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SUPPORTING INFORMATION

Selective Cobalt-Catalyzed Reduction of Terminal Alkenes and Alkynes Using $(EtO)_2Si(Me)H$ as a Stoichiometric Reductant

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General experimental. All air- and moisture sensitive manipulations were carried out using standard vacuum line and Schlenk techniques, or in a drybox containing a purified nitrogen or argon. Solvents were distilled from the appropriate drying agents under nitrogen. All glassware was cleaned using base (KOH, *i*PrOH) then acid (HClaq) baths. Analytical TLC was performed on E. Merck pre-coated (0.25 mm) silica gel 60 F254 plates. Flash column chromatography was carried out on silica gel 40 (Sorbtech Chemicals), Gas chromatographic analysis was conducted on an Agilent 7820A using hydrogen as the carrier gas, equipped with a methyl silicone column (30 m X 0.32 mm, 0.25 μm film thickness). Cobalt (II) chloride and phosphine ligands were purchased from Strem Chemicals Inc. All dienes used were synthesized in the laboratory. All silanes were purchased from Sigma Aldrich, Oakwood, Alfa Aesar and Apollo Scientific. All activating reagents were purchased from Sigma Aldrich. ^{1}H, ^{13}C NMR spectra were recorded on Bruker 400 and 600MHz, spectrometers. All spectra were obtained at ambient temperature. The chemical shifts (δ) were recorded in parts per million (ppm) and the coupling constants (*J*) in Hertz (Hz). ^{1}H and ^{13}C NMR multiplicity and coupling constants are reported where applicable. ^{1}H and ^{13}C spectra were referenced to the residual deuterated solvent peak ($CHCl_3$ 7.26 ppm, 77.32 ppm).

Ligand preparation. Bis1,6-(diaryliminoyl)pyridine ligands **5a-5d**, were prepared by a Schiff's base reaction using a modified literature methodology.[1] 2,6-Diaceylpyridine (5.00 g, 30.64 mmol) and *p*-tolunesulfonic acid (0.59 g, 3.07 mmol) were added to the 250 mL round bottom flask with a magnetic stirrer bar and toluene (150 mL) was added via syringe. Amine (9.11 g, 67.41 mmol) was added to the reaction mixture via syringe. The reaction mixture was stirred at reflux(120 ^{o}C) for 72 h under argon and water that formed was removed with a Dean-Starke trap. After 72 h reflux, the mixture was cooled to room temperature and the brown reaction mixture was washed with a solution of Na_2CO_3 and twice with water. The organic layer was separated and the combined aqueous layers were washed twice with diethyl ether. All organic layers were combined and dried with $MgSO_4$ and rotary evaporation of the resulting solution yielded a brown residue. The brown residue was crystallized from hot ethanol and was stored at -25 ^{o}C for overnight. The solid was isolated by vacuum filtration and washed several times with cold ethanol. The crystals were dried on vacuum pump for overnight. ^{1}H and ^{13}C NMR of ligands

matched what are the reported literature. Ligand **5e** was prepared according to reported literature.[2]

Synthesis of cobalt complexes: Modified literature methods were used for the preparation of complexes (*i*Pr-PDI)$CoCl_2$, (Et-PDI)$CoCl_2$, (Me-PDI)$CoCl_2$, (Cy-PDI)$CoCl_2$. Anhydrous $CoCl_2$ (48.5 mg, 0.37 mmol) and PDI ligand were taken in a flame dried 100 mL Schlenk flask and magnetic stir-bar was loaded inside the glove box with argon. The flask was removed from the glove box and purged with dry argon. Freshly distilled, degassed THF (13 mL) was added, and upon stirring at room temperature for 10 min, a brown solution was formed. The mixture was then stirred under argon for 72 h. After 72 h stirring at room temperature, freshly distilled diethyl ether (15 mL) and pentane (15 mL) were added via syringe. The solid was filtered in air and washed with cold pentane for several times. The brown solid complex was dried in pump for overnight and was used as is.

The bis-phosphine Co(II)-complexes were prepared as described earlier.[3] Anhydrous $CoCl_2$ (50.5 mg, 0.390 mmol) was added to a previously flame-dried 50-mL round two-necked bottom flask fitted with a flow control gas inlet and magnetic stir-bar loaded in a glove box under nitrogen. The nitrogen atmosphere was removed and the flask purged with dry argon. Freshly distilled, degassed THF (5 mL) was added, and upon stirring at room temperature for 15 min, a clear deep blue solution formed. A solution of diphenylphosphinopropane (DPPP, 181 mg, 0.410 mmol) in freshly distilled, degassed ether (5 mL) was added drop wise to yield a blue turbid solution. After stirring at room temperature for 15 h, 20 mL freshly distilled, degassed hexane was added in one portion to yield a blue precipitate. The resulting precipitate was filtered on a sintered glass fret under argon atmosphere, and washed with diethyl ether and hexane (1:1) mixture (3 X 5 mL) to remove any unreacted DPPP, resulting in quantitative yield of a light blue solid, which was used with no further purification.

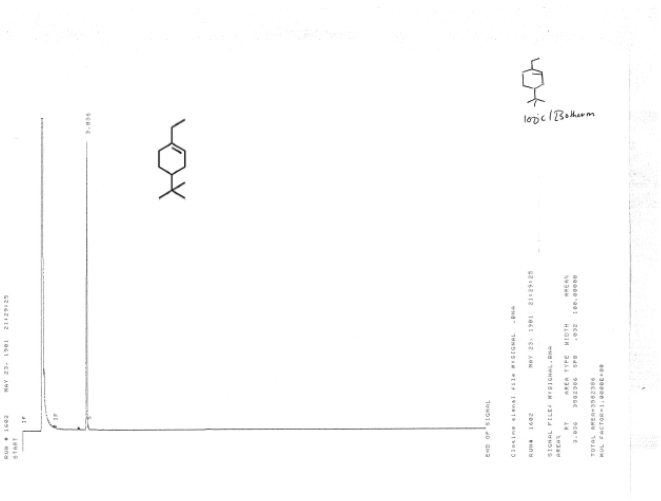
General procedure for cobalt-catalyzed reductions. An alkene (0.3 mmol) was added to a solution of cobalt (II) chloride PDI complex (9.2 mg, 0.015 mmol, 0. 05 equivalents) in anhydrous freshly distilled toluene (0.16 M) at room temperature under an atmosphere of argon. The flask was cooled at -78 ^{o}C (outside bath temperature), sodium triethyborohydride (0.03 mmol, 0.1 equivalents) in toluene was added, followed by a silane (0.33 mmol, 1.1 equivalents) and the reaction mixture was stirred at room temperature for 5 h. After completion the reaction,

it was filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yieldswere determined after purification by silica chromatography using hexane as eluent and removal of the solvent.

Procedure for reduction reaction under low catalyst loading (neat substrate). *Reduction of 4-phenyl-1-butene (with 0.1 mol% catalyst).* The alkene (4-phenyl-1-butene, 0.5 g, 3.79 mmol) was added to a solution of PDI-cobalt(II) chloride complex (2.3 mg, 0.003 mmol, 0. 001 equivalents) at room temperature under an atmosphere of argon. The flask was cooled at -78 ^{o}C, sodium triethyborohydride (0.007 mmol, 0.002 equivalents, 1 M in toluene) was added, followed by a silane (4.17 mmol, 1.1 equivalents, 0.56 mL). The flask was removed from the cold bath and placed in a oil bath preheated to 40 ^{o}C (~ in 2-3 min). The reaction mixture was stirred at 40 ^{o}C for 1 h. After completion (GC) of the reaction, it was filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yield (93%) was determined after purification by silica chromatography using hexane as eluent. The product was 98% by GC with the remaining <2% identified (GC-MS) as the isomerized 4-phenyl-2-butene, originally present in the starting material.

A parallel reaction done exactly as the previous one was evaporated to dryness and the white residue (~ 129 mg) was identified as higher molecular weight (up to MW 544) compounds by MALDI.

Reduction of 4-tert-butyl-1-vinylcyclohexene (0.1 mol% catalyst). The alkene (0.5 g, 3.05 mmol) was added to a solution of PDI-cobalt (II) chloride complex (2.0 mg, 0.003 mmol, 0. 001 equivalents) at room temperature under an atmosphere of argon. The flask was cooled at -78 ^{o}C, sodium triethyborohydride (0.006 mmol, 0.002 equivalents, 1 M in toluene) was added, followed by a



$(EtO)_2MeSiH$ (3.35 mmol, 0.45 mL, 1.1 equivalents). The flask was removed from the cold bath and placed in an oil bath preheated to 40 ^oC (~ in 2-3 min). The reaction mixture was stirred at 40 ^oC for 1 h. After completion the reaction (GC), it was filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yield (96%) was determined after purification by silica chromatography using hexane as eluent.

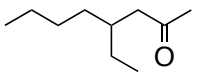
Cobalt-catalyzed reduction of dodecadiene at 50 psi H_2 (Eq 4). The diene (1.21 mmol) was added to a solution of cobalt (II) chloride PDI complex (36.8 mg, 0.06 mmol, 0. 05 equivalents) in anhydrous toluene (0.16 M) at -78 ^oC under an atmosphere of argon. Sodium triethyborohydride (0.12 mmol, 0.1 equivalents) was added to the reaction mixture. The reaction mixture was then transferred to the 20 mL white cap vial equipped with magnetic stir bar and the vial was placed inside a Fisher-Porter tube. The tube was sealed, evacuated and purged three times with hydrogen gas and then filled to 50 psi. A glass shield was placed in front of the tube and mixture was stirred for 5 h (monitor by GC, opening of the tube should be done very carefully releasing the pressure and the tube was sealed, evacuated and purged every single time). After 5 h, the solution was filtered through a short pad of silica with pentane to get the hydrogenated products(s) identified by NMR and GC MS.

Reduction of dodeca-1,3-diene at 1 atm of hydrogen using a H_2-filled balloon (Eq 4). A 50 mL Schlenk flask equipped with magnetic stirring bar was flame dried and purged with argon. The flask was charged with Cobalt (II) chloride PDI complex (36.8 mg, 0.06 mmol, 0. 05 equivalents). Anhydrous toluene (0.16 M) was added via syringe and the flask was cooled at -78 ^oC under an atmosphere of argon. Diene (1.21 mmol) followed by sodium triethyborohydride (0.12 mmol, 0.1 equivalents) was added to a solution at -78 ^oC. The flow control valve was closed to argon and hydrogen balloon was placed through the rubber septum. The flask was then removed from the bath and allowed to stirrer at room temperature for 5 h. The progress of reaction was monitored by GC. After 5 h, the solution was filtered through a short pad of silica with pentane to get the hydrogenated product.

Cobalt-catalyzed reduction of the silyl enol ether (32) under 50 psi H_2 (Scheme 2). The diene (0.08 mmol) was added to a solution of cobalt (II) chloride PDI complex (2.8 mg, 0.05

mmol, 0. 05 equivalents) in anhydrous toluene (0.16 M) at -78 ^{o}C under an atmosphere of argon. Sodium triethyborohydride (0.09 mmol, 0.1 equivalents) was added to the reaction mixture. The reaction mixture was then transferred to the 20 mL white cap vial equipped with magnetic stir bar through cannula transfer and the vial was placed inside a Fisher-Porter tube. The tube was sealed, evacuated and purged three times with hydrogen gas and then filled to 50 psi. A glass shield was placed in front of the tube and mixture was stirred for 5 h (monitor by GC, opening of the tube should be done very carefully releasing the pressure and the tube was sealed, evacuated and purged every single time). After 5 h, the solution was filtered through a short pad of silica with pentane to get the hydrogenated product, which was analyzed by GC and NMR.

Hydrogenation of 32 using Wilkinson's reagent and hydrogen. 4-Ethyl-2-octanone (19 from 32): To



a Fisher-Porter tube equipped with a magnetic stir bar was added 0.068 g (0.300 mmol) **23** and 0.0424 g (0.0458 mmol) Wilkinson's catalyst in 5 mL DCM. The tube was sealed, evacuated and purged three times with hydrogen gas, and then pressurized to 35 psi of hydrogen gas. The solution stirred at room temperature for 24 h. The gas was evacuated, the solution filtered through a short plug of silica gel to remove the catalyst and the solvent removed. The crude product was purified using 5% diethyl ether / 95% pentanes to get the product as a clear oil 0.044 g (94% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 2.32 (d, 2H, *J* = 6.8 Hz), 2.11 (s, 3H), 1.80 (quintet, 1H, *J* = 6.4 Hz), 1.20-1.32 (m, 8H), 0.81-0.89 (two triplets superimposed, 3H each).

^{13}C NMR (100 MHz, $CHCl_3$) δ 209.4, 48.4, 35.3, 33.1, 30.3, 29.7, 28.8, 26.3, 22.9, 14.0, 10.8.

GC-MS (*methyl silicone*): *m/z* 156.30 ([M+]). exact mass calculated for $C_{10}H_{20}O$ 156.27.

$[\alpha]_D^{23}$ (*c* = 0.264, $CHCl_3$) + 0.80 [from (*S,S*-BDPP)].

Cobalt-catalyzed reduction of alkyne 36a-d to alkene with 1 equivalents of $(EtO)_2SiMeH$ to produce 37a-d (Scheme 3). A 50 mL Schlenk flask equipped with magnetic stirring bar was flame dried and purged with argon. The flask was charged with cobalt (II) chloride PDI complex (16.6 mg, 0.03 mmol, 0. 01 equivalents). The flask was cooled at -78 ^{o}C under an atmosphere of argon. Alkyne (3.63 mmol) was added to the solution. Sodium triethyborohydride (0.07 mmol, 0.02 equivalents) and silane (3.99 mmol, 1.1 equivalents) was added to a solution at -78 ^{o}C. The reaction mixture was transferred to the pre heated oil bath (40 ^{o}C). The reaction mixture was stirred at 40 ^{o}C for 1h. The progress of reaction was monitored by

GC and GC-MS. The solution was then filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yield was determined after purification by silica chromatography using hexane as eluent. With 2 equivalents of silane the alkanes **38a-d** are produced in quantitative yield.

Cobalt-catalyzed reduction of alkyne 36a to alkane 38a with 2 equivalents of $(EtO)_2SiMeH$ (Scheme 4). A 50 mL Schlenk flask equipped with magnetic stirring bar was flame dried and purged with argon. The flask was charged with cobalt (II) chloride PDI complex (16.6 mg, 0.03 mmol, 0. 01 equivalents). The flask was cooled at -78 ^{o}C under an atmosphere of argon. Alkyne (3.63 mmol) was added to the solution. Sodium triethyborohydride (0.07 mmol, 0.02 equivalents) and silane (3.99 mmol, 1.1 equivalents) was added to a solution at -78 ^{o}C. The reaction mixture was transferred to the pre-heated oil bath (40 ^{o}C). The reaction mixture was stirred at 40 ^{o}C for 1h. The progress of reaction was monitored by GC and GC-MS, revealing only the alkene. After 1 h, additional silane (3.99 mmol, 1.1 equivalents) was added via syringe and the reaction mixture was further stirred at 40 ^{o}C for 90 minutes. The solution was then filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yield was determined after purification by silica chromatography using hexane as eluent.

Procedure for the attempted reduction in the presence of triethyl borane (Et_3B) and ($^{i\text{-}Pr}$PDI)Co(I)Cl. 3-Phenoxystyrene (0.26 mmol) was added to a solution of PDI-cobalt(I) chloride[4] complex (7.8 mg, 0.01 mmol, 0.05 equivalents) in anhydrous toluene (0.16 M) at room temperature under an atmosphere of argon. The flask was cooled at -78 ^{o}C, triethylborane (0.02 mmol, 0.1 equivalents, 1 M hexane) was added, followed by a $(EtO)_2MeSiH$ (0.30 mmol, 1.1 equivalents) and the reaction mixture was stirred at room temperature for 4 h. At the end of 12 h, it was filtered through the pad of silica by adding hexane and was concentrated under vacuum. No product was detected by GC.

Attempted reduction of alkene using $^{i\text{Pr}}$PDI-Co(I)Cl. 3-Phenoxystyrene (0.26 mmol) was added to a solution of PDI-cobalt(I) chloride complex (prepared by reduction of the Co(II)complex with Zn, 7.8 mg, 0.01 mmol, 0.05 equivalents)[4] in anhydrous toluene (0.16 M) at room temperature under an atmosphere of argon. The flask was cooled at -78 ^{o}C, sodium

triethyborohydride (0.01 mmol, 0.05 equivalents) was added, followed by a $(EtO)_2MeSiH$ (0.30 mmol, 1.1 equivalents) and the reaction mixture was stirred at room temperature for 4 h. After completion the reaction, it was filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yield (96%) was determined after purification by silica chromatography using hexane as eluent. Purity was ascertained by GC and ^1H NMR.

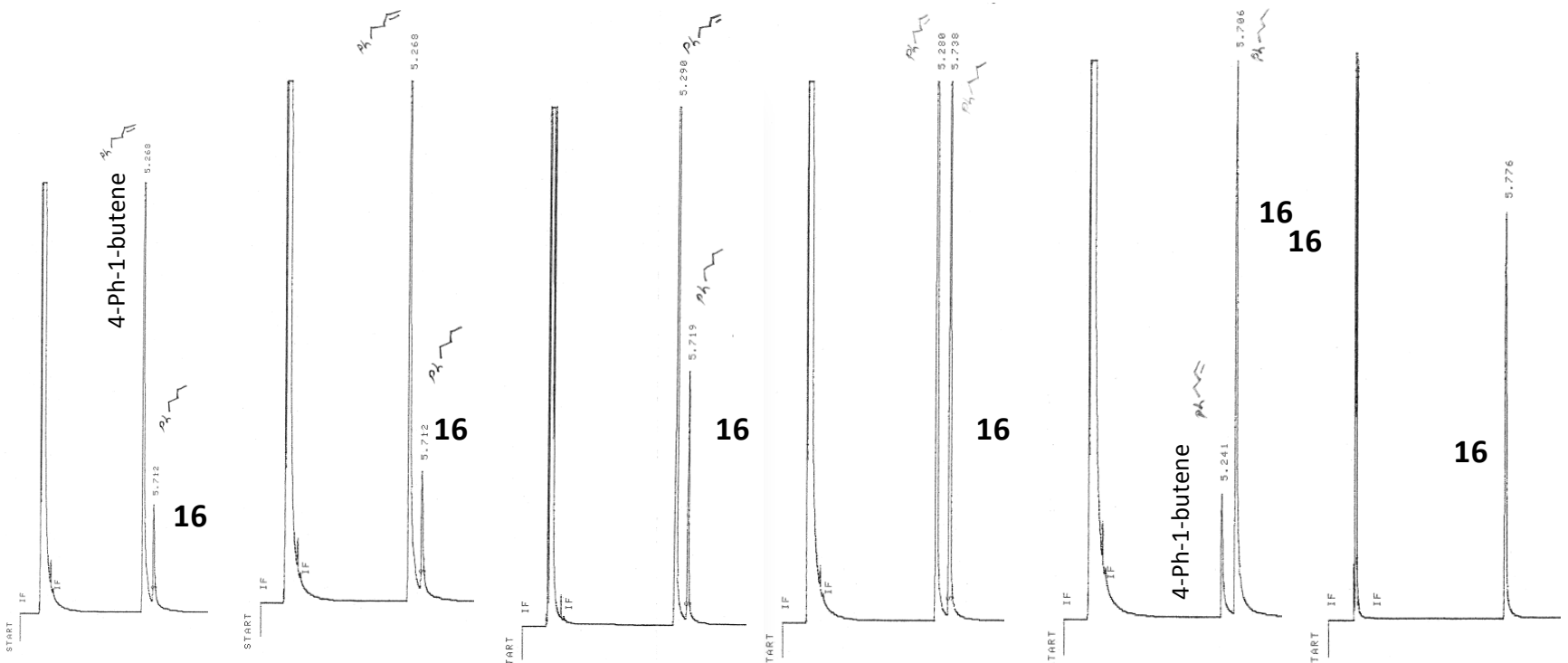
Procedure for deuterium labeling studies. An alkene (0.26 mmol) was added to a solution of cobalt (II) chloride PDI complex (7.8 mg, 0.01 mmol, 0. 05 equivalents) in anhydrous deuterated toluene (0.16 M) at room temperature under an atmosphere of argon. The flask was cooled at -78 ^oC, sodium triethyborohydride (0.03 mmol, 0.1 equivalents, 1 M solution in toluene) was added, followed by $(EtO)_2MeSiH$ (0.30 mmol, 1.1 equivalents) and the reaction mixture was stirred at room temperature for 5 h. After completion the reaction, it was filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yield (97%) was determined after purification by silica chromatography using hexane as eluent. There was no D-incorporation as judged by NMR and mass spectrometry.

A similar experiment was conducted in THF-d8. The converted product (~ 34%) was isolated and analyzed by GC and GCMS and showed similar results.

Cobalt-catalyzed reduction of 3-phenoxystyrene in the presence of D_2O. A 50 mL Schlenk flask equipped with magnetic stirring bar was flame dried and purged with argon. The flask was charged with cobalt (II) chloride PDI complex (3.2 mg, 0.005 mmol, 0. 01 equivalents). The flask was cooled at -78 ^oC under an atmosphere of argon. Alkene (0.51 mmol) was added to the solution. Sodium triethyborohydride (0.01 mmol, 0.02 equivalents) and silane (0.56 mmol, 1.1 equivalents) was added to a solution at -78 ^oC. D_2O (0.51 mmol, 1 equivalents) was finally added and the reaction mixture was transferred to the pre-heated oil bath (40 ^oC). The reaction mixture was stirred at 40 ^oC for 1h. After 1 h, it was filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yield (98%) was determined after purification by silica chromatography using hexane as eluent. There was no D-incorporation as judged by NMR and mass spectrometry.

Reduction of 3-phenoxystyrene in the presence of 4 Å molecular sieves. A 50 mL Schlenk flask equipped with magnetic stirring bar was flame dried and purged with argon. The flask was charged with cobalt (II) chloride PDI complex (3.2 mg, 0.005 mmol, 0. 01 equivalents) and 4 Å MS. The flask was cooled at -78 ^{o}C under an atmosphere of argon. Alkene (0.51 mmol) was added to the solution. Sodium triethyborohydride (0.01 mmol, 0.02 equivalents) and silane (0.56 mmol, 1.1 equivalents) was added to a solution at -78 ^{o}C. The reaction mixture was transferred to the pre heated oil bath (40 ^{o}C). The reaction mixture was stirred at 40 ^{o}C for 1h. After 1 h, it was filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yield (97%) was determined after purification by silica chromatography using hexane as eluent.

Reduction of 4-phenyl-1-butene using various equivalents of silanes (Eq 5). Six 50 mL Schlenk flask equipped with magnetic stirring bar were flame dried and purged with argon. Each flask was charged with cobalt (II) chloride PDI complex (9.3 mg, 0.01 mmol, 0. 01 equivalents). The flask was cooled at -78 ^{o}C under an atmosphere of argon. Alkene (1.51 mmol) was added to the each solution. Sodium triethyborohydride (0.03 mmol, 0.02 equivalents) and the silane (0.15 mmol for 10 mol%, 0.30 mmol for 20 mol%, 0.60 mmol for 40 mol%, 0.90 mmol for 60 mol%, 1.20 mmol for 80 mol% and 1.66 mmol for 0.11 equivalents) was added to each solution at -78 ^{o}C. The reaction mixtures were transferred to the pre heated oil bath (40 ^{o}C). The reaction mixtures were stirred at 40 ^{o}C for 1h. The progress of reaction was monitored by GC and GC-MS. The reaction were quenched and the product mixture in each case was then filtered through the pad of silica by adding hexane and was concentrated under vacuum. Isolated yield was determined after purification by silica chromatography using hexane as eluent. The product(s) was analyzed by GC (see the attached chromatograms).

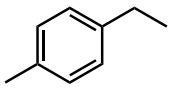


(0.1, 11) (0.2, 22) (0.4, 43) (0.6, 64) (0.8, 83) (1.1, >99)

[in bracket: (silane equivalent, % conversion to the product **16**), see Eq 5]

^{1}H and ^{13}C NMR, GC Retention Times of Reduction Products

1-ethyl-4-methylbenzene (10a)



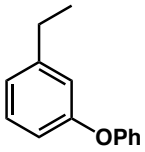
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.23 (s, 4H), 2.75 (q, $^{3}J_{H,\ H}$ = 7.6 Hz, 2H), 2.46 (s, 3H), 1.36 (t, $^{3}J_{H,\ H}$ = 7.6 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 141.3, 135.0, 129.1, 127.8, 28.5, 21.0, 15.8.

GC (methyl silicone column, 80 ^{0} C/ Isotherm) RT for product = 3.55 min

GC-MS (methyl silicone): m/z ([M+) 120.10; exact mass calculated for C_9H_{12} = 120.09

1-ethyl-3-phenoxybenzene (10b)



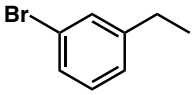
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.31 (m, 2H), 7.24 (t, ^{3}J H, H = 7.6 Hz, 1H), 7.11-7.07 (m, 1H), 7.03-7.00 (m, 2H), 6.96-6.94 (m, 1H), 6.88-6.87 (m, 1H), 6.83-6.80 (m, 1H), 2.63 (q, ^{3}J H, H = 7.6 Hz, 2H), 1.23 (t, ^{3}J H, H = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 157.8, 146.9, 130.3, 130.1, 123.6, 123.4, 119.4, 119.1, 116.7, 29.4, 15.9.

GC (methyl silicone column, 140 ^{0} C/) RT for product = 5.88 min

GC-MS (methyl silicone): m/z ([M+) 198.12; exact mass calculated for $C_{14}H_{14}O$ 198.10

1-bromo-3-ethylbenzene (10c)



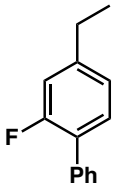
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.29 (m, 2H), 7.16-7.11 (m, 2H), 2.62(q, ^{3}J H, H = 7.6 Hz, 2H), 1.23 (t, ^{3}J H, H = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 131.2, 130.1, 128.9, 126.7, 122.5, 24.8, 15.9.

GC (methyl silicone column, 100 ^{0} C/) RT for product = 3.52 min

GC-MS (methyl silicone): m/z ([M+) 184.12; exact mass calculated for C_8H_9Br 183.99

4-ethyl-2-fluoro-1, 1'-biphenyl (11)



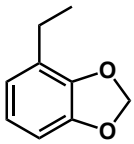
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57-7.55 (m, 2H), 7.46-7.42 (m, 2H), 7.38-7.34 (m, 2H), 7.07-6.99 (m, 2H), 2.70 (q, ^{3}J H, H = 7.6 Hz, 2H), 1.29 (t, ^{3}J H, H = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 161.2, 158.8, 146.1, 136.2, 130.7, 129.2, 128.6, 127.6, 124.0, 115.4, 28.6, 15.4.

GC (methyl silicone column, 140 ^{0} C/ Isotherm) RT for product = 9.28 min

GC-MS (methyl silicone): m/z ([M+) 200.12; exact mass calculated for $C_{14}H_{13}F$ 200.10

5-ethylbenzo[d][1,3]dioxole (12)



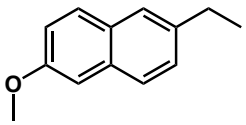
^{1}H NMR (600 MHz, $CDCl_3$) δ 6.74- 6.72 (m, 1H), 6.70-6.69 (m, 1H), 6.65-6.63 (m, 1H), 5.92 (s, 2H), 2.57 (q, ^{3}J, H, H = 7.6 Hz, 2H), 1.20 (t, ^{3}J, H, H = 7.6 Hz, 3H),

^{13}C NMR (150 MHz, $CDCl_3$) 147.6, 145.5, 138.3, 120.5, 108.5, 108.2, 100.8, 28.7, 16.1.

GC (methyl silicone column, 100 ^{0} C/ Isotherm) RT for product = 6.24 min

GC-MS (methyl silicone): m/z ([M+) 150.10; exact mass calculated for $C_9H_{10}O_2$ = 150.07

2-ethyl-6-methoxynaphthalene (13)



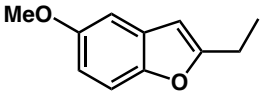
^{1}H (400 MHz, $CDCl_3$) δ 7.69-7.56 (m, 3H), 7.33-7.31 (m, 1H), 7.14-7.11 (m, 2H), 3.91 (s, 3H), 2.79 (q, ^{3}J H, H= 7.6 Hz, 2H), 1.32 (t, ^{3}J H, H= 7.6 Hz, 3H).

^{13}C (100 MHz, $CDCl_3$) δ 157.3, 139.6, 133.1, 129.4, 129.1, 127.7, 126.9, 125.6, 118.8, 105.9, 55.5, 29.0, 15.8.

GC (methyl silicone column, 140 ^{0} C/Isotherm) RT for product = 7.48 min

GC-MS (methyl silicone): m/z ([M+) 186.12; exact mass calculated for $C_{13}H_{14}O$ 186.10

2-ethyl-5-methoxybenzofuran (14)



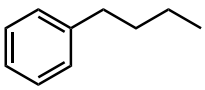
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.27 (d, 1H), 6.96-6.95 (d, 1H), 6.81-6.78 (dd, 1H), 6.31-6.30 (d, 1H), 3.82 (s, 3H), 2.80-2.74 (m, 2H), 1.31 (t, $^{3}J_{H,H}$ = 7.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0, 155.8, 149.7, 129.7, 111.4, 111.1, 103.3, 101.3, 65.9, 56.0, 22..0, 12.0.

GC (*methyl silicone column*, 120 ^{0} C/ Isotherm) RT for product = 7.85 min

GC-MS (*methyl silicone*): *m/z* ([M+) 176.10; exact mass calculated for $C_{11}H_{12}O_2$ 176.08

Butylbenzene (16)



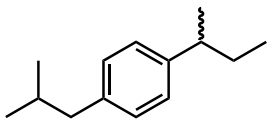
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.19 (m, 3H), 7.15 – 7.12 (m, 2H), 2.58 (t, ^{3}J H, H = 7.7 Hz, 2H), 1.61-1.54 (m, 2H), 1.37-1.28 (m, 2H), 0.90 (t, ^{3}JH, H = 7.3 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ143.0, 128.6, 128.4, 125.7, 35.8, 33.8, 22.5, 14.1.

GC (methyl silicone column, 80 ^{0} C/ Isotherm) RT for product = 3.27 min

GC-MS (methyl silicone): m/z ([M+) 134.10; exact mass calculated for $C_{10}H_{14}$ 134.11

1-(*iso*-butyl)-4-isobutylbenzene (17b)



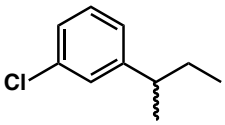
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14-7.07 (m, 4H), 2.64-2.55 (m, 1H), 2.47 (d, $^{3}J_{H,H}$ = 7.5 Hz, 2H), 1.93-1.83 (m, 1H), 1.65-1.57 (m, 2H), 1.26 (d, $^{3}J_{H,H}$ = 6.9 Hz, 3H), 0.93 (d, $^{3}J_{H,H}$ = 6.6 Hz, 6H), 0.85 (t, $^{3}J_{H,H}$ = 7.4 Hz, 3H)

^{13}C NMR (100 MHz, $CDCl_3$) δ 145.1, 139.2, 129.2, 127.0, 45.4, 41.5, 31.5, 30.5, 22.7, 22.1, 12.5.

GC (*methyl silicone column*, 100 ^{0} C/Isotherm) RT for product = 7.15 min

GC-MS (*methyl silicone*): *m/z* ([M+) 190.10; exact mass calculated for $C_{14}H_{22}$ 190.17

1-(*sec*-butyl)-3-chlorobenzene (17a)



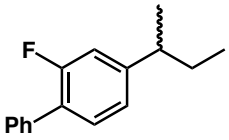
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 - 7.14 (m, 4H), 2.62 – 2.53 (m, 1H), 1.62-1.55 (m, 2H), 1.30 (d, $^{3}J_{H,H}$ = 6.9 Hz, 3H), 0.90 (t, $^{3}J_{H,H}$ = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 149.9, 134.2, 129.6, 127.4, 126.1, 125.5, 41.7, 31.2, 21.8, 12.3.

GC (*methyl silicone column*, 100 ^{0} C/Isotherm) RT for product = 3.52 min

GC-MS (*methyl silicone*): *m/z* ([M+) 168.10; exact mass calculated for $C_{10}H_{13}Cl$ 168.07

4-(*sec*-butyl)-2-fluoro-1, 1'-biphenyl (18)



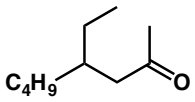
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 - 7.57 (m, 2H), 7.48-7.44 (m, 2H), 7.40-7.35 (m, 2H), 7.07 - 6.99 (m, 2H), 2.71 – 2.62 (m, 1H), 1.69-1.62 (m, 2H), 1.30 (d, $^{3}J_{H,\,H}$ = 6.9 Hz, 3H), 0.90 (t, $^{3}J_{H,\,H}$ = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 158.6, 149.6, 136.1, 130.5, 129.0, 128.5, 127.4, 123.2, 114.4, 41.4, 31.1, 21.7, 12.3.

GC (*methyl silicone column*, 180 ^{0} C/Isotherm) RT for product = 3.14 min

GC-MS (*methyl silicone*): *m/z* ([M+) 228.10; exact mass calculated for $C_{16}H_{17}F$ 228.13

4-ethyloctan-2-one (19)



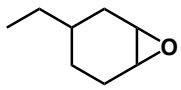
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.32 (d, J = 6.8 Hz, 2H), 2.11 (s, 3H), 1.80(q, J = 6.4 Hz, 1H), 1.32 – 1.20 (m, 8H), 0.89 – 0.81(two triplets superimposed, 3H each).

^{13}C NMR (100 MHz, $CDCl_3$) δ 209.4, 48.4, 35.3, 33.1, 30.3, 29.7, 28.8, 26.3, 22.9, 14.0, 10.8.

GC (*methyl silicone column*, 100 ^{0} C/Isotherm) RT for product = 2.56 min

GC-MS (methyl silicone): m/z 156.30 ([M+]). exact mass calculated for $C_{10}H_{20}O$ 156.27.

(*cis, trans*)-3-ethyl-7-oxabicyclo[4.1.0]heptane (23)



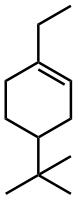
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.79- 3.69 (m, 4H), 1.19-1.1.42 (m, 16H), 0.83- 0.78 (m, 2H), 0.12- 0.04 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 36.3, 34.8, 34.7, 31.8, 29.2, 27.1, 25.5, 22.8, 21.6, 20.9, 18.9, 14.2, 11.6.

GC (methyl silicone column, 100 ^{0} C/ Isotherm) RT for product = 4.01 min and 4.06 min in the ratio of 59: 41

GC-MS (methyl silicone): m/z ([M+) 126.19; exact mass calculated for $C_8H_{14}O$ = 126.10

4-(*tert*-butyl)-1-ethylcyclohex-1-ene (25a)



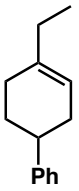
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.39-5.38 (m, 1H), 2.04 – 1.92 (m, 4H,), 1.83-1.72(m, 2H), 1.32-1.26 (m, 1H), 1.23-1.11 (m, 2H), 0.98 (t, ^{3} JH, H = 7.5 Hz, 3H), 0.86 (s, 9H)

^{13}C NMR (100 MHz, $CDCl_3$) δ 139.6, 119.8, 44.6, 32.5, 30.4, 30.1, 27.5, 27.0, 24.6, 12.7.

GC (*methyl silicone column*, 100 ^{0} C/ Isotherm) RT for product = 3.56 min

GC-MS (*methyl silicone*): *m/z* ([M+) 166.17; exact mass calculated for $C_{12}H_{22}$ 166.10

4-ethyl-1, 2, 3, 6-tetrahydro-1, 1'-biphenyl (25b)



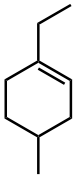
^{1}H (400 MHz, $CDCl_3$) δ 7.31-7.27 (m, 2H), 7.23-7.16 (m, 3H), 5.48-5.47 (m, 1H), 2.78 – 2.70 (m, 1H,), 2.19-1.92 (m, 6H), 1.81-1.70 (m, 2H), 1.02 (t, ^{3} JH, H = 7.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 139.5, 128.4, 127.0, 125.9, 119.1, 53.5, 40.4, 33.6, 30.3, 29.1, 12.4.

GC (*methyl silicone column*, 100^{o}C/ 10 min, rate = 20 ^{o}C, 250 ^{o}C = 40 min) RT for product = 12.97 min

GC-MS (*methyl silicone*): *m/z* ([M+) 186.10; exact mass calculated for $C_{14}H_{18}$ 186.14

1-ethyl-4-methylcyclohex-1-ene (25c)



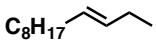
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.32-5.31 (m, 1H), 2.05-1.88(m, 6H), 1.68-1.63 (m, 1H), 1.60-1.54 (m, 2H), 0.95 (t, ^{3}J H, H= 7.4 Hz, 6H), 0.91(d, ^{3}J H, H= 6.3 Hz, 3H)

^{13}C NMR (100 MHz, $CDCl_3$) δ 139.3, 119.0, 34.0, 31.5, 30.4, 29.8, 28.7, 28.5, 12.5

GC (*methyl silicone column*, 40 ^{0} C/ Isotherm) RT for product = 4.97 min

GC-MS (*methyl silicone*): *m/z* ([M+) 124.13; exact mass calculated for C_9H_{16} 124.10

(*E*)-dodec-3-ene (27a)



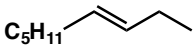
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.47-5.35 (m, 2H), 2.03-1.94(m, 4H), 1.34-1.27 (m, 12 H), 0.98-0.94 (m, 3H), 0.90-0.84(m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 132.1, 129.7, 32.9, 32.2, 30.0, 29.8, 29.6, 29.5, 25.9, 23.0, 14.4, 14.3.

GC (*methyl silicone column*, 100^{o}C/Isotherm) RT for product = 3.66 min

GC-MS (*methyl silicone*): *m/z* ([M+) 168.10; exact mass calculated for $C_{12}H_{24}$ 168.16

(*E*)-non-3-ene (27b)



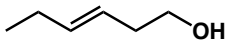
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.46-5.35 (m, 2H), 2.02-1.94(m, 4H), 1.34-1.27 (m, 6 H), 0.98-0.86(m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 131.8, 129.3, 32.5, 31.9, 29.7, 29.3, 25.5, 22.6, 13.9.

GC (*methyl silicone column*, 50 ^{o} C/ Isotherm) RT for product = 2.97 min.

GC-MS (*methyl silicone*): *m/z* ([M+) 126.05; exact mass calculated for C_9H_{18} 126.14

(*E*)-hex-3-en-1-ol (27c)



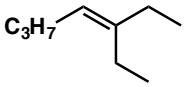
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.53- 5.46 (m, 1H), 5.33- 5.26 (m, 1H) 3.58 (t, $^{3}J_{H, H}$ = 6.64Hz, 2H), 2.30-2.25 (m, 2H), 2.22 (s, 1H), 2.07- 2.00 (m, 2H), 0.93 (t, $^{3}J_{H, H}$ = 7.54Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 134.9, 124.7, 62.4, 30.9, 20.8, 14.5.

GC (methyl silicone column, 140 ^{0} C/ Isotherm) RT for product = 3.99 min

GC-MS (methyl silicone): m/z ([M+) 100.10; exact mass calculated for $C_6H_{12}O$ = 100.09

3-ethylhept-3-ene (29)



^{1}H NMR (400 MHz, $CDCl_3$) δ 5.08 (t, ^{3}J H, H= 7.1 Hz, 1H), 2.09-1.95(m, 6H), 1.43-1.29(m, 2H), 0.95 (t, ^{3}J H, H= 7.4 Hz, 6H), 0.91(t, ^{3}J H, H= 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.1, 123.0, 31.9, 30.0, 29.9, 23.0, 22.9, 14.4.

GC (*methyl silicone column*, 50 ^{0} C/Isotherm) RT for product = 3.27 min

GC-MS (*methyl silicone*): *m/z* ([M+) 220.10; exact mass calculated for C_9H_{18} 126.14

(2*S*,4*S*,*E*)-2-ethyl-1-ethylidene-4-isopropylcyclohexane and (2*S*,4*R*,*E*)-2-ethyl-1-ethylidene-4-isopropylcyclohexane (30)

The ^{1}H NMR and ^{13}C NMR spectra of the diastereomers were recorded in a mixture.

^{1}H NMR (600 MHz, $CDCl_3$) δ 5.19-5.23 (m, 1H each), 2.62-2.65 (m, 1H, ent-*trans*-), 2.35-2.40 (m, 1H,, 2.13-2.20 (m, 1H each), 1.99 (dt, 1H, *J* = 3.2 Hz, 13.6 Hz, one diastereomer), 1.91 (ddd, 1H, *J* = 2.7 Hz, 8.0 Hz, 13.3 Hz, one diastereomer), 1.66-1.74 (m, 2H, both diastereomers merged), 1.56-1.58 (m, 6H, both diastereomers merged), 1.52-1.54 (m, 1H, one diastereomer), 1.49-1.52 (m, 1H, one diastereomer), 1.44-1.48 (m, 2H, both diastereomers merged), 1.34-1.42 (m, 3H, both diastereomers merged), 1.28-1.33 (m, 3H, both diastereomers merged), 1.10-1.18 (m, 2H, both diastereomers merged), 0.94-1.03 (m, 2H, both diastereomers merged), 0.89 (q, 3H each, *J* = 7.5 Hz, both diastereomer merged), 0.82-0.86 (m, 12H, both diastereomers merged).

^{13}C NMR (150 MHz, $CDCl_3$) δ 142.87, 142.32, 116.95, 116.16, 39.82, 39.25, 38.26, 37.21, 34.88, 32.81, 32.74, 32.38, 32.33, 31.59, 30.54, 28.12, 25.37, 22.33, 20.18, 19.88, 19.70, 14.05, 13.15, 12.76, 12.21, 11.96.

GC (*cyclodex-B*, 110 ^{o}C): See attached GC spectra for diastereomeric ratio and *ee.*

R_t of reduced product from HV-product using Co(dppp)Cl_2 ligand: 50.18 min and 48.35 min

R_t reduced product of H- product using Co[(*S,S*)-BDPP]Cl_2 complex : 50.16 min and 48.01 min.

(*Z*)-4-ethylnon-2-ene (31)

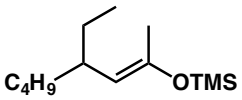
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.59-5.55 (m, 1H), 5.18 - 512 (m, 1H), 2.38 – 2.21(m, 1H), 1.70 – 1.67 (m, 3H), 1.47 – 1.22 (m, 10H), 1.07 – 0.94 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 136.2, 123.7, 38.8, 35.9, 32.4, 31.9, 28.9, 27.3, 22.9, 14.4, 12.0.

GC (*methyl silicone column*, 50 ^{0} C/Isotherm) RT for product = 6.42 min

GC-MS (*methyl silicone*): *m/z* ([M+) 154.10; exact mass calculated for $C_{11}H_{22}$ 154.17

(*E*)-((4-ethyloct-2-en-2-yl)oxy)trimethylsilane (33)



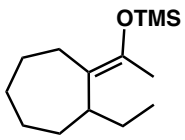
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.35 (dq, ^{3}J H, H = 0.8 Hz, 10.1 Hz, 1H), 1.95 – 1.84 (m, 1H), 1.72 (d, ^{3}J H, H= 0.9 Hz, 3H), 1.44 – 1.21 (m, 5H), 1.18-1.08 (m, 3H), 0.90-0.82 (m, 6H), 0.18 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.4, 114.2, 39.7, 36.1, 29.7, 29.4, 22.9, 18.3, 14.1, 11.9, 0.3.

GC (methyl silicone column, 100 ^{0} C/ Isotherm) RT for product = 4.56 min

GC-MS (methyl silicone): m/z ([M+) 228.10; exact mass calculated for $C_{10}H_{12}$ 228.19

(*E*)-(1-(2-ethylcycloheptylidene)ethoxy)trimethylsilane (35)



^{1}H NMR (400 MHz, $CDCl_3$) δ 2.53-2.48 (m, 1H), 2.30-2.22 (m, 1H), 1.93-1.86 (m, 1H), 1.79 (s, 3H), 1.76-1.60 (m, 4H), 1.49-1.43 (m, 1H), 1.36-1.18 (m, 3H), 1.13-1.06 (m, 2H), 0.82 (t, 3H, *J* = 7.4 Hz).

^{13}C NMR (100 MHz, $CDCl_3$) δ 141.7, 122.8, 41.8, 33.9, 31.6, 29.09, 29.07, 26.1, 24.7, 18.4, 12.0, 0.8.

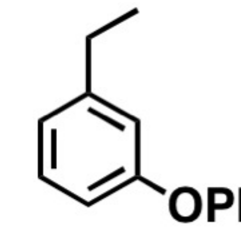
GC (*cyclosil-B,* 90 °C): R_t from dppp: 73.675 min and 74.592 min; from (*S,S*-BDPP): 73.674 min (98%) and 74.729 min (2%).

GC-MS (*methyl silicone*): m/z ([M+) 240.40; exact mass calculated for $C_{14}H_{28}OSi$ 240.19

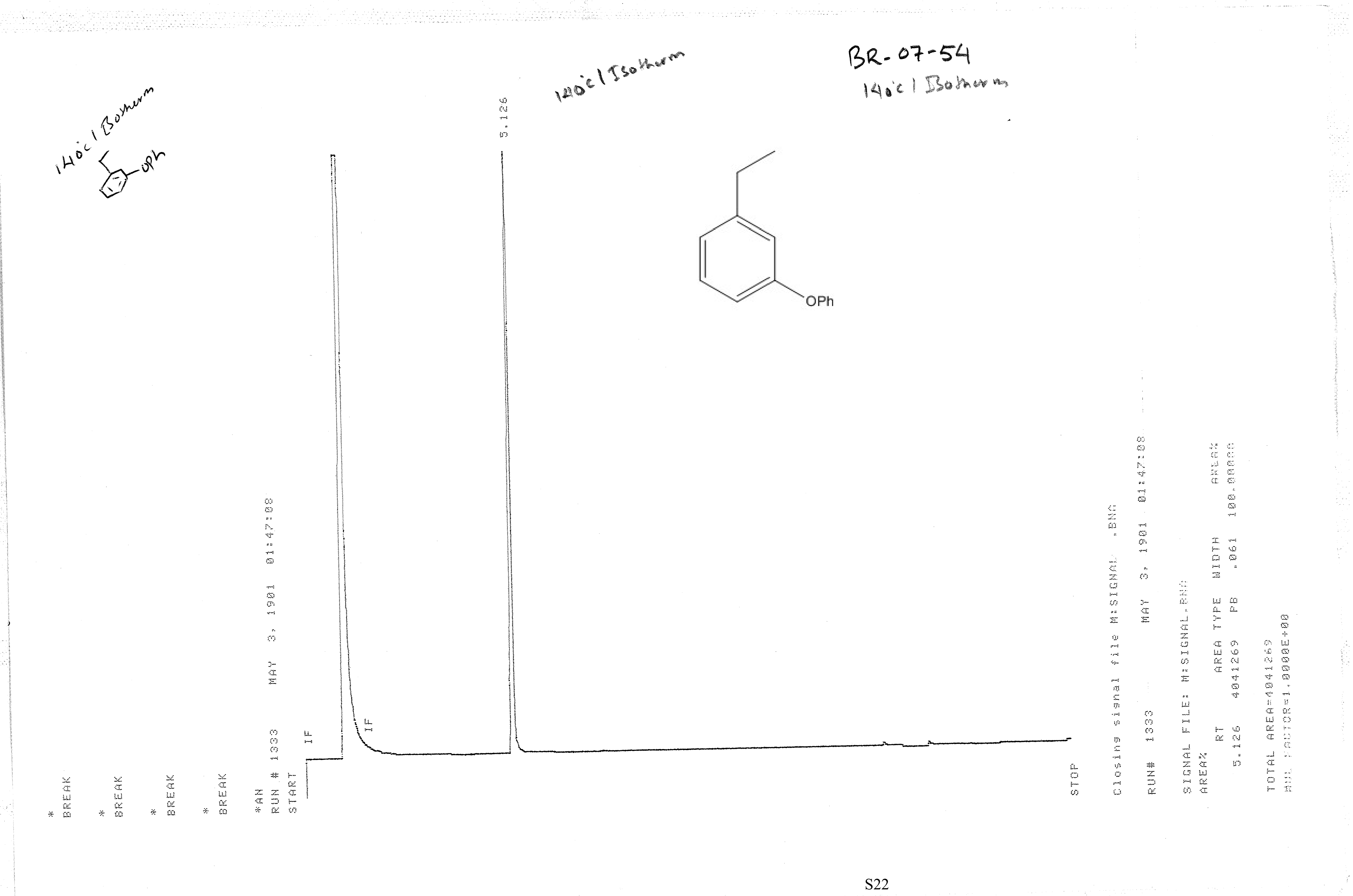
References

1. Britovsek, G. J. P.; Bruce, M.; Gibson, V. C.; Kimberley, B. S.; Maddox, P. J.; Mastroianni, S.; McTavish, S. J.; Redshaw, C.; Solan, G. A.; Strömberg, S.; White, A. J. P.; Williams, D. J. *J. Am. Chem. Soc.* **1999**, *121*, 8728.
2. Kim, H. J.; Asif, R.; Chung, D. S.; Hong, J. I. *Tetrahedron Lett.* **2003**, 44, 4335.
3. Sharma, R. K;. RajanBabu, T. V. *J. Am. Chem. Soc.* **2010**, *132*, 3295 and references cited therein.
4. (a) Gibson, V. C.; Humphries, M. J.; Tellmann, K. P.; Wass, D. F.; White, A. J. P.; Williams, D. J. *Chem. Commun.* **2001**, *2001*, 2252. (b) Kooistra, T. M.; Knijnenburg, Q.; Smits, J. M. M.; Horton, A. D.; Budzelaar, P. H. M.; Gal, A. W. *Angew. Chem. Int. Ed.* **2001**, *40*, 4719.

10a (Crude product)



10b


BR-07-54
140°C / Isotherm
5.126
OPh
START
STOP
RUN # 1333
MAY 3, 1901 01:47:08
Closing signal file M:SIGNAL .BNC
RUN# 1333
SIGNAL FILE: M:SIGNAL.BNC
AREA%
RT AREA TYPE WIDTH AREA%
5.126 4041269 PB .061 100.00000
TOTAL AREA=4041269
MUL FACTOR=1.0000E+00

```
*
BREAK

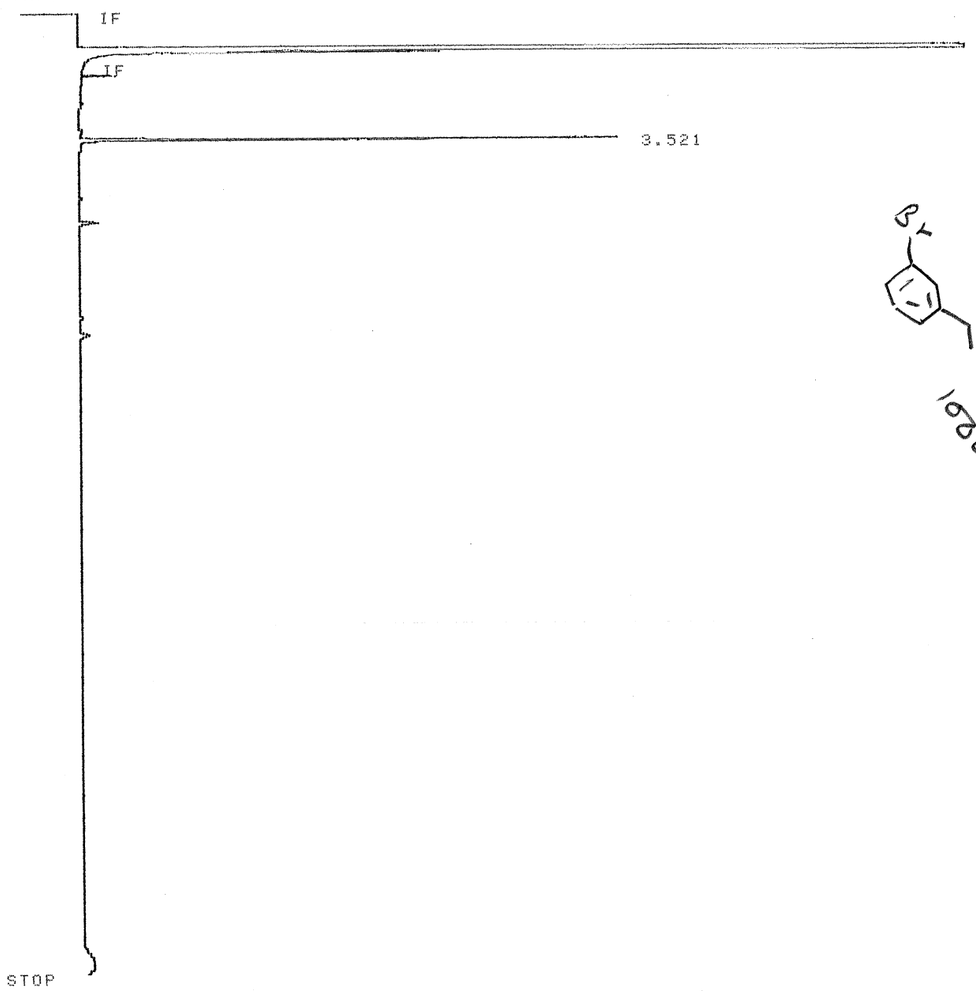
*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 2246      OCT  1, 1901  23:41:50
START
```




```
STOP

Error storing signal to M:SIGNAL  .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL  .PRA

RUN#  2246           OCT  1, 1901  23:41:50

PEAK FILE  : M:SIGNAL.PRA
AREA%
      RT      AREA TYPE  WIDTH      AREA%
   3.521   1474335  SBB   .039  100.00000

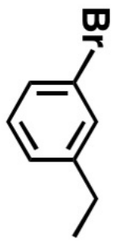
TOTAL AREA=1474335
MUL FACTOR=1.0000E+00
```


Iodic/Bromm

Iodic/Bromm

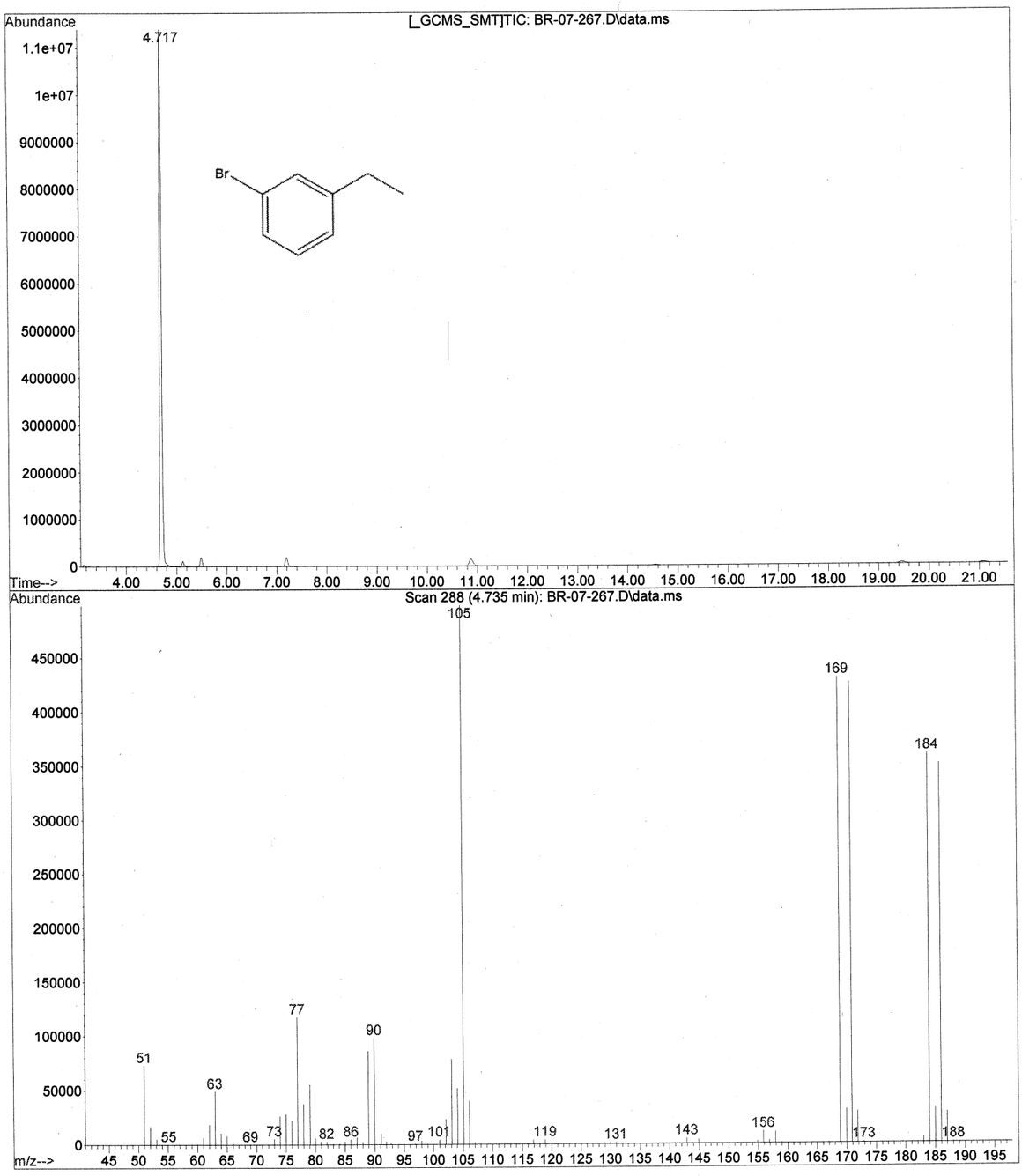
BR-07-267
Iodic/Bromm

S23

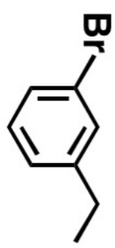


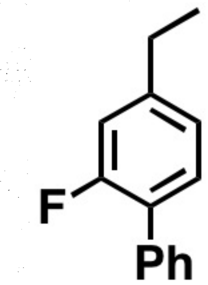
10c

File :D:\MSDChemData\Babu\Balaram\BR-07-267.D
Operator : BALARAM
Acquired : 28 Sep 2015 9:39 using AcqMethod 100-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-267
Misc Info :
Vial Number: 1

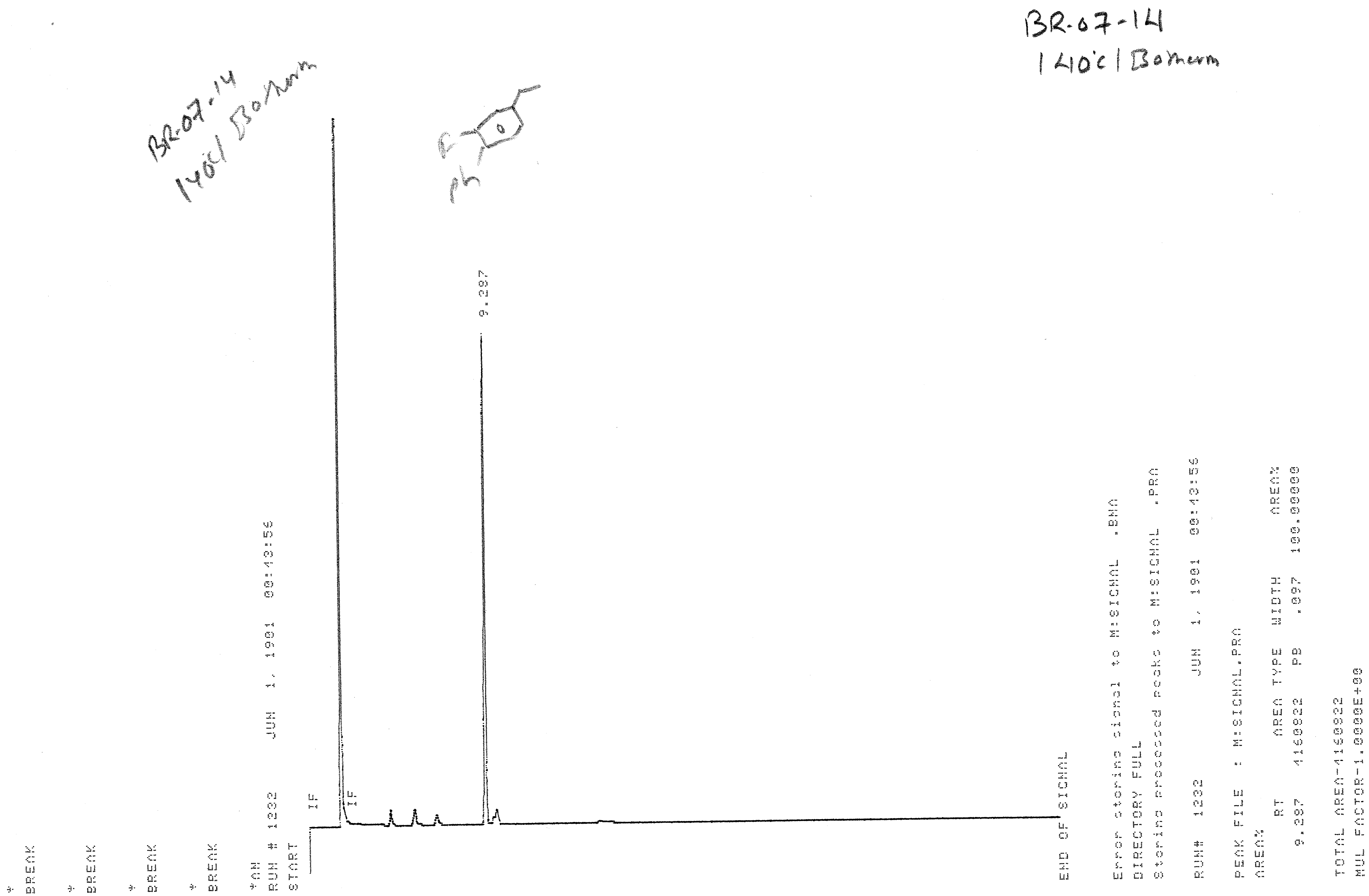


10c

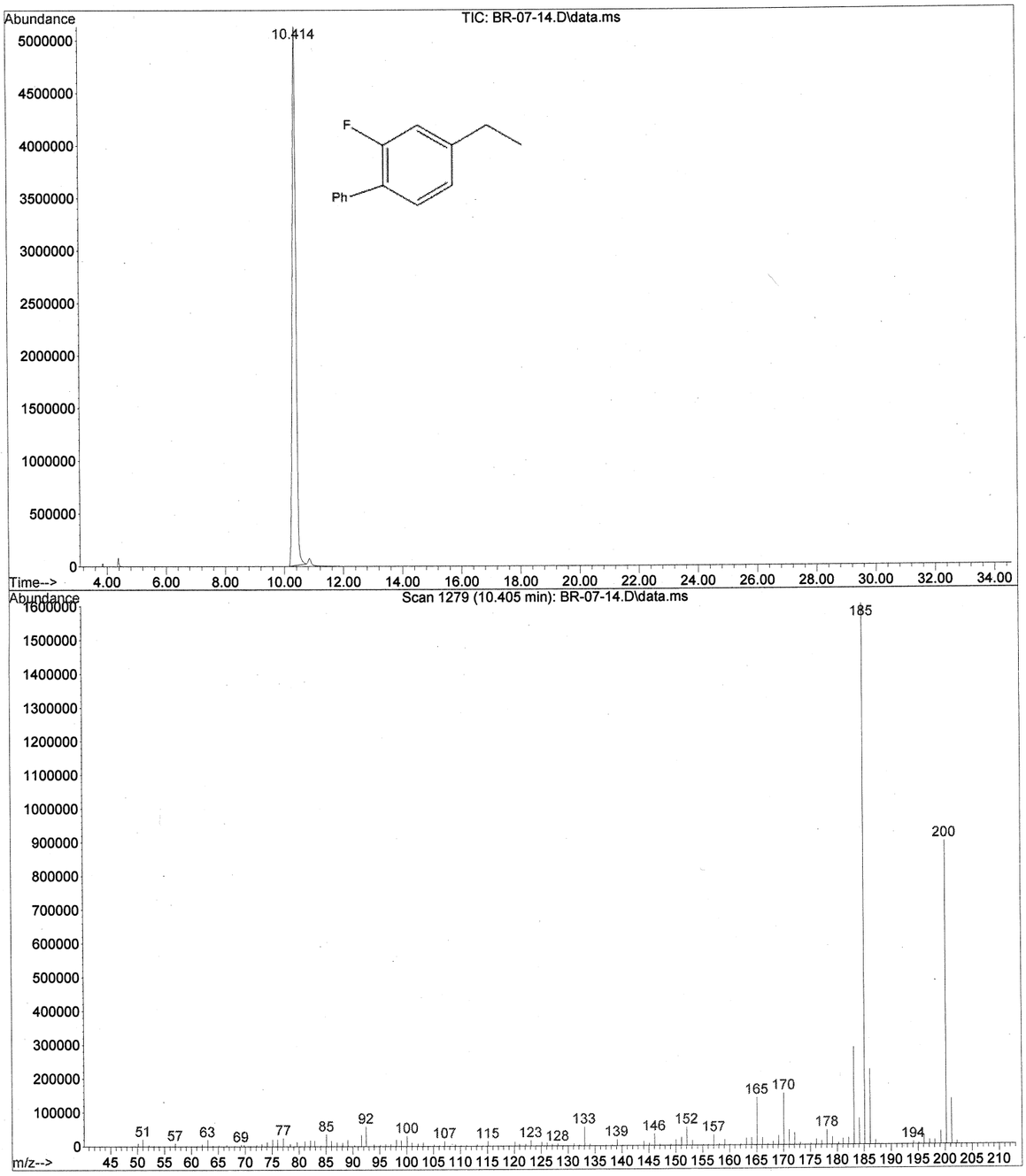




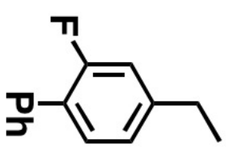
11

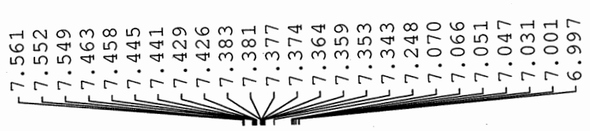

BR-07-14
140°c | Bottom
9.287
* BREAK
* BREAK
* BREAK
* BREAK
*ON
RUN # 1232   JUN 1, 1901 00:43:56
START
IF
IF
END OF SIGNAL
Error storing signal to M:SIGNAL .BNC
DIRECTORY FULL
Storing processed peaks to M:SIGNAL .PRO
RUN# 1232   JUN 1, 1901 00:43:56
PEAK FILE : M:SIGNAL.PRO
AREA%
RT   AREA TYPE WIDTH   AREA%
9.287 4160822 PB .097 100.00000
TOTAL AREA=4160822
MUL FACTOR=1.0000E+00

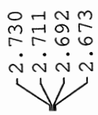
File :D:\MSDChemData\Babu\Balaram\BR-07-14.D
Operator : BALARAM
Acquired : 22 May 2015 20:13 using AcqMethod 140-Isotherm.M
Instrument : GCMS
Sample Name: BR-07-14
Misc Info :
Vial Number: 1

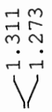


11

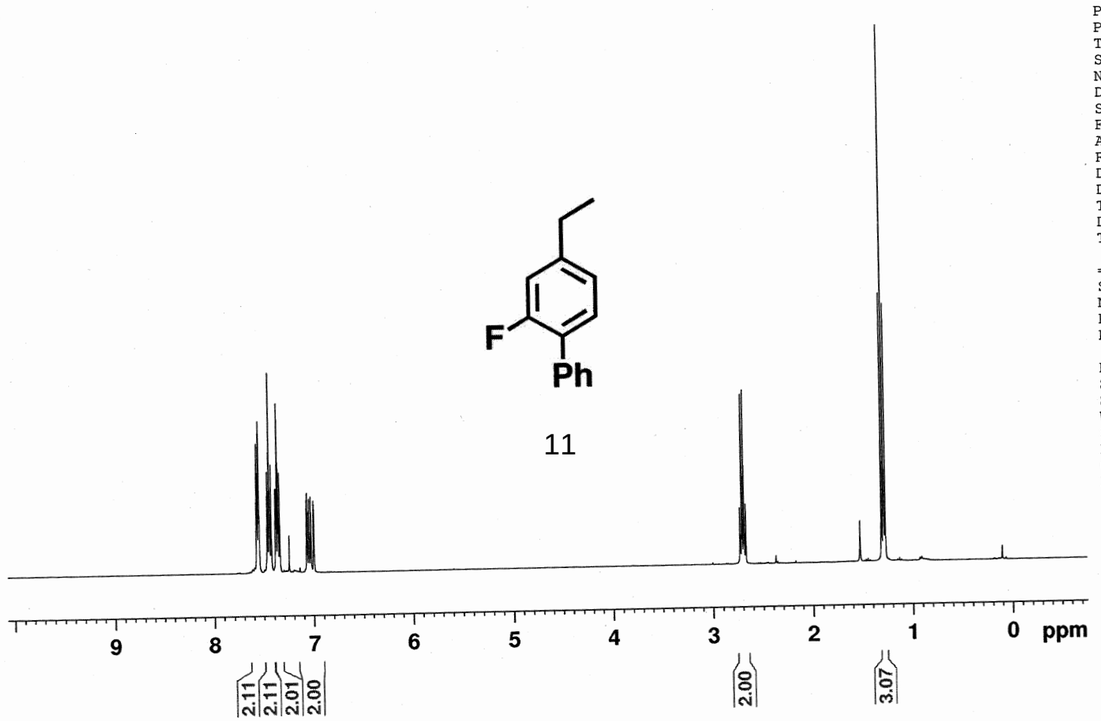








Faculty Group Rajanbabu987
PROTON_OSU CDCl3 {C:\Bruker\TopSpin3.0} raya.5 96



BRUKER

Cur	
NAME	BR-067-14
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

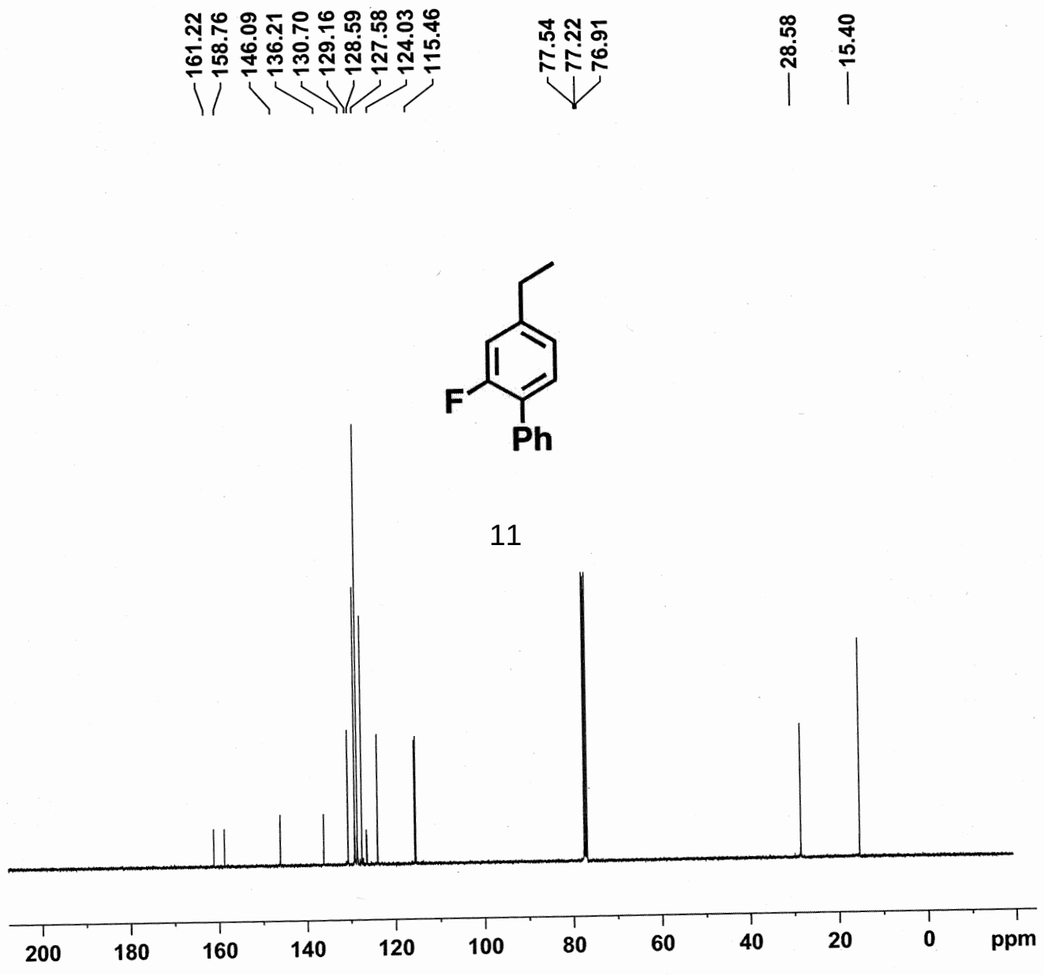
Date_	20150516
Time	1.50
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9845889 sec
RG	79.06
DW	60.800 usec
DE	6.50 usec
TE	301.5 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

SFO1	400.1724712 MHz
NUC1	1H
P1	15.00 usec
PLW1	13.19999981 W

F2 - Processing parameters

SI	65536
SF	400.1700142 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

```
Curr
NAME                 BR-067-14
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20150516
Time                      2.51
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                       2050
DW                     20.800 usec
DE                      6.50 usec
TE                     303.3 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               100.6328883 MHz
NUC1                       13C
P1                        9.50 usec
PLW1               53.29999924 W

======== CHANNEL f2 ========
SFO2               400.1716007 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2               14.60000038 W
PLW12               0.51327997 W
PLW13               0.32850000 W

F2 - Processing parameters
SI                        32768
SF                 100.6228056 MHz
WDW                          EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```

12

100°C - Bolhem

BR-07-24
100°C | Bolhem


```
MUL FACTOR-1.0000E+00

*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 1228    MAY 31, 1991  17:15:16
START
IF

IF

STOP

Error storing signal to M:SIGNAL  .BHA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL  .PRA

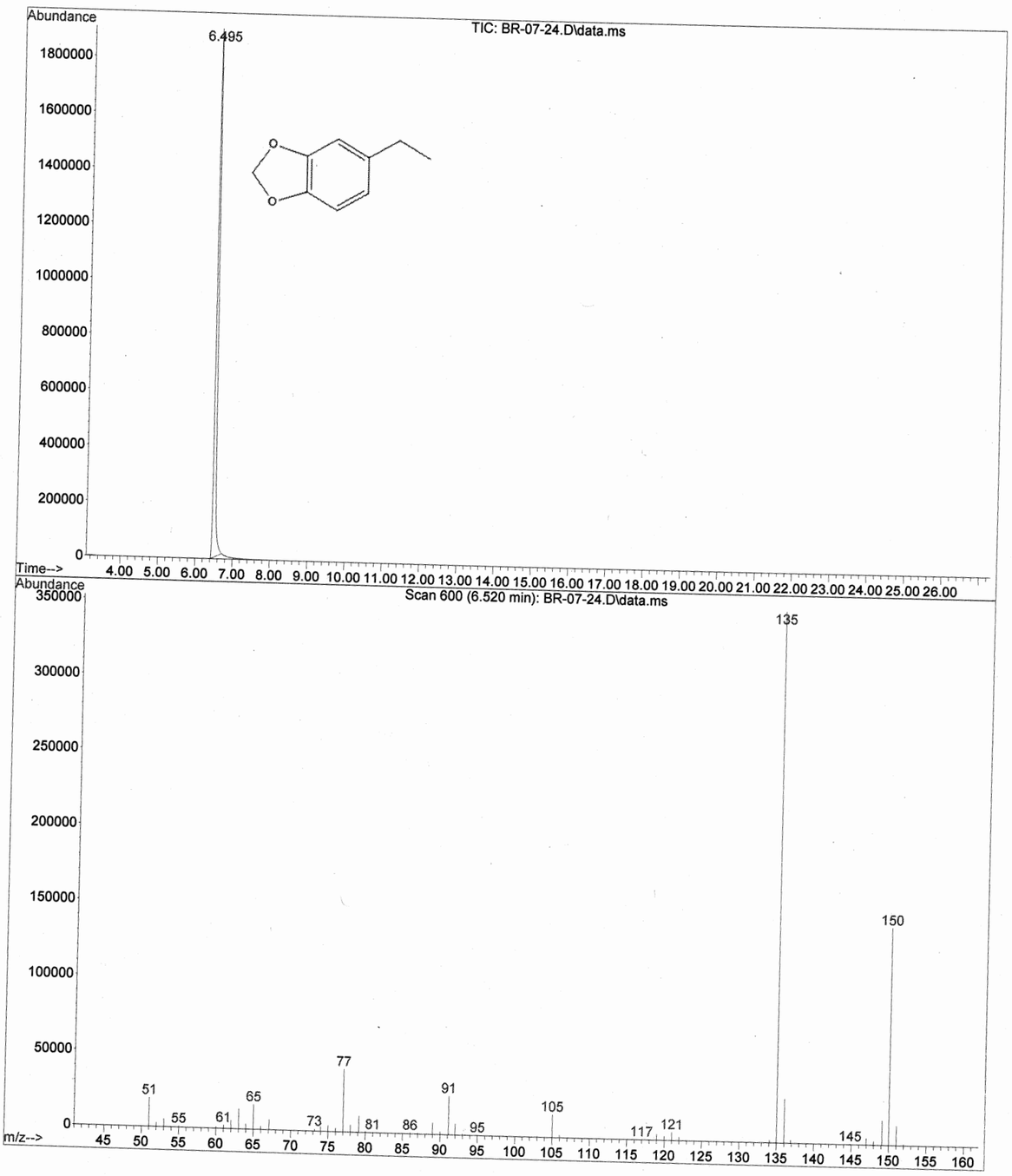
RUN# 1228          MAY 31, 1991  17:15:16

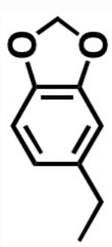
PEAK FILE : M:SIGNAL.PRA
AREA%

      RT        AREA TYPE  WIDTH        AREA%
    6.242  10163276  SBB    .093  100.00000

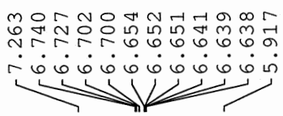
TOTAL AREA=1.0163E+07
MUL FACTOR=1.0000E+00
```

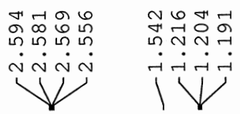
File :D:\MSDChemData\Babu\Balaram\BR-07-24.D
Operator : BALARAM
Acquired : 22 May 2015 17:54 using AcqMethod 100-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-24
Misc Info :
Vial Number: 1





12





Faculty Rajanbabu163

12

BRUKER

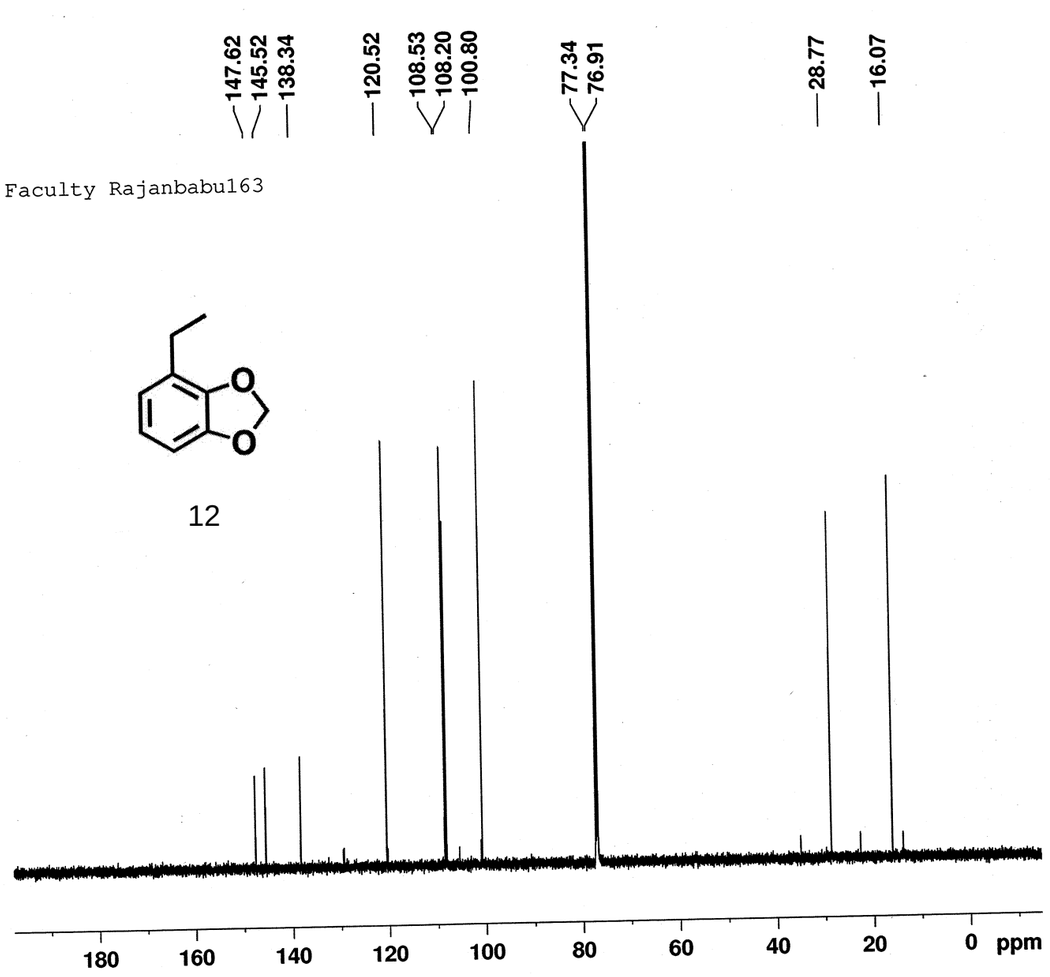
Current Data Parameters
NAME BR-07-24
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150521
Time 23.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 96152
SOLVENT CDCl3
NS 64
DS 2
SWH 12019.230 Hz
FIDRES 0.125002 Hz
AQ 3.9999232 sec
RG 120.04
DW 41.600 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.0478245 MHz
NUC1 1H
P1 10.00 usec
PLW1 25.10000038 W

F2 - Processing parameters
SI 65536
SF 600.0450121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Faculty Rajanbabu163



12

BRUKER

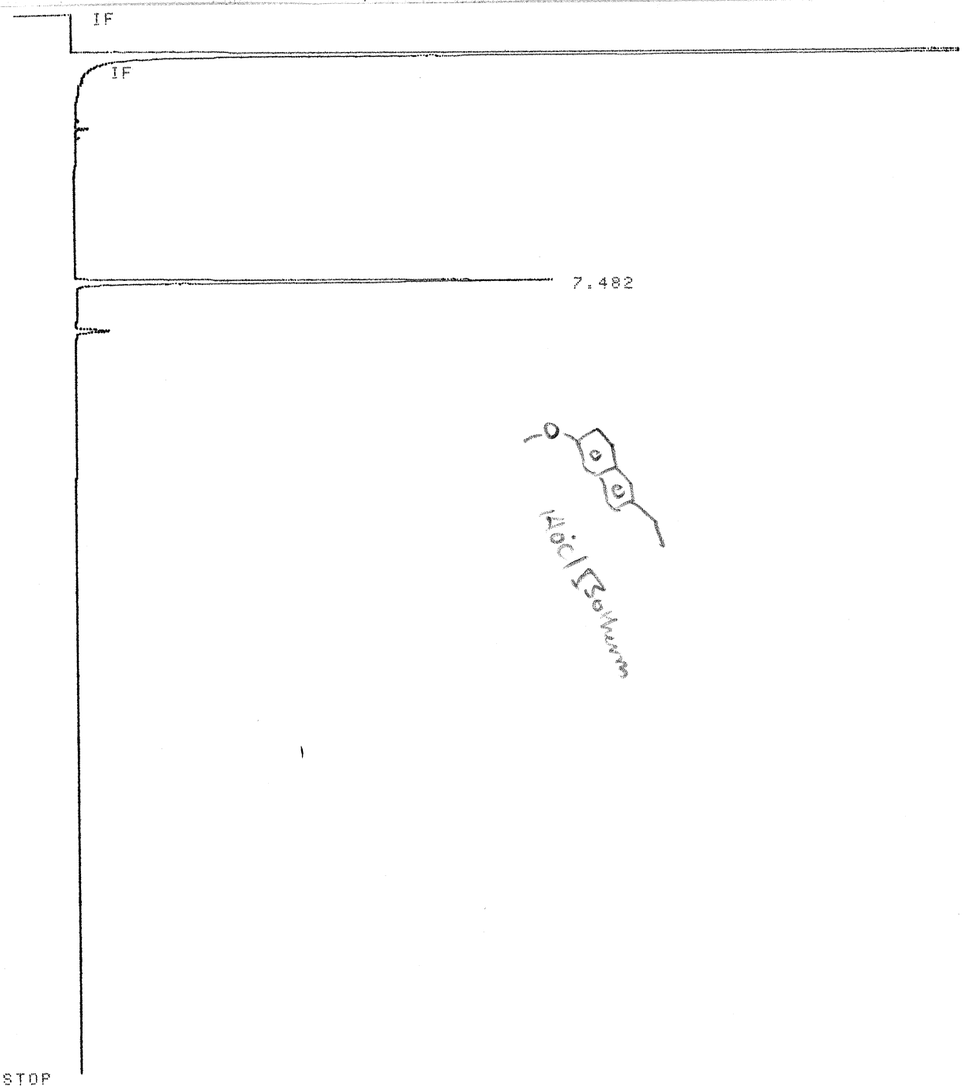
```
Cur
NAME                 BR-07-24
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_                20150522
Time                     0.24
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                36057.691 Hz
FIDRES              0.550197 Hz
AQ                  0.9087659 sec
RG                     189.17
DW                     13.867 usec
DE                       6.50 usec
TE                      300.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             150.8965227 MHz
NUC1                     13C
P1                     12.00 usec
PLW1              78.16000366 W

======== CHANNEL f2 ========
SFO2             600.0474002 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                  80.00 usec
PLW2              28.00000000 W
PLW12              0.43750000 W
PLW13              0.28000000 W

F2 - Processing parameters
SI                       32768
SF               150.8814185 MHz
WDW                         EM
SSB       0
LB                      1.00 Hz
GB        0
PC                      1.40
```



14oC Isotherm

STOP

Error storing signal to M:SIGNAL .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL .PRA

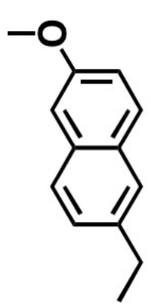
RUN# 2235 SEP 30, 1901 15:32:33

PEAK FILE : M:SIGNAL.PRA
AREA%

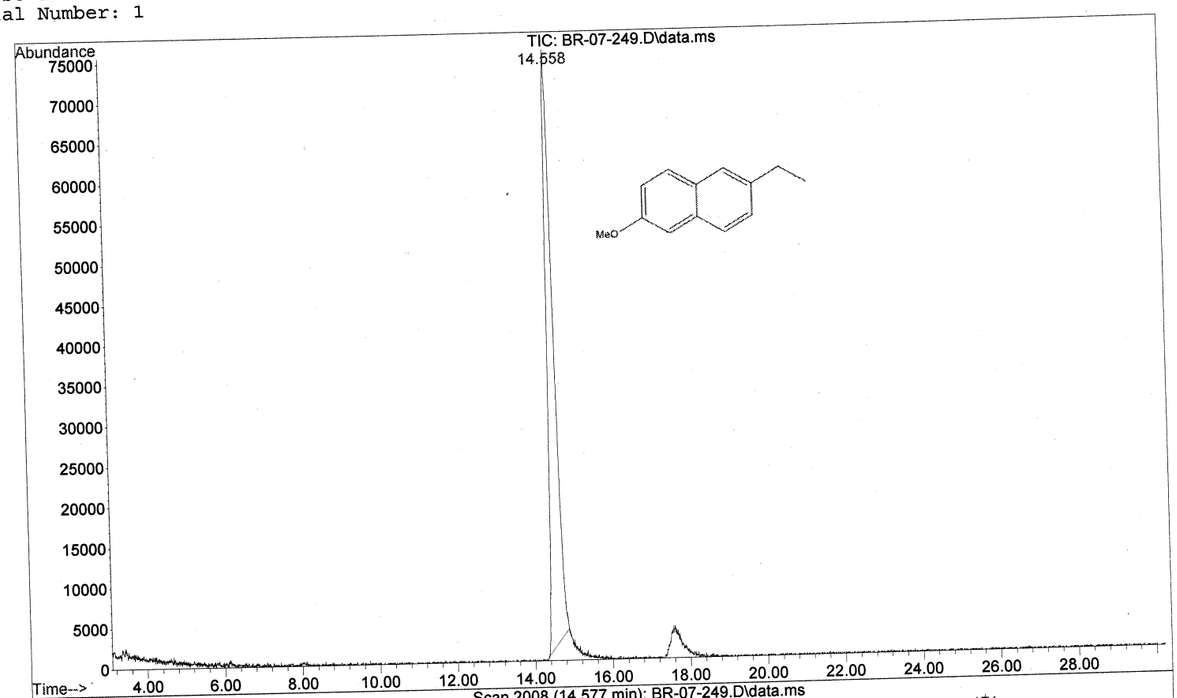
RT	AREA	TYPE	WIDTH	AREA%
7.482	1363001	PB	.082	100.00000

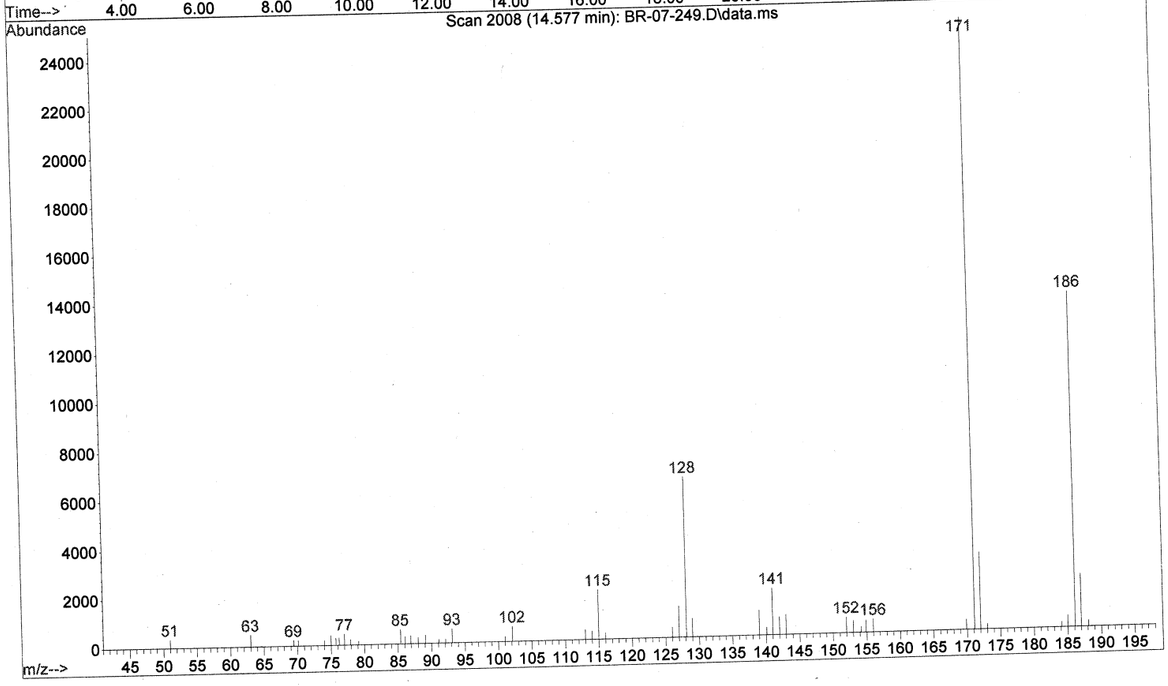
TOTAL AREA=1363001
MUL FACTOR=1.0000E+00

BR-07-249
14oC Isotherm



File :D:\MSDChemData\Babu\Balaram\BR-07-249.D
Operator : BALARAM
Acquired : 17 Sep 2015 11:15 using AcqMethod 140-Isotherm.M
Instrument : GCMS
Sample Name: BR-07-249
Misc Info :
Vial Number: 1





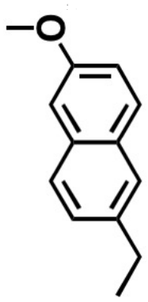
Storing processed peaks to M:SIGNAL .PRA

RUN# 2035 SEP 6, 1901 16:55:52

PEAK FILE : M:SIGNAL.PRA
AREA%

RT	AREA	TYPE	WIDTH	AREA%
7.850	17843888	PB	.189	100.00000

TOTAL AREA=1.7844E+07
MUL FACTOR=1.0000E+00



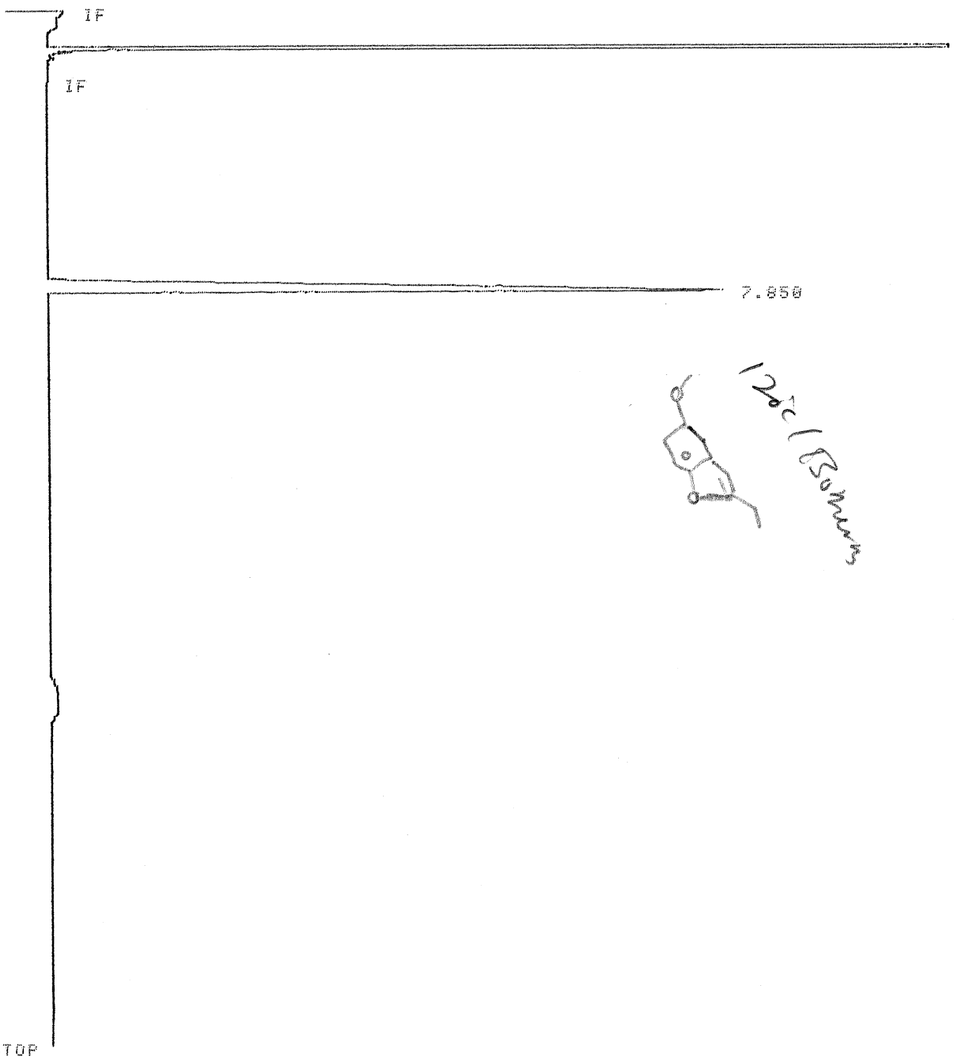
```
*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 2035      SEP  6, 1901  16:55:52
START
```



120c / Bothum

BR-07-231
120c / Bothum

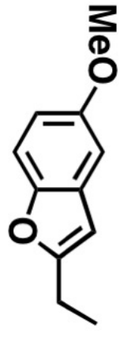
```
STOP

Error storing signal to M:SIGNAL  .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL  .PRA

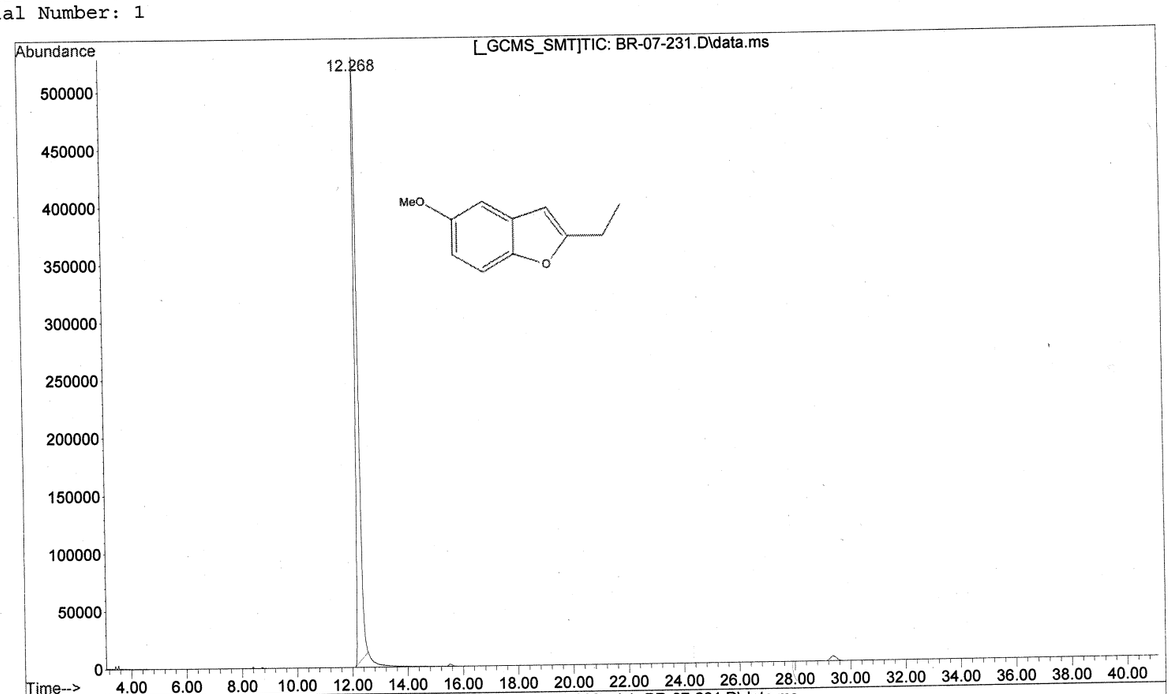
RUN#  2035          SEP  6, 1901  16:55:52

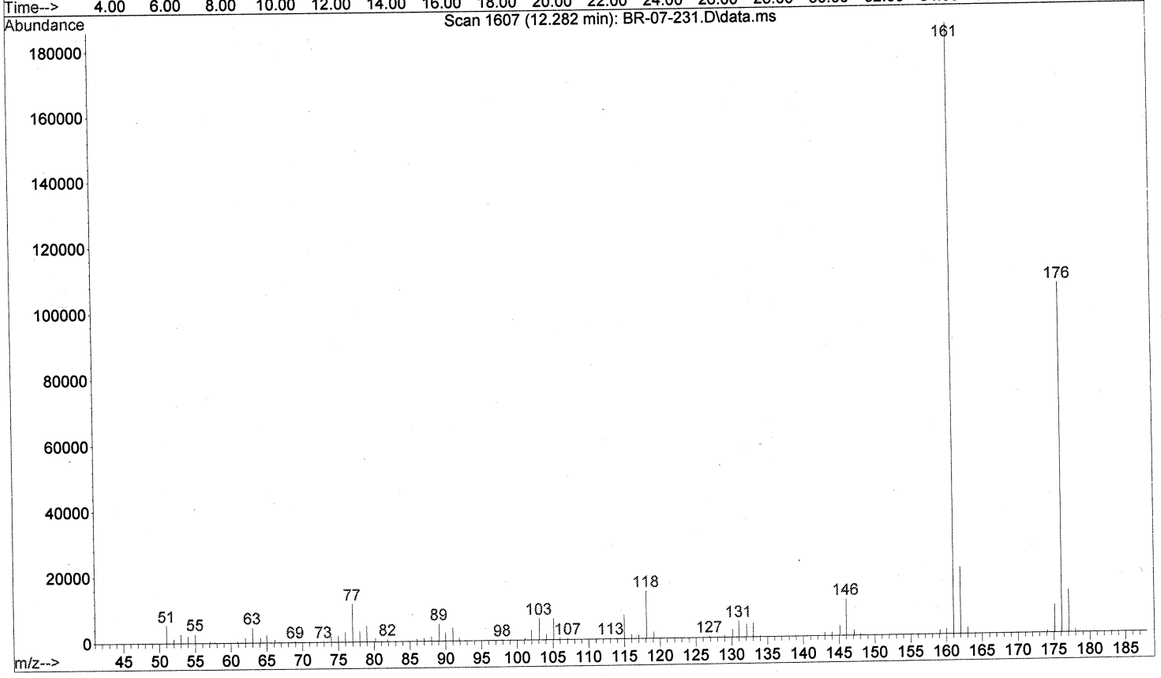
PEAK FILE  : M:SIGNAL.PRA
AREA%
      RT      AREA TYPE  WIDTH      AREA%
   7.850  17843888   PB   .189  100.00000

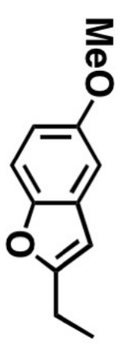
TOTAL AREA=1.7844E+07
MUL FACTOR=1.0000E+00
```



File :D:\MSDChemData\Babu\Balaram\BR-07-231.D
Operator : BALARAM
Acquired : 29 Aug 2015 14:56 using AcqMethod 120-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-231
Misc Info :
Vial Number: 1







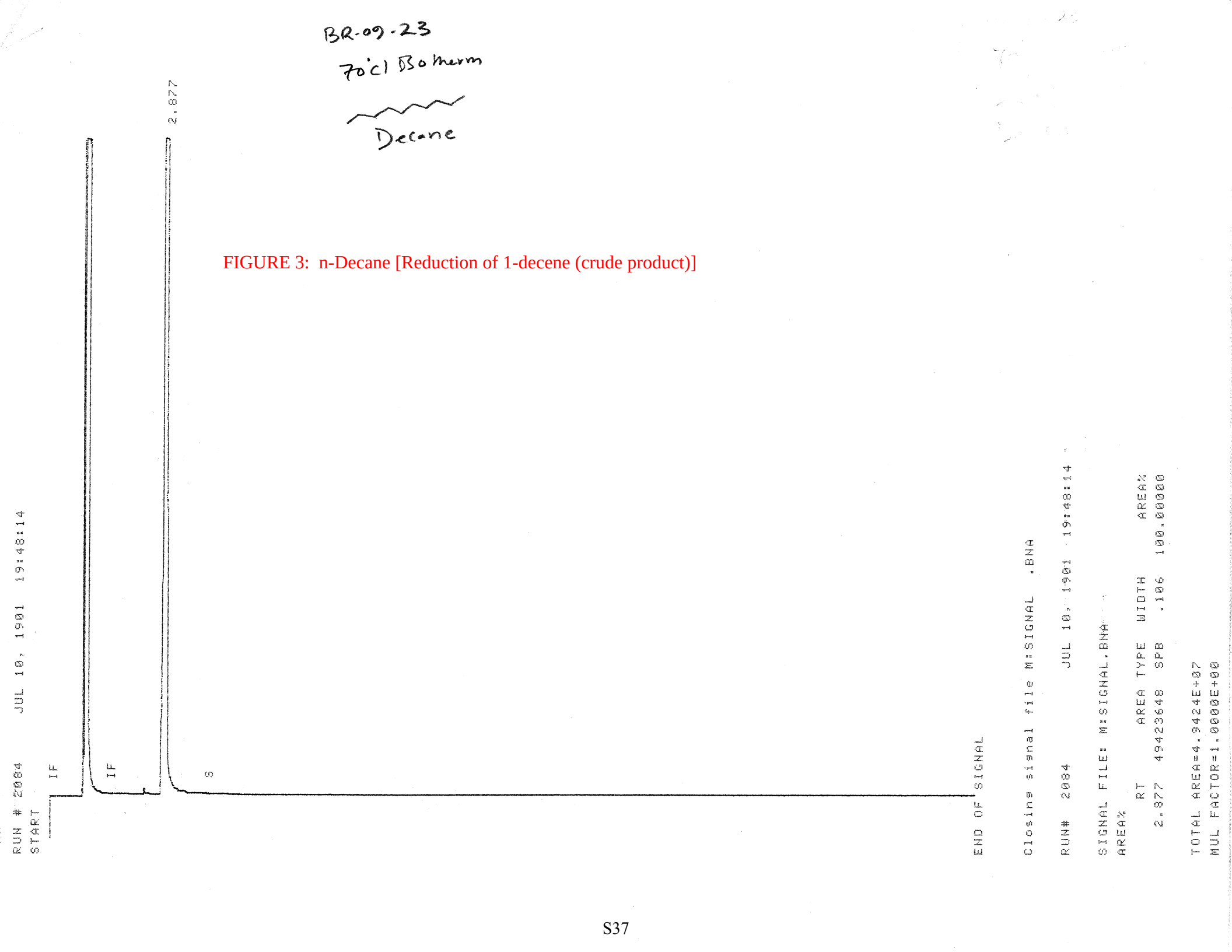
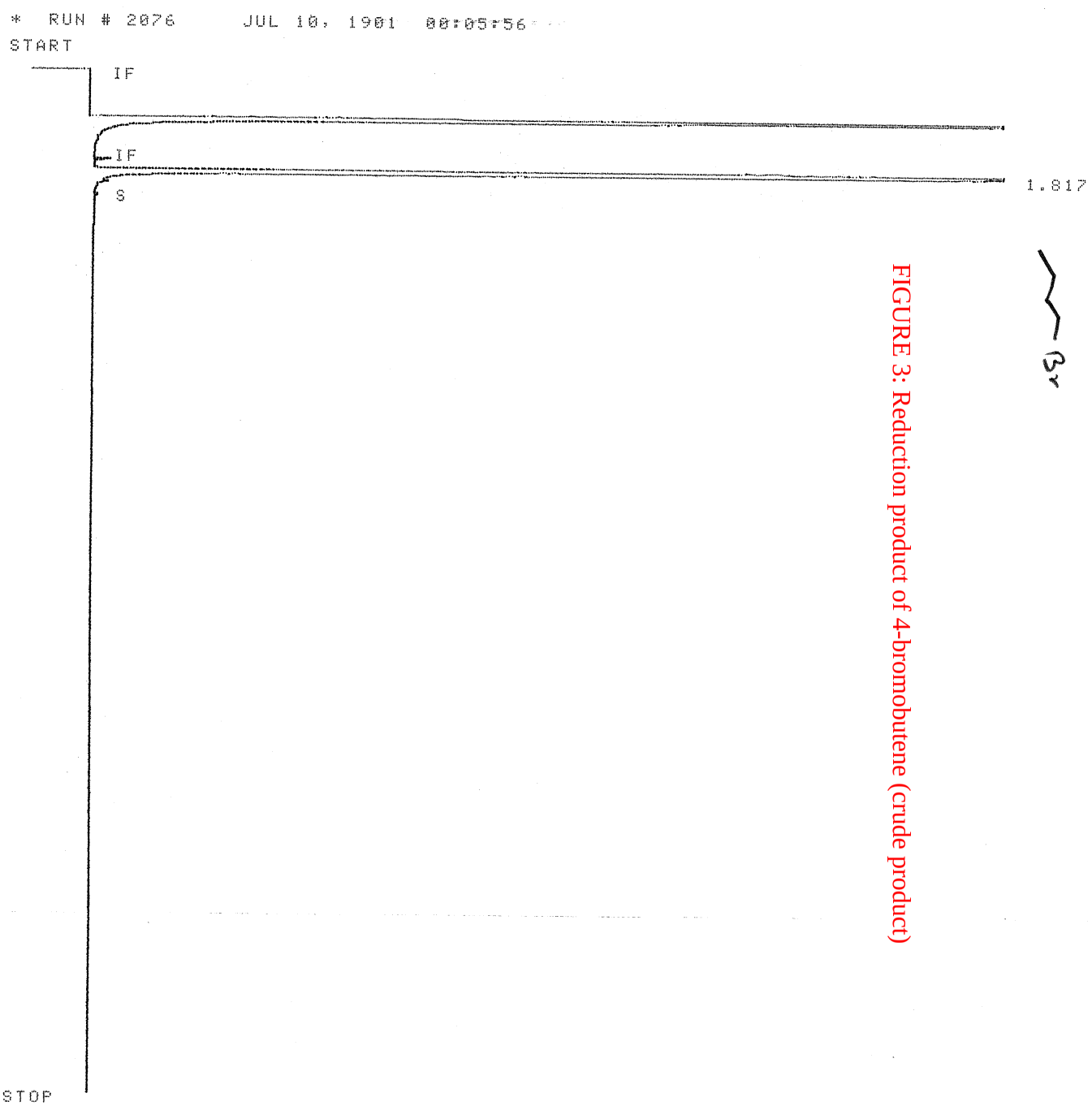


FIGURE 3: n-Decane [Reduction of 1-decene (crude product)]



Closing signal file M:SIGNAL .BNC

RUN# 2076 JUL 10, 1901 00:05:56

SIGNAL FILE: M:SIGNAL.BNC
AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.817	30931072	SPB	.066	100.00000

TOTAL AREA=3.0931E+07
MUL FACTOR=1.0000E+00

BR-09-12
60°C (Isomers)
Br

FIGURE 3: Reduction product of 4-bromobutene (crude product)

FIGURE 3: Reduction product of 3-butene-1-ol (crude product)

BQ-09-29

35°C / Isotherm

FIGURE 3: Reduction product of allyl amine (crude product)

Closing signal file M:SIGNAL .BNA

RUN# 2110 JUL 12, 1901 22:45:30

SIGNAL FILE: M:SIGNAL.BNA

AREA%

RT	AREA TYPE	WIDTH	AREA%
2.222	27854864 SPB	.060	100.00000

TOTAL AREA=2.7855E+07

MUL FACTOR=1.0000E+00

BR-09-50

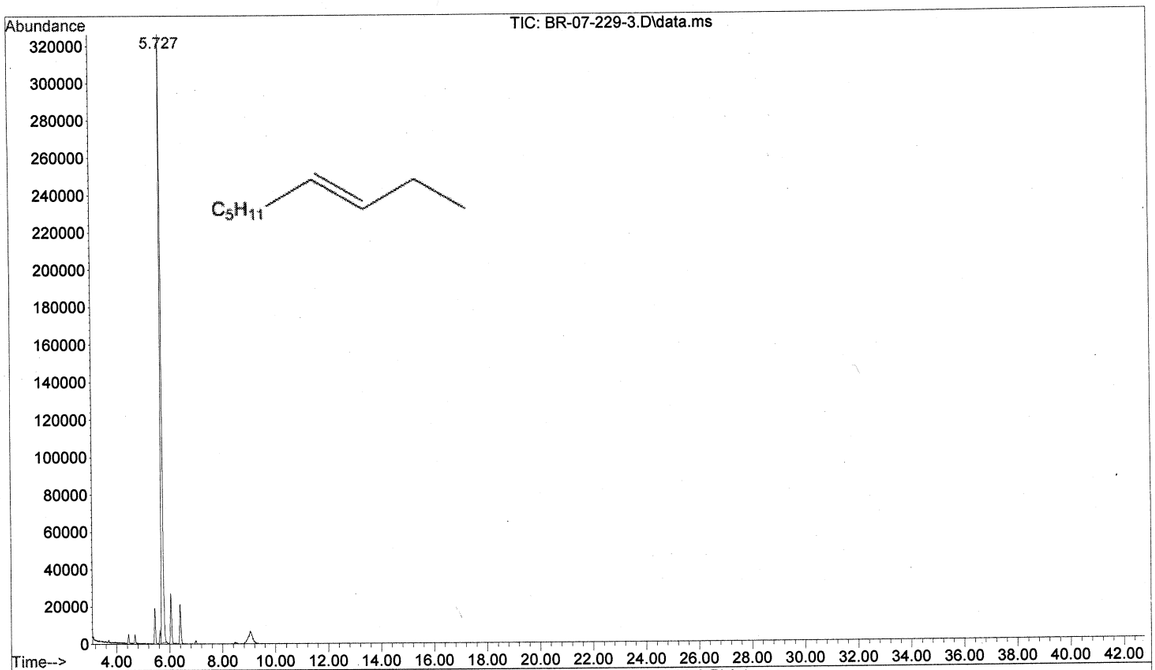
100 cl Bottoms

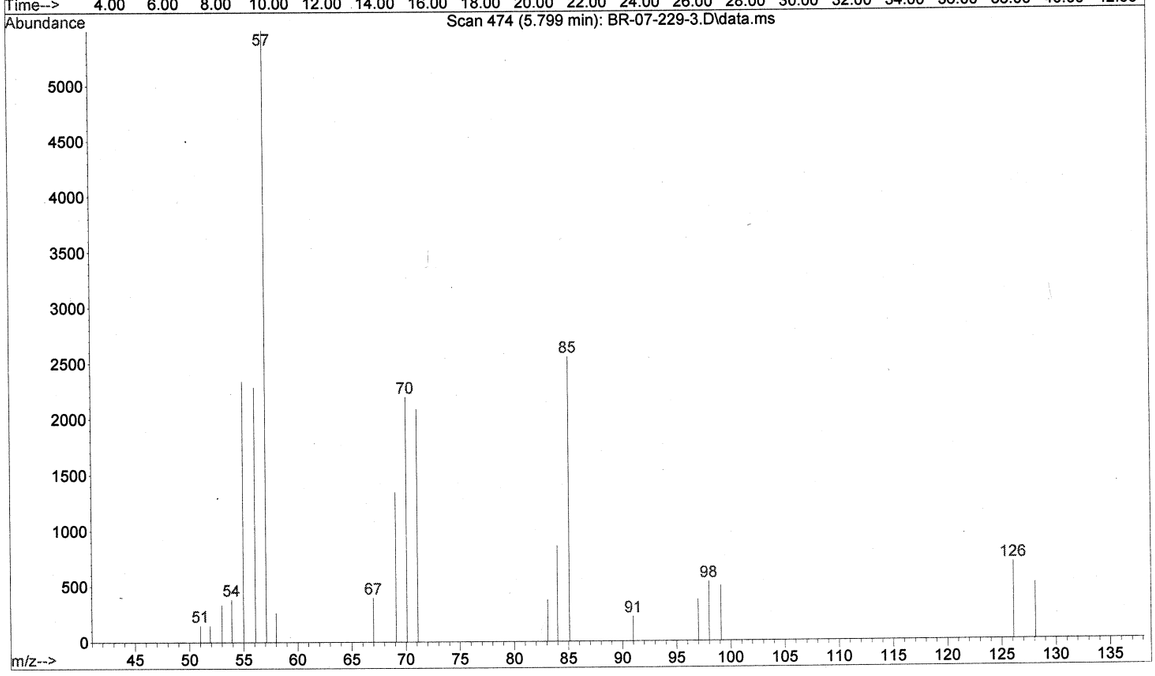
FIGURE 3: Reduction product of cis- and trans- 4-vinyl-1,2-epoxycyclohexane (crude product)

C5H11
27b

BR-07-229
50°C / Bohrem
C5H11
50°C / Bohrem
2.978
*
BREAK
*
BREAK
*
BREAK
*HH
RUN # 2082 SEP 11, 1901 19:26:18
START
IF
IF
STOP
Error storing signal to M:SIGNAL .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL .PRA
RUN# 2082 SEP 11, 1901 19:26:18
PEAK FILE : M:SIGNAL.PRA
AREA%
RT AREA TYPE WIDTH AREA%
2.978 18648848 SBB .047 100.00000
TOTAL AREA=1.8649E+07
MUL FACTOR=1.0000E+00

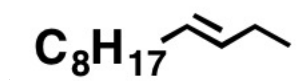
File :D:\MSDChemData\Babu\Balaram\BR-07-229-3.D
Operator : BALARAM
Acquired : 31 Aug 2015 13:27 using AcqMethod 50-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-229-3
Misc Info :
Vial Number: 1





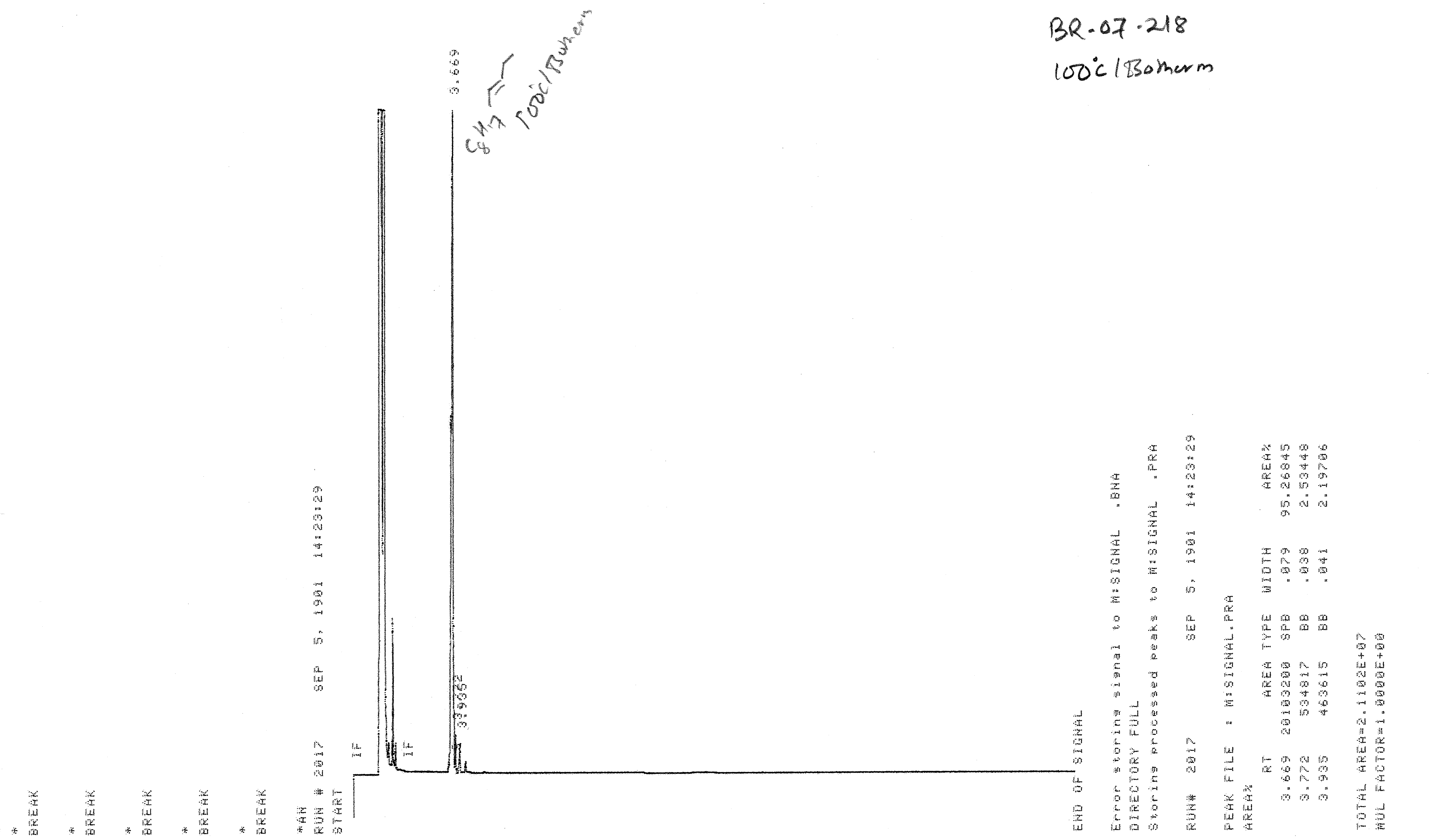
C_5H_{11}

27b



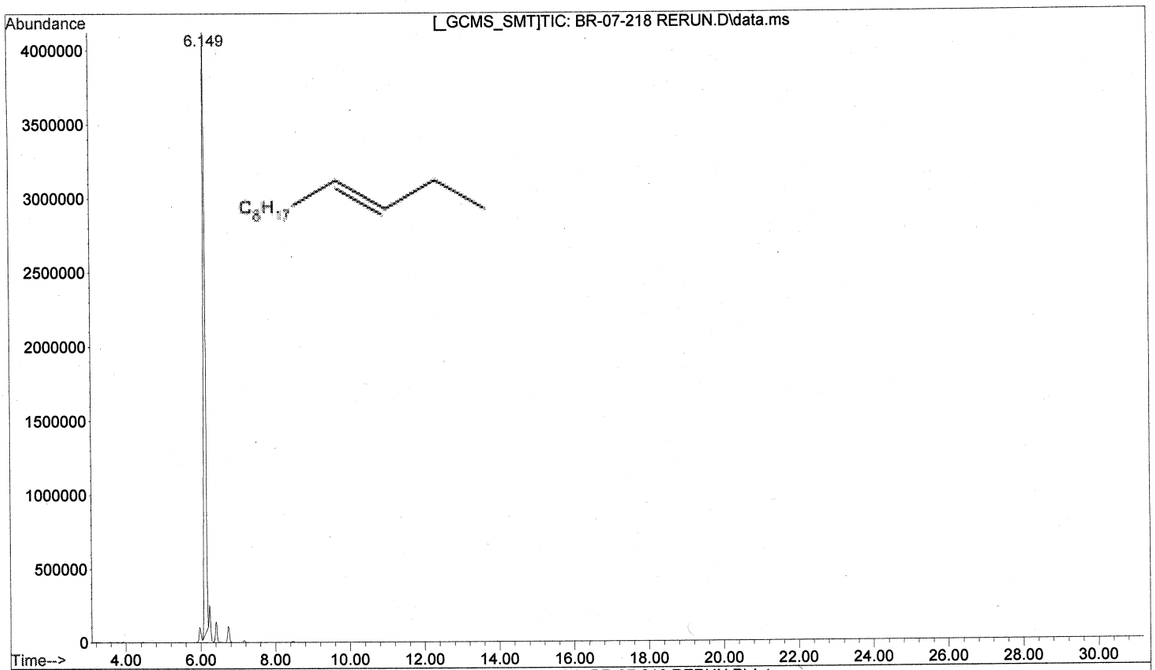
C_8H_{17}

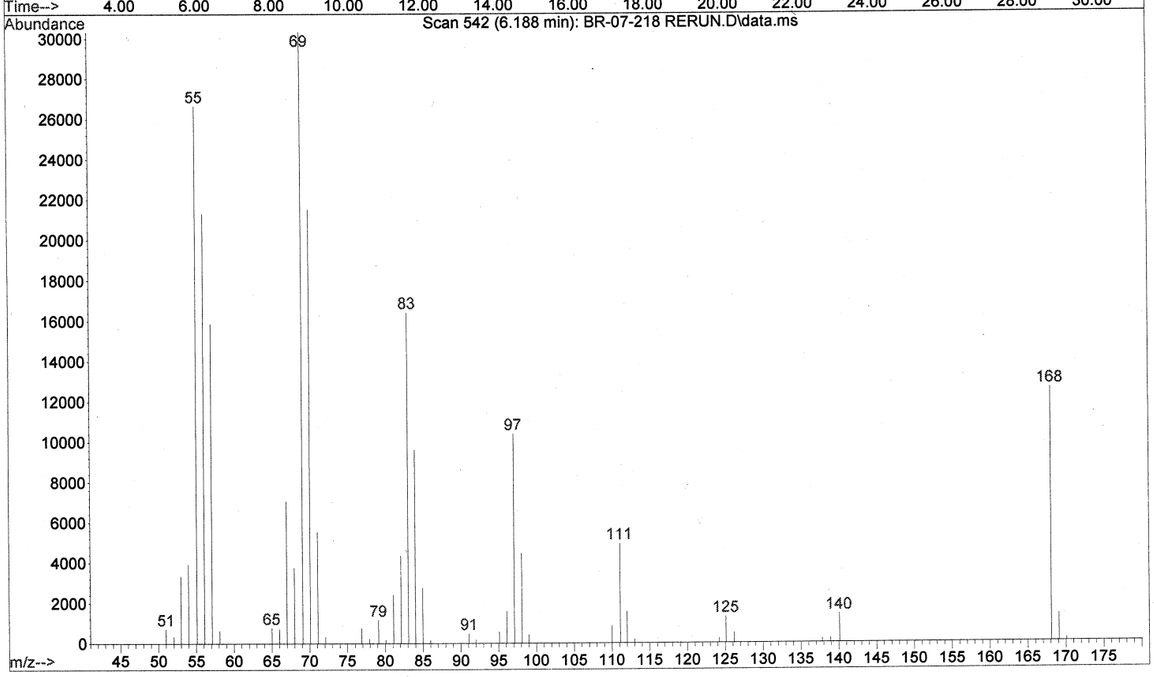
27a



BR-07-218
100°C / Bohem
* BREAK
* BREAK
* BREAK
* BREAK
* BREAK
*AN
RUN # 2017 SEP 5, 1901 14:23:29
START
IF
IF
3.669
3.772
END OF SIGNAL
Error storing signal to M:SIGNAL .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL .PRA
RUN# 2017 SEP 5, 1901 14:23:29
PEAK FILE : M:SIGNAL.PRA
AREA%
RT AREA TYPE WIDTH AREA%
3.669 20103200 SPB .079 95.26845
3.772 534817 BB .038 2.53448
3.935 463615 BB .041 2.19706
TOTAL AREA=2.1102E+07
MUL FACTOR=1.0000E+00

File :D:\MSDChemData\Babu\Balaram\BR-07-218 RERUN.D
Operator : BALARAM
Acquired : 27 Aug 2015 10:58 using AcqMethod 100-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-218 RERUN
Misc Info :
Vial Number: 1





C_8H_{17}

27a

29

50°C / Isotherm

BR-07-230

50°C / Isotherm


```
*
BREAK

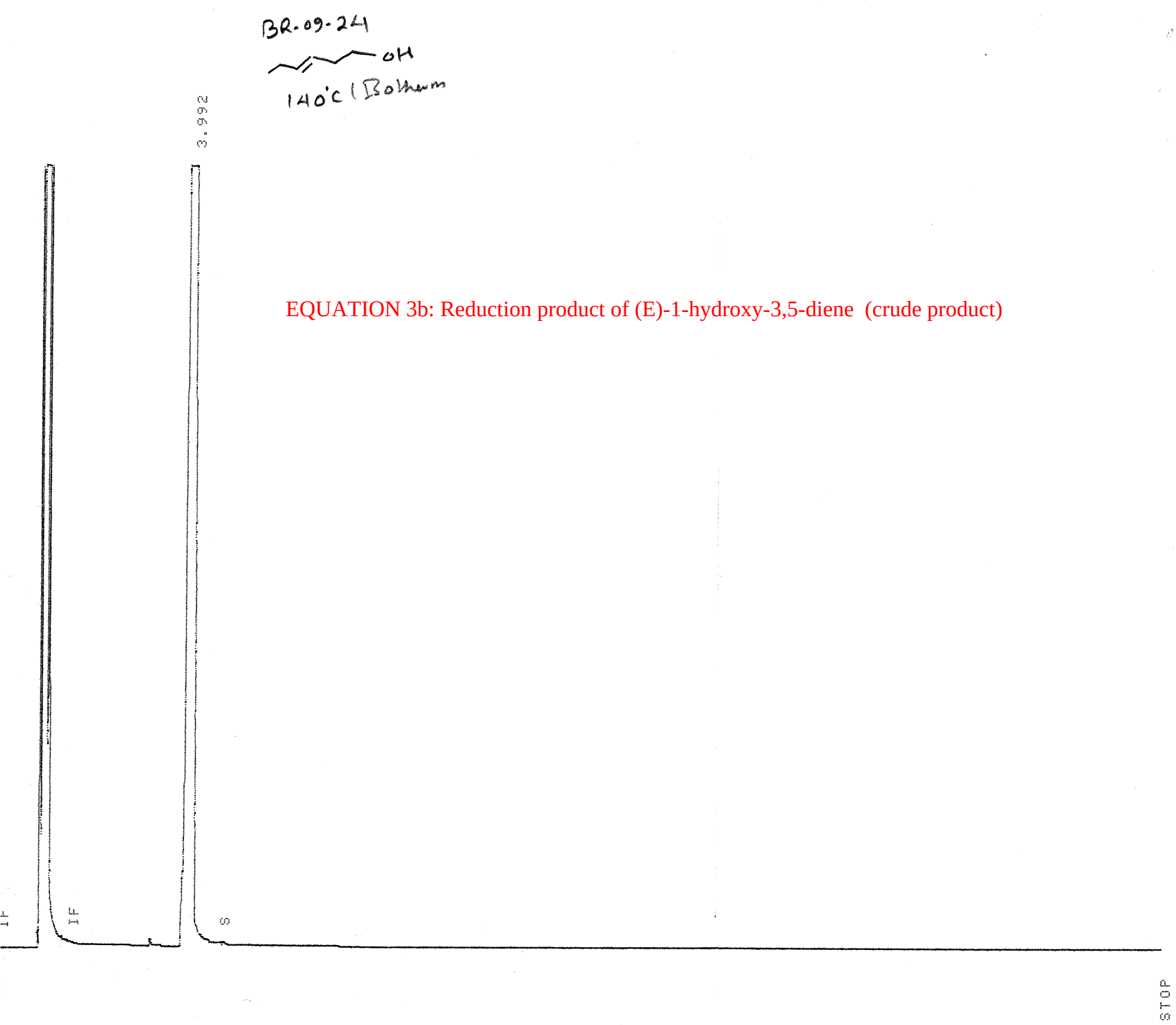
*AN
RUN # 1475    MAY 14, 1901   23:10:54
START
END OF SIGNAL

Closing signal file M:SIGNAL   .BNA

RUN#  1475          MAY 14, 1901   23:10:54

SIGNAL FILE: M:SIGNAL.BNA
AREA%    RT      AREA TYPE  WIDTH      AREA%
   3.278   2445093  SPB   .035  100.00000

TOTAL AREA=2445093
MUL FACTOR=1.0000E+00
```



EQUATION 3b: Reduction product of (E)-1-hydroxy-3,5-diene (crude product)

EQUATION 4: Co-Catalyzed hydrogenation (at 1 atmosphere of H2) of (E)-1,3-dodecadiene (crude product)

BR-09-38
100°c / Bolhum
H_2 (50 PSI)

C_8H_{12}

e.4.

EQUATION 4: Co-Catalyzed hydrogenation (at 3.5 atmosphere of H2) of (E)-1,3-dodecadiene (crude product)

RUN # 2047 JUL 5, 1901 01:22:28

Closing signal file M:SIGNAL .BNA

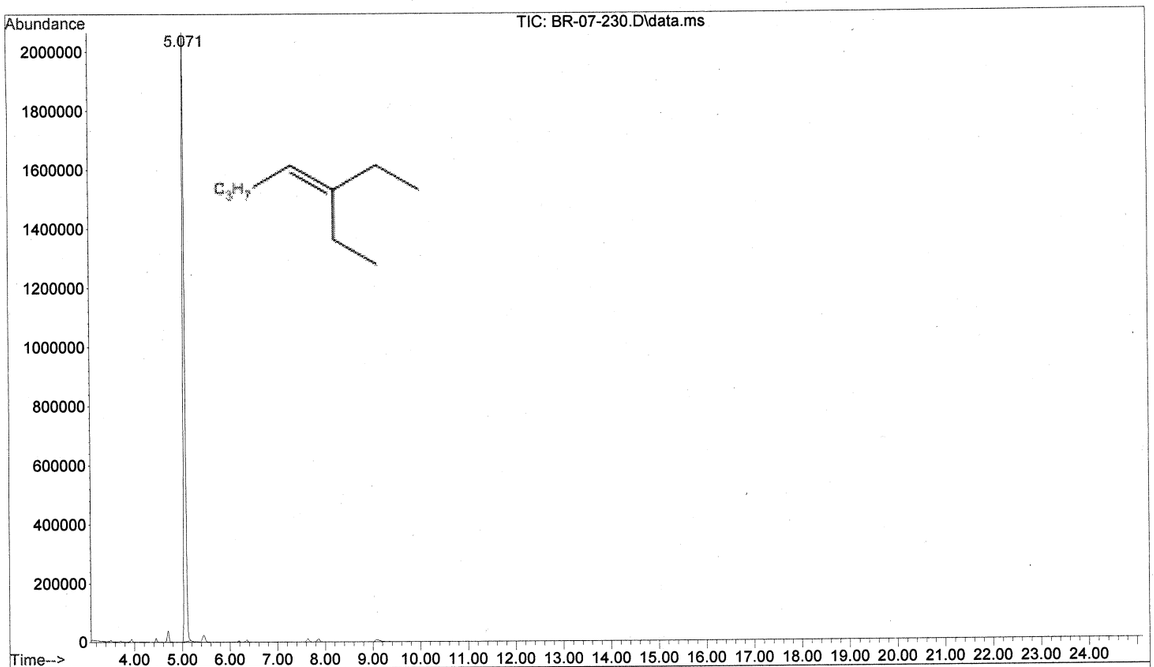
RUN# 2047 JUL 5, 1901 01:22:28

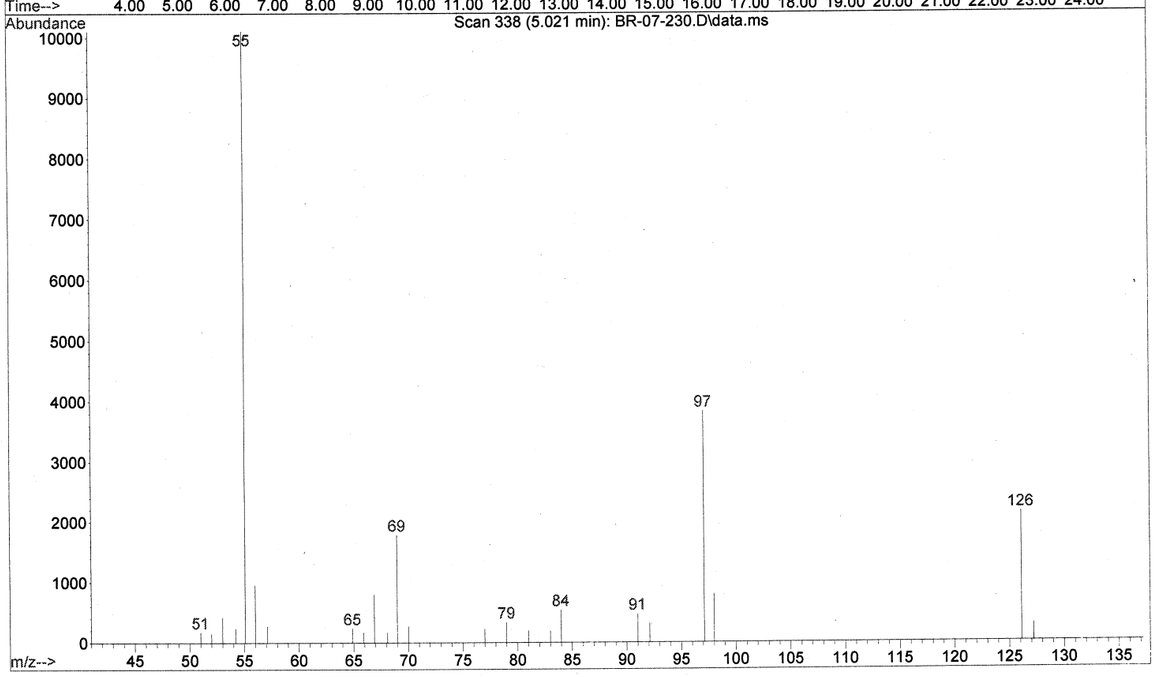
SIGNAL FILE: M:SIGNAL.BNA
AREA%

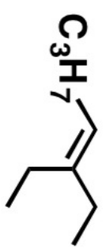
RT	AREA TYPE	WIDTH	AREA%
3.227	75133504 SBB	.159	100.00000

TOTAL AREA=7.5134E+07
MUL FACTOR=1.0000E+00

File :D:\MSDChemData\Babu\Balaram\BR-07-230.D
Operator : BALARAM
Acquired : 28 Aug 2015 20:15 using AcqMethod 50-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-230
Misc Info :
Vial Number: 1







```
*
BREAK

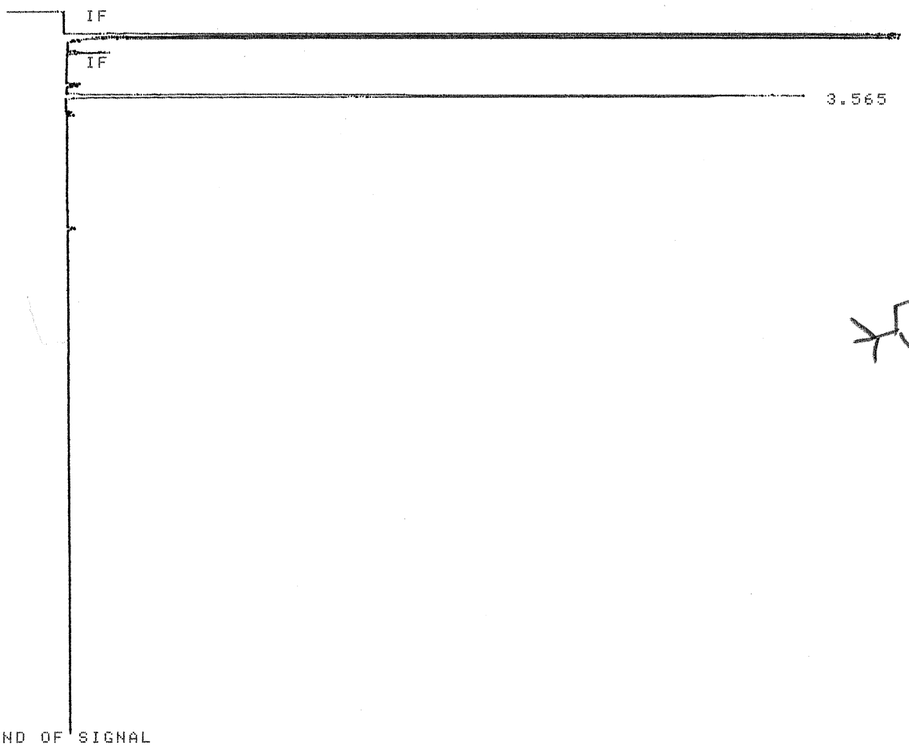
*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 1746      AUG  9, 1901  16:30:04
START
```



```
END OF SIGNAL

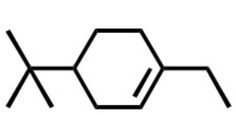
Error storing signal to M:SIGNAL  .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL  .PRA

RUN#  1746          AUG  9, 1901  16:30:04

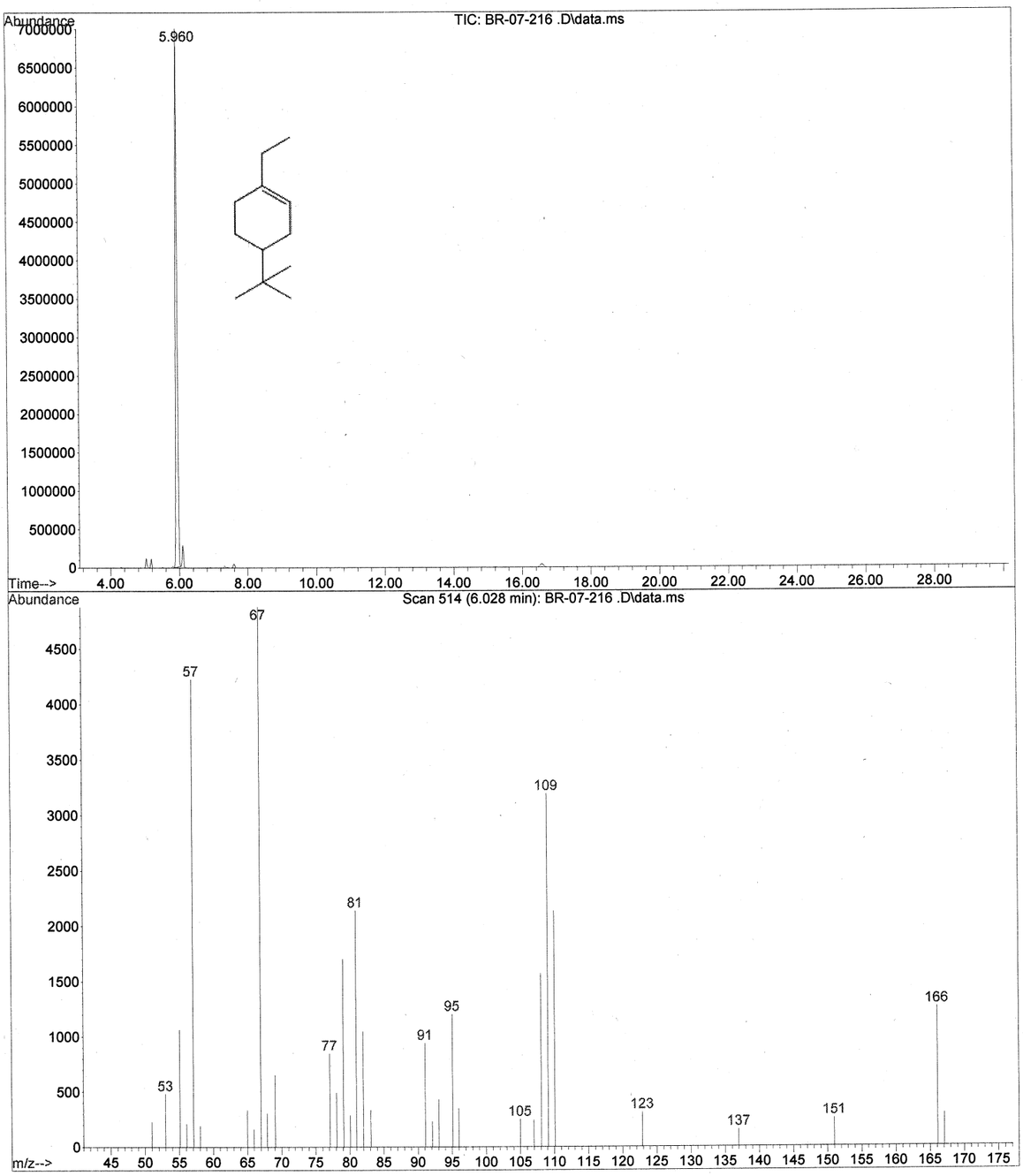
PEAK FILE  : M:SIGNAL.PRA
AREA%
      RT      AREA TYPE  WIDTH      AREA%
   3.565  29982176  SPB   .064  100.00000

TOTAL AREA=2.9982E+07
MUL FACTOR=1.0000E+00
```

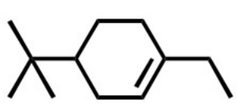
BR.07-216
100°C / Bohem

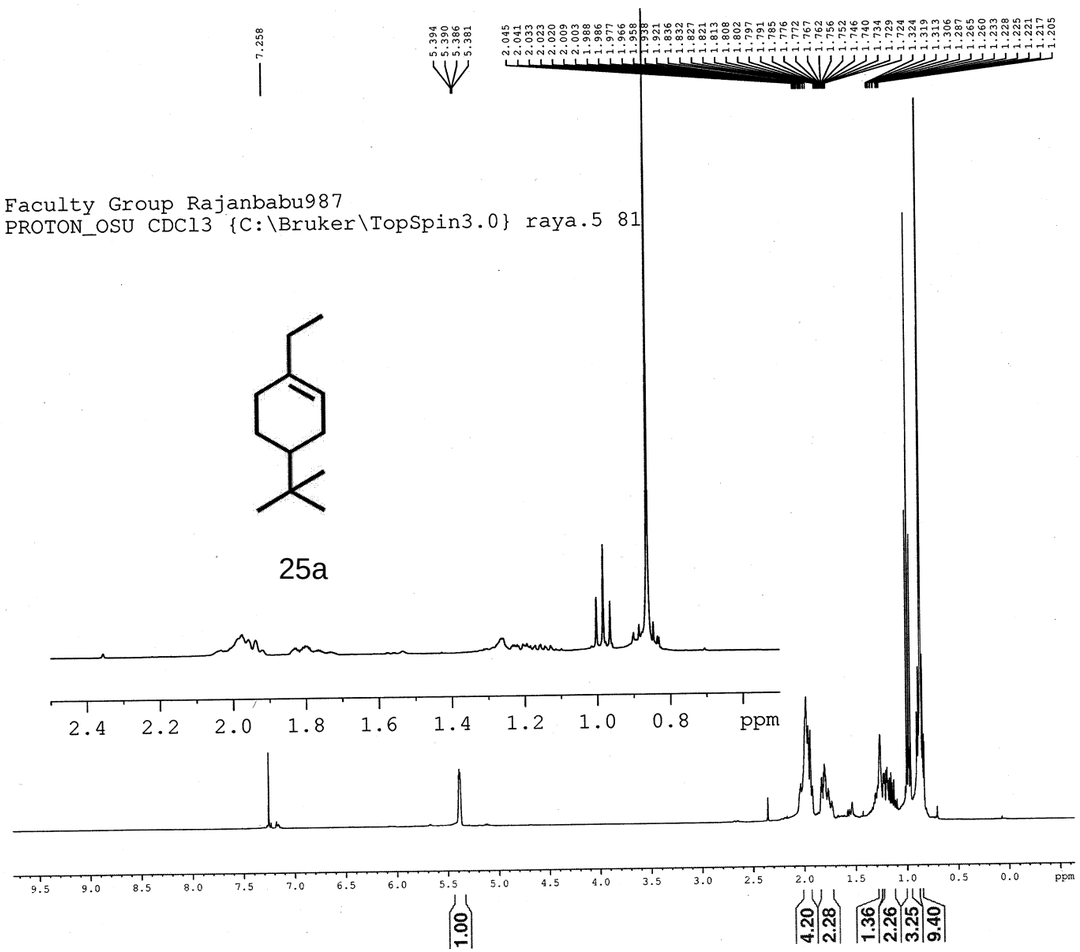


File :D:\MSDChemData\Babu\Balaram\BR-07-216 .D
Operator : BALARAM
Acquired : 31 Jul 2015 12:22 using AcqMethod 100-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-216
Misc Info :
Vial Number: 1



25a





BRUKER

Current Data Parameters
NAME BR-07-216 Final
EXPNO 1
PROCNO 1

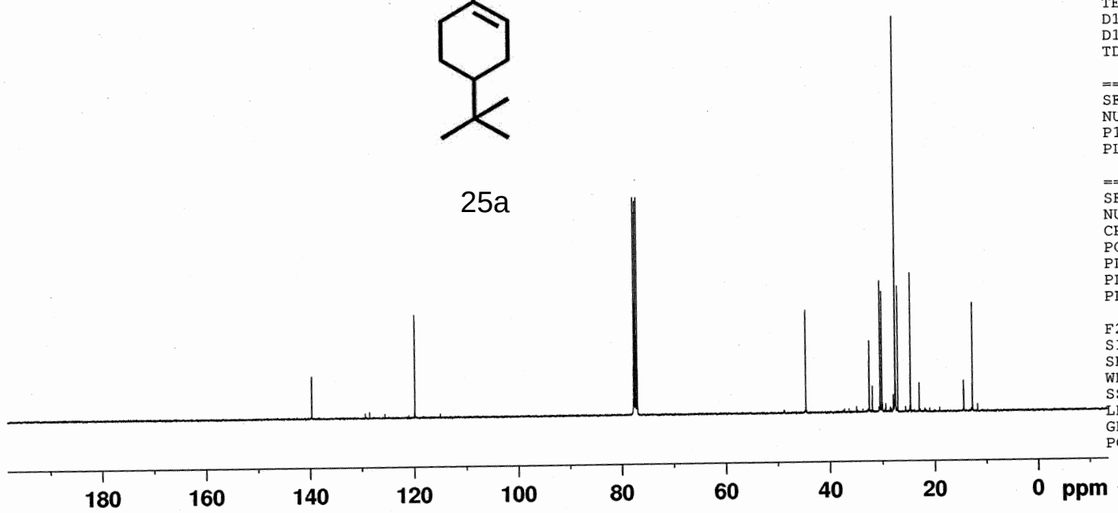
F2 - Acquisition Parameters
Date_ 20150827
Time 19.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 68.54
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1724712 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.19999981 W

F2 - Processing parameters
SI 65536
SF 400.1700105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Faculty Group Rajanbabu987
C13CPD CDCl3 {C:\Bruker\TopSpin3.0} raya.5 81

25a



BRUKER

Curr	
NAME	BR-07-216 Final
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20150828
Time	3.49
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631488 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	300.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

SFO1	100.6328883 MHz
NUC1	13C
P1	9.50 usec
PLW1	53.29999924 W

======== CHANNEL f2 ========

SFO2	400.1716007 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	14.60000038 W
PLW12	0.51327997 W
PLW13	0.32850000 W

F2 - Processing parameters

SI	32768
SF	100.6227953 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

```
*
BREAK

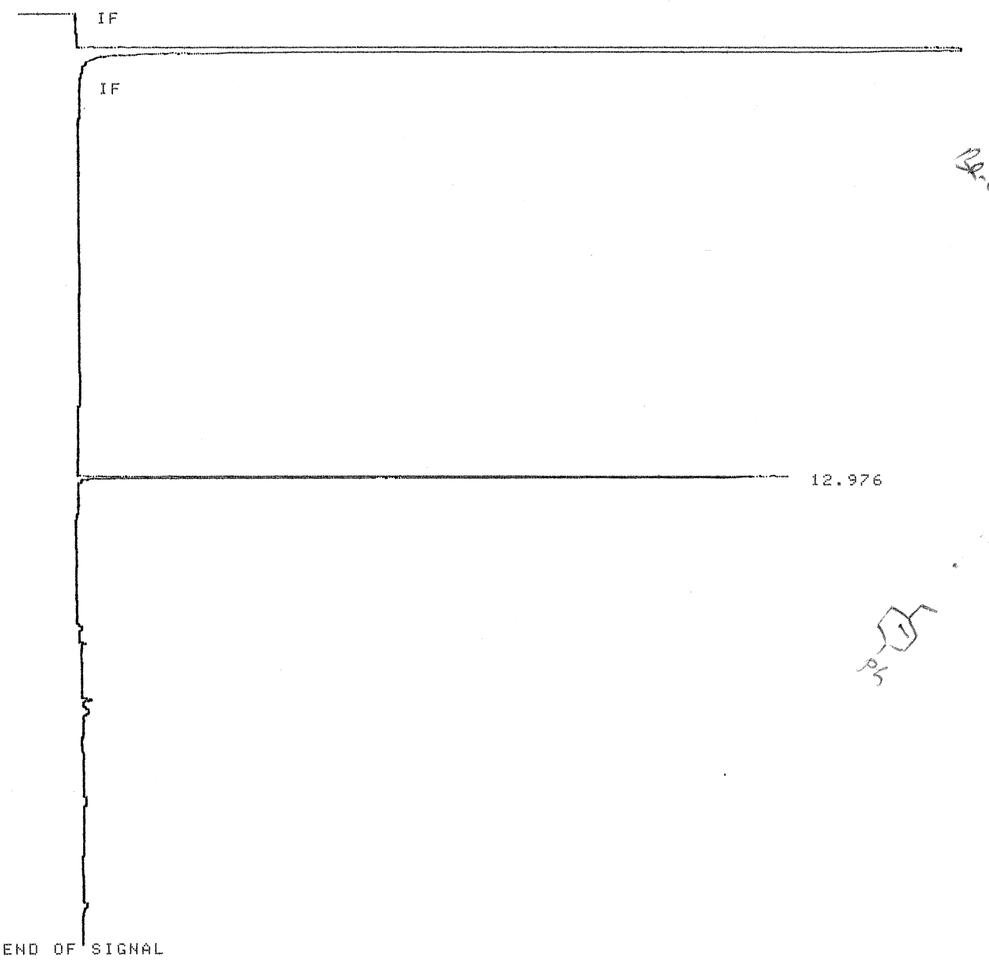
*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 1725      AUG  7, 1901   15:27:46
START
```



BR-07-212


```
END OF SIGNAL

Error storing signal to M:SIGNAL  .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL  .PRA

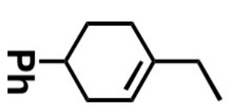
RUN#  1725          AUG  7, 1901   15:27:46

PEAK FILE  : M:SIGNAL.PRA
AREA%
      RT       AREA TYPE  WIDTH       AREA%
  12.976     782077  SBB    .031   100.00000

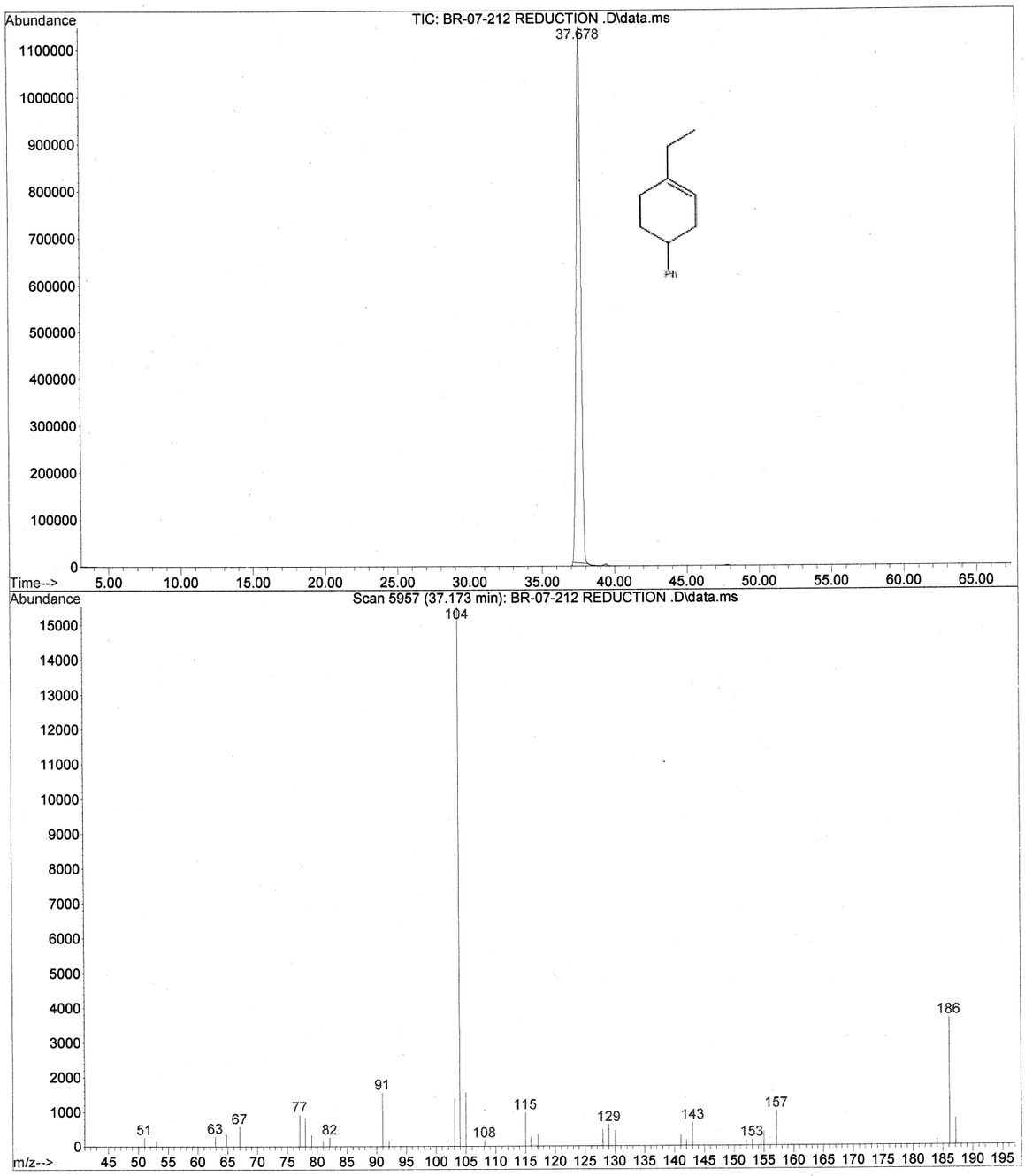
TOTAL AREA= 782077
MUL FACTOR=1.0000E+00
```

BR-07-212
100°C - 10 min
Int. 20°C
250°C - 40 min

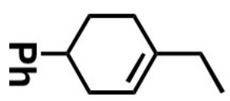
25b

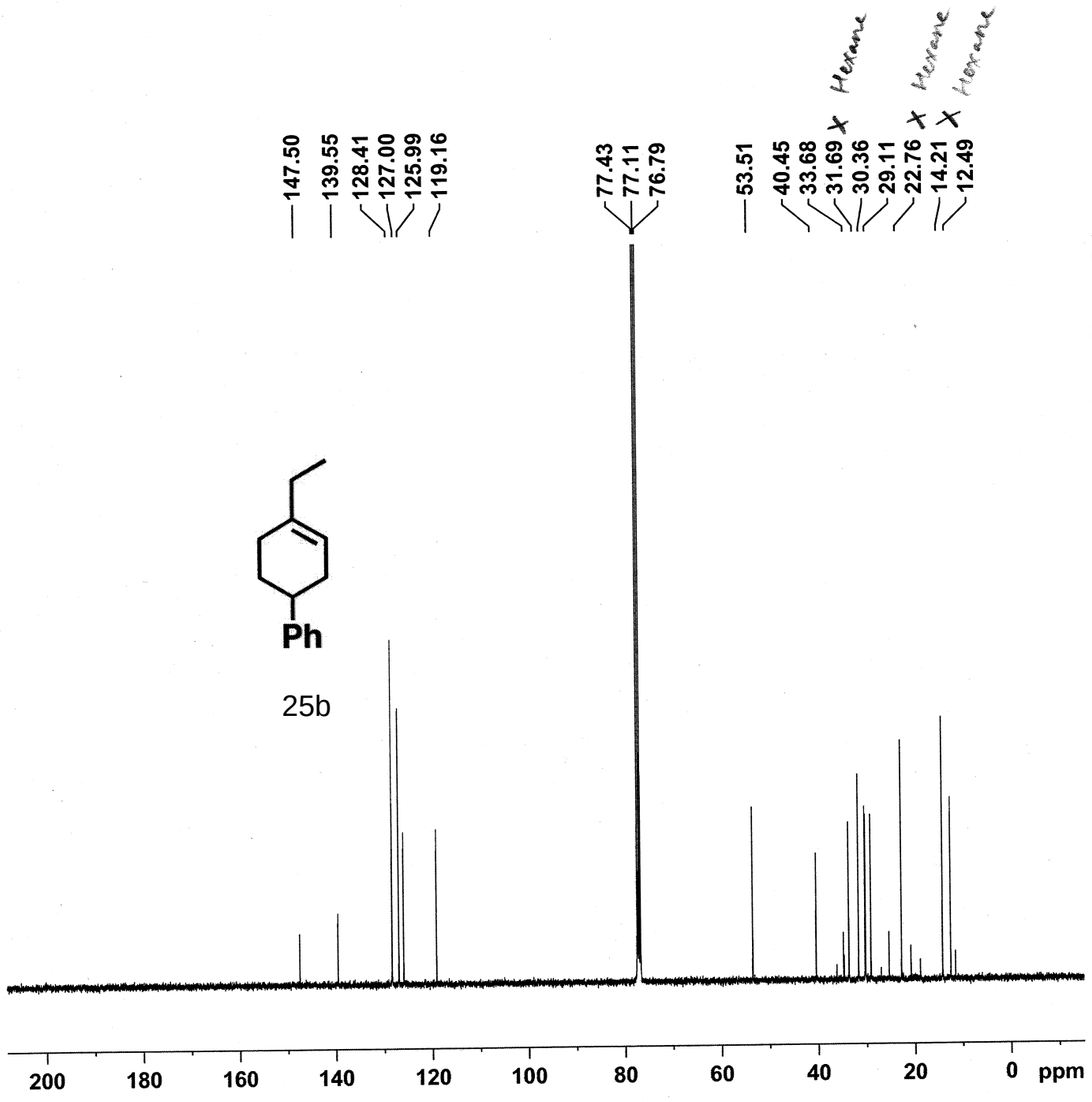


```
File       :D:\MSDChemData\Babu\Balaram\BR-07-212 REDUCTION .D
Operator   : BALARAM
Acquired   :  1 Aug 2015  16:32     using AcqMethod 100-ISOTHERM.M
Instrument :    GCMS
Sample Name: BR-07-212 REDUCTION
Misc Info  :
Vial Number: 1
```



25b





BRUKER

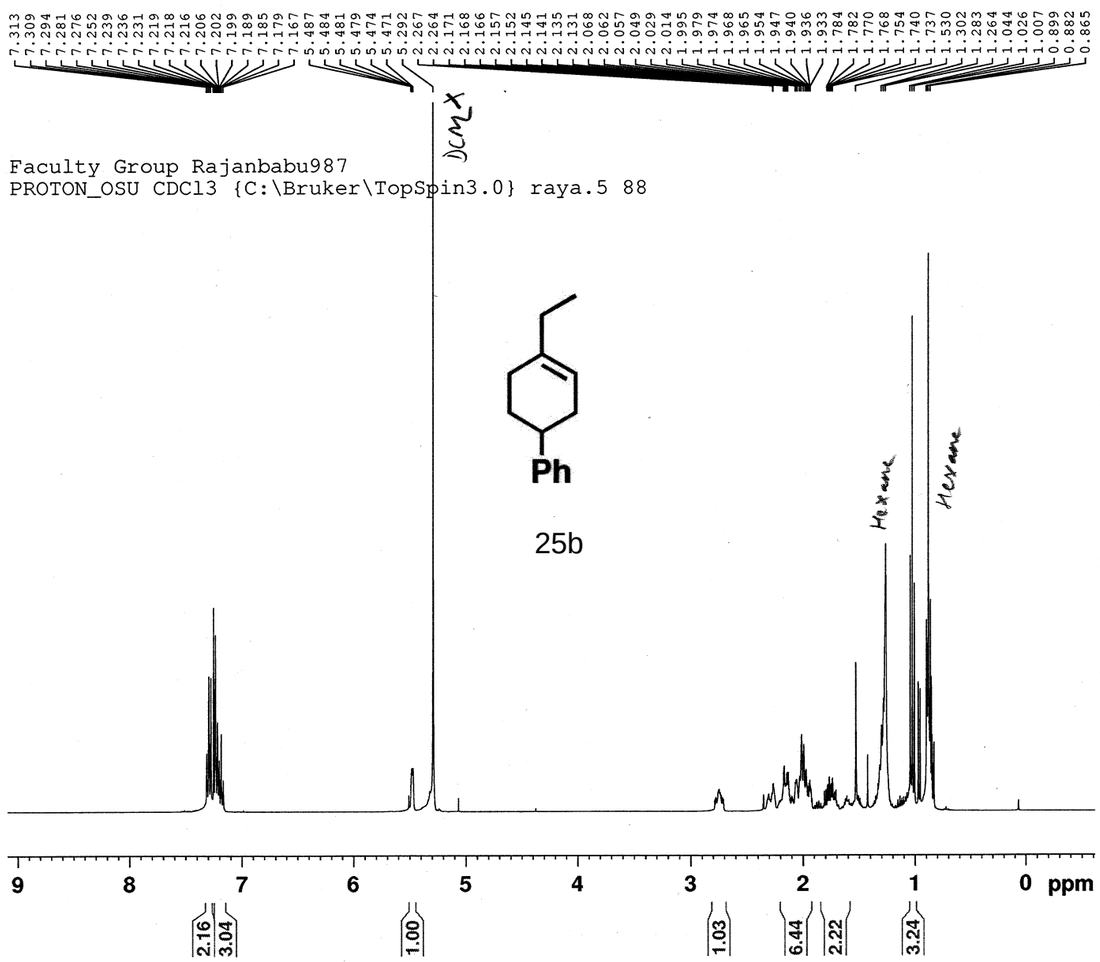
```
Cur:
NAME                BR-07-212 reduction
EXPNO                                 2
PROCNO                                1

F2 - Acquisition Parameters
Date_                          20150730
Time                               3.48
INSTRUM                           spect
PROBHD    5 mm PABBO BB/
PULPROG                         zgpg30
TD                               65536
SOLVENT                          CDCl3
NS                                2048
DS                                   4
SWH                        24038.461 Hz
FIDRES                      0.366798 Hz
AQ                          1.3631488 sec
RG                                2050
DW                           20.800 usec
DE                            6.50 usec
TE                           300.0 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                                  1

======== CHANNEL f1 ========
SFO1                     100.6328883 MHz
NUC1                              13C
P1                            9.50 usec
PLW1                     53.29999924 W

======== CHANNEL f2 ========
SFO2                     400.1716007 MHz
NUC2                              1H
CPDPRG[2                     waltz16
PCPD2                         80.00 usec
PLW2                     14.60000038 W
PLW12                     0.51327997 W
PLW13                     0.32850000 W

F2 - Processing parameters
SI                                32768
SF                       100.6228168 MHz
WDW                                  EM
SSB       0
LB                             1.00 Hz
GB        0
PC                             1.40
```



Faculty Group Rajanbabu987
PROTON_OSU CDCl3 {C:\Bruker\TopSpin3.0} raya.5 88

```
Cur...  ...  ...
NAME      BR-07-212 reduction
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20150729
Time              14.34
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  64
DS                   2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9845889 sec
RG               120.36
DW               60.800 usec
DE                6.50 usec
TE               300.0 K
D1           1.00000000 sec
TD0                  1

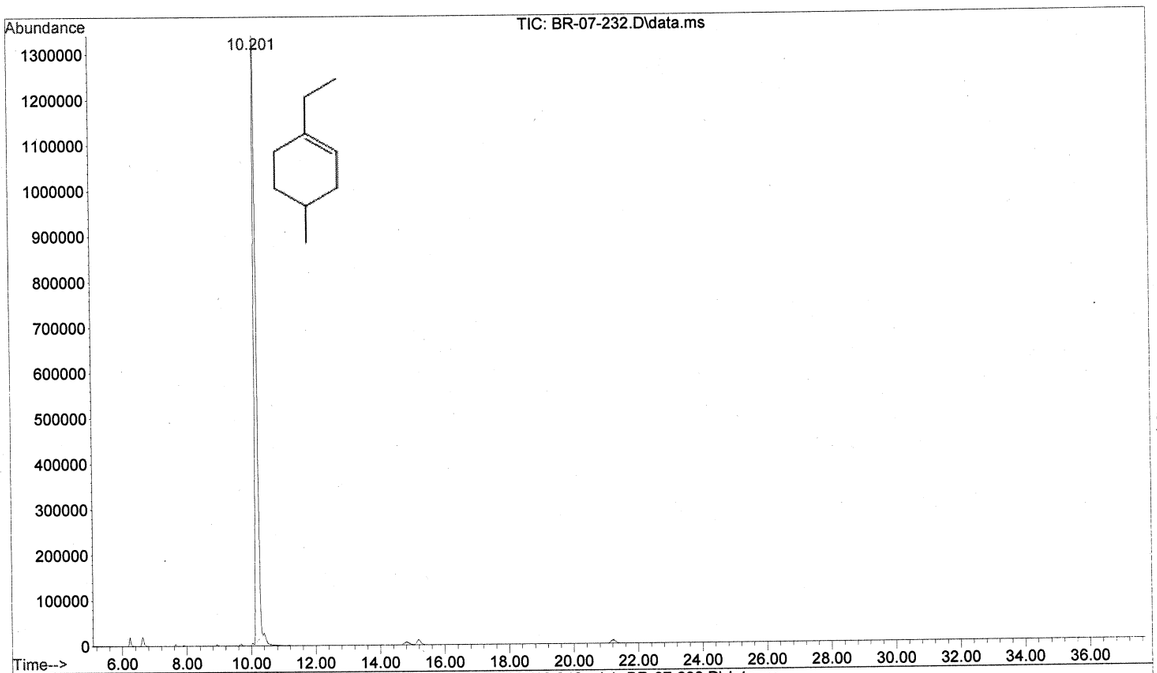
======== CHANNEL f1 ========
SFO1        400.1724712 MHz
NUC1               1H
P1               15.00 usec
PLW1        13.19999981 W

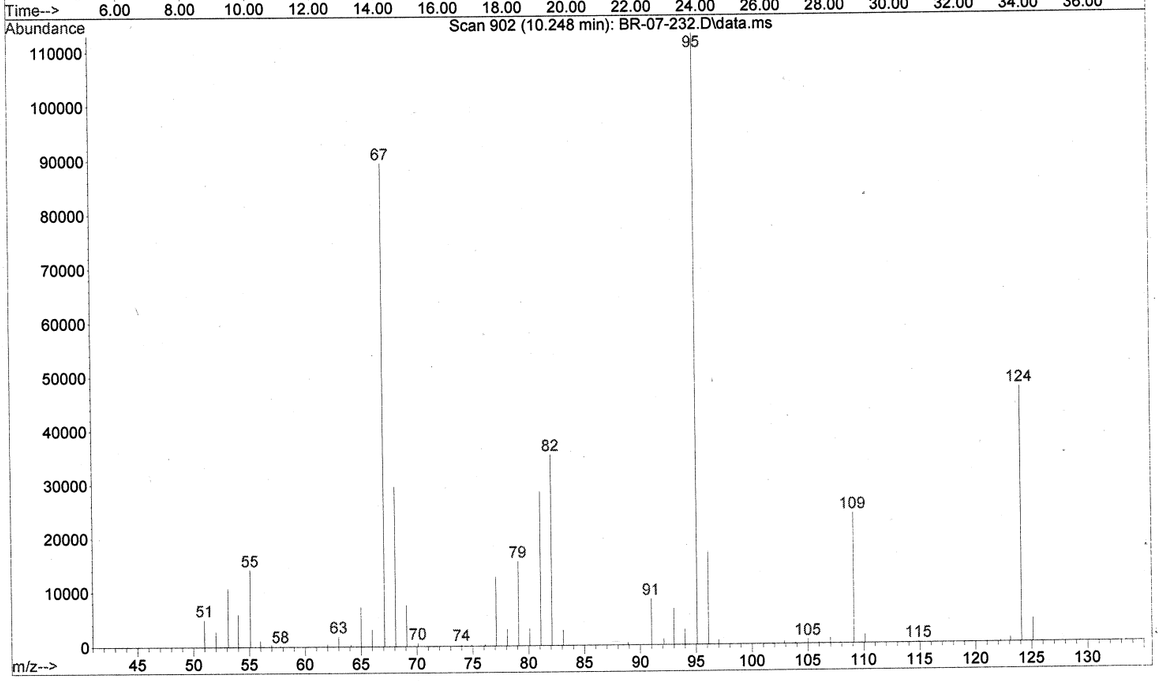
F2 - Processing parameters
SI               65536
SF          400.1700129 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```

* BREAK

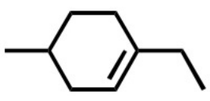
* BREAK

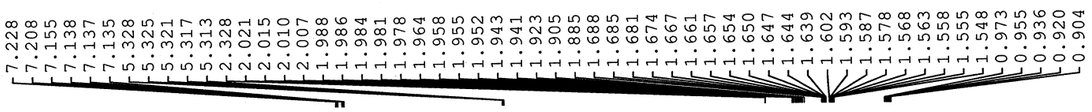
File :D:\MSDChemData\Babu\Balaram\BR-07-232.D
Operator : BALARAM
Acquired : 2 Sep 2015 14:59 using AcqMethod 40-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-232
Misc Info :
Vial Number: 1

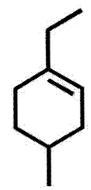




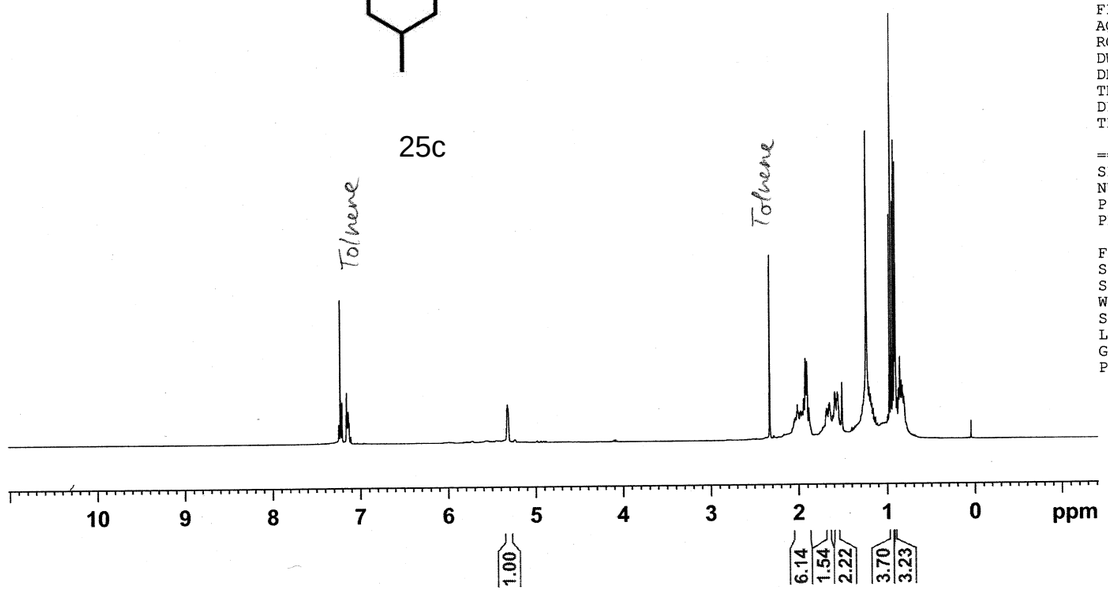
25c







25c

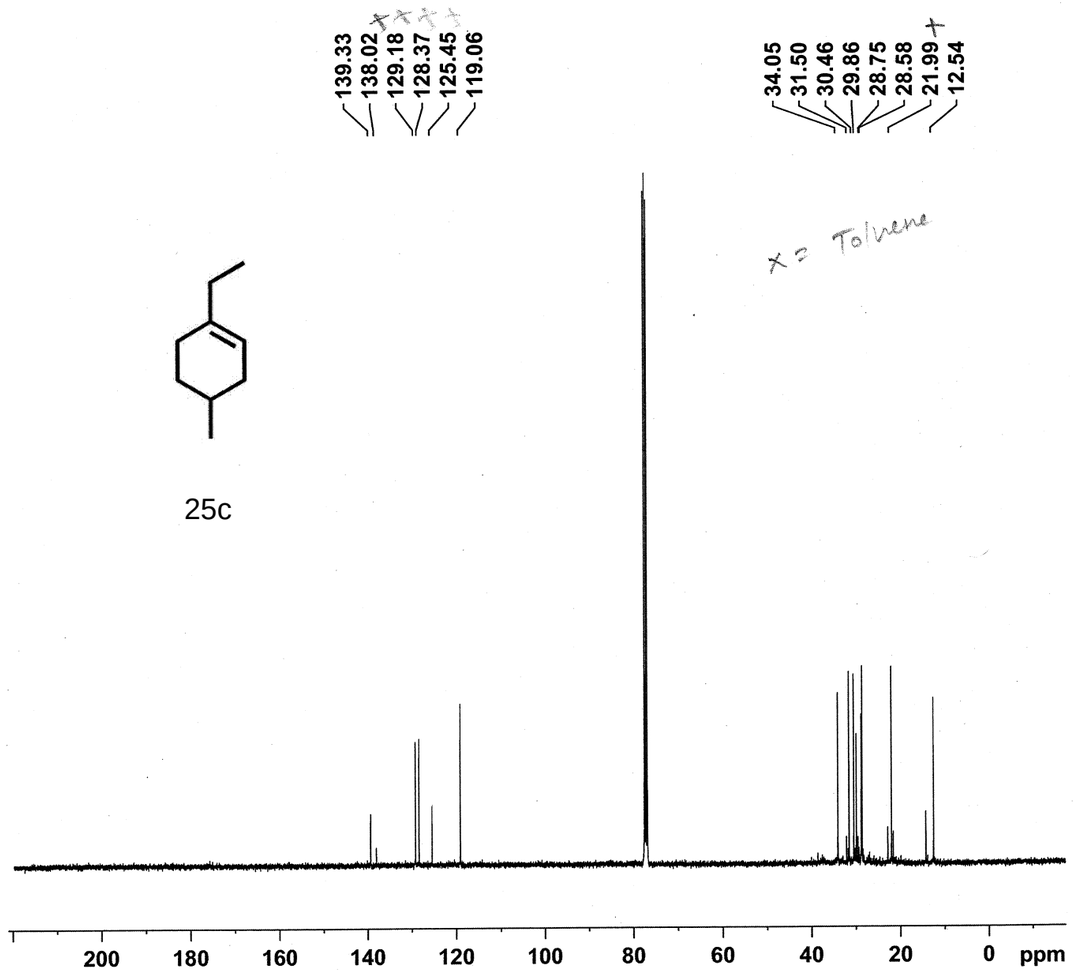



```
Curr
NAME                BR-07-232 Second
EXPNO                             1
PROCNO                            1

F2 - Acquisition Parameters
Date_                      20150904
Time                          12.24
INSTRUM                       spect
PROBHD    5 mm PABBO BB/
PULPROG                       zg30
TD                           65536
SOLVENT                      CDCl3
NS                              64
DS                               2
SWH                       8223.685 Hz
FIDRES                    0.125483 Hz
AQ                        3.9845889 sec
RG                            99.77
DW                           60.800 usec
DE                            6.50 usec
TE                           300.0 K
D1                     1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
SFO1                 400.1724712 MHz
NUC1                           1H
P1                           15.00 usec
PLW1                13.19999981 W

F2 - Processing parameters
SI                           65536
SF                   400.1700226 MHz
WDW                             EM
SSB                 0
LB                            0.30 Hz
GB                  0
PC                            1.00
```



25c

```
Curre
NAME        BR-07-232 Second
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20150905
Time                   5.12
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     2048
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631488 sec
RG                     1820
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6328883 MHz
NUC1                    13C
P1                    9.50 usec
PLW1            53.29999924 W

======== CHANNEL f2 ========
SFO2            400.1716007 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                80.00 usec
PLW2            14.60000038 W
PLW12            0.51327997 W
PLW13            0.32850000 W

F2 - Processing parameters
SI                     32768
SF              100.6228118 MHz
WDW                       EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

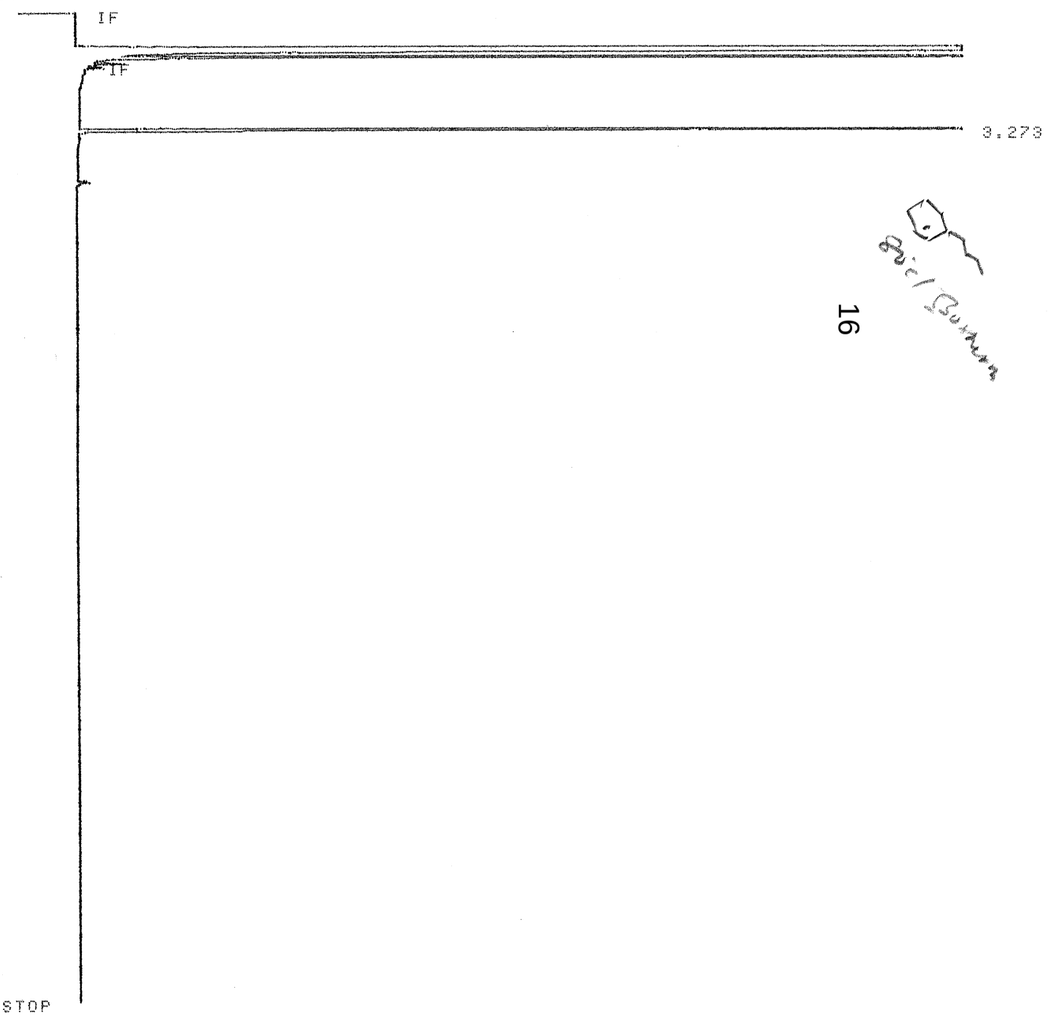
```
*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 2203      SEP 25, 1901   17:24:51
START
```




```
STOP

Error storing signal to M:SIGNAL   .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL   .PRA

RUN#  2203          SEP 25, 1901   17:24:51

PEAK FILE  : M:SIGNAL.PRA
AREA%
      RT       AREA TYPE  WIDTH       AREA%
   3.273    2292638  SPB    .038   100.00000

TOTAL AREA=2292638
MUL FACTOR=1.0000E+00


*
BREAK

*
BREAK
```

16

Boc / Boehm

16

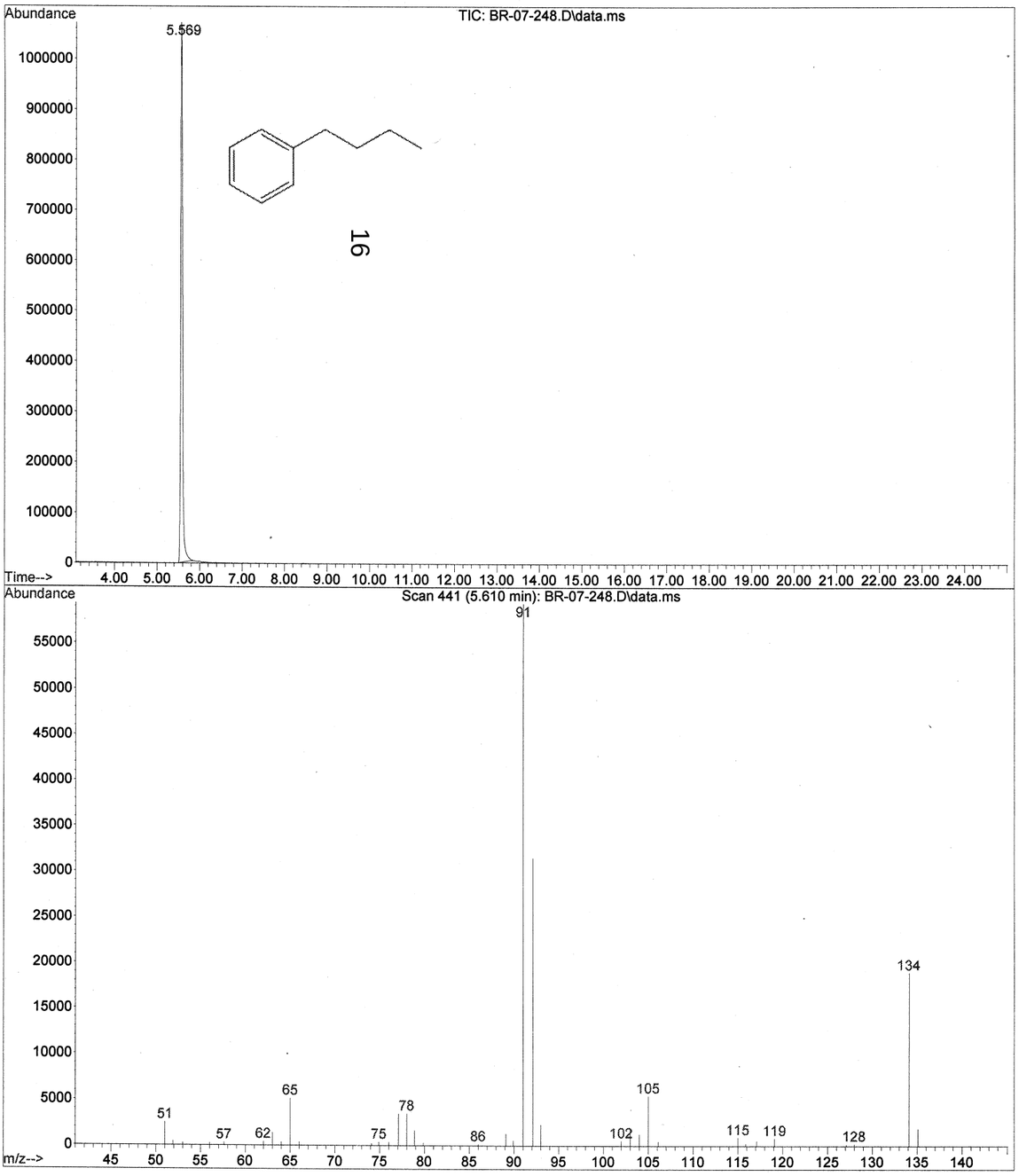
BR-08-248

Boc / Boehm

(crude 0.001 equiv. catalyst)

16

File :D:\MSDChemData\Babu\Balaram\BR-07-248.D
Operator : BALARAM
Acquired : 16 Sep 2015 12:19 using AcqMethod 80-Isotherm.M
Instrument : GCMS
Sample Name: BR-07-248
Misc Info :
Vial Number: 1



```
*
BREAK

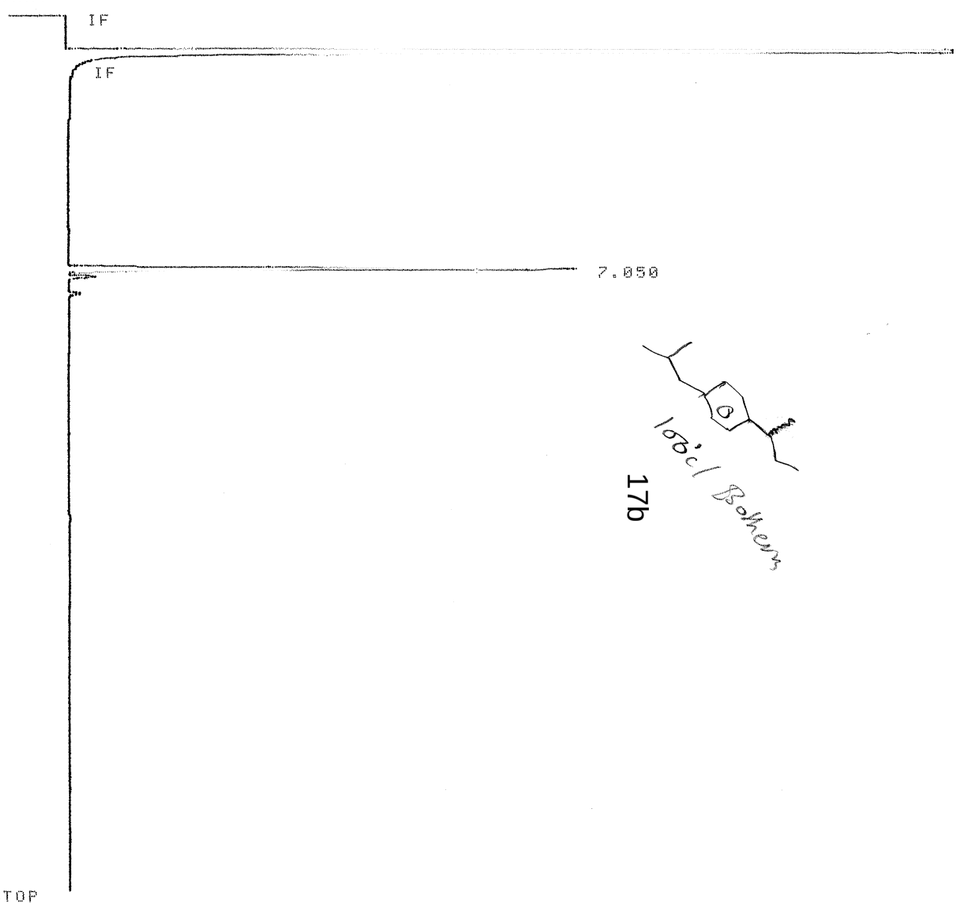
*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 2250      OCT  2, 1901  15:55:26
START
```



17b

BR-07-244
10Dic1 Bohrem

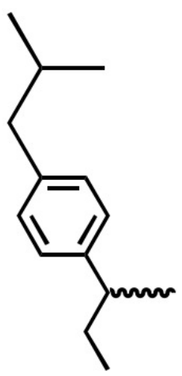
```
STOP

Error storing signal to M:SIGNAL  .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL  .PRA

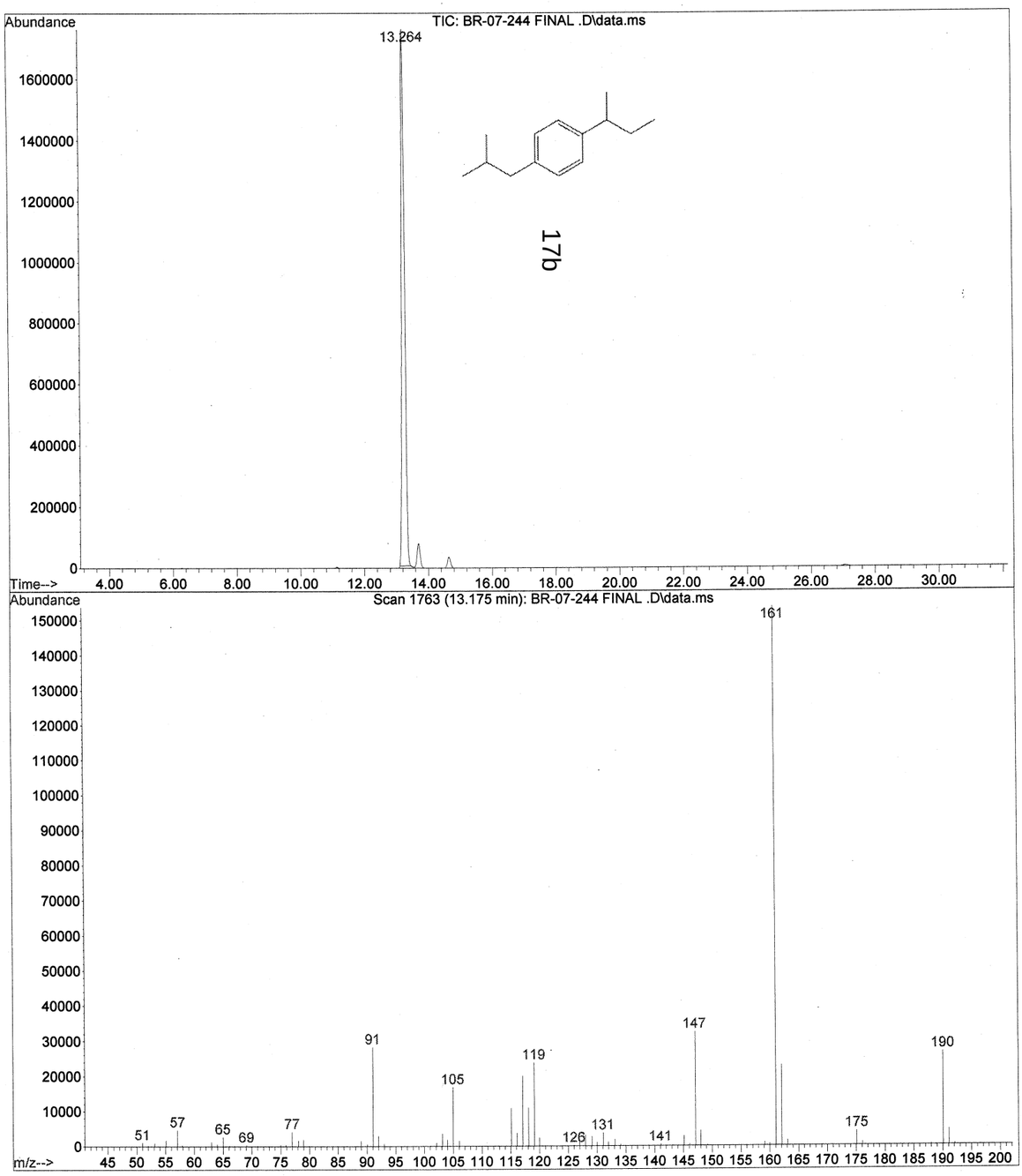
RUN#  2250          OCT  2, 1901  15:55:26

PEAK FILE  : M:SIGNAL.PRA
AREA%
      RT       AREA TYPE  WIDTH      AREA%
   7.050   2665261   PB   .075  100.00000

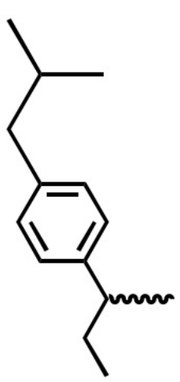
TOTAL AREA-2665261
MUL FACTOR-1.0000E+00
```

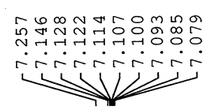


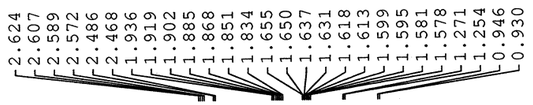
File :D:\MSDChemData\Babu\Balaram\BR-07-244 FINAL .D
Operator : BALARAM
Acquired : 23 Sep 2015 10:42 using AcqMethod 100-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-244 FINAL
Misc Info :
Vial Number: 1



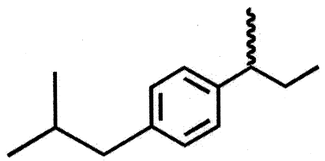
17b



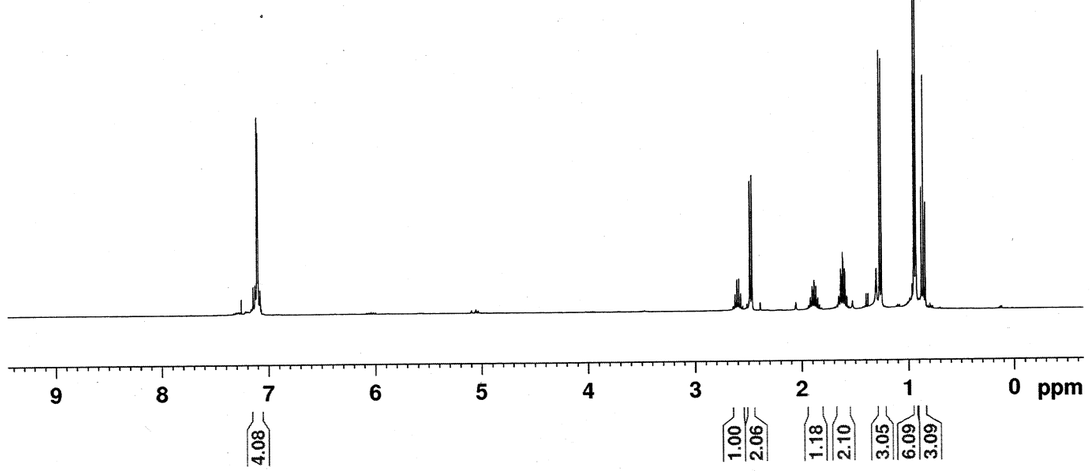




Faculty Group Rajanbabu987
PROTON_OSU CDCl3 {C:\Bruker\TopSpin3.0} raya.5 1



17b



Current Data Parameters
NAME BR-07-244
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150911
Time 23.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 32.11
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

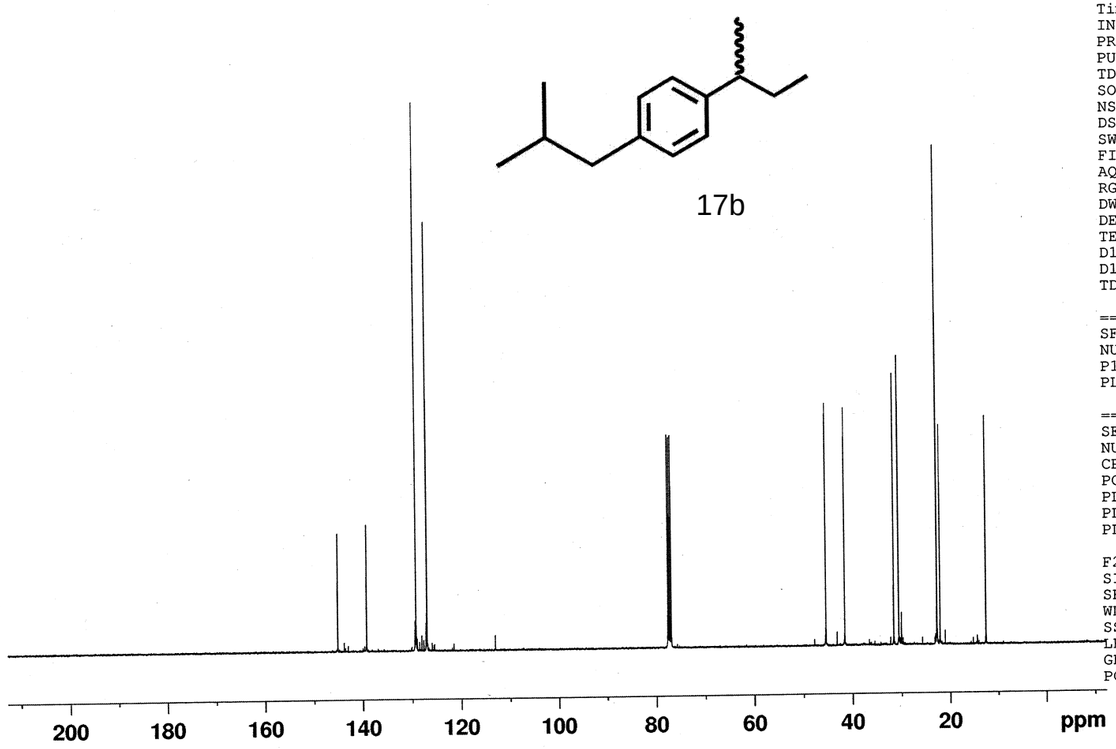
======== CHANNEL f1 ========
SFO1 400.1724712 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.19999981 W

F2 - Processing parameters
SI 65536
SF 400.1700108 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

145.19
139.28
129.25
127.02
77.64
77.32
77.00
45.41
41.57
31.58
30.56
22.75
22.12
12.58

Faculty Group Rajanbabu987
C13CPD CDCl3 {C:\Bruker\TopSpin3.0} raya.5 1

17b



BRUKER

Current Data Parameters
NAME BR-07-244
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150912
Time 1.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 1030
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6328883 MHz
NUC1 13C
P1 9.50 usec
PLW1 53.29999924 W

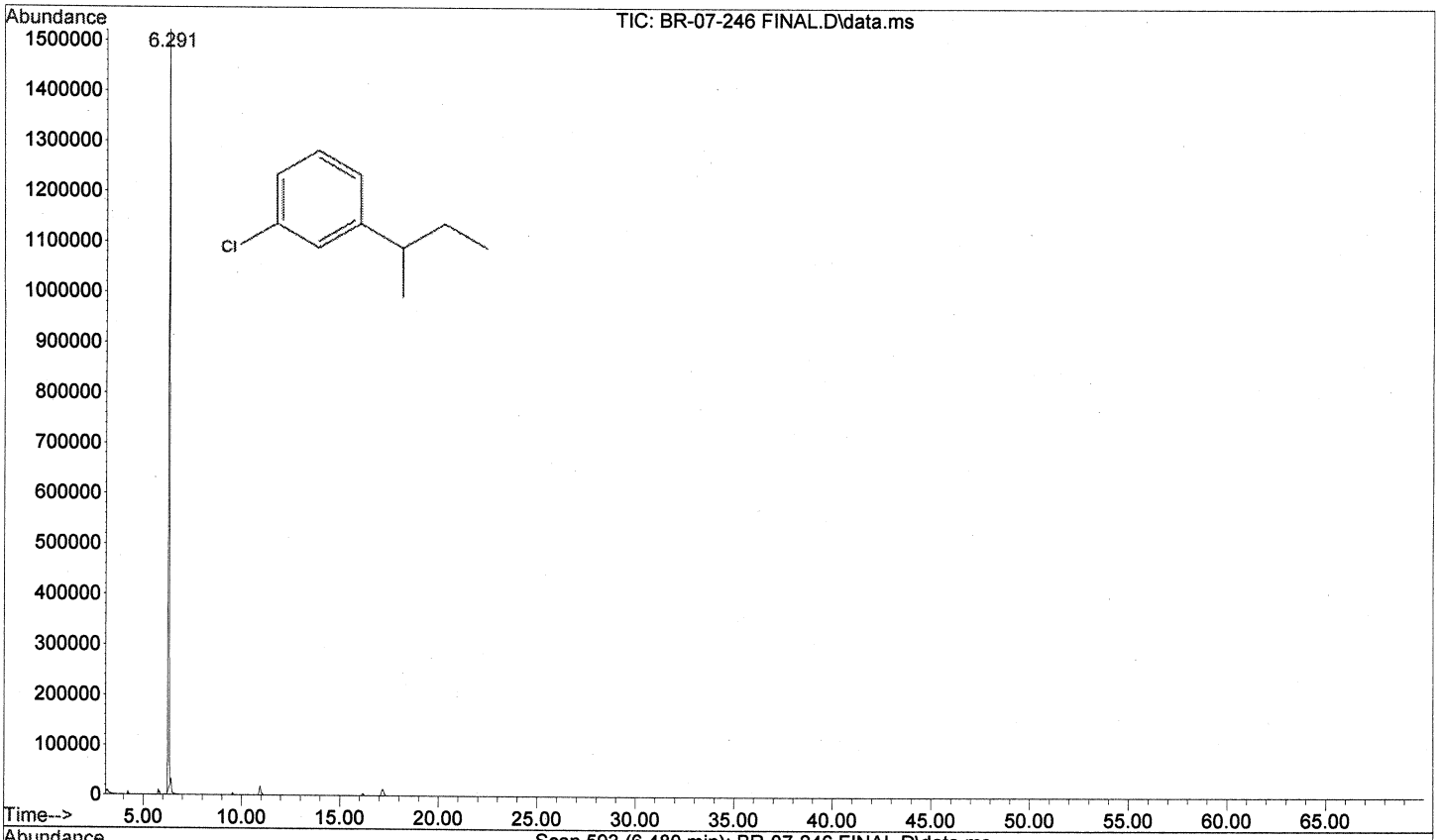
======== CHANNEL f2 ========
SFO2 400.1716007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 14.60000038 W
PLW12 0.51327997 W
PLW13 0.32850000 W

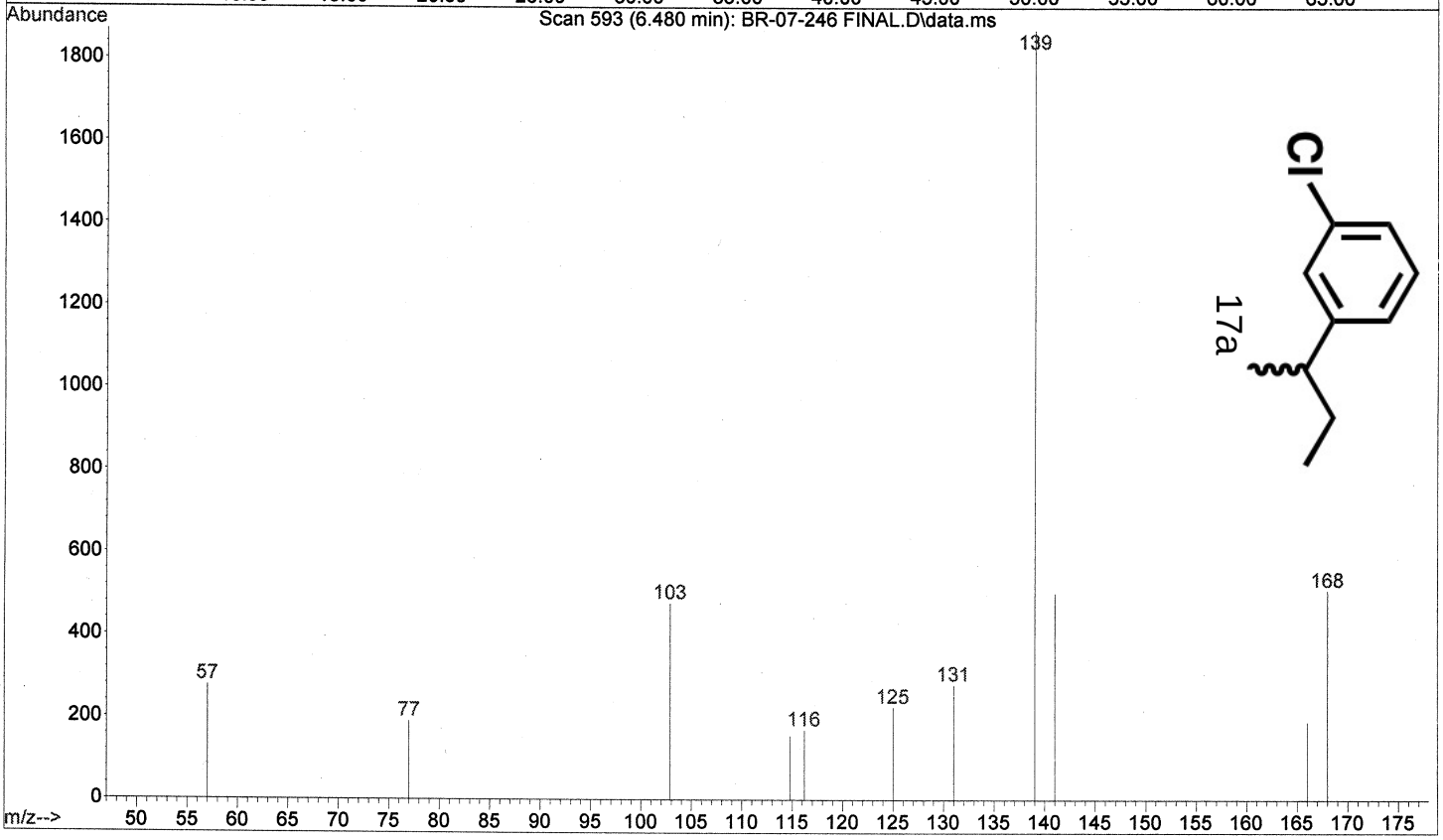
F2 - Processing parameters
SI 32768
SF 100.6227979 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

BQ-07-246
100°C / 80 mmHg

17a

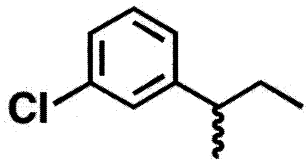
File :D:\MSDChemData\Babu\Balaram\BR-07-246 FINAL.D
Operator : BALARAM
Acquired : 22 Sep 2015 20:07 using AcqMethod 100-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-246 Final
Misc Info :
Vial Number: 1





17a

Faculty Group Rajanbabu987
PROTON_OSU CDCl3 {C:\Bruker\TopSpin3.0} raya.5 44



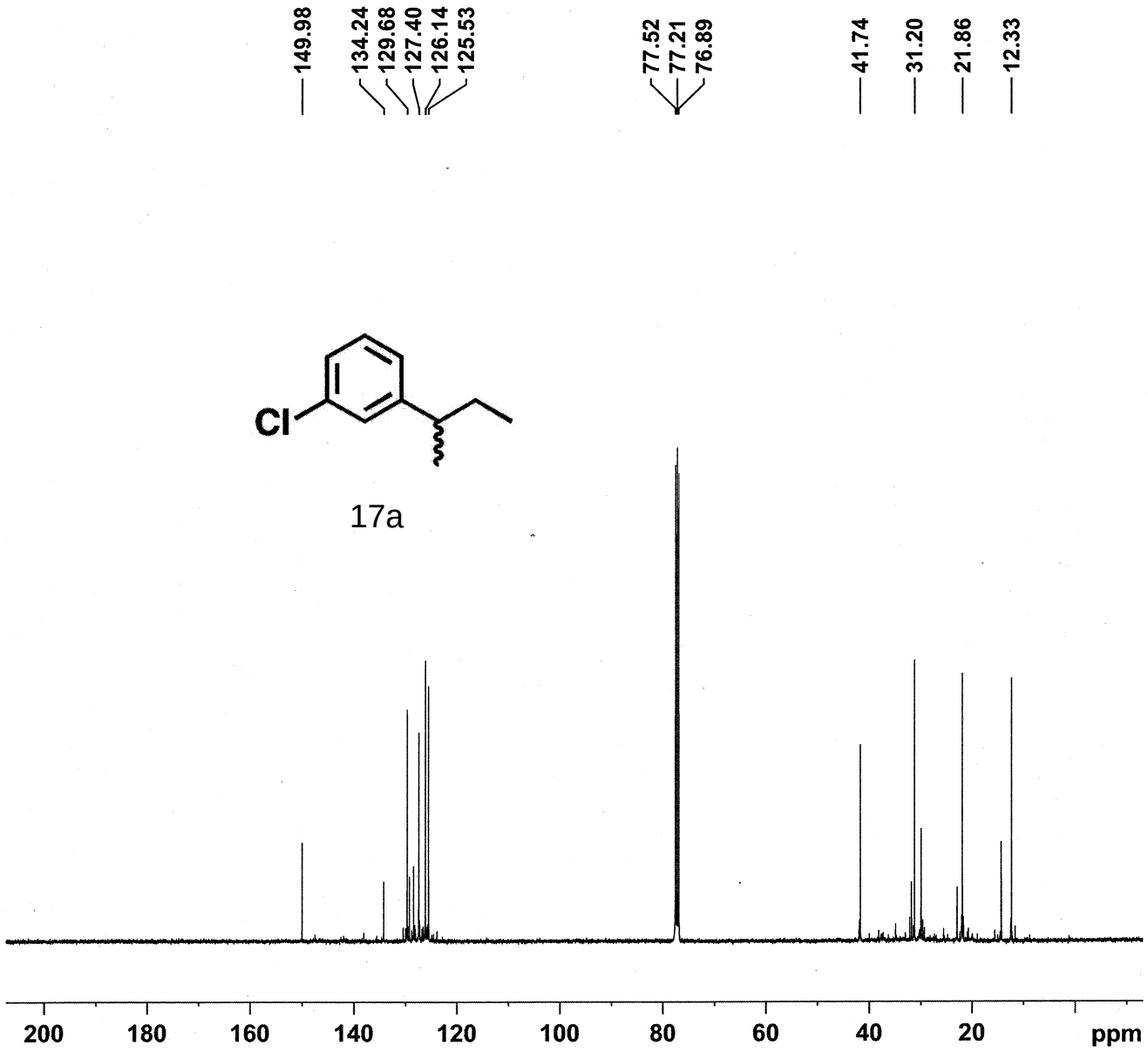
17a

Current Data Parameters
NAME BR-07-246
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150912
Time 1.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 61.71
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1724712 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.19999981 W

F2 - Processing parameters
SI 65536
SF 400.1700112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



17a

```
Current Data Parameters
NAME                  BR-07-246
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150912
Time                       3.35
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        2048
DS                           4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                        2050
DW                      20.800 usec
DE                       6.50 usec
TE                      300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               100.6328883 MHz
NUC1                       13C
P1                        9.50 usec
PLW1               53.29999924 W

======== CHANNEL f2 ========
SFO2               400.1716007 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2               14.60000038 W
PLW12               0.51327997 W
PLW13               0.32850000 W

F2 - Processing parameters
SI                        32768
SF                 100.6228079 MHz
WDW                          EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```

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*
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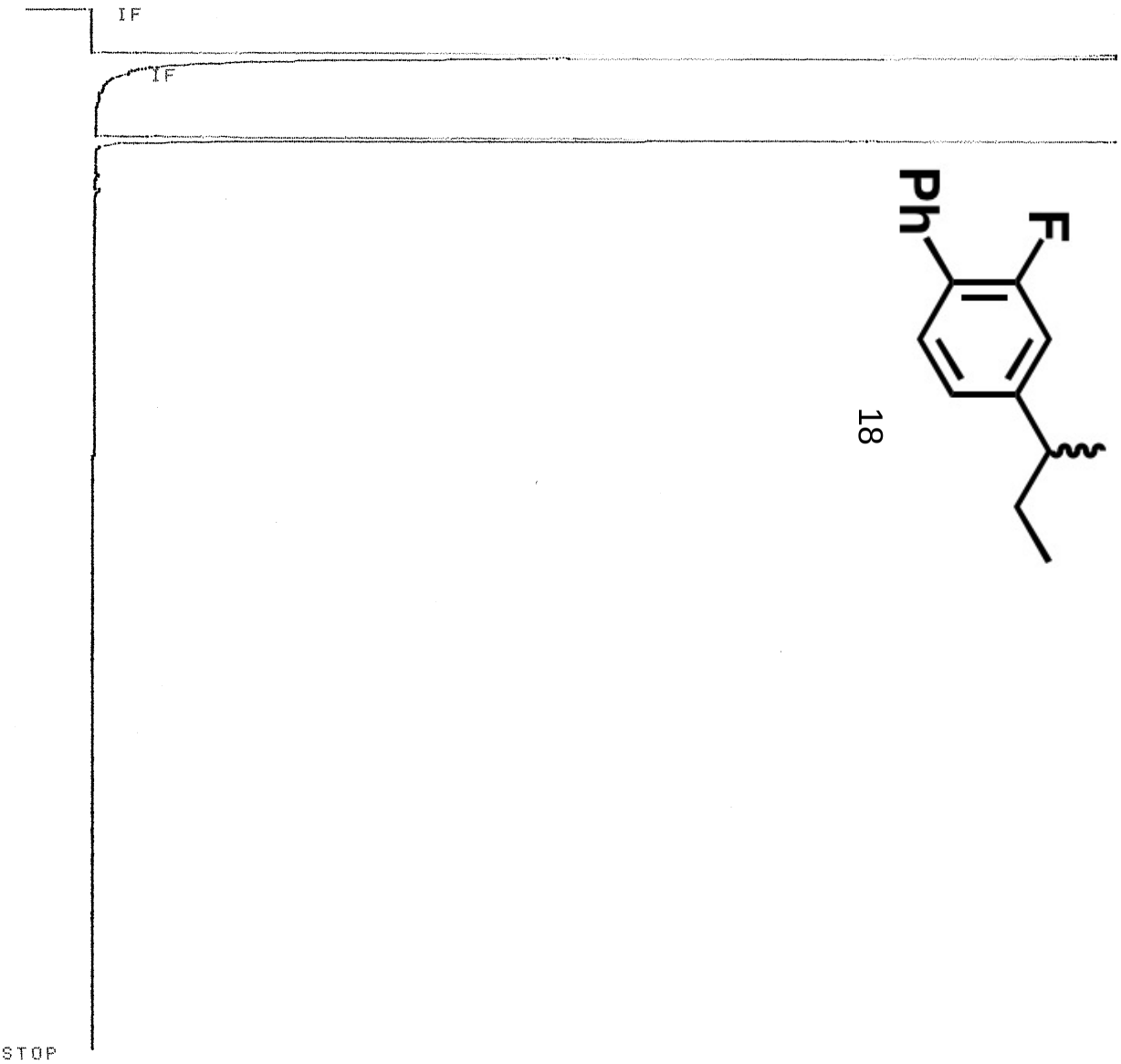
*
*
*AN
RUN # 2251      OCT  2, 1901  16:52:02
START
```



18

```
STOP

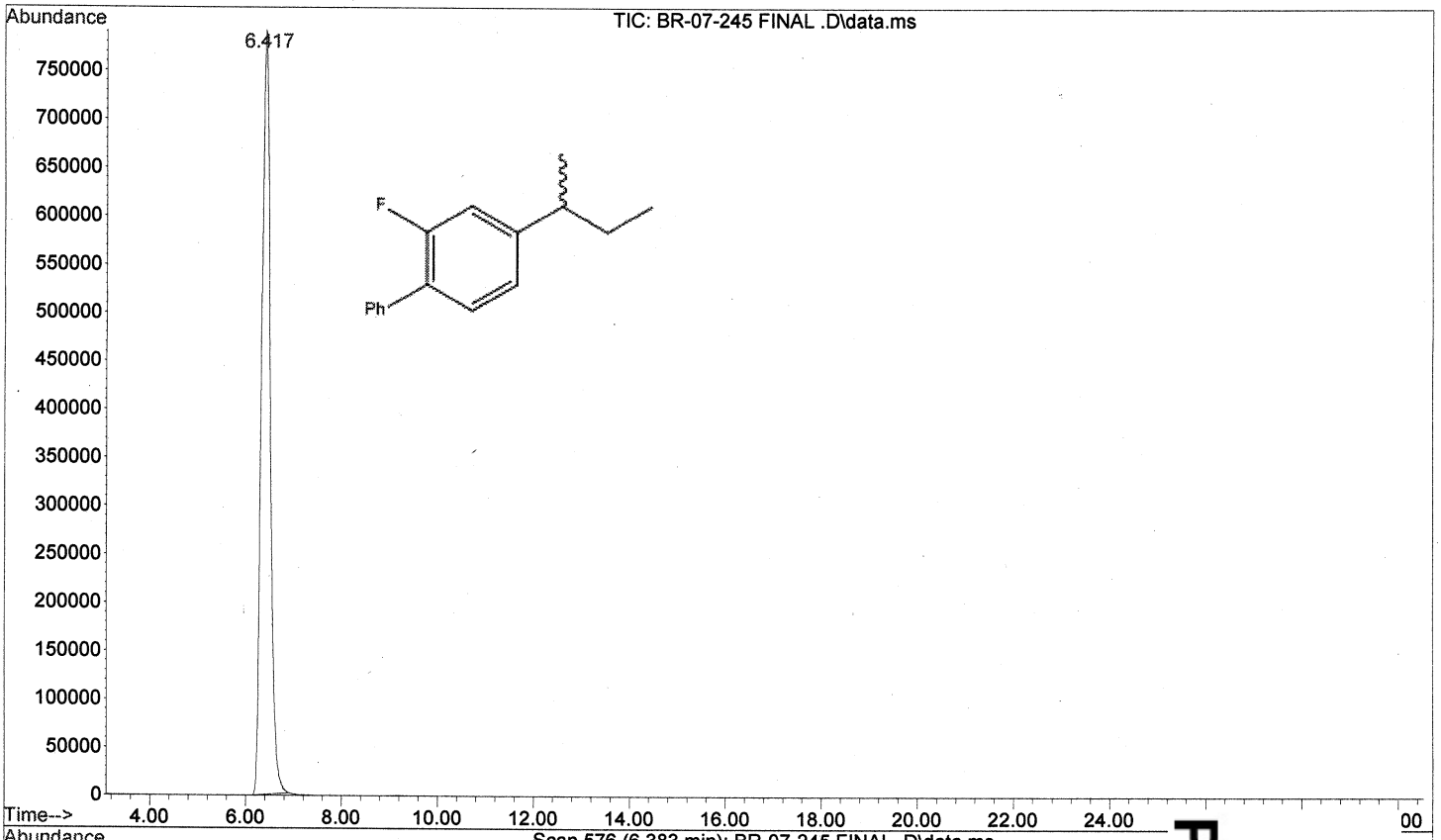
Error storing signal to M:SIGNAL  .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL  .PRA

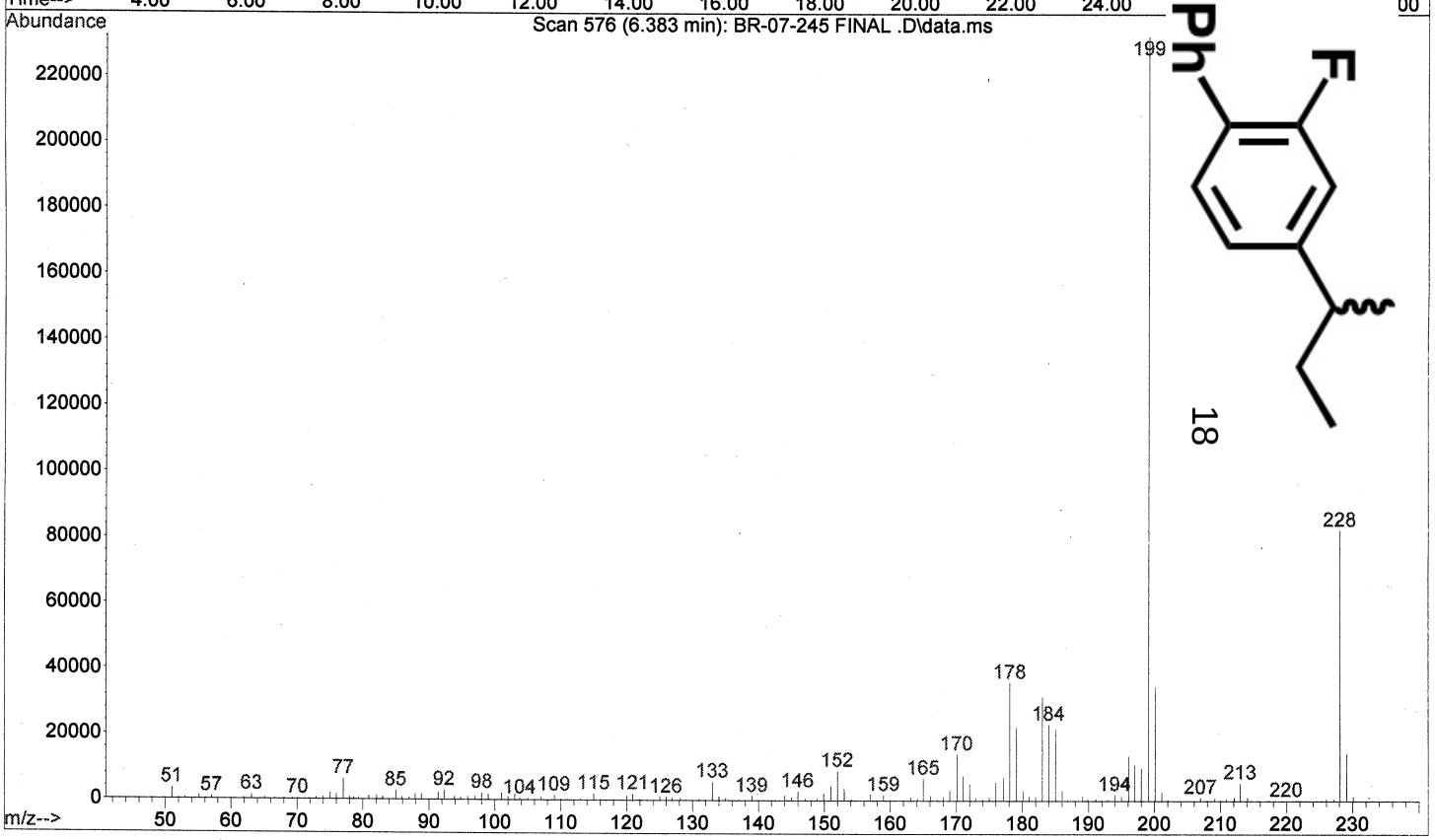
RUN#  2251          OCT  2, 1901  16:52:02

PEAK FILE  : M:SIGNAL.PRA
AREA%
      RT       AREA TYPE  WIDTH       AREA%
```

BR-07-2
180°C / 13

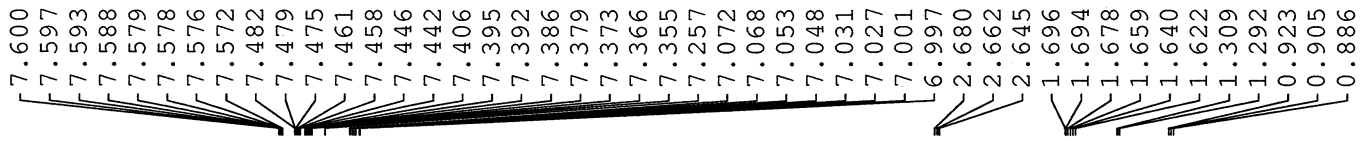
File :D:\MSDChemData\Babu\Balaram\BR-07-245 FINAL .D
Operator : BALARAM
Acquired : 23 Sep 2015 11:43 using AcqMethod 180-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-245 FINAL
Misc Info :
Vial Number: 1



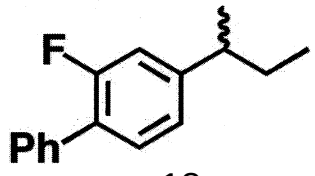


Ph F

18



Faculty Group Rajanbabu987
PROTON_OSU CDCl3 {C:\Bruker\TopSpin3.0} raya.5 26



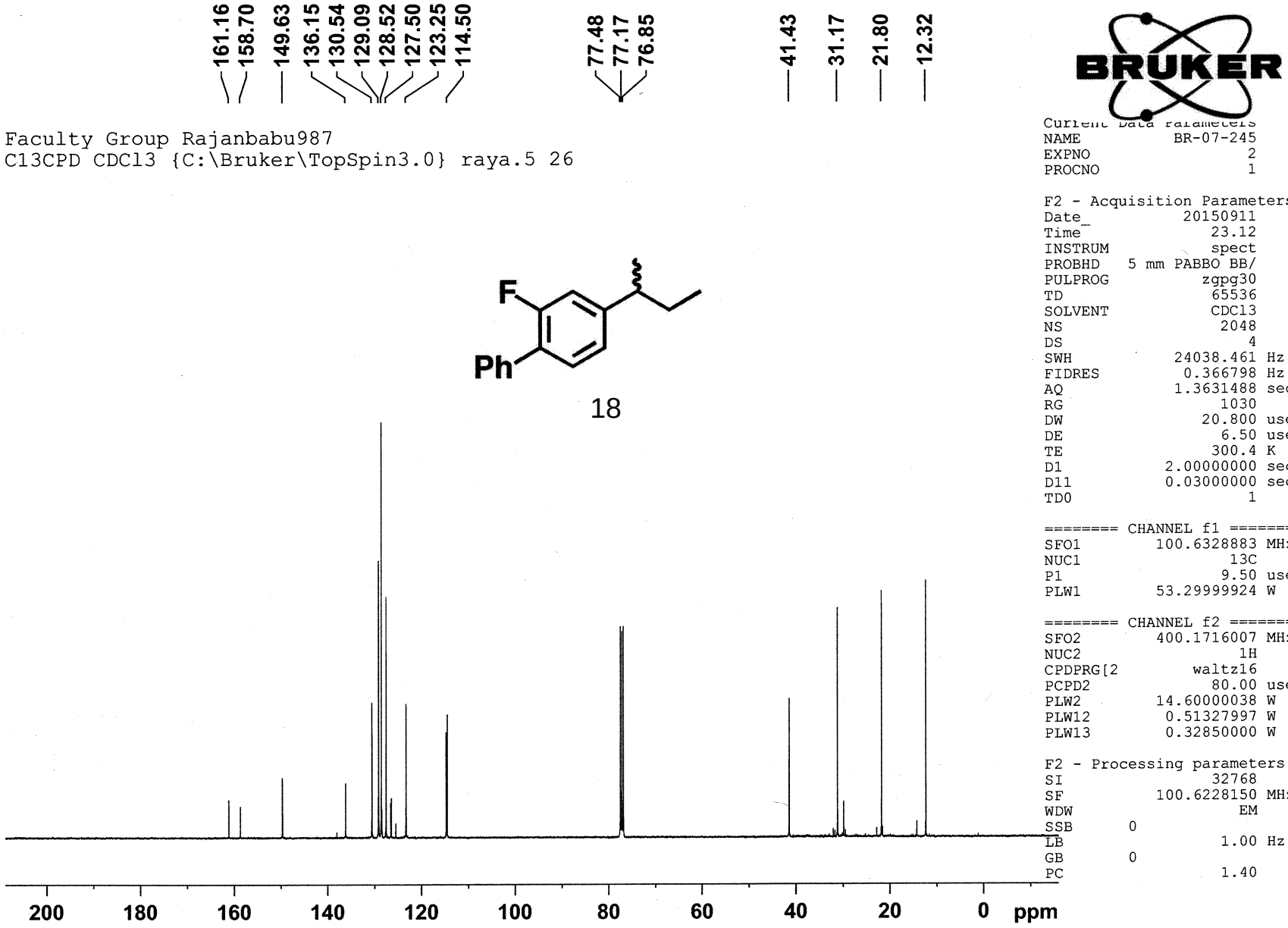
18

Current Data Parameters
NAME BR-07-245
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150911
Time 21.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 32.11
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1724712 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.19999981 W

F2 - Processing parameters
SI 65536
SF 400.1700110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


161.16
158.70
149.63
136.15
130.54
129.09
128.52
127.50
123.25
114.50
77.48
77.17
76.85
41.43
31.17
21.80
12.32
Faculty Group Rajanbabu987
C13CPD CDCl3 {C:\Bruker\TopSpin3.0} raya.5 26
F
Ph
18
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME BR-07-245
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150911
Time 23.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 1030
DW 20.800 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6328883 MHz
NUC1 13C
P1 9.50 usec
PLW1 53.29999924 W
======== CHANNEL f2 ========
SFO2 400.1716007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 14.60000038 W
PLW12 0.51327997 W
PLW13 0.32850000 W
F2 - Processing parameters
SI 32768
SF 100.6228150 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

BREAK

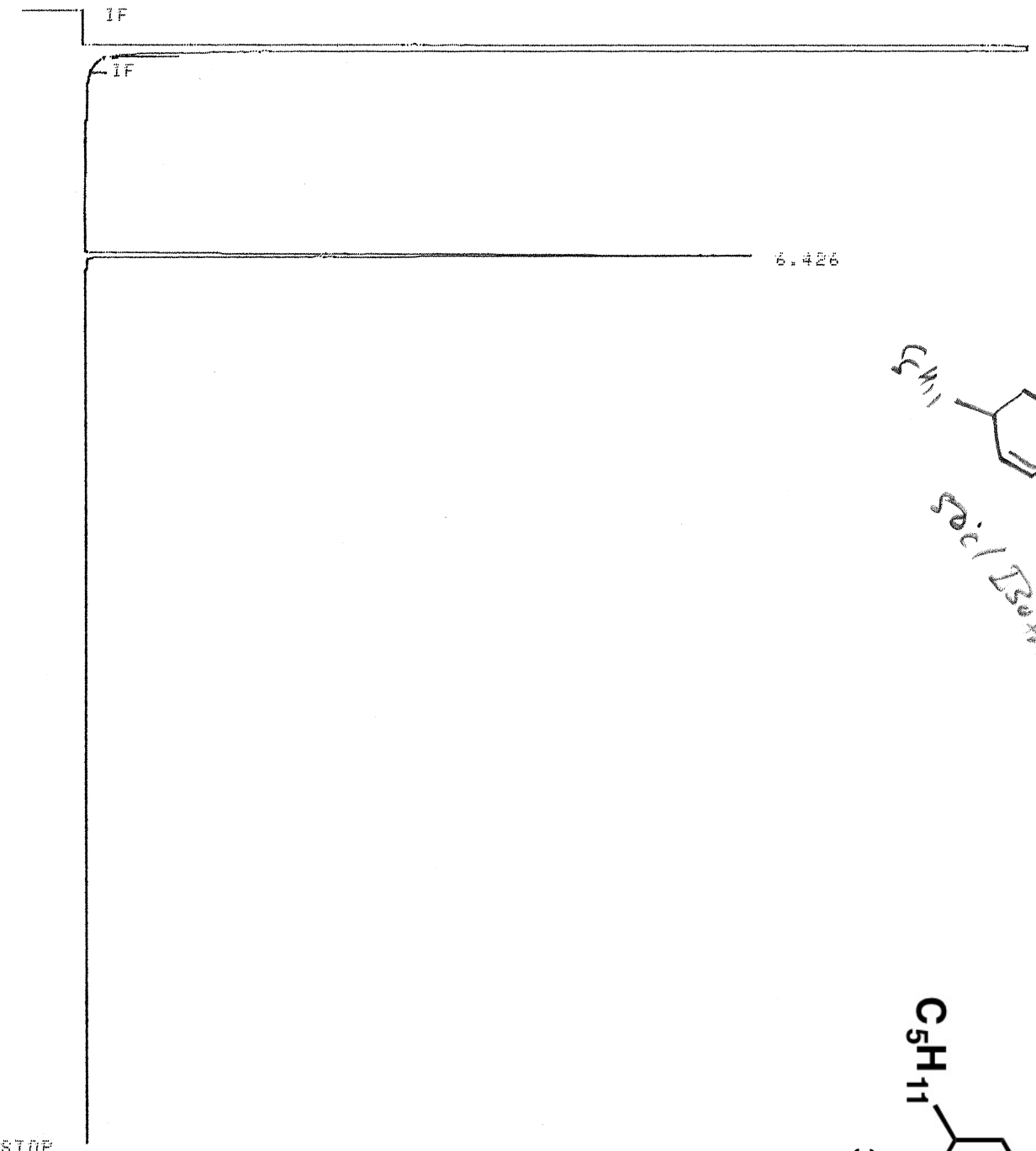
*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 2116 SEP 13, 1901 22:36:45
START



STOP

Error storing signal to M:SIGNAL .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL .PRA

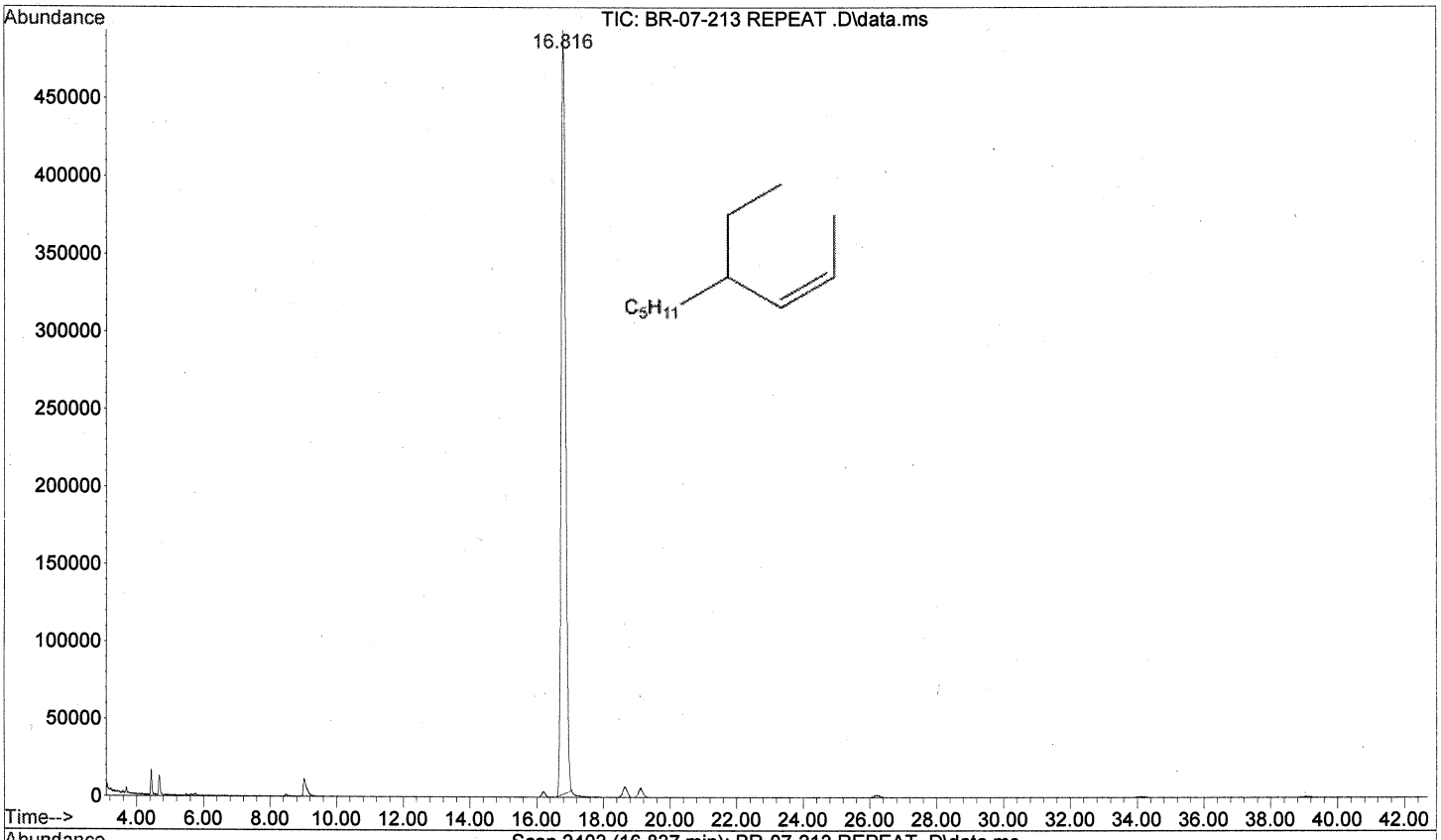
sec / Butyl

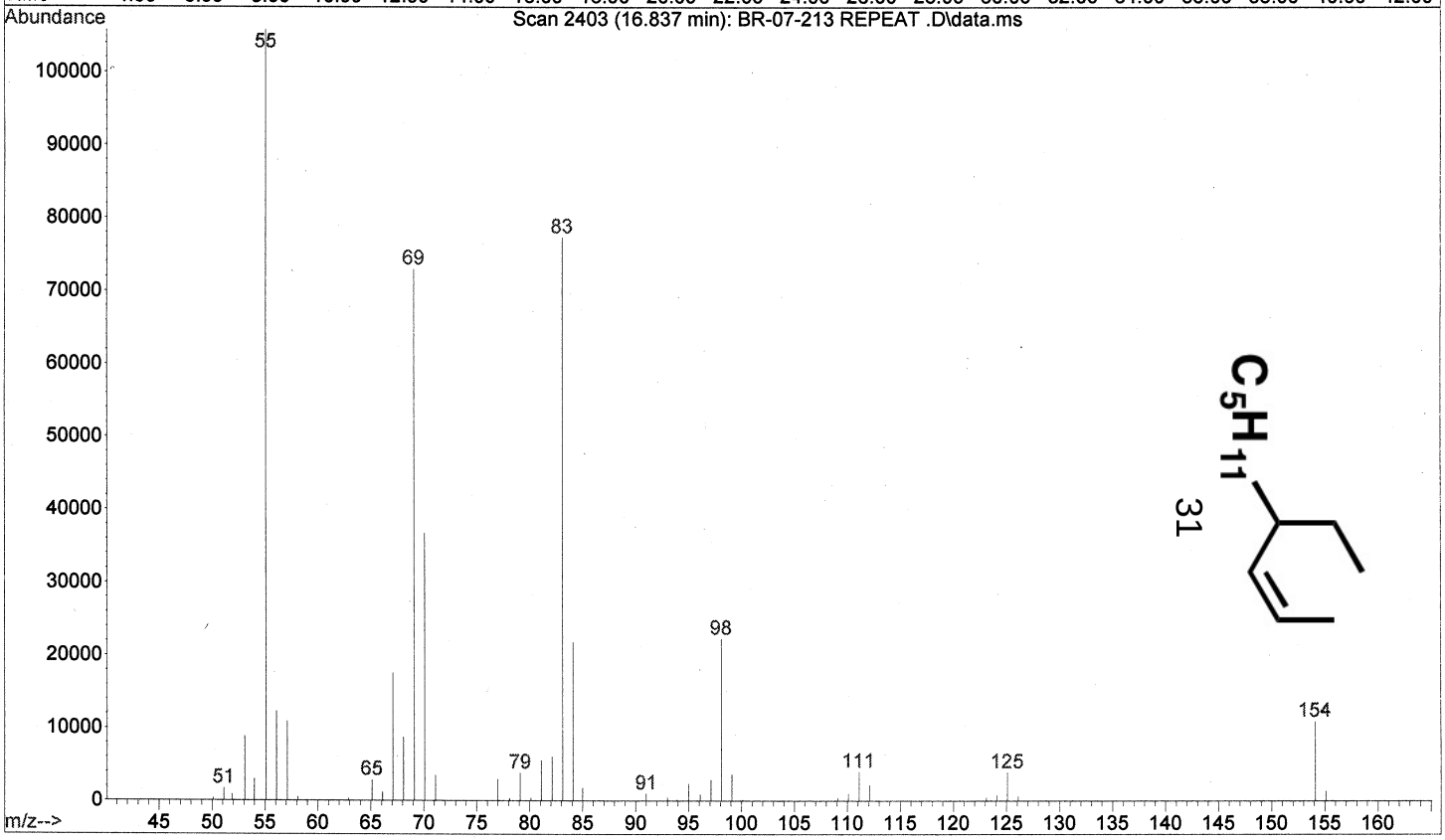
31

S76

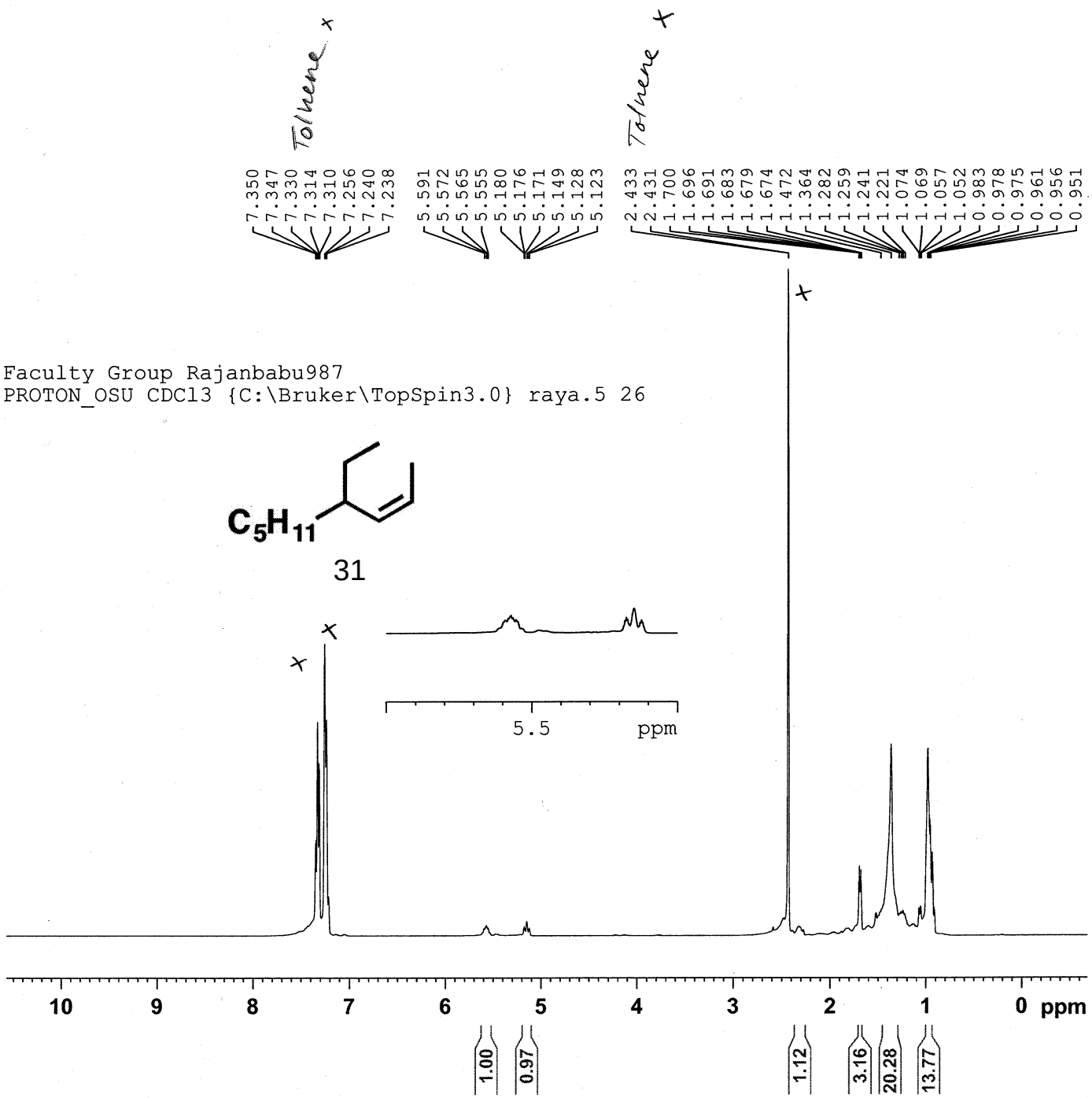
BR-
52

File :D:\MSDChemData\Babu\Balaram\BR-07-213 REPEAT .D
Operator : BALARAM
Acquired : 31 Jul 2015 13:15 using AcqMethod 50-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-213 REPEAT
Misc Info :
Vial Number: 1





31

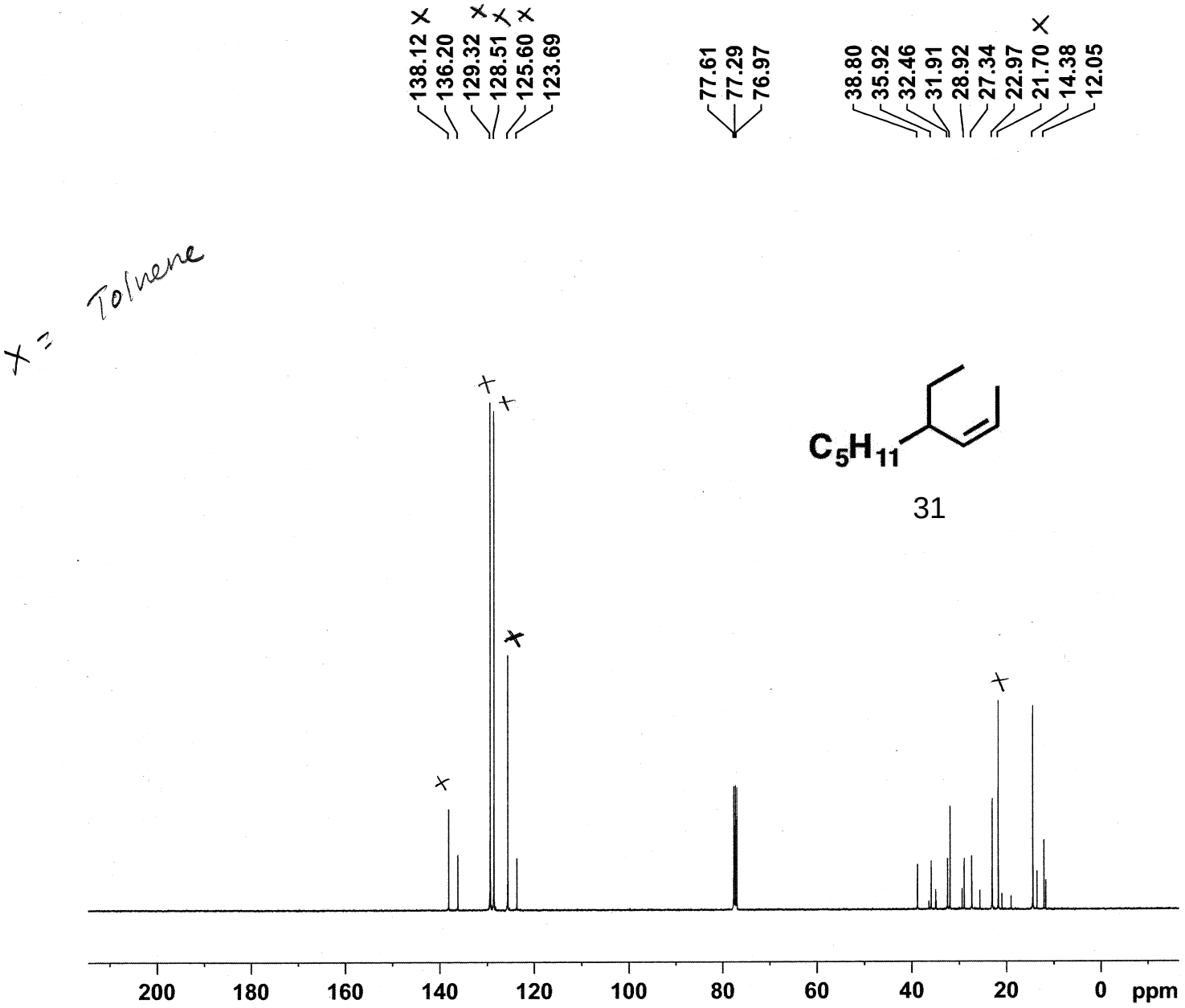



```
Curr
NAME          BR-07-213 Rerun
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20150801
Time                   19.35
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       64
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9845889 sec
RG                     32.11
DW                    60.800 usec
DE                     6.50 usec
TE                    300.0 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            400.1724712 MHz
NUC1                      1H
P1                     15.00 usec
PLW1            13.19999981 W

F2 - Processing parameters
SI                    65536
SF              400.1700138 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```




```
Current Data Parameters
NAME      BR-07-213 Rerun
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150802
Time                0.31
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6328883 MHz
NUC1                13C
P1                9.50 usec
PLW1         53.29999924 W

======== CHANNEL f2 ========
SFO2         400.1716007 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         14.60000038 W
PLW12         0.51327997 W
PLW13         0.32850000 W

F2 - Processing parameters
SI                 32768
SF           100.6228053 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

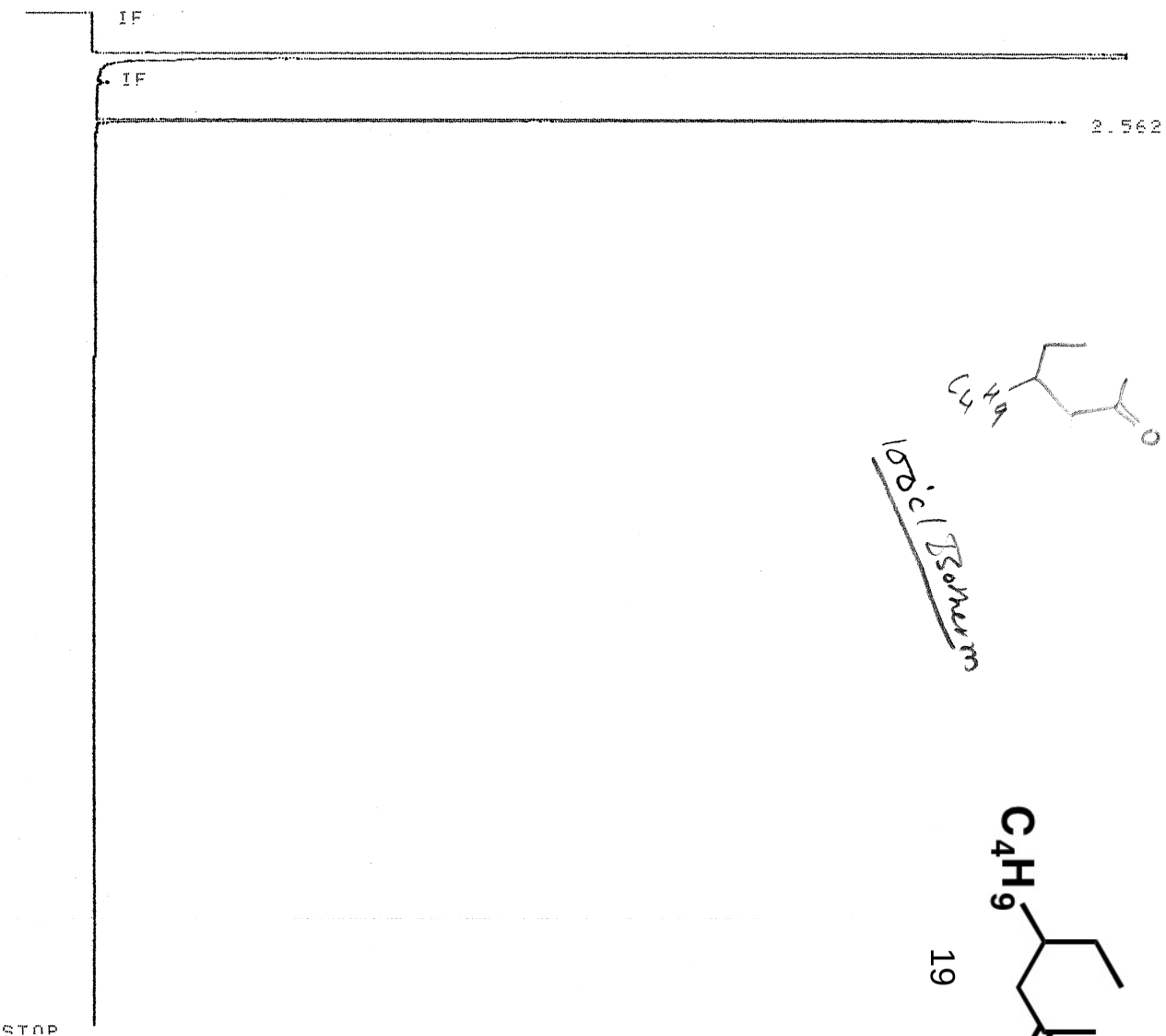
```
*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 2279      OCT  6, 1901  17:09:58
START
```




```
STOP

Error storing signal to M:SIGNAL  .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL  .PRA

RUN#  2279          OCT  6, 1901  17:09:58

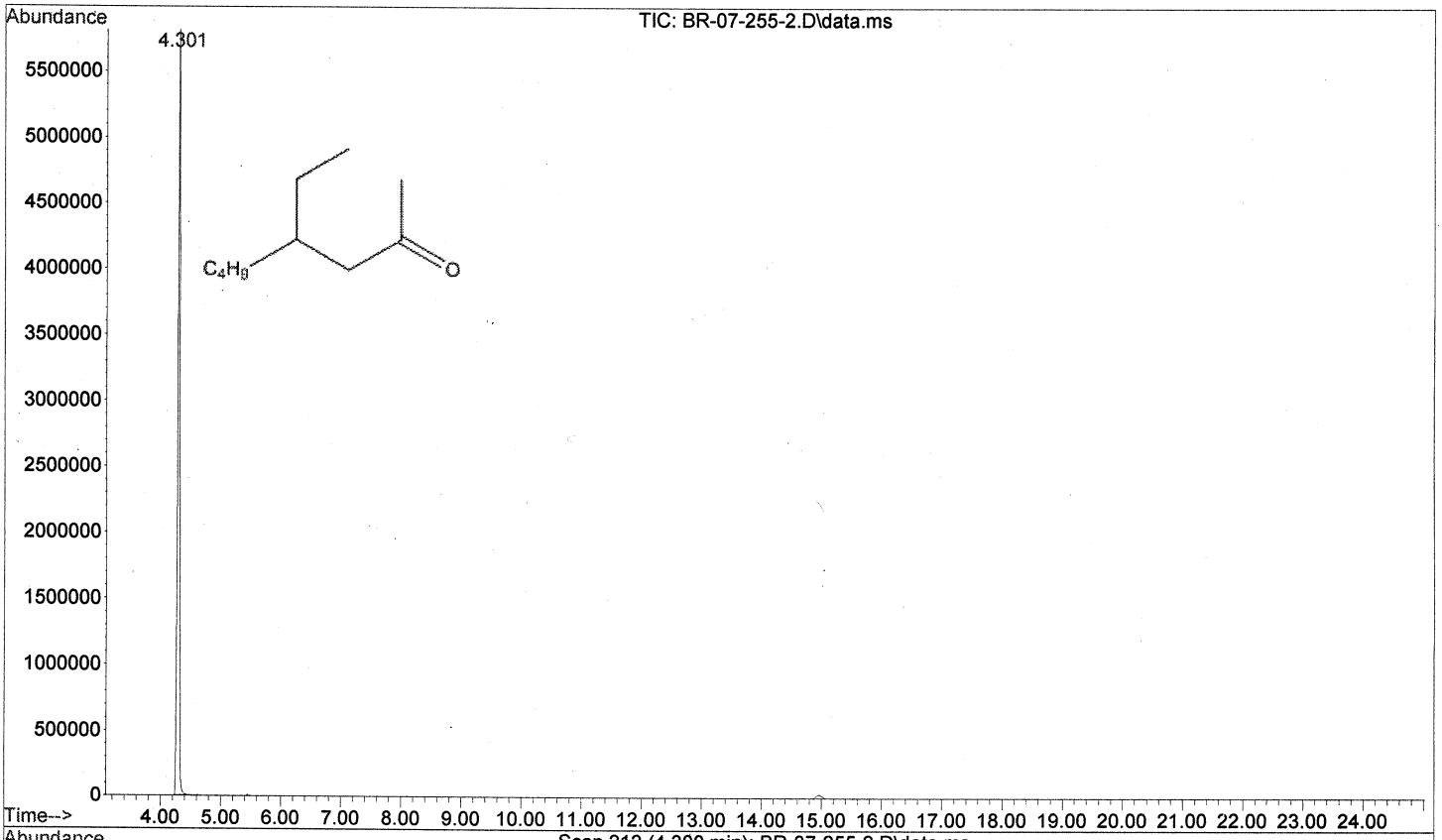
PEAK FILE  = M:SIGNAL.PRA
AREA%
      RT       AREA TYPE  WIDTH       AREA%
   2.562    3455360  SPB    .030  100.00000
```

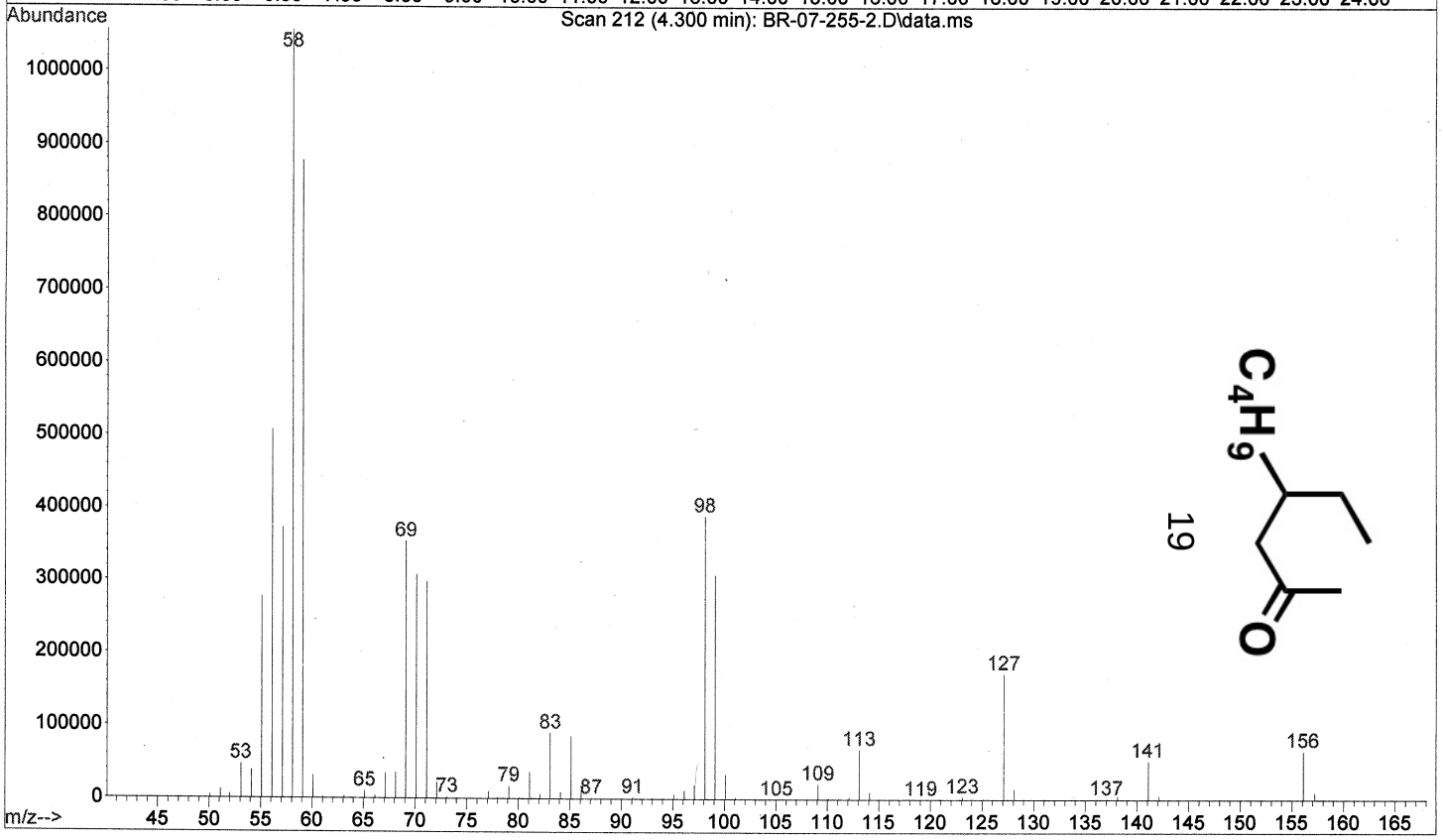

100% Bornem

19

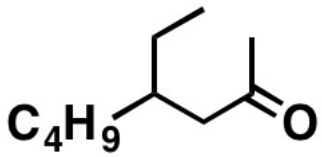
BR-07-...
100% Bo...

File :D:\MSDChemData\Babu\Balaram\BR-07-255-2.D
Operator : BALARAM
Acquired : 26 Sep 2015 15:43 using AcqMethod 100-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-255-2
Misc Info :
Vial Number: 1

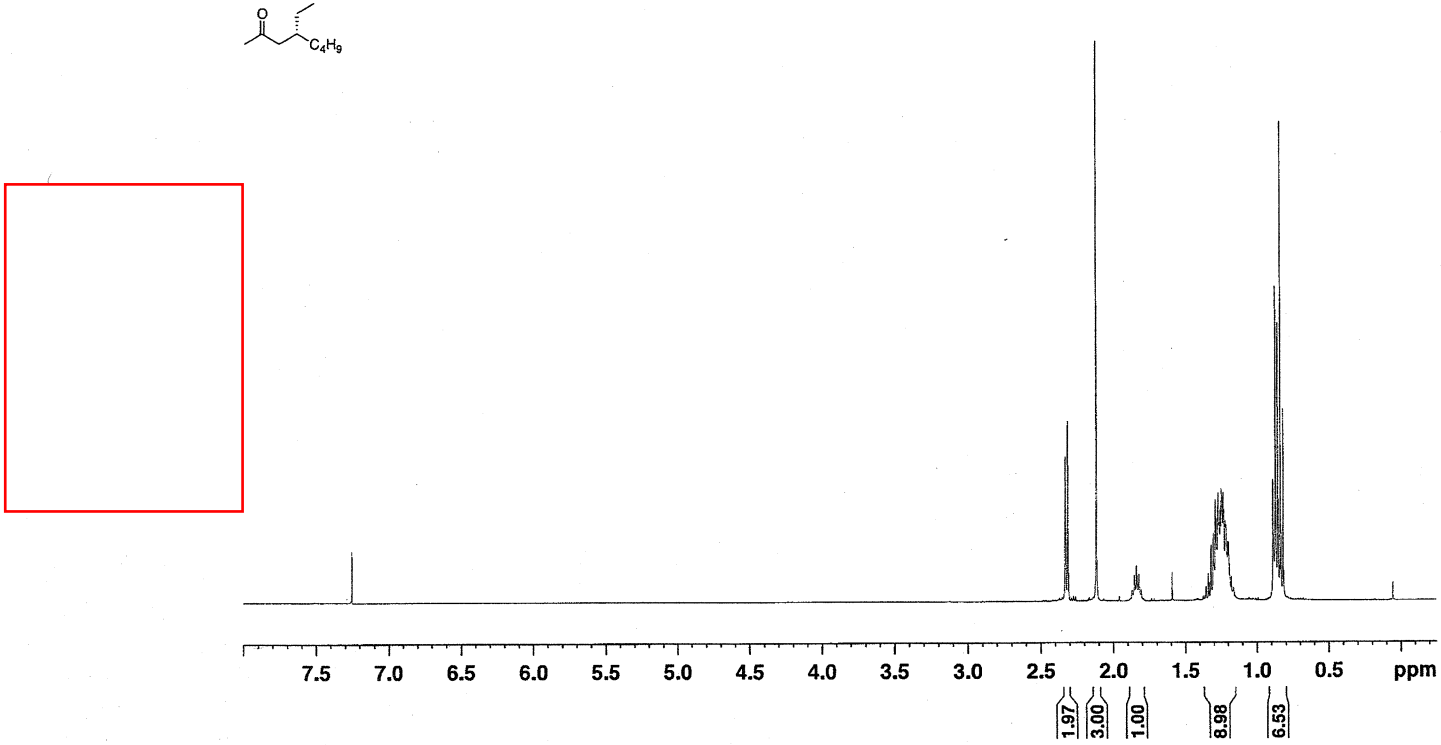




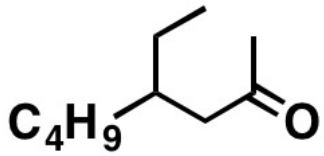
19



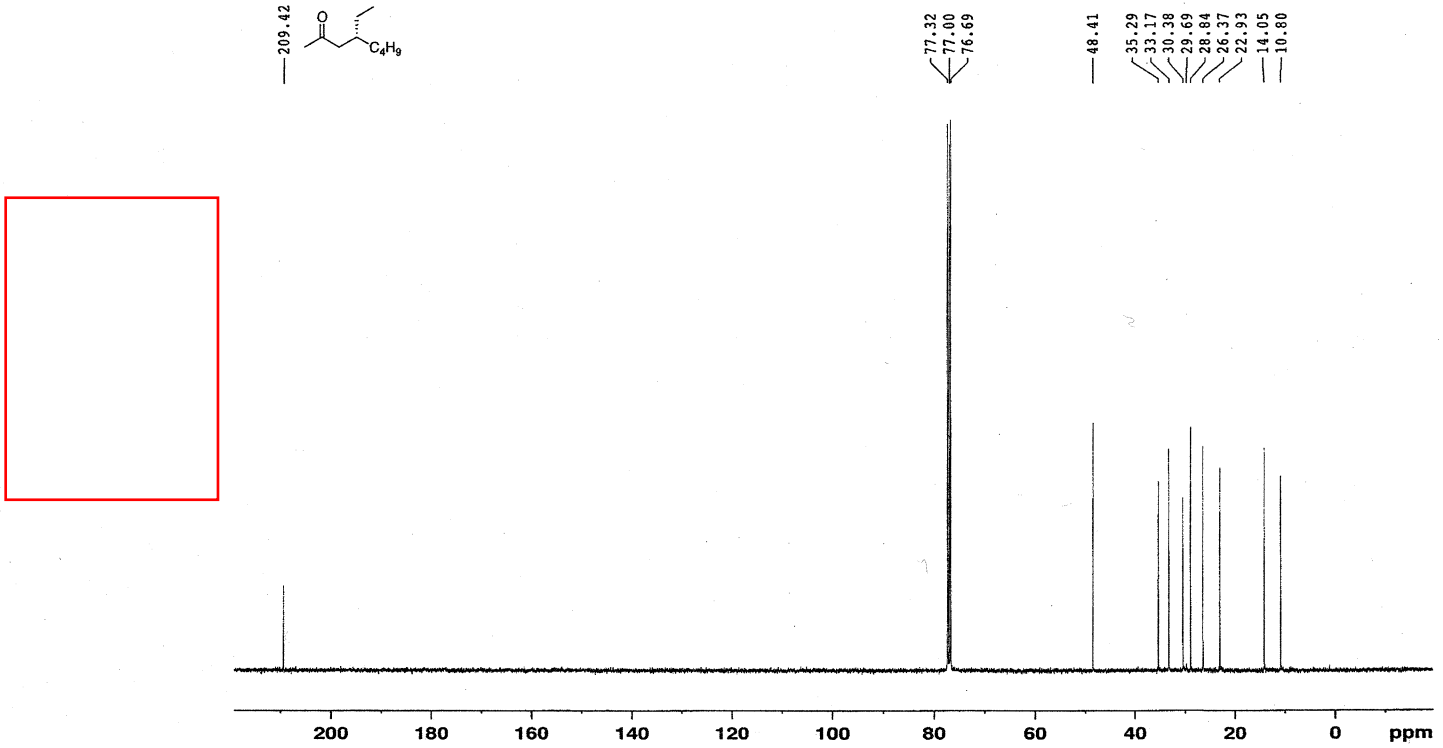
19



^{1}H NMR (400 MHz, $CDCl_3$) Spectra of Compound



19

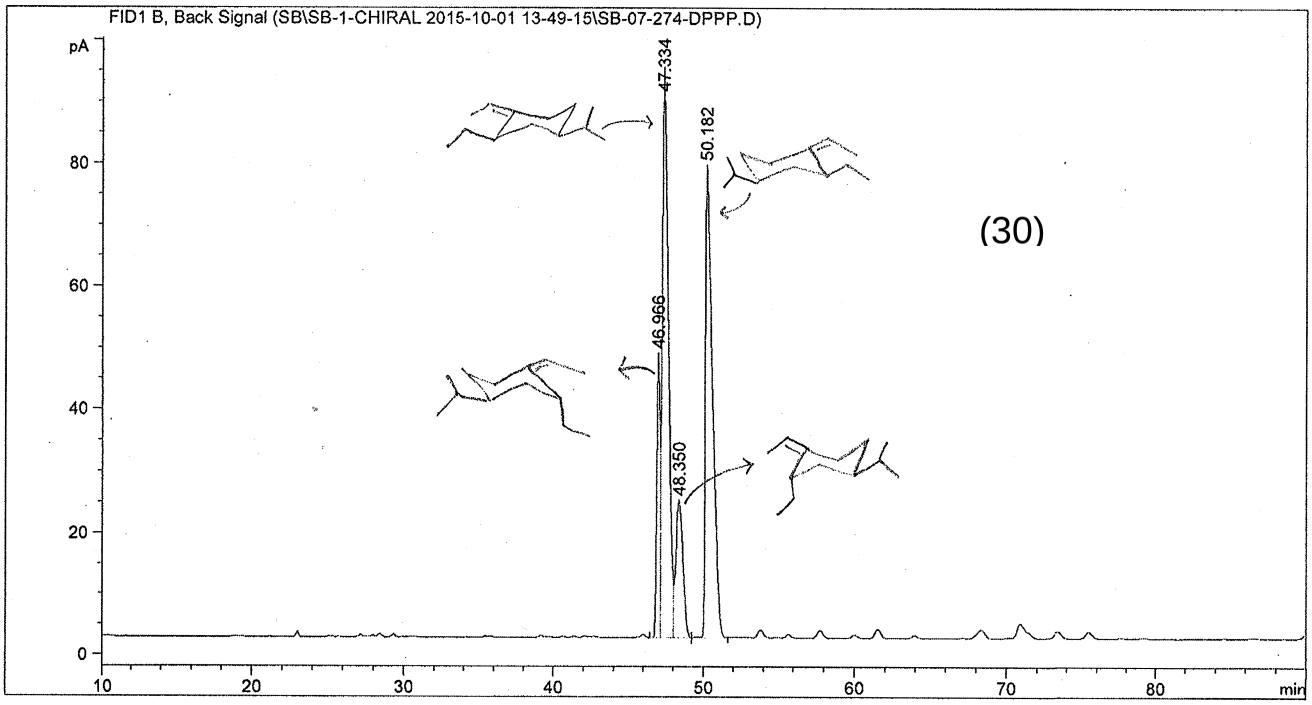


^{13}C NMR (100 MHz, $CDCl_3$) Spectra of Compound

Data File C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2015-10-01 13-49-15\SB-07-274-DPPP.D
Sample Name: SB-07-274-DPPP.D

dppp - reduction
(80°C - Isotherm)
Cyclosil B

```
==========================================================================
Acq. Operator    : BISWAS                          Seq. Line :   1
Acq. Instrument  : Babu                             Location : Vial 201
Injection Date   : 10/1/2015 1:50:27 PM                Inj :   1
                                                  Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 4 µl
Acq. Method      : C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2015-10-01 13-49-15\80_C-ISO_CHIRAL.M
Last changed     : 10/1/2015 3:17:53 PM by BISWAS
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\STANDBY.M
Last changed     : 10/1/2015 10:44:37 AM by Kendra
                   (modified after loading)
Method Info      : Checkout 7820A at OSU; front channel
```




```
==========================================================================
                        Area Percent Report
==========================================================================

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

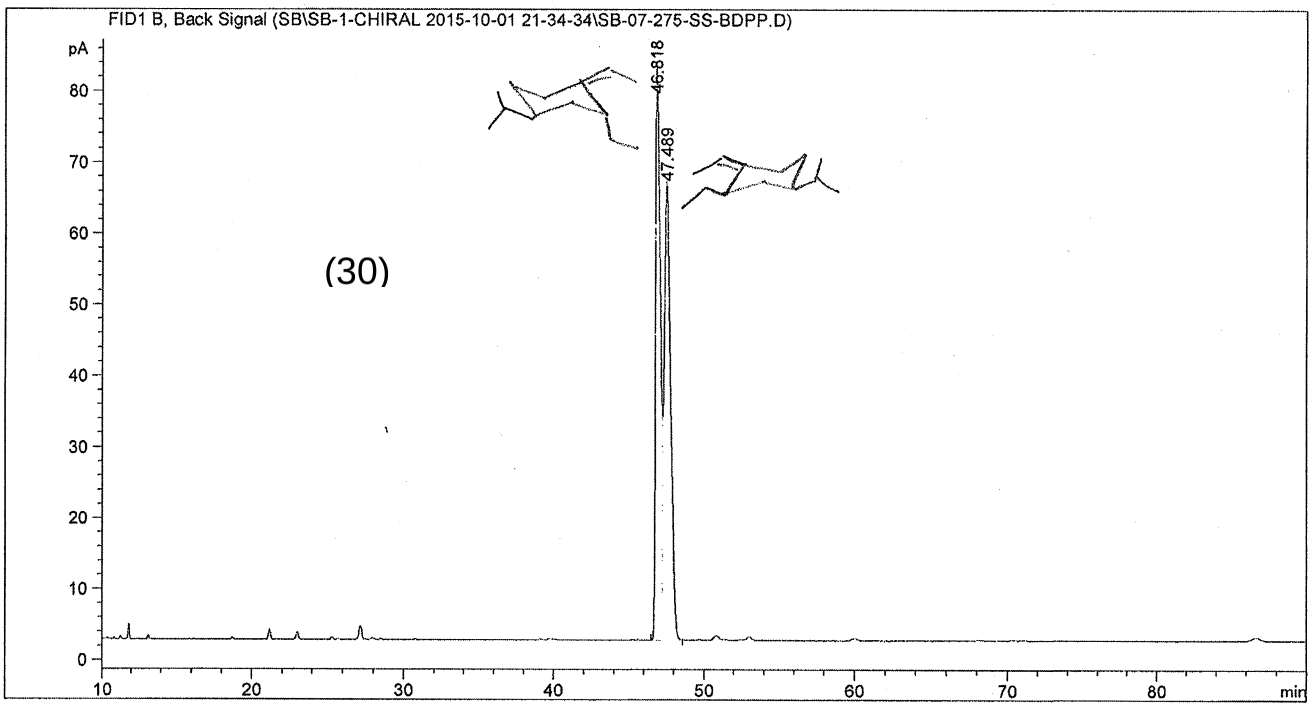

Signal 1: FID1 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  46.966 VV    0.2232  695.23895   46.30341 10.69985
   2  47.334 VV    0.3756 2573.46802   92.10998 39.60612
   3  48.350 VV    0.4284  736.79749   22.50037 11.33944
   4  50.182 VV    0.4152 2492.14819   76.83704 38.35459
```

Data File C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2015-10-01 21-34-34\SB-07-275-SS-BDPP.D
Sample Name: SB-07-275-SS-BDPP.D

(5S-BDPP)-reduction
80°C-Isotherm
Cyclosil-B

```
==========================================================================
Acq. Operator    : BISWAS                          Seq. Line :   1
Acq. Instrument  : Babu                             Location : Vial 201
Injection Date   : 10/1/2015 9:35:43 PM                 Inj :   1
                                                  Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method      : C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2015-10-01 21-34-34\80_C-ISO_CHIRAL.M
Last changed     : 10/1/2015 9:34:34 PM by BISWAS
Analysis Method  : C:\CHEM32\1\METHODS\STANDBY.M
Last changed     : 10/2/2015 12:19:48 PM by Kendra
                   (modified after loading)
Method Info      : Checkout 7820A at OSU; front channel
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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

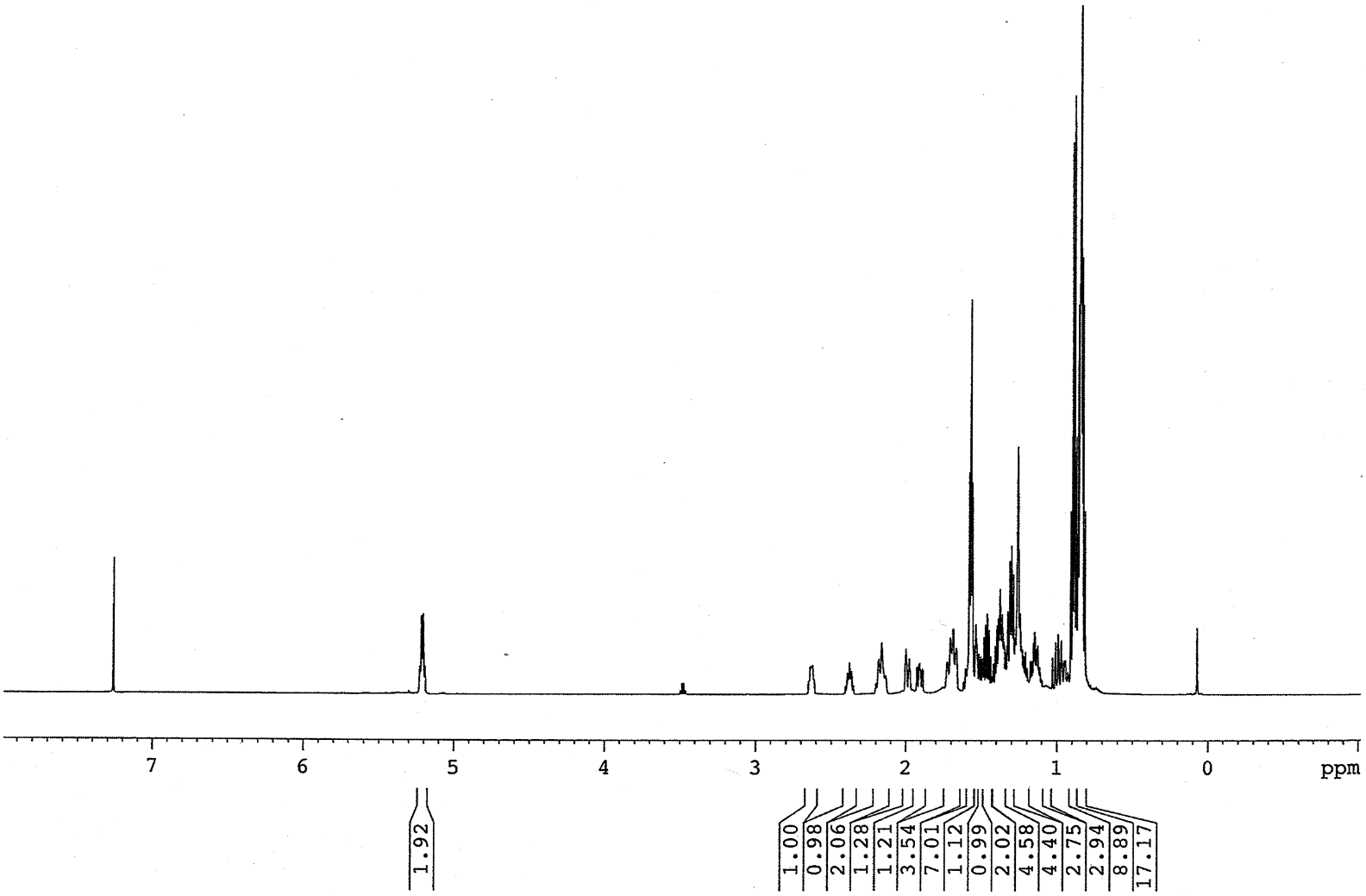

Signal 1: FID1 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  46.818 BV    0.3108 1848.43604   80.12138 48.90229
   2  47.489 VB    0.4043 1931.42004   63.81955 51.09771

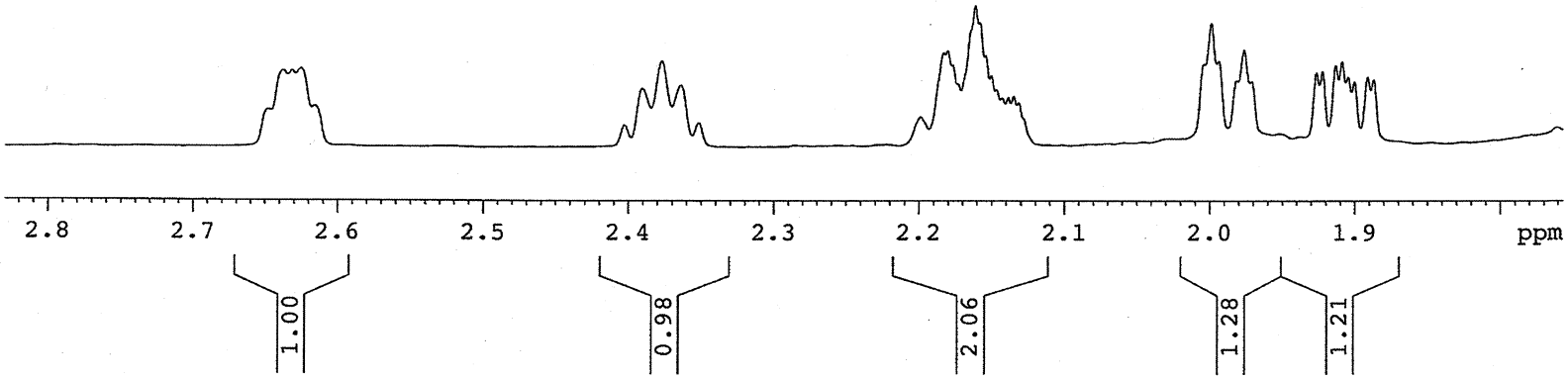
Totals :                  3779.85608  143.94093
```

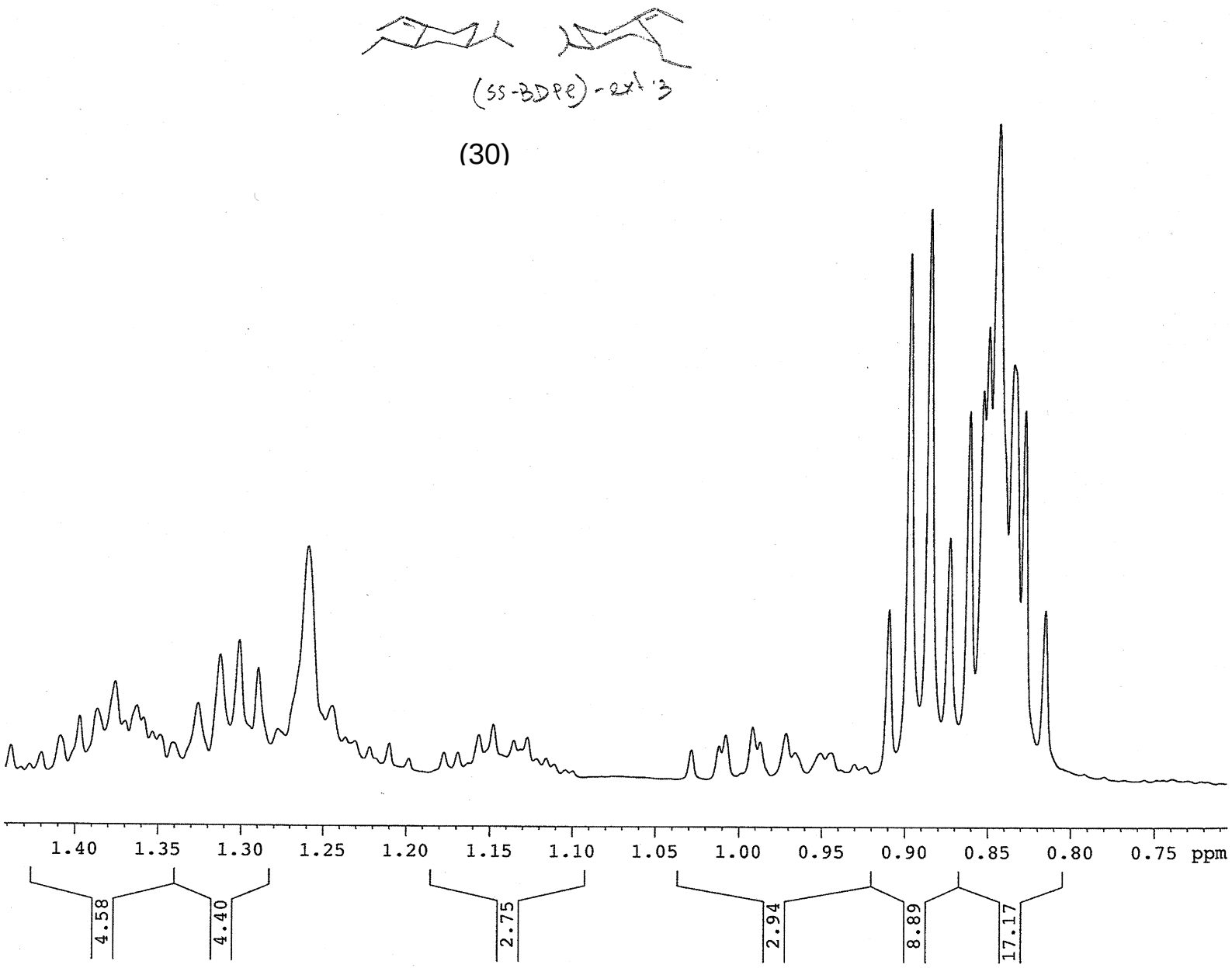

(From SS-BDPP)

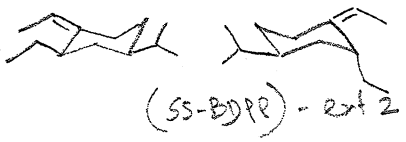
(30)


7
6
5
4
3
2
1
0
ppm
1.92
1.00
0.98
2.06
1.28
1.21
3.54
7.01
1.12
0.99
2.02
4.58
4.40
2.75
2.94
8.89
17.17

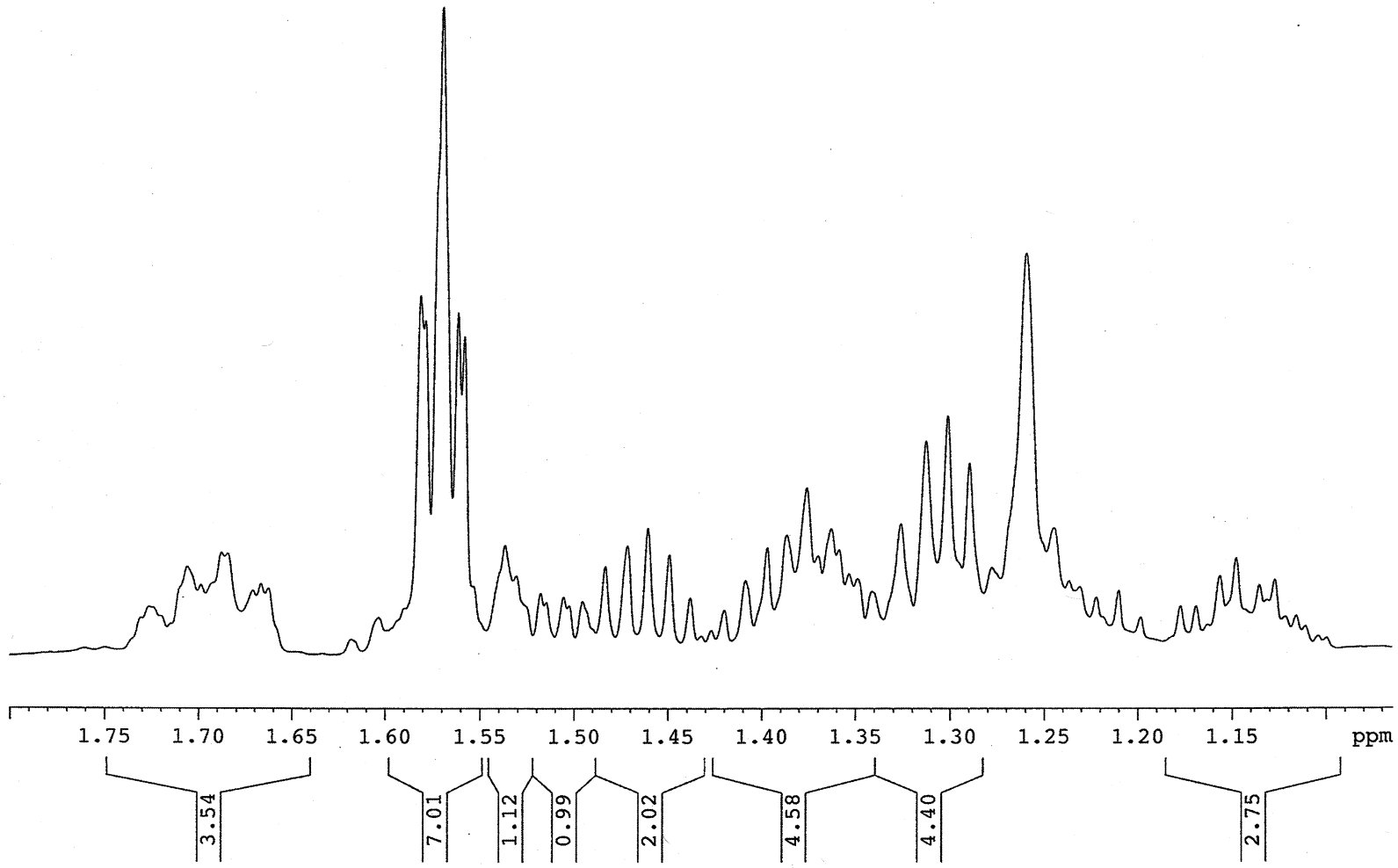
(30)




(SS-BDPE)-ex1.3
(30)
1.40
1.35
1.30
1.25
1.20
1.15
1.10
1.05
1.00
0.95
0.90
0.85
0.80
0.75
ppm
4.58
4.40
2.75
2.94
8.89
17.17

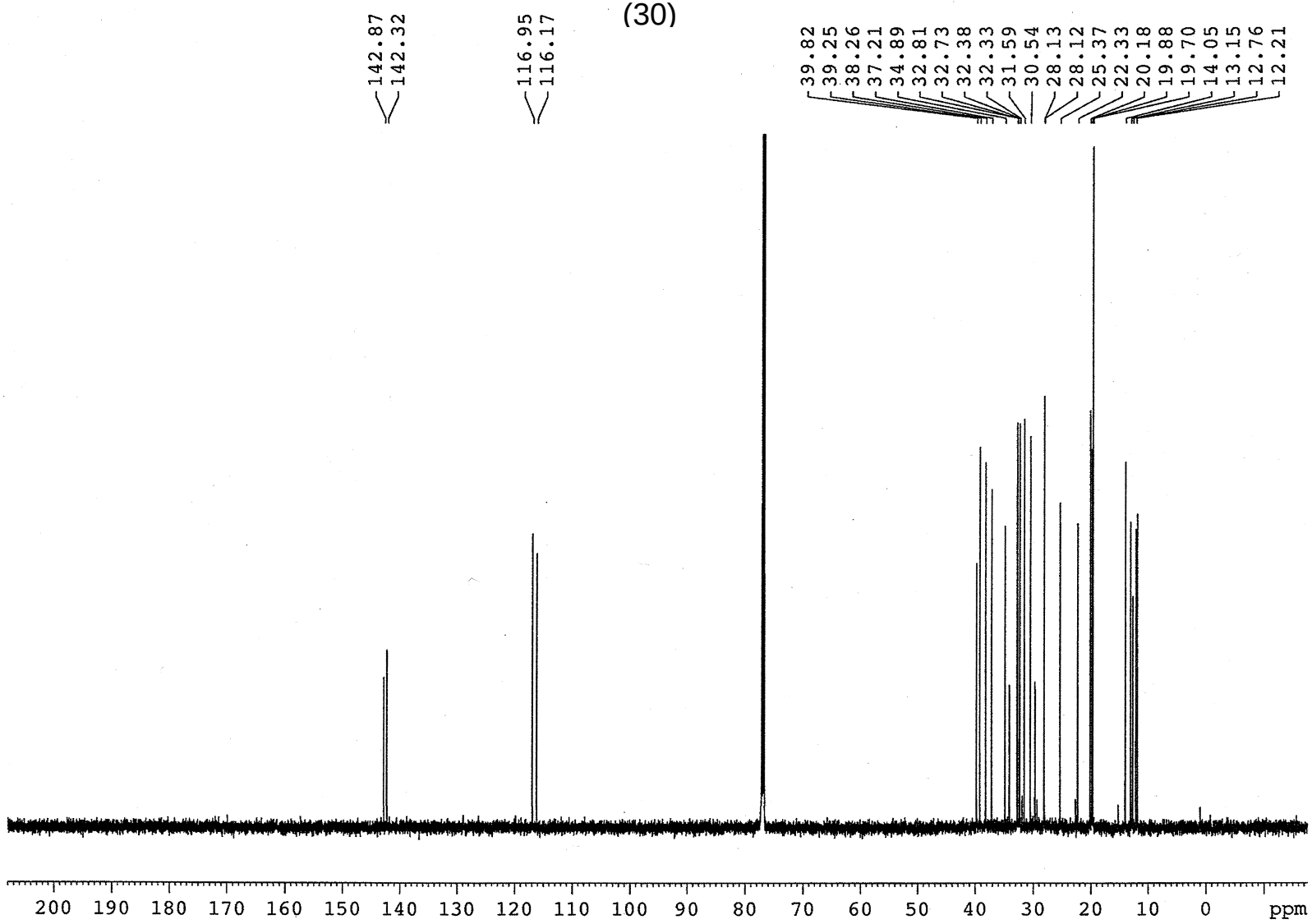

(SS-BDPP) - ent 2

(30)


1.75
1.70
1.65
1.60
1.55
1.50
1.45
1.40
1.35
1.30
1.25
1.20
1.15
ppm
3.54
7.01
1.12
0.99
2.02
4.58
4.40
2.75


(From ss-BDPP)

(30)


142.87
142.32
116.95
116.17
39.82
39.25
38.26
37.21
34.89
32.81
32.73
32.38
32.33
31.59
30.54
28.13
28.12
25.37
22.33
20.18
19.88
19.70
14.05
13.15
12.76
12.21
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

```
*
BREAK

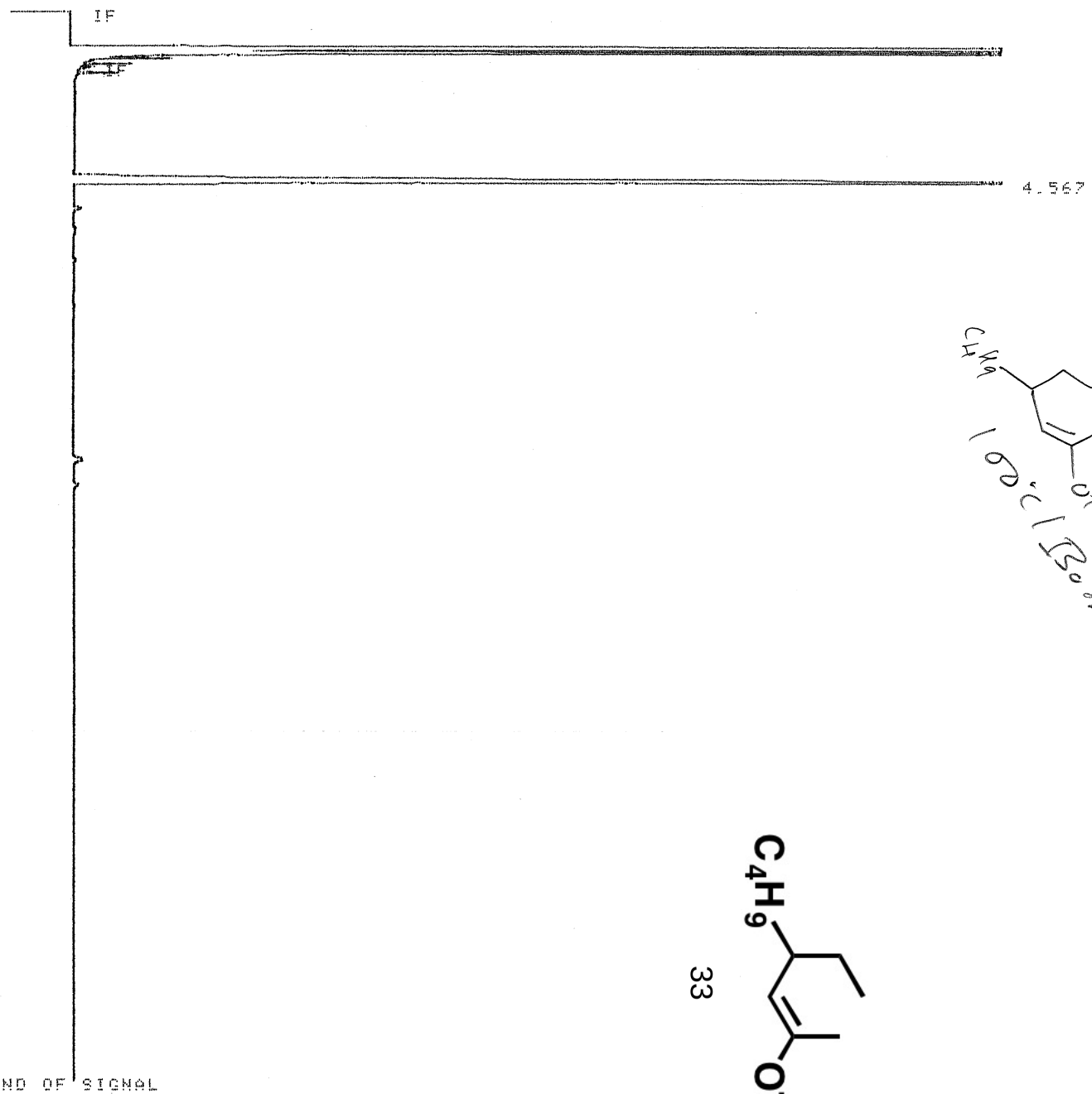
*
BREAK

*
BREAK

*
BREAK

*
BREAK

*AN
RUN # 2253      OCT  2, 1901  18:55:10
START
```

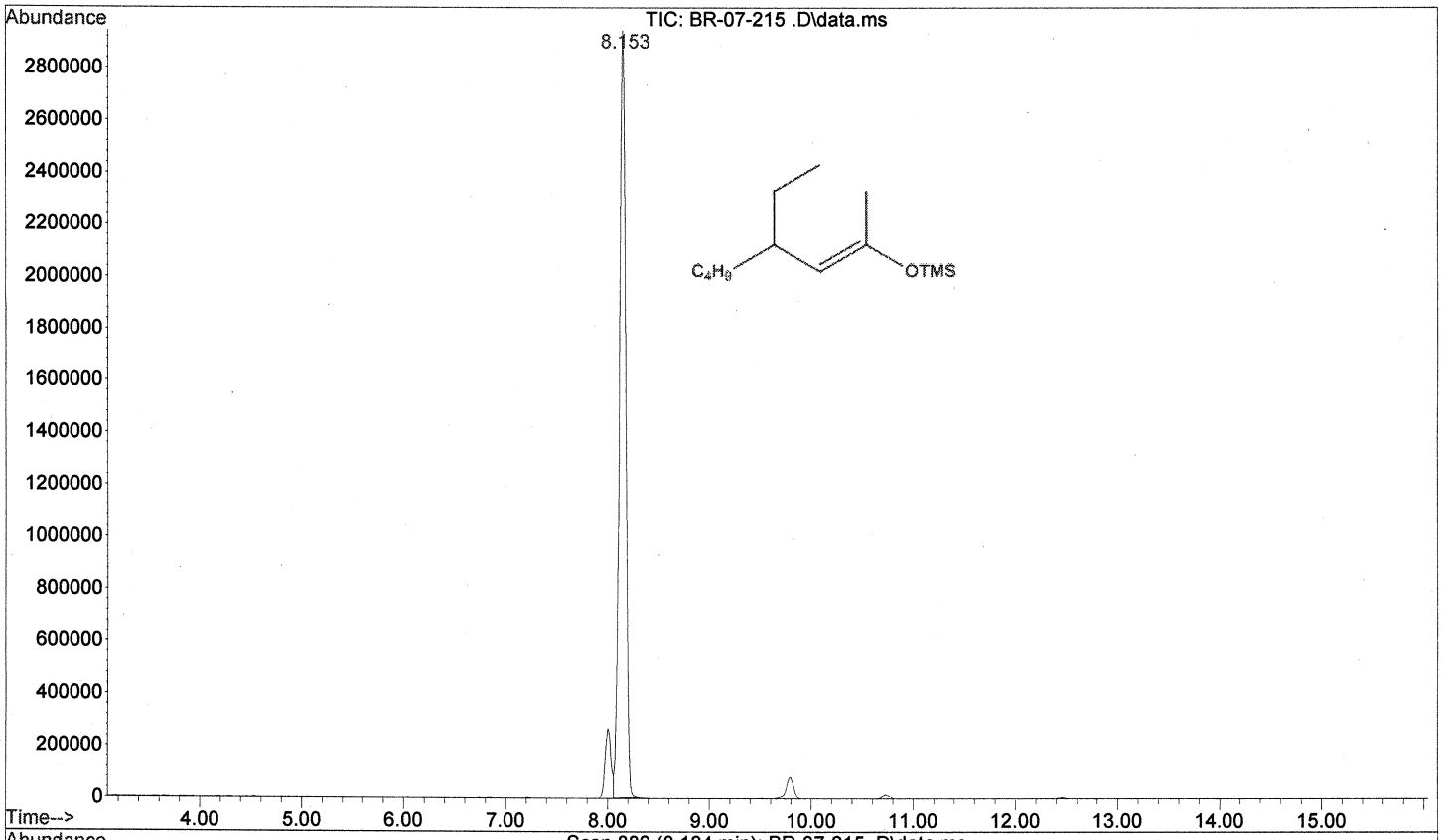


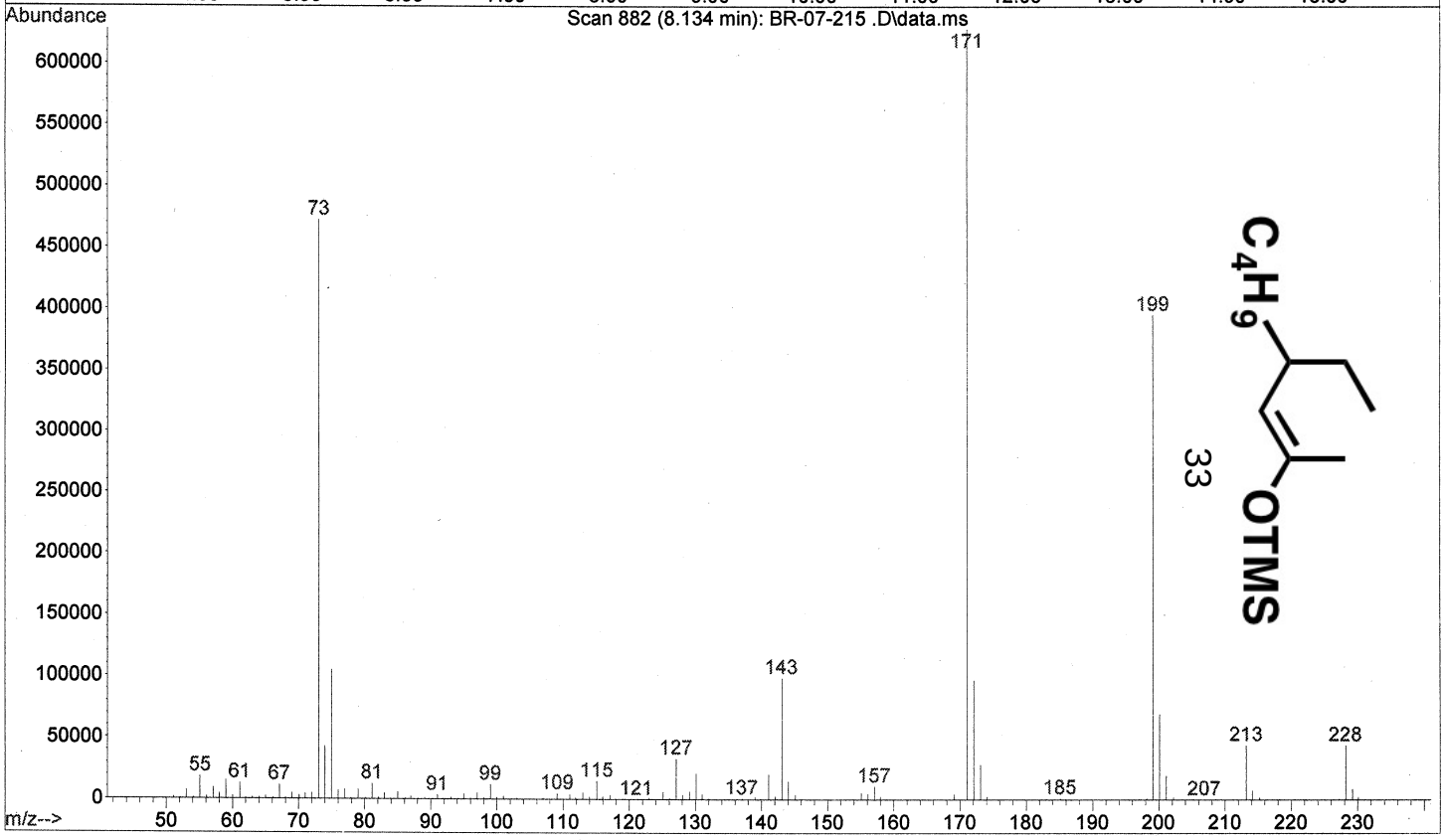

```
END OF SIGNAL

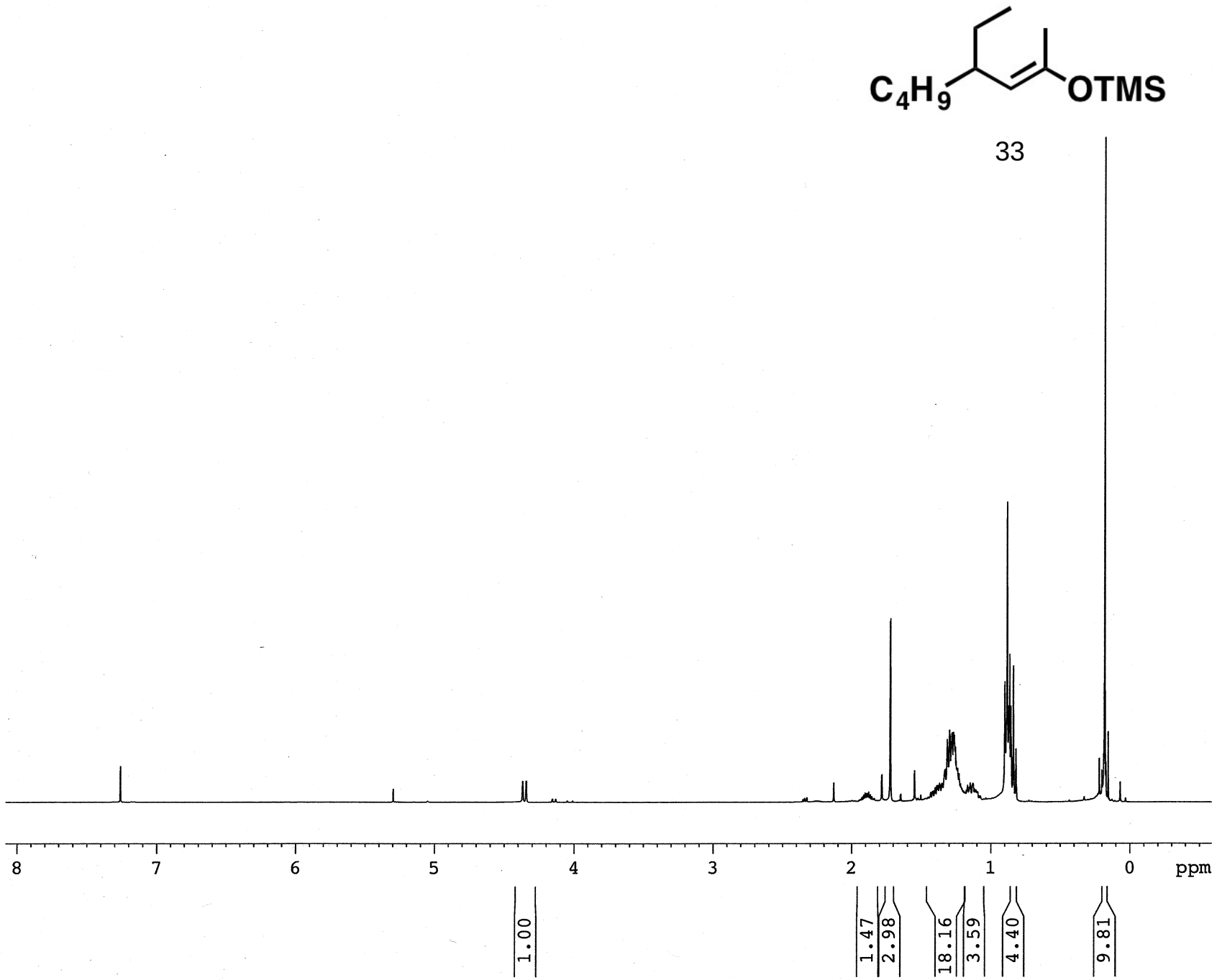
Error storing signal to M:SIGNAL  .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL  .PRA
```

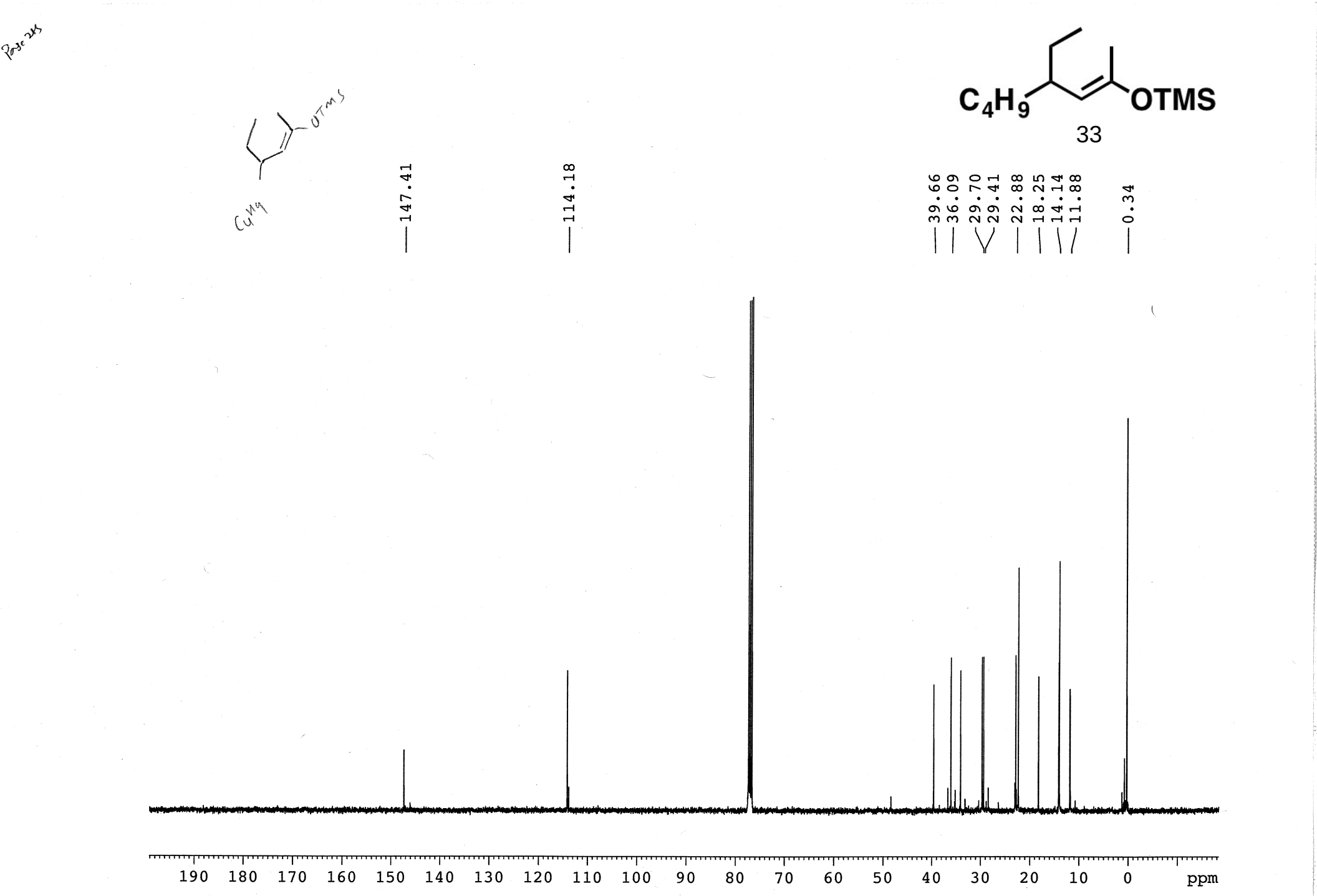

33

File :D:\MSDChemData\Babu\Balaram\BR-07-215 .D
Operator : BALARAM
Acquired : 31 Jul 2015 10:30 using AcqMethod 100-ISOTHERM.M
Instrument : GCMS
Sample Name: BR-07-215
Misc Info :
Vial Number: 1

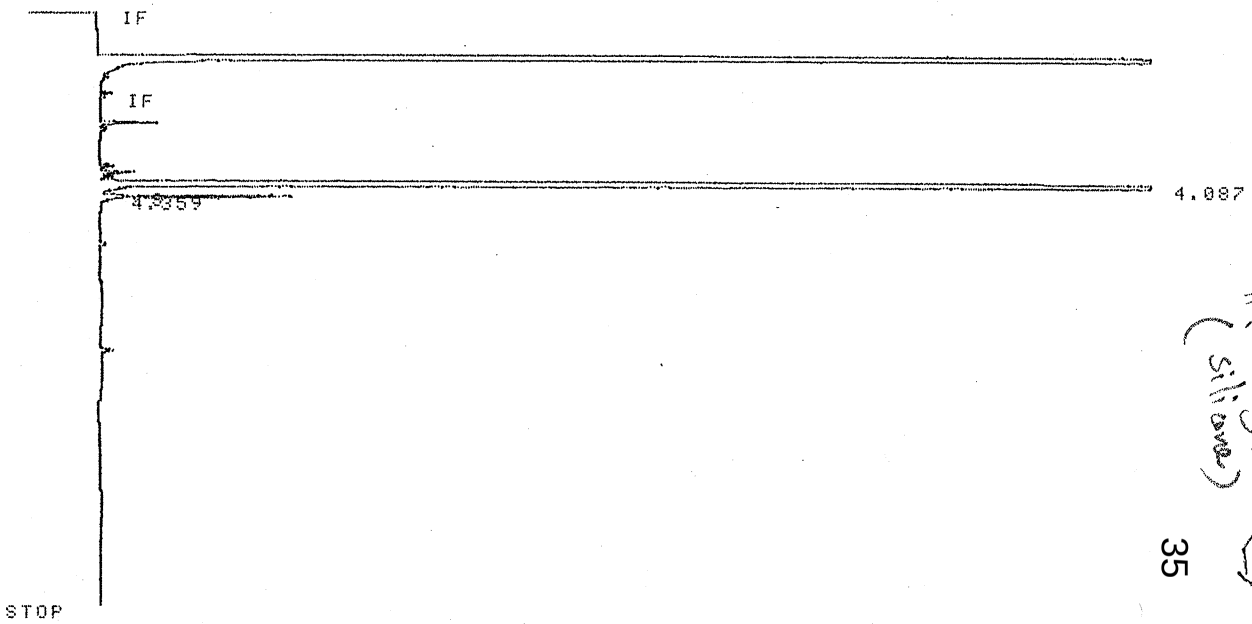





C4H9
OTMS
33
8
7
6
5
4
3
2
1
0
ppm
1.00
1.47
2.98
18.16
3.59
4.40
9.81


C4H9
OTMS
33
147.41
114.18
39.66
36.09
29.70
29.41
22.88
18.25
14.14
11.88
0.34
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

RUN # 1822 AUG 16, 1901 17:39:08
START



STOP

Error storing signal to M:SIGNAL .BNA
DIRECTORY FULL
Storing processed peaks to M:SIGNAL .PRA

RUN# 1822 AUG 16, 1901 17:39:08

PEAK FILE : M:SIGNAL.PRA
AREA%

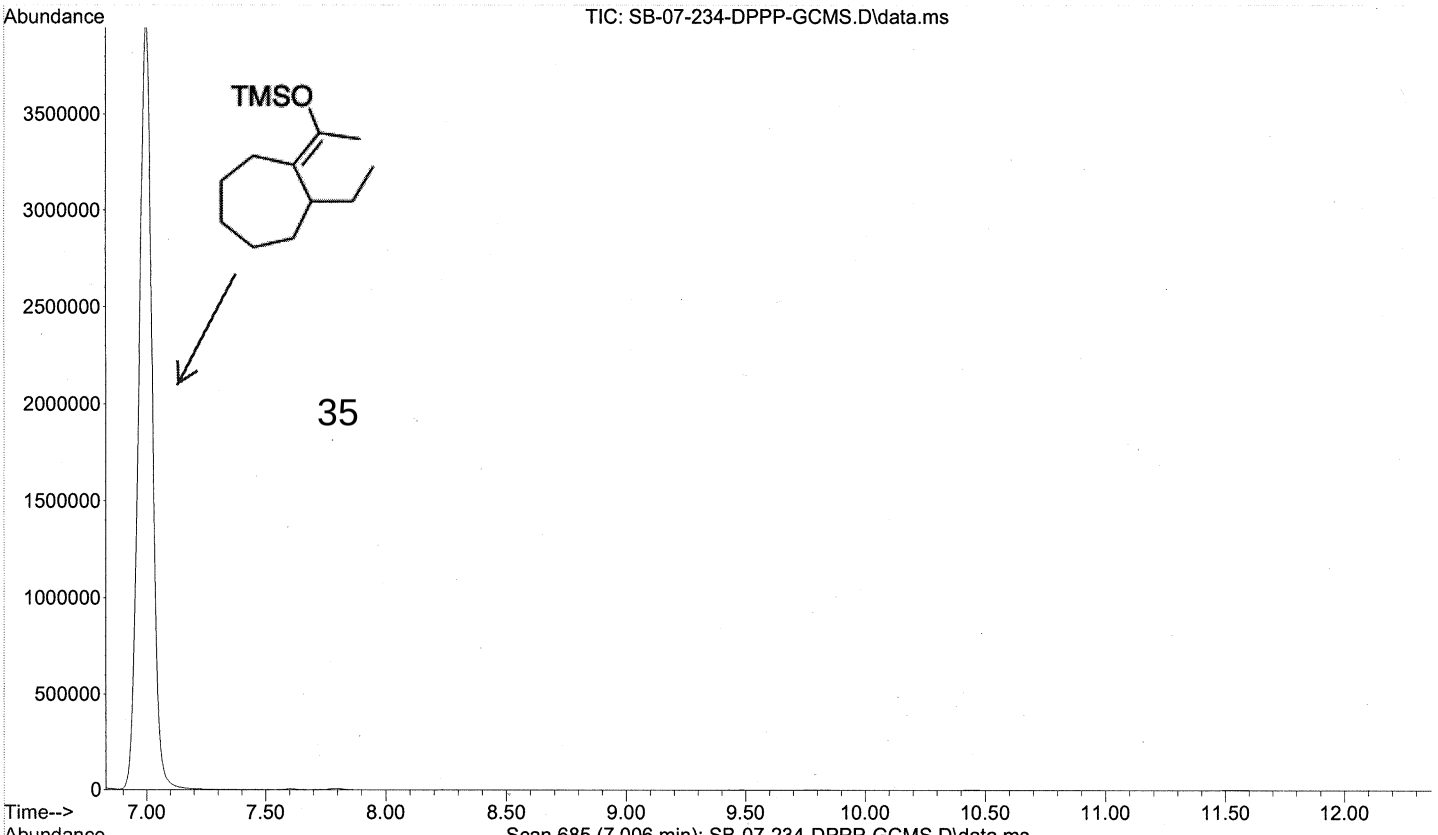
RT	AREA	TYPE	WIDTH	AREA%
4.087	12656624	SHB	.059	98.05914
4.359	250508	BB	.044	1.94085

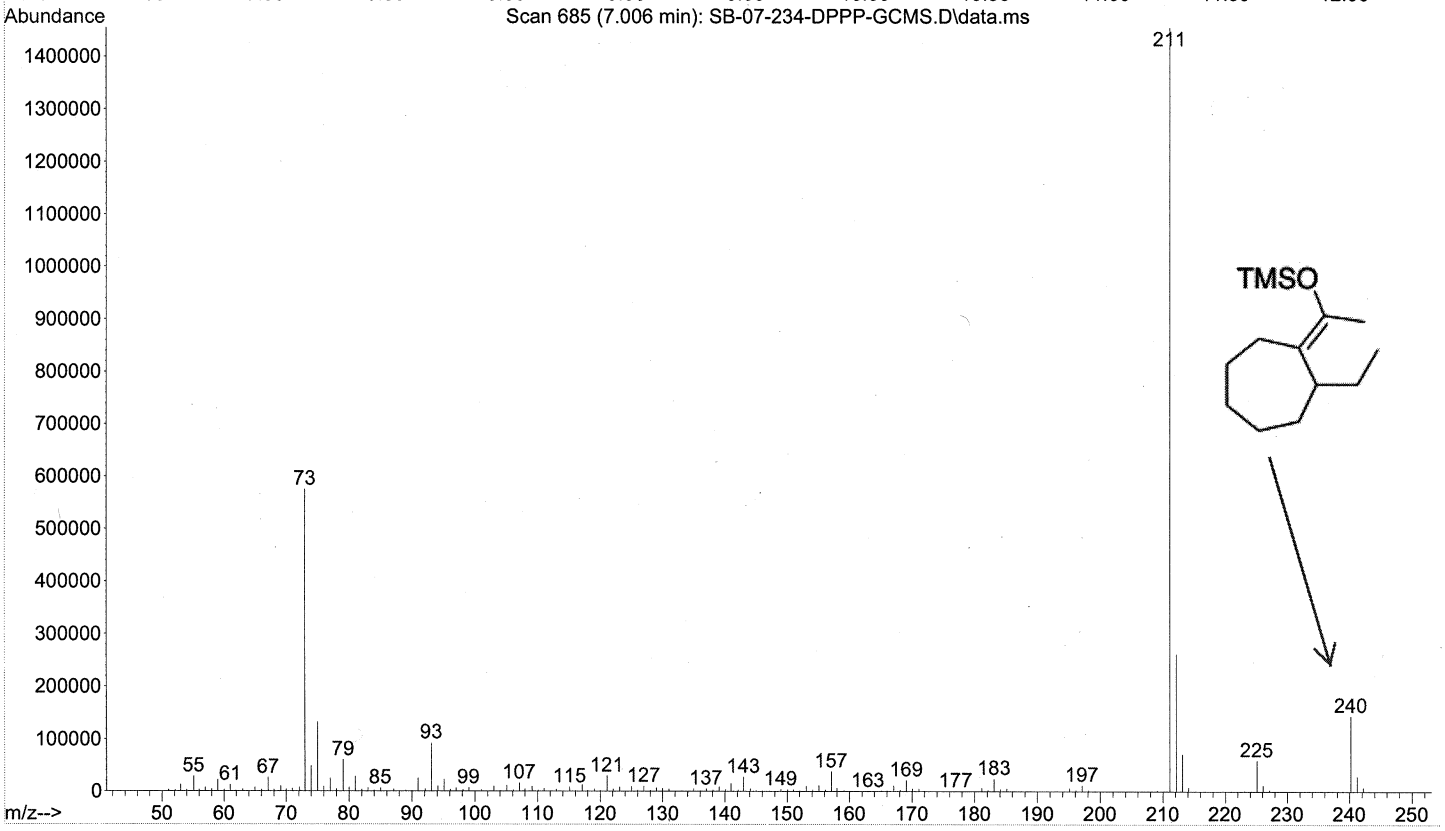
TOTAL AREA=1.2907E+07
MUL FACTOR=1.0000E+00

130°C - 6ft HP-methyl (silicone)

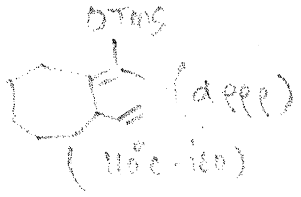
(SS-BDPP)

File :D:\MSDChemData\Babu\biswas\130ramp250.M\SB-07-234-DPPP-GCMS.D
Operator : BISWAS
Acquired : 20 Nov 2015 23:17 using AcqMethod 130RAMP250.M
Instrument : GCMS
Sample Name: SB-07-234-DPPP-GCMS
Misc Info :
Vial Number: 1





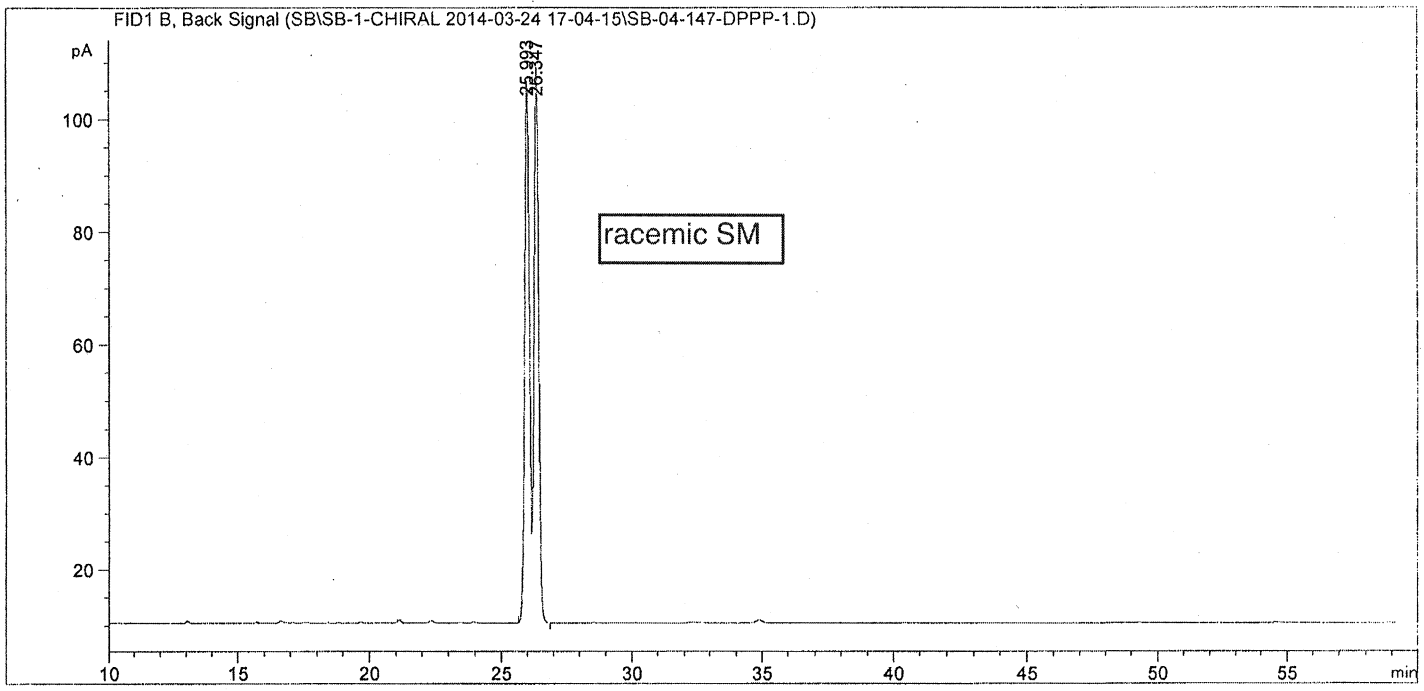
Data File C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2014-03-24 17-04-15\SB-04-147-DPPP-1.D
Sample Name: SB-04-147-DPPP-1.D



(Gedsil-B)

26

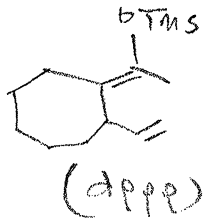
```
==========================================================================
Acq. Operator    : BISWAS                          Seq. Line :   1
Acq. Instrument  : Babu                             Location : Vial 201
Injection Date   : 3/24/2014 5:05:22 PM                 Inj :   1
                                                  Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 4 µl
Acq. Method      : C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2014-03-24 17-04-15\110_C-ISO_CHIRAL.M
Last changed     : 3/24/2014 5:45:53 PM by BISWAS
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\STANDBY.M
Last changed     : 4/29/2014 5:11:59 PM by BISWAS
                   (modified after loading)
Method Info      : Checkout 7820A at OSU; front channel
```




```
==========================================================================
                        External Standard Report
==========================================================================

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

Data File C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2014-03-24 17-04-15\SB-04-147-DPPP-1.D
Sample Name: SB-04-147-DPPP-1.D



==========

Area Percent Report

==========

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

Signal 1: FID1 B, Back Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	25.993	BV	0.1953	1181.58130	95.68957	49.75679
2	26.347	VB	0.1935	1193.13232	97.79988	50.24321

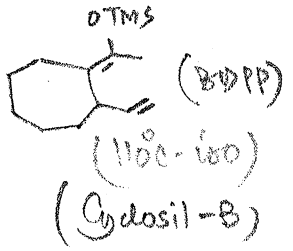
Totals : 2374.71362 193.48945

==========

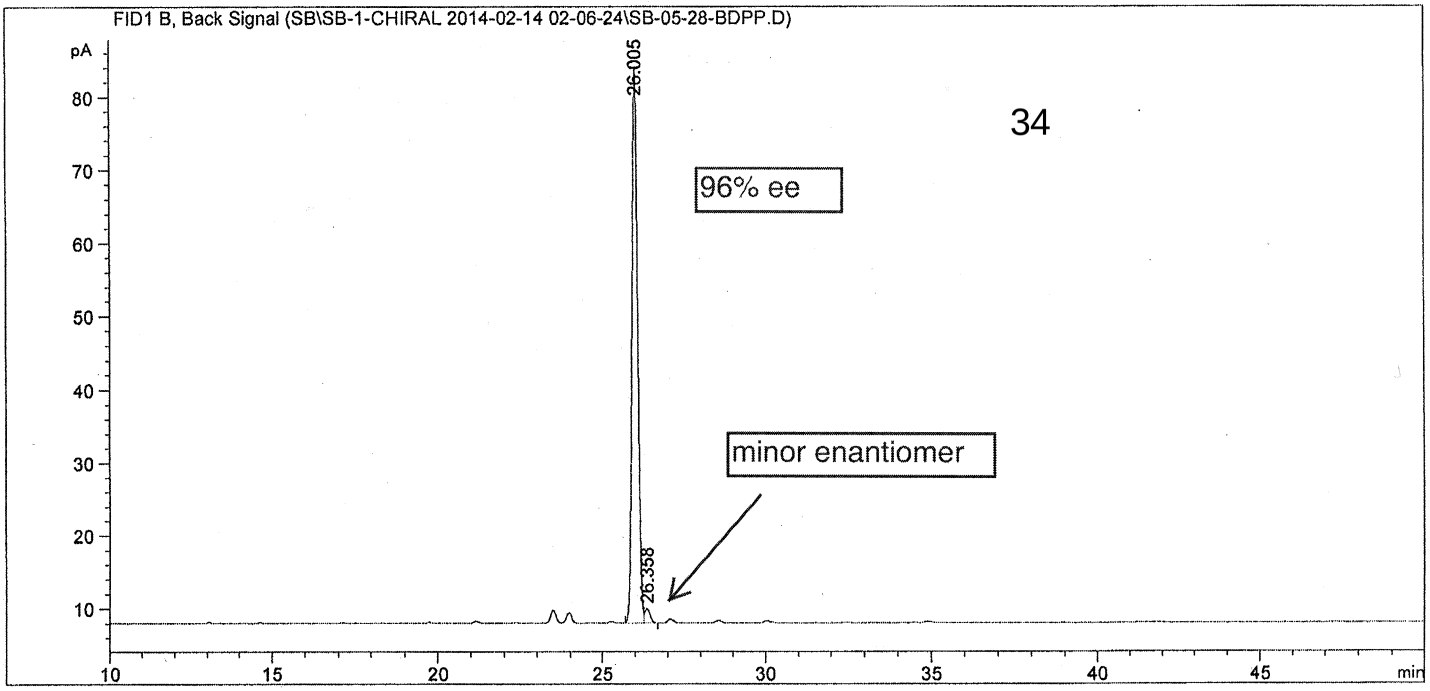
*** End of Report ***

S98

Data File C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2014-02-14 02-06-24\SB-05-28-BDPP.D
Sample Name: SB-05-28-BDPP.D




```
================================================================================
Acq. Operator    : BISWAS                          Seq. Line :   1
Acq. Instrument  : Babu                             Location : Vial 201
Injection Date   : 2/14/2014 2:07:33 AM                 Inj :   1
                                                  Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 4 µl
Acq. Method      : C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2014-02-14 02-06-24\110_C-ISO_CHIRAL.M
Last changed     : 2/14/2014 2:06:23 AM by BISWAS
Analysis Method  : C:\CHEM32\1\METHODS\35_CYCLODEX-B.M
Last changed     : 2/25/2014 12:42:11 AM by Kendra Dewese
                   (modified after loading)
```




```
================================================================================
                          Area Percent Report
================================================================================

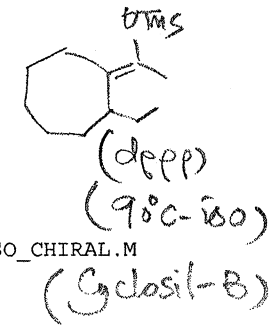
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  26.005 BV    0.1928  916.15771   75.49691 97.75248
   2  26.358 VB    0.1722   21.06422    1.92939  2.24752

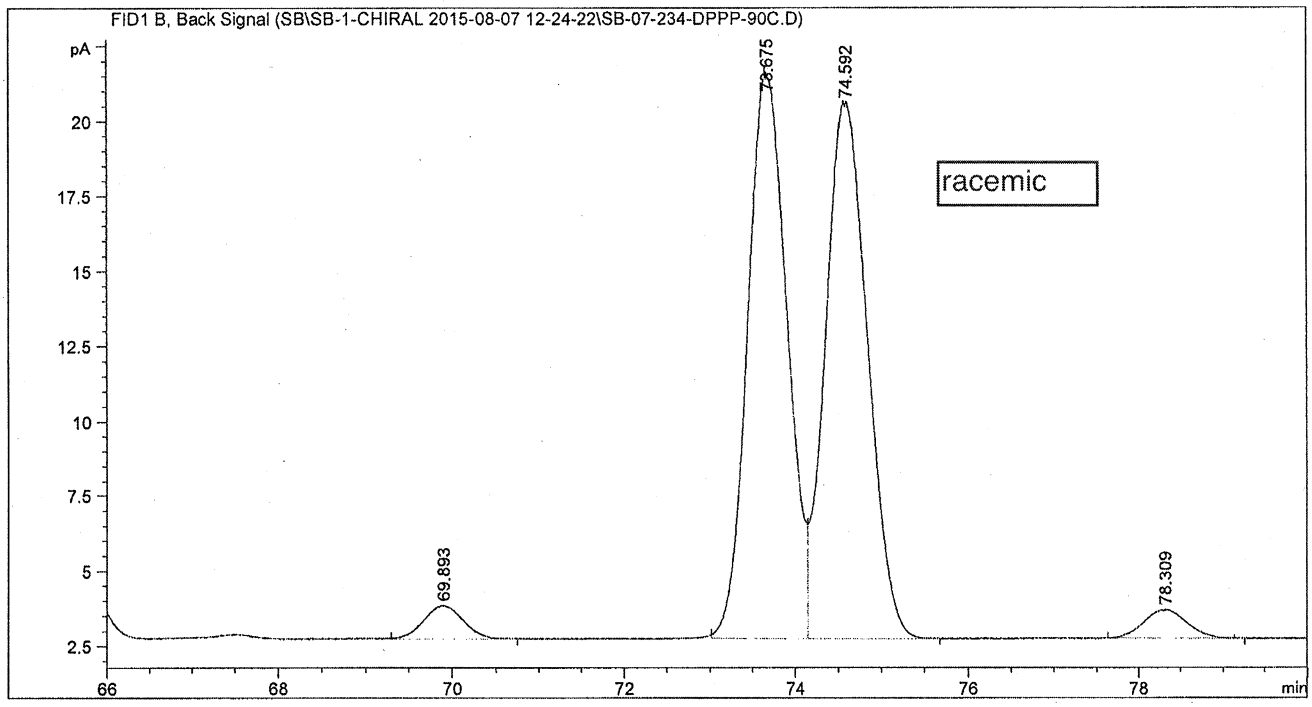
Totals :                   937.22193   77.42630
```

Sample Name: SB-07-234-DPPP-90C.D



35

```
==========================================================================
Acq. Operator    : BISWAS                          Seq. Line :   1
Acq. Instrument  : Babu                             Location : Vial 201
Injection Date   : 8/7/2015 12:25:32 PM                 Inj :   1
                                                  Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 4 µl
Acq. Method      : C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2015-08-07 12-24-22\90_C-ISO_CHIRAL.M
Last changed     : 8/7/2015 2:26:45 PM by BISWAS
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\STANDBY.M
Last changed     : 8/7/2015 3:09:11 PM by BISWAS
                   (modified after loading)
Method Info      : Checkout 7820A at OSU; front channel
```




```
==========================================================================
                        Area Percent Report
==========================================================================

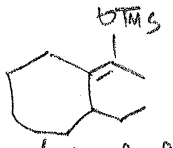
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  23.690 BB    0.1682    3.60211 3.40510e-1  0.28258
   2  69.893 BB    0.4947   34.46913    1.09678  2.70402
   3  73.675 BV    0.5011  599.07330   18.73468 46.99584
   4  74.592 VB    0.5235  603.44257   17.80017 47.33860
```


S100

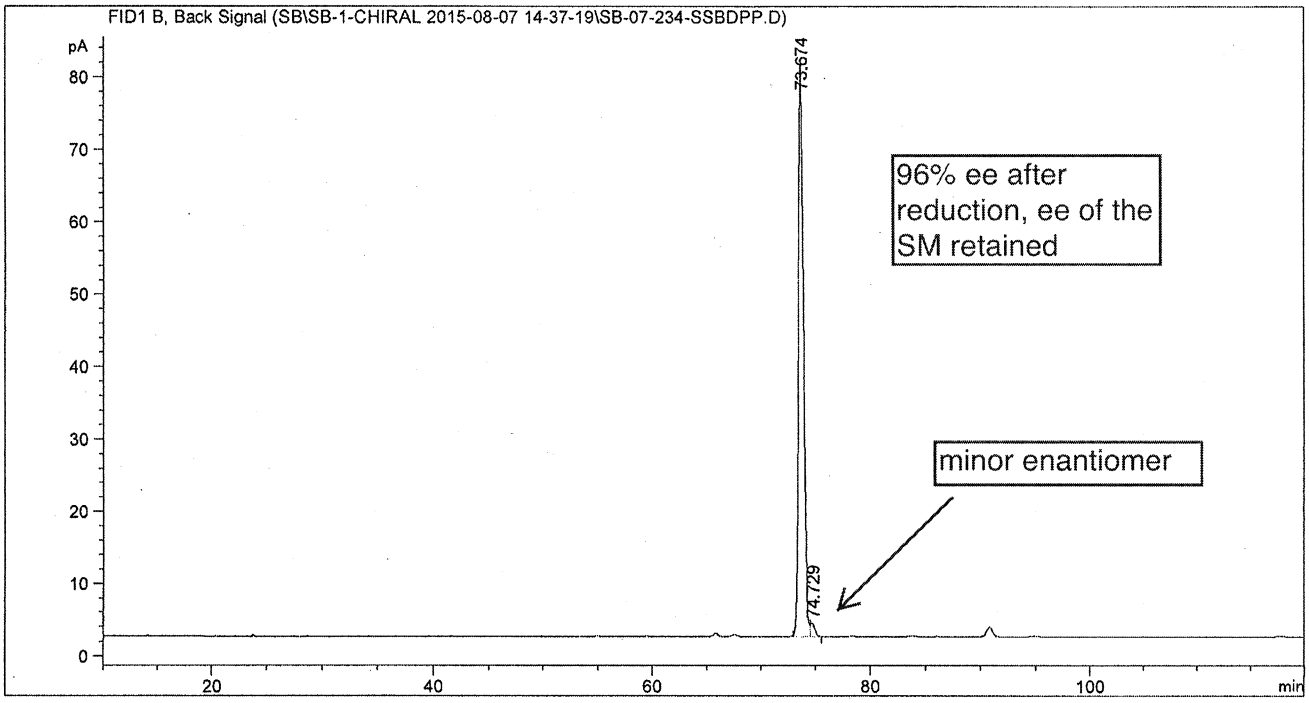
Data File C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2015-08-07 14-37-19\SB-07-234-SSBDPP.D
Sample Name: SB-07-234-SSBDPP.D



(ss-BDPP)
(90°C-iso)
Cyclosil-B

35

```
==========================================================================
Acq. Operator    : BISWAS                          Seq. Line :   1
Acq. Instrument  : Babu                             Location : Vial 201
Injection Date   : 8/7/2015 2:39:20 PM                  Inj :   1
                                                  Inj Volume : 1 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 4 µl
Acq. Method      : C:\CHEM32\1\DATA\SB\SB-1-CHIRAL 2015-08-07 14-37-19\90_C-ISO_CHIRAL.M
Last changed     : 8/7/2015 4:56:27 PM by BISWAS
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\STANDBY.M
Last changed     : 8/7/2015 4:57:31 PM by BISWAS
                   (modified after loading)
Method Info      : Checkout 7820A at OSU; front channel
```




```
==========================================================================
                        Area Percent Report
==========================================================================

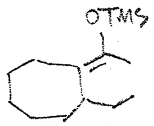
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 B, Back Signal

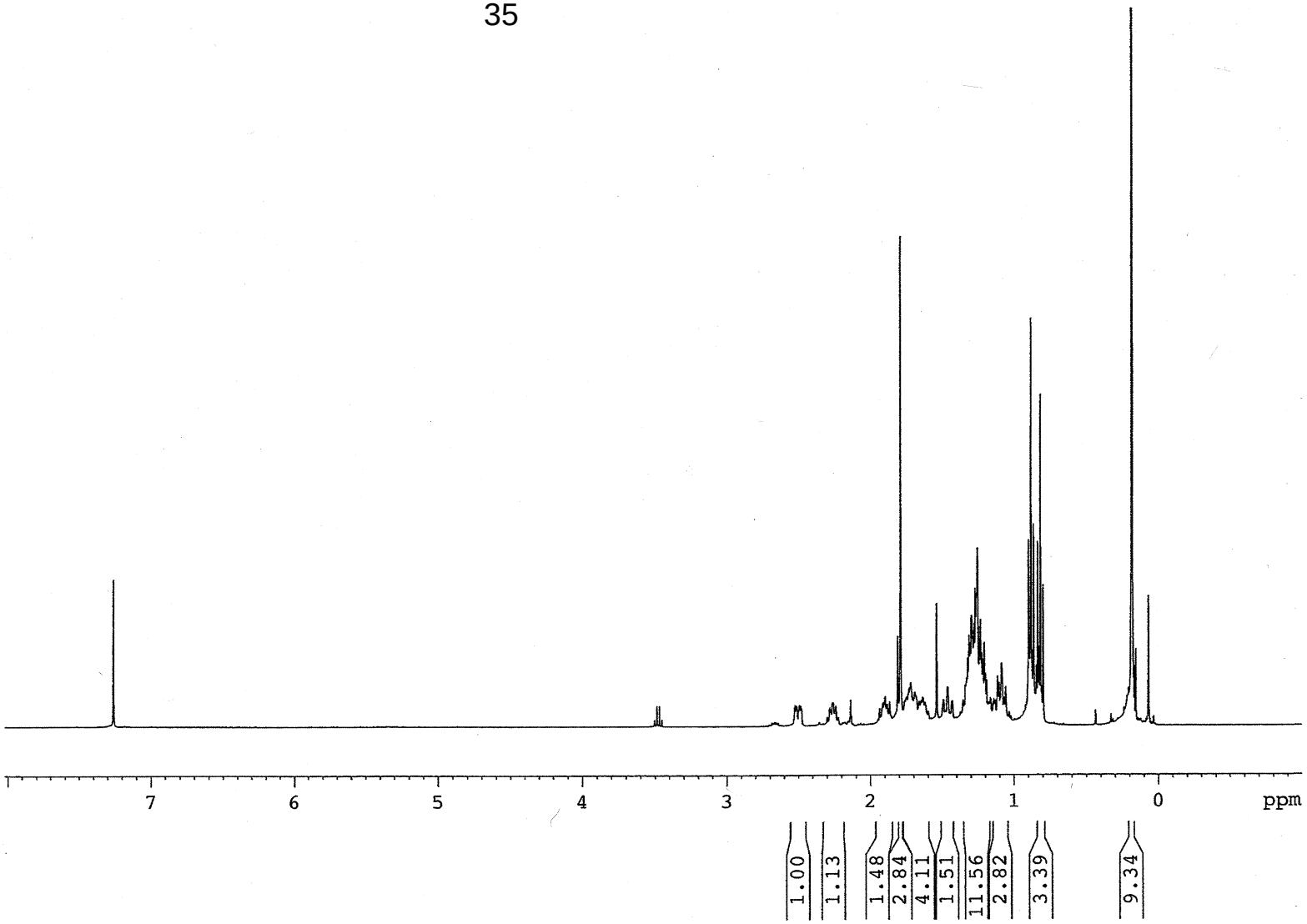
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  73.674 BV    0.5138 2592.39551   78.39325 98.01167
   2  74.729 VB    0.4446   52.59119    1.82713  1.98833

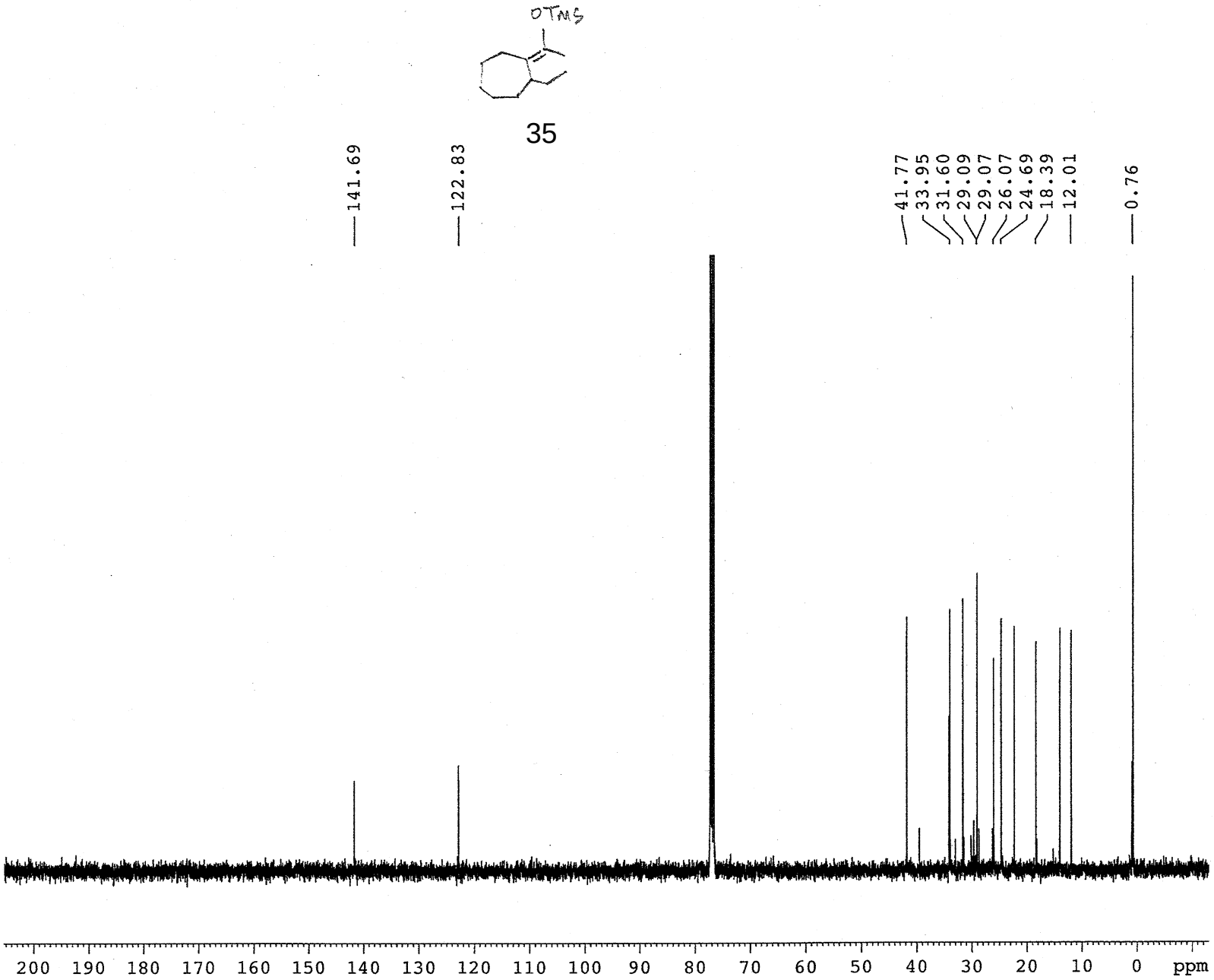
Totals :                  2644.98670   80.22037
```

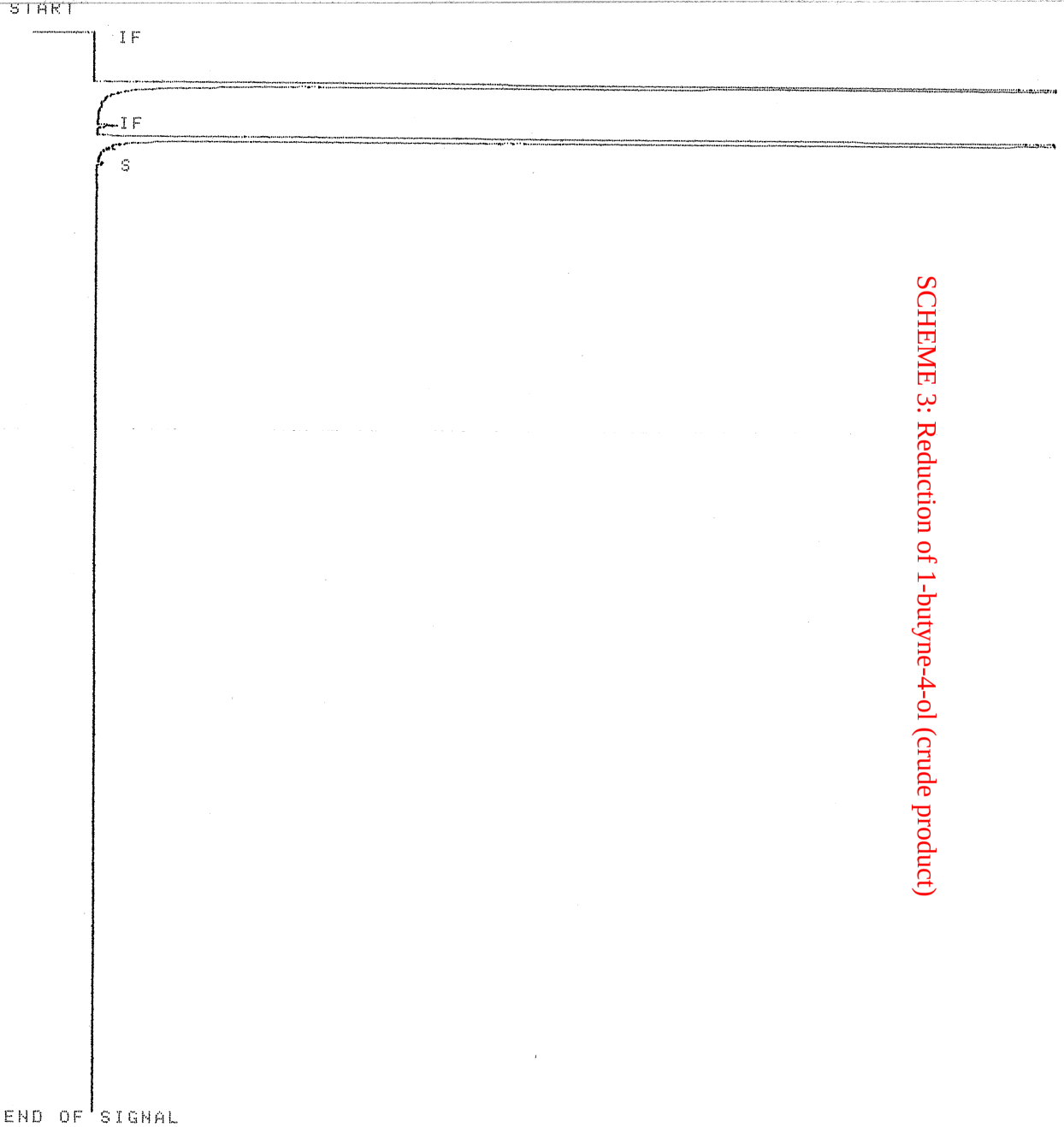

S101



35




OTMS
35
141.69
122.83
41.77
33.95
31.60
29.09
29.07
26.07
24.69
18.39
12.01
0.76
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



END OF SIGNAL

Closing signal file M:SIGNAL .BNA

RUN# 2076 JUL 10, 1901 00:05:56

SIGNAL FILE: M:SIGNAL.BNA
AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.817	30931072	SPB	.066	100.00000

TOTAL AREA=3.0931E+07
MUL FACTOR=1.0000E+00

SCHEME 3: Reduction of 1-butyne-4-ol (crude product)