789
Totals :				3091.58559	85.45356	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

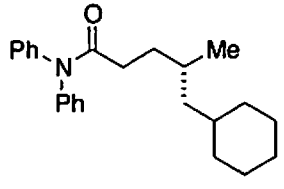
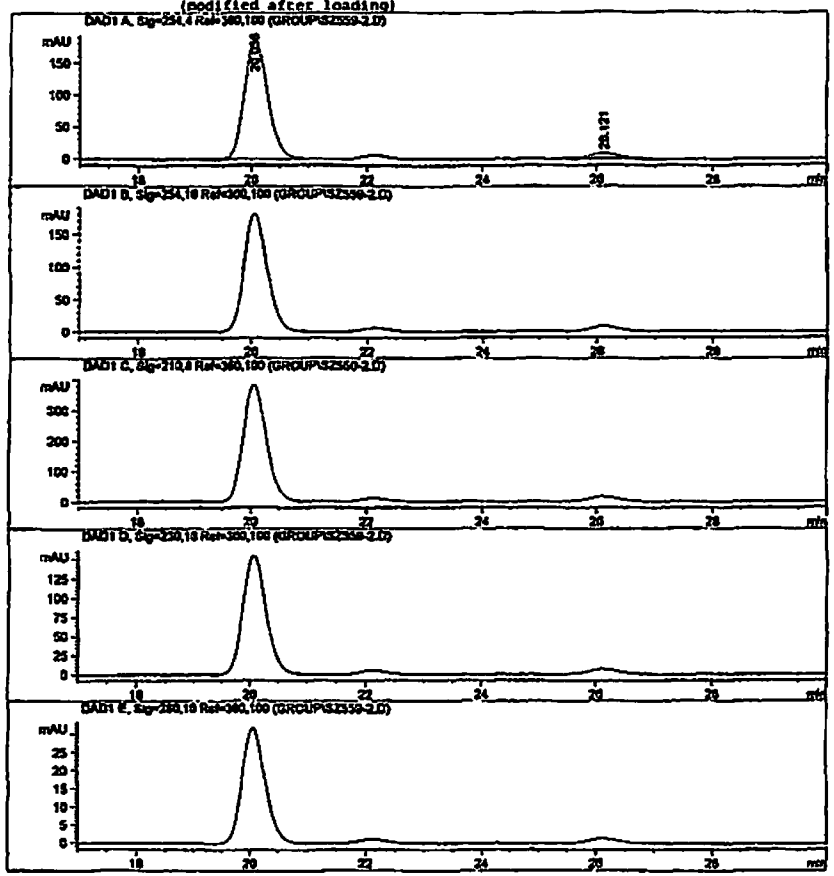


Table 1, entry 2

with (*R,R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ559-2.D

Injection Date : 7/26/2010 2:21:14 PM Seq. Line : 3
Sample Name : Location : Vial 2
Acq. Operator : jtm Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence : Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\ADH-0240.M
Last changed : 8/11/2009 4:43:29 PM by SZ
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 6:36:52 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ559-2.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.056	BB	0.4384	5290.59473	185.70091	94.7646
2	26.121	BP	0.4531	292.28845	7.91065	5.2354
Totals :				5582.88318	193.61157	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

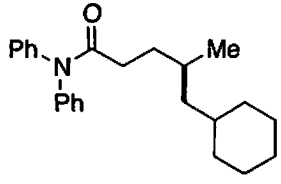


Table 1, entry 2

with (*S,S*)-**1**

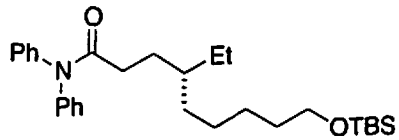
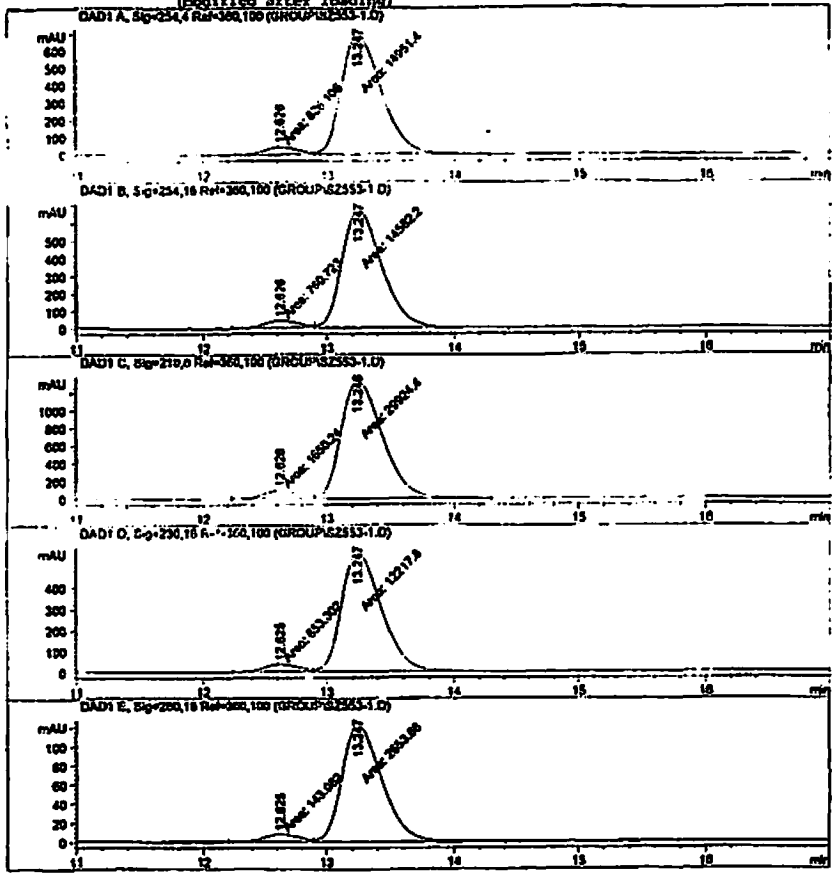


Table 1, entry 3

with (*R,R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ553-1.D

Injection Date	: 7/6/2010 9:39:03 PM
Sample Name	:
Acq. Operator	: jtm
Acq. Instrument	: Instrument 1
Different Inj Volume from Sequence !	Actual
Acq. Method	: C:\HPCHEM\1\METHODS\ADH-0130.M
Last changed	: 5/8/2009 8:39:29 AM by MM
Analysis Method	: C:\HPCHEM\1\METHODS\AD-00530.M
Last changed	: 7/7/2010 5:40:16 PM by jtm
	(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ553-1.D

Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.626	MM	0.3194	826.10608	43.11390	5.2360
2	13.247	MM	0.3614	1.49513e4	689.46777	94.7640
Totals :				1.57779e4	732.58168	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.626	MM	0.3117	760.72339	40.67119	4.9581
2	13.247	MM	0.3619	1.45822e4	671.61914	95.0419
Totals :				1.53429e4	712.29033	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.626	MM	0.3151	1650.24500	87.28162	5.2265
2	13.246	MM	0.3765	2.99244e4	1324.67798	94.7735
Totals :				3.15746e4	1411.95959	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.625	MM	0.3140	653.30212	34.58416	5.0757
2	13.247	MM	0.3622	1.22178e4	562.17419	94.9243
Totals :				1.28711e4	596.75835	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.625	MM	0.3173	143.08186	7.51659	5.1160
2	13.247	MM	0.3636	2653.65576	121.63618	94.8840
Totals :				2796.73763	129.15277	

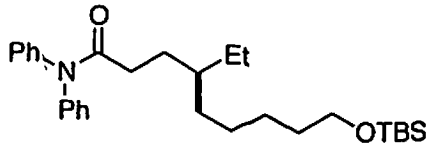


Table 1, entry 3

with (*S,S*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ553-2.D

```
Injection Date  : 7/6/2010 10:10:19 PM
Sample Name     :
Acq. Operator   : jtm
Acq. Instrument : Instrument 1
Different Inj Volume from Sequence :     Actual
Acq. Method     : C:\HPCHEM\1\METHODS\ADH-0130.M
Last changed    : 5/8/2009 6:39:29 AM by HM
Analysis Method : C:\HPCHEM\1\METHODS\AD-00530.M
Last changed    : 7/7/2010 5:14:46 PM by jtm
                  (modified after loading)
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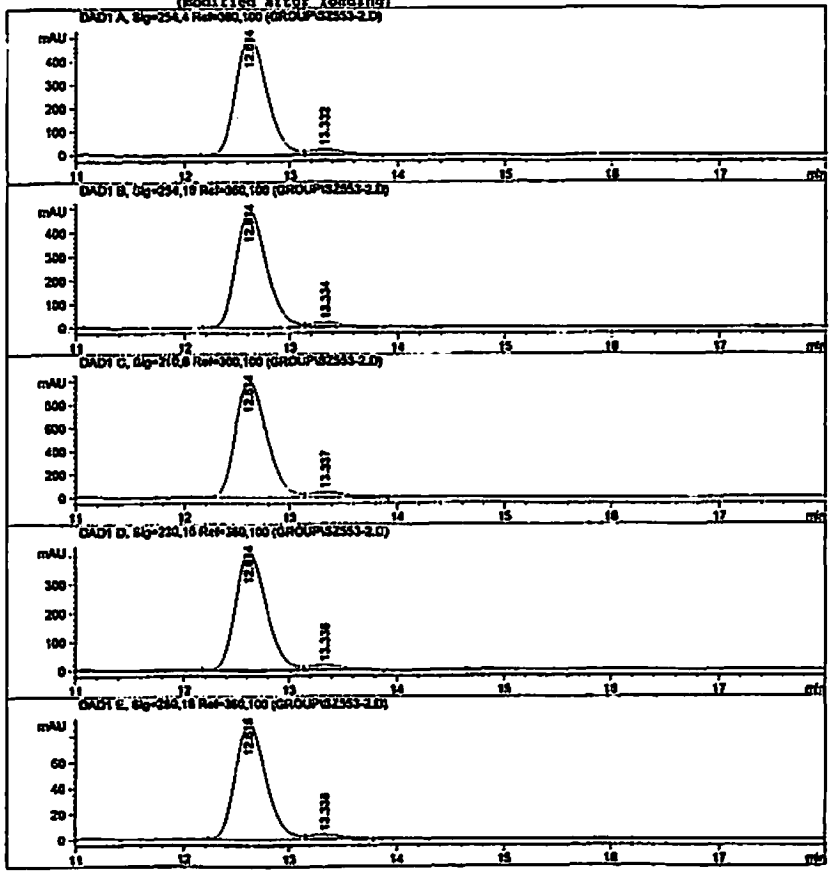


Data File C:\HPCHEM\1\DATA\GROUP\SZ553-2.D

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.614	BV	0.3161	1.04088e4	509.94598	95.9835
2	13.332	VB	0.3103	435.56693	20.63039	4.0165
Totals :				1.08444e4	530.57637	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.614	BV	0.3161	1.01299e4	496.34854	95.9857
2	13.334	VB	0.3141	423.65262	20.08257	4.0143
Totals :				1.05535e4	516.43111	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.614	BV	0.3222	2.08797e4	1005.61359	95.8138
2	13.337	VB	0.3149	912.25574	42.76027	4.1862
Totals :				2.17919e4	1048.37385	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.614	BV	0.3159	8451.90039	414.44778	95.9681
2	13.336	VB	0.3088	355.08801	16.78893	4.0319
Totals :				8806.98840	431.23672	

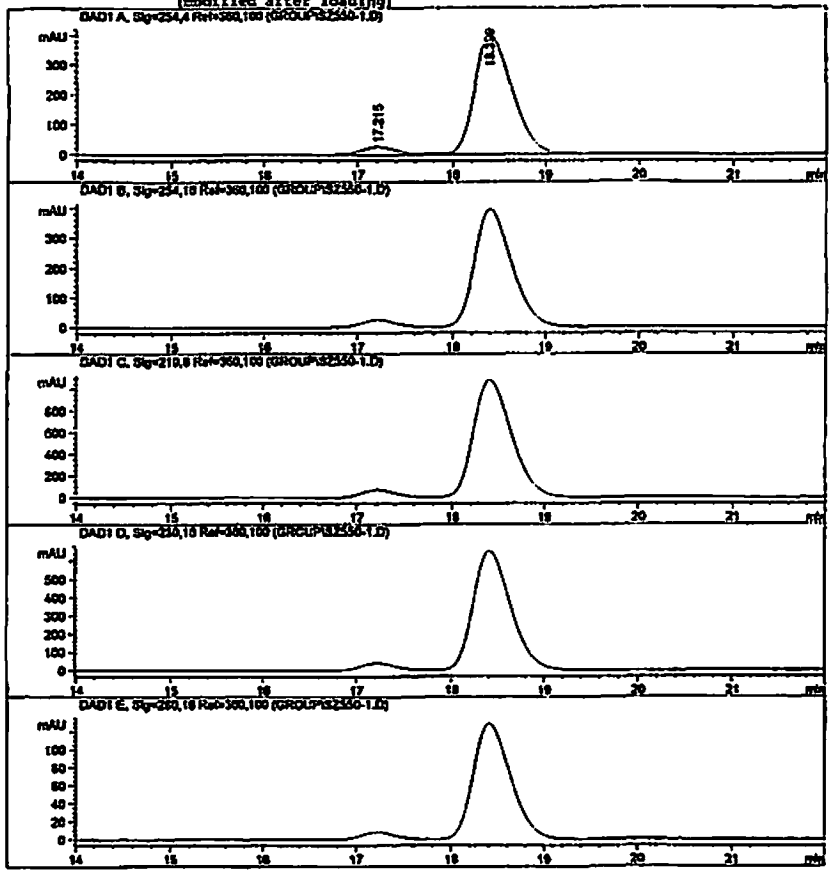
Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.615	BB	0.3145	1831.75488	89.62212	96.1938
2	13.338	BB	0.2974	72.47832	3.62323	3.8062
Totals :				1904.23321	93.24535	

Data File C:\HPCHEM\1\DATA\GROUP\SZ550-1.D

Injection Date : 7/6/2010 7:54:51 PM Seq. Line : 3
Sample Name : Location : Vial 45
Acq. Operator : jtm Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\ADH-1030.M
Last changed : 4/21/2010 11:34:12 AM by jtm
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 6:47:13 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ550-1.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.215	BV	0.3652	639.39001	25.44100	5.3728
2	18.399	VB	0.4366	1.12611e4	399.87430	94.6272
Totals :				1.19005e4	425.31530	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

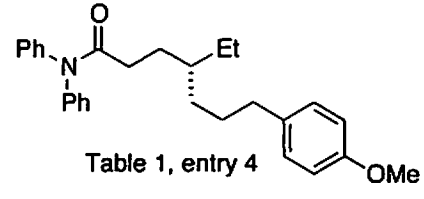
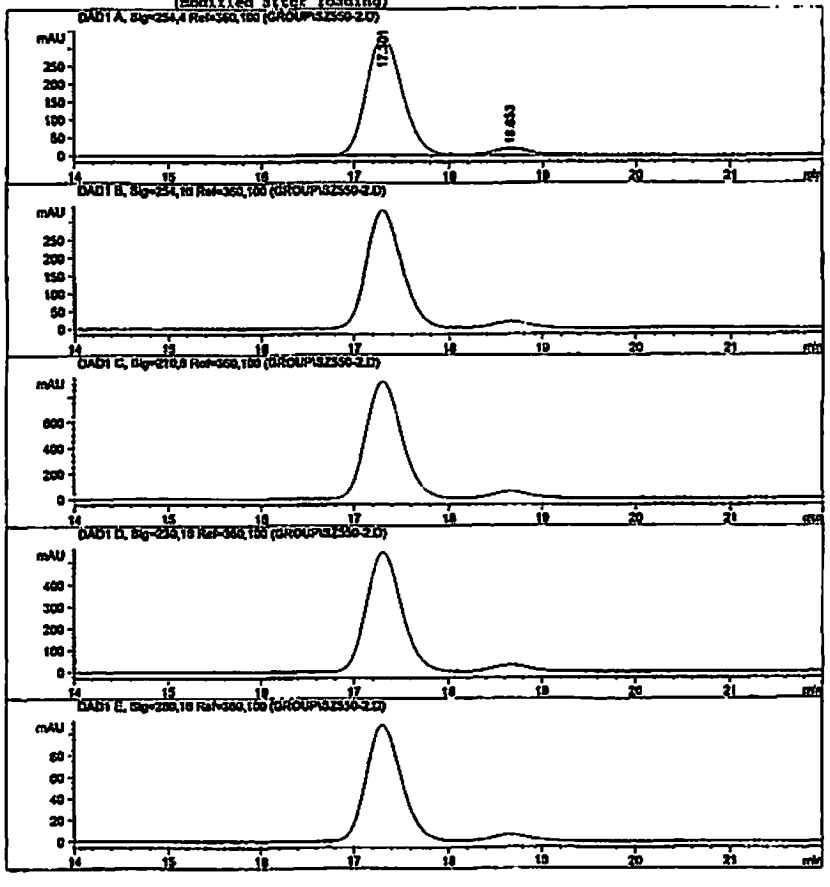


Table 1, entry 4

with (*R,R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ550-2.D

Injection Date	: 7/6/2010 8:26:10 PM	Seq. Line	: 4
Sample Name	:	Location	: Vial 46
Acq. Operator	: jtm	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence !	Actual Inj Volume	: 1 µl	
Acq. Method	: C:\HPCHEM\1\METHODS\ADH-1030.M		
Last changed	: 4/21/2010 11:34:12 AM by jtm		
Analysis Method	: C:\HPCHEM\1\METHODS\OJH-0060.M		
Last changed	: 8/21/2011 6:46:16 PM by SN		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ550-2.D

Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.301	BB	0.3987	8683.53613	334.72333	94.0779
2	18.653	BP	0.4140	546.61829	19.09062	5.9221
Totals :				9230.15442	353.81395	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

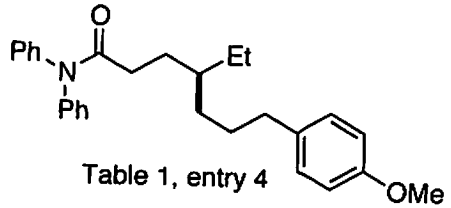
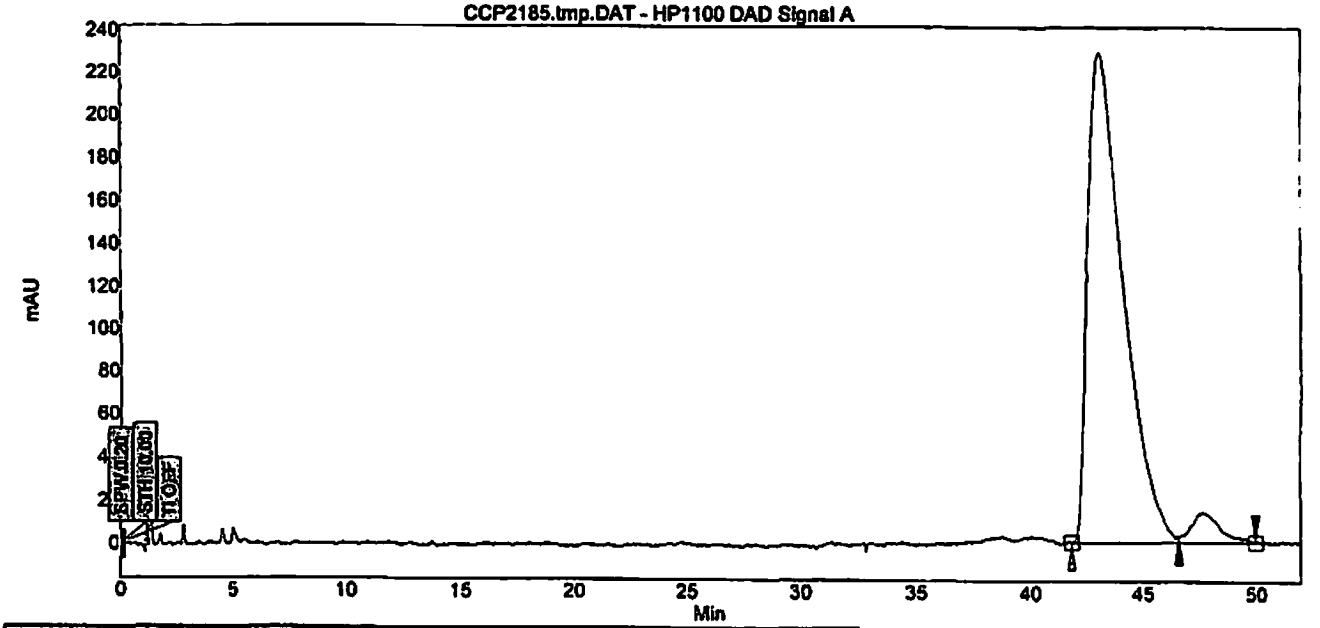


Table 1, entry 4

with (*S,S*)-1

sz7871ad101 Vial 20 Method: AD-H 10% MeOH



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	41.84	43.03	46.58	0.00	95.25	228.3	416.8	95.246
2	UNKNOWN	46.58	47.61	49.99	0.00	4.75	14.3	20.8	4.754
Total						100.00	242.5	437.6	100.000

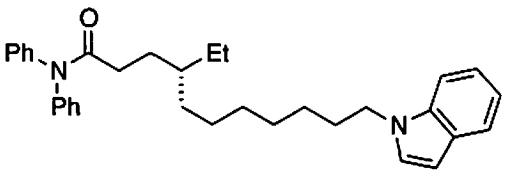
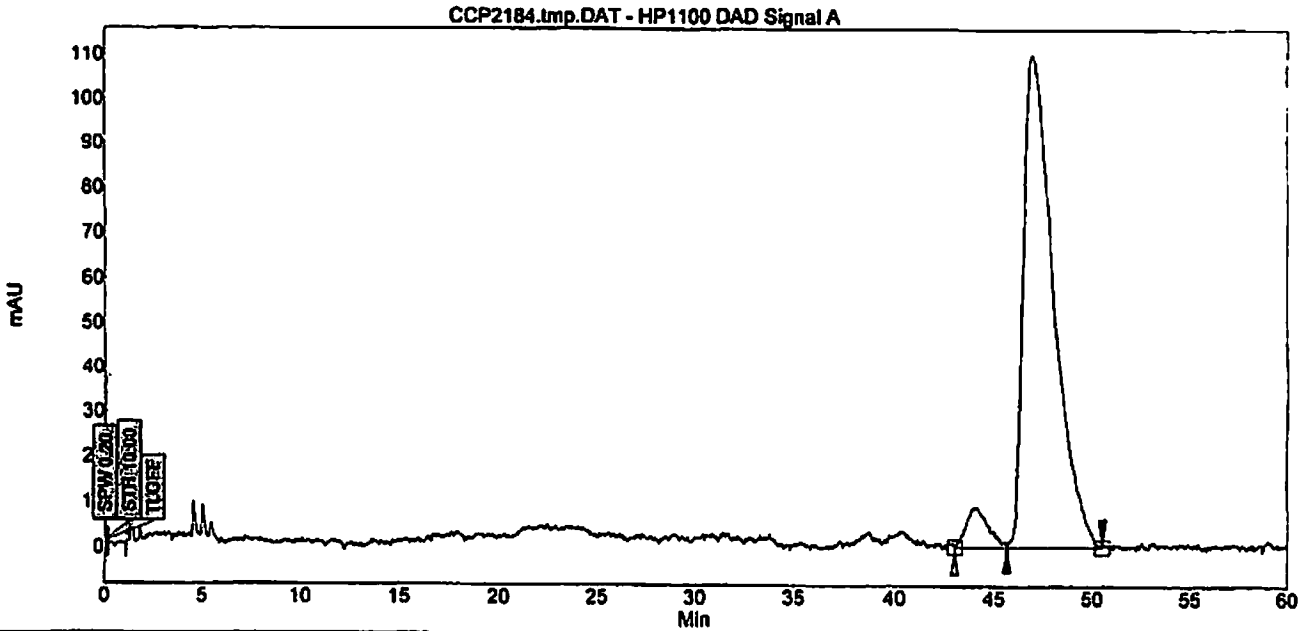


Table 1, entry 5

with (*R*,*R*)-**1**

sz7872ad100 Vial 21 Method: AD-H 10% MeOH



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	43.02	44.15	45.65	0.00	5.45	8.9	11.3	5.449
2	UNKNOWN	45.65	46.98	50.53	0.00	94.55	109.7	195.3	94.551
Total						100.00	118.6	206.6	100.000

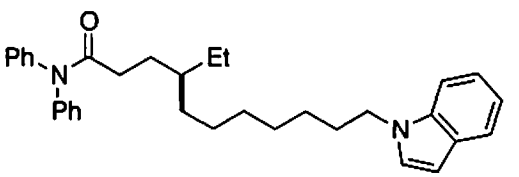
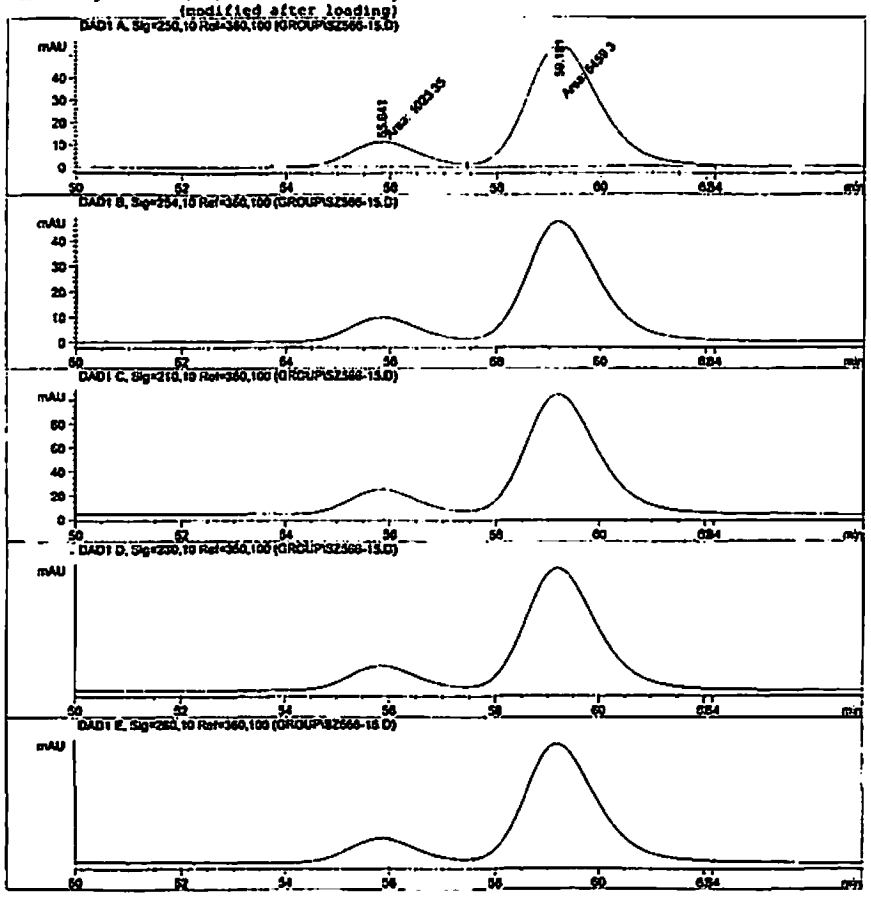


Table 1, entry 5

with (*S*,*S*)-**1**

Data File C:\HPCHEM\1\DATA\GROUP\SZ566-15.D

Injection Date	: 7/26/2010 8:26:12 PM	Seq. Line	: 2
Sample Name	:	Location	: Vial 91
Acq. Operator	: JTM	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 15 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\AD-05-80.M		
Last changed	: 12/23/2004 8:14:11 AM by GROUP		
Analysis Method	: C:\HPCHEM\1\METHODS\OJ-00590.M		
Last changed	: 8/15/2011 11:43:55 AM by NB		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ566-15.D

Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	55.841	MF	1.5247	1023.34711	11.18632	15.7859
2	59.181	FM	1.6700	5459.30371	54.48517	84.2141
Totals :				6482.65082	65.67149	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

*** End of Report ***

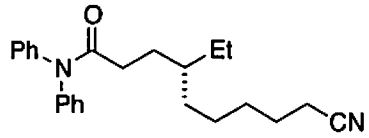
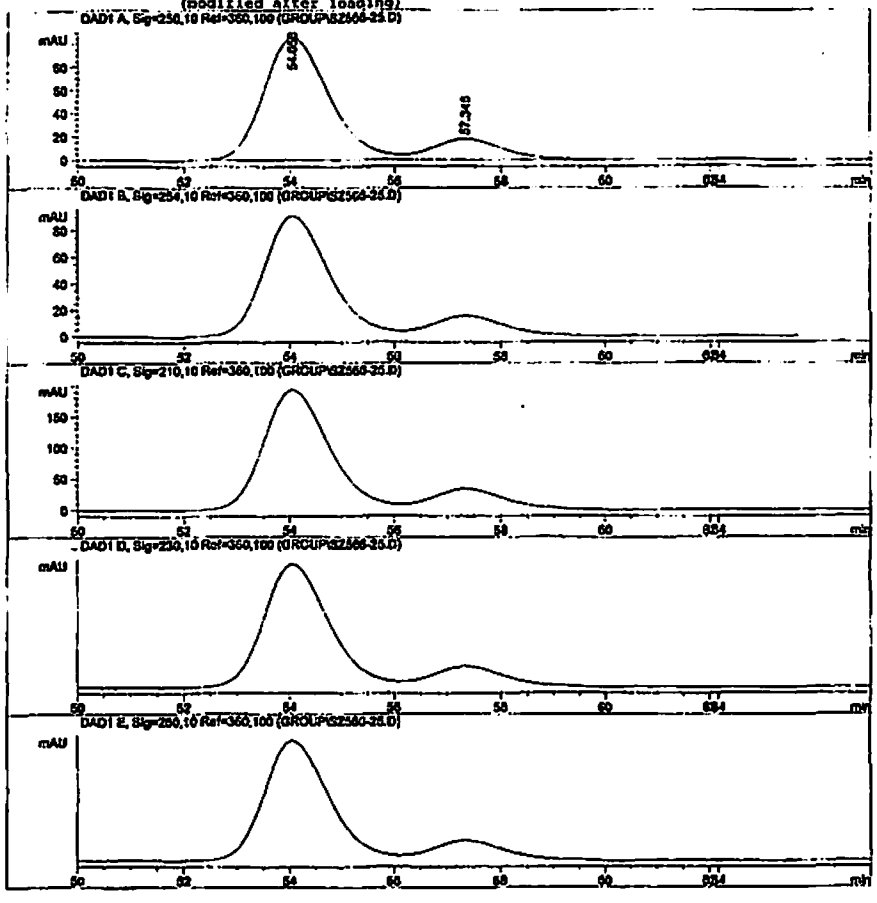


Table 1, entry 6

with (*R*,*R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ566-25.D

Injection Date	: 7/26/2010 9:47:24 PM	Seq. Line	: 3
Sample Name	:	Location	: Vial 92
Acq. Operator	: JTM	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 15 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\AD-05-80.M		
Last changed	: 12/23/2004 8:14:11 AM by GROUP		
Analysis Method	: C:\HPCHEM\1\METHODS\OJ-00590.M		
Last changed	: 8/21/2011 6:47:42 PM by MB		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ566-25.D

Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	
Use Multiplier & Dilution Factor with ISTDs			

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	54.050	BV	1.2603	9117.47656	105.08060	84.8560
2	57.346	VB	1.1079	1627.16858	17.48224	15.1440
Totals :				1.07446e4	122.56284	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

*** End of Report ***

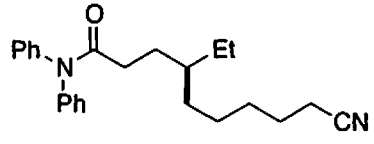
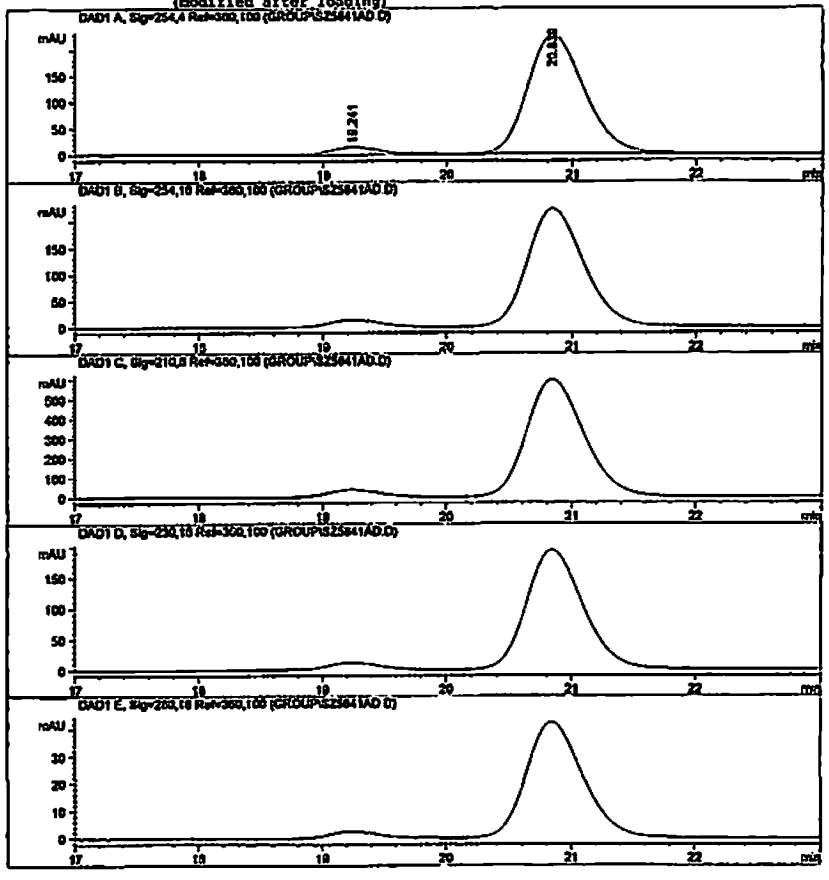


Table 1, entry 6

with (*S*,*S*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ5841AD.D

Injection Date : 8/8/2010 7:34:41 PM Seq. Line : 2
Sample Name : Location : Vial 1
Acq. Operator : jtm Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\ADH-0360.M
Last changed : 7/20/2009 9:10:15 AM by SZ
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 6:56:06 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ5841AD.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.241	BB	0.4219	412.67188	14.50753	5.4395
2	20.839	BB	0.4834	7173.89209	226.54938	94.5605
Totals :				7586.56396	241.05691	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

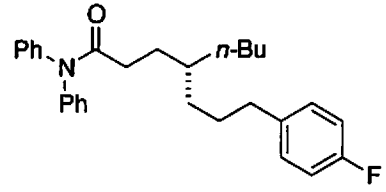
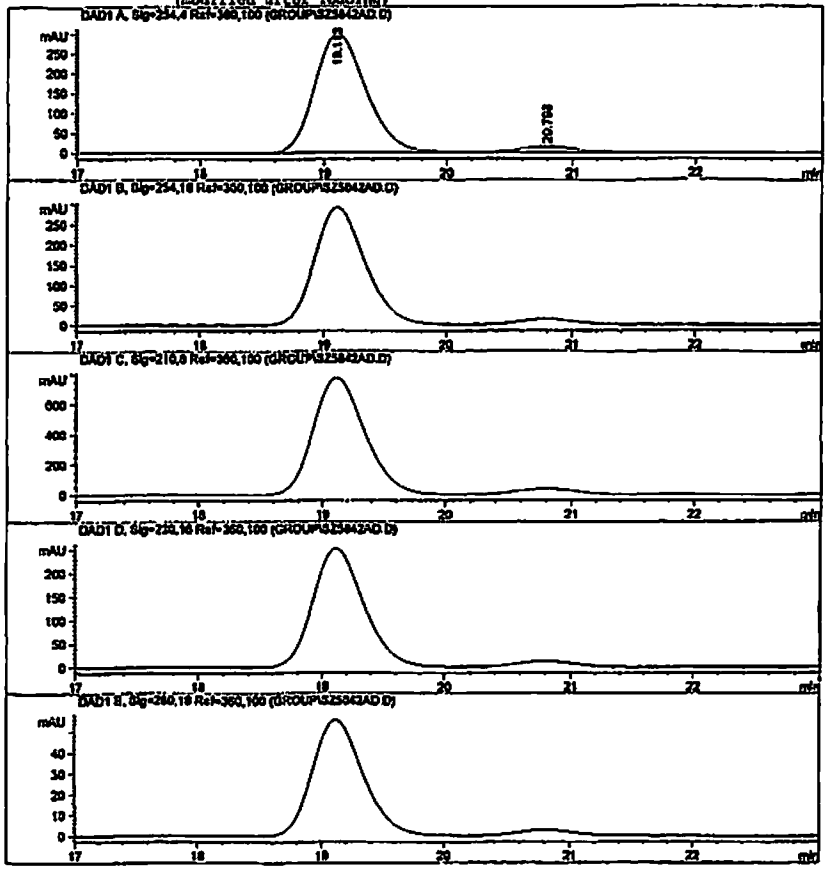


Table 1, entry 7

with (*R*,*R*)-**1**

Data File C:\HPCHEM\1\DATA\GROUP\SZ5842AD.D

Injection Date : 8/8/2010 8:35:53 PM Seq. Line : 3
Sample Name : Location : Vial 2
Acq. Operator : jtm Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\ADB-0360.M
Last changed : 7/20/2009 9:10:15 AM by SZ
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 6:56:32 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ5842AD.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.113	BB	0.4508	8640.99219	297.52673	95.2334
2	20.798	PB	0.4458	432.49255	14.10294	4.7666
Totals :				9073.48474	311.62968	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

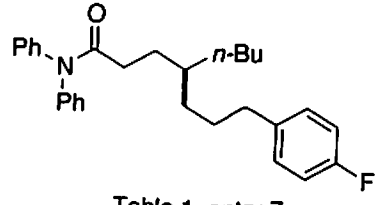
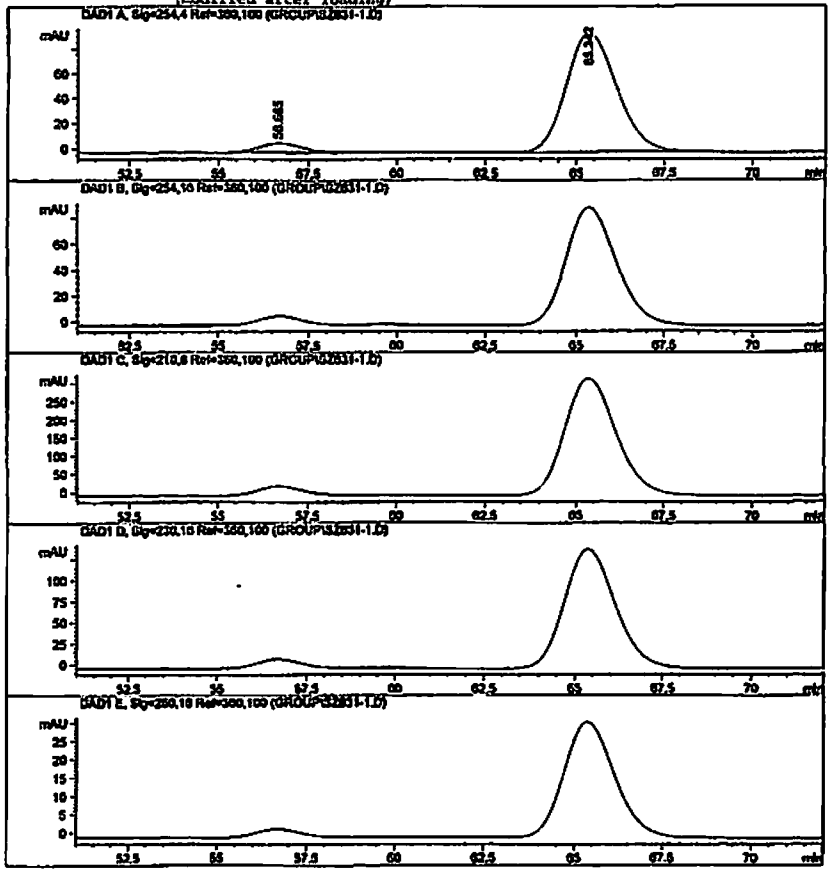


Table 1, entry 7

with (*S*,*S*)-**1**

Data File C:\HPCHEM\1\DATA\GROUP\SZ631-1.D

Injection Date	: 10/1/2010 12:30:55 AM	Seq. Line	: 3
Sample Name	:	Location	: Vial 3
Acq. Operator	: JTM	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 2 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ADR-0380.M		
Last changed	: 9/30/2010 10:46:00 PM by JTM		
Analysis Method	: C:\HPCHEM\1\METHODS\OJK-0060.M		
Last changed	: 8/21/2011 6:57:12 PM by SZ		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ631-1.D

Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	56.665	BP	0.9421	594.51666	7.42511	5.9815
2	65.342	BB	1.2129	9344.80273	94.88984	94.0185
Totals :				9939.31940	102.31495	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

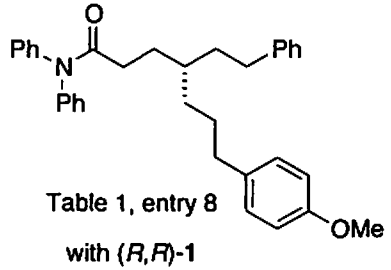
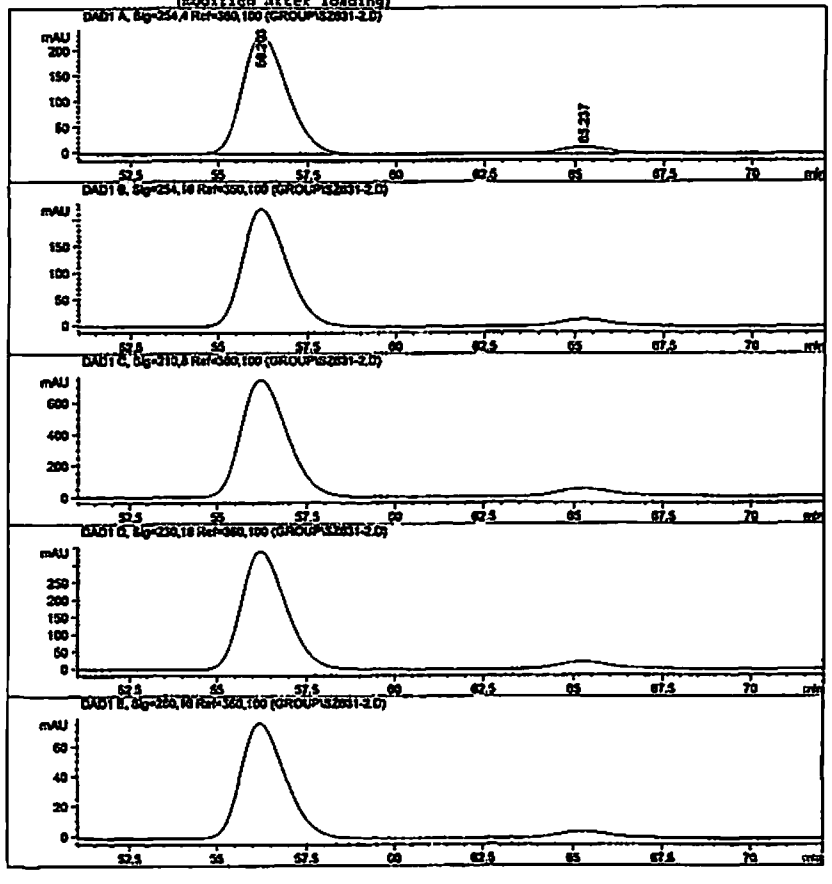


Table 1, entry 8

with (*R*,*R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ631-2.D

Injection Date : 9/30/2010 11:09:37 PM Seq. Line : 2
Sample Name : Location : Vial 2
Acq. Operator : JTM Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\ADH-0380.M
Last changed : 9/30/2010 10:46:00 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 6:58:09 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ631-2.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	56.203	BB	1.2502	1.99559e4	232.30559	94.0507
2	65.237	BB	1.1199	1262.33008	13.38977	5.9493
Totals :				2.12189e4	245.69536	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

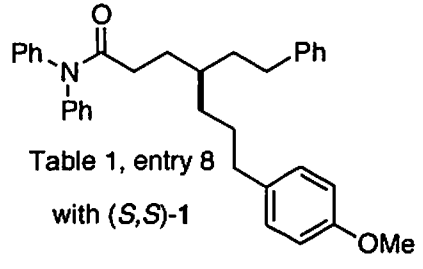
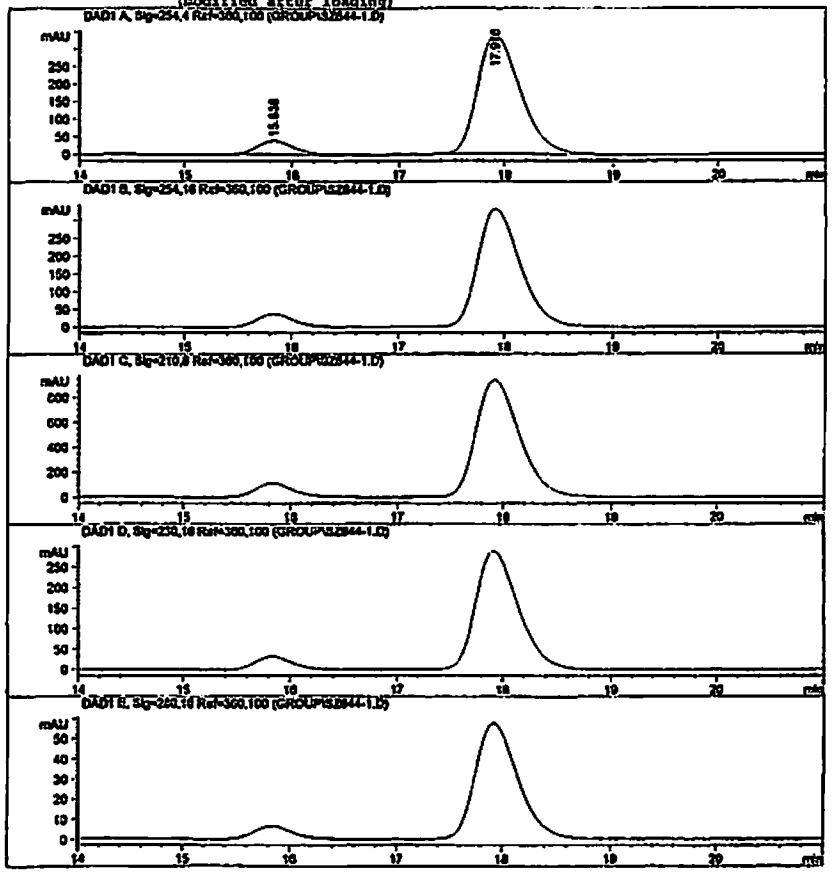


Table 1, entry 8

with (*S*,*S*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ644-1.D

Injection Date	: 12/9/2010 7:19:52 PM	Seq. Line	: 7
Sample Name	:	Location	: Vial 61
Acq. Operator	: JTH	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence !	Actual Inj Volume	: 1 µl	
Acq. Method	: C:\HPCHEM\1\METHODS\ADH-0340.M		
Last changed	: 6/16/2009 1:35:44 PM by SZ		
Analysis Method	: C:\HPCHEM\1\METHODS\OJH-0060.M		
Last changed	: 8/21/2011 6:58:40 PM by SW		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ644-1.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.836	BB	0.3652	914.17950	38.48144	8.8374
2	17.916	BB	0.4318	9430.27246	339.75735	91.1626
Totals :				1.03445e4	378.23879	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

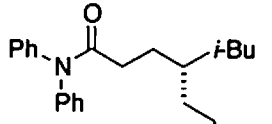
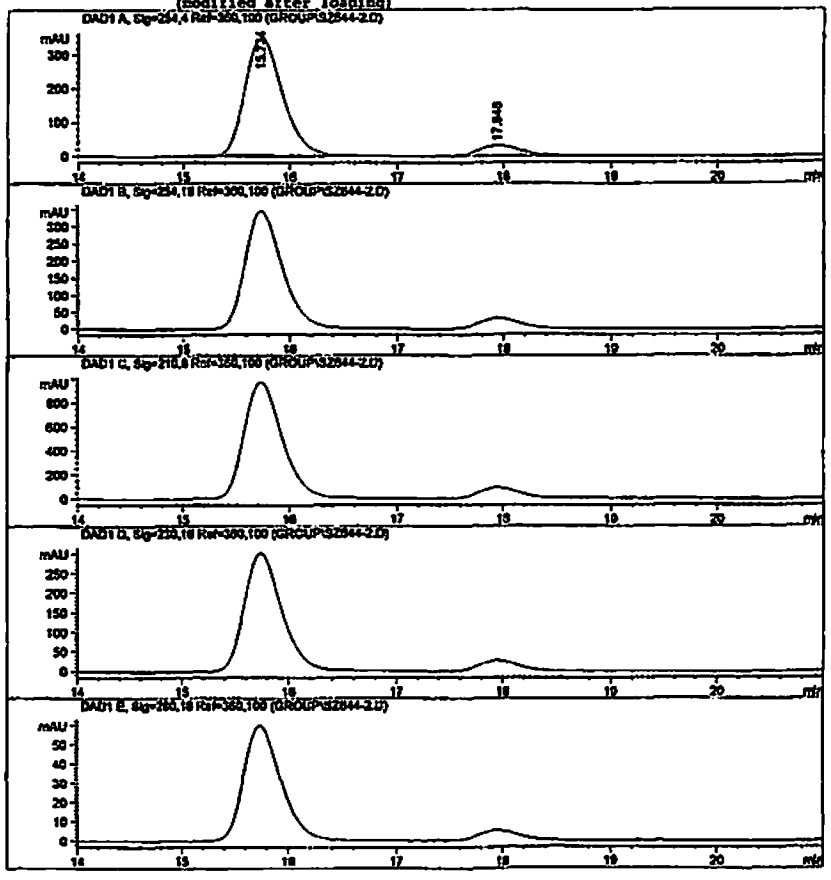


Table 1, entry 9

with (*R*,*R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ644-2.D

Injection Date	: 12/9/2010 8:01:09 PM	Seq. Line	: 8
Sample Name	:	Location	: Vial 62
Acq. Operator	: JTM	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ADH-0340.M		
Last changed	: 6/16/2009 1:35:44 PM by SZ		
Analysis Method	: C:\HPCHEM\1\METHODS\OJH-0060.M		
Last changed	: 8/21/2011 6:59:44 PM by SN		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ644-2.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.734	PB	0.3758	8592.07715	353.19318	91.3477
2	17.946	BB	0.3938	813.83118	31.25548	8.6523
Totals :				9405.90833	384.44866	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

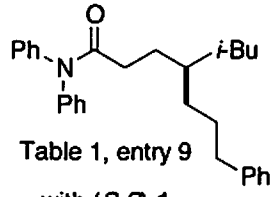
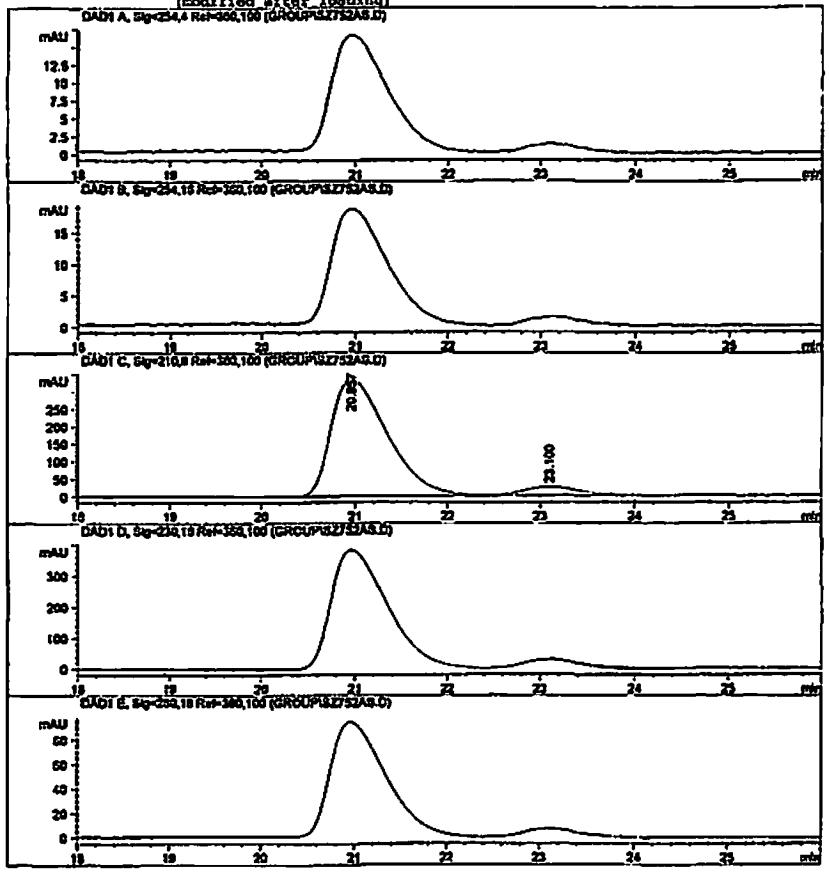


Table 1, entry 9

with (*S*,*S*)-1

Data File C:\HPCHEM\1\DATA\GROUP\S2752AS.D

Injection Date : 5/30/2011 8:46:11 PM Seq. Line : 24
Sample Name : Location : Vial 1
Acq. Operator : JTM Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence : Actual Inj Volume : 2 µl
Acq. Method : C:\HPCHEM\1\METHODS\ASH-0240.M
Last changed : 9/17/2009 10:57:40 AM by JC
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 7:00:29 PM by SH
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\S2752AS.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.645	PB	0.0805	6.42441	1.00594	100.0000
Totals :				6.42441	1.00594	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

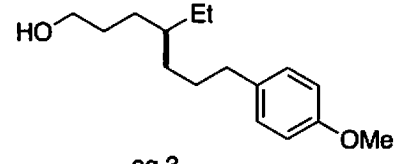
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.957	VB	0.6282	1.38881e4	334.61560	92.9735
2	23.100	BP	0.5470	1049.60107	25.56639	7.0265
Totals :				1.49377e4	360.18199	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

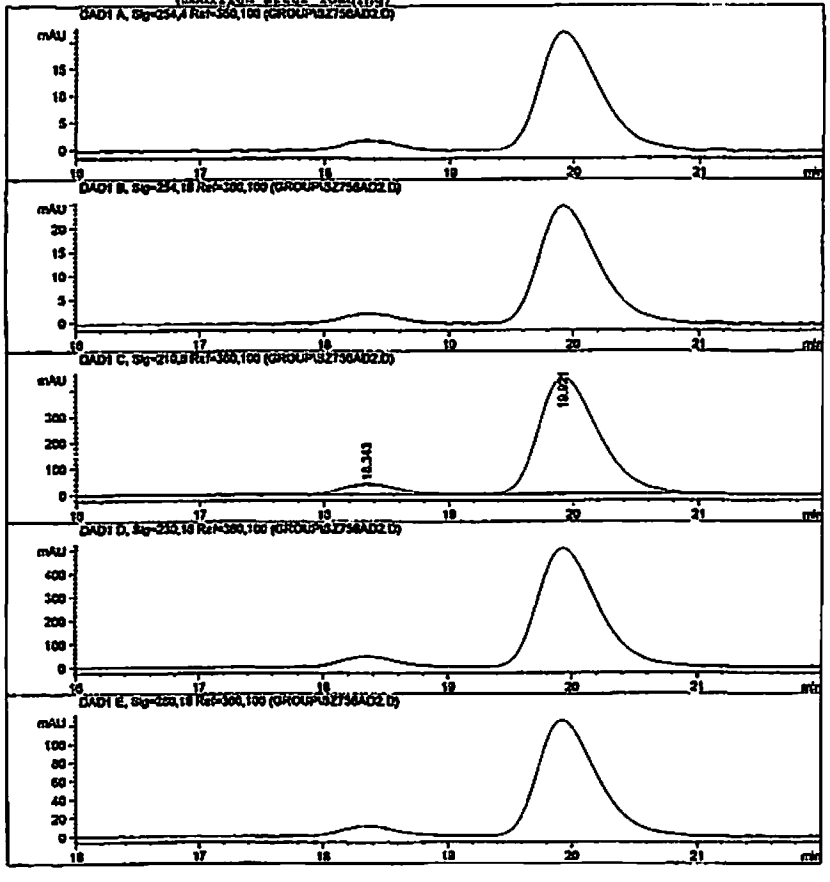
*** End of Report ***



eq 3

Data File C:\HPCHEM\1\DATA\GROUP\SZ756AD2.D

Injection Date : 5/28/2011 12:36:43 PM Seq. Line : 3
Sample Name : Location : Vial 3
Acq. Operator : JTH Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 2 µl
Acq. Method : C:\HPCHEM\1\METHODS\ADH-0260.M
Last changed : 3/11/2011 10:30:30 AM by JTH
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 7:01:32 PM by SW
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ756AD2.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

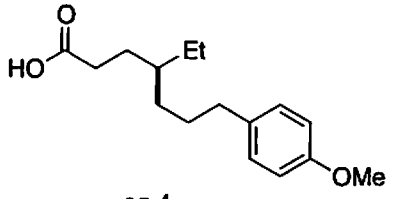
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.343	VP	0.4695	1263.04602	39.66729	7.8361
2	19.921	VB	0.5069	1.48552e4	447.93698	92.1639
Totals :				1.61182e4	487.60427	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

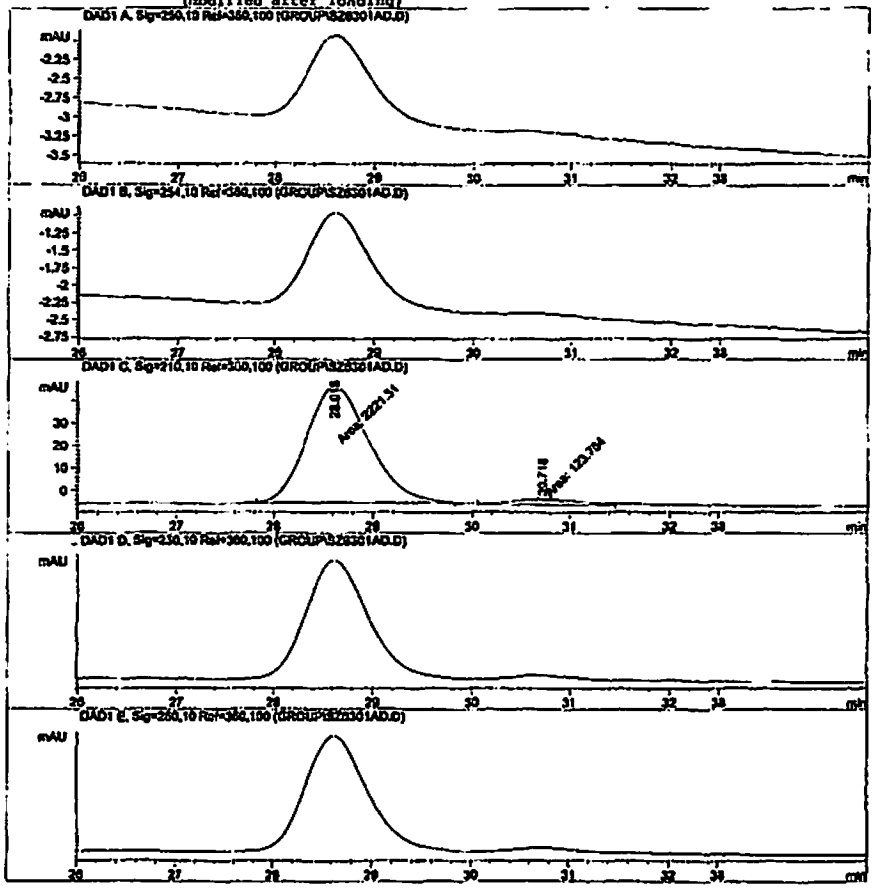
*** End of Report ***



eq 4

Data File C:\HPCHEM\1\DATA\GROUP\SZ6301AD.D

Injection Date : 10/9/2010 3:57:09 PM Seq. Line : 4
Sample Name : Location : Vial 81
Acq. Operator : JTH Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 15 µl
Different Inj Volume from Sequence : Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\AD-OJ-60.M
Last changed : 8/18/2010 8:28:57 AM by JTH
Analysis Method : C:\HPCHEM\1\METHODS\OJ-00590.M
Last changed : 8/21/2011 6:48:19 PM by NB
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ6301AD.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

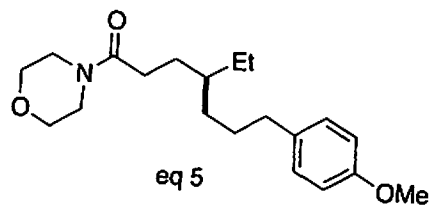
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.618	MM	0.7168	2221.30688	51.65020	94.7216
2	30.718	MM	0.8117	123.78417	2.54177	5.2784
Totals :				2345.09106	54.19197	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

*** End of Report ***

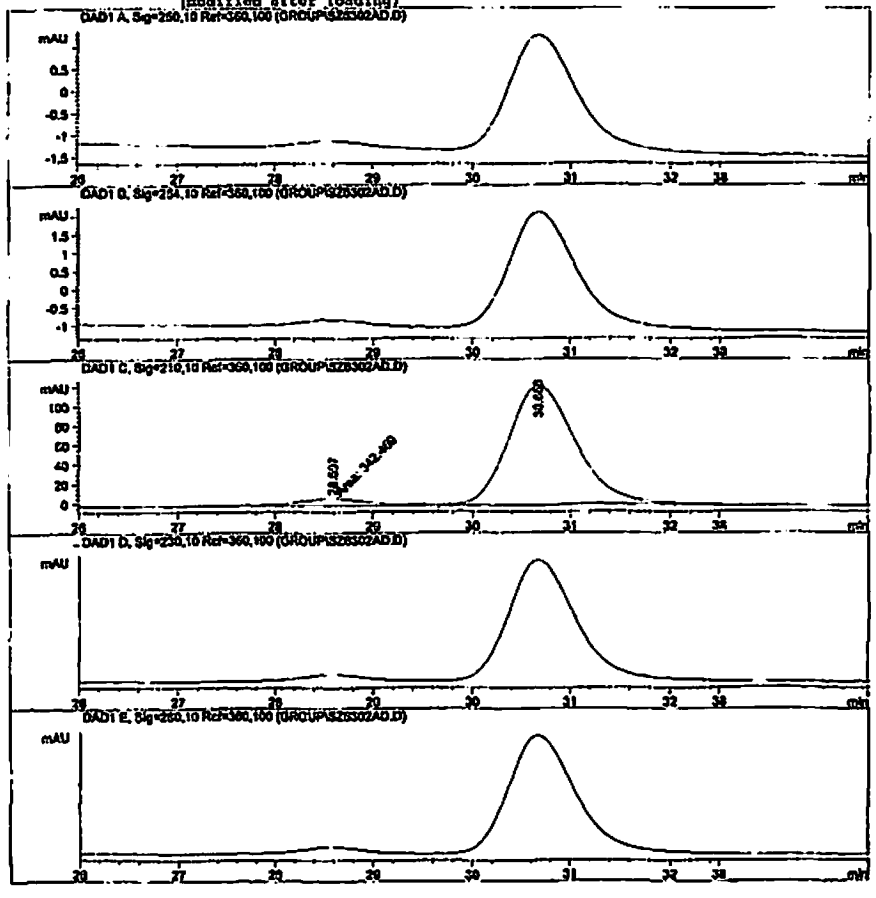


eq 5

Instrument 1 8/21/2011 7:05:19 PM NB

Data File C:\HPCHEM\1\DATA\GROUP\SZ6302AD.D

Injection Date	: 10/9/2010 5:35:48 PM	Seq. Line	: 6
Sample Name	:	Location	: Vial 82
Acq. Operator	: JTM	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 15 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 10 µl
Acq. Method	: C:\HPCHEM\1\METHODS\AD-03-40.M		
Last changed	: 2/18/2007 7:39:36 PM by GROUP		
Analysis Method	: C:\HPCHEM\1\METHODS\OJ-00590.M		
Last changed	: 8/21/2011 7:05:22 PM by NB		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ6302AD.D

Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.597	MM	0.7733	342.46872	7.38128	5.5867
2	30.666	BB	0.7313	5787.55469	122.46820	94.4133

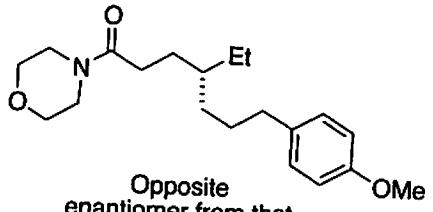
Totals : 6130.02341 129.84948

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

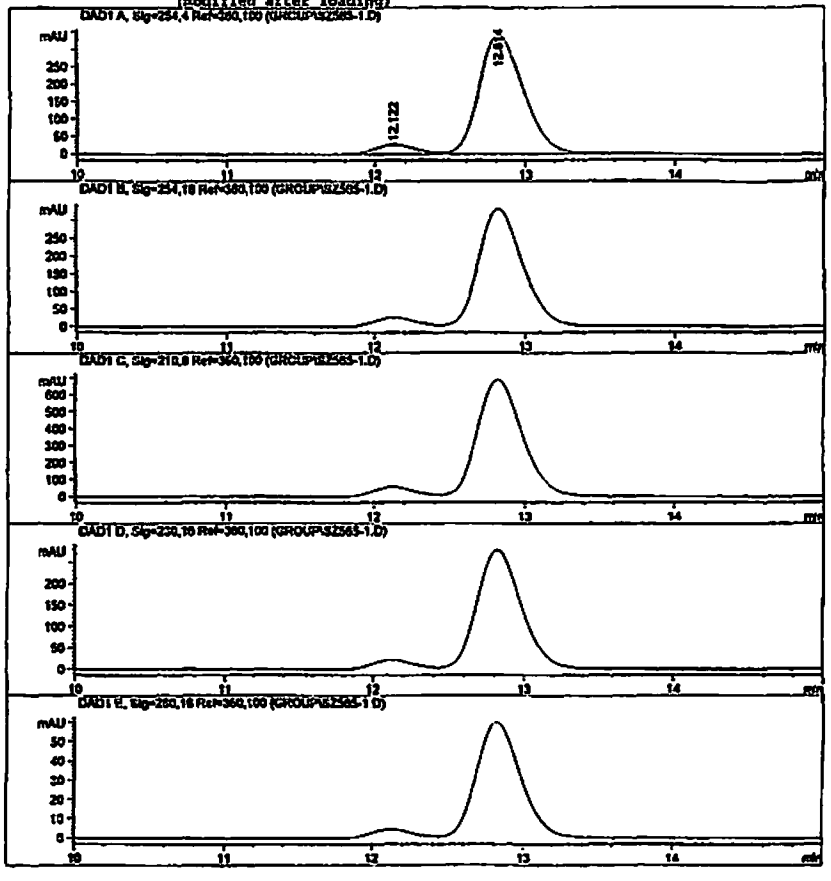
*** End of Report ***



Opposite enantiomer from that shown in eq 5

Data File C:\HPCHEM\1\DATA\GROUP\SZ565-1.D

Injection Date : 7/22/2010 2:05:48 PM Seq. Line : 2
Sample Name : Location : Vial 1
Acq. Operator : jtm Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\ADII-0130.M
Last changed : 5/8/2009 8:39:29 AM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 7:02:46 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ565-1.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.122	PV	0.2875	503.80266	26.97778	6.6146
2	12.814	VB	0.3201	7101.46631	342.20755	93.3854
Totals :				7604.46896	369.18533	

Results obtained with enhanced integrator!

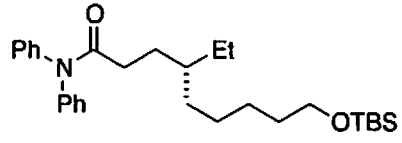
Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

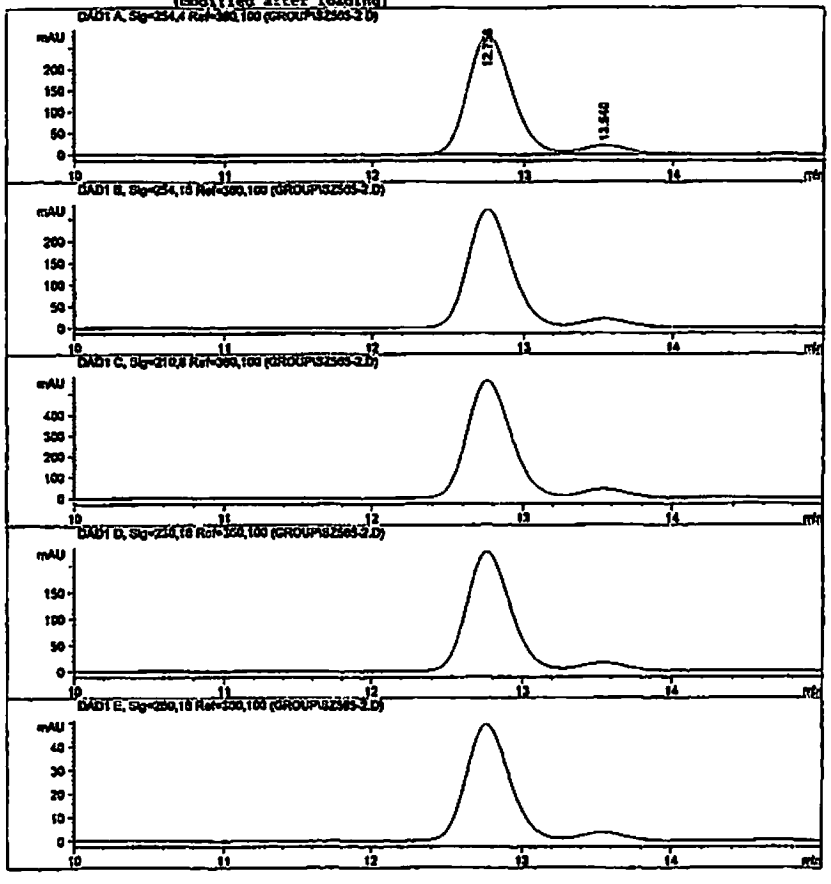


eq 6

with (*R,R*)-**1**

Data File C:\HPCHEM\1\DATA\GROUP\SZ565-2.D

Injection Date	: 7/22/2010 2:37:02 PM	Seq. Line	: 3
Sample Name	:	Location	: Vial 2
Acq. Operator	: jtm	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ADH-0130.M		
Last changed	: 5/8/2009 8:39:29 AM by NN		
Analysis Method	: C:\HPCHEM\1\METHODS\OJH-0060.M		
Last changed	: 8/21/2011 7:15:12 PM by SN		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ565-2.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.758	BV	0.3127	5623.28369	279.52429	92.9098
2	13.540	VB	0.3295	429.13089	20.06857	7.0902
Totals :				6052.41458	299.59286	

Results obtained with enhanced integrator!

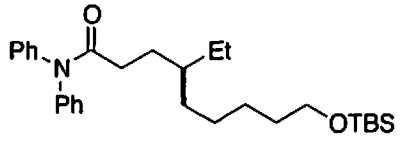
Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

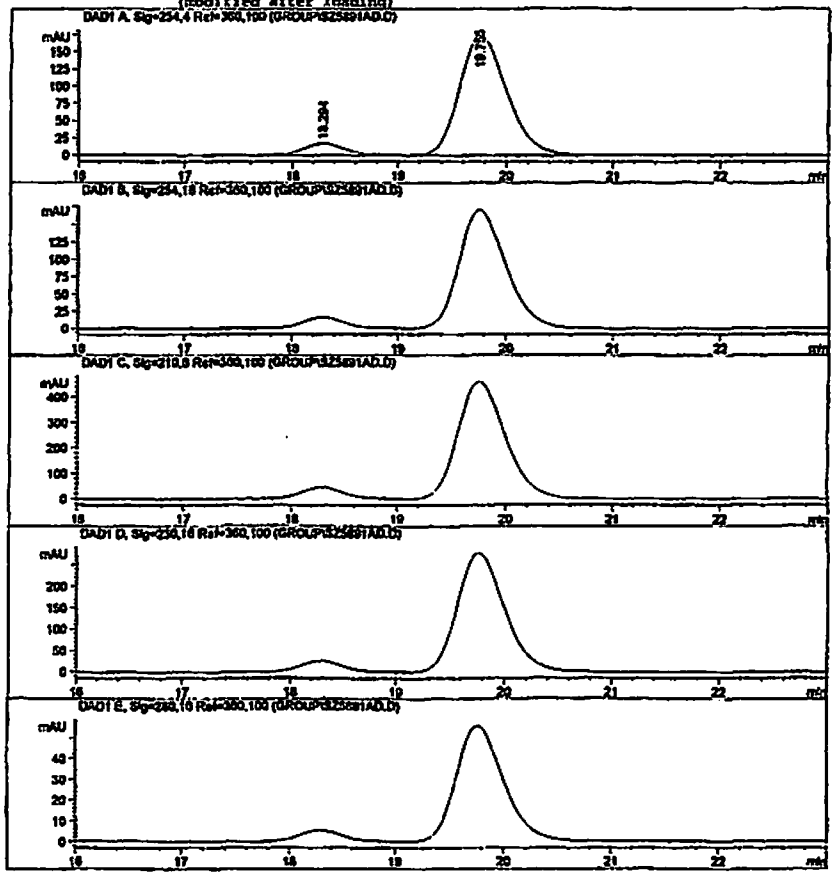


eq 6

with (*S*,*S*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ5891AD.D

Injection Date	: 8/27/2010 6:01:17 AM	Seq. Line	: 38
Sample Name	:	Location	: Vial 71
Acq. Operator	: jtm	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence :	Actual	Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ADH-1040.M		
Last changed	: 5/8/2009 8:41:57 AM by NM		
Analysis Method	: C:\HPCHEM\1\METHODS\OJH-0060.M		
Last changed	: 8/21/2011 7:15:55 PM by SN		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ5891AD.D

Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.294	VV	0.3466	499.66953	17.11603	8.7025
2	19.755	VV	0.4447	5242.00391	173.41225	91.2975
Totals :				5741.67343	190.52828	

Results obtained with enhanced integrator!

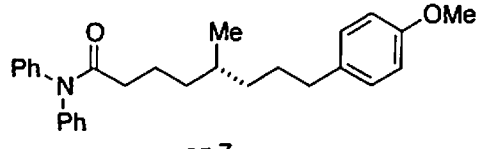
Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

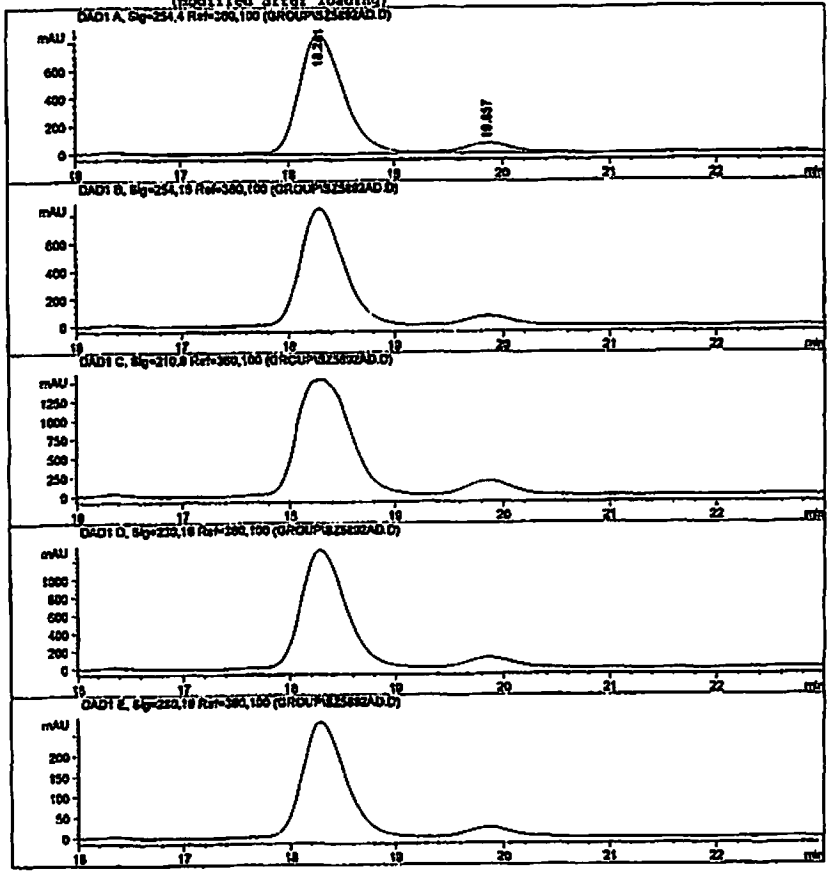


eq 7

with (*R*,*R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ5892AD.D

Injection Date : 8/27/2010 6:42:36 AM Seq. Line : 39
Sample Name : Location : Vial 72
Acq. Operator : jtm Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\ADB-1040.M
Last changed : 5/8/2009 8:41:57 AM by RM
Analysis Method : C:\HPCHEM\1\METHODS\OJB-0060.M
Last changed : 8/21/2011 7:20:07 PM by SH
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ5892AD.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.281	VV	0.4367	2.49516e4	859.37195	91.8934
2	19.857	VB	0.4449	2201.17993	70.77409	8.1066
Totals :				2.71528e4	930.14603	

Results obtained with enhanced integrator!

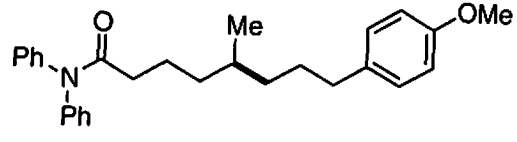
Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

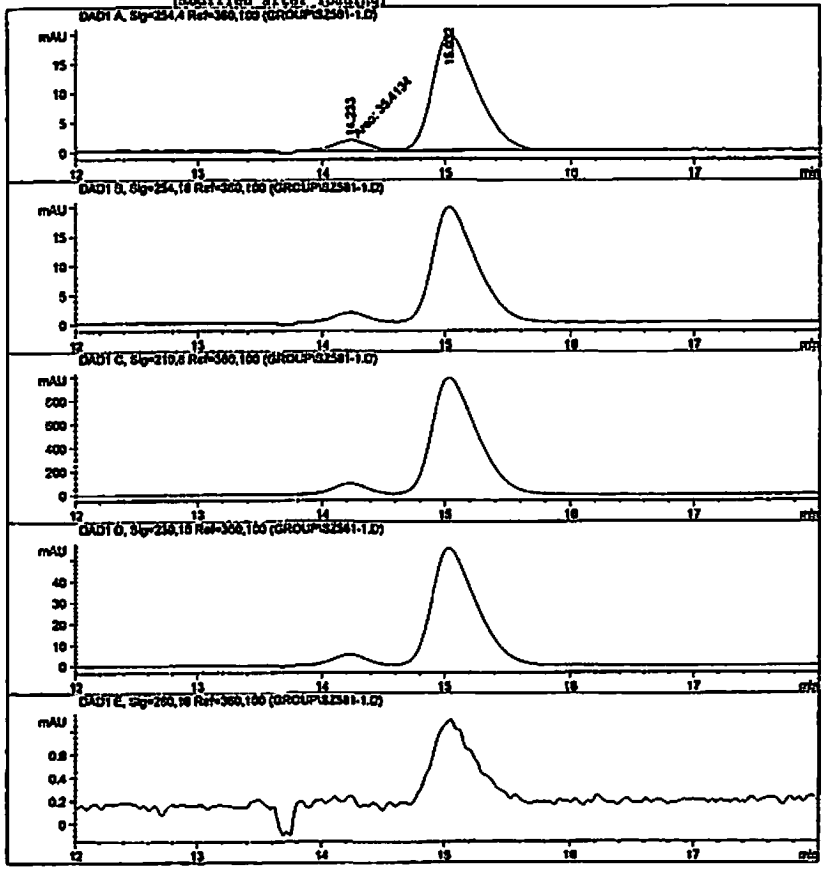


eq 8

with (*S*,*S*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ581-1.D

Injection Date	: 8/9/2010 11:27:21 PM	Seq. Line	: 6
Sample Name	:	Location	: Vial 1
Acq. Operator	: jtm	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\OJH-0140.M		
Last changed	: 5/8/2009 8:25:25 AM by MM		
Analysis Method	: C:\HPCHEM\1\METHODS\OJH-0060.M		
Last changed	: 8/21/2011 7:21:46 PM by SH		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ581-1.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.233	MM	0.3305	35.41337	1.78586	6.8279
2	15.032	VP	0.3551	483.24210	19.77509	93.1721
Totals :				518.65547	21.56096	

Results obtained with enhanced integrator!

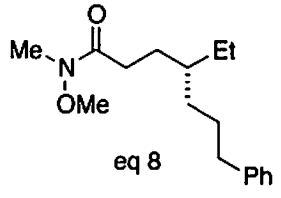
Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

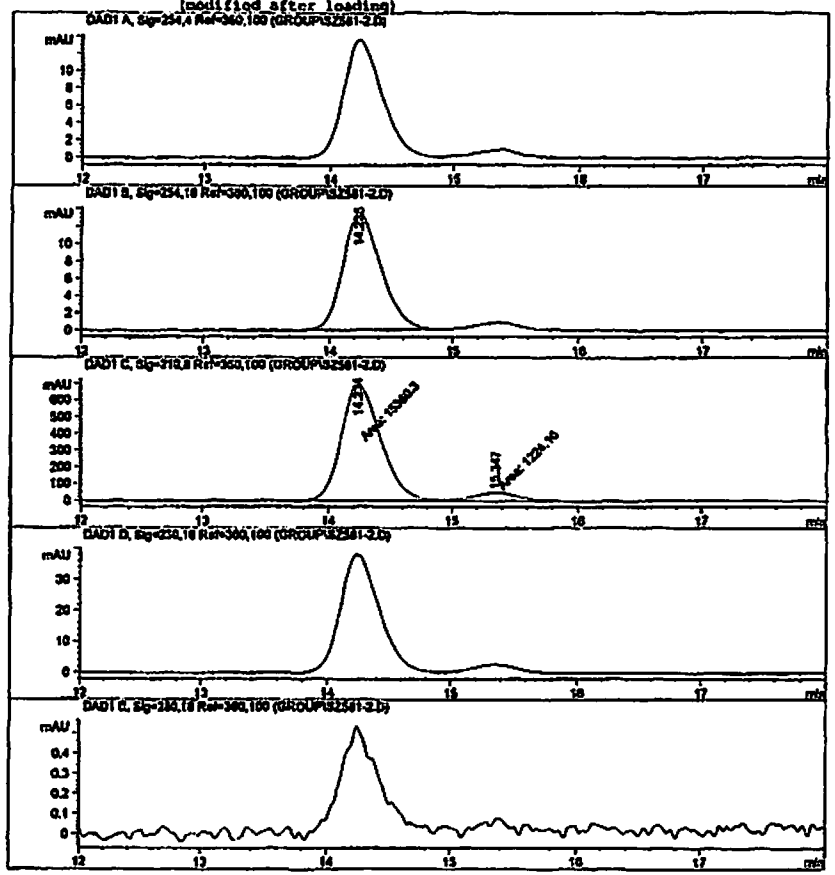


eq 8

with (*R,R*)-**1**

Data File C:\HPCHEM\1\DATA\GROUP\SZ581-2.D

Injection Date	: 8/10/2010 12:08:38 AM	Seq. Line	: 7
Sample Name	:	Location	: Vial 2
Acq. Operator	: jtm	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence :		Actual Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\OJH-0140.M		
Last changed	: 5/8/2009 8:25:25 AM by HM		
Analysis Method	: C:\HPCHEM\1\METHODS\OJH-0060.M		
Last changed	: 8/21/2011 7:25:35 PM by SN		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ581-2.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.235	BB	0.3206	290.23990	13.51022	100.0000
Totals :				290.23990	13.51022	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

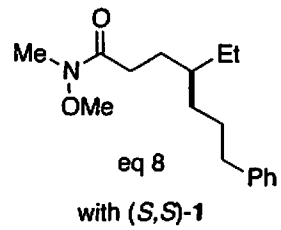
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.234	MF	0.3672	1.53803e4	698.07281	92.6275
2	15.347	FM	0.4122	1224.16309	49.50098	7.3725
Totals :				1.66045e4	747.57380	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

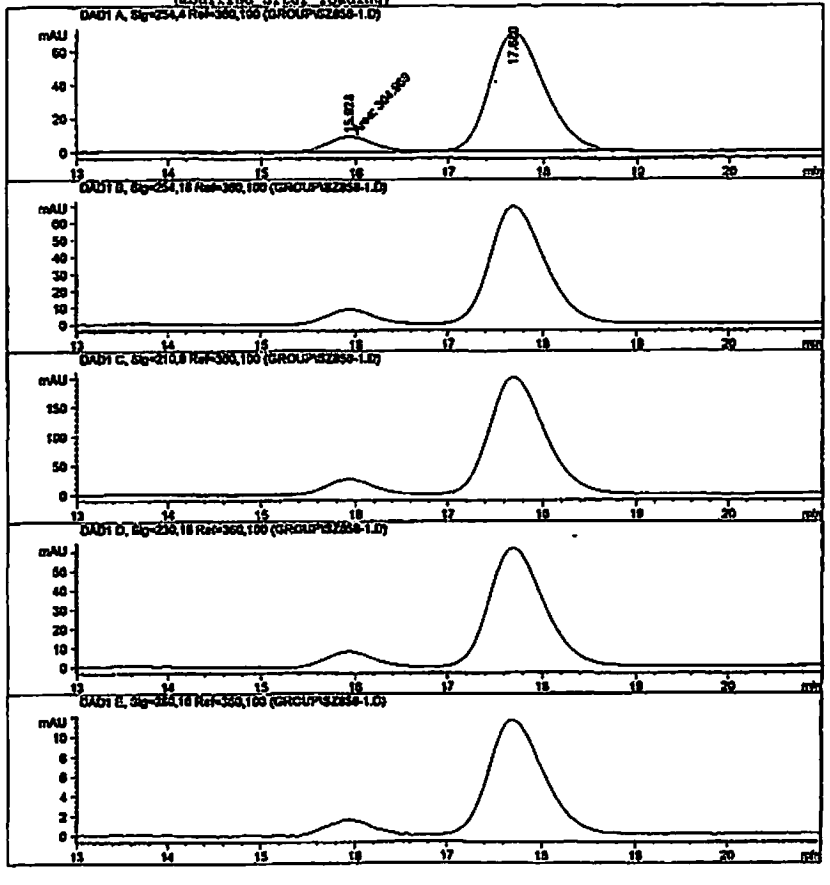


eq 8

with (*S*,*S*)-**1**

Data File C:\HPCHEM\1\DATA\GROUP\SZ858-1.D

Injection Date : 7/20/2011 6:52:06 AM Seq. Line : 33
Sample Name : Location : Vial 17
Acq. Operator : SN Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence : Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\ODH-0330.M
Last changed : 3/8/2011 6:39:45 PM by JTN
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 7:27:09 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ858-1.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.928	MM	0.5573	304.96912	9.12026	9.7416
2	17.689	BB	0.5851	2825.63208	70.57677	90.2584
Totals :				3130.60120	79.69703	

Results obtained with enhanced integrator!

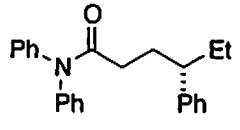
Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

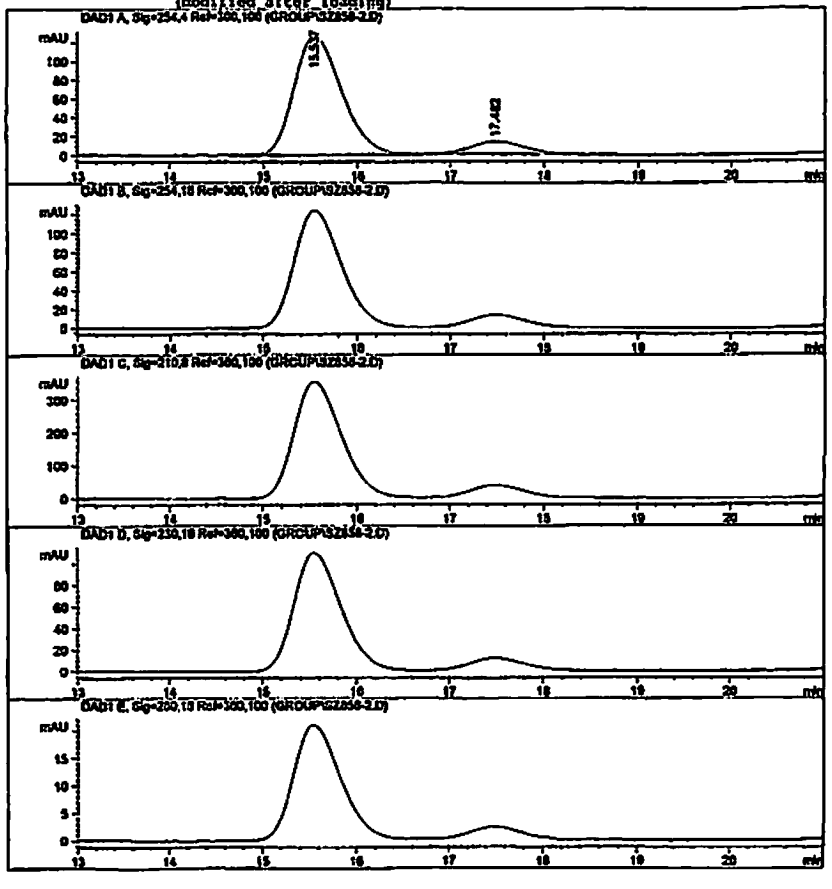


eq 9

with (*R,R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ858-2.D

Injection Date : 7/20/2011 7:23:19 AM Seq. Line : 34
Sample Name : Location : Vial 18
Acq. Operator : SN Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\OJH-0330.M
Last changed : 3/8/2011 6:39:45 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 7:30:19 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ858-2.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.537	FB	0.5250	4430.60596	127.00677	88.9048
2	17.482	BB	0.5154	552.93109	13.80100	11.0952
Totals :				4983.53705	140.80777	

Results obtained with enhanced integrator!

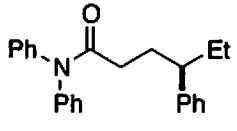
Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

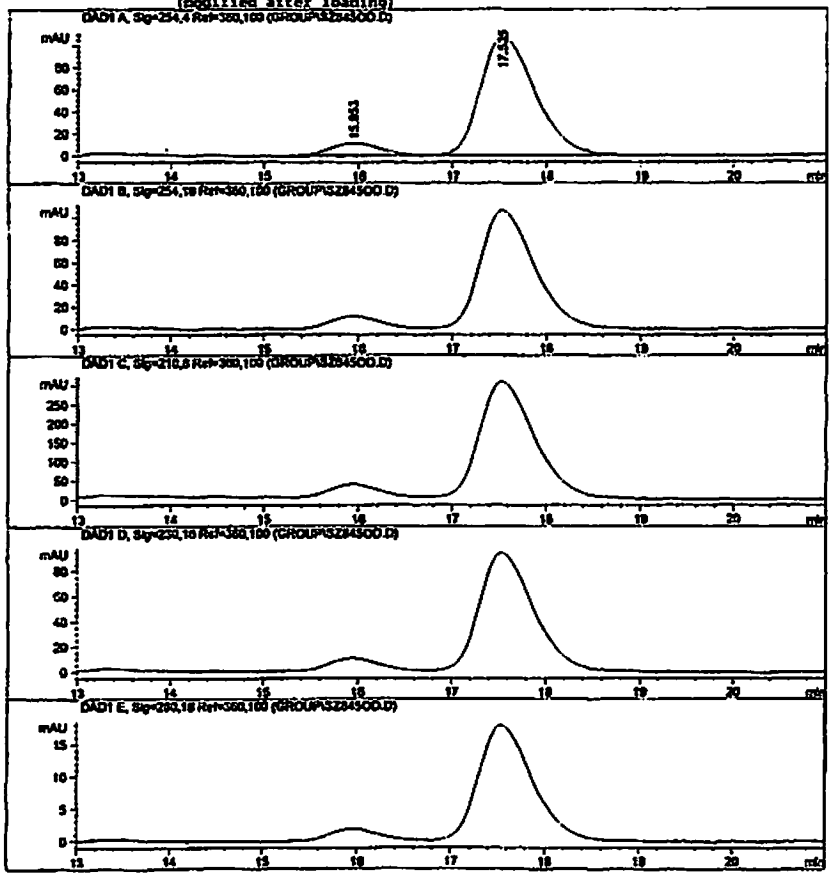


eq 9

with (*S*,*S*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ845OD.D

Injection Date	: 7/10/2011 3:46:08 PM	Seq. Line	: 5
Sample Name	:	Location	: Vial 41
Acq. Operator	: JTH	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence !	Actual Inj Volume	: 1 µl	
Acq. Method	: C:\HPCHEM\1\METHODS\ODH-0360.M		
Last changed	: 3/8/2011 6:33:52 PM by JTH		
Analysis Method	: C:\HPCHEM\1\METHODS\OJH-0060.M		
Last changed	: 8/21/2011 7:32:38 PM by SN		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ845OD.D

Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.953	PV	0.4370	412.78586	11.66039	8.7014
2	17.535	VB	0.6076	4331.09912	108.05372	91.2986
Totals :				4743.88498	119.71411	

Results obtained with enhanced integrator!

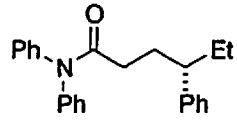
Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

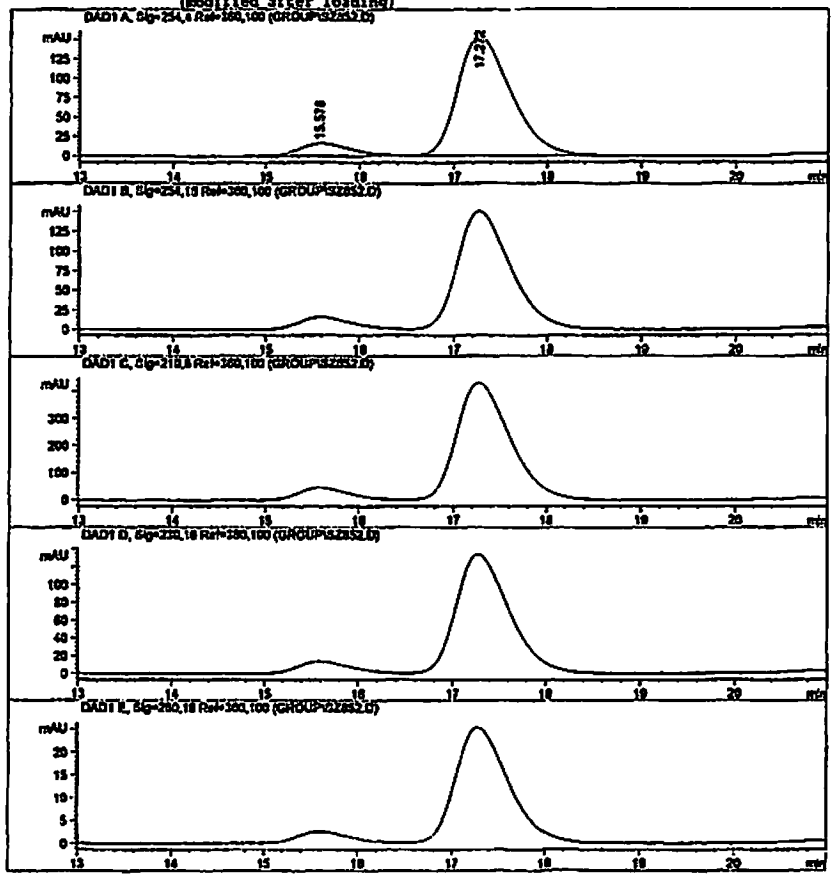


eq 10

with (*R*,*R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ852.D

Injection Date : 7/20/2011 7:54:31 AM Seq. Line : 35
Sample Name : Location : Vial 19
Acq. Operator : SN Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\OJH-0330.M
Last changed : 3/8/2011 6:39:45 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\OJH-0060.M
Last changed : 8/21/2011 7:32:38 PM by SN
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ852.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.578	BV	0.4615	612.44531	16.10928	9.1519
2	17.272	VB	0.5904	6079.54639	154.05106	90.8481
Totals :				6691.99170	170.16034	

Results obtained with enhanced integrator!

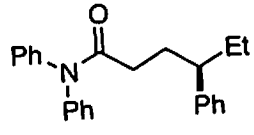
Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

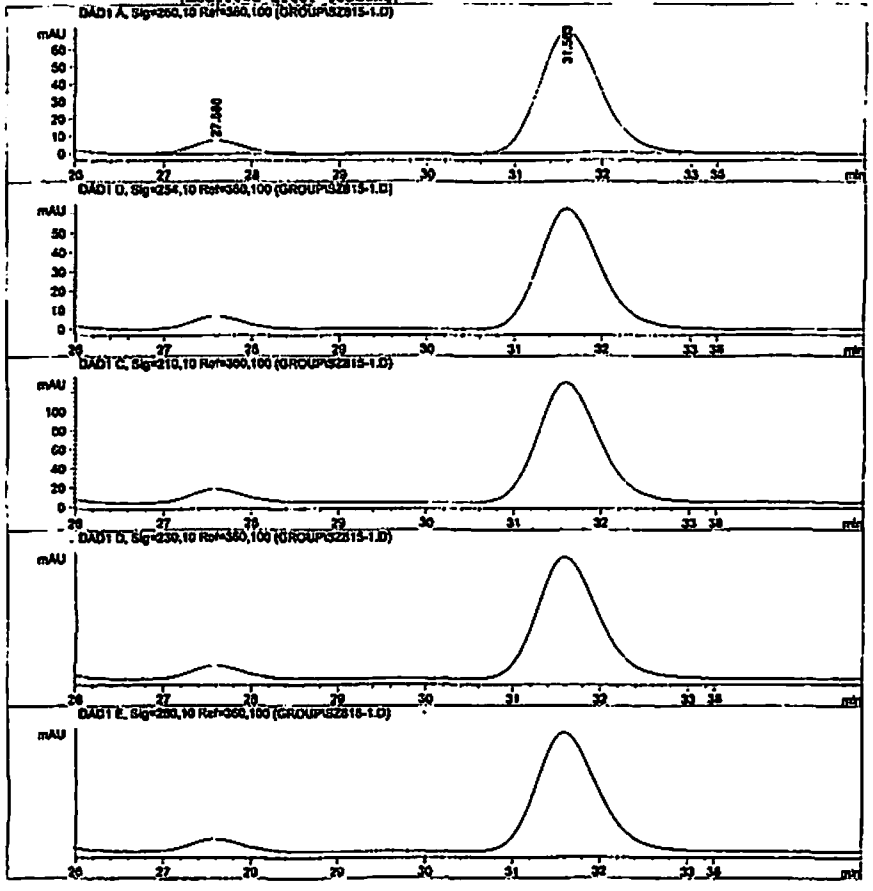


eq 10

with (*S,S*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ815-1.D

Injection Date	: 5/30/2011 11:30:20 AM	Seq. Line	: 2
Sample Name	:	Location	: Vial 21
Acq. Operator	: NB	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 15 µl
Different Inj Volume from Sequence !		Actual Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\AD-02-40.M		
Last changed	: 8/10/2010 9:22:11 AM by JTH		
Analysis Method	: C:\HPCHEM\1\METHODS\OJ-00590.M		
Last changed	: 8/21/2011 7:07:23 PM by NB		
	(modified after loading)		



Data File C:\HPCHEM\1\DATA\GROUP\SZ815-1.D

Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.586	BB	0.6131	309.12506	7.52697	8.2894
2	31.583	BB	0.7569	3420.02271	70.13905	91.7106
Totals :				3729.14777	77.66602	

Results obtained with enhanced integrator!

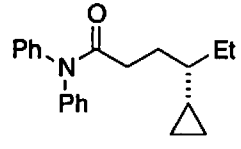
Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Signal 5: DAD1 E, Sig=200,10 Ref=360,100

*** End of Report ***

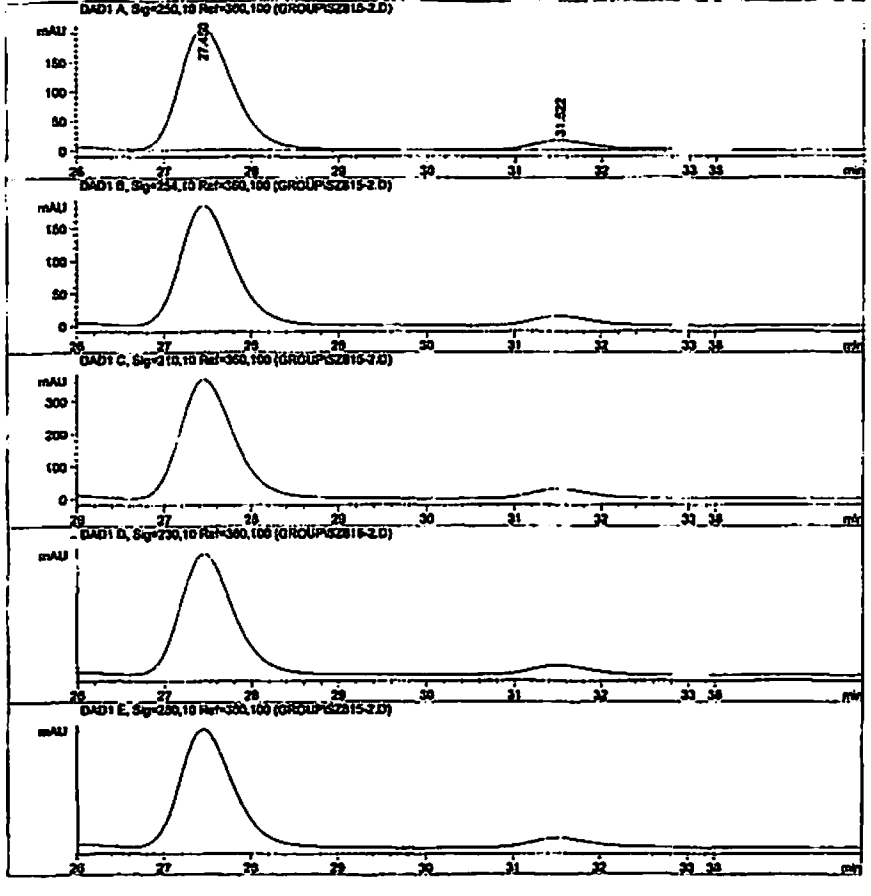


eq 11

with (*R*,*R*)-1

Data File C:\HPCHEM\1\DATA\GROUP\SZ815-2.D

Injection Date : 5/30/2011 12:11:31 PM Seq. Line : 3
Sample Name : Location : Vial 22
Acq. Operator : MB Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 15 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 1 µl
Acq. Method : C:\HPCHEM\1\METHODS\AD-02-40.M
Last changed : 8/10/2010 9:22:11 AM by JTH
Analysis Method : C:\HPCHEM\1\METHODS\OJ-00590.M
Last changed : 8/21/2011 7:34:04 PM by MB
(modified after loading)



Data File C:\HPCHEM\1\DATA\GROUP\SZ815-2.D

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.450	VB	0.6592	8912.49609	207.45563	92.1683
2	31.522	BB	0.7161	757.31165	16.06155	7.8317
Totals :				9669.80774	223.51717	

Results obtained with enhanced integrator!

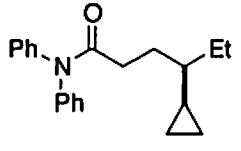
Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

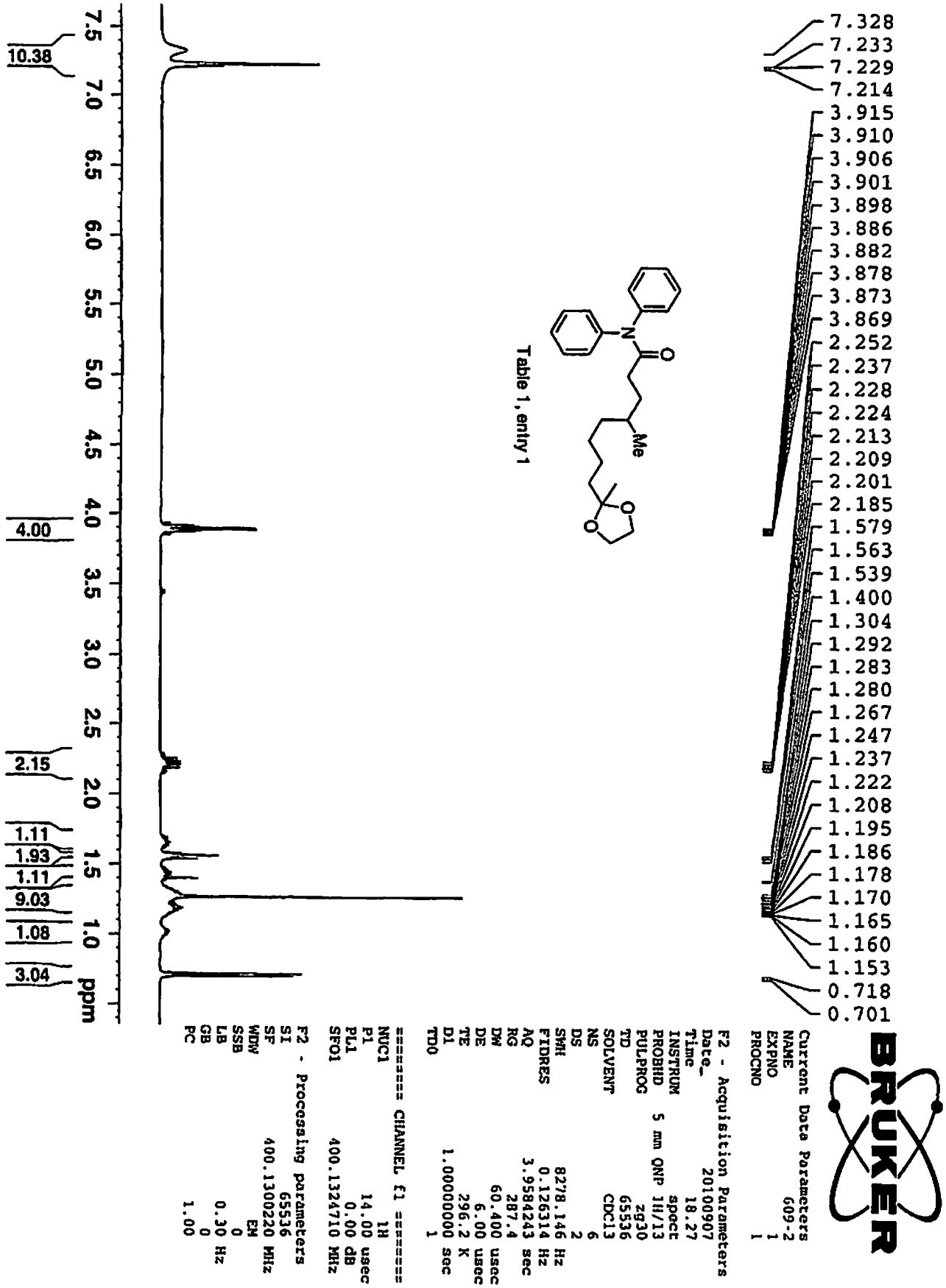
*** End of Report ***

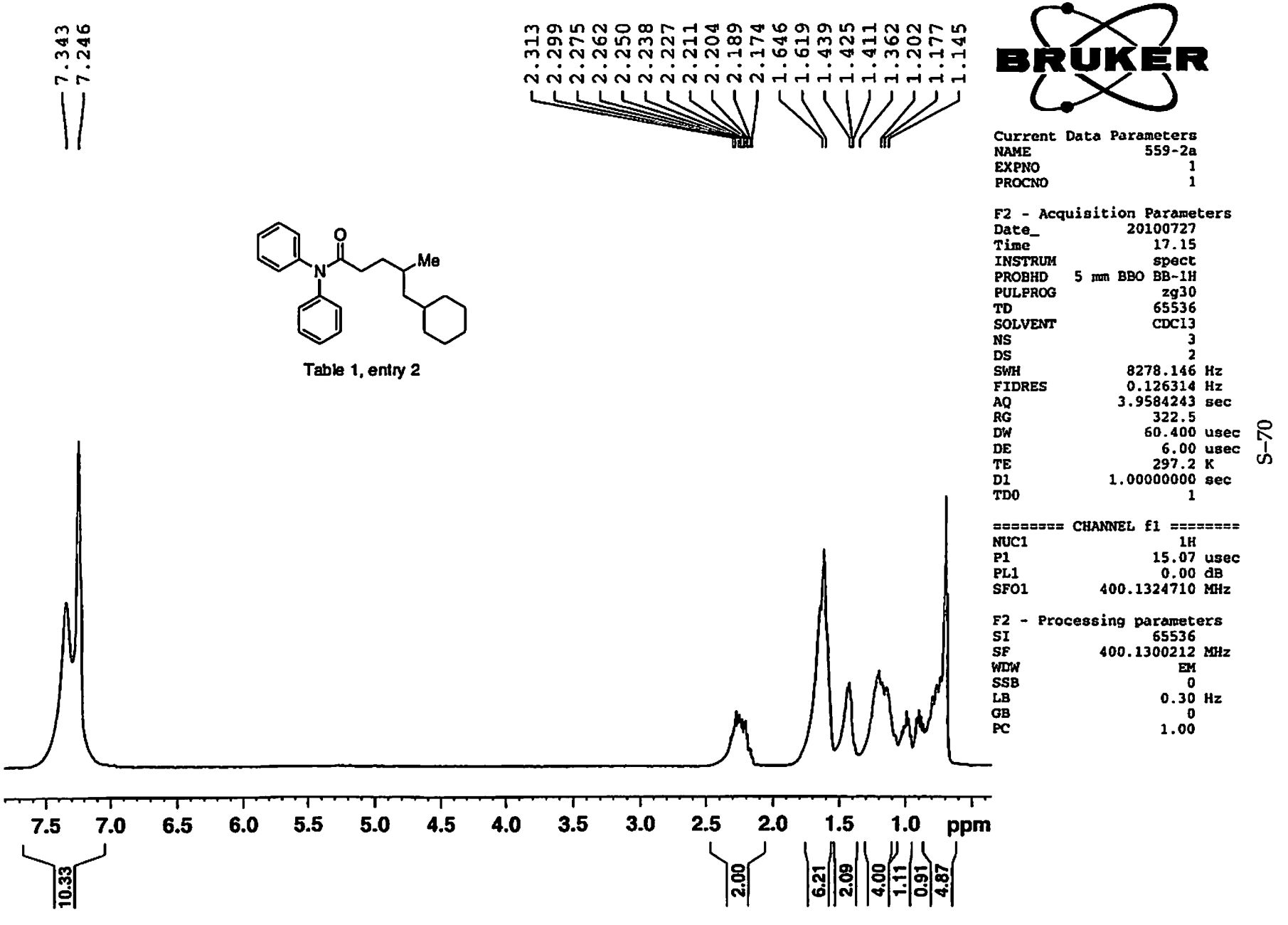


eq 11

with (*S,S*)-1

VIII. ^{1}H NMR Spectra




7.343
7.246
2.313
2.299
2.275
2.262
2.250
2.238
2.227
2.211
2.204
2.189
2.174
1.646
1.619
1.439
1.425
1.411
1.362
1.202
1.177
1.145
BRUKER
Current Data Parameters
NAME 559-2a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100727
Time 17.15
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.07 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300212 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
Me
Table 1, entry 2
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
10.33
2.00
6.21
2.09
4.00
1.11
0.91
4.87

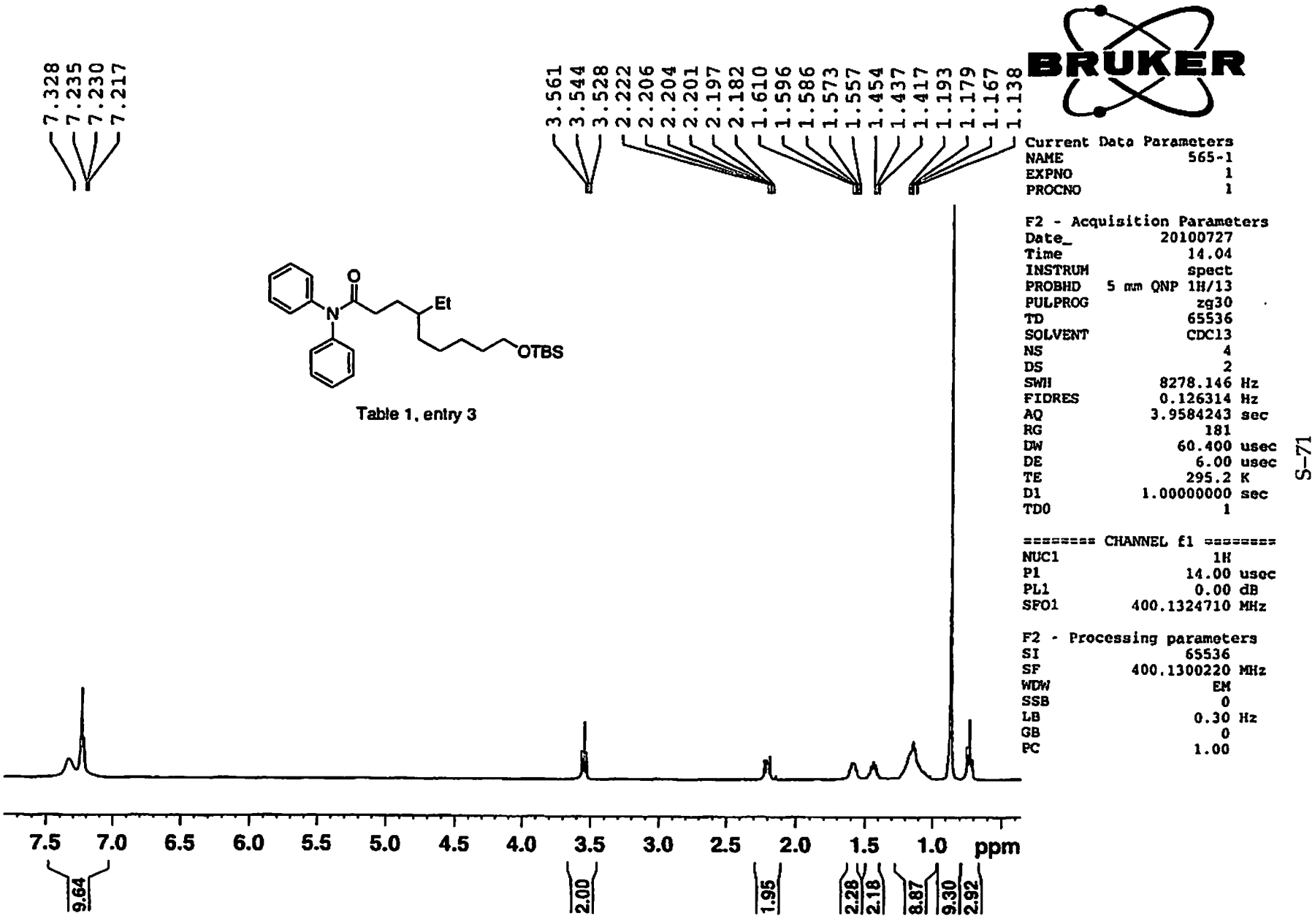
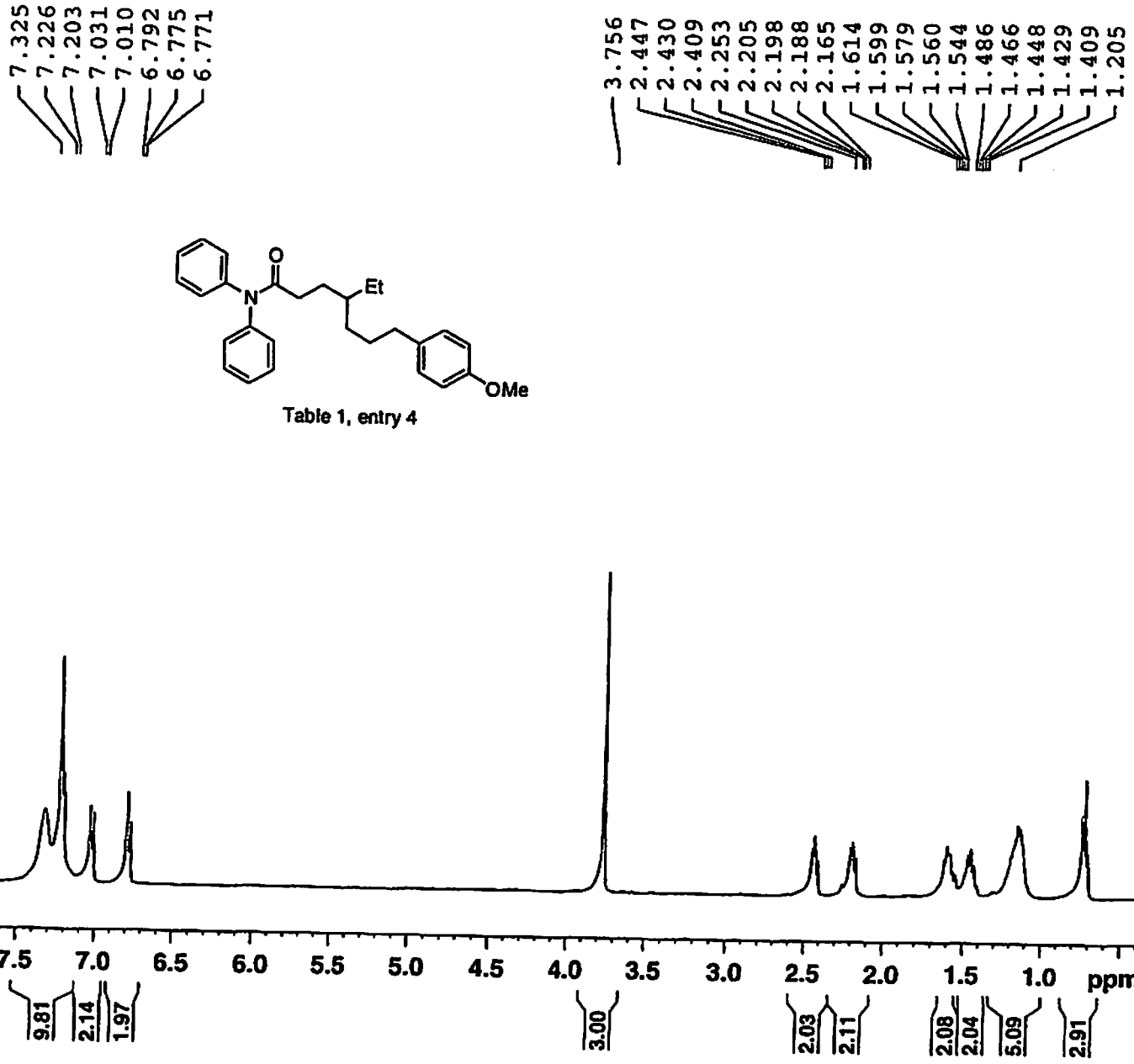
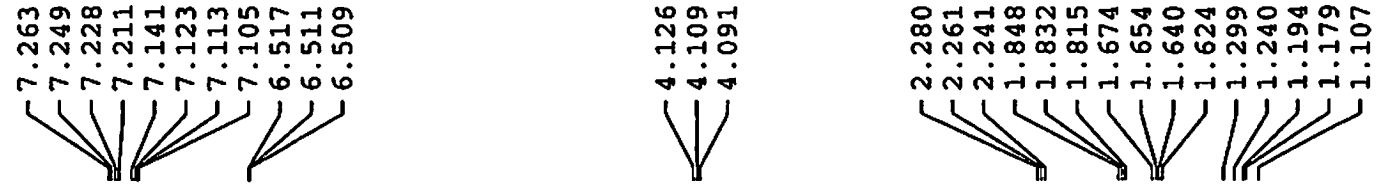


Table 1, entry 3


7.325
7.226
7.203
7.031
7.010
6.792
6.775
6.771
3.756
2.447
2.430
2.409
2.253
2.205
2.198
2.188
2.165
1.614
1.599
1.579
1.560
1.544
1.486
1.466
1.448
1.429
1.409
1.205
BRUKER
Current Data Parameters
NAME 550-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100708
Time 14.30
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.07 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300212 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
Et
OMe
Table 1, entry 4
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
9.81
2.14
1.97
3.00
2.03
2.11
2.08
2.04
5.09
2.91



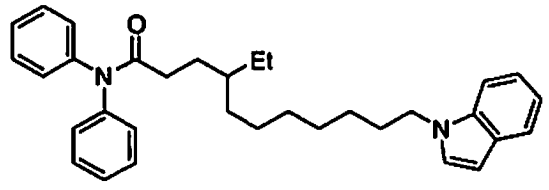
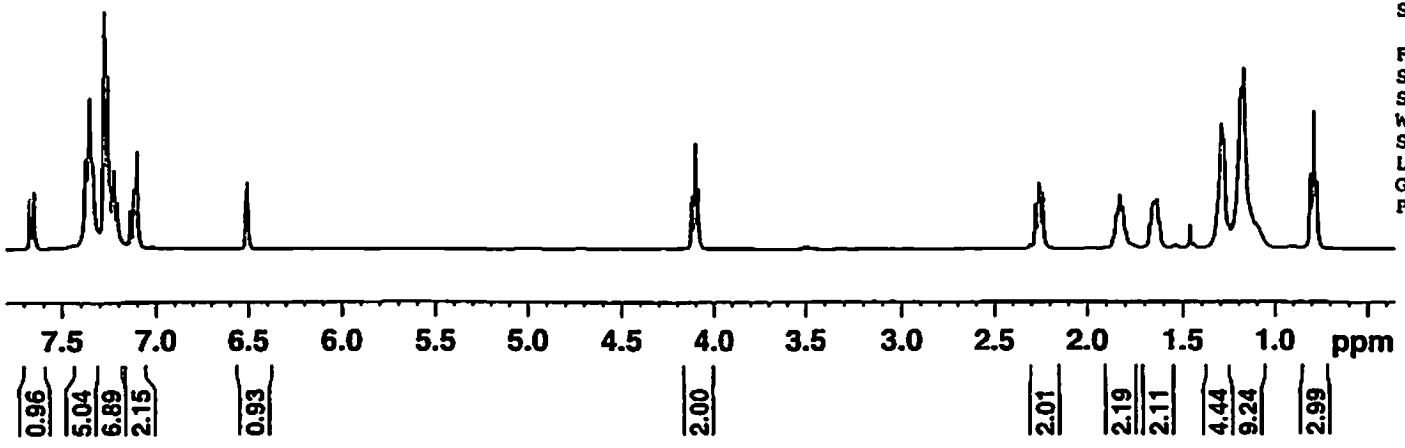


Table 1, entry 5



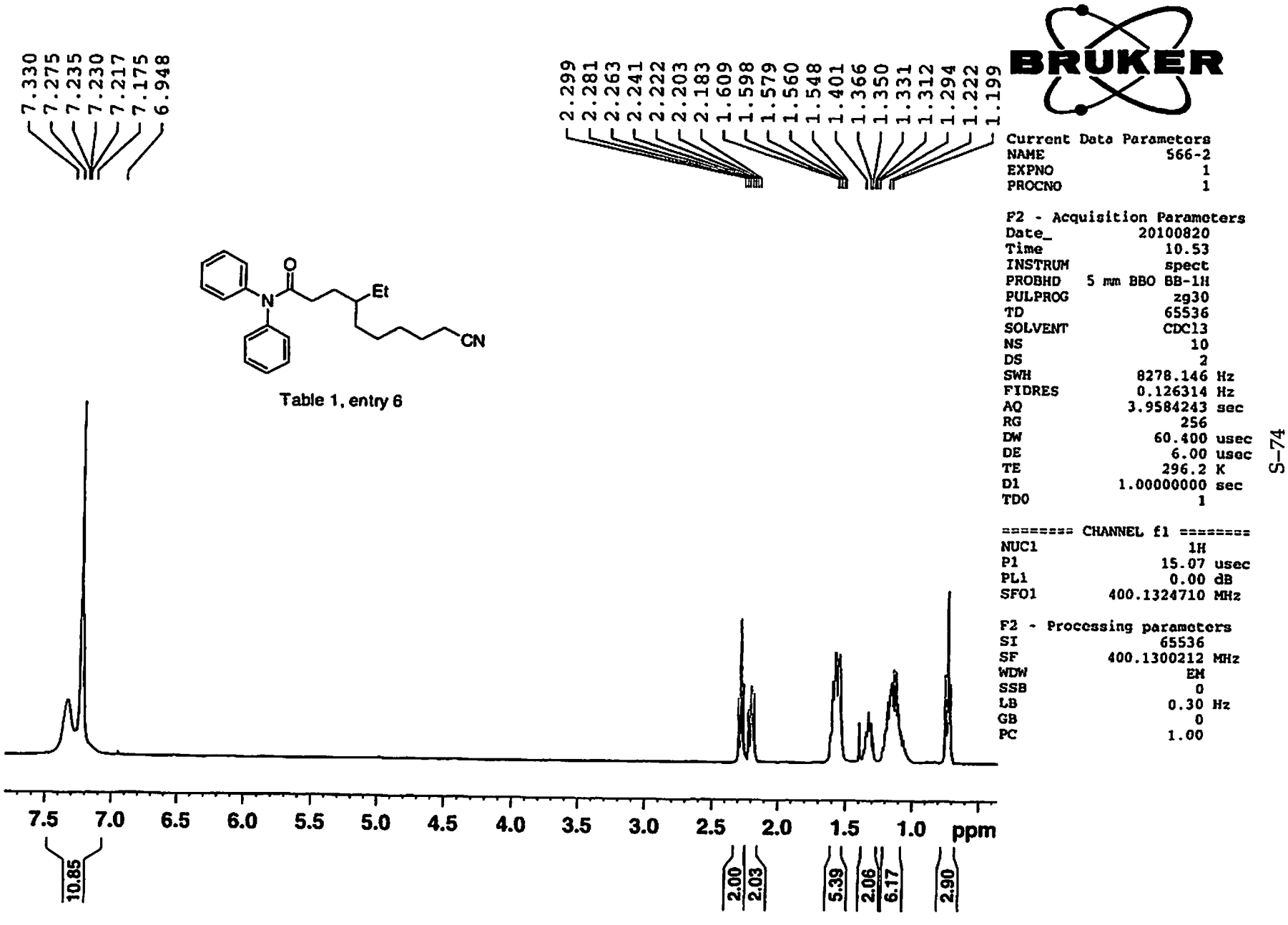
BRUKER

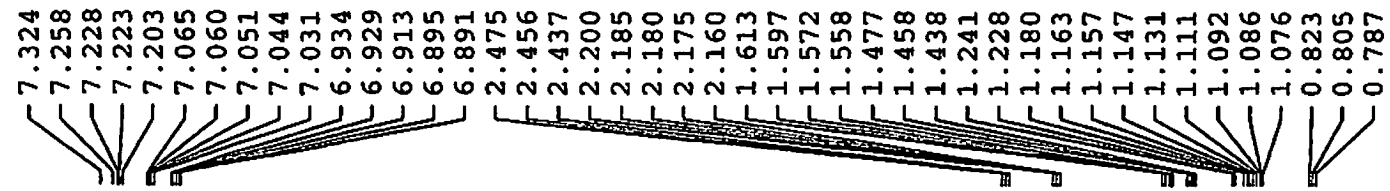
```
Current Data Parameters
NAME          787-1proton
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110511
Time                9.41
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    10
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  25.4
DW                60.400 usec
DE                 6.00 usec
TE                296.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.07 usec
PL1                0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300212 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```


7.330
7.275
7.235
7.230
7.217
7.175
6.948
2.299
2.281
2.263
2.241
2.222
2.203
2.183
1.609
1.598
1.579
1.560
1.548
1.401
1.366
1.350
1.331
1.312
1.294
1.222
1.199
BRUKER
Current Data Parameters
NAME 566-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100820
Time 10.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.07 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300212 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
Et
CN
Table 1, entry 6
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
10.85
2.00
2.03
5.39
2.06
6.17
2.90



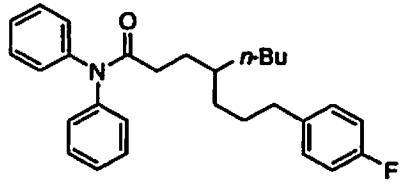
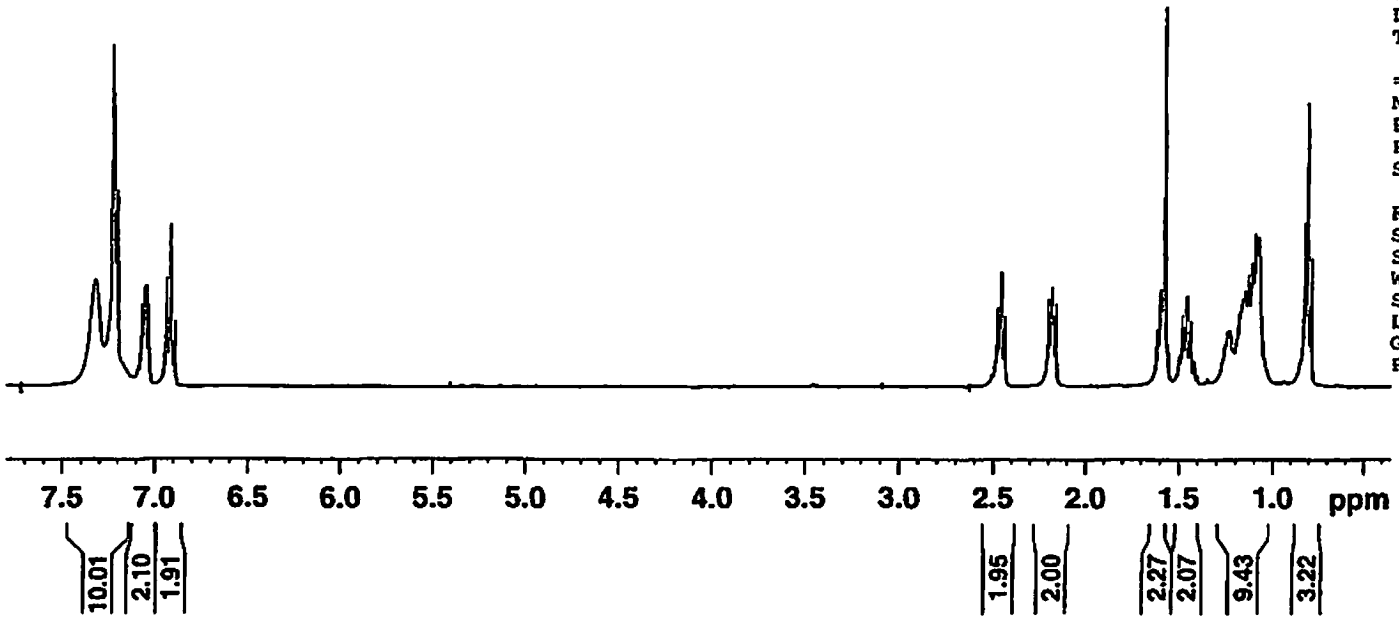


Table 1, entry 7




```
Current Data Paramoters
NAME                  584-2
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20100901
Time                 11.27
INSTRUM              spect
PROBHD    5 mm QNP 1H/13
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                        7
DS                        2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                      256
DW                 60.400 usec
DE                  6.00 usec
TE                  295.2 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
PL1                  0.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF             400.1300220 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```

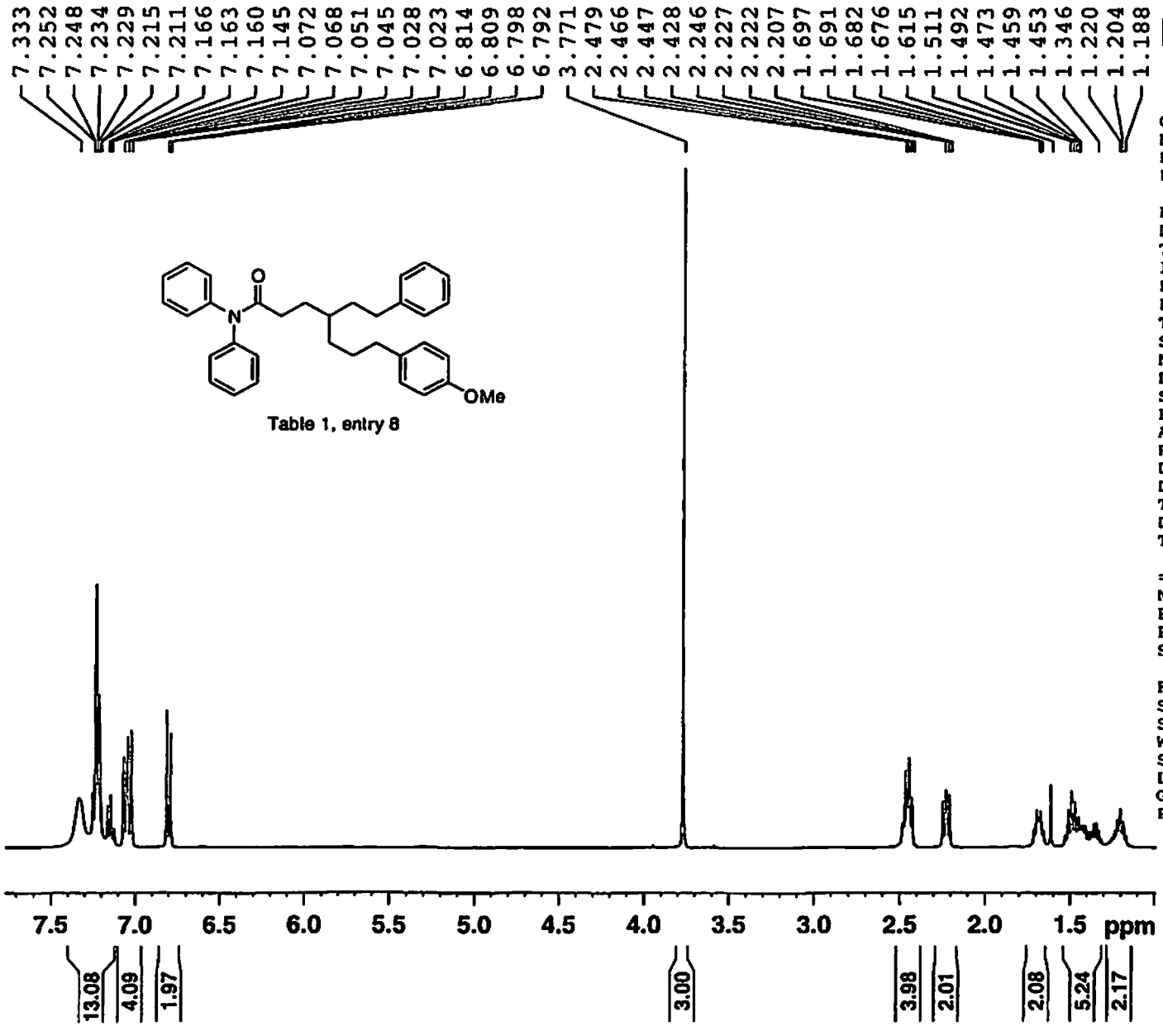


Table 1, entry 8

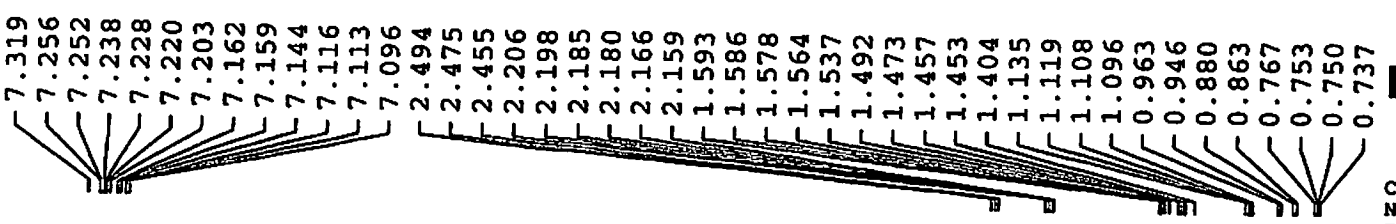
BRUKER

Current Data Parameters
NAME 631-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100929
Time 18.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

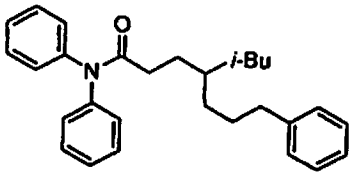


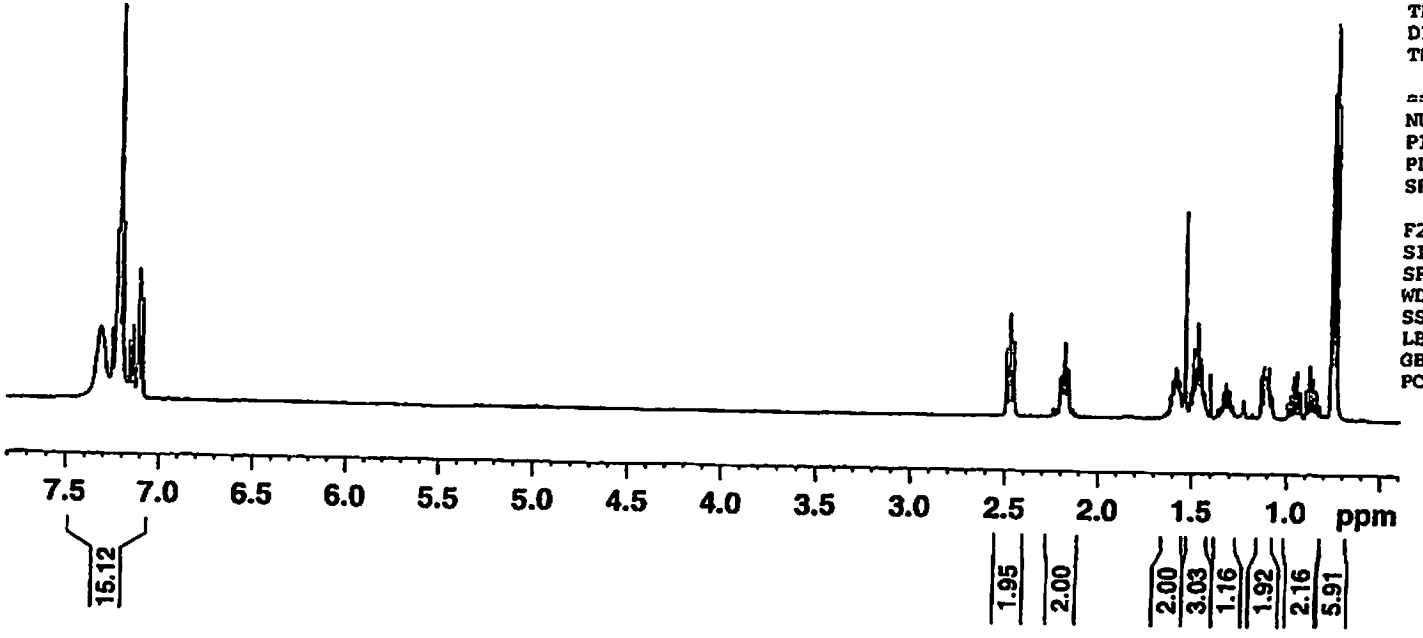
Table 1, entry 9

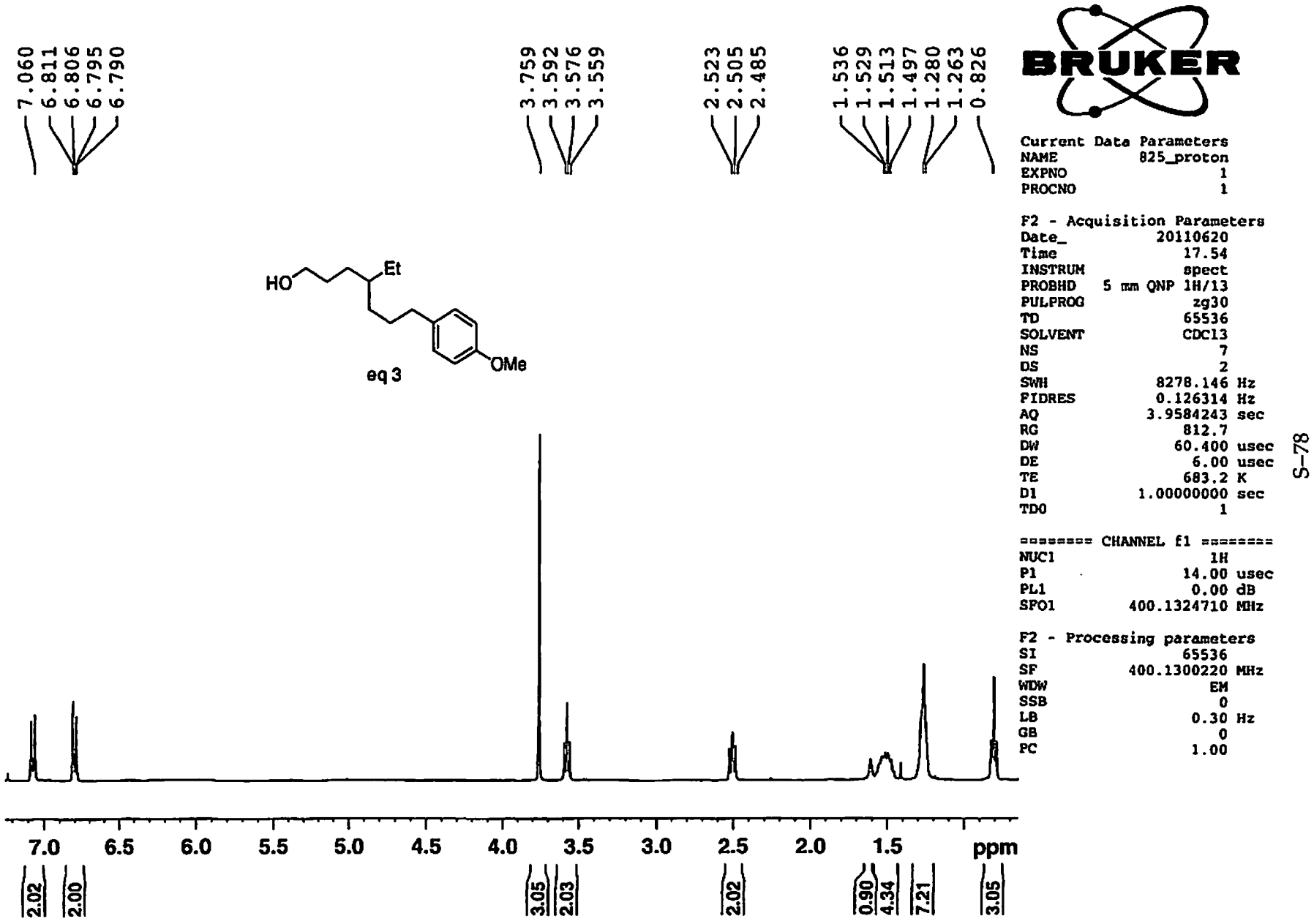
```
Current Data Parameters
NAME                  664-1
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20101210
Time                 11.31
INSTRUM              spect
PROBHD   5 mm BBO BB-1H
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                        2
DS                        2
SWH               8278.146 Hz
FIDRES               0.126314 Hz
AQ                3.9584243 sec
RG                   203.2
DW                   60.400 usec
DE                    6.00 usec
TE                   296.2 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    15.07 usec
PL1                    0.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF             400.1300212 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```





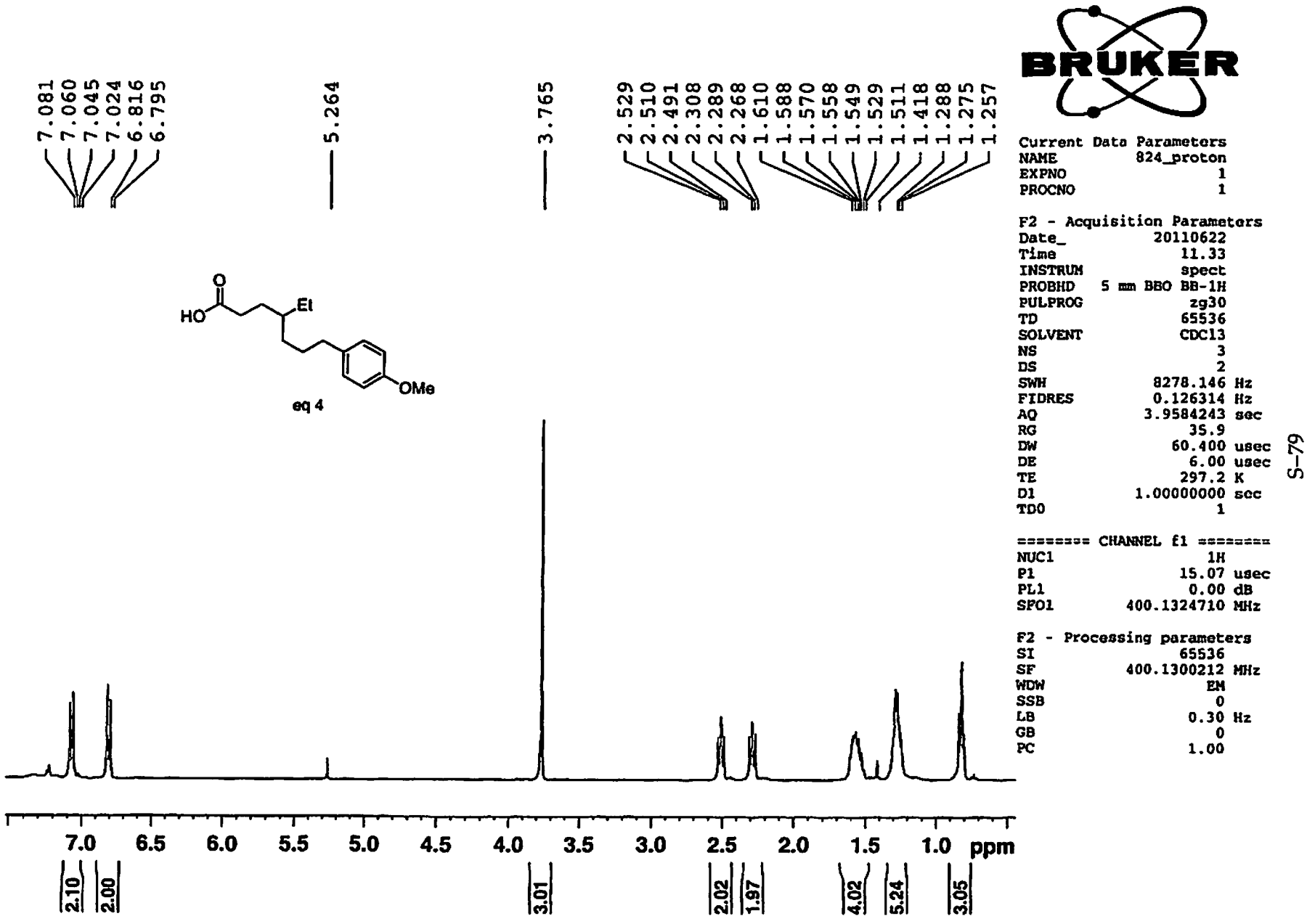
BRUKER

Current Data Parameters
NAME 825_proton
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110620
Time 17.54
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 812.7
DW 60.400 usec
DE 6.00 usec
TE 683.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



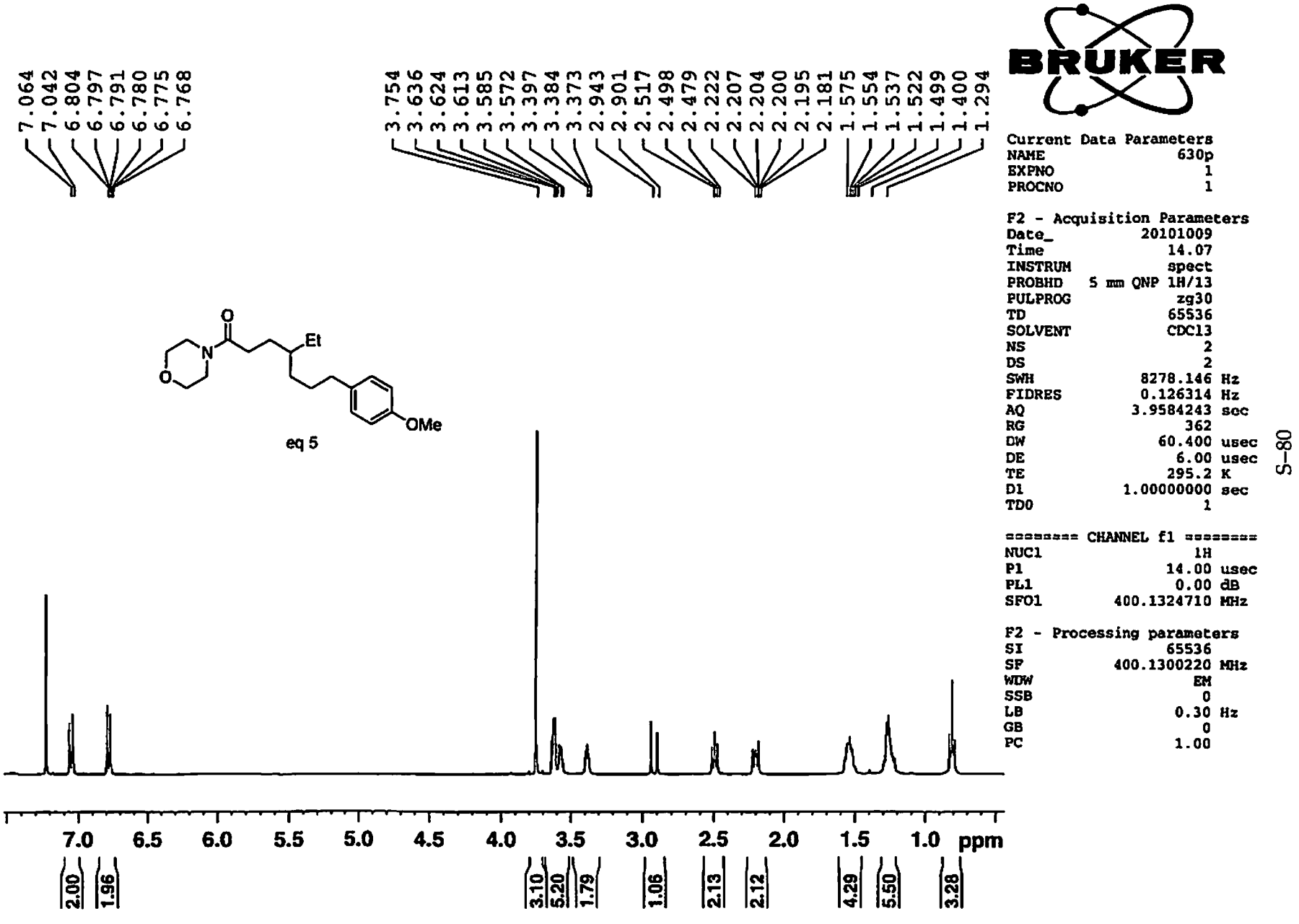
BRUKER

Current Data Parameters
NAME 824_proton
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110622
Time 11.33
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 35.9
DW 60.400 usec
DE 6.00 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.07 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300212 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



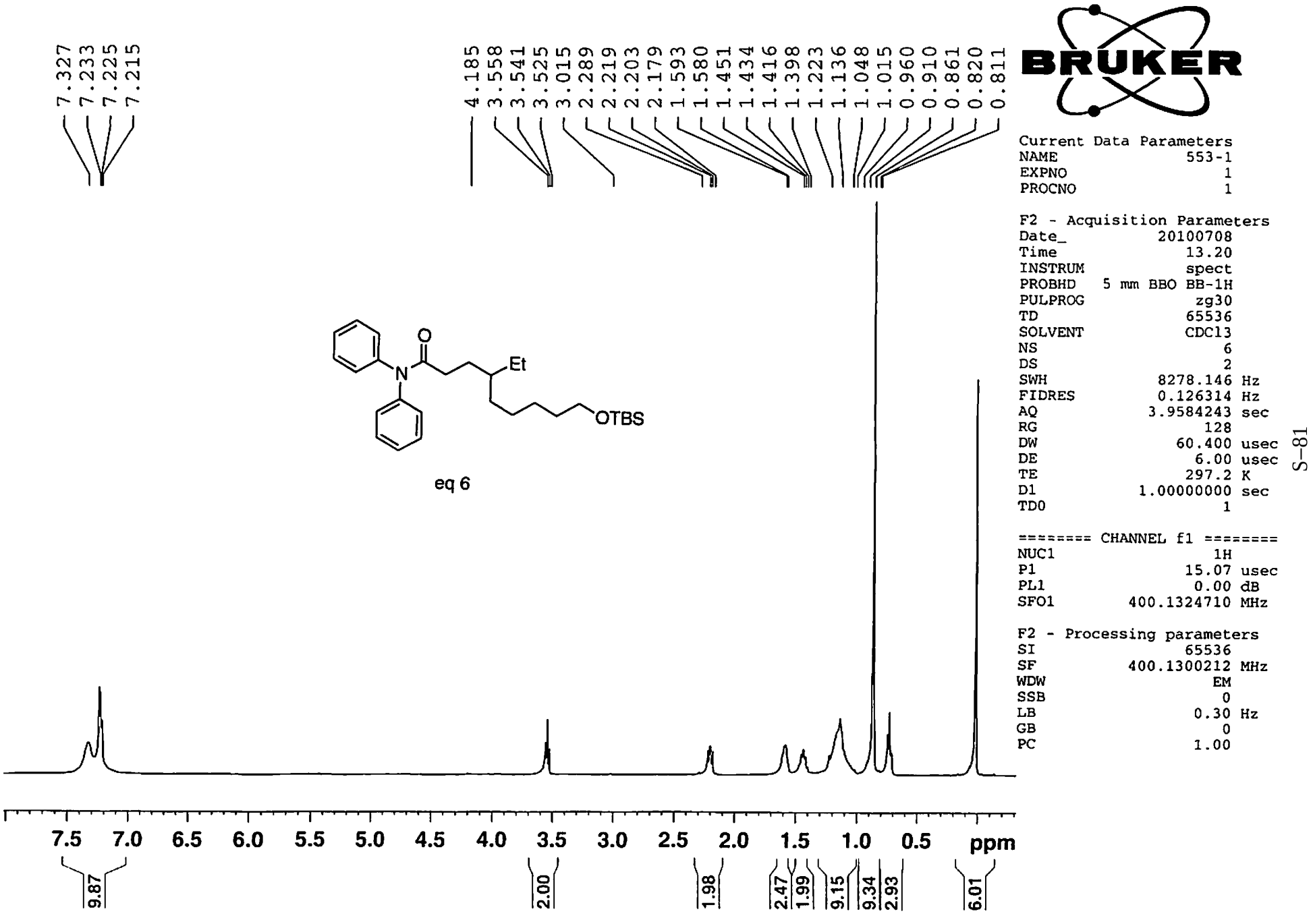
BRUKER

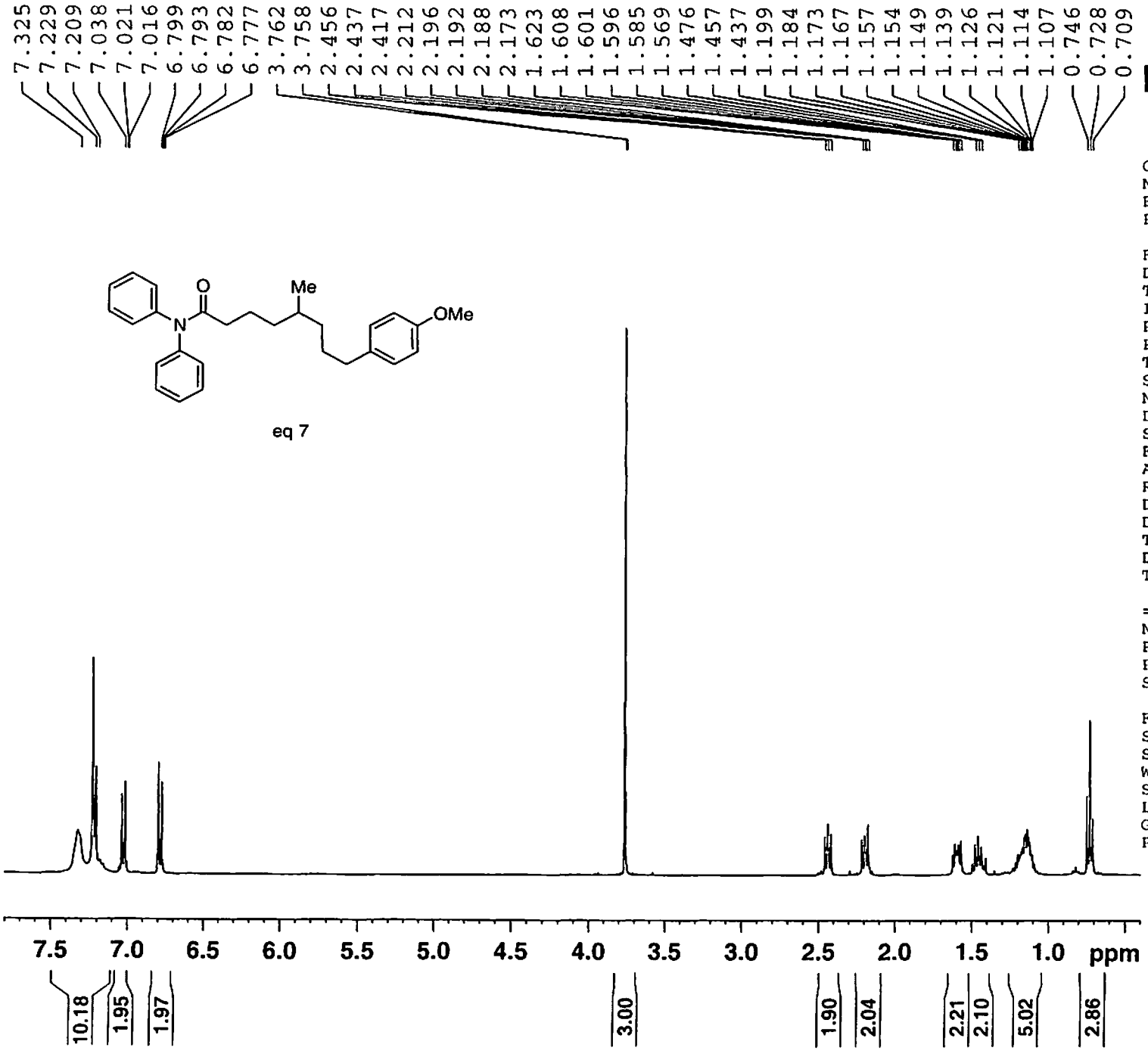
Current Data Parameters
NAME 630p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101009
Time 14.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


7.327
7.233
7.225
7.215
4.185
3.558
3.541
3.525
3.015
2.289
2.219
2.203
2.179
1.593
1.580
1.451
1.434
1.416
1.398
1.223
1.136
1.048
1.015
0.960
0.910
0.861
0.820
0.811
BRUKER
Current Data Parameters
NAME 553-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100708
Time 13.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.07 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300212 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
Et
OTBS
eq 6
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
9.87
2.00
1.98
2.47
1.99
9.15
9.34
2.93
6.01

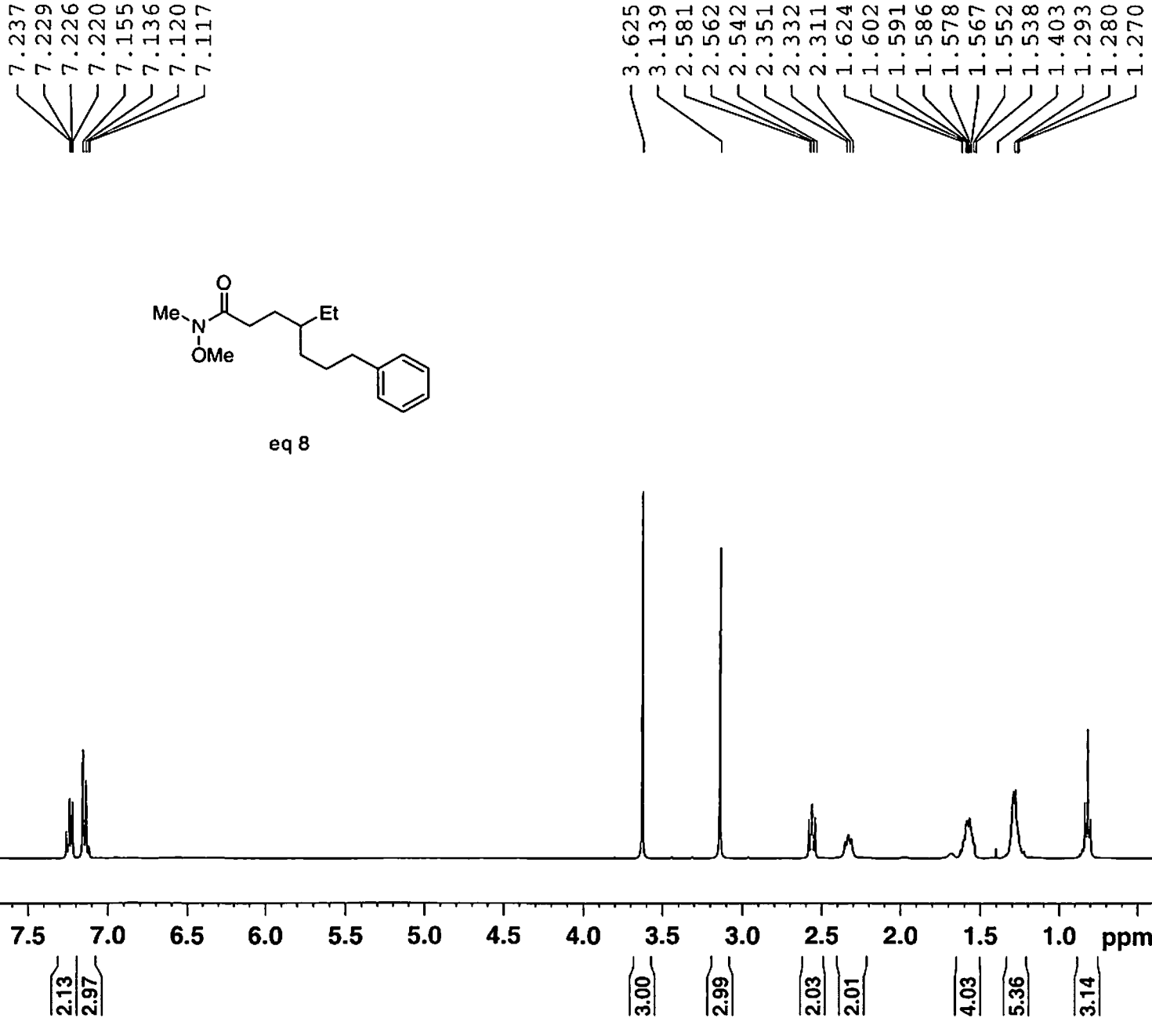


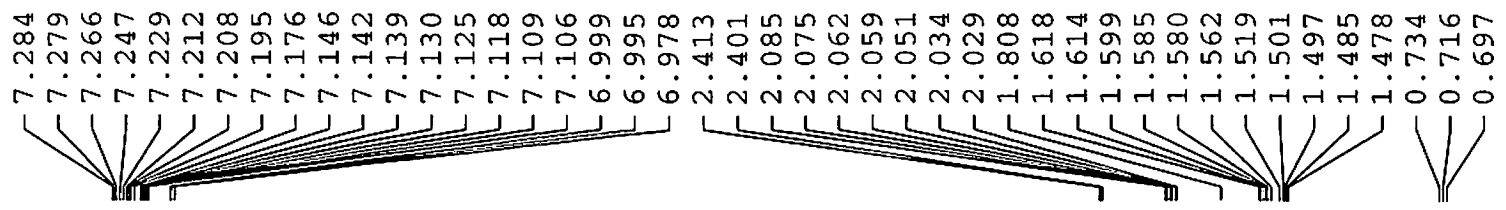

```
Current Data Parameters
NAME          589-1proton
EXPNO                   1
PROCNO                  1

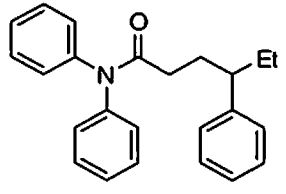
F2 - Acquisition Parameters
Date_           20110310
Time               14.31
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    10
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 287.4
DW                60.400 usec
DE                 6.00 usec
TE                683.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300220 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  1.00
```


7.237
7.229
7.226
7.220
7.155
7.136
7.120
7.117
3.625
3.139
2.581
2.562
2.542
2.351
2.332
2.311
1.624
1.602
1.591
1.586
1.578
1.567
1.552
1.538
1.403
1.293
1.280
1.270
BRUKER
Current Data Parameters
NAME 581-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100929
Time 18.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
OMe
O
Et
eq 8
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.13
2.97
3.00
2.99
2.03
2.01
4.03
5.36
3.14





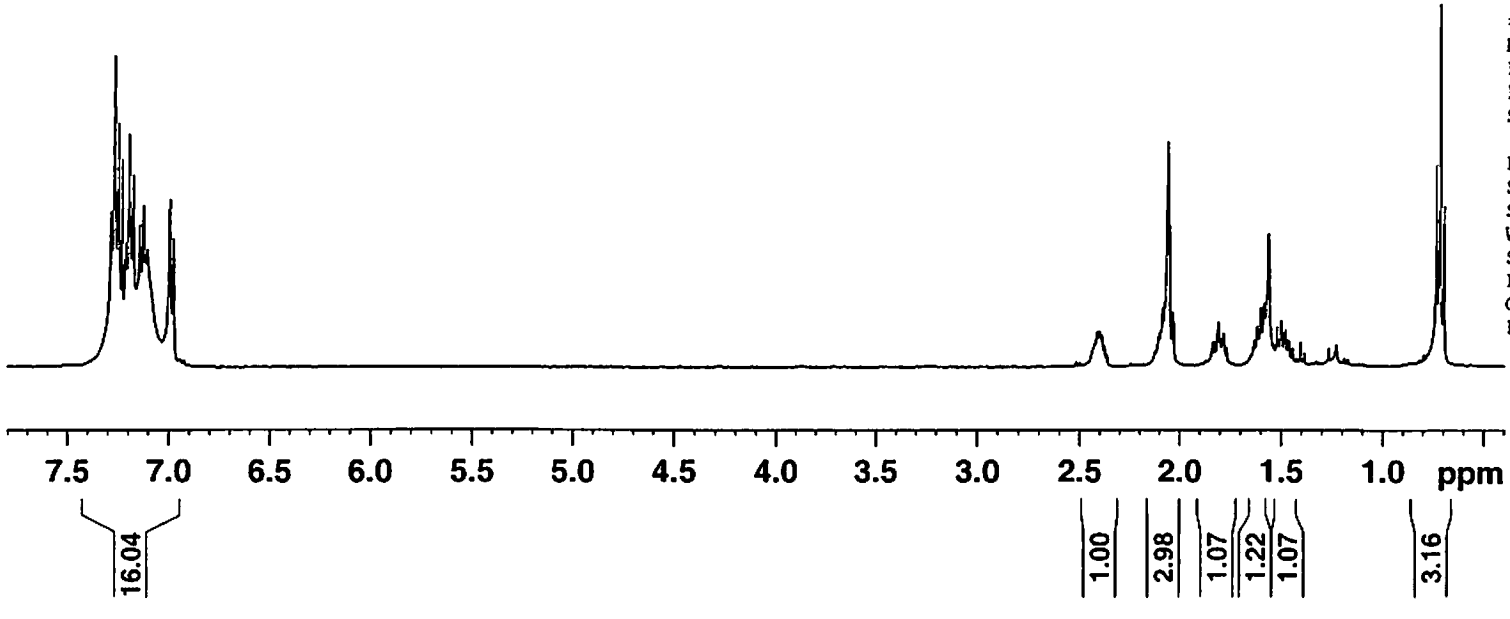
eq 9

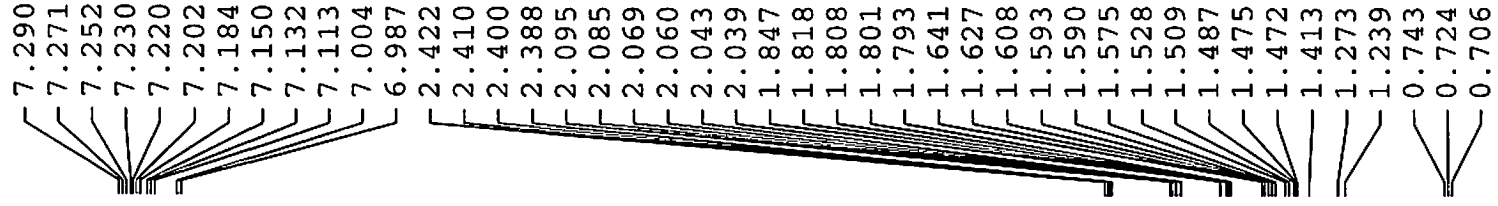
```
Current Data Parameters
NAME            852proton
EXPNO                   1
PROCNO                  1

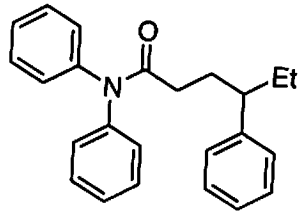
F2 - Acquisition Parameters
Date_            20110720
Time                17.53
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                  5160.6
DW                 60.400 usec
DE                  6.00 usec
TE                 683.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

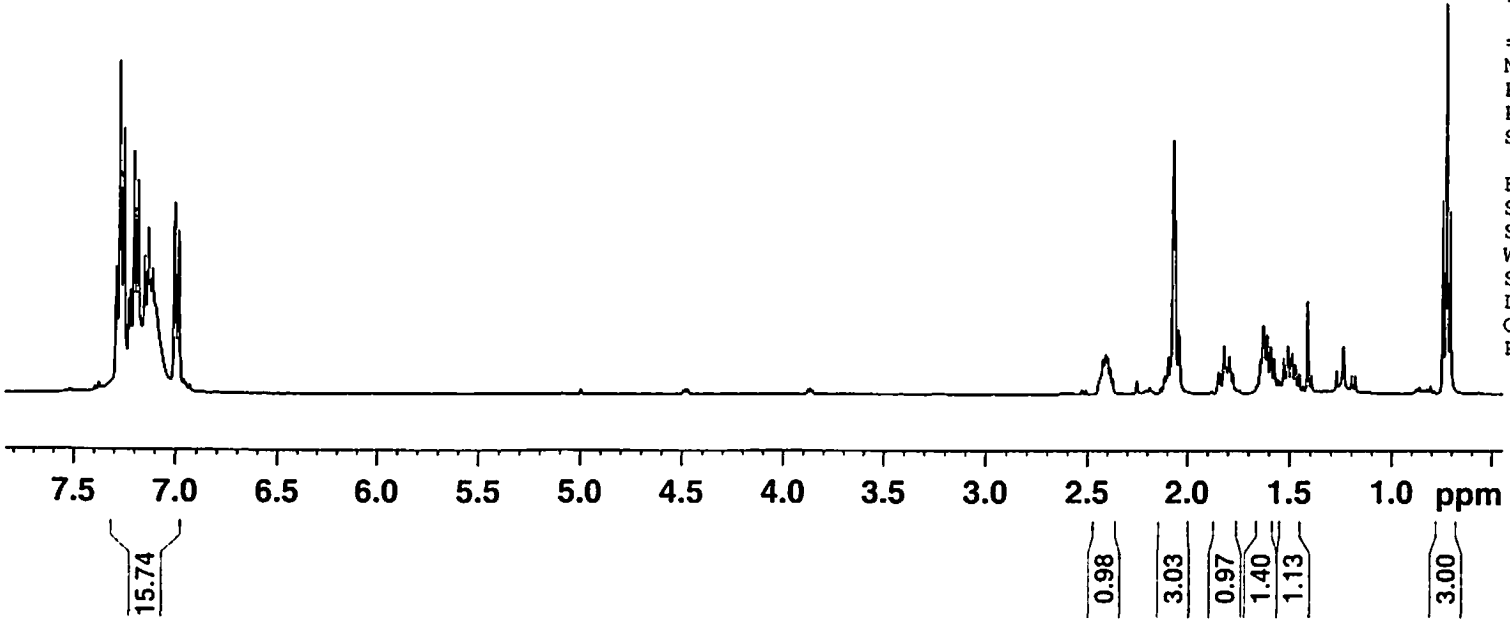
F2 - Processing parameters
SI                  65536
SF           400.1300220 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```







eq 10

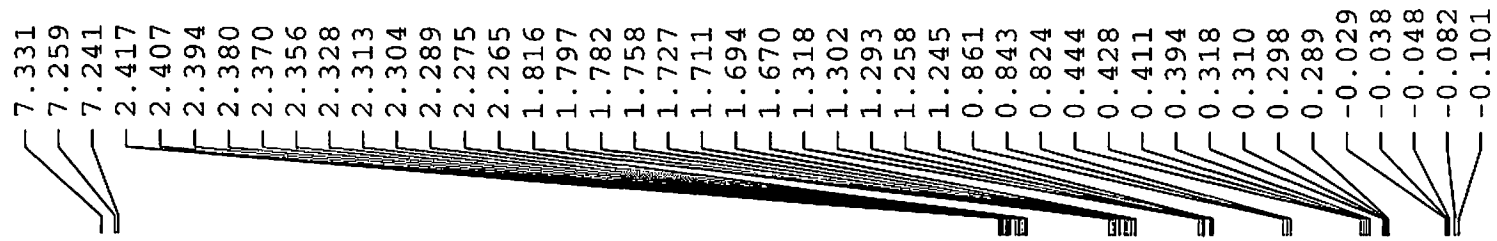


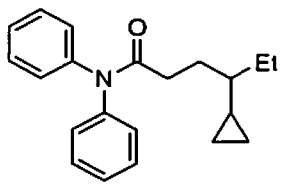
Current Data Parameters
NAME 858-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110822
Time 15.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 3251
DW 60.400 usec
DE 6.00 usec
TE 683.2 K
D1 1.00000000 sec
TD0 1

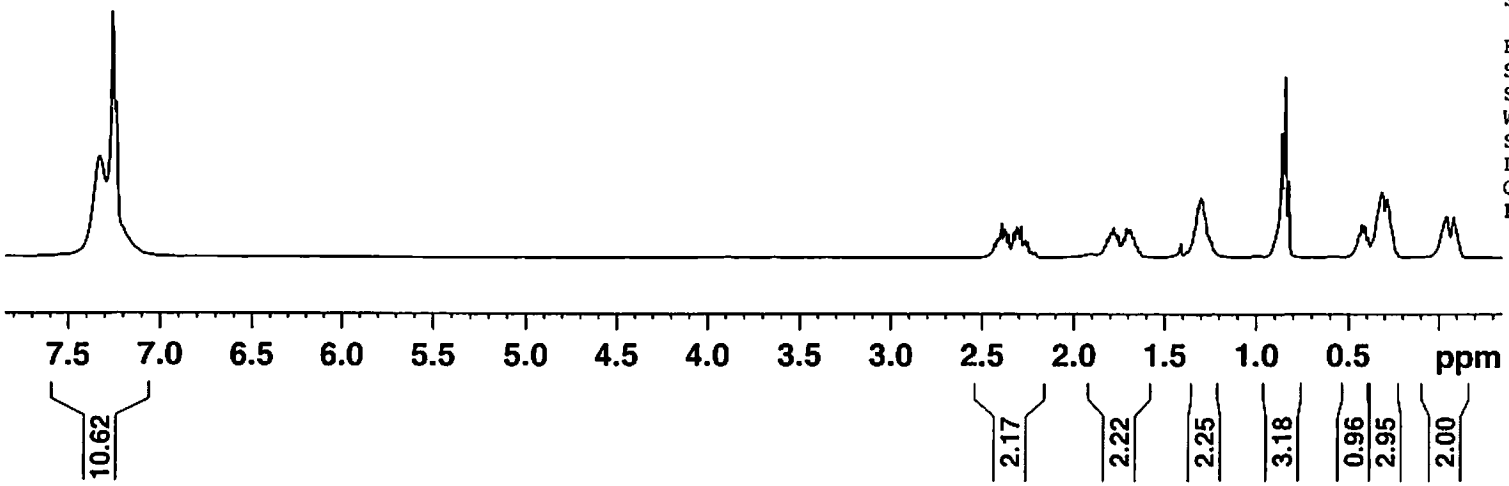
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





eq 11



BRUKER

```
Current Data Parameters
NAME          815-2proton
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20110530
Time               14.14
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                     4
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                   114
DW                60.400 usec
DE                 6.00 usec
TE                683.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300220 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```