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SUPPORTING INFORMATION

Palladium nanoparticles in glycerol: clear-cut catalyst for one-pot multi-step processes applied in the synthesis of heterocyclic compounds

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Experimental part

General. All preparations and manipulations were performed using standard Schlenk techniques under argon atmosphere. Unless stated otherwise, commercially compounds were used without further purification. Glycerol was treated under vacuum at 80 °C overnight prior to use. NMR spectra were recorded on a Bruker Avance 300 spectrometer at 293 K (299.7 MHz for ^{1}H NMR and 75.5 MHz for ^{13}C NMR). GC analyses were carried out on an Agilent GC6890 with a flame ionization detector, using a SGE BPX5 column composed by 5% of phenylmethylsiloxane. IR spectra were recorded in the range of 4000-400 cm-1 on a Perkin Elmer Spectrum One FT-IR. Mass chromatograms were carried out by the "Service commun de spectrométrie de masse" of the "Institute de Chimie de Toulouse de l'Université Paul Sabatier". Electronic impact (EI) and Chemical ionization (CI using methane or ammonia as reactant gas), on a TSQ 7000 Thermo Electron apparatus and electrospray (ES) on a API-365 MS/MS Spectrometer (Perkin Elmer Sciex). Gas chromatography analyses were performed with a Perkin–Elmer Clarus 500 chromatograph fitted with a FID and MS-detector, using dodecane as internal standard. The column employed was SGE BPX5 (30 m x 0.32 mm x 0.25 mm) phase composed by 5% of phenyl methylsiloxane. The injector temperature was 250 °C and the flow 2 mL/min. Temperature programme: 40 °C for 2 min; 10 °C/min to 300 °C hold for 5 min. SFC analyses (CO_2/methanol, 4 mL/min) were carried out at 35 °C using a Chiralpak OJ-H 5 μm column and a UV PDA detector. TEM images of particles dispersed in glycerol and in solid state were obtained from transmission electron microscopes JEOL JEM 1400 running at 120 kV and JEOL JEM 2100F running at 200 kV equipped with X PGT analyzer (detection of light elements, resolution 135 eV), at the "Service Commun de Microscopie Electronique de l'Université Paul Sabatier, TEMSCAN". A drop of solution was deposited on a holey carbon grid and the excess of glycerol was removed in order to obtain a film as thin as possible. The nanoparticles size distribution and average diameter were directly determined from TEM images by Image-J software associated to a Microsoft Excel macro developed by Christian Pradel.

Crystallographic data collection and structure determination
The data were collected at low temperature (193 K) on a Bruker-AXS APEX II QUAZAR diffractometer equipped with a 30W air-cooled microfocus source (**a1**, **a30**, **s40u**, **r33** and **g8**) and on a Bruker-AXS SMART APEX II diffractometer (**n35** and **a17u**), using MoKα radiation (λ = 0.71073Å). Phi- and omega- scans were used. The data were integrated with SAINT, and an empirical absorption correction with SADABS was applied.[1] The structures were solved by direct methods (SHELXS-97)[2] and refined using the least-squares method on F^2[2]. All non-H atoms were refined with anisotropic displacement parameters. The H atoms were refined isotropically at calculated positions using a riding model.
CCDC-1020806 (**a1**), CCDC-1020807 (**a30**), CCDC-1020808 (**s40u**), CCDC-1020809 (**r33**), CCDC-1020810 (**n35**), CCDC-1020811 (**a17u**) and CCDC-1020812 (**g8**) contain the

supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.a-c.uk/data_request/cif.

References

[1] SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
[2] G. M. Sheldrick, *Acta Cryst*., 2008, **A64**, 112-122.

Synthesis of Palladium Nanoparticles in glycerol

Pd(OAc)2 (5.10^{-2} mmol, 11.2 mg) both in the presence of m-TPPTS (5.10^{-2} mmol, 28.4 mg), were dissolved in 5 mL of glycerol and stirred at room temperature under argon in a Fischer-Porter bottle until complete dissolution. The system was then pressurised with 3bar of dihydrogen and stirred at 60 °C overnight, leading to a black solution. After releasing the residual gas, the solution was washed with pentane (2x5 mL) under argon atmosphere and dried under reduced pressure for 1 h.

General Procedure for Synthesis of N-Substituted Phthalimides catalyzed by PdNPs in glycerol.

o-Dihaloarene/o-iododerivative (0.4 mmol), amine (0.6 mmol), and DABCO (1 mmol) were consecutively added to 1 mL of a solution of preformed nanoparticles in glycerol. The resulting mixture was stirred at room temperature under argon in a Fisher-Porter bottle. The system was then pressurised with carbon monoxide (0.5 bar) and stirred at 120 °C for 30 min. The mixture was then the cooled to room temperature. The catalytic mixture was extracted with dichloromethane (3 x 10 mL) and the combined organic phases were dried over anhydrous Na2SO4, filtered and the solvent evaporated under reduced pressure. The product was purified by short column chromatography on silica gel. All the products were previously reported, and identified by comparison of their 1H and 13C NMR spectra and GC-MS data with those of authentic samples.

Pd-catalysed domino Sonogashira reaction/Cycloisomerization process. Synthesis of Benzo[b]furan in glycerol

Aryl halide (0.4 mmol), the corresponding terminal alkyne (0.6 mmol), t-BuOK (1 mmol) were consecutively added to 1 mL of a solution of preformed nanoparticles in glycerol. The resulting mixture was heated at 100 °C during 2 h and then cooled to room temperature. The organic products were extracted from the catalytic mixture with dichloromethane (3 x 10 mL) and the combined organic phases were dried over anhydrous Na2SO4, filtered and the solvent evaporated under reduced pressure. The product was purified by short-column chromatography on silica gel. All the products were previously reported, and identified by comparison of their 1H and 13C NMR spectra and GC-MS data with those of authentic samples.

Pd-catalysed sequential processes: carbonylative cyclization followed by domino reaction Sonogashira/Cycloisomerization

1,2-Diiodobenzene (0.4 mmol, 131.96 mg), 4-pentyn-1-amine (0.6 mmol, 49.87 mg), and DABCO (1 mmol, 112.17 mg) were consecutively added to 1 mL of a solution of preformed nanoparticles in glycerol (0.01 mmol Pd, 2.5 mol%). The resulting mixture was stirred at room temperature under argon in a Fisher-Porter bottle. The system was then pressurised with carbon monoxide (0.5 bar) and stirred at 120 °C for 30 min and then cooled to room temperature. The system were added 2-Iodophenol (0.4 mmol, 88.01 mg). The resulting mixture was heated at 100 °C for 2 h. The mixture was then cooled to room temperature. The organic products were extracted from the catalytic mixture with dichloromethane or dichloromethane (3×10 mL), and the combined organic phases were dried over anhydrous Na2SO4, filtered and the solvent was

evaporated under reduced pressure. The product was purified by short-column chromatography on silica gel. Yield: for xx, 111.10 mg (91%)

Pd-catalysed sequential process: carbonylative cyclization followed by hydrogenation
o-dihaloarene/o-iododerivative (0.4 mmol), amine (0.6 mmol), and DABCO (1 mmol) were consecutively added to 1 mL of a solution of preformed nanoparticles in glycerol (0.01 mmol Pd, 2.5 mol%). The resulting mixture was stirred at room temperature under argon in a Fisher-Porter bottle. The system was then pressurised with carbon monoxide (0.5 bar) and stirred at 120 °C for 30 min and then cooled to room temperature. The system was then pressurized with dihydrogen (3 bar) and stirred at 100 °C for 2 h. The mixture was then cooled to room temperature. The catalytic mixture was extracted with dichloromethane (3 x 10 mL) and the combined organic phases were dried over anhydrous Na2SO4, filtered and the solvent evaporated under reduced pressure. The product was purified by short-column chromatography on silica gel. The corresponding product was identified by comparison of its 1H and 13C NMR spectra and GC–MS data with those of an authentic sample.

Pd-catalysed domino process: Sonogashira coupling/Cycloisomerization process. Synthesis of isoindolinone in glycerol
2-Iodo-benzamide (0.4 mmol), terminal alkyne (0.6 mmol), and t-BuOK (1 mmol) were consecutively added to 1 mL of a solution of preformed nanoparticles in glycerol (0.01 mmol Pd, 2.5 mol%). The resulting mixture was heated at 100 °C during 2 h and then cooled to room temperature. The organic products were extracted from the catalytic mixture with dichloromethane (3 x 10 mL) and the combined organic phases were dried over anhydrous Na2SO4, filtered and the solvent evaporated under reduced pressure. The product was purified by short-column chromatography on silica gel. All the products were previously reported, and identified by comparison of their 1H and 13C NMR spectra and GC-MS data with those of authentic samples.

Pd-catalysed sequential process: domino Sonogashira/Cycloisomerization process followed by hydrogenation
2-Iodo-benzamide (0.4 mmol), terminal alkyne (0.6 mmol), and t-BuOK (1 mmol) were consecutively added to 1 mL of a solution of preformed nanoparticles in glycerol (0.01 mmol Pd, 2.5 mol%). The resulting mixture was heated at 100 °C during 2 h and then cooled to room temperature. The system was then pressurized with dihydrogen (3 bar) and stirred at 100 °C for 2 h. The mixture was then cooled to room temperature. The catalytic mixture was extracted with dichloromethane (3 × 10 mL) and the combined organic phases were dried over anhydrous Na2SO4, filtered and the solvent evaporated under reduced pressure. The product was purified by short-column chromatography on silica gel. The corresponding product was identified by comparison of its 1H and 13C NMR spectra and GC–MS data with those of an authentic sample.

Pd/Cu-catalysed sequential process: domino carbonylative cyclization followed by Azide–Alkyne cycloaddition
1,2-Diiodobenzene (0.4 mmol, 131.96 mg), 4-pentyn-1-amine (0.6 mmol, 49.87 mg), and DABCO (1 mmol, 112.17 mg) were consecutively added to 1 mL of a solution of preformed nanoparticles in glycerol (0.01 mmol Pd, 2.5 mol%). The resulting mixture was stirred at room temperature under argon in a Fisher-Porter bottle. The system was then pressurised with carbon monoxide (0.5 bar) and stirred at 120 °C for 30 min and then cooled to room temperature. The system were added consecutively to a solution of preformed Cu2ONP in glycerol (1 mL, 0.01 mmol of Cu) and benzyl azide (0.4 mmol). The resulting mixture was heated at 100 °C for 2 h. The mixture was then cooled to room temperature. The organic products were extracted from

the catalytic mixture with dichloromethane or dichloromethane (3×10 mL), and the combined organic phases were dried over anhydrous Na2SO4, filtered and the solvent was evaporated under reduced pressure. The product was purified by short-column chromatography on silica gel. Yield: for xx, 128.70 mg (93%)

Pd/Cu-catalysed sequential process: domino Sonogashira/Cycloisomerization followed by Azide–Alkyne cycloaddition

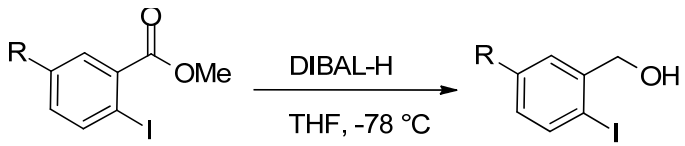
2-Iodophenol (0.4 mmol, 88.01 mg), 1,7-Octadiyne (0.6 mmol, 63.70 mg), t-BuOK (1 mmol, 112.21 mg) were consecutively added to 1 mL of a solution of preformed nanoparticles in glycerol (0.01 mmol Pd, 2.5 mol%). The resulting mixture was heated at 100 °C during 2 h and then cooled to room temperature. The system were added consecutively to a solution of preformed Cu_2ONP in glycerol (1 mL, 0.01 mmol of Cu) and benzyl azide (0.4 mmol). The resulting mixture was heated at 100 °C for 2 h. The mixture was then cooled to room temperature. The organic products were extracted from the catalytic mixture with dichloromethane or dichloromethane (3×10 mL), and the combined organic phases were dried over anhydrous Na_2SO_4, filtered and the solvent was evaporated under reduced pressure. The product was purified by short-column chromatography on silica gel. Yield: for xx, 125.70 mg (95%)

General Procedure for Recycling the Catalytic Phase

A typical experimental procedure to recycle the catalytic phase is described. The glycerol phase from the previous run was maintained for 30 min under dynamic vacuum while stirring at 100 °C. Then, the corresponding reagents were added to the glycerol phase. The catalytic mixture was the treated under the corresponding conditions applied for the first run, the final product was extracted with dichloromethane and analyzed by GC and NMR.

Synthesis of starting materials

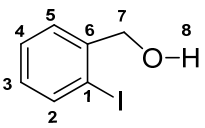
a) Synthesis of benzylic alcohols (*Org. Lett.*, 2012, **14**, 3264)



R= I(1), H(2)

To solution of 1 or 2 (3.79 mmol) in THF (25mL) was added DIBAL-H (9.5 mL, 9.47 mmol) at -78 °C. After completion, the mixture was added 1M NaOH (2mL) and MgSO4. The reaction mixture was filtered and concentrated under reduce pressure. The residue was purified by flash chromatography on silica (30% EtOAc/hexane) to give a white solid.

(2-iodophenyl)methanol. Yield 95%. The spectroscopic data was in good agreement with that reported previously.

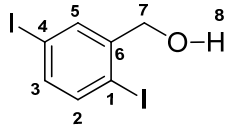


^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 2.02 (br, $1H_8$), 4.68 (s, $2H_7$), 6.97 (m, $1H_{arom}$), 7.34 (m, $1H_{arom}$), 7.45 (m, $1H_{arom}$), 7.84 (m, $1H_{arom}$).

$^{13}C\{^1H\}$ NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 69.4(C_7), 97.6(C_{arom}), 128.5(C_{arom}), 128.6(C_{arom}), 129.4(C_{arom}), 139.3(C_{arom}), 142.6(C_{arom}).

M/S (EI) : t_r= 9.67 min, calc. for C_7H_7IO: 233.95. Found: (*m/z*) = 233.71 (M^+, 100).

2,5-diiodophenyl)methanol. Yield 95%. The spectroscopic data was in good agreement with that reported previously. [1]



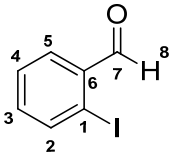
^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 2.08 (br, $1H_8$), 4.63 (s, $2H_7$), 7.27 (d, $1H_{arom}$), 7.50 (d, $1H_{arom}$), 7.80 (s, $1H_{arom}$).

$^{13}C\{^1H\}$ NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 68.7(C_7), 94.4(C_{arom}), 96.3(C_{arom}), 137.1(C_{arom}), 138.2(C_{arom}), 140.8(C_{arom}), 144.8(C_{arom}).

M/S (EI) : t_r= 12.77 min, calc. for **$C_7H_6I_2O$**: 359.8. Found: (*m/z*) = 359.9 (M^+, 100).

a) Synthesis of 2-iodobenzylalcohol (J. Tummatorn and G. Dudley, *Org. Lett*., 2011, **13**, 1572)

To a solution of 2-iodobenzylalcohol (1.31 g, 5.60 mmol) in DCM was added PCC (1.33 g, 6.16 mmol). after being stirred at room temperature for 2 h, the reaction solvent was removed in vacuum and the residue was dissolved with 30% EtOAc/hexane and filtered through a silica gel plug. Evaporation gave 1.26 g (97%) of 2-iodobenzaldehyde as a white solid.

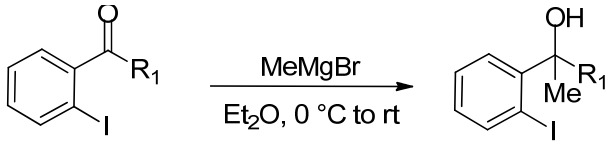


^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 7.28 (td, $1H_{arom}$), 7.47 (t, $1H_{arom}$), 7.88 (dd, $1H_{arom}$), 7.96 (d, $1H_{arom}$), 10.07 (s, 1H8).

$^{13}C\{^1H\}$ NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 100.7(C_{arom}), 128.7(C_{arom}), 130.2(C_{arom}), 135.1(C_{arom}), 135.4(C_{arom}), 140.6(C_{arom}), 195.7(C_7).

M/S (EI) : t_r= 9.07 min, calc. for **C_7H_5IO**: 231.9. Found: (*m/z*) = 231.1 (M^+, 100).

c) Synthesis of 2-iodophenylalcohols

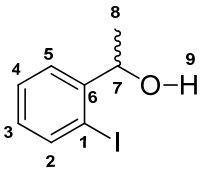


R= H, OMe

R_1= H, Me

A solution of methyl Grignard reagent in Et_2O (4.5 mmol) was added to a stirred solution of *o*-iodobenzaldehyde or methyl-o-iodobenzoate (3.0 mmol) in Et_2O (15 mL) at 0 °C. The mixture was warmed to room temperature over 1 h, monitoring through TLC (30% EtOAc in hexanes). The reaction was quenched through the addition of saturated aqueous NH_4Cl (15 mL), the aqueous phase was extracted with Et_2O (3*20 mL). The combined organic phases were dried (Na_2SO_4) and concentrated in vacuum. The residue was purified through FCC (SiO_2: 30% EtOAc in hexanes) to yield a white solid.

1-(2-iodophenyl)ethanol. Yield 95%. The spectroscopic data was in good agreement with that reported previously (D. Sälinger and R. Brückner, *Chem. Eur. J*., 2009, **15**, 6688)

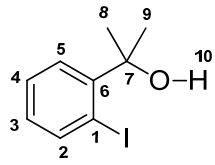


^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 1.38 (d, $3H_8$), 2.23 (br, $1H_9$), 4.95 (q, $1H_7$), 6.85 (m, $1H_{arom}$), 7.29 (m, $1H_{arom}$), 7.46 (m, $1H_{arom}$), 7.73 (m, $1H_{arom}$).

$^{13}C\{^1H\}$ NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 23.9(C_8), 73.7(C_7), 97.3(C_{arom}), 126.0(C_{arom}), 128.5(C_{arom}), 129.3(C_{arom}), 139.2(C_{arom}), 147.5(C_{arom}).

M/S (EI) : t_r= 9.53 min, calc. for **C_8H_9IO**: 247.71. Found: (*m/z*) = 247.54 (M^+, 100).

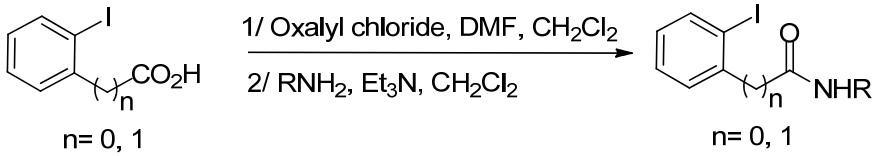
2-(2-iodophenyl)propan-2-ol. Yield 91%. The spectroscopic data was in good agreement with that reported previously (R. Moss, K. Jespersen and J. Westbrook, *J. Am. Chem. Soc*., 1989, **111**, 250)



^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 1.65 (s, $6H_{8,9}$), 2.58 (br, $1H_{10}$), 6.78 (m, $1H_{arom}$), 7.22 (m, $1H_{arom}$), 7.55 (m, $1H_{arom}$), 7.85 (m, $1H_{arom}$).
$^{13}C\{^1H\}$ NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 29.7($C_{8,9}$), 72.1(C_7), 93.3(C_{arom}), 126.8(C_{arom}), 128.1(C_{arom}), 128.6(C_{arom}), 142.8(C_{arom}), 148.6(C_{arom}).

M/S (EI) : t_r= 9.94 min, calc. for **$C_9H_{11}IO$**: 261.9. Found: (*m/z*) = 261.2 (M^+, 100).

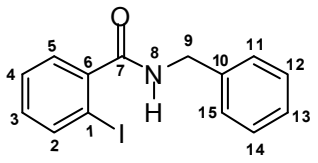
c) Synthesis of 2-iodobenzamides



n= 0, 1 n= 0, 1

To a solution of the corresponding carboxylic acid (10 mmol) in CH_2Cl_2 (20ml) was added oxalyl chloride (1.29 ml, 15 mmol) and DMF (73µL, 0.95 mmol). Stirring was continued till the solid disappeared. The volatile was removed under reduced pressure. Then CH_2Cl_2 (20ml) was added followed by addition of the corresponding amine (10 mmol) and triethylamine (1.39 ml, 10 mmol). After being stirred at room temperature for another one hour, the mixture was quenched with water and the aqueous phase was extracted with CH_2Cl_2. The combined organic phase was washed with 10% HCl (aq.), Water, saturated $NaHCO_3$ (aq.) and brine, successively, and dried over Na_2SO_4. Evaporation of the solvent afforded the corresponding amide used without further purification.

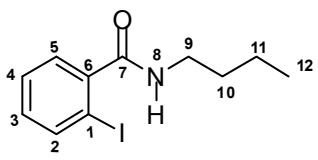
N-benzyl-2-iodobenzamide. Yield 99%. The spectroscopic data was in good agreement with that reported previously (B. Yao, C. Jaccoud, Q. Wang and J. Zhu, *Chem. Eur. J*., 2012, **18**, 5864)



^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 4.64 (s, $2H_9$), 6.16 (br, $1H_8$), 7.07 (m, $1H_{arom}$), 7.30 (m, $7H_{arom}$), 7.84 (d, $1H_{arom}$).
$^{13}C\{^1H\}$ NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 44.3(C_9), 92.5(C_{arom}), 127.8(C_{arom}), 128.2(C_{arom}), 128.4(C_{arom}), 128.8(C_{arom}), 131.2(C_{arom}), 137.7(C_{arom}), 139.9(C_{arom}), 142.1(C_{arom}), 169.4(C_7).

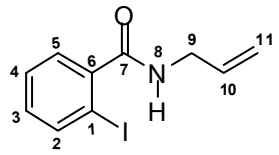
M/S (EI) : t_r= 15.32 min, calc. for **$C_{14}H_{12}INO$**: 337.16. Found: (*m/z*) = 337.18 (M^+, 100).

N-butyl-2-iodobenzamide. Yield 97%. The NMR spectral data was in good agreement with that reported previously (*N*. Schröder, J. Wencel-Delord, and F. Glorius, *J. Am. Chem. Soc.*, 2012, **134**, 8298)



^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 0.80 (t, $3H_{12}$), 1.22 (m, $2H_{11}$), 1.44 (q, $2H_{10}$), 3.22 (q, $2H_9$), 6.82 (br, $1H_8$), 6.91 (m, $1H_{arom}$), 7.13 (m, $2H_{arom}$), 7.67 (d, $1H_{arom}$).
$^{13}C\{^1H\}$ NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 13.4(C_{12}), 19.9(C_{11}), 30.8(C_{10}), 39.4(C_9), 92.4(C_{arom}), 127.5(C_{arom}), 127.7(C_{arom}), 130.3(C_{arom}), 139.1(Carom), 142.1(C_{arom}), 159.4(C_{arom}), 169.0(C_7).

M/S (EI) : t_r= 12.93 min**,** calc. for **$C_{11}H_{14}INO$**: 303.01. Found: (*m/z*) = 303.07 (M^+, 100).

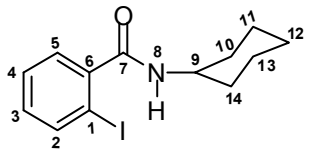


Yield 97%. The NMR spectral data was in good agreement with that reported previously (S. Couty, B. Liegault, C. Meyer and J. Cossy, *Tetrahedron*, 2006, **62**, 3882)

^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 4.02 (d, $2H_9$), 5.24 (d, $2H_{11}$), 5.90 (m, $1H_{10}$), 6.09 (br, $1H_8$), 7.03 (m, $1H_{arom}$), 7.33 (m, $1H_{arom}$), 7.83 (d, $2H_{arom}$).

^{13}C{^{1}H} NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 42.2(C_9), 92.5(C_{arom}), 117.1(C_{11}), 128.1(C_{arom}), 128.4(C_{arom}), 131.1(C_{arom}), 133.7(C_{10}), 139.9(C_{arom}), 142.1(C_{arom}), 169.2(C_7).

M/S (EI) : t_r= 12.63 min**,** calc. for **$C_{10}H_{10}INO$**: 287.21. Found: (*m/z*) = 287.04 (M^+, 100).

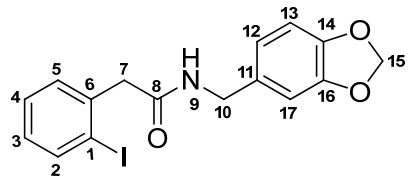


Yield 97%. The NMR spectral data was in good agreement with that reported previously.

^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 1.12 (m, $4H_{11,13}$), 1.60 (m, $2H_{12}$), 1.80 (m, $4H_{10,14}$), 2.52 (m, $1H_9$), 7.62 (m, $1H_{arom}$), 7.80 (m, $1H_{arom}$), 7.97 (m, $2H_{arom}$), 8.03 (br, $1H_8$).

^{13}C{^{1}H} NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 24.8(C_{12}), 24.9(C_{13}), 25.7(C_{11}), 32.3(C_{14}), 32.4(C_{10}), 51.6(C_9), 92.8(C_{arom}), 127.7(C_{arom}), 130.7(C_{arom}), 131.2(C_{arom}), 141.5(C_{arom}), 142.4(C_{arom}), 167.2(C_7).

M/S (EI) : t_r= 15.04 min**,** calc. for **$C_{13}H_{16}INO$**: 329.03. Found: (*m/z*) = 329.14 (M^+, 100).

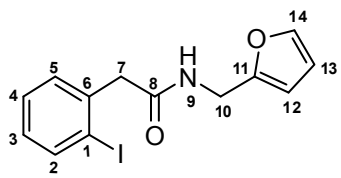


Yield 91%. The NMR spectral data was in good agreement with that reported previously.

^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 3.85 (s, $2H_{10}$), 4.24 (s, $2H_7$), 6.07 (s, $2H_{15}$), 6.76 (m, $2H_{arom}$), 7.03 (m, $3H_{arom}$), 7.32 (m, $1H_{arom}$), 7.83 (m, $1H_{arom}$), 8.03 (br, $1H_9$).

^{13}C{^{1}H} NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 39.0(C_7), 43.9(C_{10}), 98.8(C_{arom}), 101.2(C_{15}), 107.7(C_{arom}), 109.2(C_{arom}), 128.1(C_{arom}), 129.2(C_{arom}), 130.6(C_{arom}), 131.2(C_{arom}), 145.9(C_{arom}), 146.8(C_{arom}), 144.8(C_{arom}), 171.9(C_8).

M/S (EI) : t_r= 16.57 min**,** calc. for **$C_{16}H_{14}INO_3$**: 395.01. Found: (*m/z*) = 395.19 (M^+, 100).



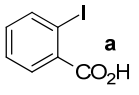
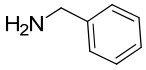
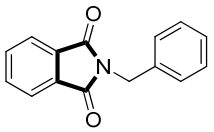
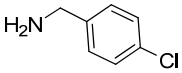
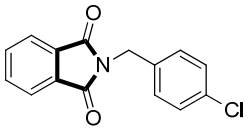
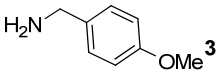
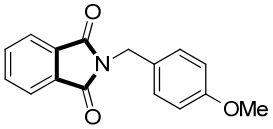
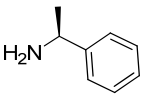
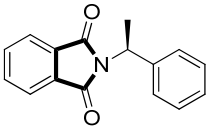
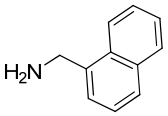
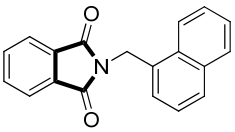
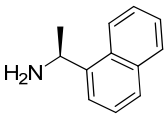
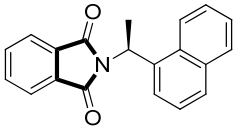
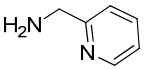
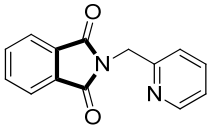
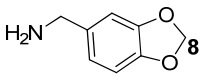
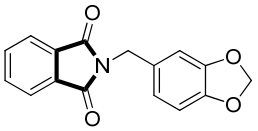
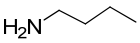
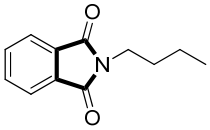
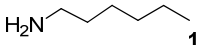
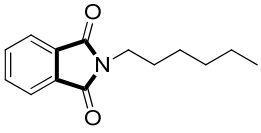
Yield 91%. The NMR spectral data was in good agreement with that reported previously.

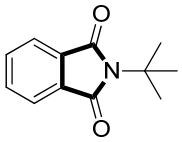
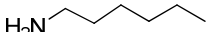
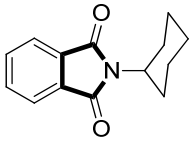
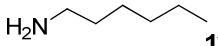
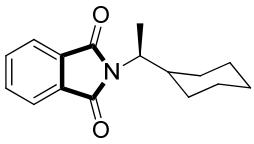
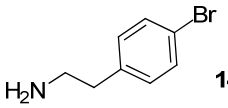
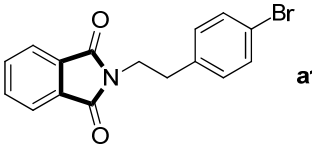
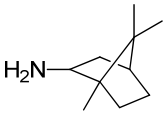
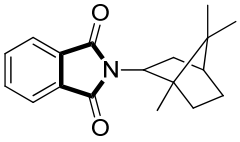
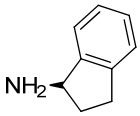
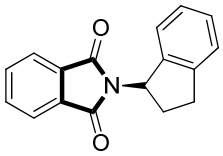
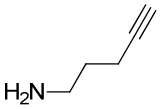
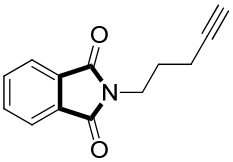
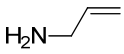
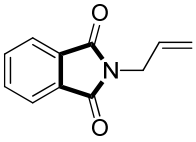
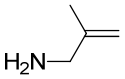
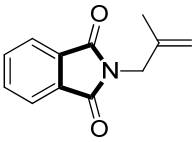
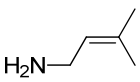
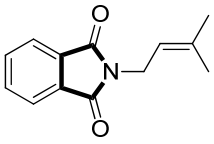
^{1}H NMR (300 MHz, $CDCl_3$, 298 K): δ (ppm) = 3.74 (s, $2H_7$), 4.43 (d, $2H_{10}$), 5.78 (br, $1H_9$), 6.30 (d, $2H_{12,13}$), 6.98 (m, $1H_{14}$), 7.34 (m ,$3H_{arom}$), 7.87 (d, $1H_{arom}$).

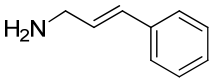
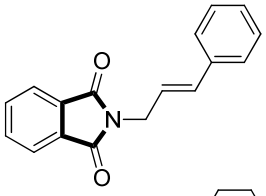
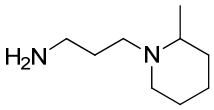
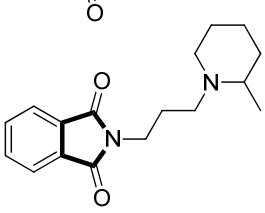
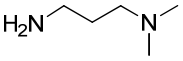
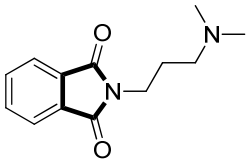
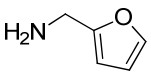
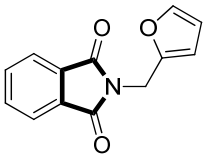
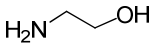
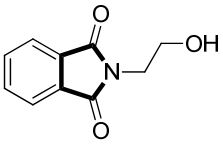
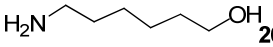
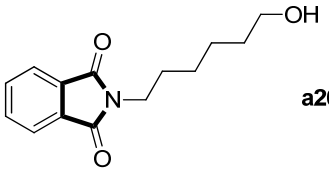
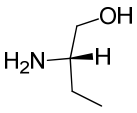
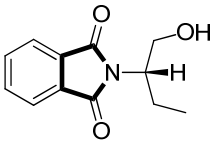
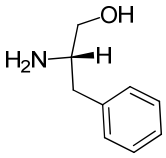
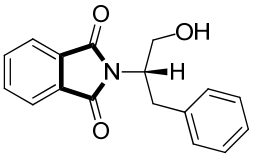
^{13}C{^{1}H} NMR (76.5 MHz, $CDCl_3$, 298 K): δ (ppm) = 36.8(C_{10}), 48.6(C_7), 101.5(C_{12}), 107.4(C_{13}), 110.5(C_{arom}), 128.9(C_{arom}), 129.2(C_{arom}), 131.0(C_{arom}), 138.1(C_{arom}), 140.2(C_{arom}), 142.3(C_{14}), 151.4(C_{11}), 169.6(C_8).

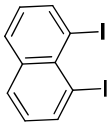
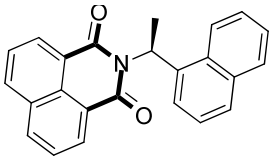
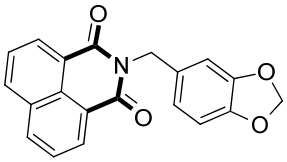
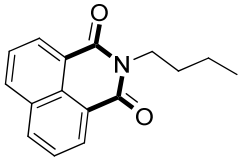
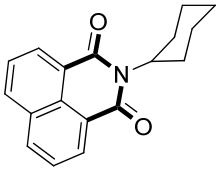
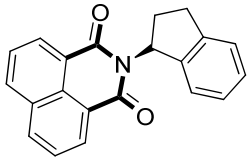
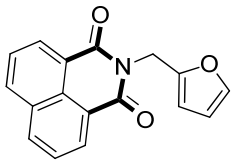
M/S (EI) : t_r= 14.94 min**,** calc. for **$C_{13}H_{12}INO_2$**: 341.19. Found : (*m/z*) = 341.20 (M^+, 100).

Table S1. Pd-catalysed three-component carbonylative cyclisations for the synthesis of N-substituted isoindol-1,3-diones (**a1-a28**) and isoquinolin-1,3-diones (**g** derivatives).[a]

Entry	Substrat	Amine	Product	Yield (%)[b]
1	 **a**	 **1**	 **a1**	98
2	**a**	 **2**	 **a2**	98
3	**a**	 **3**	 **a3**	95
4	**a**	 **4**	 **a4**	90
5	**a**	 **5**	 **a5**	90
6	**a**	 **6**	 **a6**	90
7	**a**	 **7**	 **a7**	96
8	**a**	 **8**	 **a8**	95
9	**a**	 **9**	 **a9**	98
10	**a**	 **10**	 **a10**	96

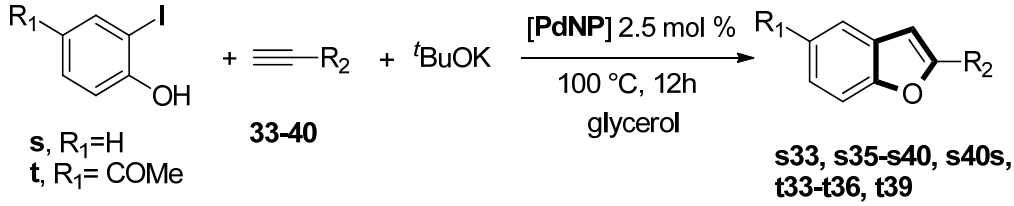
11	**a**		**11**		**a11**	92
12	**a**		**12**		**a12**	98
13	**a**		**13**		**a13**	96
14	**a**		**14**		**a14**	95
15	**a**		**15**		**a15**	92
16	**a**		**16**		**a16**	90
17	**a**		**17**		**a17**	90
18	**a**		**18**		**a18**	91
19	**a**		**19**		**a19**	96
20	**a**		**20**		**a20**	91

21	**a**		**21**		**a21**	94
22	**a**		**22**		**a22**	91
23	**a**		**23**		**a23**	95
24	**a**		**24**		**a24**	91
25	**a**		**25**		**a25**	92
26	**a**		**26**		**a26**	92
27	**a**		**27**		**a27**	96
28	**a**		**28**		**a28**	90

29	 **g**	**6**		**g6**	93
30	**g**	**(8)**		**g8**	95
31	**g**	**(9)**		**g9**	98
32	**g**	**12**		**g12**	91
33	**g**	**16**		**g16**	91
34	**g**	**24**		**g24**	97

[a] Results from duplicate experiments. Reaction conditions: 0.4 mmol of 2-iodobenzoic acid or 1,8 diiodonaphtalene, 0.4 mmol of amine in glycerol (1 mL, 13.6 mmol) at 120 °C, 0.5 bar CO for 0.5h, using 2.5 mol% **PdNP**. [b] Isolated yields after column chromatography; compounds identified by ^{1}H and ^{13}C NMR and GC-MS.

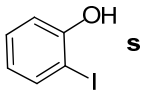
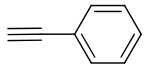
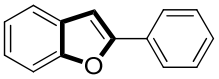
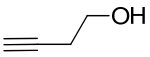
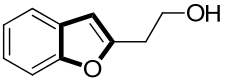
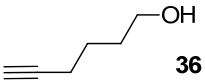
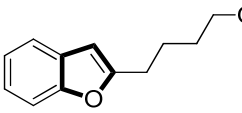
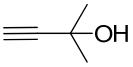
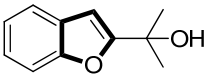
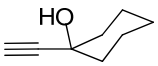
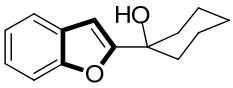
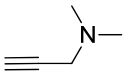
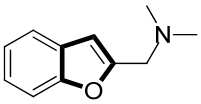
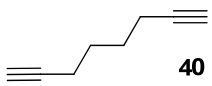
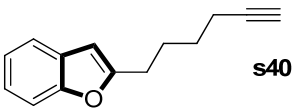
Table S2. Synthesis of 2-substituted benzofurans (s33, s35-s40, t33-t36 and t39-t40) by Pd-catalysed Sonogashira coupling followed by intramolecular cyclisation.[a]

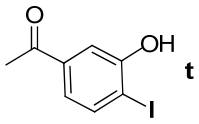
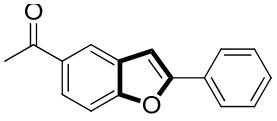
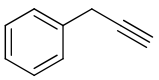
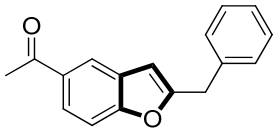
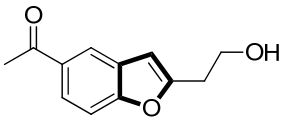
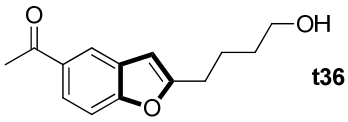
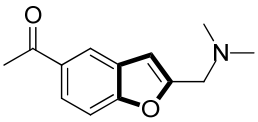
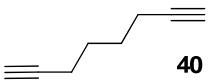
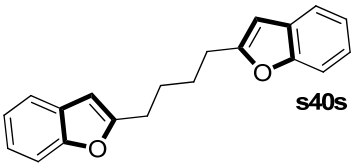


s, R_1=H
t, R_1= COMe

33-40

s33, s35-s40, s40s, t33-t36, t39

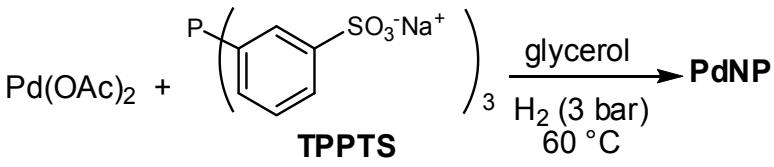
Entry	Substrat	R_2	Product	Yield (%)[b]
1	 **s**	 **33**	 **s33**	91
2	**s**	 **35**	 **s35**	93
3	**s**	 **36**	 **s36**	98
4	**s**	 **37**	 **s37**	91
5	**s**	 **38**	 **s38**	97
6	**s**	 **39**	 **s39**	97
7	**s**	 **40**	 **s40**	96

8	 **t**	**33**		**t33**	98
9	**t**	 **34**		**t34**	95
10	**t**	**35**		**t35**	94
11	**t**	**36**		**t36**	93
12	**t**	**39**		**t39**	92
13	**s**	 **40**		**s40s**	91

[a] Results from duplicate experiments. Reaction conditions: 0.4 mmol of iodophenol or 4'-hydroxy-3'-iodoacetophenone and 0.6 mmol of alkyne in glycerol (1 mL, 13.6 mmol) at 100 °C for 12 h, using 2.5 mol% **PdNP**.
[b] Isolated yields after column chromatography; compounds identified by ^{1}H and ^{13}C NMR and GC-MS.

Table S3. Crystallographic data for **a1**, **a30**, **s40u**, **r33**, **n35**, **a17u** and **g8**.

	a1	**a30**	**s40u**	**r33**	**n35**	**a17u**	**g8**
chemical formula	$C_{15}H_{11}NO_2$	$C_{22}H_{20}N_2O_4$	$C_{21}H_{21}N_3O$	$C_{23}H_{16}O$	$C_{22}H_{23}NO_2$	$C_{20}H_{18}N_4O_2$	$C_{20}H_{13}NO_4$
*M*r	237.25	376.4	331.41	308.36	333.41	346.38	331.31
crystal system	triclinic	monoclinic	monoclinic	monoclinic	triclinic	triclinic	triclinic
space group	$P\ \bar{1}$	$P\ 2_1/n$	$P\ c$	$P\ 2_1$	$P\ \bar{1}$	$P\ \bar{1}$	$P\ \bar{1}$
a [Å]	7.1432(4)	7.0179(8)	18.2723(16)	11.008(3)	9.1264(5)	5.6257(7)	7.1728(2)
b [Å]	8.4932(4)	4.6365(4)	5.5654(4)	7.5883(19)	9.7169(6)	19.7469(19)	8.0196(2)
c [Å]	10.1874(5)	29.074(3)	8.7266(7)	19.571(5)	10.2731(7)	23.768(2)	12.8455(4)
α [°]	99.388(2)	90	90	90	95.504(4)	79.737(6)	84.142(2)
β [°]	97.577(2)	92.757(3)	103.633(4)	90.988(10)	91.157(4)	88.280(7)	89.687(2)
γ [°]	106.771(2)	90	90	90	101.221(4)	82.746(7)	87.901(2)
V [Å^3]	573.27(5)	944.93(17)	862.43(12)	1634.6(7)	888.78(10)	2577.2(5)	734.56(4)
Z	2	2	2	4	2	6	2
Reflections collected	8247	7457	11512	11659	13707	44315	12101
Independent reflections	2080 [R(int) = 0.0205]	1947 [R(int) = 0.0402]	3137 [R(int) = 0.0256]	5736 [R(int) = 0.08]	3588 [R(int) = 0.0591]	8707 [R(int) = 0.1375]	2691 [R(int) = 0.0279]
ρ_{calc} [g cm^{-3}]	1.374	1.323	1.276	1.253	1.246	1.339	1.498
μ ($Mo_{K\alpha}$) [mm^{-1}]	0.092	0.092	0.080	0.075	0.079	0.090	0.106
crystal size (mm^3)	0.10 x 0.08 x 0.08	0.18 x 0.16 x 0.04	0.24 x 0.24 x 0.20	0.16x 0.08 x 0.02	0.20 x 0.12 x 0.08	0.40 x 0.08 x 0.03	0.12 x 0.10 x 0.06
GOF on F²	1.048	1.048	1.039	0.972	1.079	1.075	1.028
R ($I > 2\sigma(I)$)	0.0340	0.0445	0.0412	0.0704	0.0587	0.0759	0.0379
*w*R² (all data)	0.0899	0.1093	0.1114	0.1720	0.2170	0.1814	0.1044
Largest difference peak and hole [e Å^{-3}]	0.230 and -0.190	0.159 and -0.170	0.312 and -0.227	0.243 and -0.197	0.326 and -0.319	0.219 and -0.271	0.161 and -0.253



Scheme S1. Synthesis of PdNPs in neat glycerol.

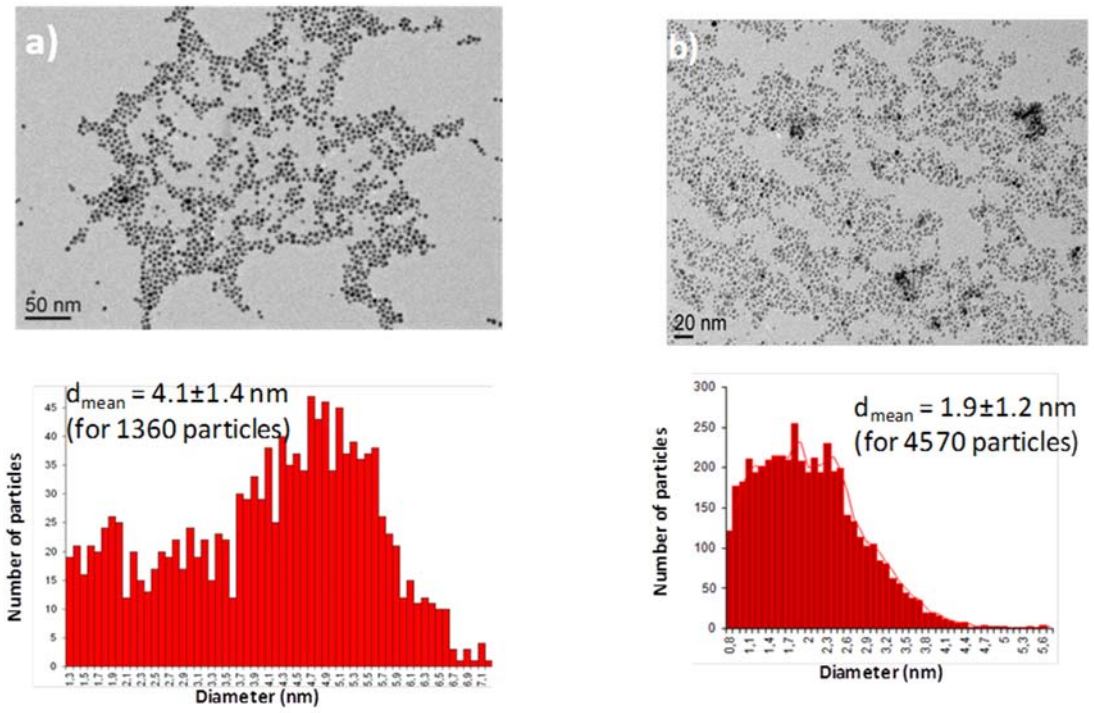
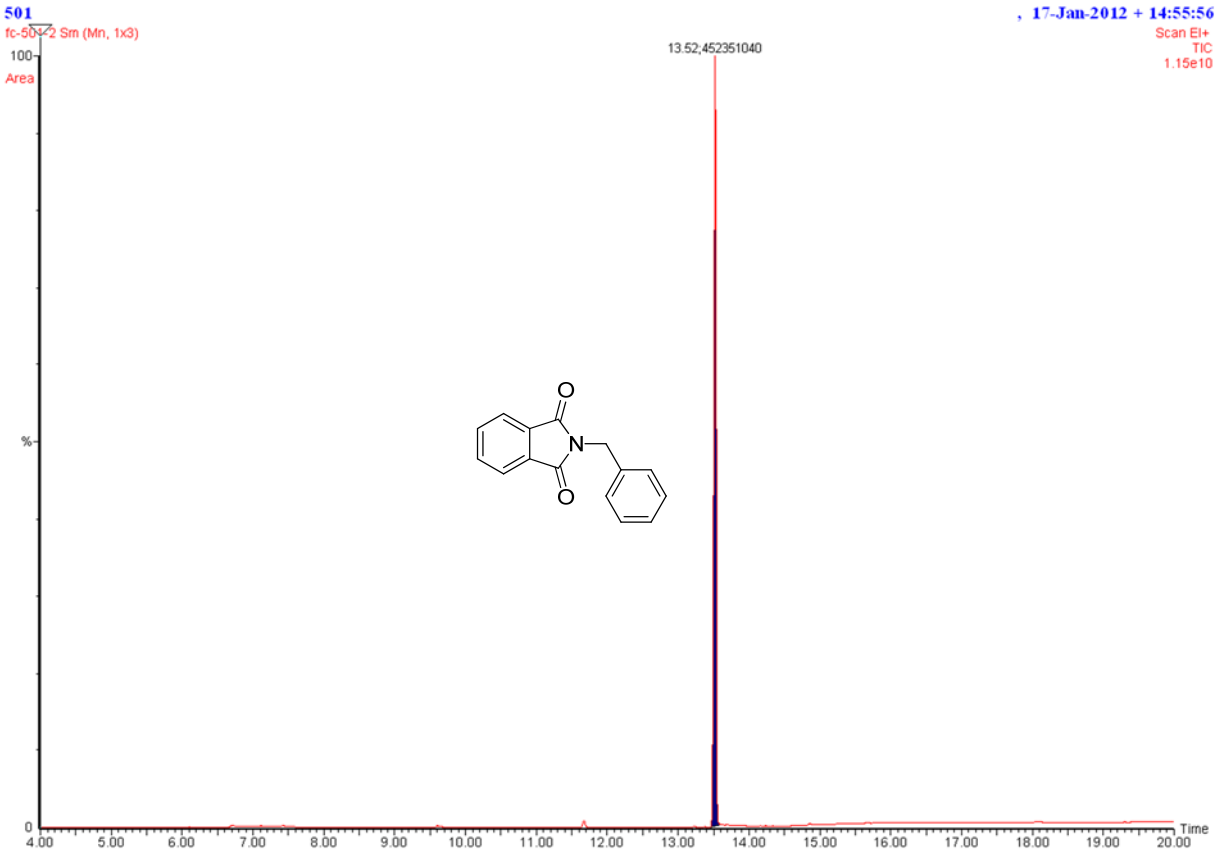
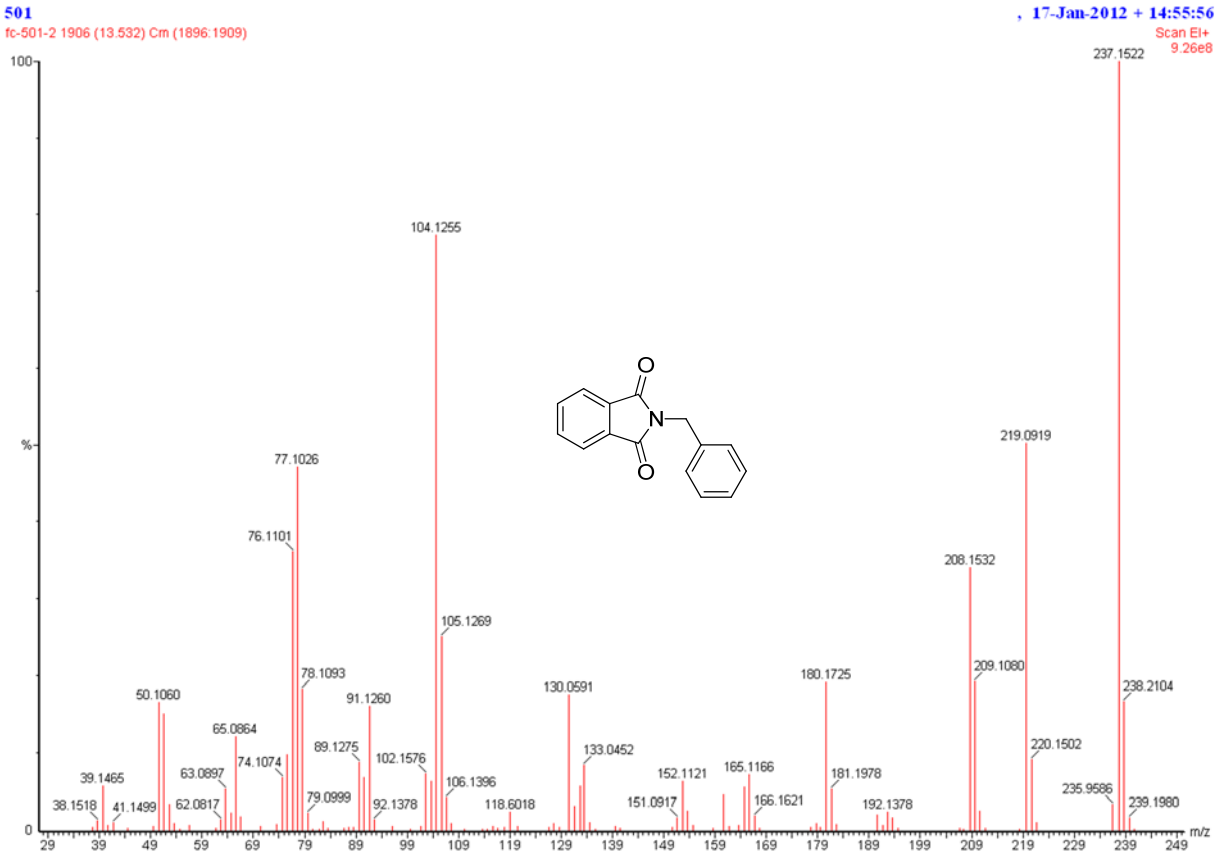


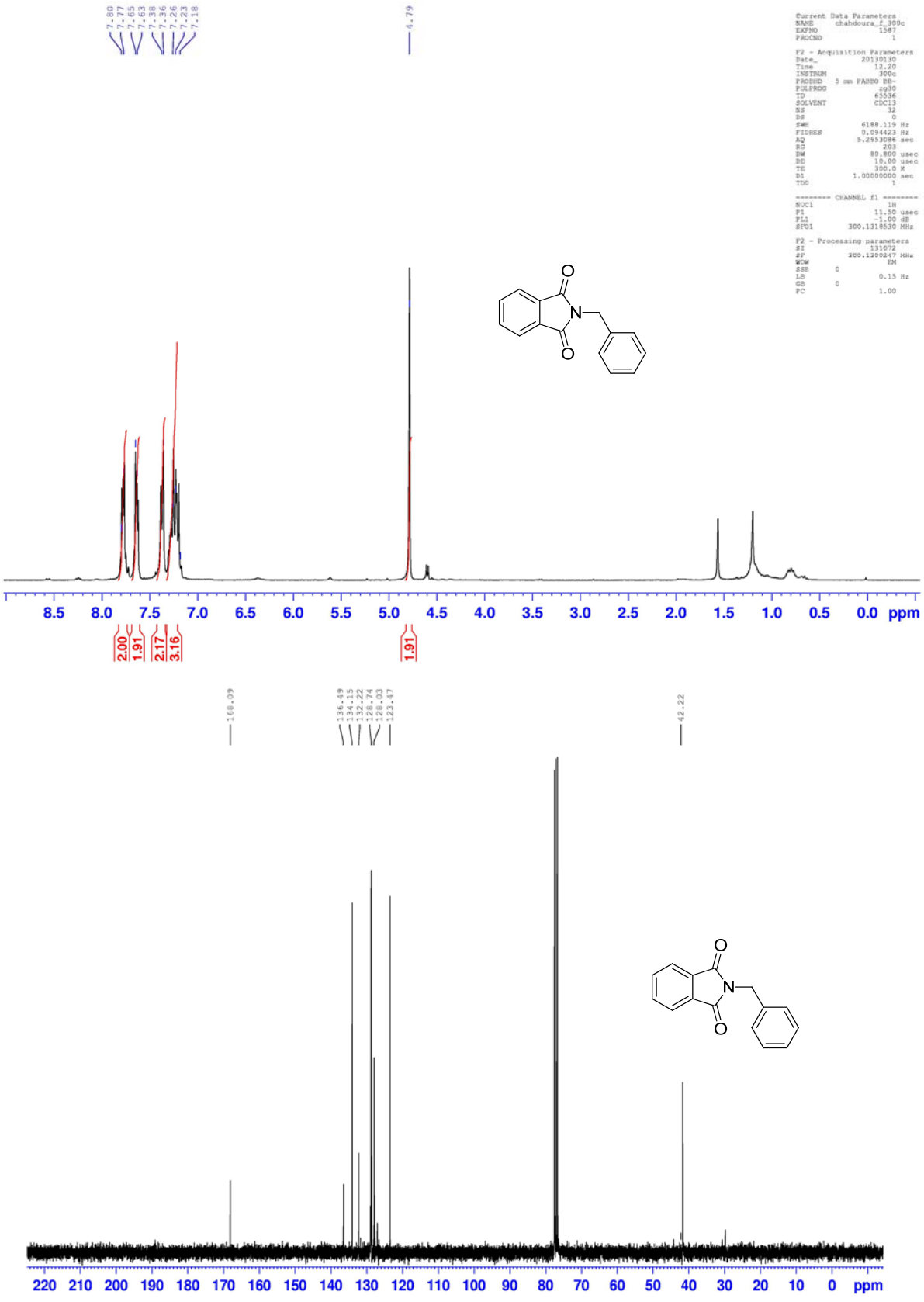
Figure S1 TEM images corresponding to the **PdNP** in neat glycerol (a) and after the fifth recycling run (b).

SPECTRA OF SYNTHESISED HETEROCYCLES

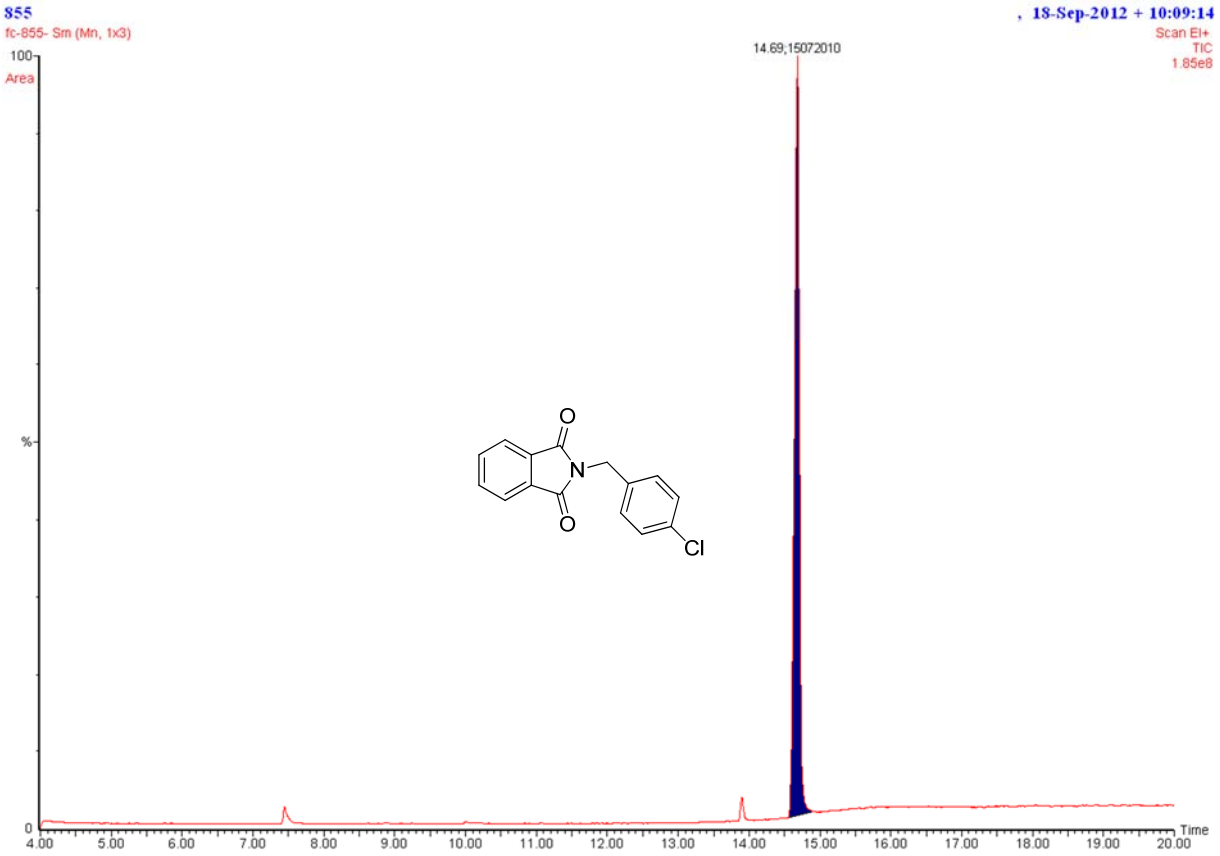
following the order of appearance in the main text

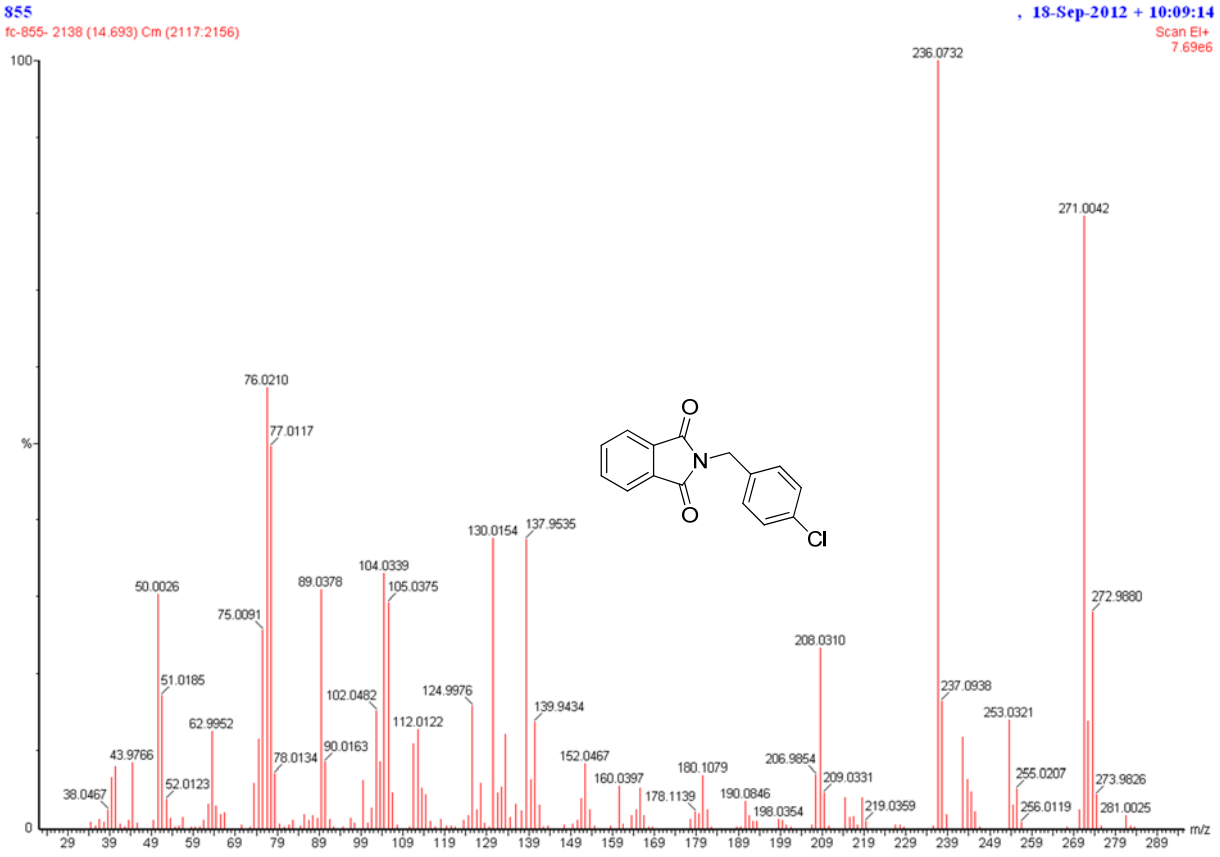

501
fc-501-2 Sm (Mn, 1x3)
100
Area
%
0
13.52;452351040
, 17-Jan-2012 + 14:55:56
Scan EI+
TIC
1.15e10
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
Time

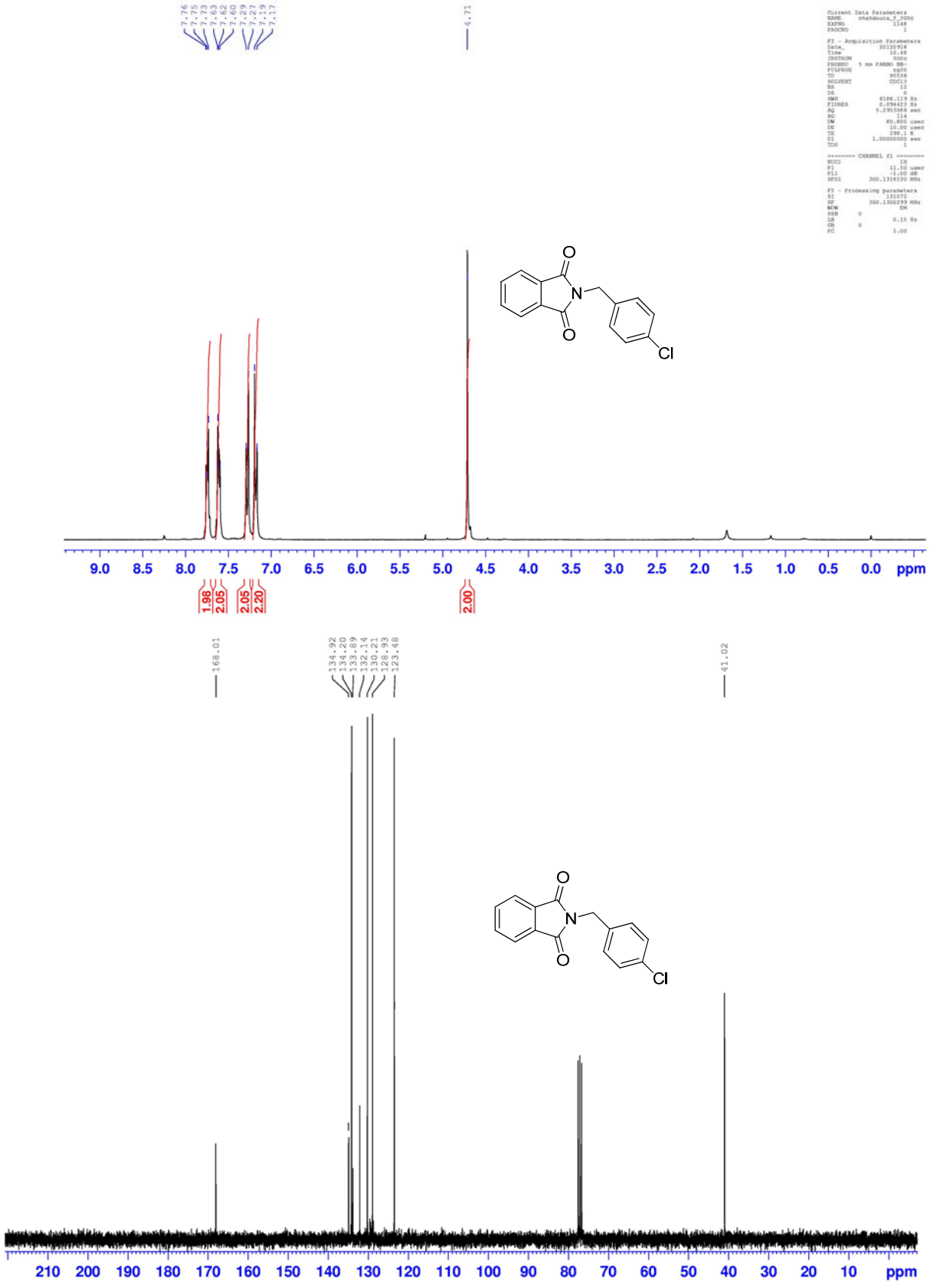

501
fc-501-2 1906 (13.532) Cm (1896:1909)
, 17-Jan-2012 + 14:55:56
Scan EI+
9.26e8
100
%
0
237.1522
104.1255
219.0919
77.1026
76.1101
208.1532
105.1269
209.1080
78.1093
180.1725
238.2104
50.1060
130.0591
91.1260
65.0864
89.1275
133.0452
220.1502
74.1074
102.1576
152.1121
165.1166
181.1978
39.1465
63.0897
106.1396
235.9586
38.1518
41.1499
62.0817
79.0999
92.1378
118.6018
151.0917
166.1621
192.1378
239.1980
29 39 49 59 69 79 89 99 109 119 129 139 149 159 169 179 189 199 209 219 229 239 249
m/z



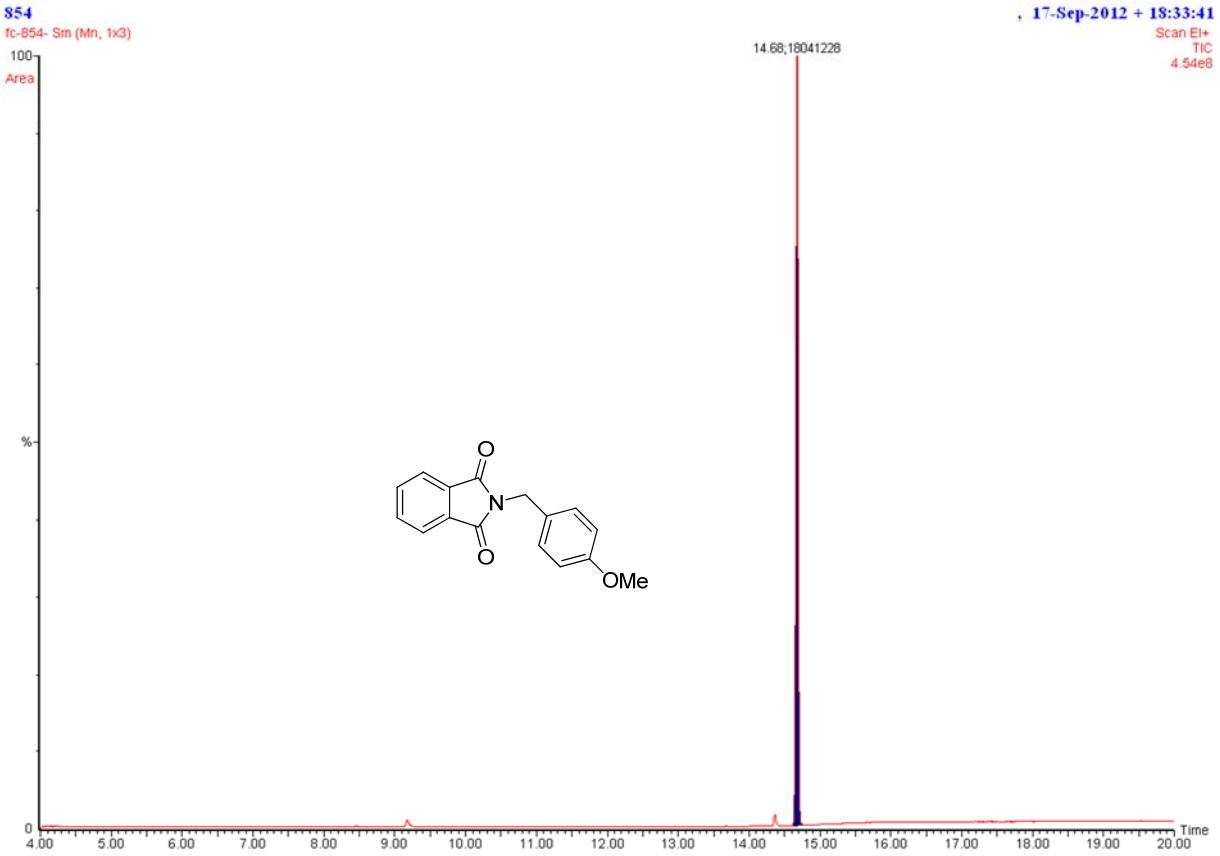
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a1**

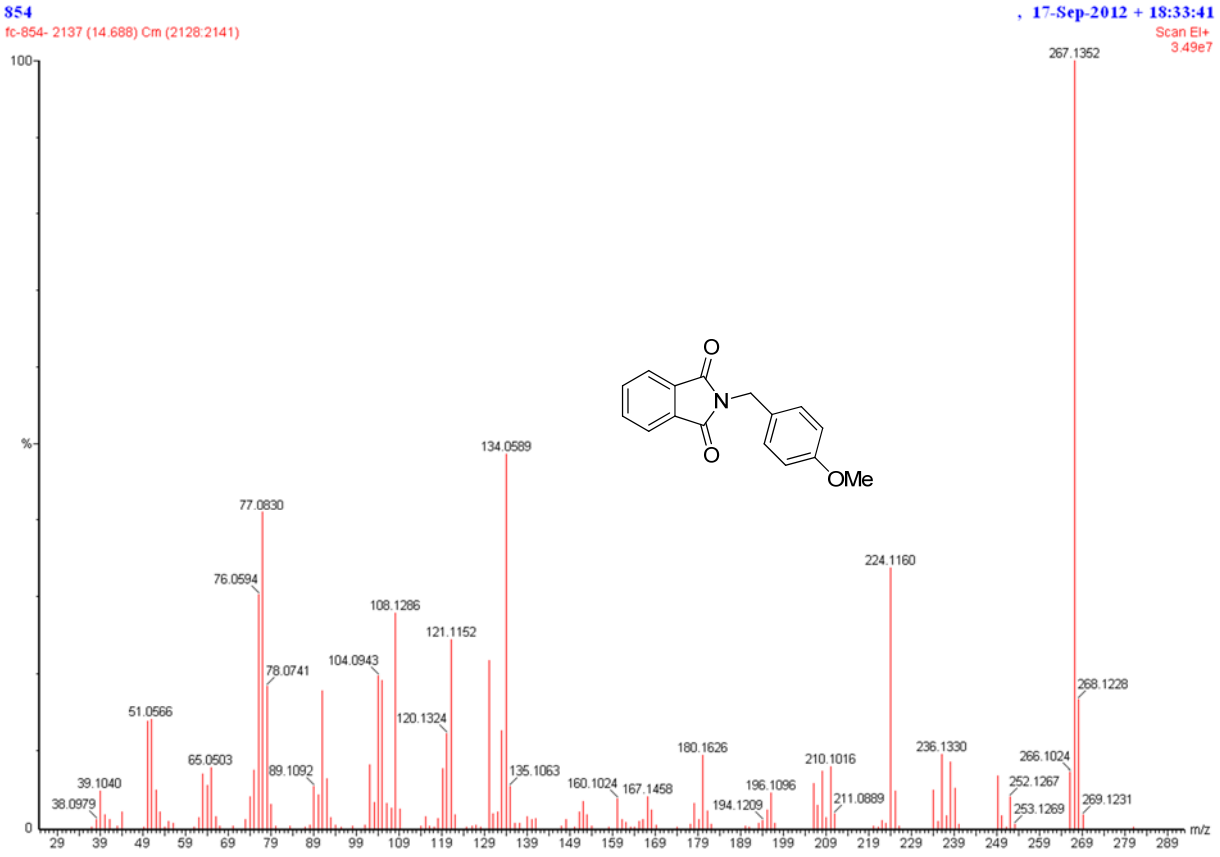

855
fc-855- Sm (Mn, 1x3)
, 18-Sep-2012 + 10:09:14
Scan EI+
TIC
1.85e8
Area
14.69;15072010
%
Time
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
O
N
O
Cl

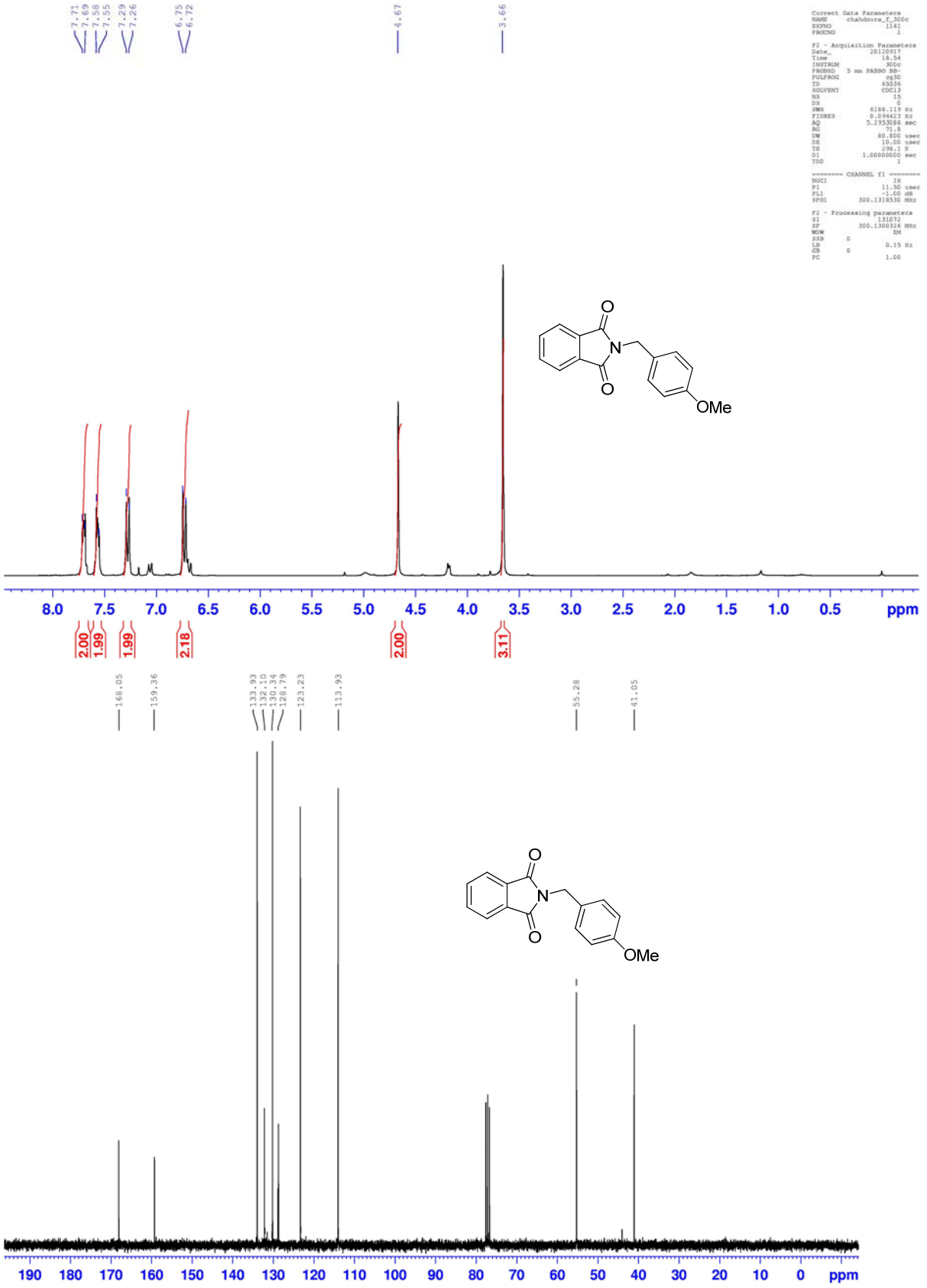

855
fc-855- 2138 (14.693) Cm (2117:2156)
, 18-Sep-2012 + 10:09:14
Scan EI+
7.69e6
%
m/z
236.0732
271.0042
272.9880
76.0210
77.0117
130.0154
137.9535
104.0339
105.0375
89.0378
50.0026
75.0091
208.0310
51.0185
102.0482
124.9976
237.0938
253.0321
139.9434
62.9952
112.0122
43.9766
78.0134
90.0163
152.0467
160.0397
180.1079
206.9854
209.0331
255.0207
38.0467
52.0123
178.1139
190.0846
198.0354
219.0359
273.9826
256.0119
281.0025
O
N
O
Cl



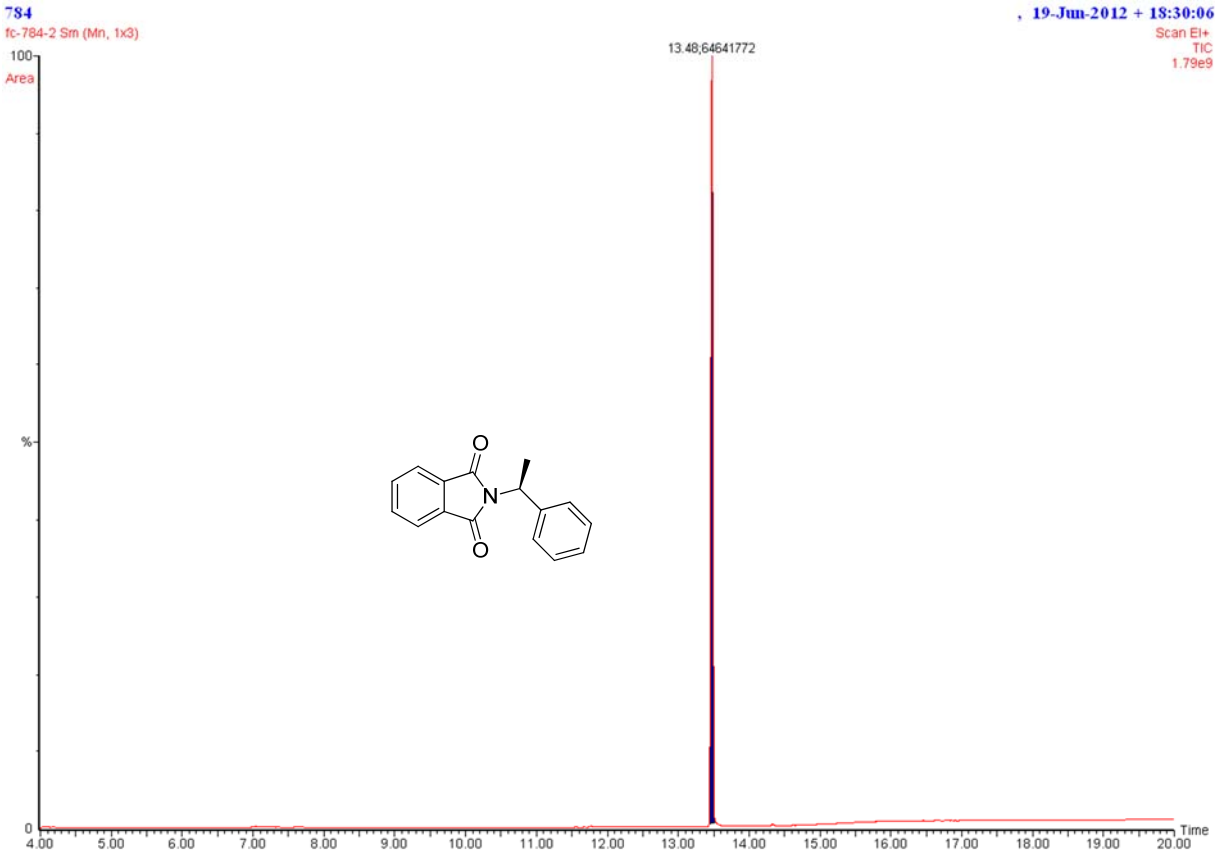
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a2**

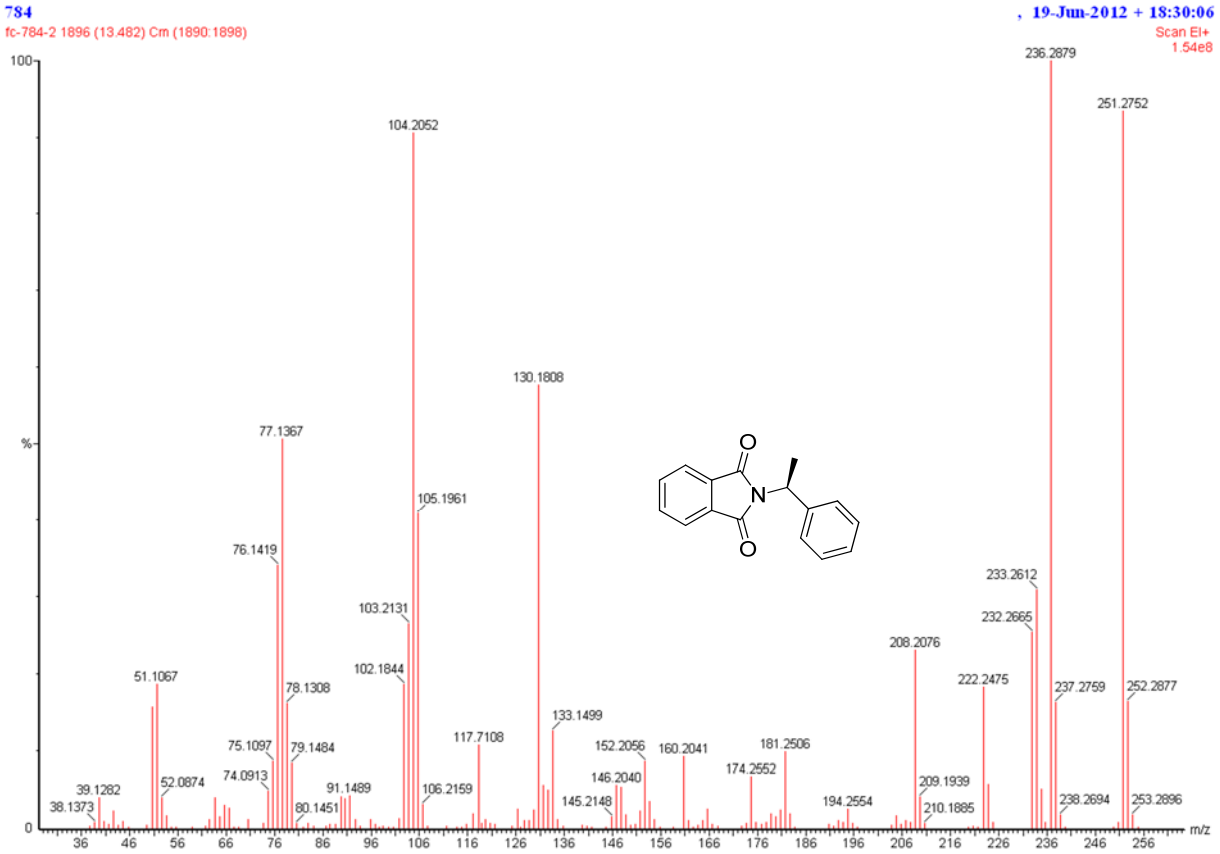

854
, 17-Sep-2012 + 18:33:41
fc-854- Sm (Mn, 1x3)
Scan EI+
TIC
4.54e8
14.68;18041228
100
Area
%
0
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
Time
O
N
O
OMe

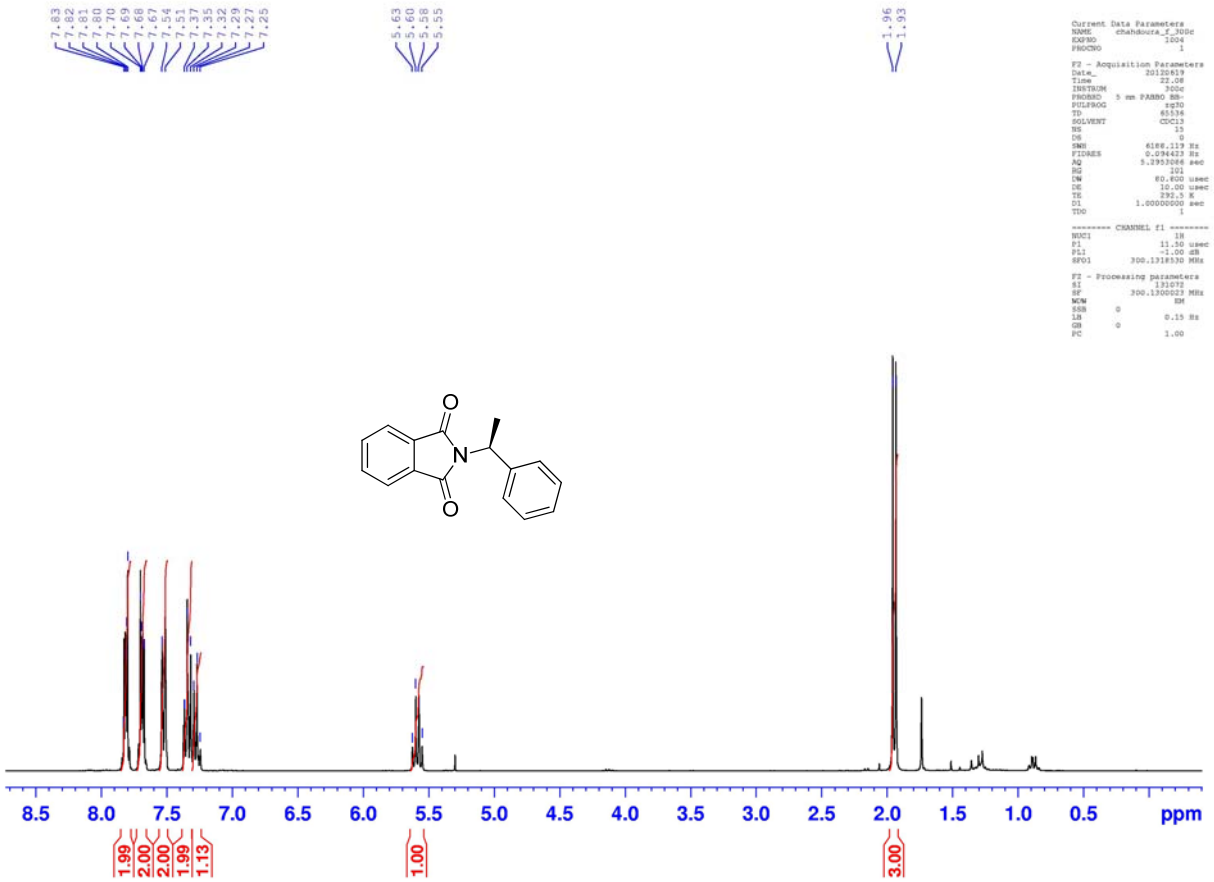

854
, 17-Sep-2012 + 18:33:41
fc-854- 2137 (14.688) Cm (2128:2141)
Scan EI+
3.49e7
100
%
0
267.1352
134.0589
77.0830
224.1160
76.0594
108.1286
121.1152
104.0943
78.0741
268.1228
51.0566
120.1324
180.1626
210.1016
236.1330
266.1024
65.0503
89.1092
135.1063
160.1024
167.1458
196.1096
252.1267
39.1040
38.0979
194.1209
211.0889
253.1269
269.1231
29 39 49 59 69 79 89 99 109 119 129 139 149 159 169 179 189 199 209 219 229 239 249 259 269 279 289
m/z
O
N
O
OMe

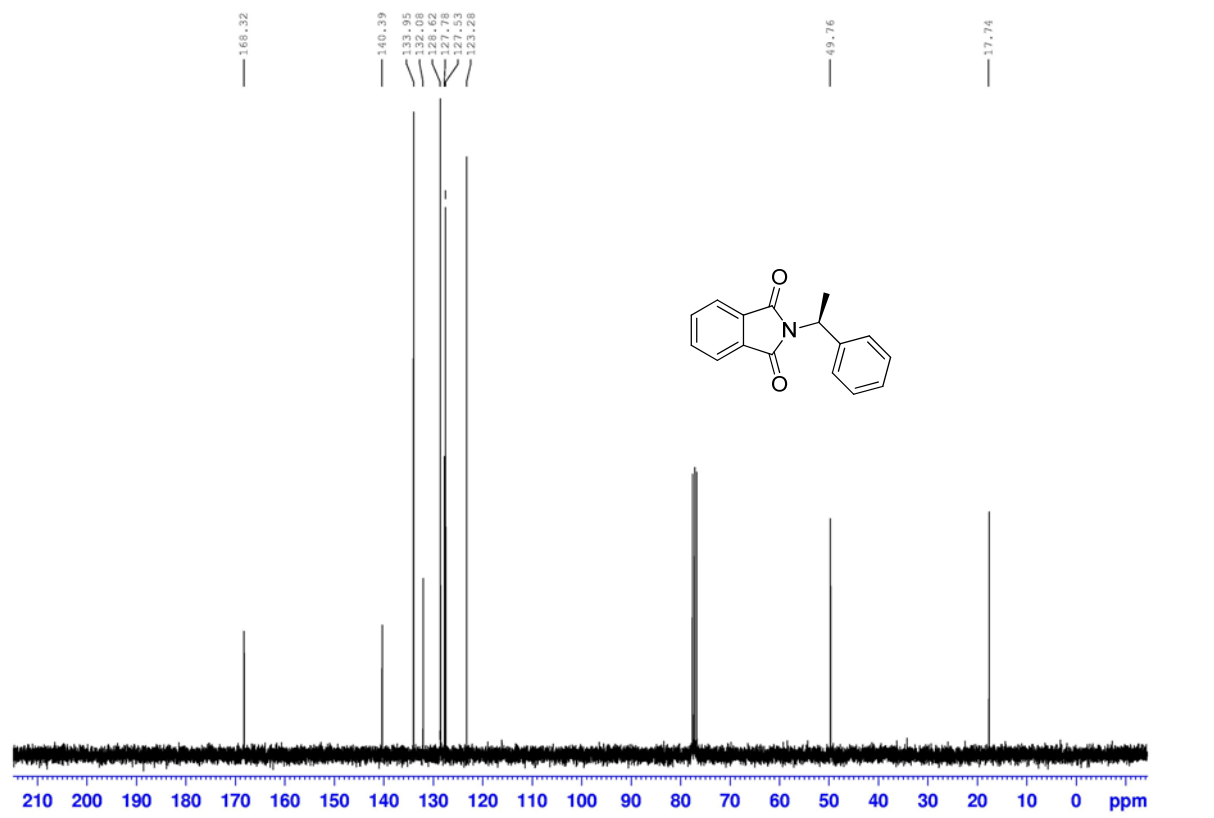


GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a3**


784
fc-784-2 Sm (Mn, 1x3)
, 19-Jun-2012 + 18:30:06
Scan EI+
TIC
1.79e9
13.48;64641772
100
Area
%
0
Time
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
O
N
O

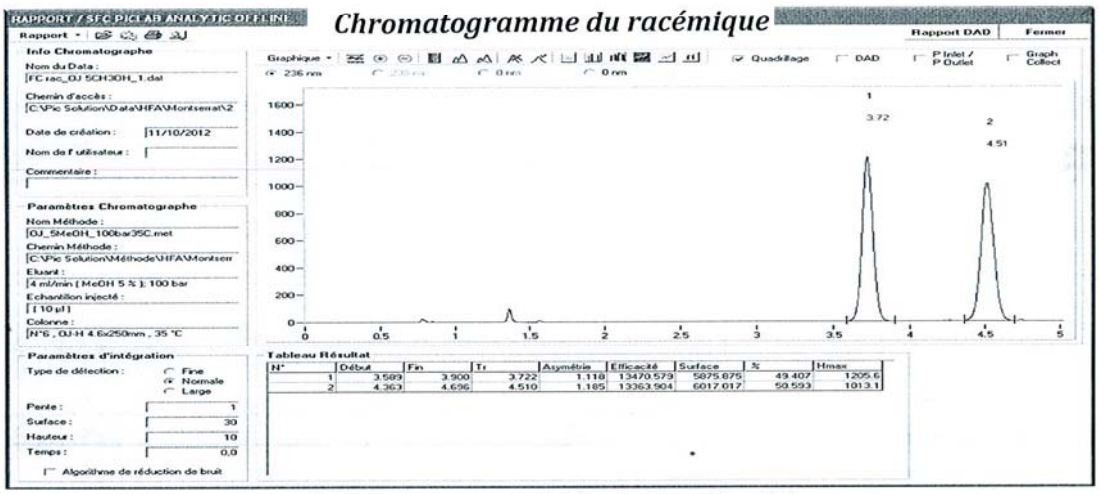

784
fc-784-2 1896 (13.482) Cm (1890:1898)
, 19-Jun-2012 + 18:30:06
Scan EI+
1.54e8
100
%
0
m/z
236.2879
251.2752
104.2052
130.1808
77.1367
105.1961
76.1419
103.2131
102.1844
233.2612
232.2665
208.2076
222.2475
51.1067
78.1308
237.2759
252.2877
133.1499
117.7108
75.1097
79.1484
152.2056
160.2041
181.2506
74.0913
146.2040
174.2552
52.0874
39.1282
91.1489
106.2159
145.2148
209.1939
194.2554
38.1373
80.1451
210.1885
238.2694
253.2896
36 46 56 66 76 86 96 106 116 126 136 146 156 166 176 186 196 206 216 226 236 246 256
O
N
O


7.83
7.82
7.81
7.80
7.70
7.69
7.68
7.67
7.54
7.51
7.37
7.35
7.32
7.29
7.27
7.25
5.63
5.60
5.58
5.55
1.96
1.93
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.99
2.00
2.00
1.99
1.13
1.00
3.00

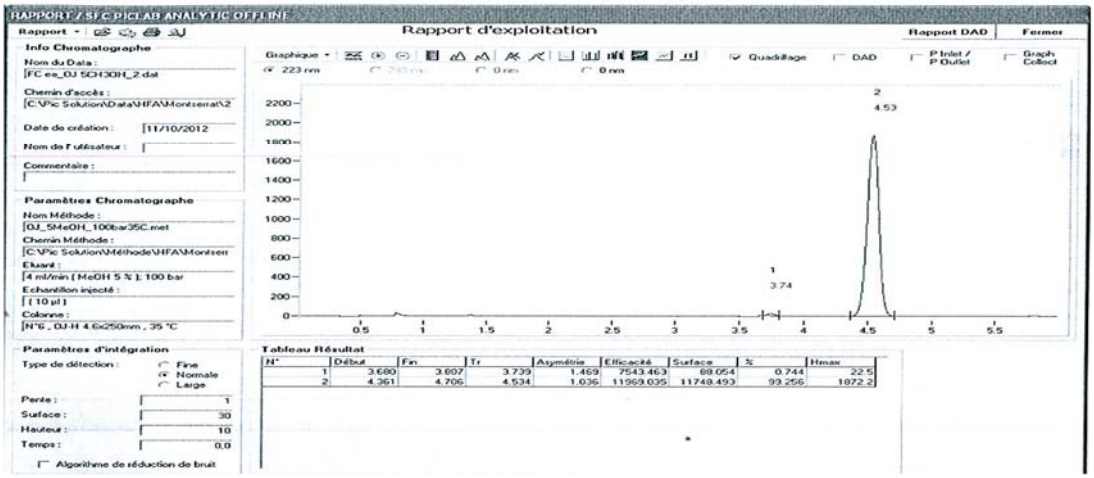

168.32
140.39
133.95
132.08
128.62
127.78
127.53
123.28
49.76
17.74
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

SFC analyses:

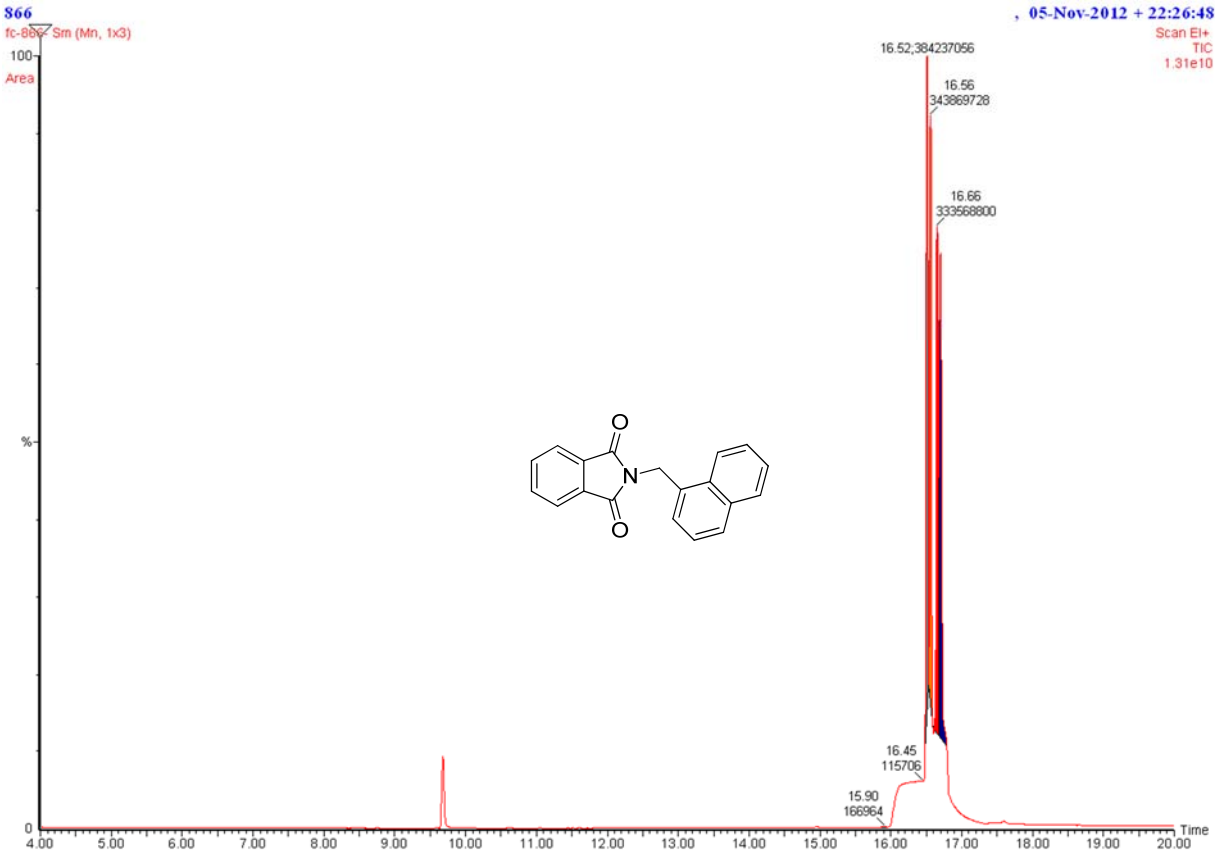
Colonne	Chiralpak OJ-H 5 µm (4.6 x 250) mm
Débit total CO2 + co-solvant (mL/min)	4
Co-solvant	CH3OH
% Co-solvant	5
Température (°C)	35
Pin (bar)	138
Pout (bar)	100
λ (nm)	236 (λmax = 223, 285)
t_R (min)	3.72 et 4.51
Durée du run (min)	6

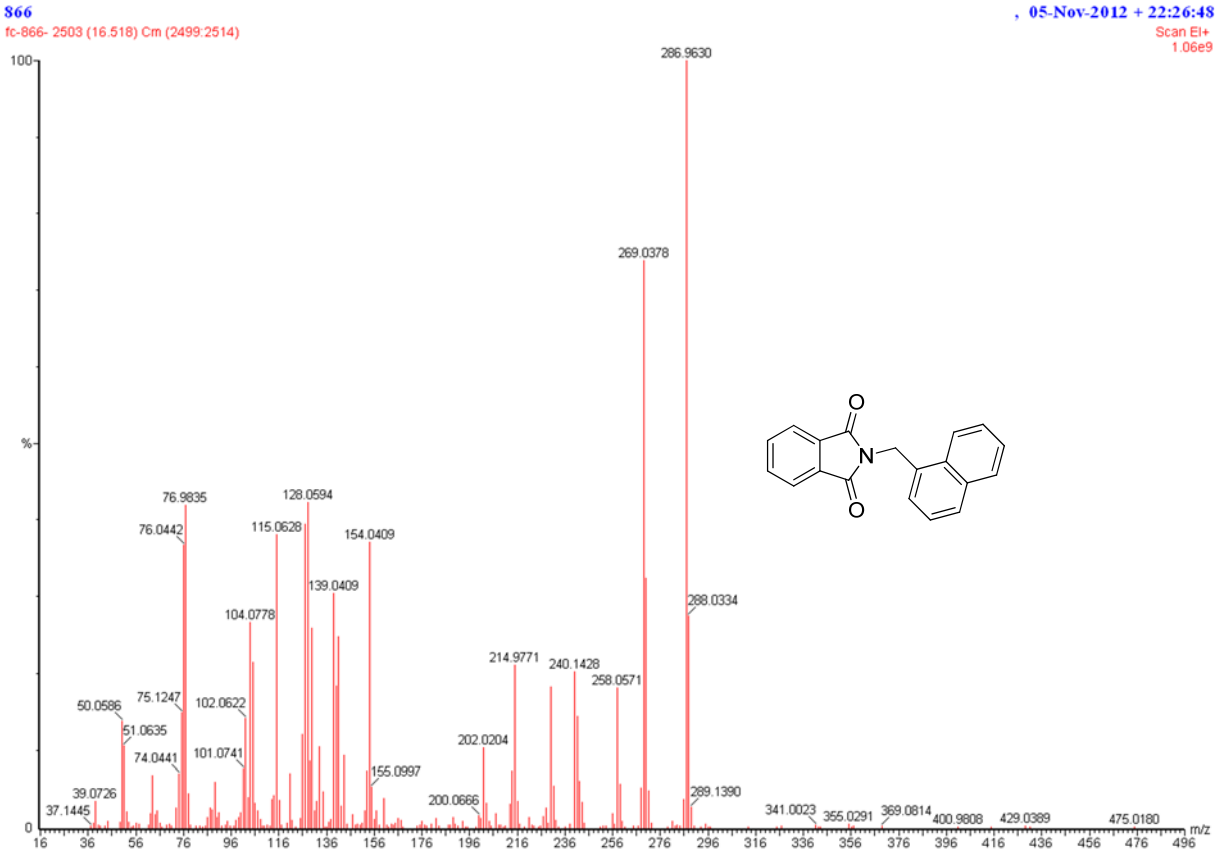


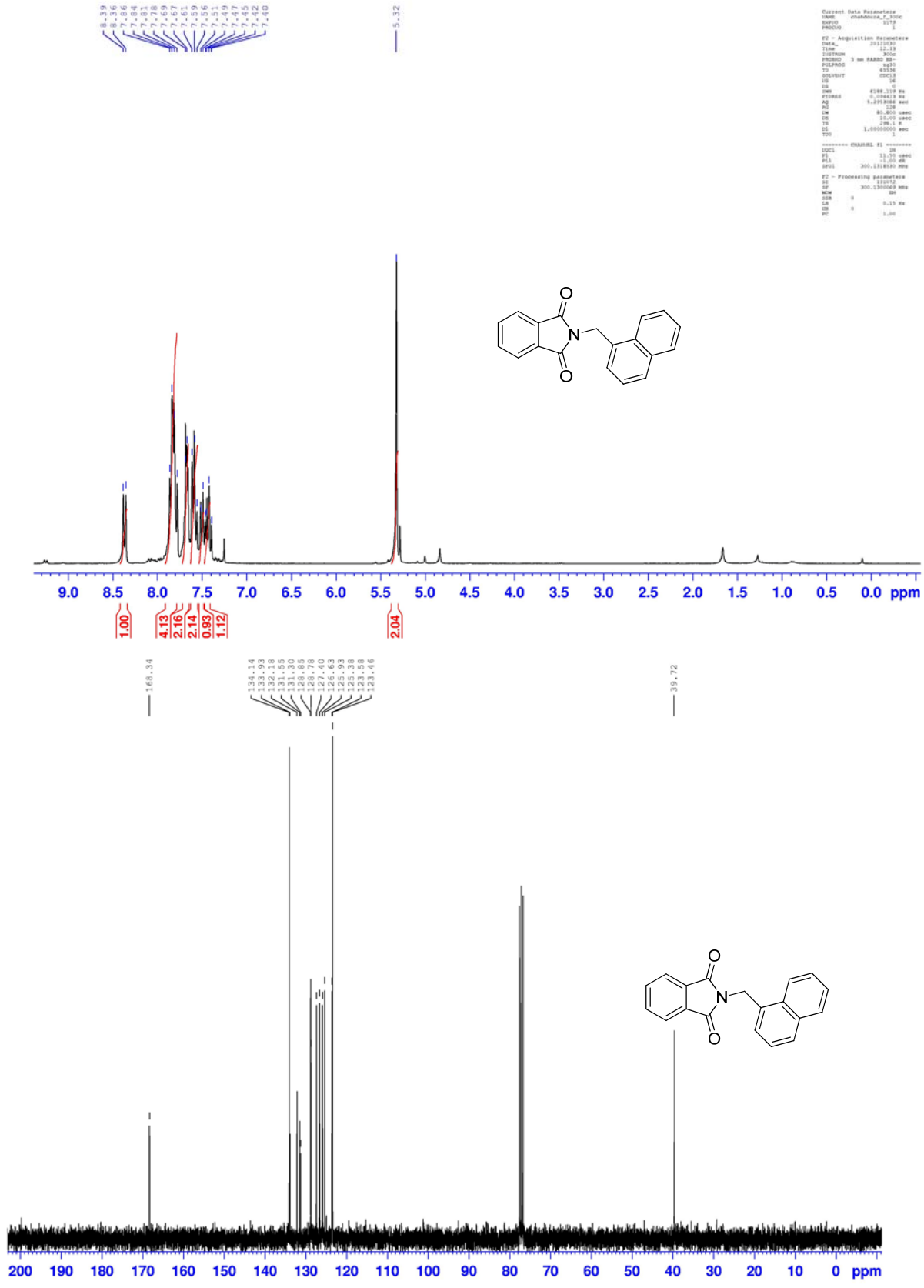
Chromatogramme de l'excès énantiomérique



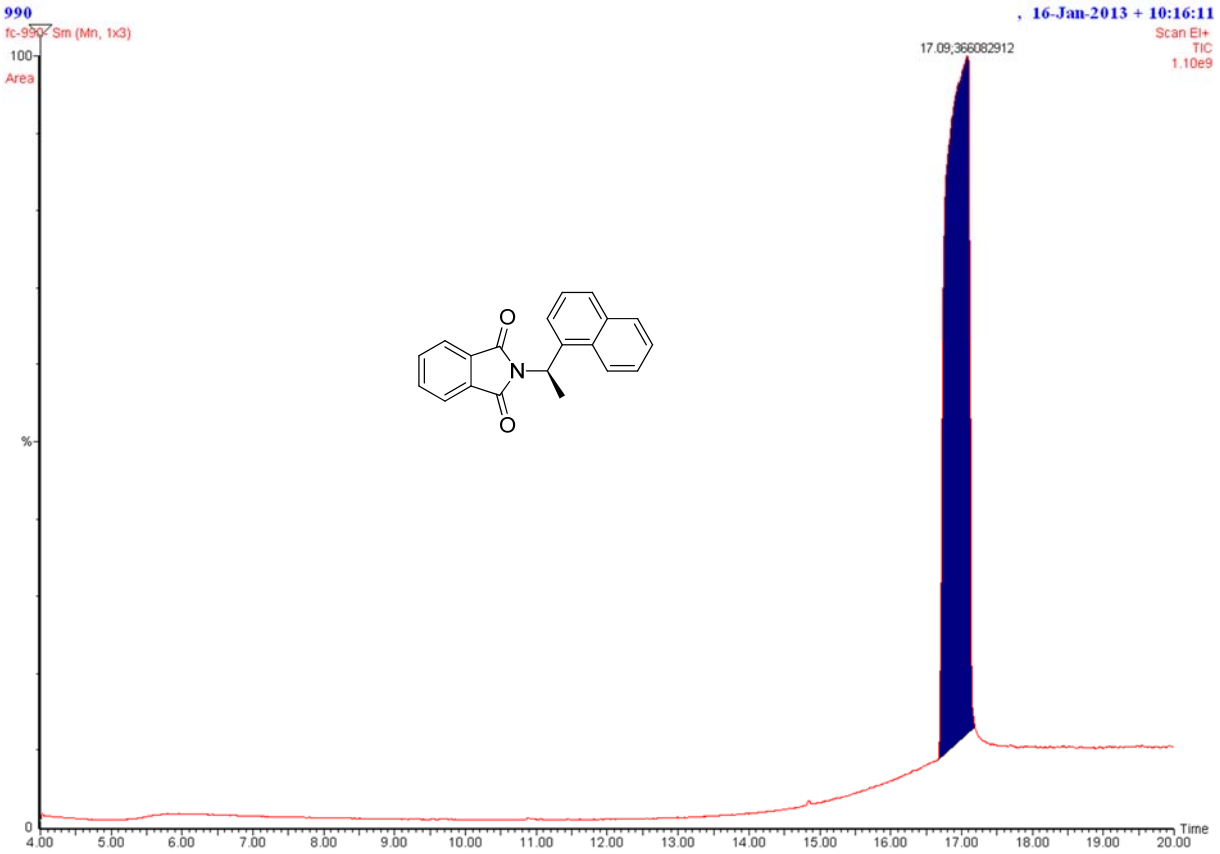
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) spectra in $CDCl_3$, and SFC analysis (bottom) for **a4**

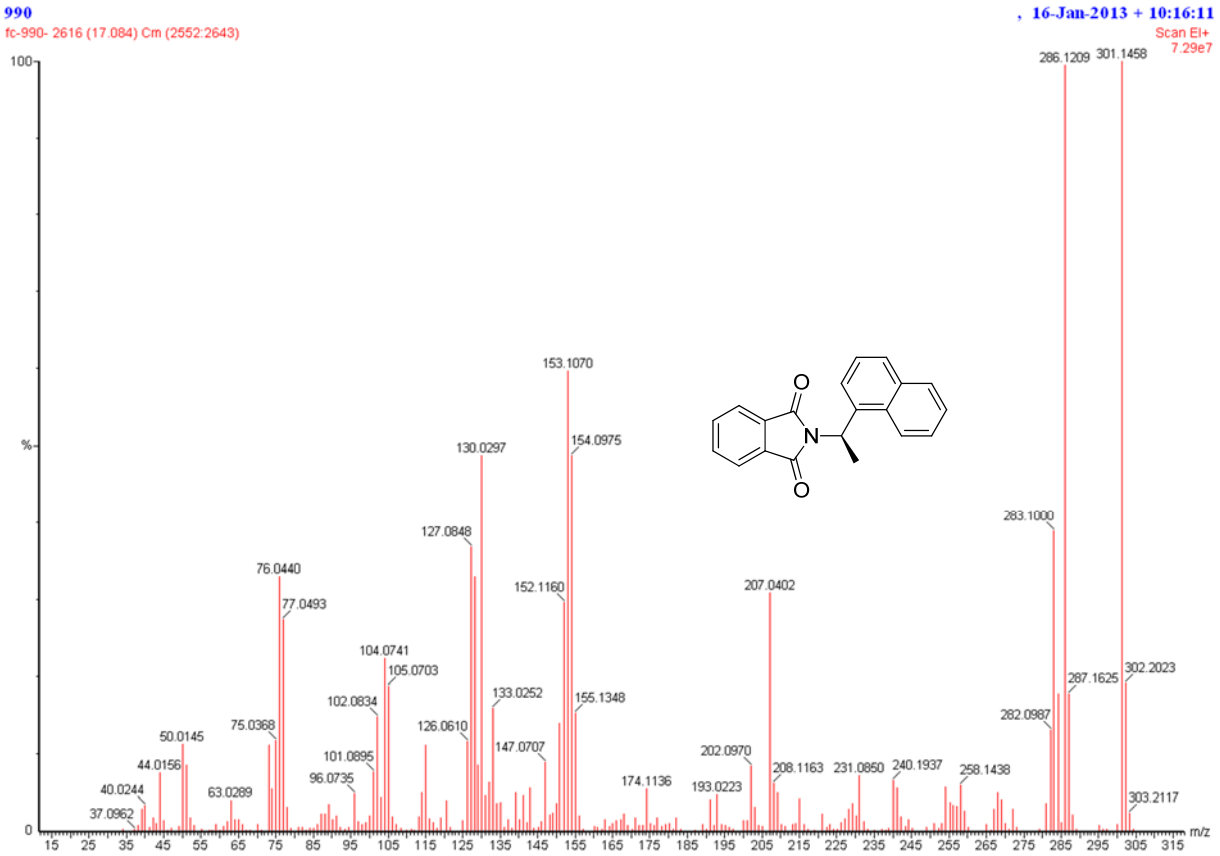

866
, 05-Nov-2012 + 22:26:48
Scan EI+
TIC
1.31e10
Area
%
16.52;384237056
16.56
343869728
16.66
333568800
16.45
115706
15.90
166964
Time

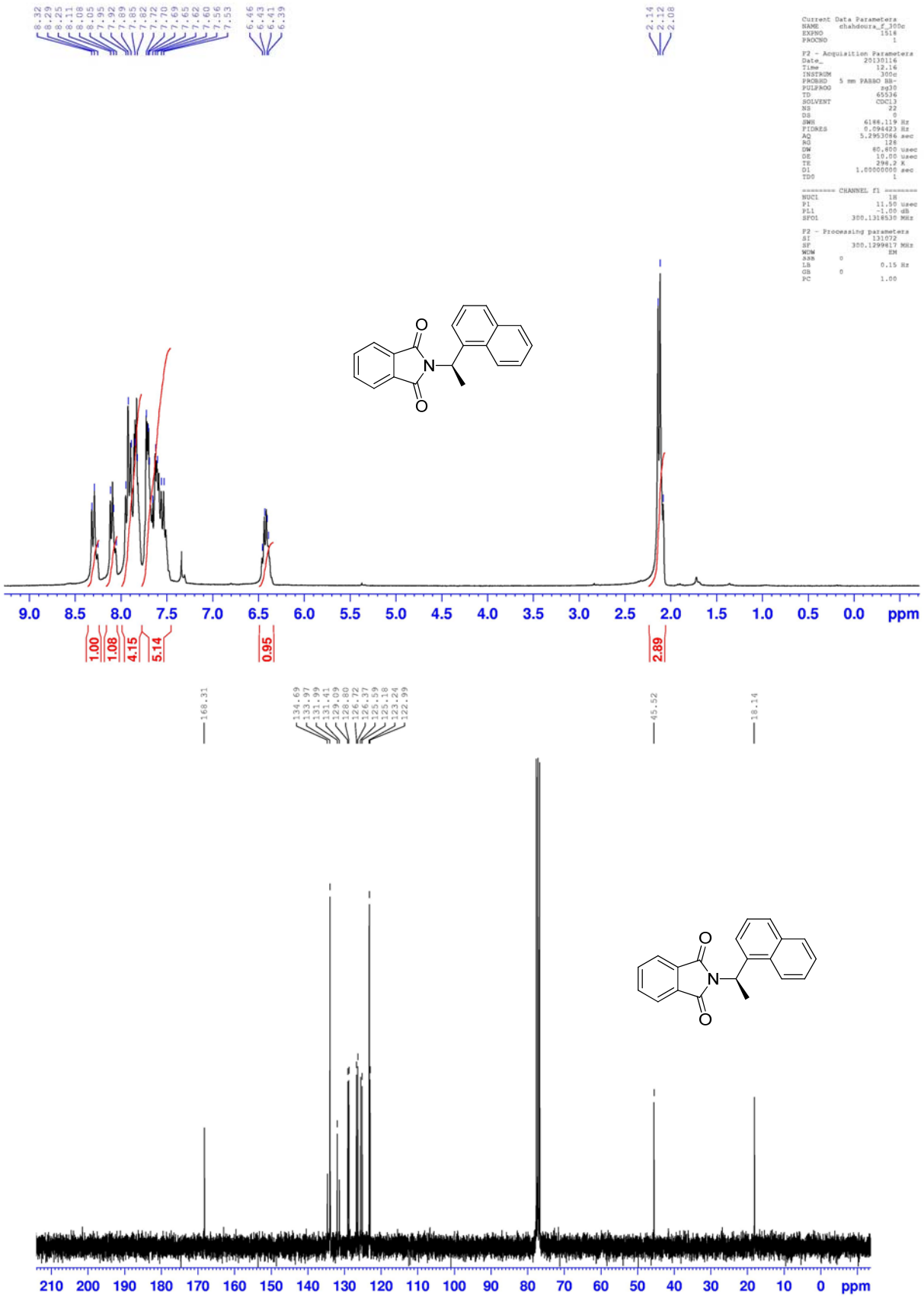

866
, 05-Nov-2012 + 22:26:48
fc-866- 2503 (16.518) Cm (2499:2514)
Scan EI+
1.06e9
%
286.9630
269.0378
288.0334
76.9835
128.0594
76.0442
115.0628
154.0409
139.0409
104.0778
214.9771
240.1428
258.0571
50.0586
75.1247
102.0622
51.0635
202.0204
74.0441
101.0741
155.0997
39.0726
37.1445
200.0666
289.1390
341.0023
355.0291
369.0814
400.9808
429.0389
475.0180
m/z



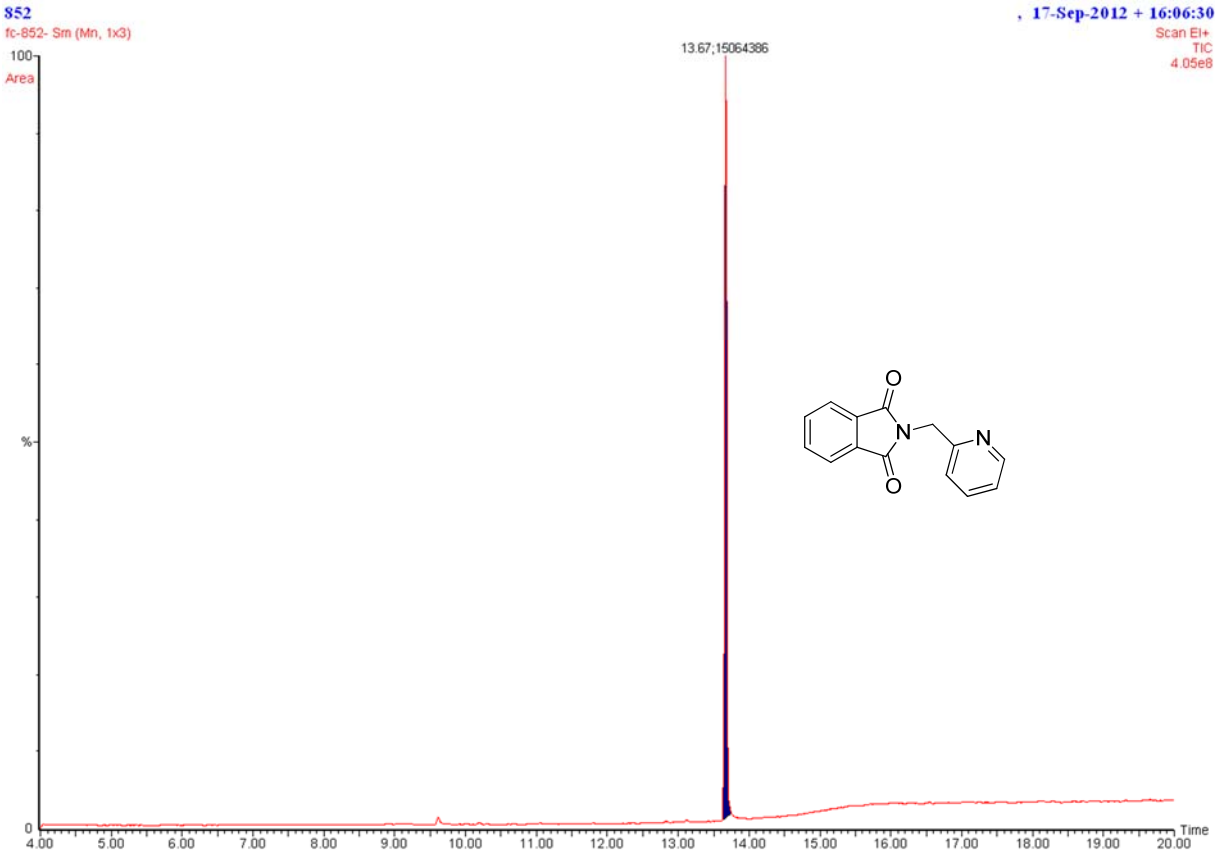
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a5**

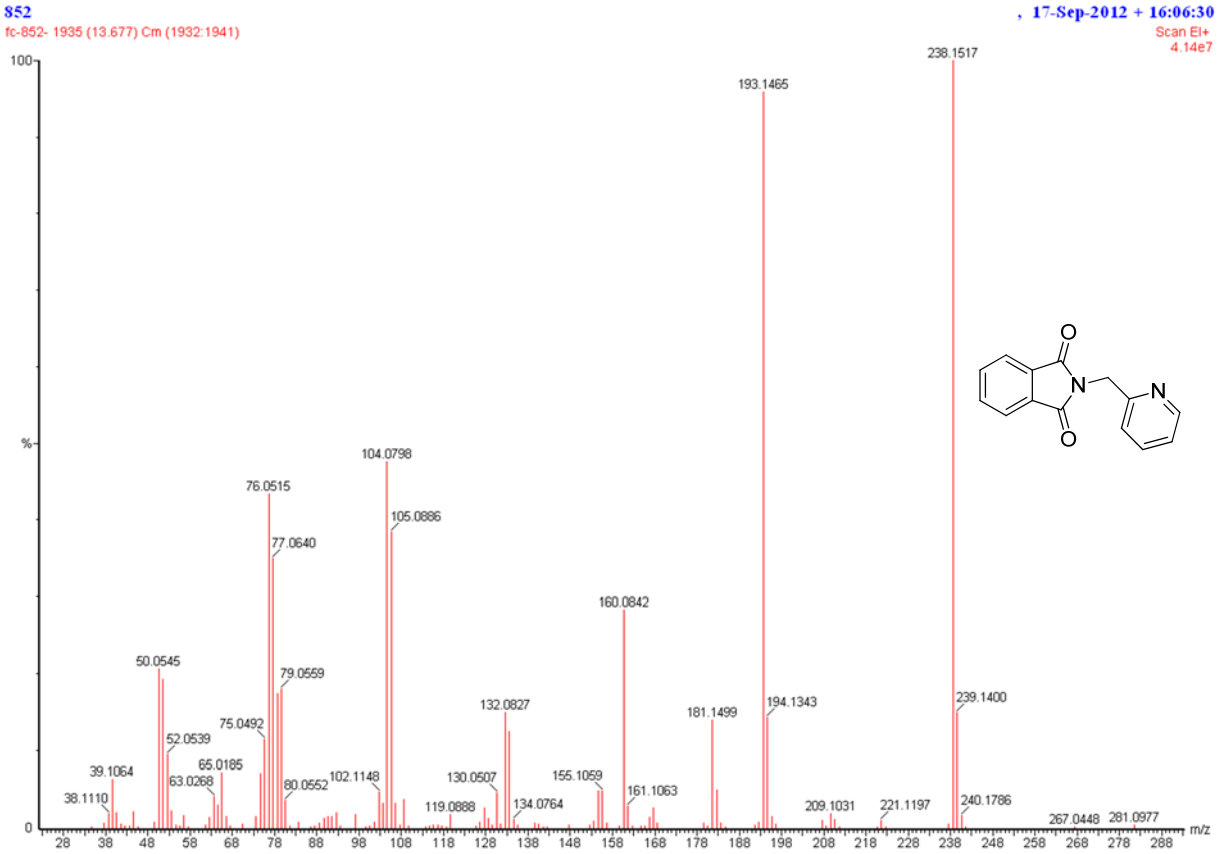

990
fc-990 Sm (Mn, 1x3)
Area
100
%
0
, 16-Jan-2013 + 10:16:11
Scan EI+
TIC
1.10e9
17.09;366082912
O
N
O
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
Time

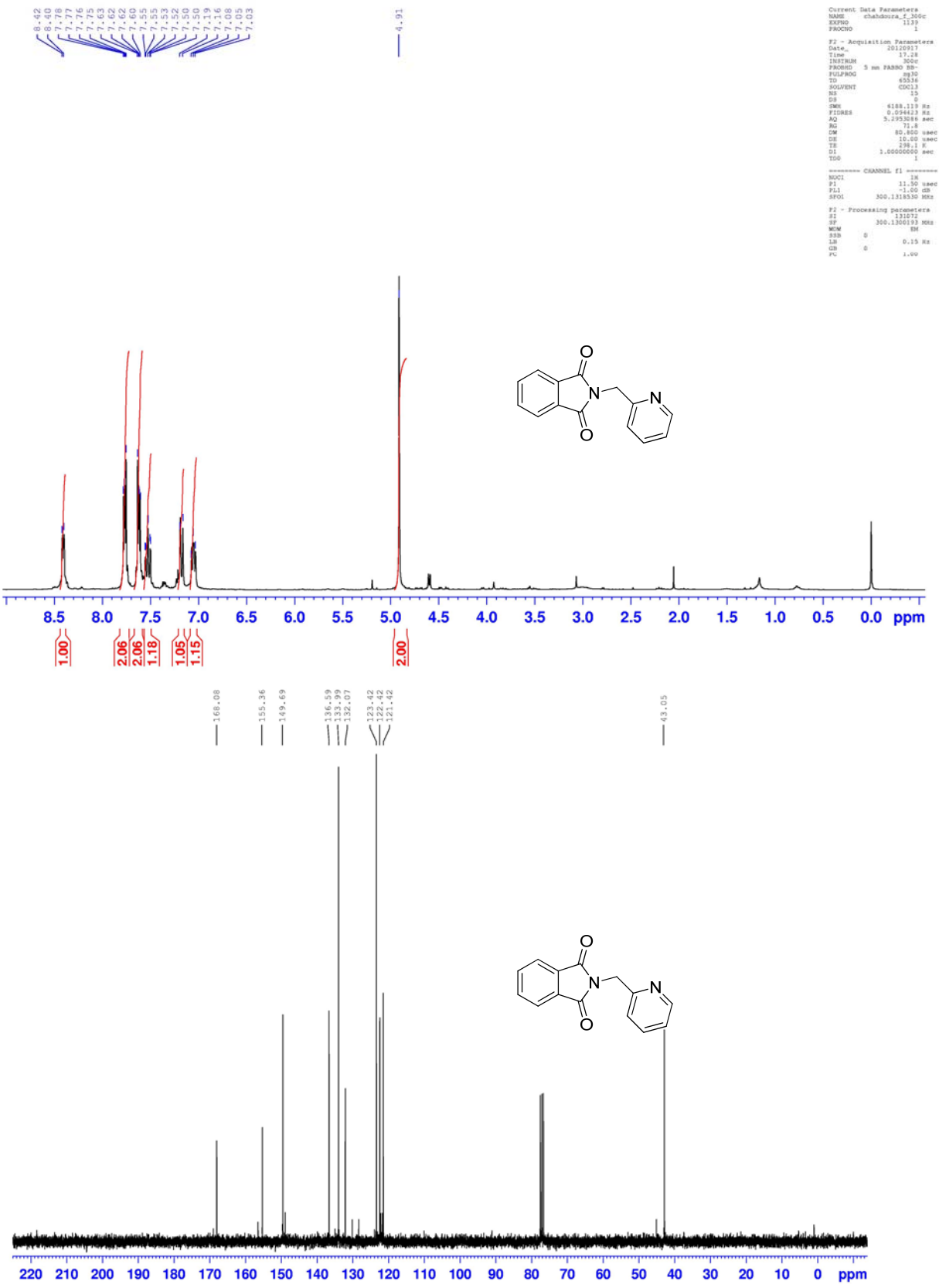

990
fc-990- 2616 (17.084) Cm (2552:2643)
, 16-Jan-2013 + 10:16:11
Scan EI+
7.29e7
100
%
0
O
N
O
286.1209
301.1458
283.1000
153.1070
154.0975
130.0297
127.0848
76.0440
77.0493
152.1160
207.0402
104.0741
105.0703
133.0252
155.1348
102.0834
126.0610
75.0368
50.0145
147.0707
202.0970
287.1625
302.2023
282.0987
44.0156
101.0895
96.0735
208.1163
231.0850
240.1937
258.1438
40.0244
63.0289
174.1136
193.0223
303.2117
37.0962
15 25 35 45 55 65 75 85 95 105 115 125 135 145 155 165 175 185 195 205 215 225 235 245 255 265 275 285 295 305 315
m/z



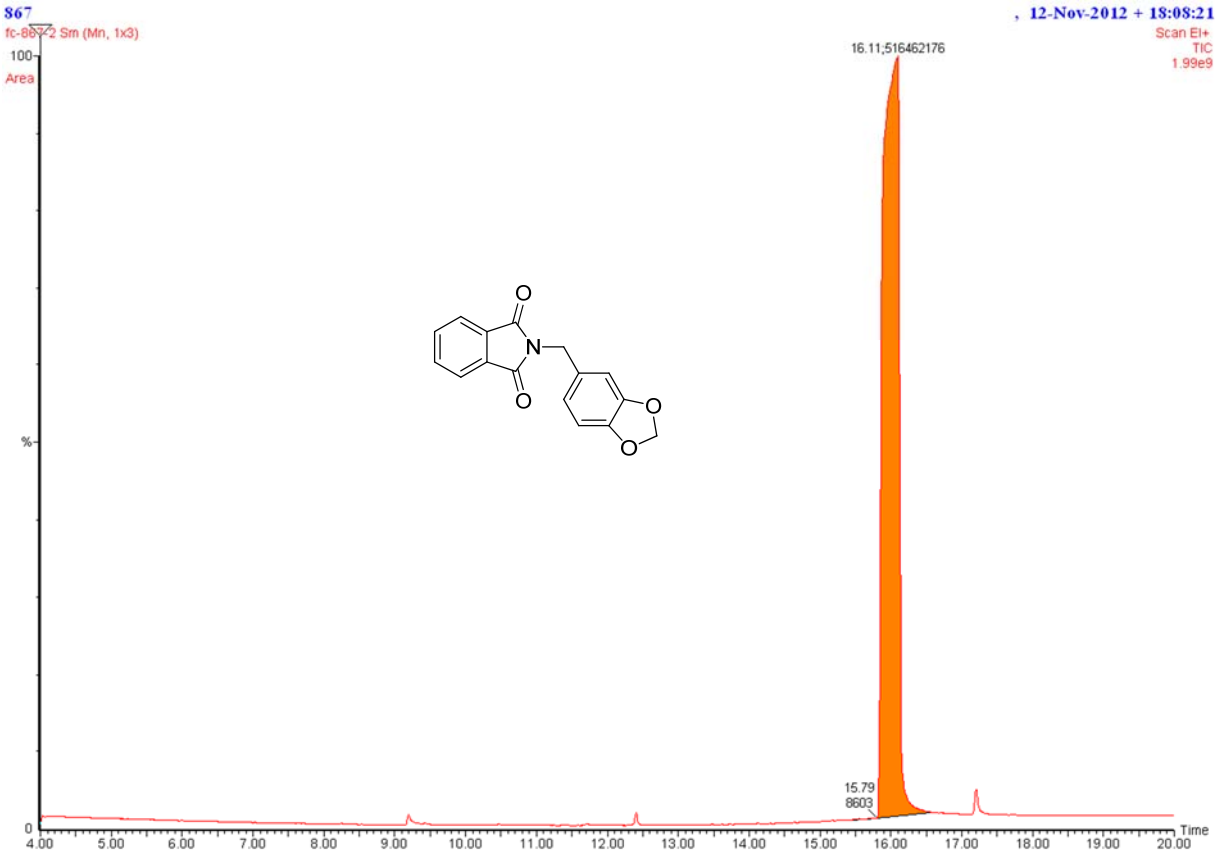
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a6**

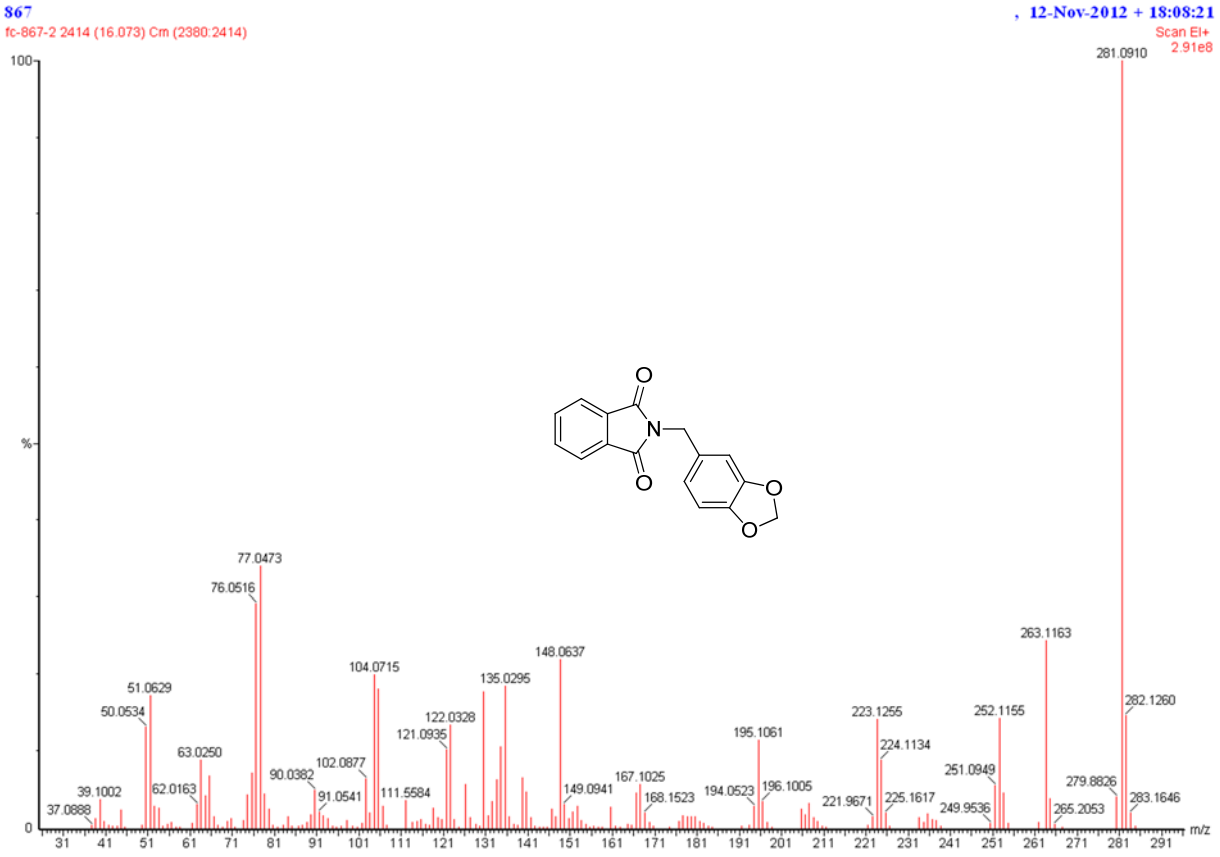

852
fc-852- Sm (Mn, 1x3)
, 17-Sep-2012 + 16:06:30
Scan EI+
TIC
4.05e8
Area
13.67;15064386
%
Time

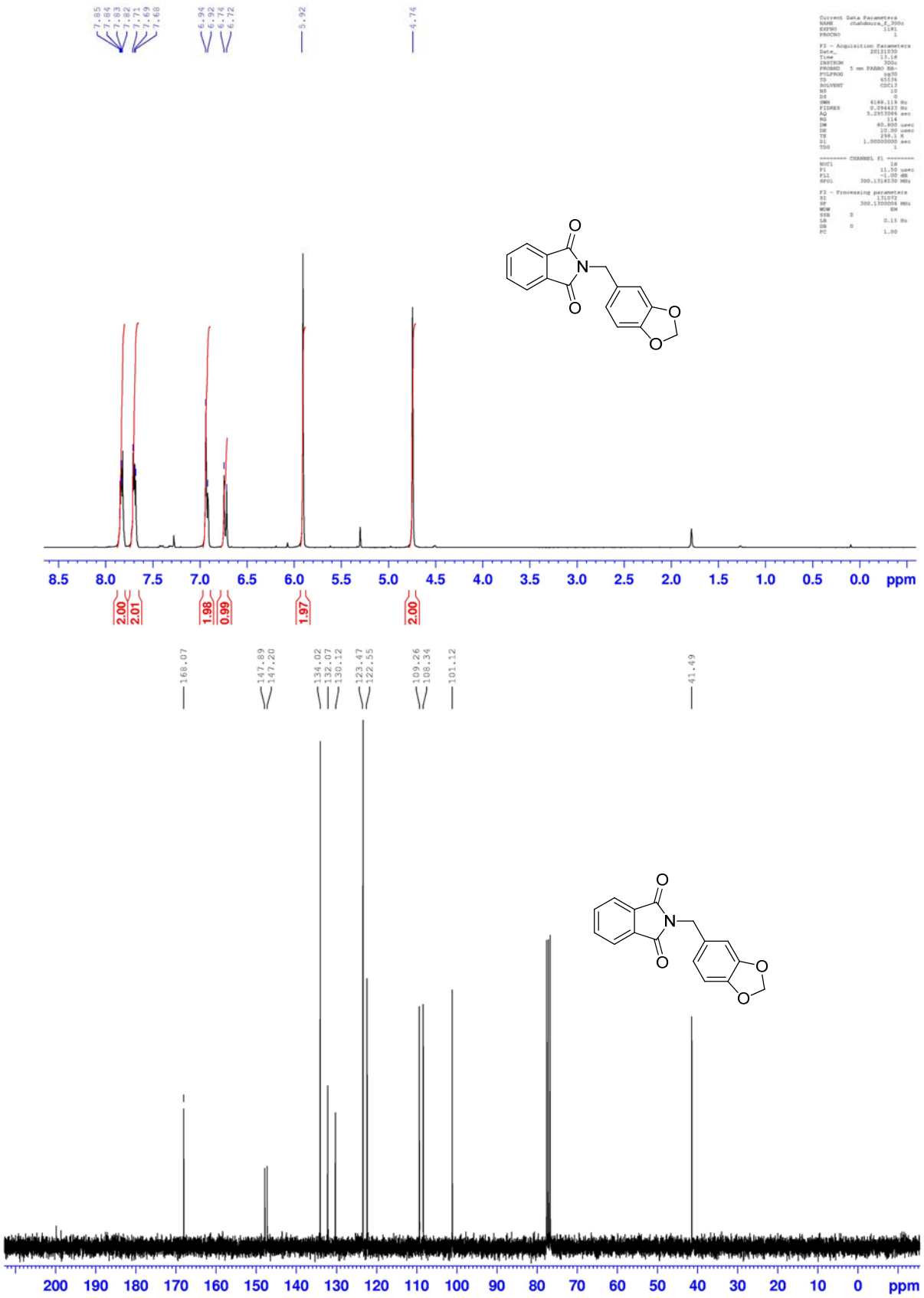

852
fc-852- 1935 (13.677) Cm (1932:1941)
, 17-Sep-2012 + 16:06:30
Scan EI+
4.14e7
%
238.1517
193.1465
104.0798
76.0515
105.0886
77.0640
160.0842
50.0545
79.0559
132.0827
181.1499
194.1343
239.1400
75.0492
52.0539
65.0185
39.1064
63.0268
102.1148
80.0552
130.0507
155.1059
161.1063
38.1110
119.0888
134.0764
209.1031
221.1197
240.1786
267.0448
281.0977
m/z



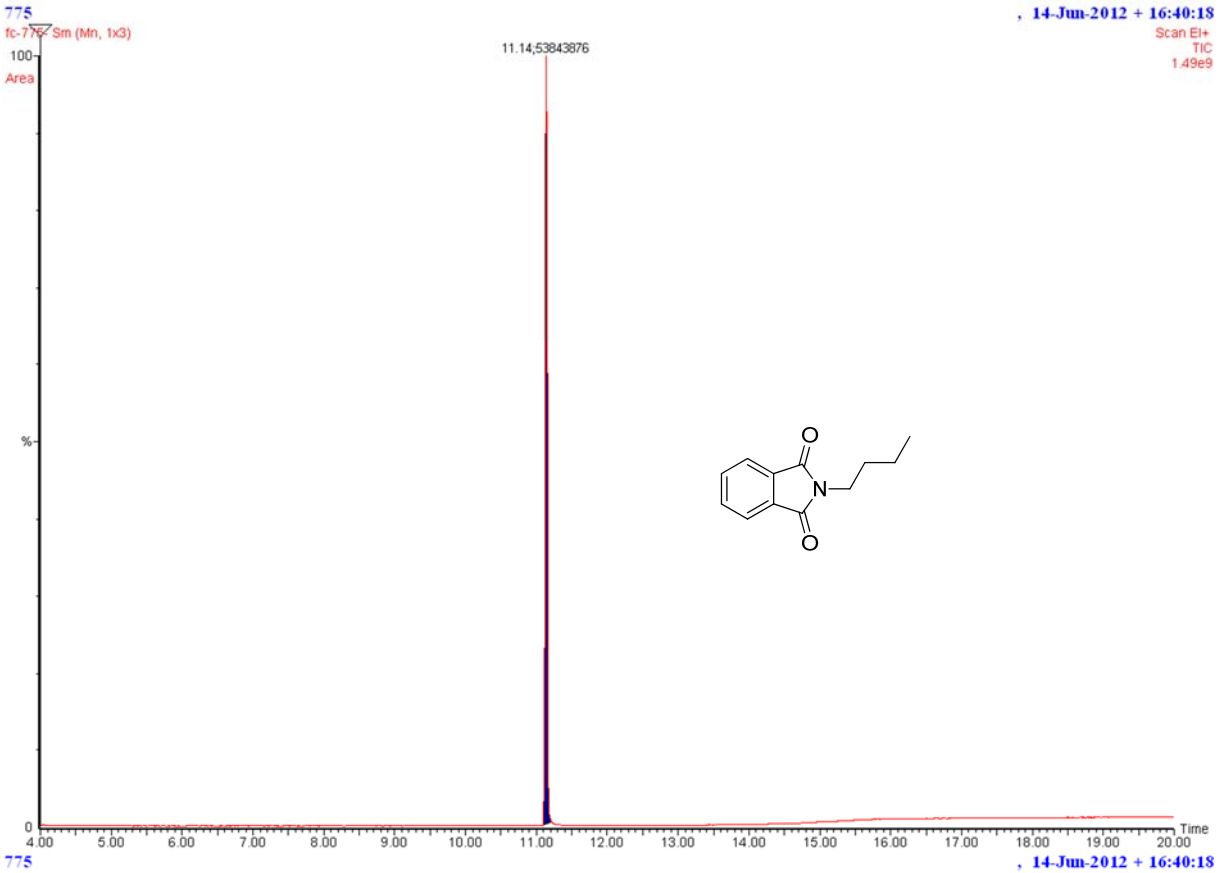
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a7**

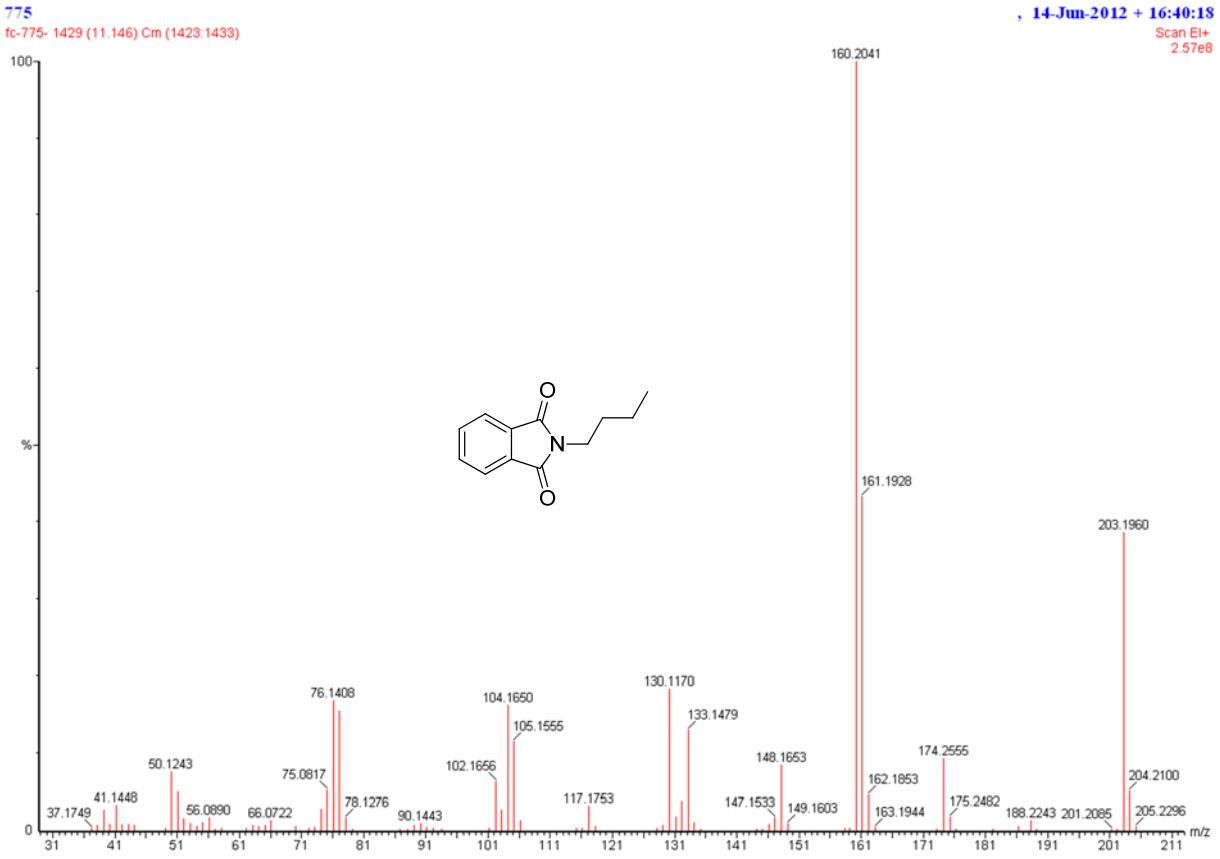

867
, 12-Nov-2012 + 18:08:21
Scan EI+
TIC
1.99e9
Area
100
%
0
16.11;516462176
15.79
8603
Time
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
O
N
O
O
O

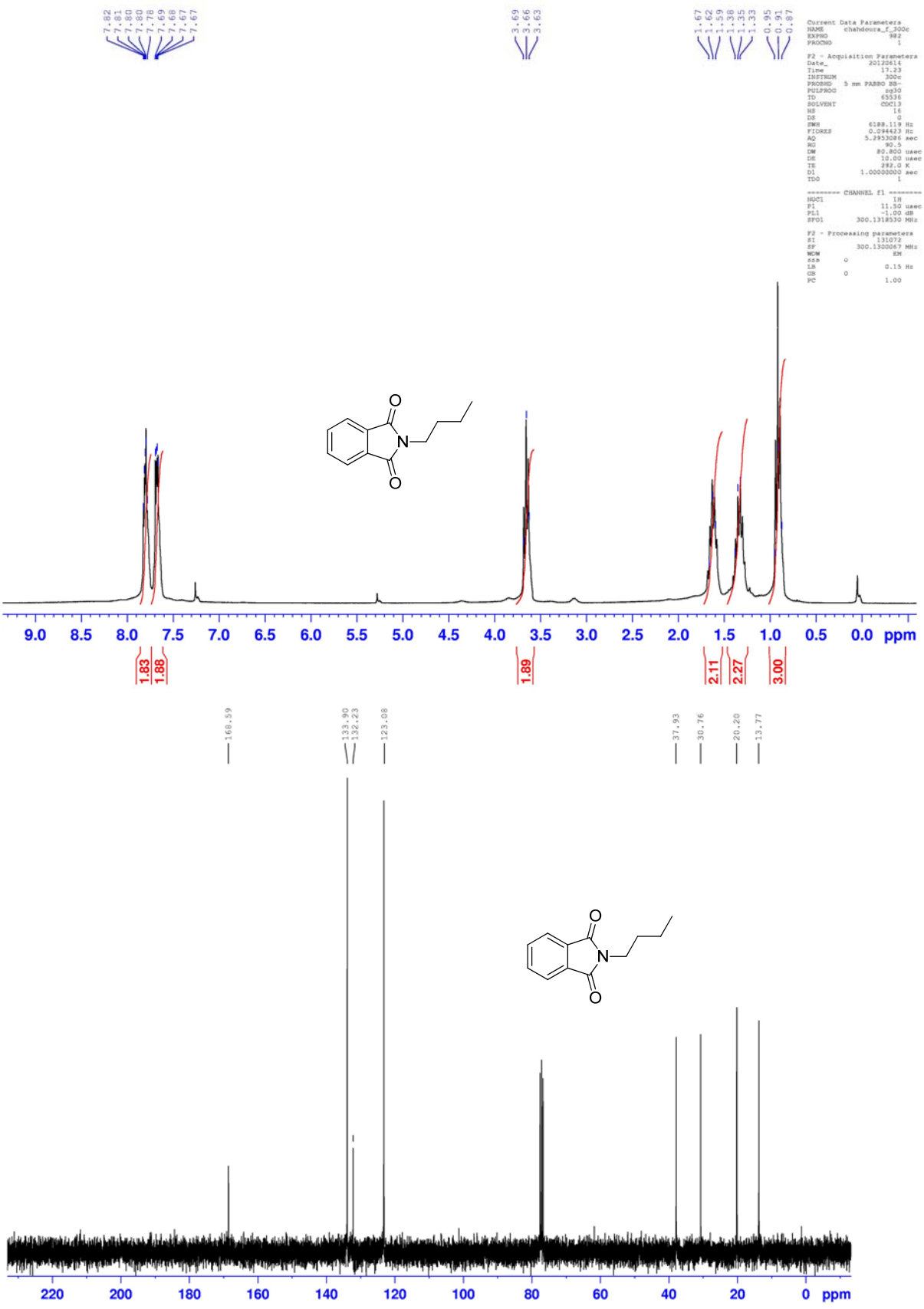

867
, 12-Nov-2012 + 18:08:21
fc-867-2 2414 (16.073) Cm (2380:2414)
Scan EI+
2.91e8
100
%
0
281.0910
77.0473
76.0516
148.0637
104.0715
135.0295
51.0629
50.0534
122.0328
121.0935
263.1163
223.1255
252.1155
282.1260
195.1061
224.1134
63.0250
102.0877
90.0382
251.0949
279.8826
39.1002
62.0163
91.0541
111.5584
149.0941
167.1025
194.0523
196.1005
221.9671
225.1617
283.1646
37.0888
168.1523
249.9536
265.2053
m/z
31
41
51
61
71
81
91
101
111
121
131
141
151
161
171
181
191
201
211
221
231
241
251
261
271
281
291
O
N
O
O
O



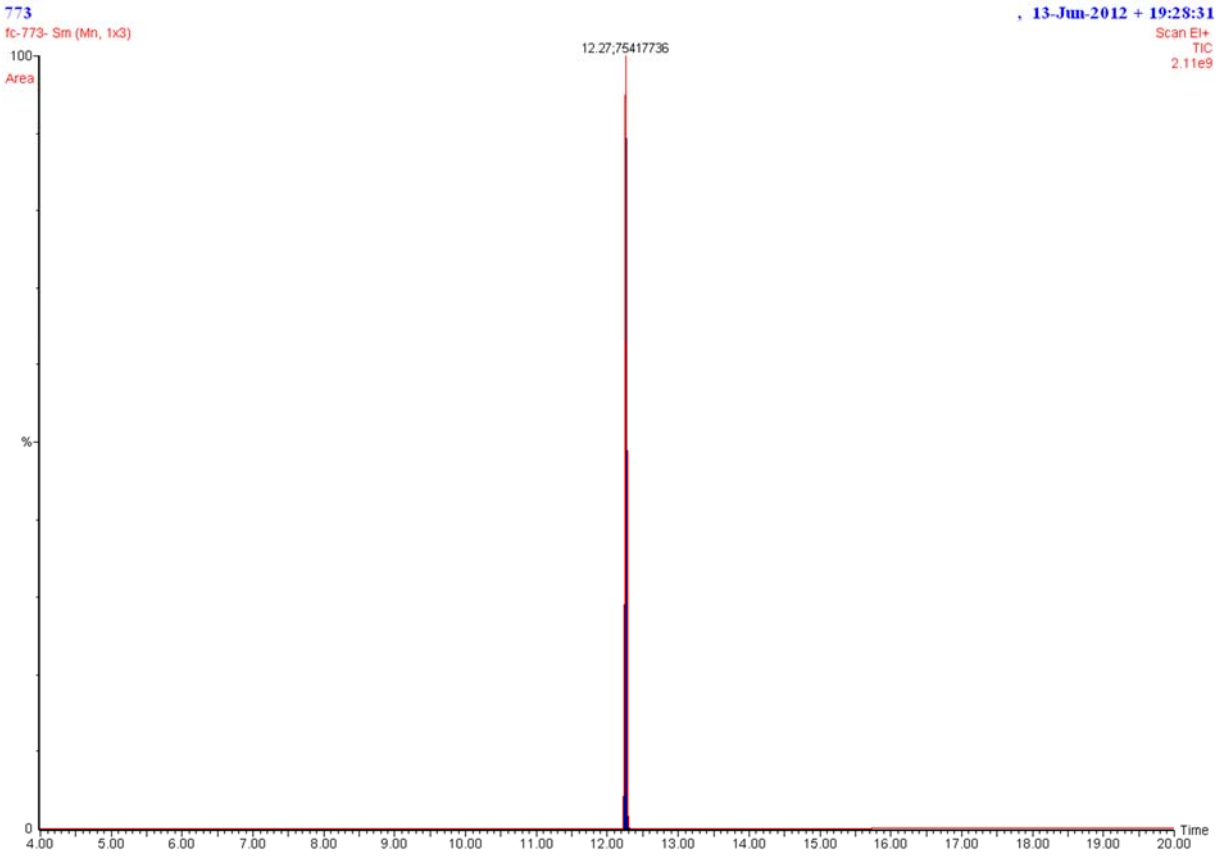
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a8**

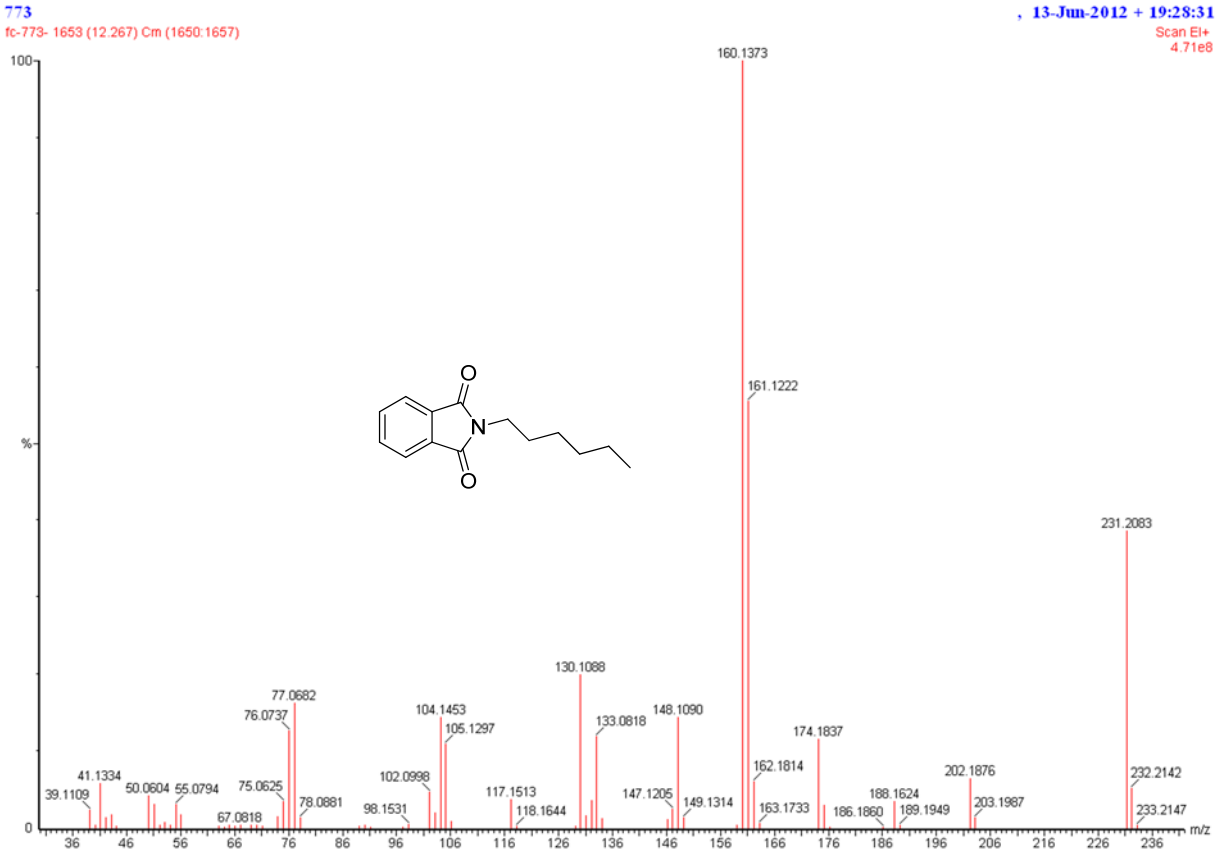

775
fc-775 Sm (Mn, 1x3)
, 14-Jun-2012 + 16:40:18
Scan EI+
TIC
1.49e9
Area
100
%
0
11.14;53843876
O
N
O
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

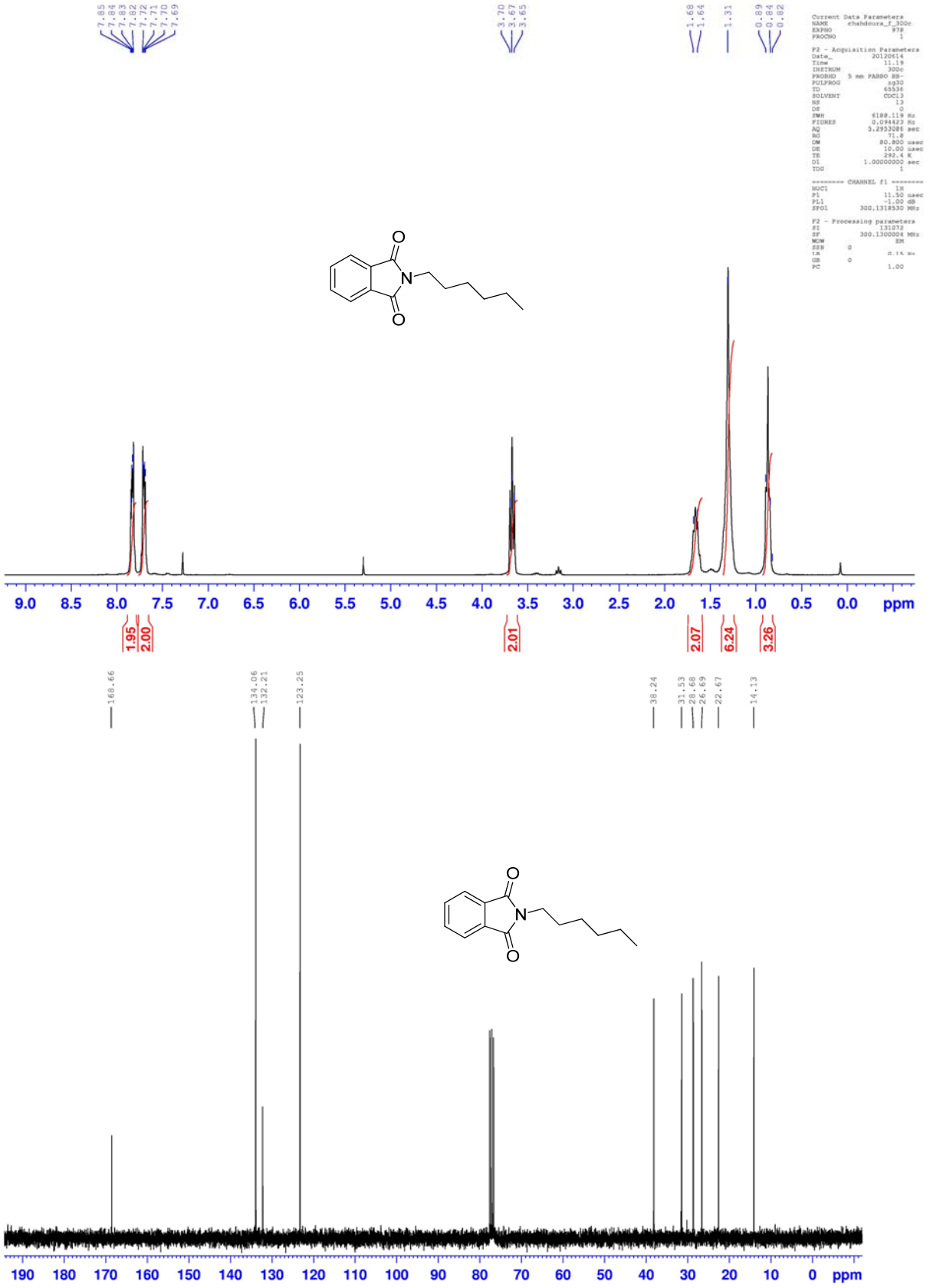

775
fc-775- 1429 (11.146) Cm (1423:1433)
, 14-Jun-2012 + 16:40:18
Scan EI+
2.57e8
100
%
0
O
N
O
160.2041
161.1928
203.1960
130.1170
76.1408
104.1650
105.1555
133.1479
148.1653
174.2555
50.1243
75.0817
102.1656
162.1853
204.2100
41.1448
78.1276
117.1753
147.1533
149.1603
175.2482
37.1749
56.0890
66.0722
90.1443
163.1944
188.2243
201.2085
205.2296
31
41
51
61
71
81
91
101
111
121
131
141
151
161
171
181
191
201
211
m/z



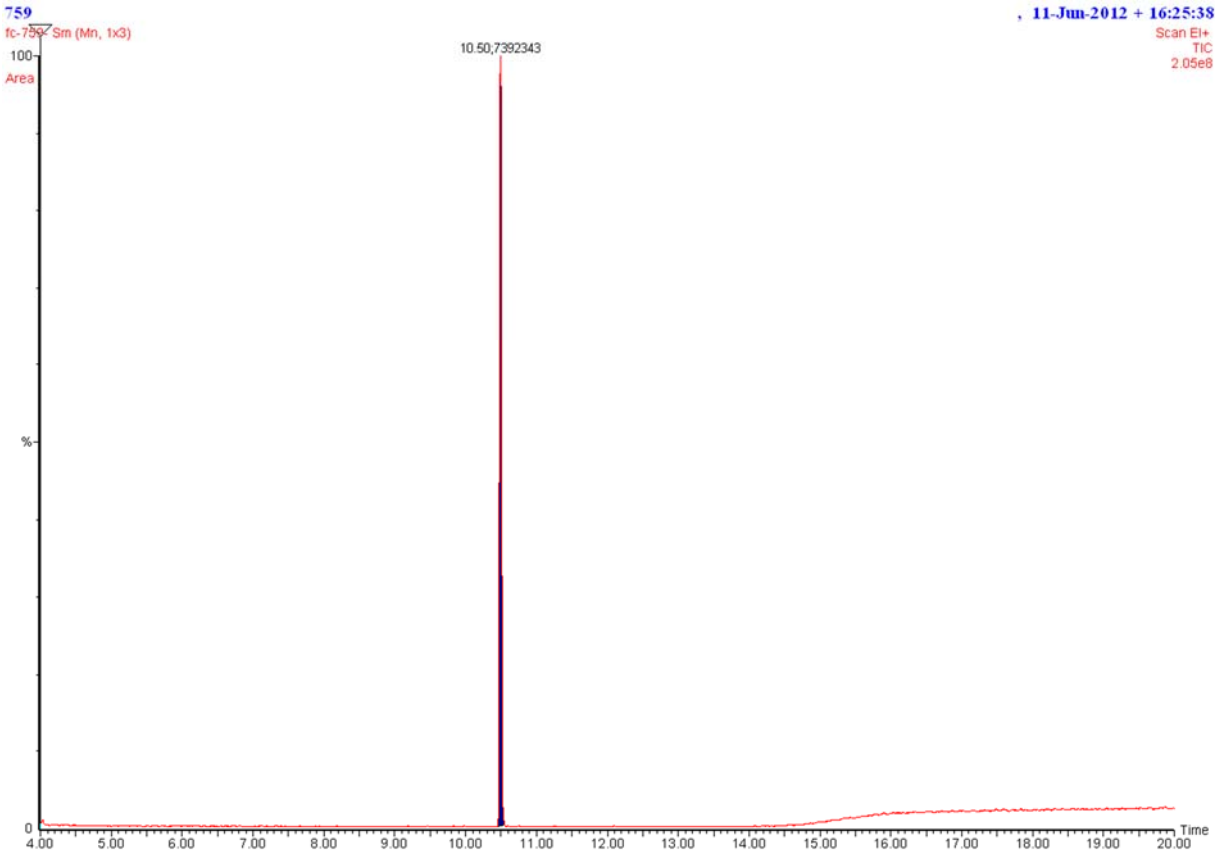
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a9**

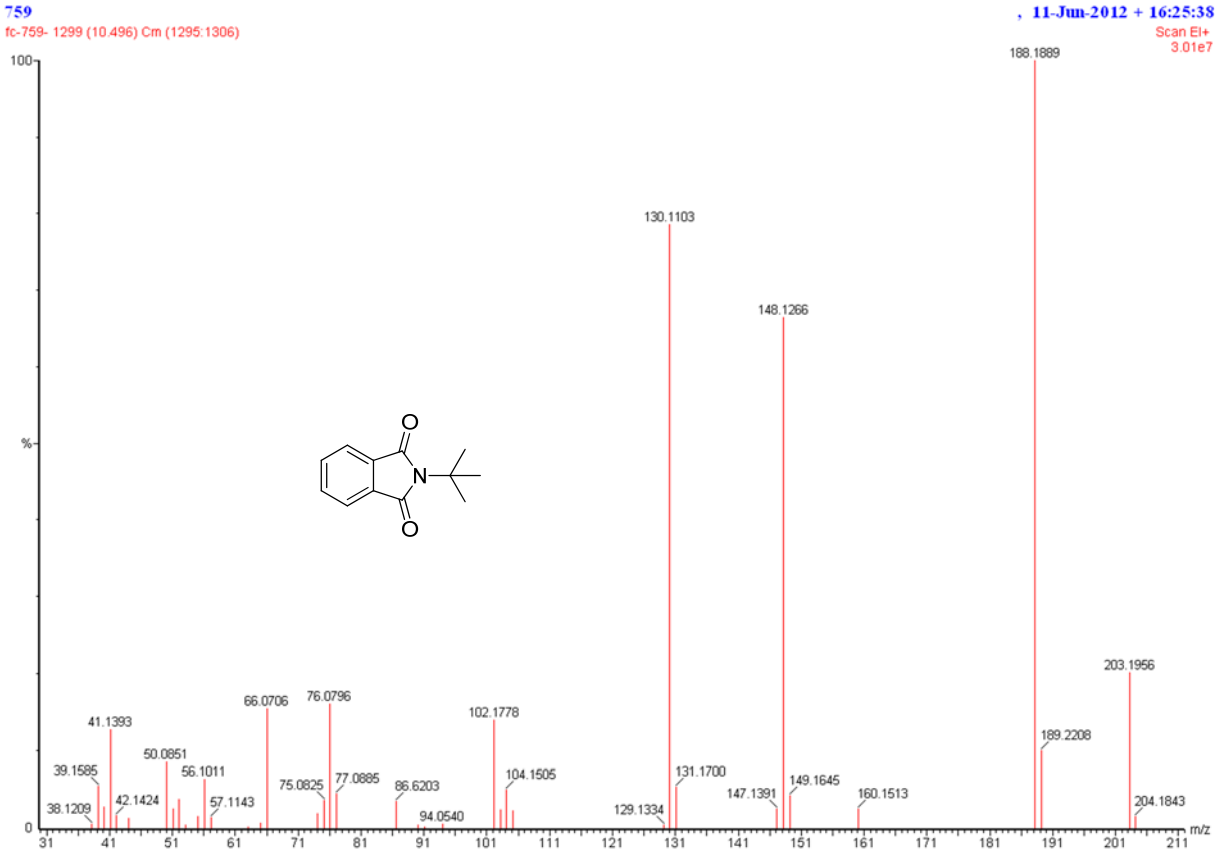

773
fc-773- Sm (Mn, 1x3)
, 13-Jun-2012 + 19:28:31
Scan EI+
TIC
2.11e9
Area
12.27;75417736
%
Time

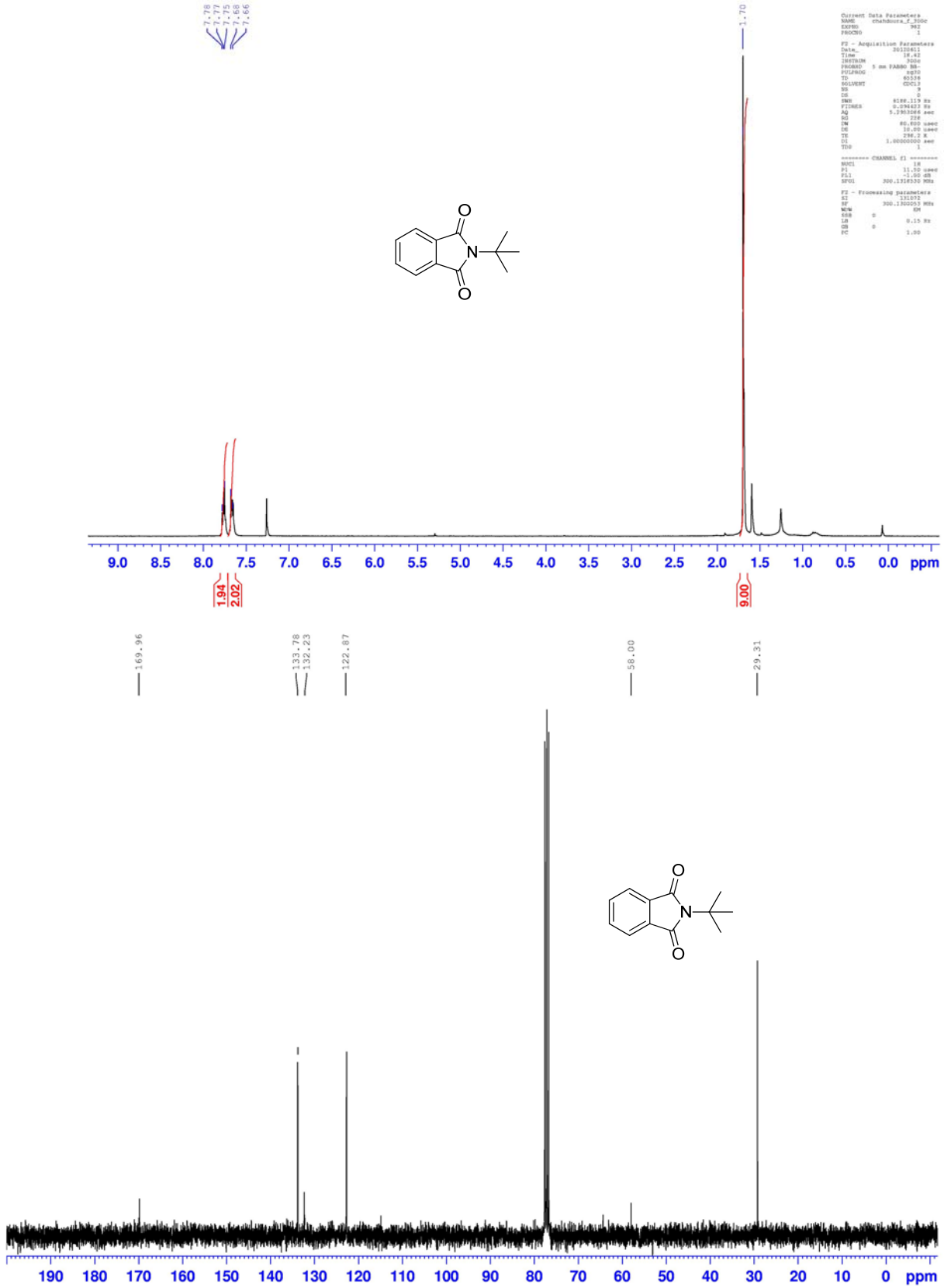

773
fc-773- 1653 (12.267) Cm (1650:1657)
, 13-Jun-2012 + 19:28:31
Scan EI+
4.71e8
%
160.1373
161.1222
231.2083
130.1088
77.0682
76.0737
104.1453
105.1297
133.0818
148.1090
174.1837
41.1334
39.1109
50.0604
55.0794
75.0625
78.0881
67.0818
102.0998
98.1531
117.1513
118.1644
147.1205
149.1314
162.1814
163.1733
186.1860
188.1624
189.1949
202.1876
203.1987
232.2142
233.2147
m/z



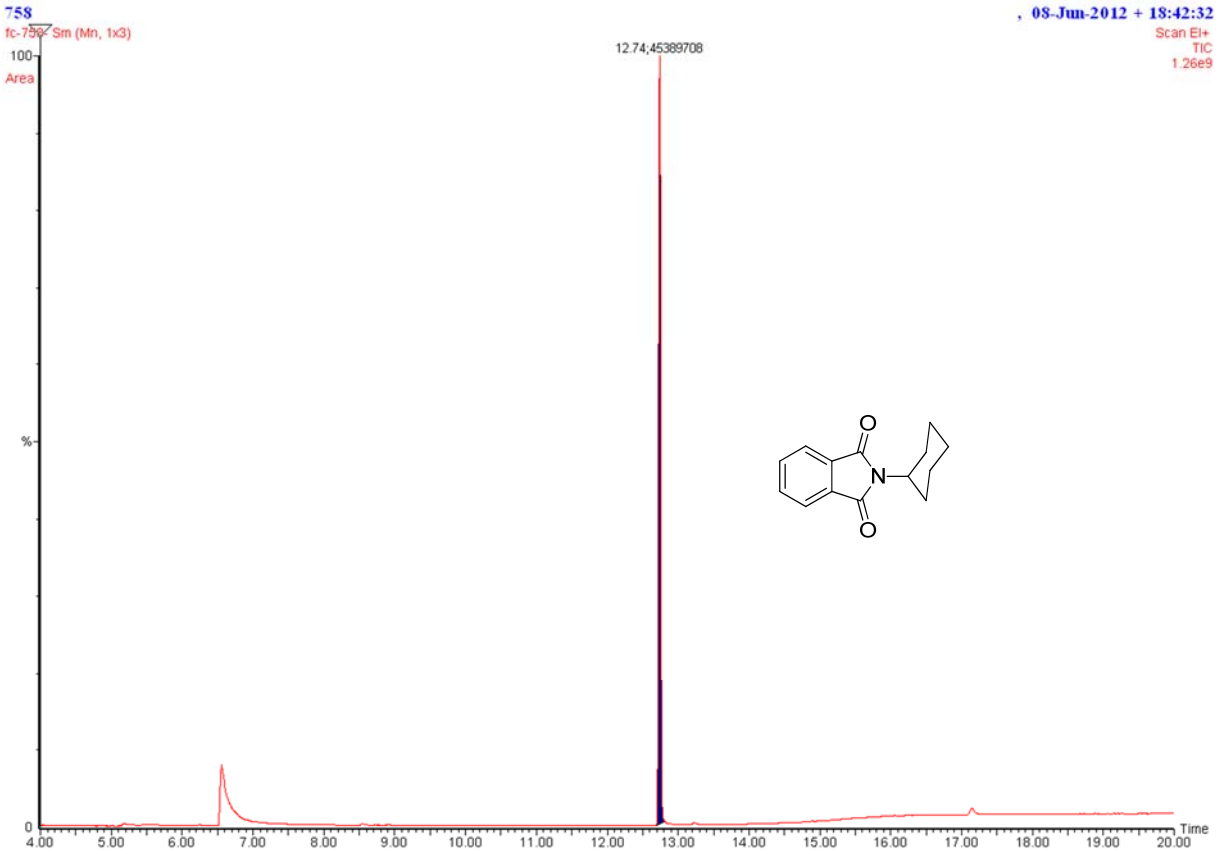
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a10**

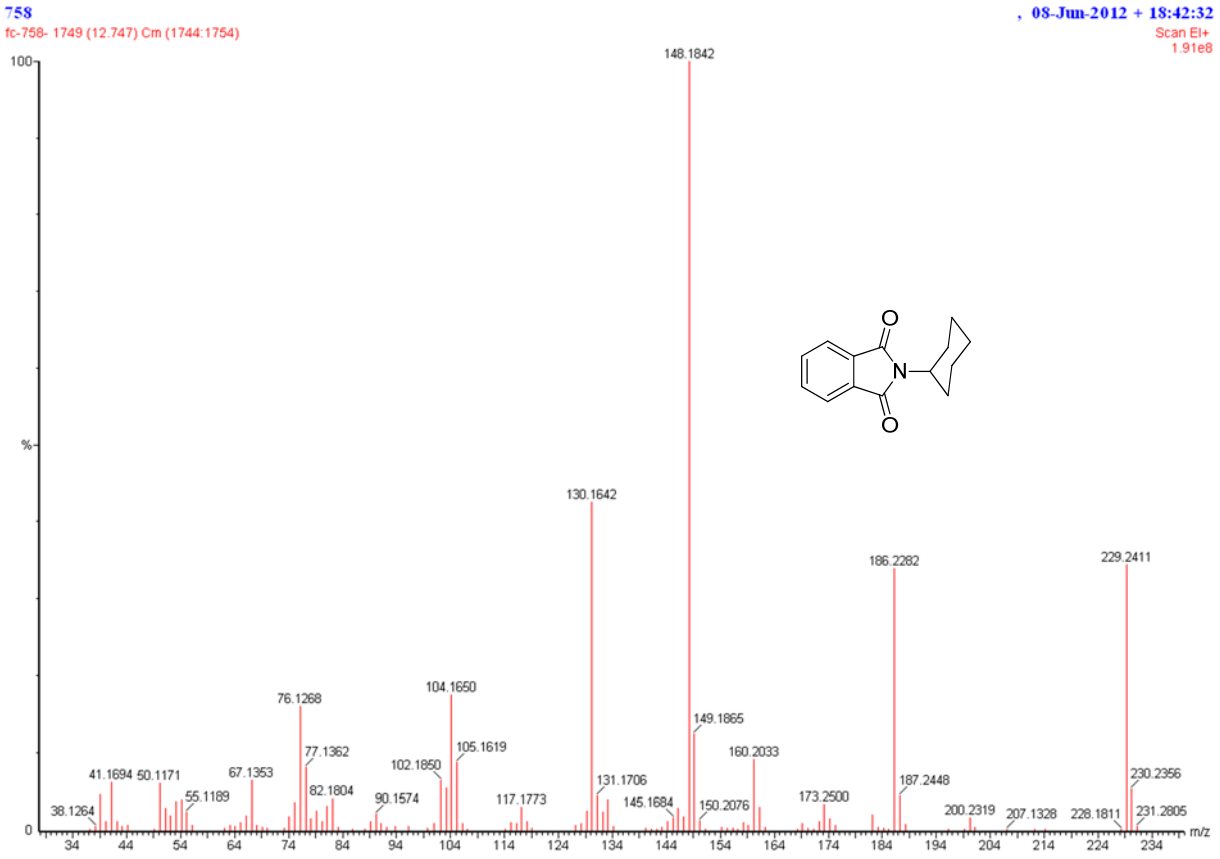

759
fc-759- Sm (Mn, 1x3)
Area
100
%
0
10.50,7392343
, 11-Jun-2012 + 16:25:38
Scan EI+
TIC
2.05e8
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
Time

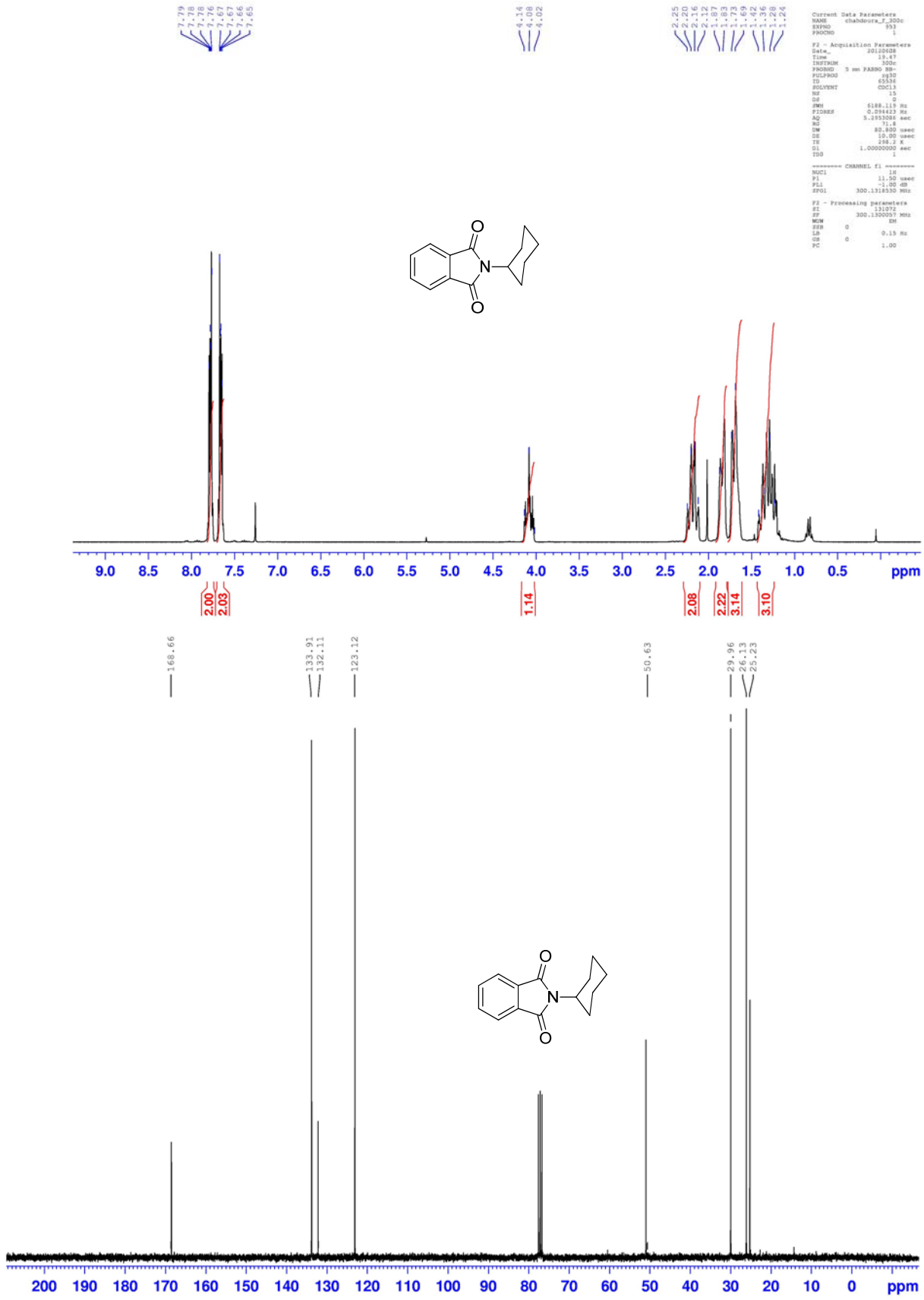

759
fc-759- 1299 (10.496) Cm (1295:1306)
, 11-Jun-2012 + 16:25:38
Scan EI+
3.01e7
100
%
0
188.1889
130.1103
148.1266
203.1956
189.2208
204.1843
160.1513
149.1645
131.1700
104.1505
102.1778
66.0706
76.0796
41.1393
50.0851
56.1011
39.1585
42.1424
38.1209
57.1143
75.0825
77.0885
86.6203
94.0540
129.1334
147.1391
O
N
O
31 41 51 61 71 81 91 101 111 121 131 141 151 161 171 181 191 201 211
m/z



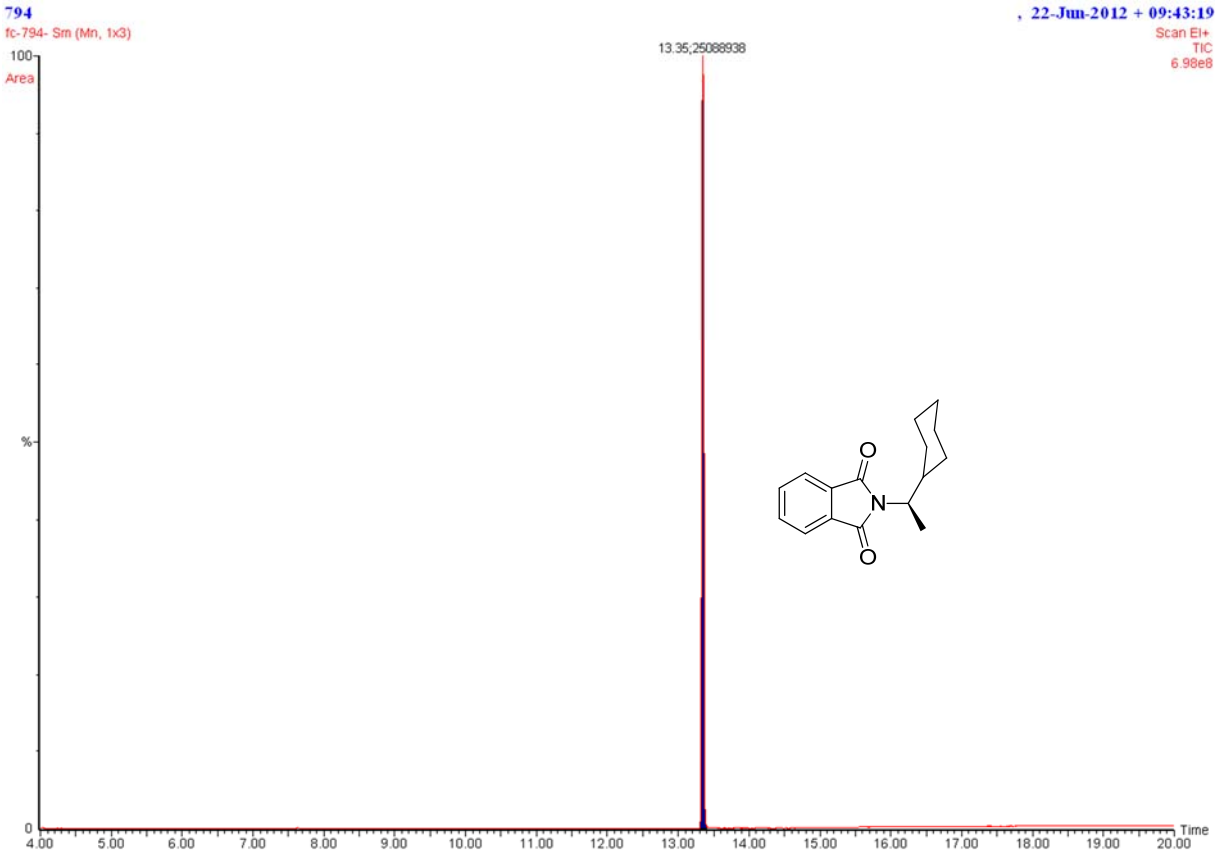
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a11**

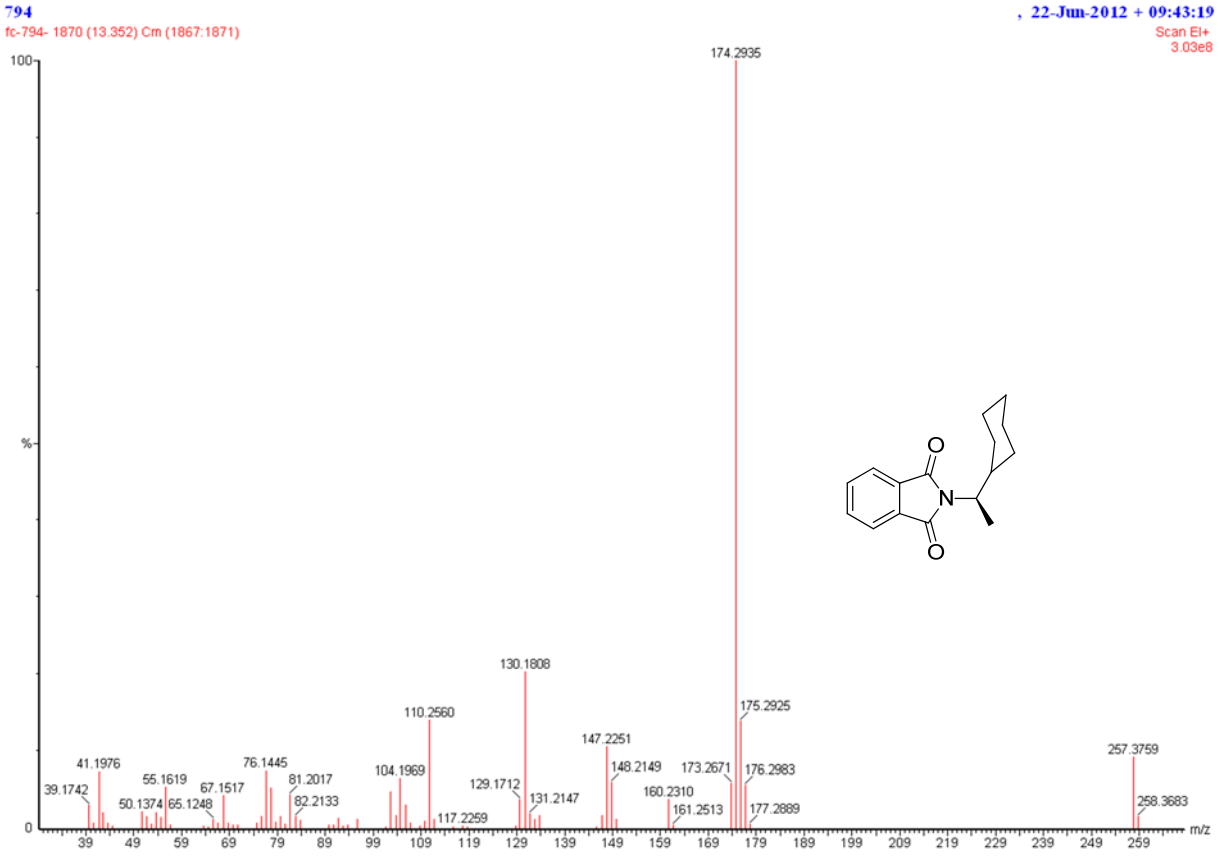

758
fc-758 Sm (Mn, 1x3)
, 08-Jun-2012 + 18:42:32
Scan EI+
TIC
1.26e9
100
Area
%
0
12.74;45389708
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N
O

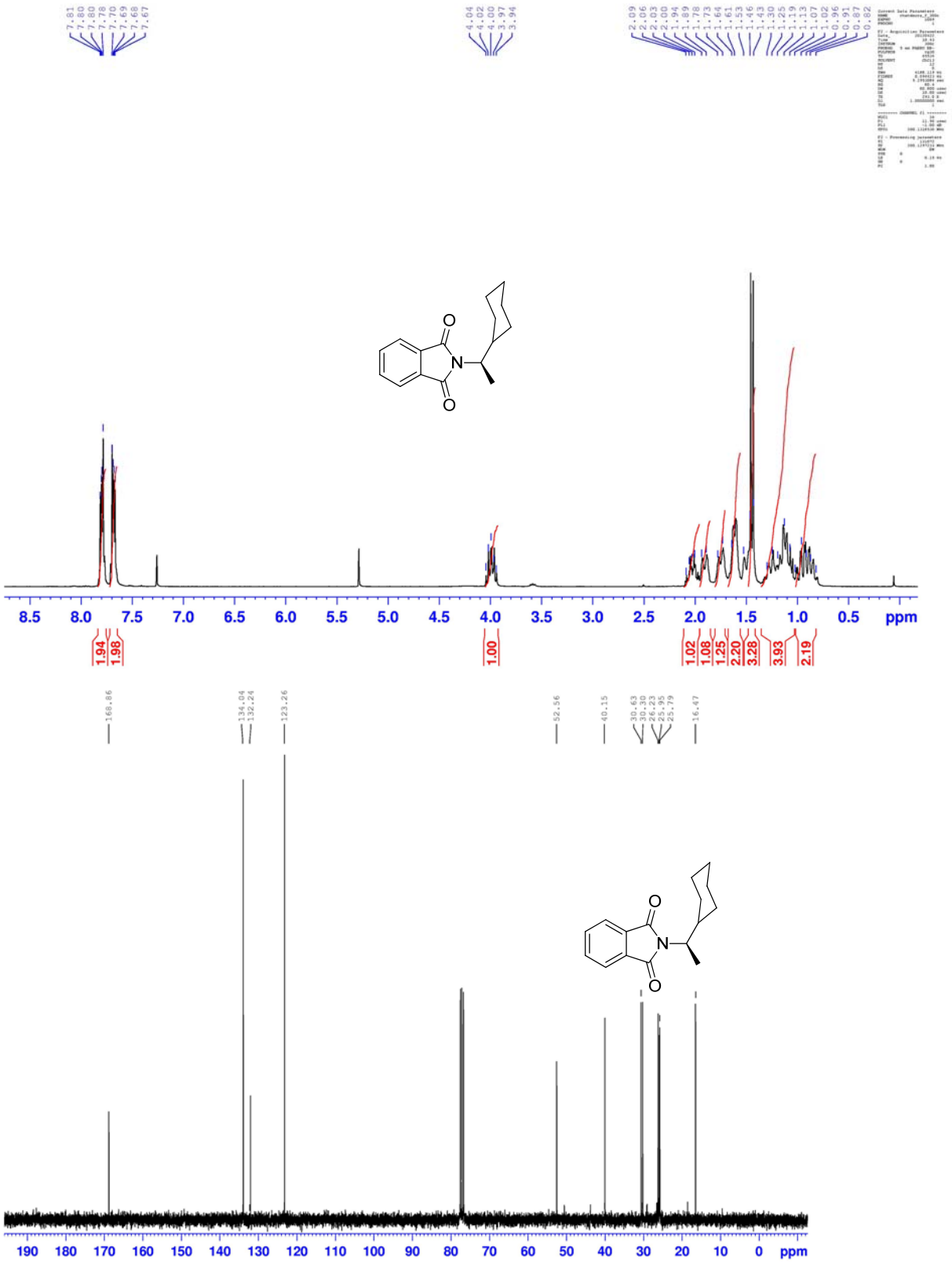

758
fc-758- 1749 (12.747) Cm (1744:1754)
, 08-Jun-2012 + 18:42:32
Scan EI+
1.91e8
100
%
0
148.1842
130.1642
186.2262
229.2411
104.1650
76.1268
149.1865
105.1619
77.1362
160.2033
102.1850
41.1694
50.1171
67.1353
131.1706
187.2448
230.2356
55.1189
82.1804
90.1574
117.1773
145.1684
150.2076
173.2500
200.2319
207.1328
228.1811
231.2805
38.1264
34
44
54
64
74
84
94
104
114
124
134
144
154
164
174
184
194
204
214
224
234
m/z
O
N
O



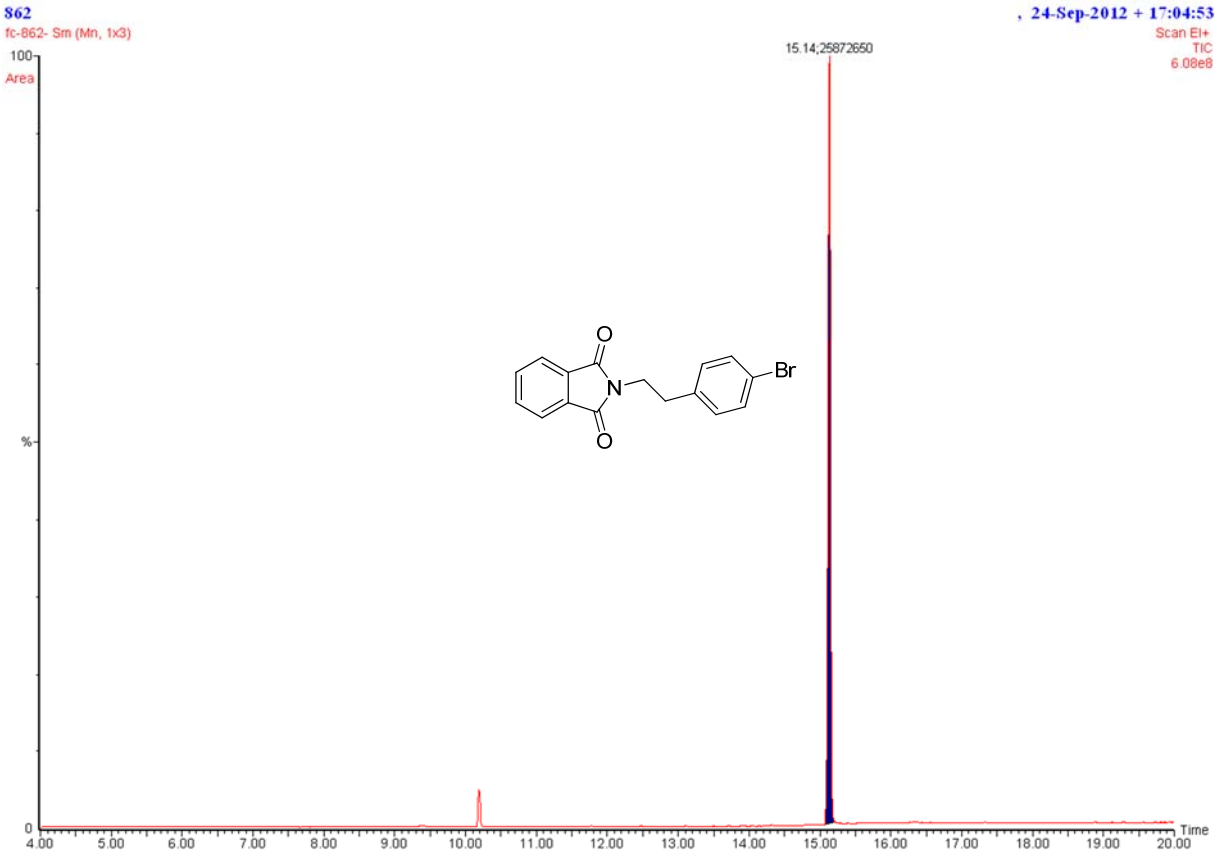
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a12**

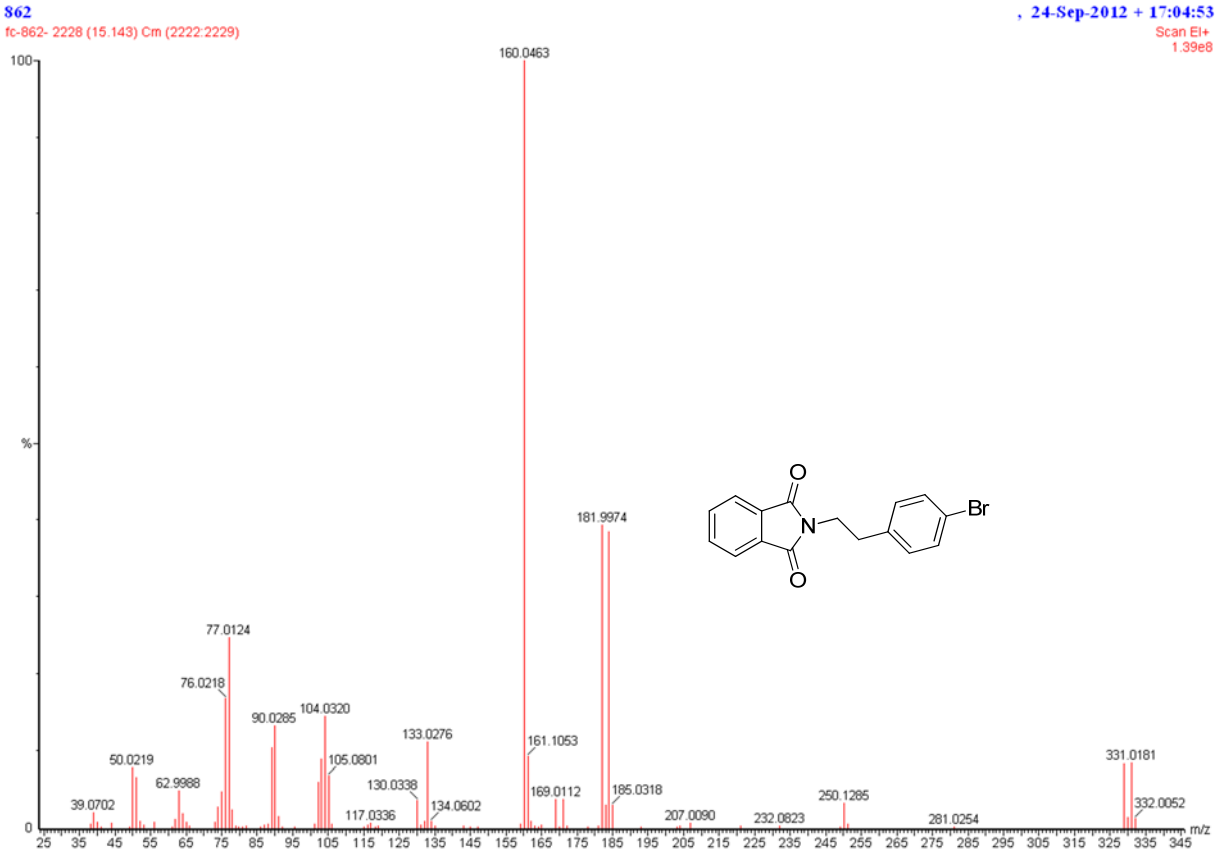

794
fc-794- Sm (Mn, 1x3)
, 22-Jun-2012 + 09:43:19
Scan EI+
TIC
6.98e8
Area
13.35;25088938
%
Time

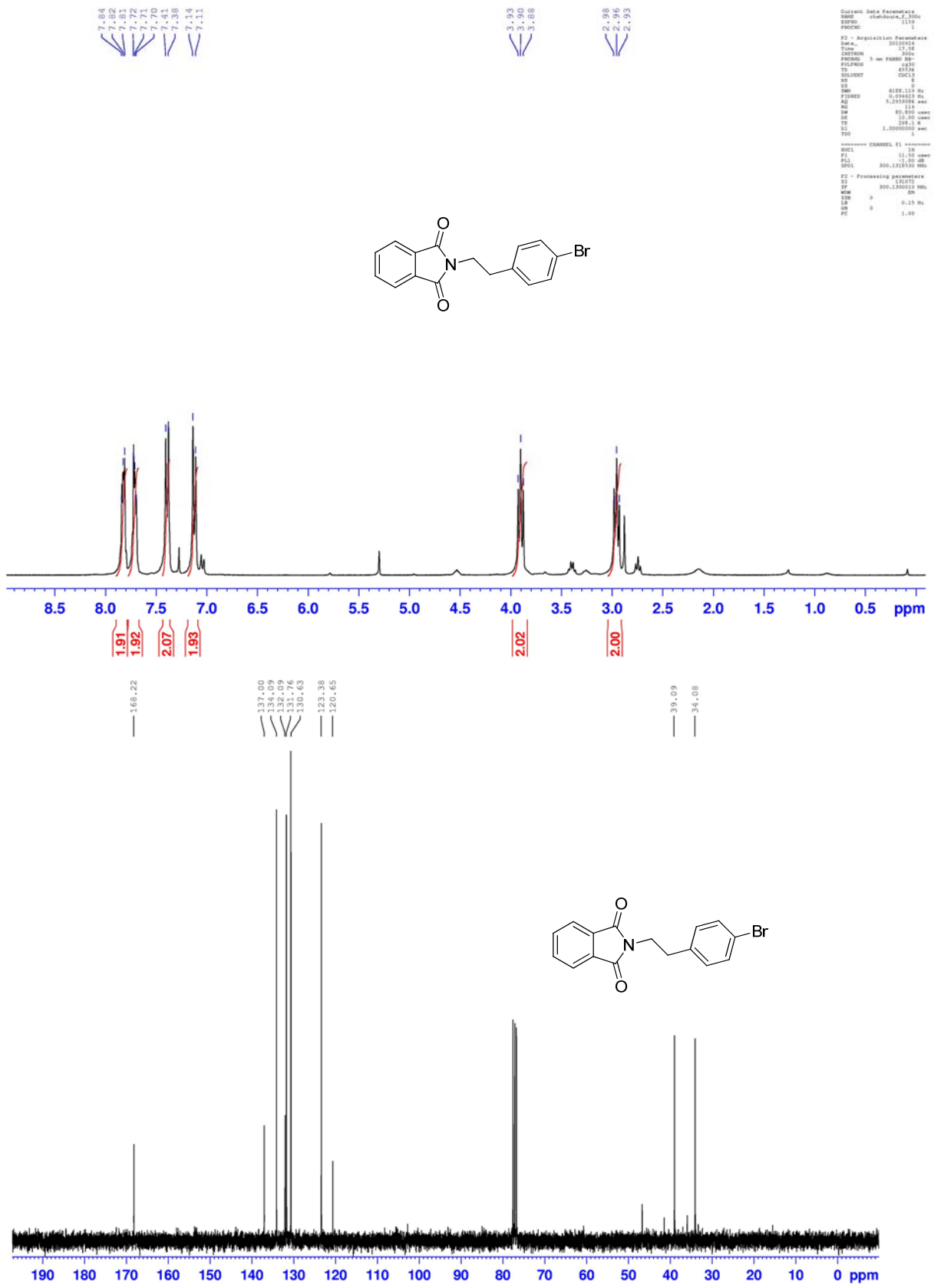

794
fc-794- 1870 (13.352) Cm (1867:1871)
, 22-Jun-2012 + 09:43:19
Scan EI+
3.03e8
%
174.2935
130.1808
110.2560
175.2925
147.2251
257.3759
41.1976
76.1445
55.1619
81.2017
104.1969
148.2149
173.2671
176.2983
39.1742
67.1517
129.1712
160.2310
258.3683
50.1374
65.1248
82.2133
131.2147
161.2513
177.2889
117.2259
m/z



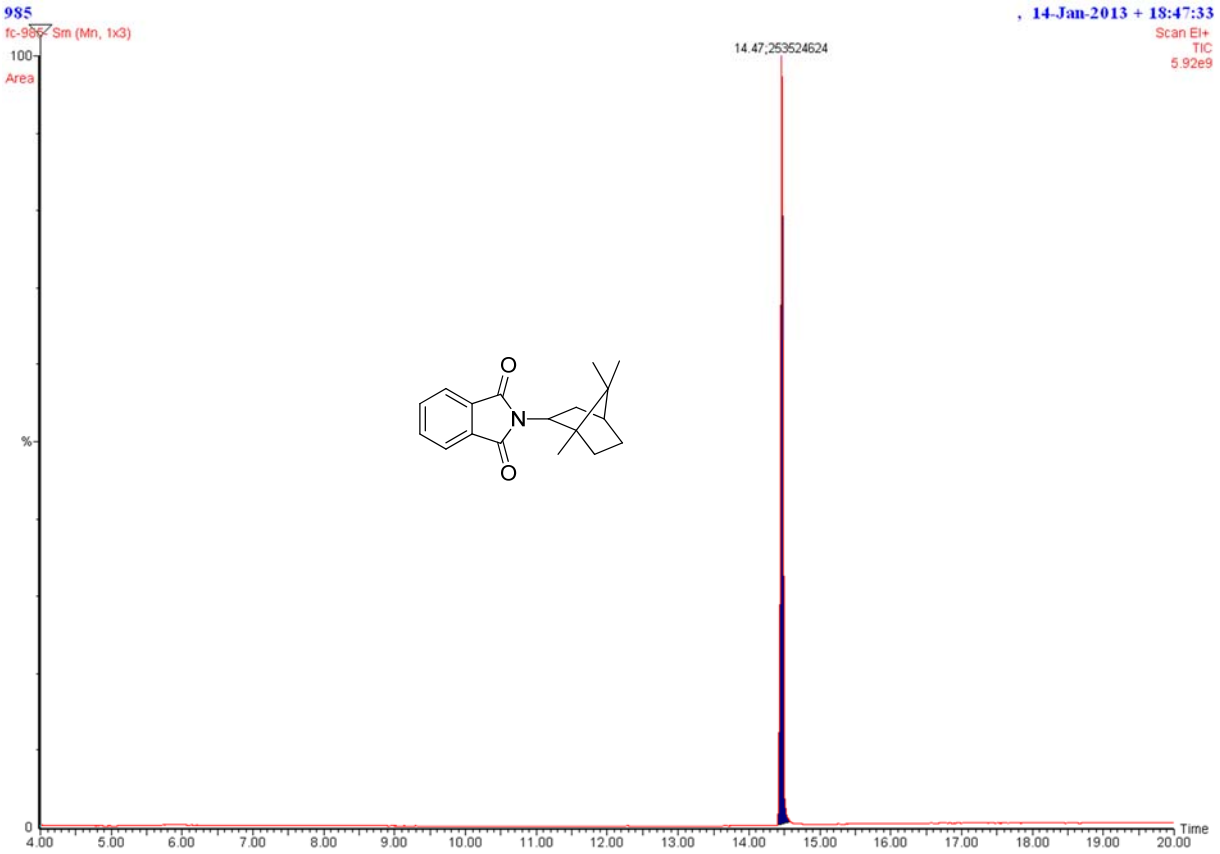
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a13**

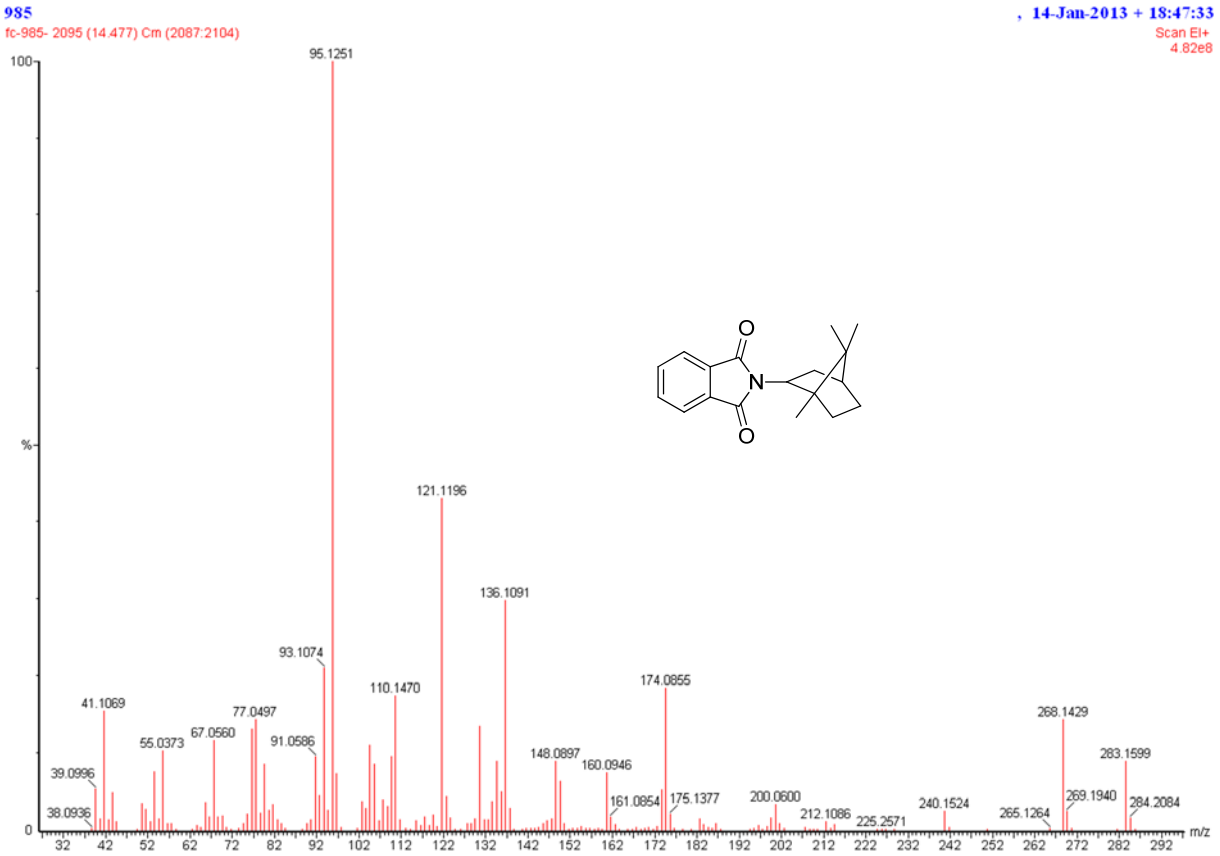

862
, 24-Sep-2012 + 17:04:53
fc-862- Sm (Mn, 1x3)
Scan EI+
TIC
6.08e8
100
Area
15.14;25872650
%
0
Time
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
O
N
O
Br

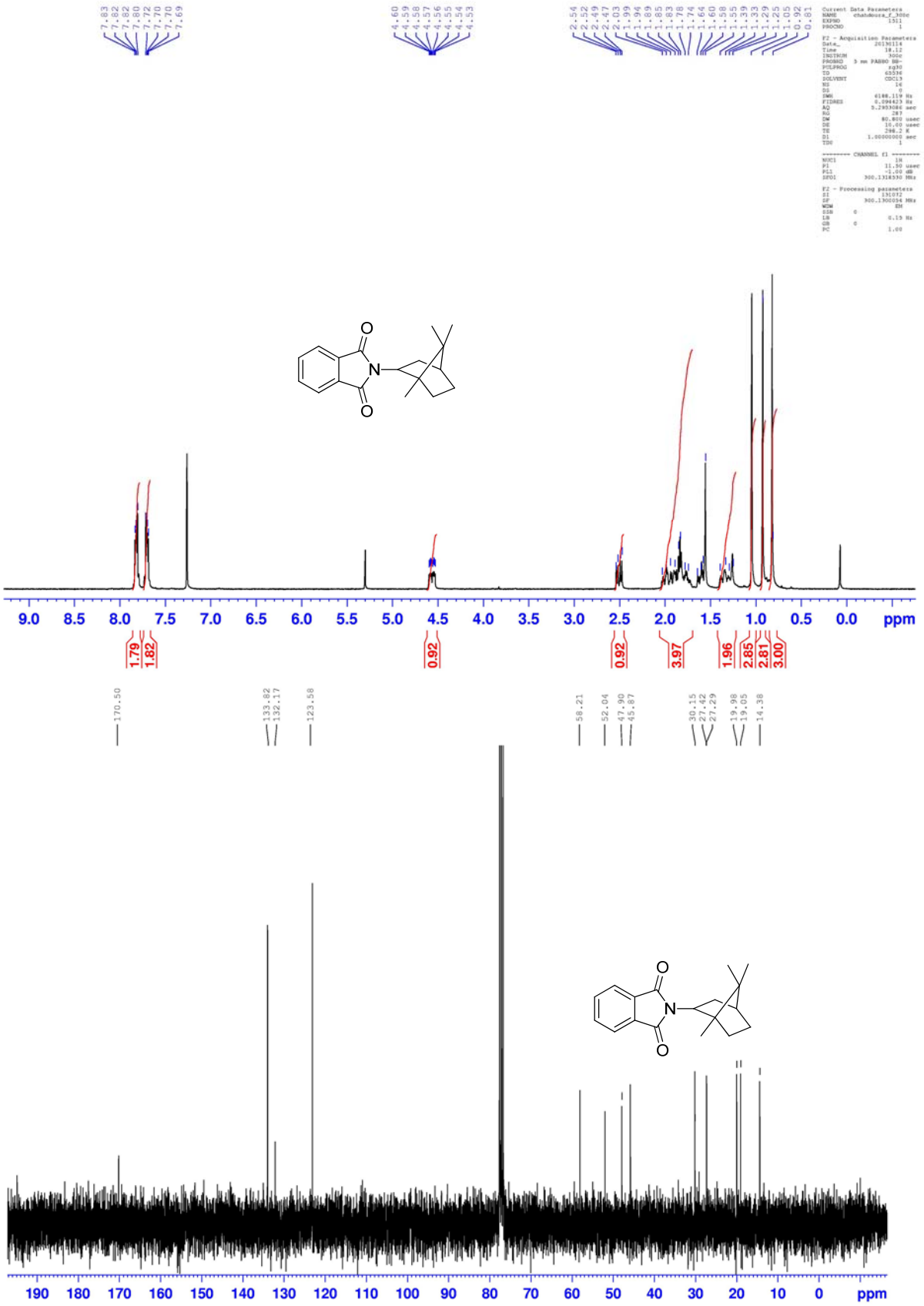

862
, 24-Sep-2012 + 17:04:53
fc-862- 2228 (15.143) Cm (2222:2229)
Scan EI+
1.39e8
100
%
0
160.0463
181.9974
77.0124
76.0218
90.0285
104.0320
133.0276
161.1053
50.0219
105.0801
62.9988
130.0338
169.0112
185.0318
39.0702
134.0602
117.0336
207.0090
232.0823
250.1285
281.0254
331.0181
332.0052
m/z
25 35 45 55 65 75 85 95 105 115 125 135 145 155 165 175 185 195 205 215 225 235 245 255 265 275 285 295 305 315 325 335 345
O
N
O
Br



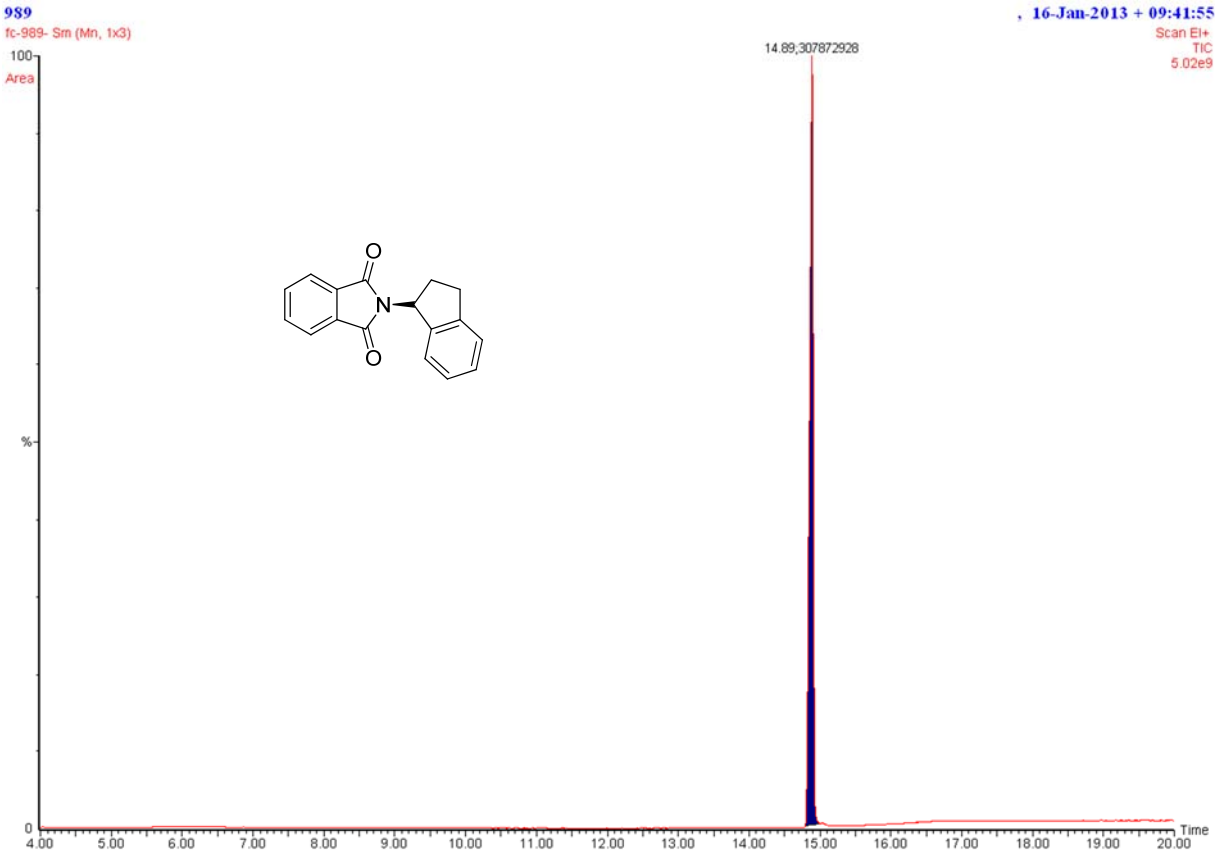
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a14**

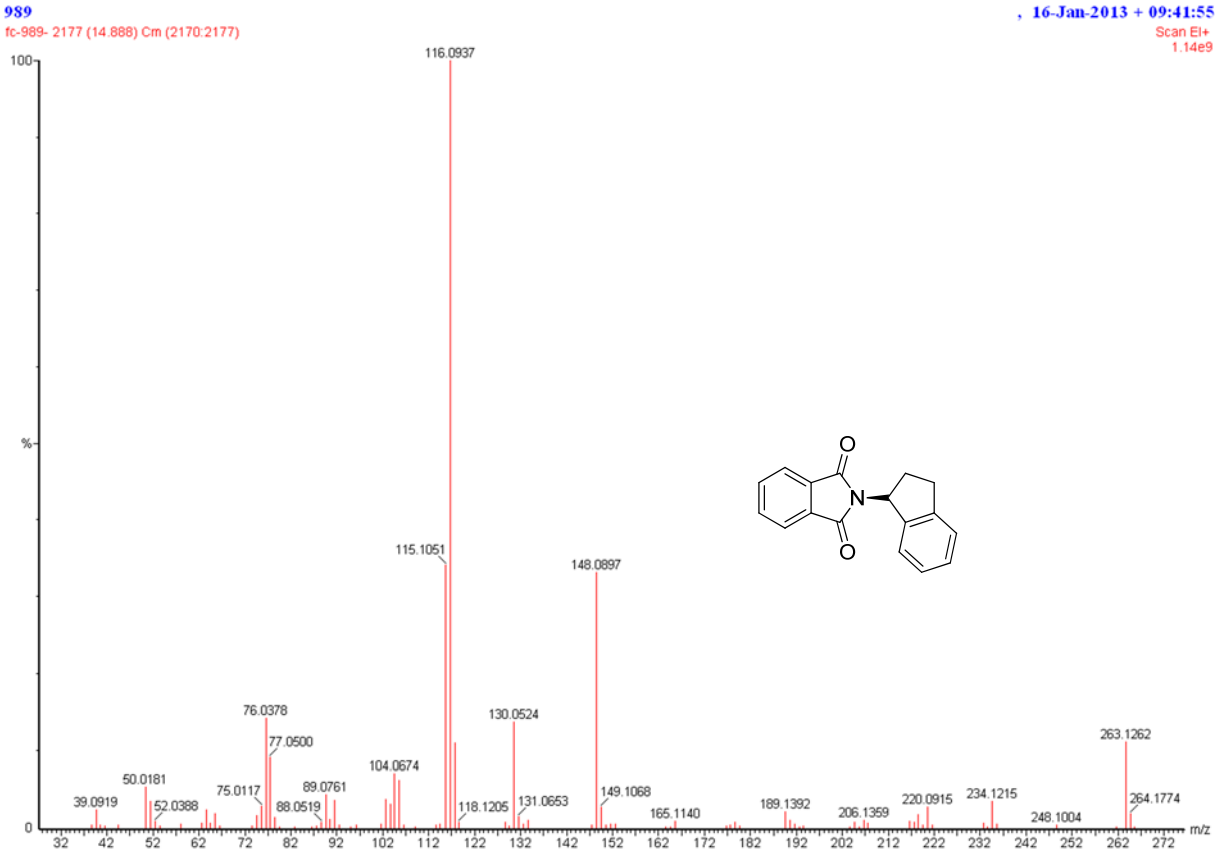

985
, 14-Jan-2013 + 18:47:33
fc-985 Sm (Mn, 1x3)
Scan EI+
TIC
5.92e9
100
Area
14.47;253524624
%
0
Time
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
O
N
O

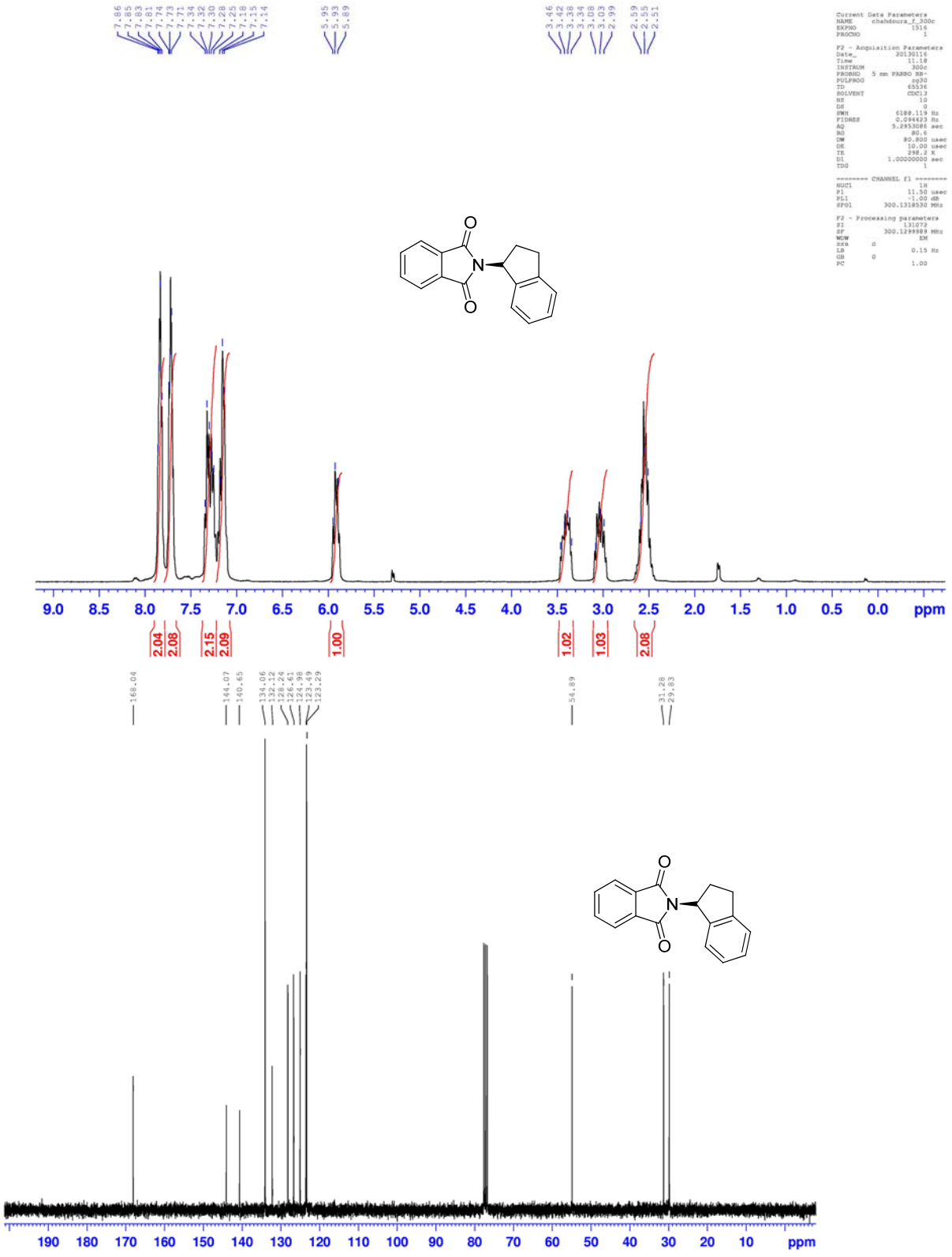

985
, 14-Jan-2013 + 18:47:33
fc-985- 2095 (14.477) Cm (2087:2104)
Scan EI+
4.82e8
100
%
0
95.1251
121.1196
136.1091
93.1074
110.1470
174.0855
41.1069
77.0497
67.0560
55.0373
91.0586
148.0897
160.0946
39.0996
38.0936
161.0854
175.1377
200.0600
212.1086
225.2571
240.1524
265.1264
268.1429
269.1940
283.1599
284.2084
m/z
32 42 52 62 72 82 92 102 112 122 132 142 152 162 172 182 192 202 212 222 232 242 252 262 272 282 292
O
N
O



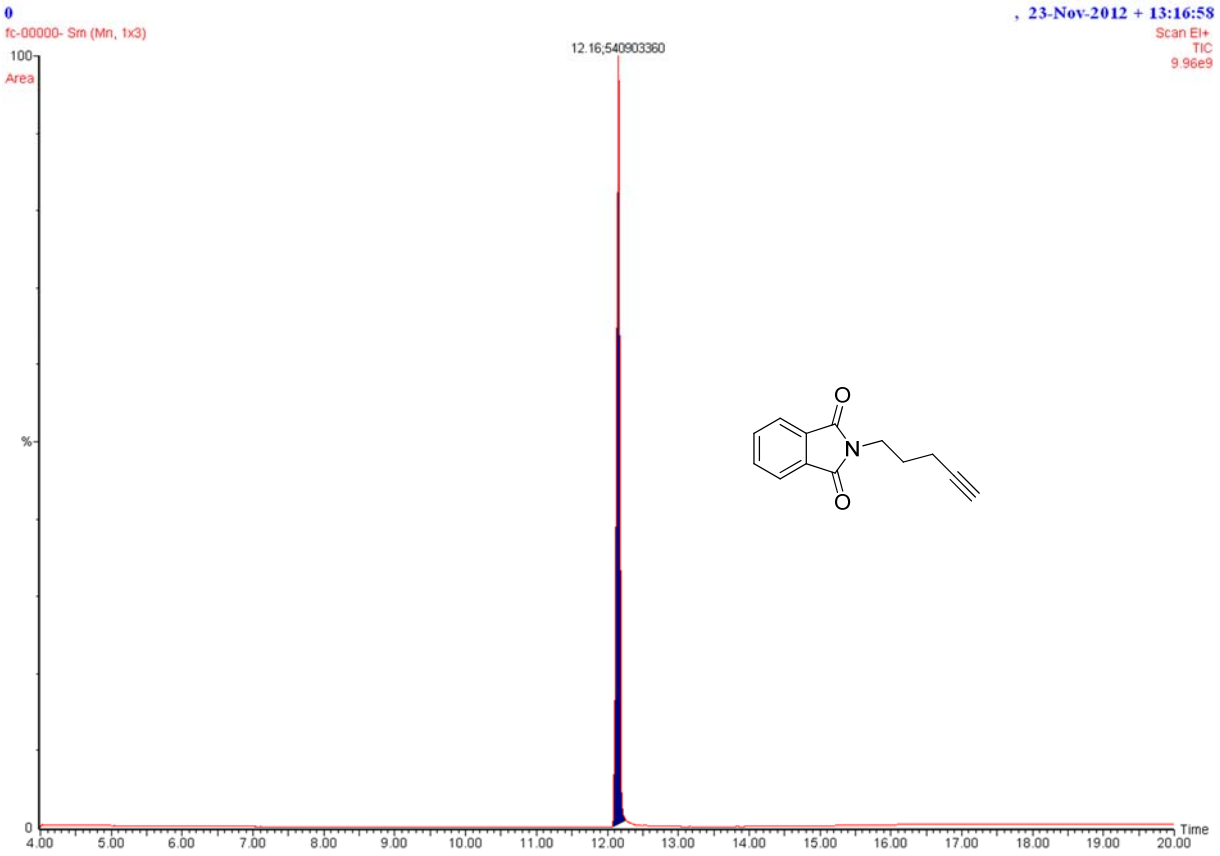
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a15**

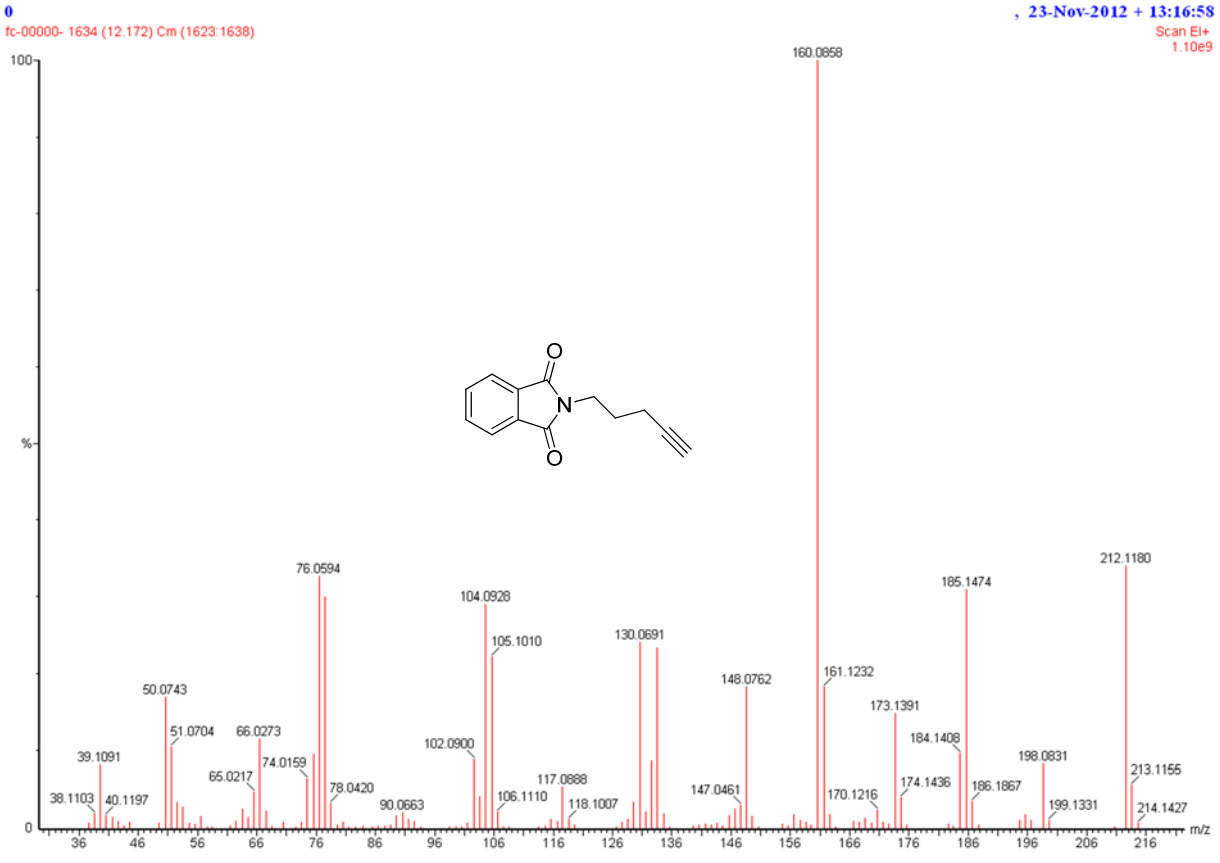

989
, 16-Jan-2013 + 09:41:55
fc-989- Sm (Mn, 1x3)
Scan EI+
TIC
5.02e9
Area
14.89;307872928
Time

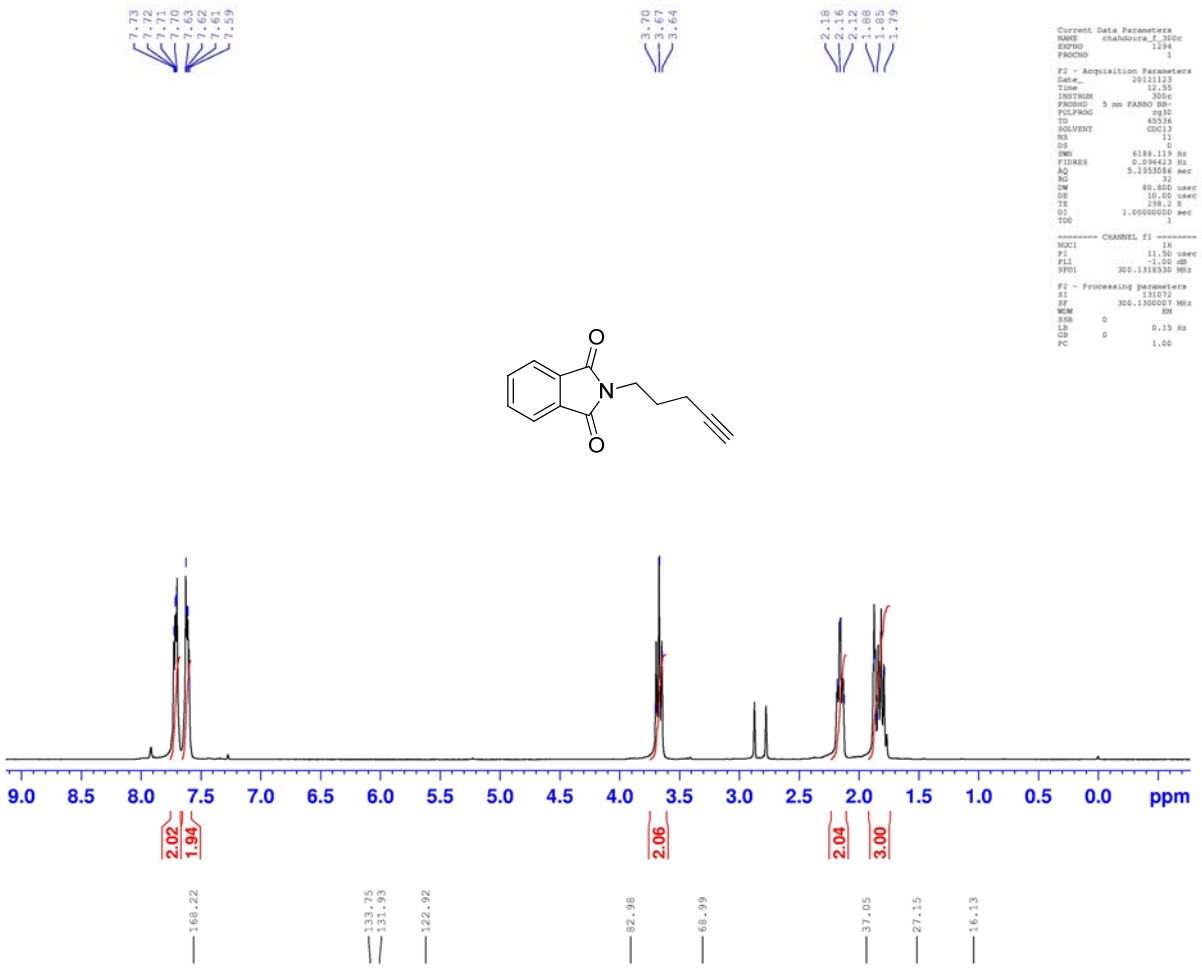

989
, 16-Jan-2013 + 09:41:55
fc-989- 2177 (14.888) Cm (2170:2177)
Scan EI+
1.14e9
116.0937
115.1051
148.0897
130.0524
76.0378
77.0500
104.0674
50.0181
39.0919
52.0388
75.0117
89.0761
88.0519
118.1205
131.0653
149.1068
165.1140
189.1392
206.1359
220.0915
234.1215
248.1004
263.1262
264.1774
m/z

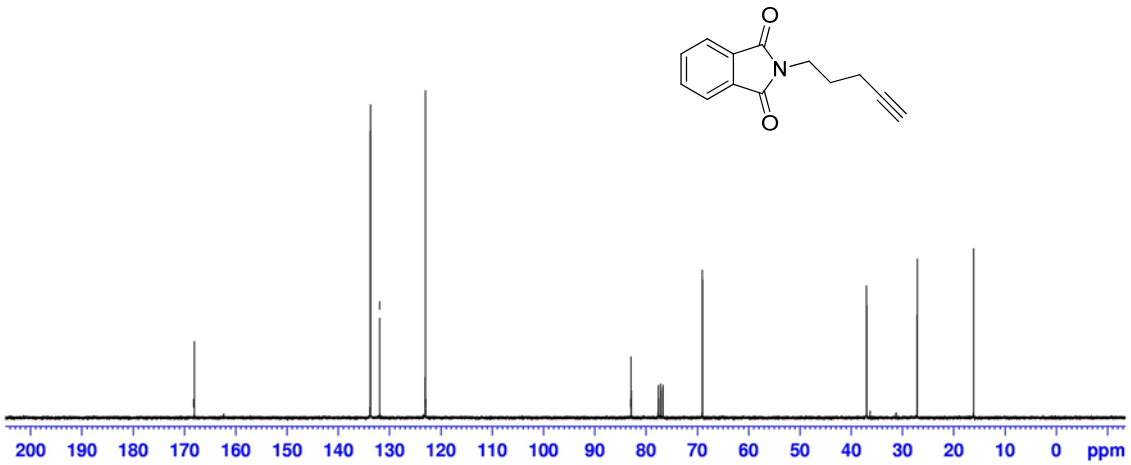


GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a16**

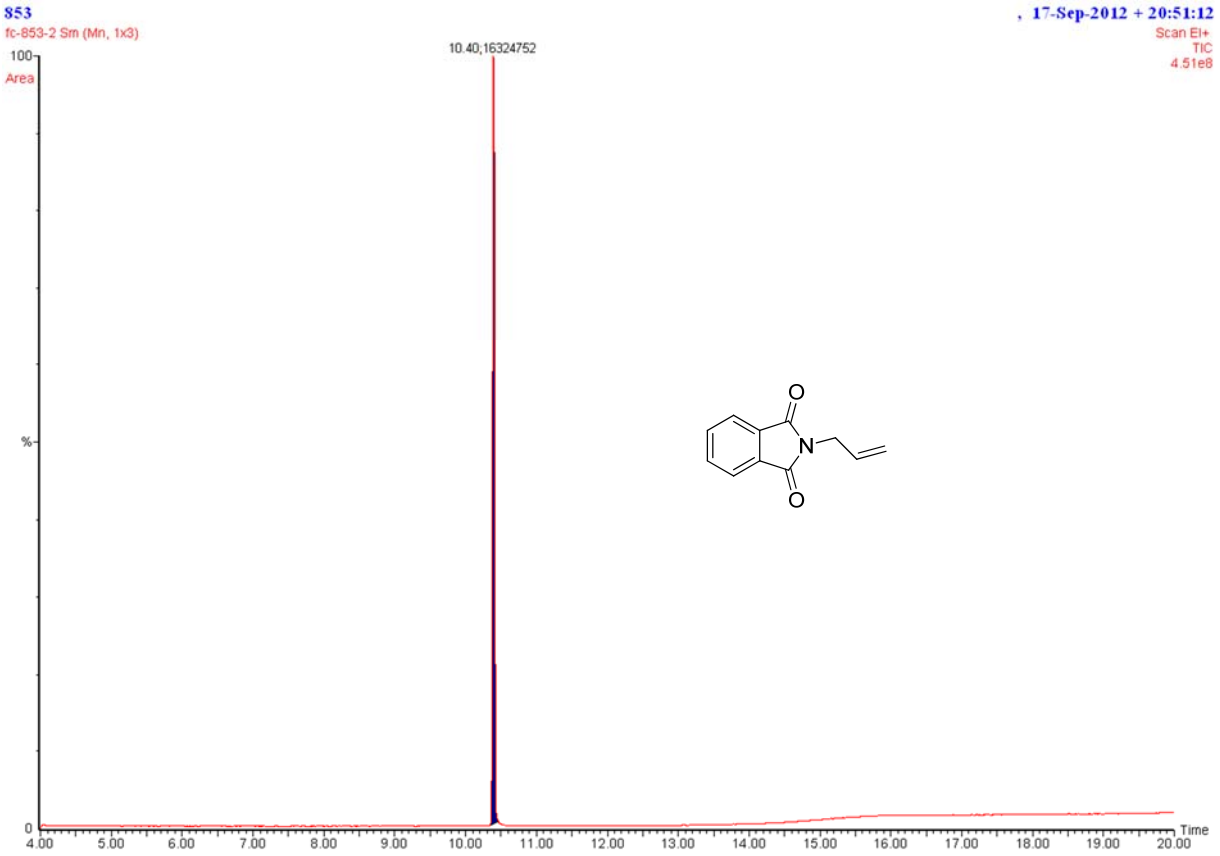

0
, 23-Nov-2012 + 13:16:58
fc-00000- Sm (Mn, 1x3)
Scan EI+
TIC
9.96e9
100
Area
12.16;540903360
%
0
Time
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
O
N
O

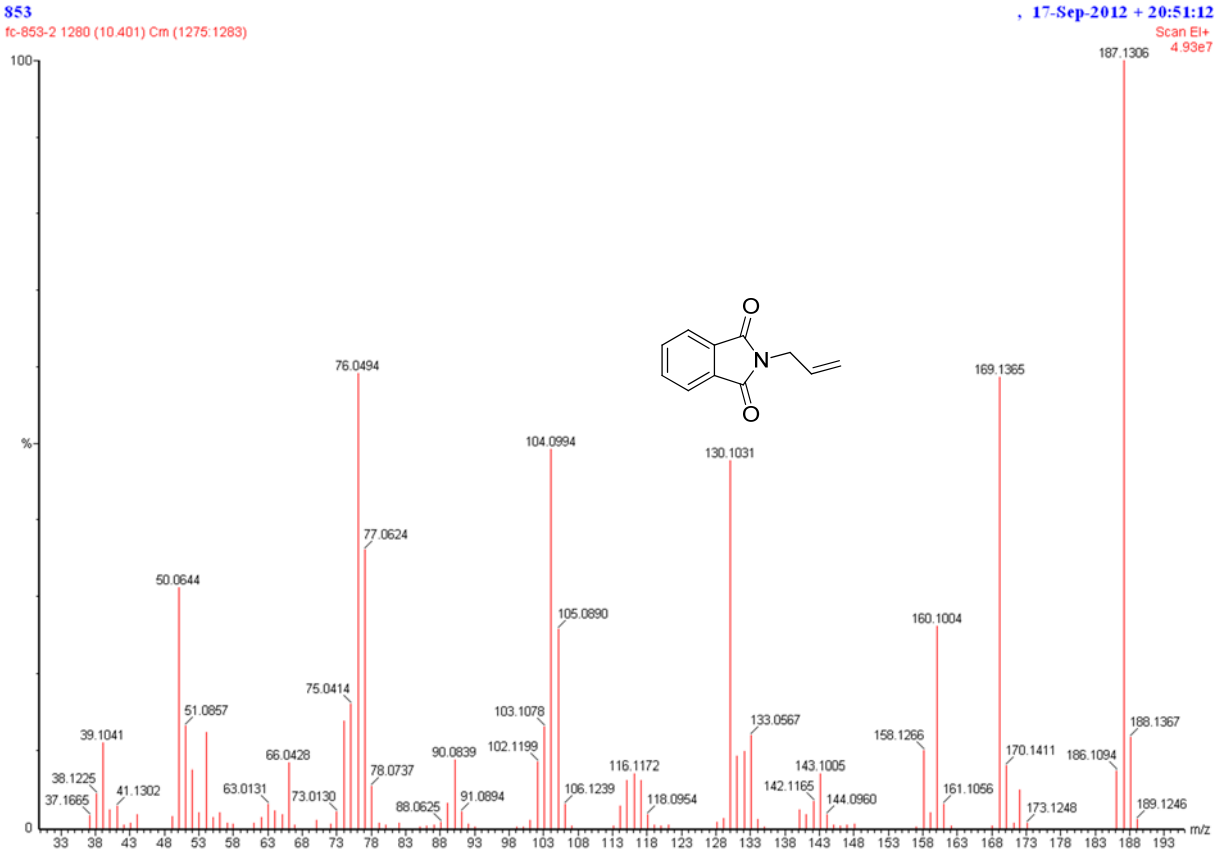

0
, 23-Nov-2012 + 13:16:58
fc-00000- 1634 (12.172) Cm (1623:1638)
Scan EI+
1.10e9
100
%
0
m/z
160.0858
212.1180
185.1474
76.0594
104.0928
105.1010
130.0691
148.0762
161.1232
173.1391
50.0743
51.0704
66.0273
74.0159
39.1091
65.0217
78.0420
102.0900
117.0888
106.1110
118.1007
147.0461
170.1216
174.1436
184.1408
186.1867
198.0831
199.1331
213.1155
214.1427
38.1103
40.1197
90.0663
36
46
56
66
76
86
96
106
116
126
136
146
156
166
176
186
196
206
216
O
N
O

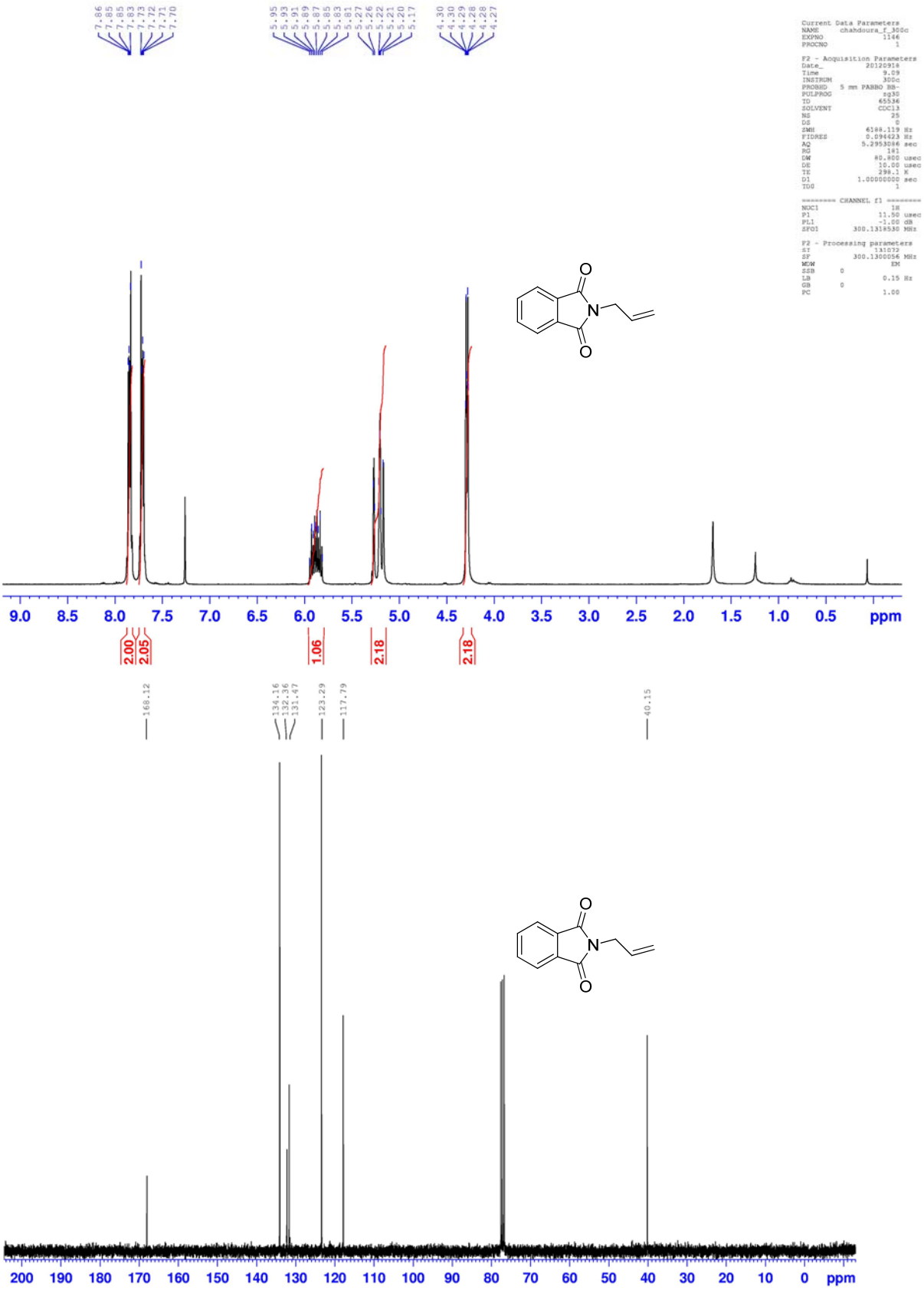




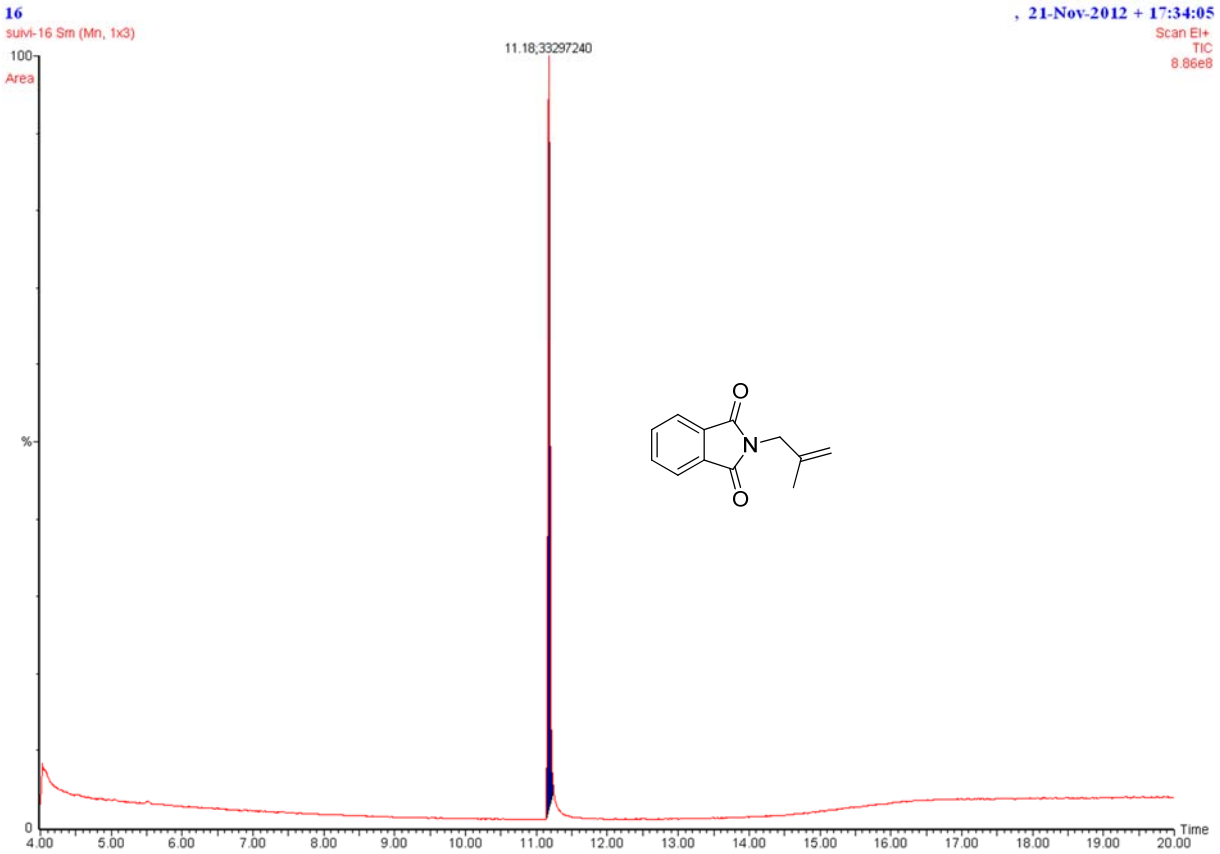
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a17**

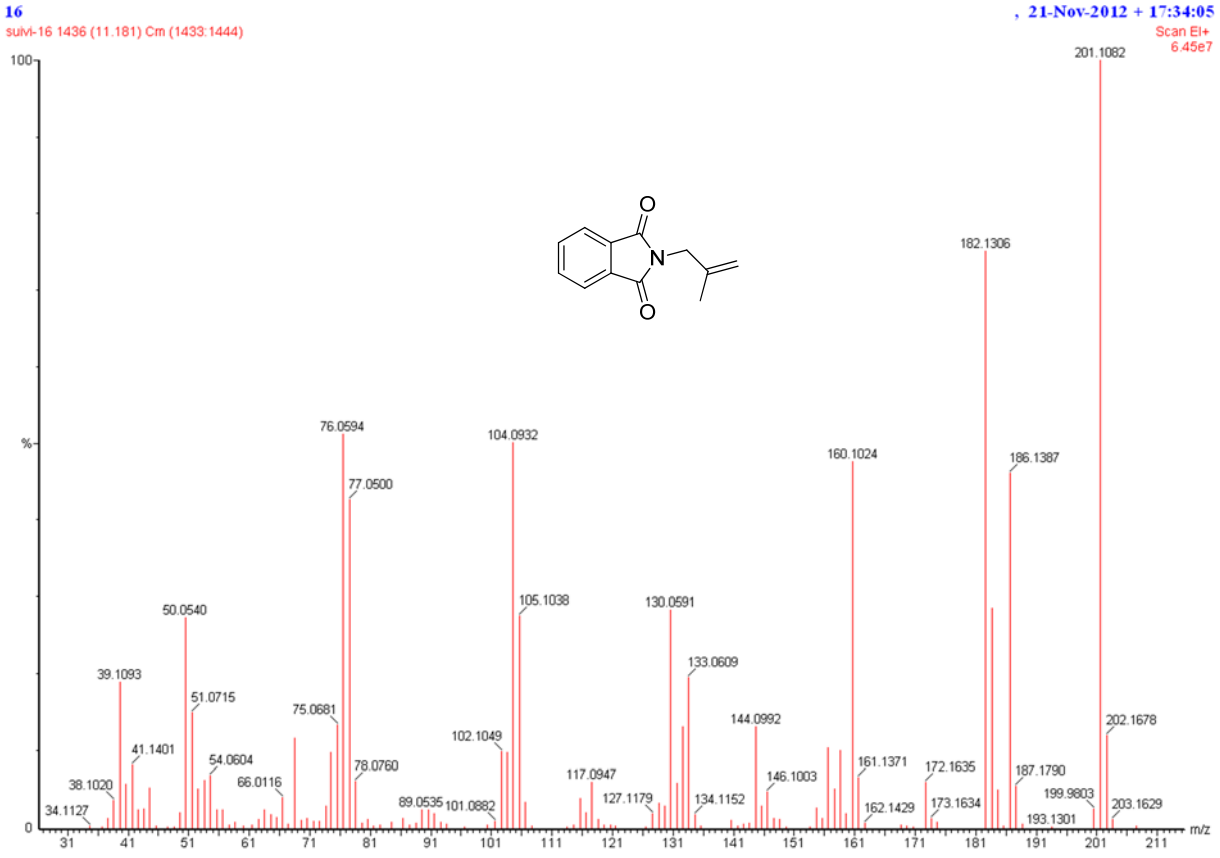

853
fc-853-2 Sm (Mn, 1x3)
, 17-Sep-2012 + 20:51:12
Scan EI+
TIC
4.51e8
10.40,16324752
100
Area
%
0
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
Time
O
N
O

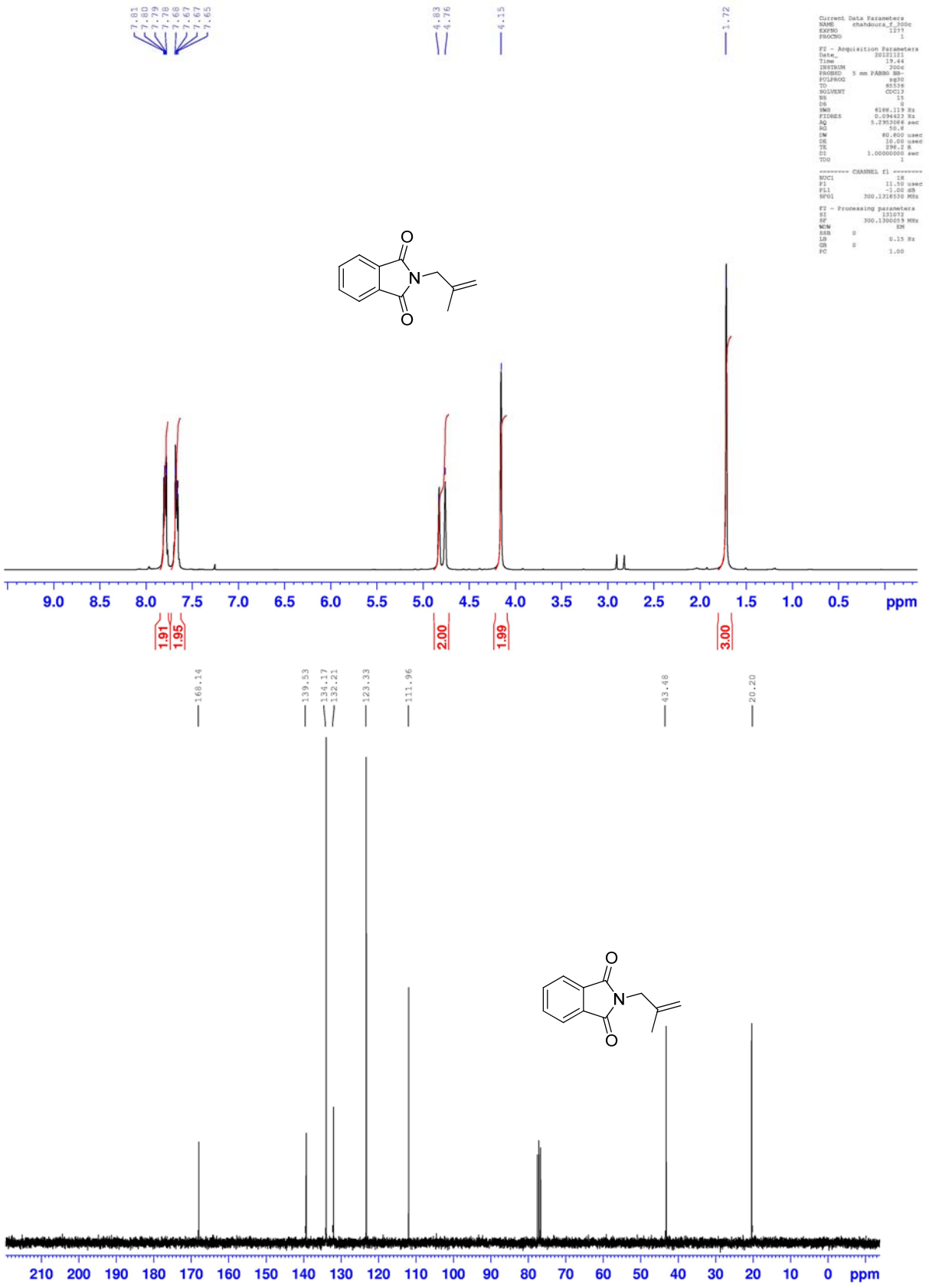

853
fc-853-2 1280 (10.401) Cm (1275:1283)
, 17-Sep-2012 + 20:51:12
Scan EI+
4.93e7
100
%
0
187.1306
76.0494
104.0994
130.1031
169.1365
77.0624
50.0644
105.0890
160.1004
75.0414
51.0857
103.1078
133.0567
188.1367
39.1041
158.1266
66.0428
102.1199
143.1005
170.1411
186.1094
90.0839
116.1172
78.0737
38.1225
41.1302
63.0131
73.0130
106.1239
142.1165
144.0960
161.1056
189.1246
37.1665
88.0625
91.0894
118.0954
173.1248
33 38 43 48 53 58 63 68 73 78 83 88 93 98 103 108 113 118 123 128 133 138 143 148 153 158 163 168 173 178 183 188 193
m/z
O
N
O



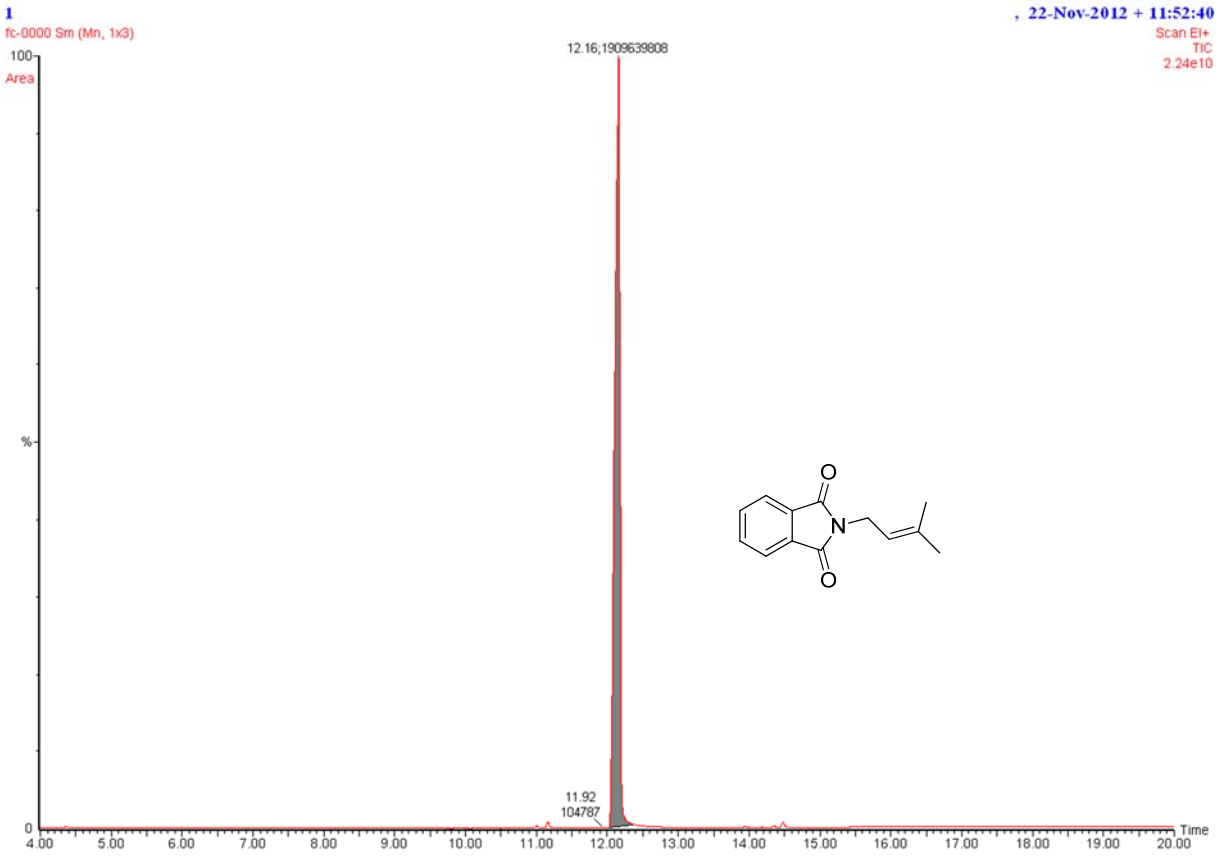
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a18**

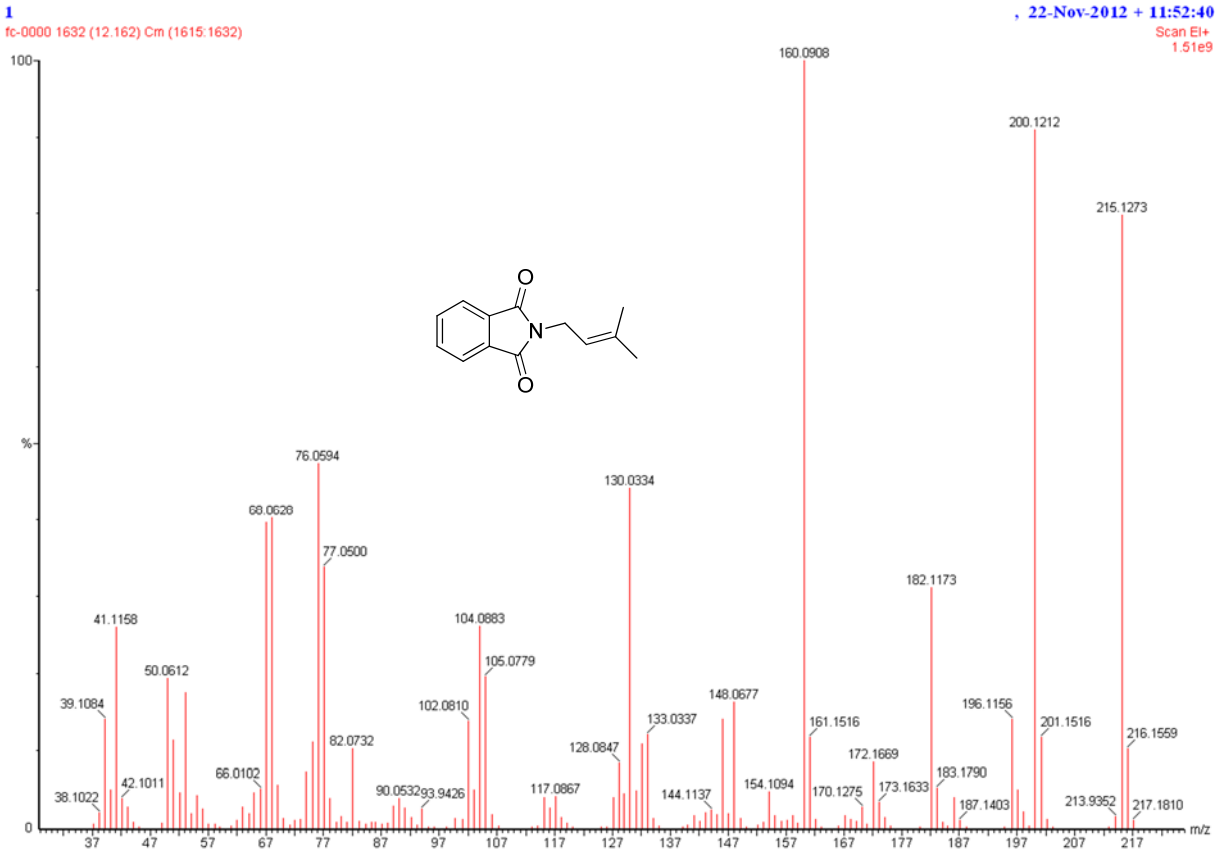

16
, 21-Nov-2012 + 17:34:05
suivi-16 Sm (Mn, 1x3)
Scan EI+
TIC
8.86e8
11.18;33297240
100
Area
%
0
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N
O

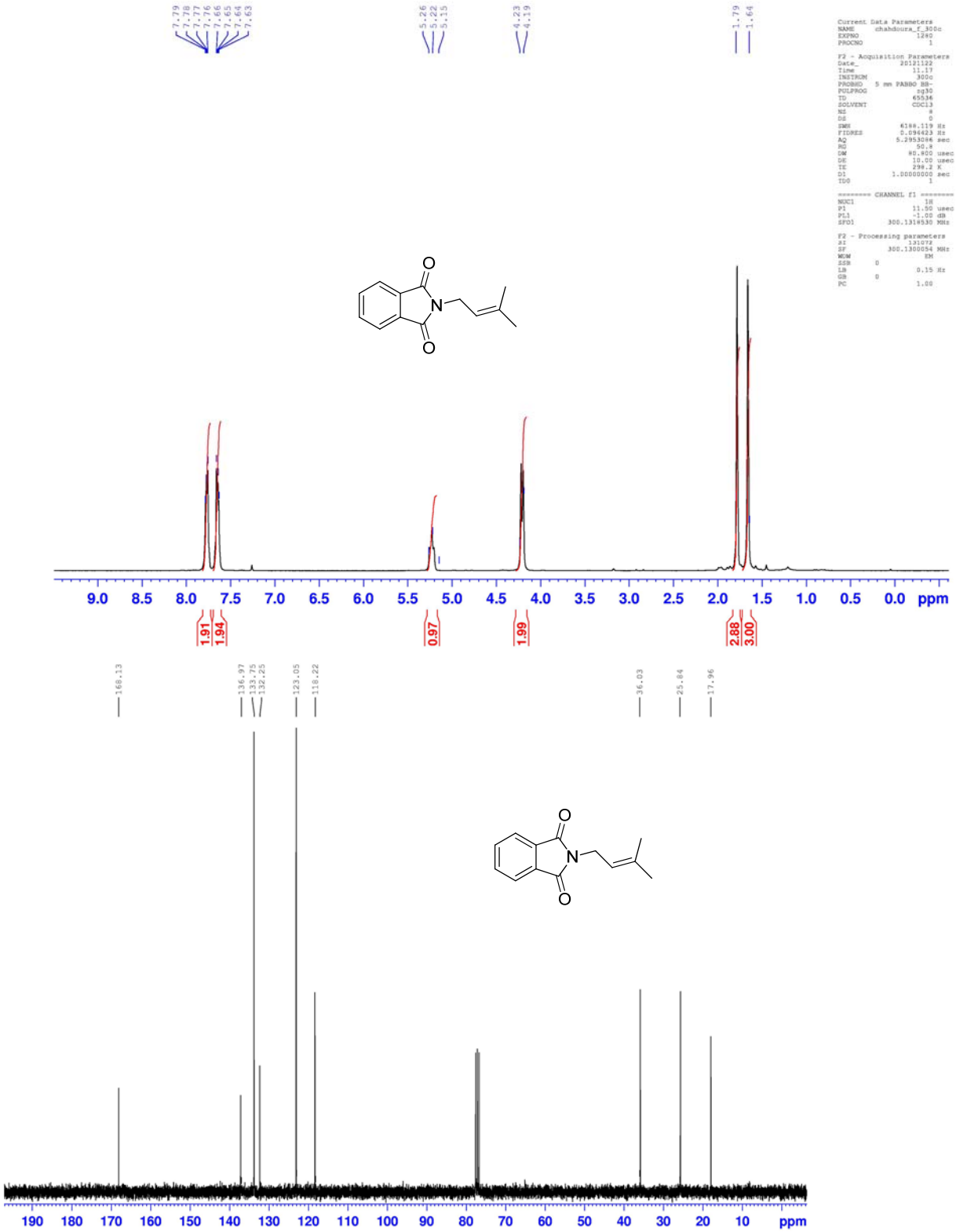

16
, 21-Nov-2012 + 17:34:05
suivi-16 1436 (11.181) Cm (1433:1444)
Scan EI+
6.45e7
100
%
0
201.1082
182.1306
76.0594
104.0932
160.1024
186.1387
77.0500
105.1038
50.0540
130.0591
133.0609
39.1093
51.0715
144.0992
202.1678
75.0681
102.1049
41.1401
54.0604
161.1371
172.1635
187.1790
78.0760
146.1003
38.1020
66.0116
117.0947
199.9803
89.0535
101.0882
127.1179
134.1152
162.1429
173.1634
203.1629
34.1127
193.1301
31
41
51
61
71
81
91
101
111
121
131
141
151
161
171
181
191
201
211
m/z
O
N
O



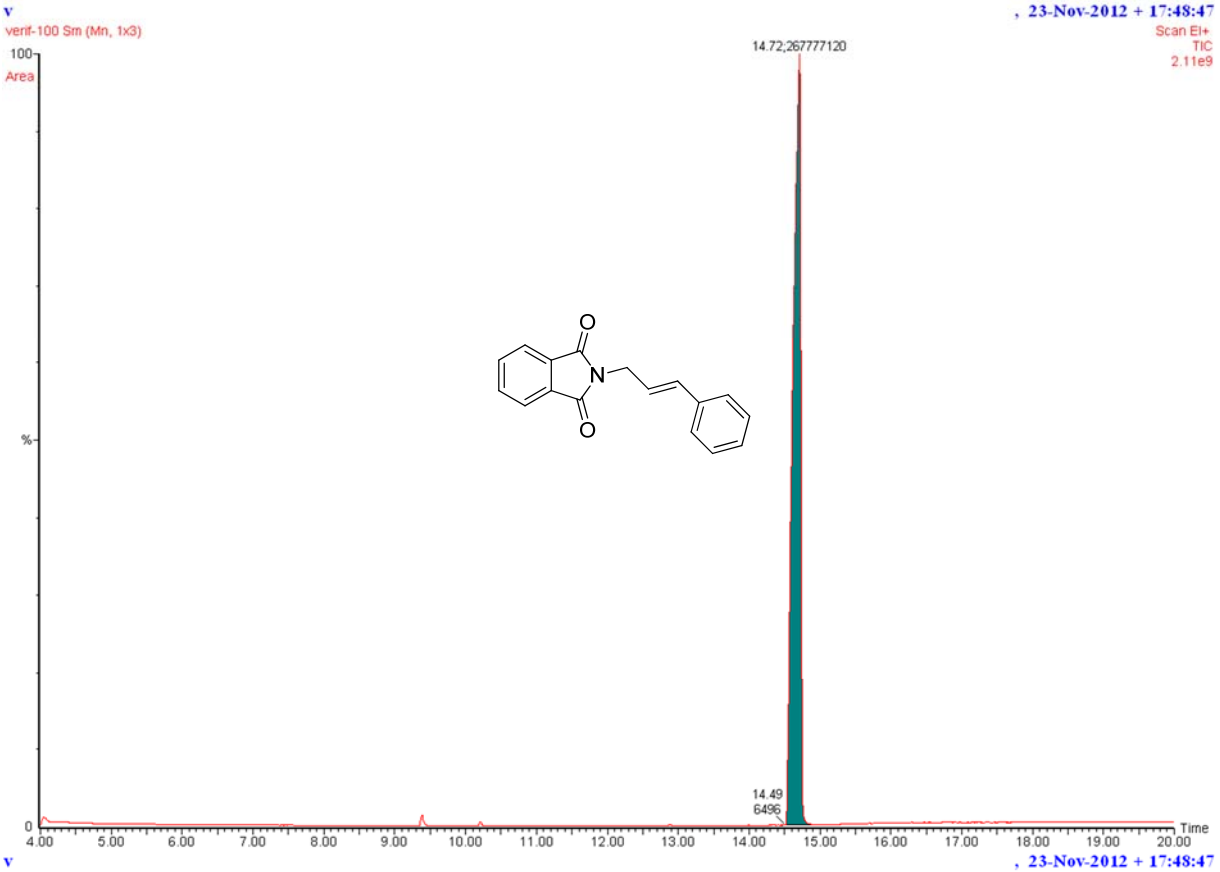
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a19**

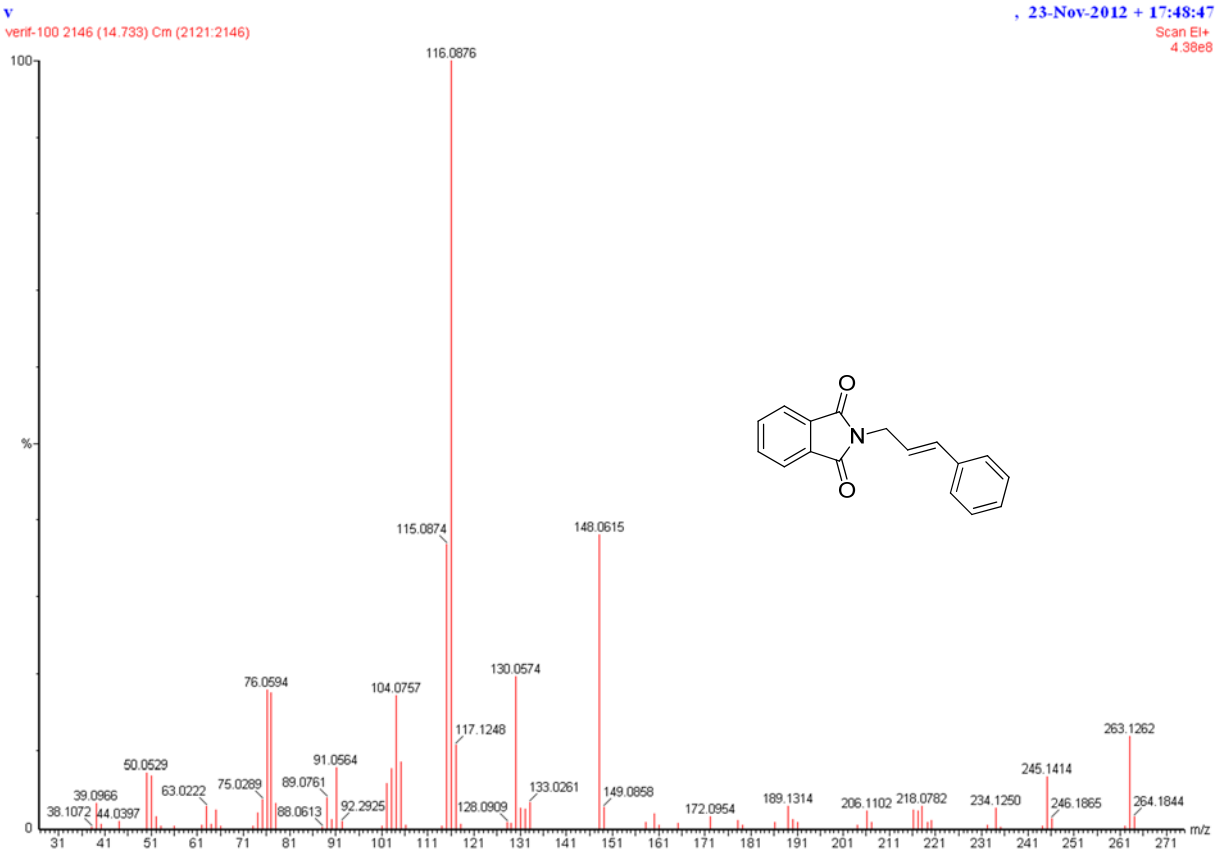

1
fc-0000 Sm (Mn, 1x3)
, 22-Nov-2012 + 11:52:40
Scan EI+
TIC
2.24e10
100
Area
%
0
12.16;1909639808
11.92
104787
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

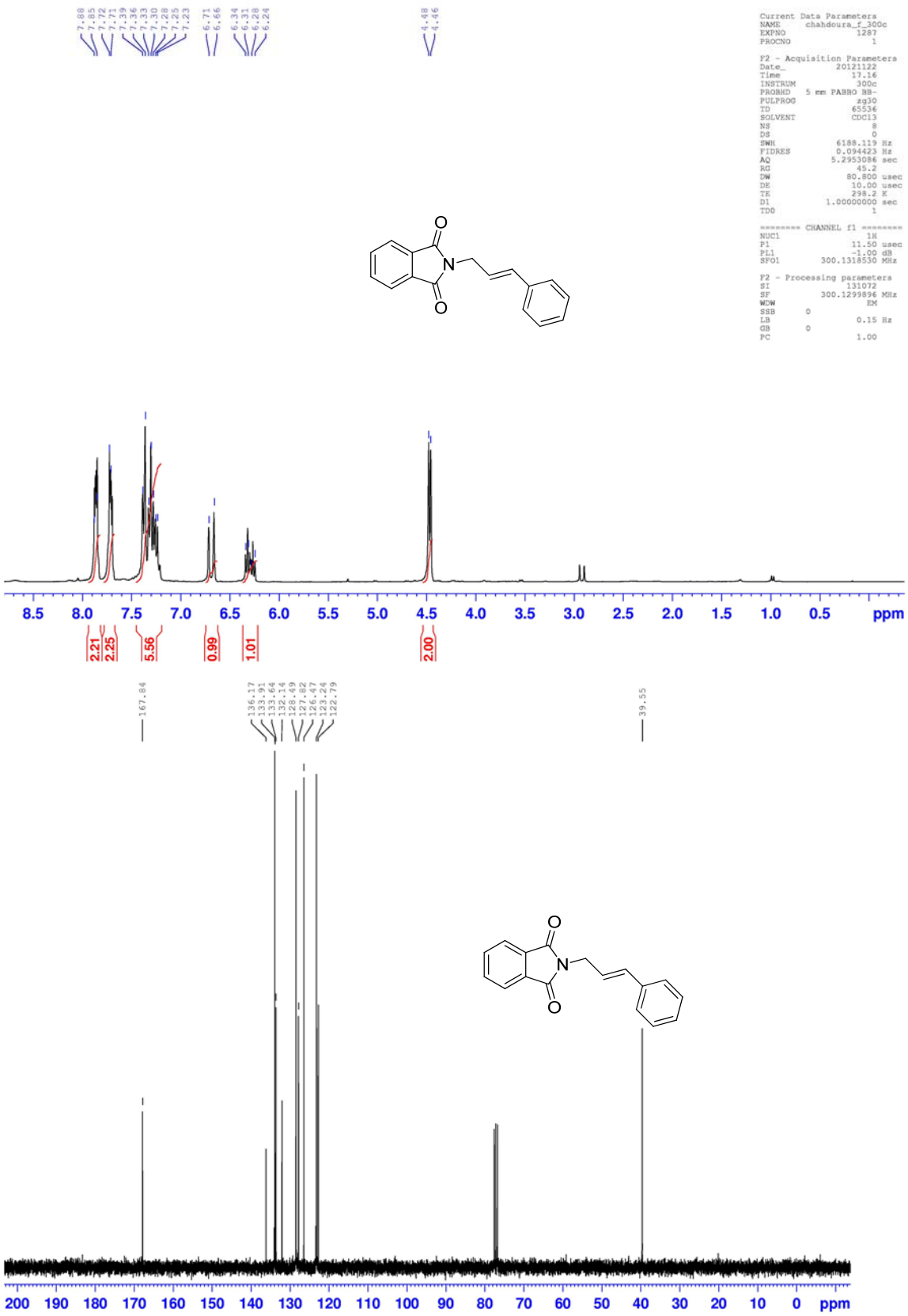

1
fc-0000 1632 (12.162) Cm (1615:1632)
, 22-Nov-2012 + 11:52:40
Scan EI+
1.51e9
100
%
0
160.0908
200.1212
215.1273
76.0594
130.0334
68.0620
77.0500
182.1173
41.1158
104.0883
105.0779
50.0612
39.1084
102.0810
148.0677
133.0337
196.1156
201.1516
161.1516
216.1559
82.0732
128.0847
172.1669
183.1790
66.0102
42.1011
38.1022
90.0532
93.9426
117.0867
144.1137
154.1094
170.1275
173.1633
187.1403
213.9352
217.1810
37
47
57
67
77
87
97
107
117
127
137
147
157
167
177
187
197
207
217
m/z



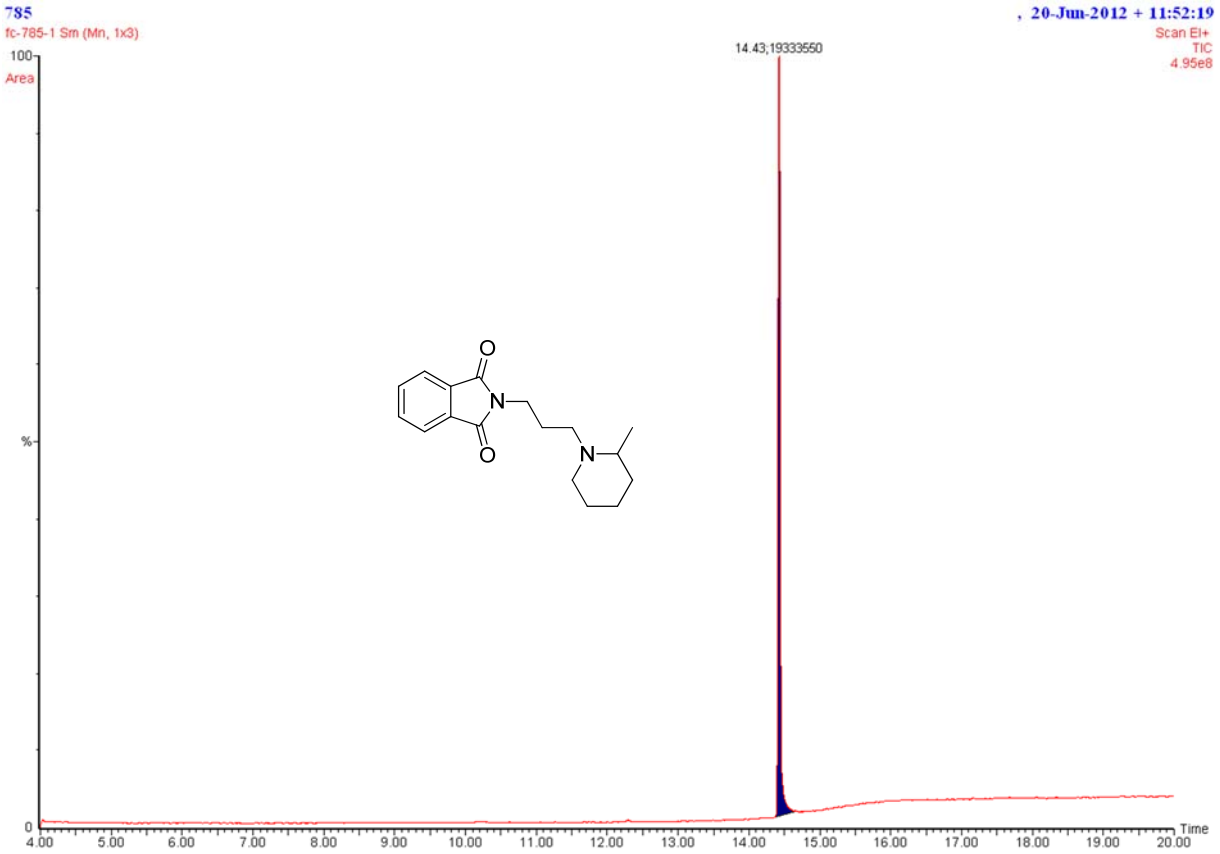
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a20**

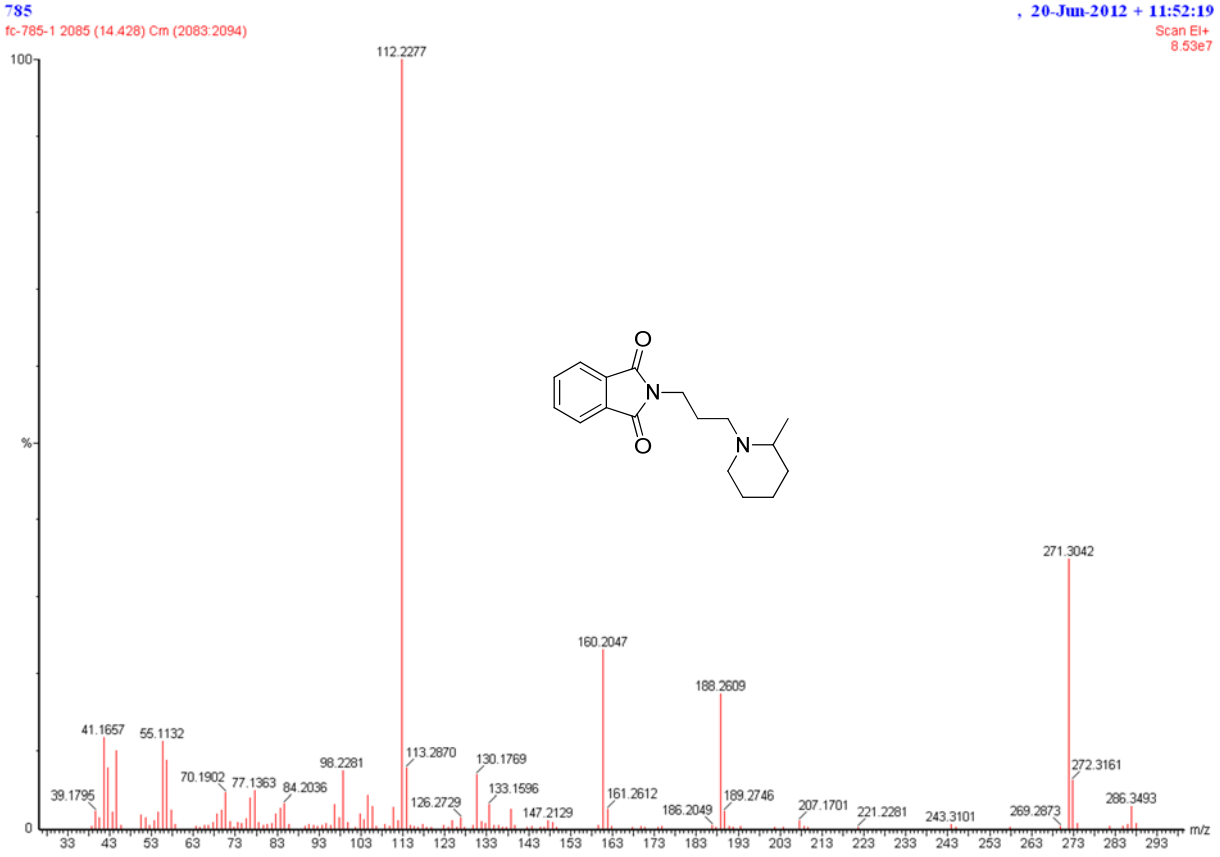

v
verif-100 Sm (Mn, 1x3)
, 23-Nov-2012 + 17:48:47
Scan EI+
TIC
2.11e9
100
Area
%
0
14.72;267777120
14.49
6496
O
N
O
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

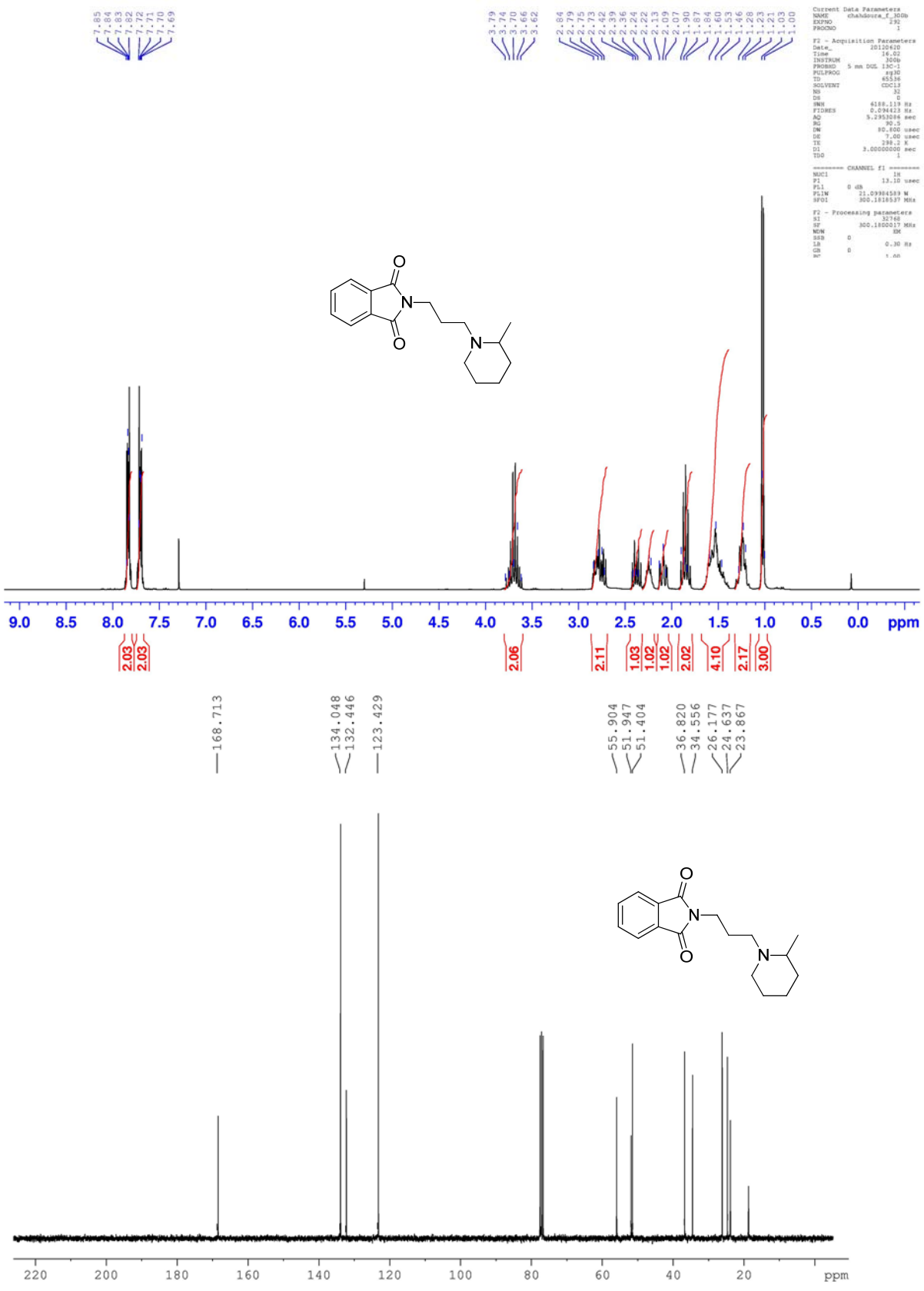

v
verif-100 2146 (14.733) Cm (2121:2146)
, 23-Nov-2012 + 17:48:47
Scan EI+
4.38e8
100
%
0
116.0876
115.0874
148.0615
130.0574
76.0594
104.0757
117.1248
91.0564
50.0529
89.0761
133.0261
149.0858
263.1262
245.1414
63.0222
75.0289
92.2925
128.0909
172.0954
189.1314
206.1102
218.0782
234.1250
246.1865
264.1844
39.0966
38.1072
44.0397
88.0613
O
N
O
31
41
51
61
71
81
91
101
111
121
131
141
151
161
171
181
191
201
211
221
231
241
251
261
271
m/z



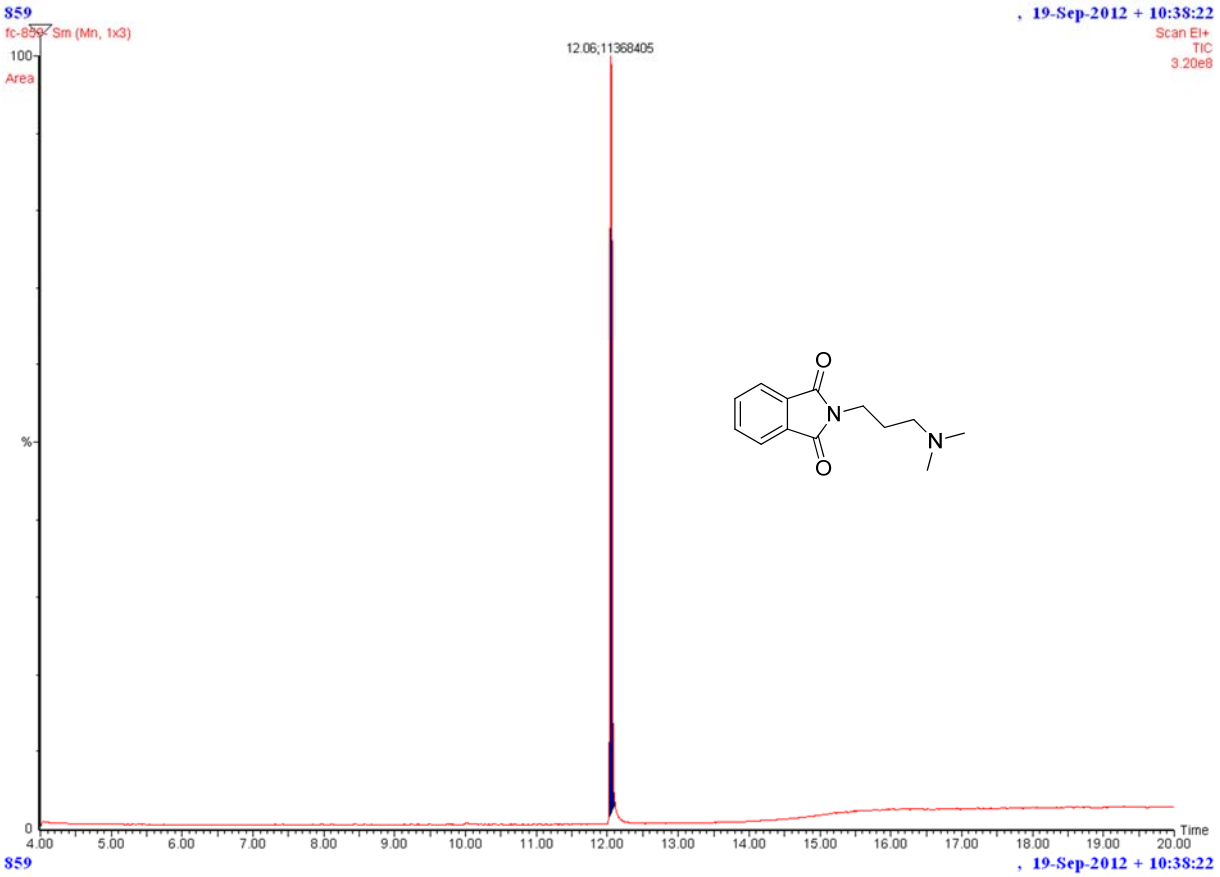
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a21**

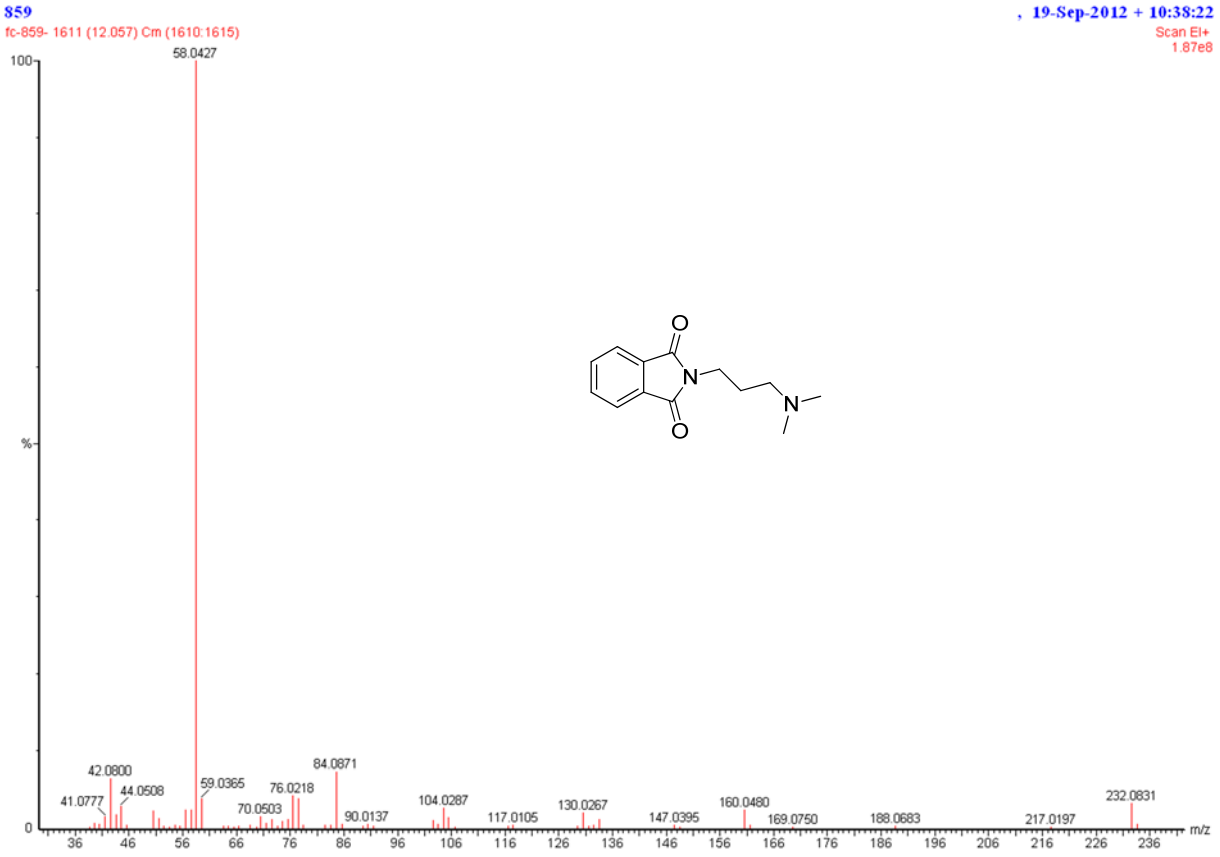

785
fc-785-1 Sm (Mn, 1x3)
, 20-Jun-2012 + 11:52:19
Scan EI+
TIC
4.95e8
100
Area
%
0
14.43;19333550
O
N
O
N
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

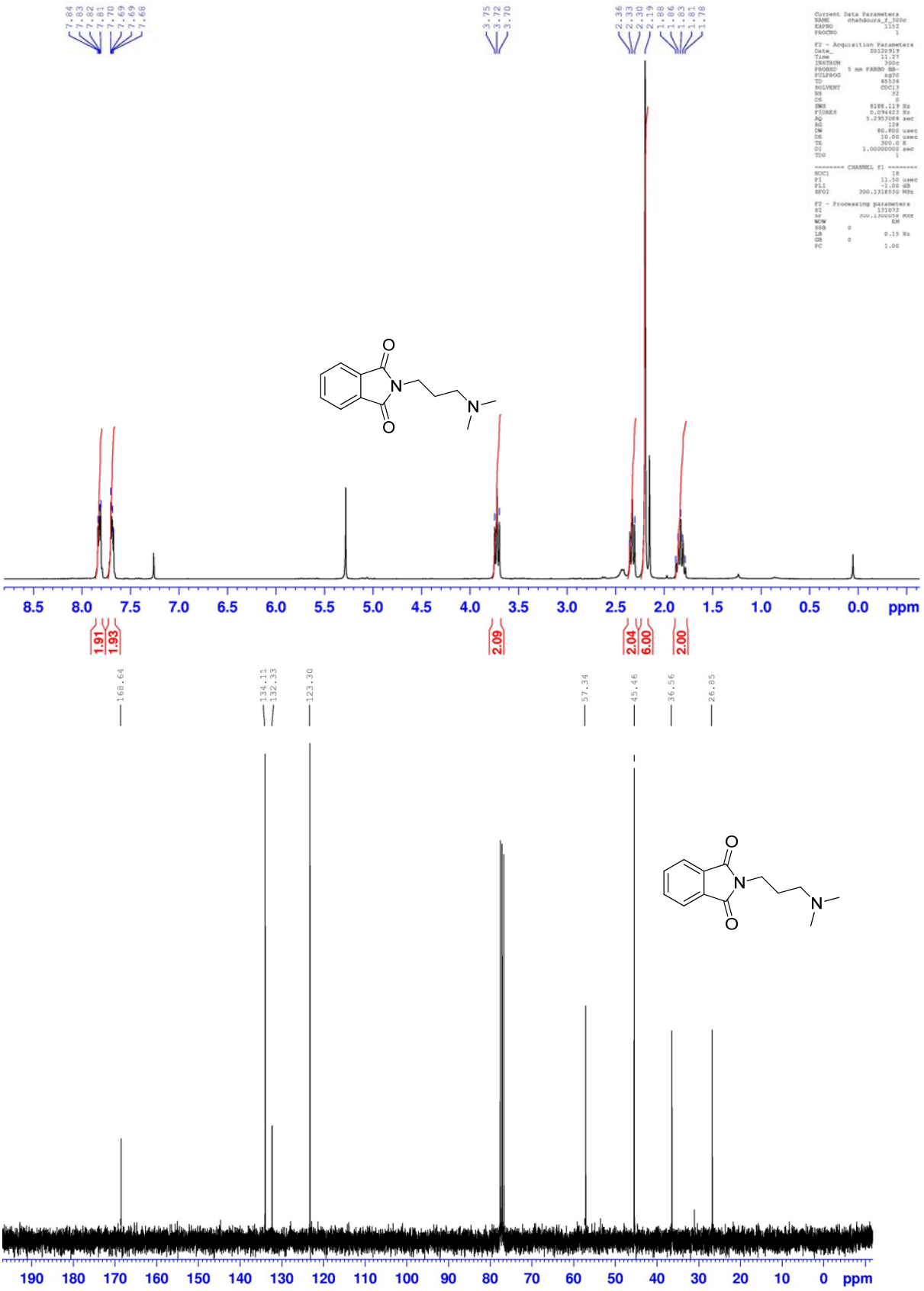

785
fc-785-1 2085 (14.428) Cm (2083:2094)
, 20-Jun-2012 + 11:52:19
Scan EI+
8.53e7
100
%
0
112.2277
271.3042
160.2047
188.2609
41.1657
55.1132
70.1902
77.1363
84.2036
98.2281
113.2870
130.1769
133.1596
39.1795
126.2729
147.2129
161.2612
186.2049
189.2746
207.1701
221.2281
243.3101
269.2873
272.3161
286.3493
O
N
O
N
33
43
53
63
73
83
93
103
113
123
133
143
153
163
173
183
193
203
213
223
233
243
253
263
273
283
293
m/z



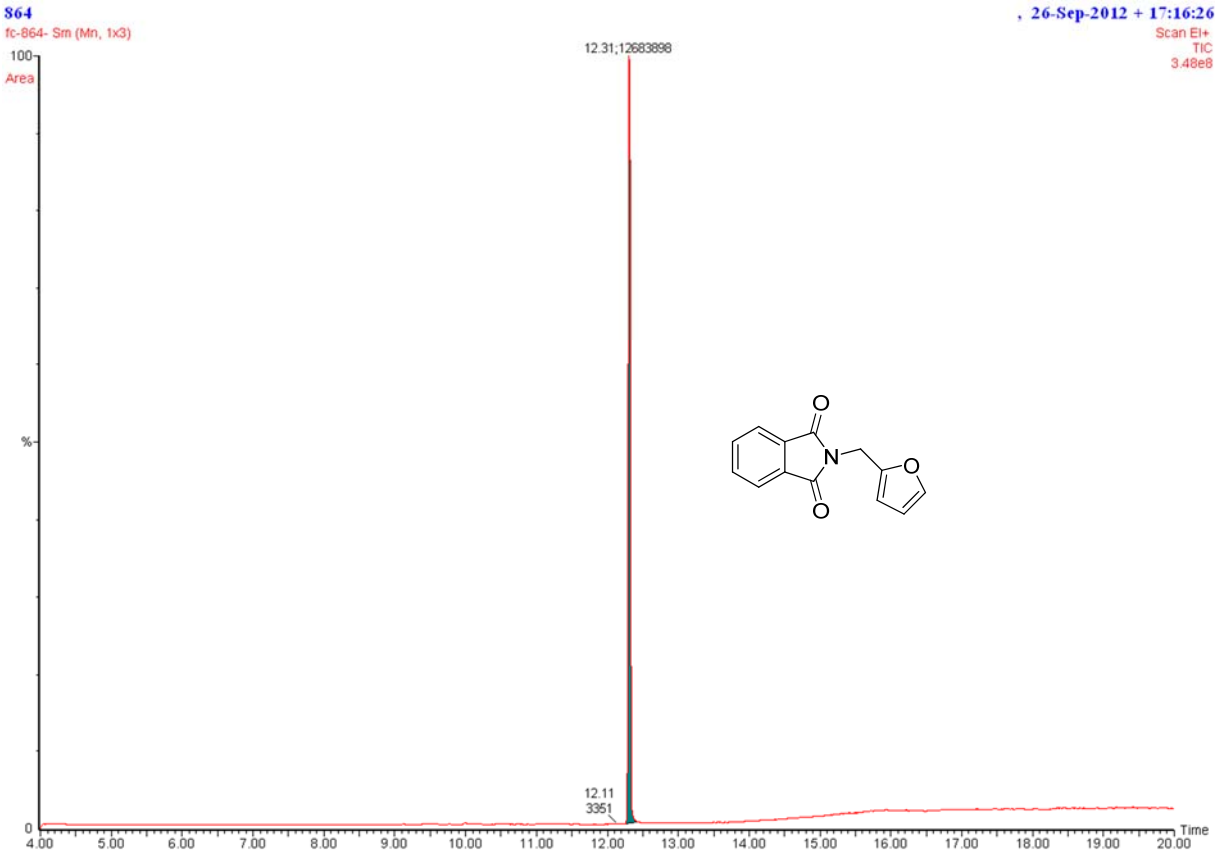
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a22**

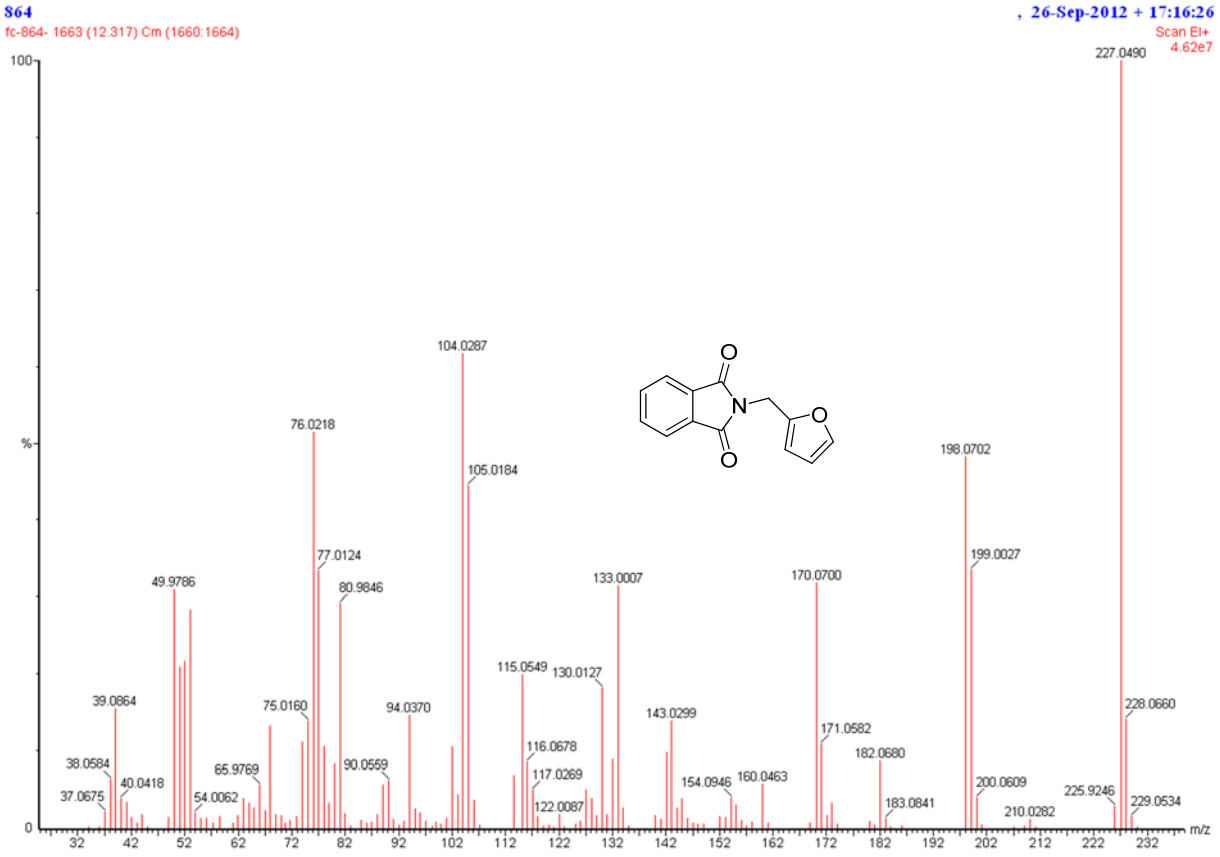

859
fc-859 Sm (Mn, 1x3)
, 19-Sep-2012 + 10:38:22
Scan EI+
TIC
3.20e8
100
Area
%
0
12.06;11368405
O
N
O
N
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

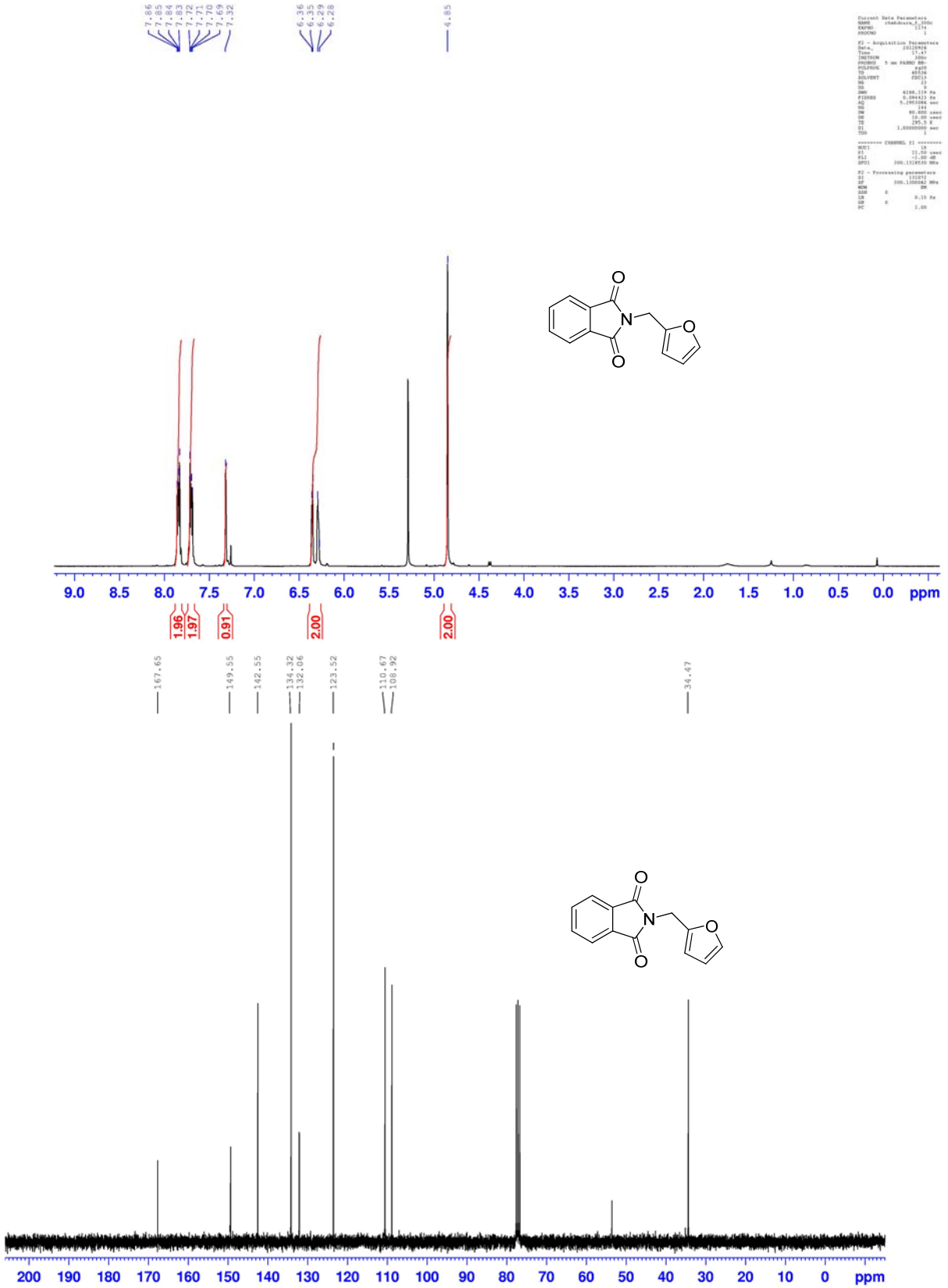

859
fc-859- 1611 (12.057) Cm (1610:1615)
, 19-Sep-2012 + 10:38:22
Scan EI+
1.87e8
100
%
0
58.0427
42.0800
44.0508
41.0777
59.0365
70.0503
76.0218
84.0871
90.0137
104.0287
117.0105
130.0267
147.0395
160.0480
169.0750
188.0683
217.0197
232.0831
O
N
O
N
36
46
56
66
76
86
96
106
116
126
136
146
156
166
176
186
196
206
216
226
236
m/z



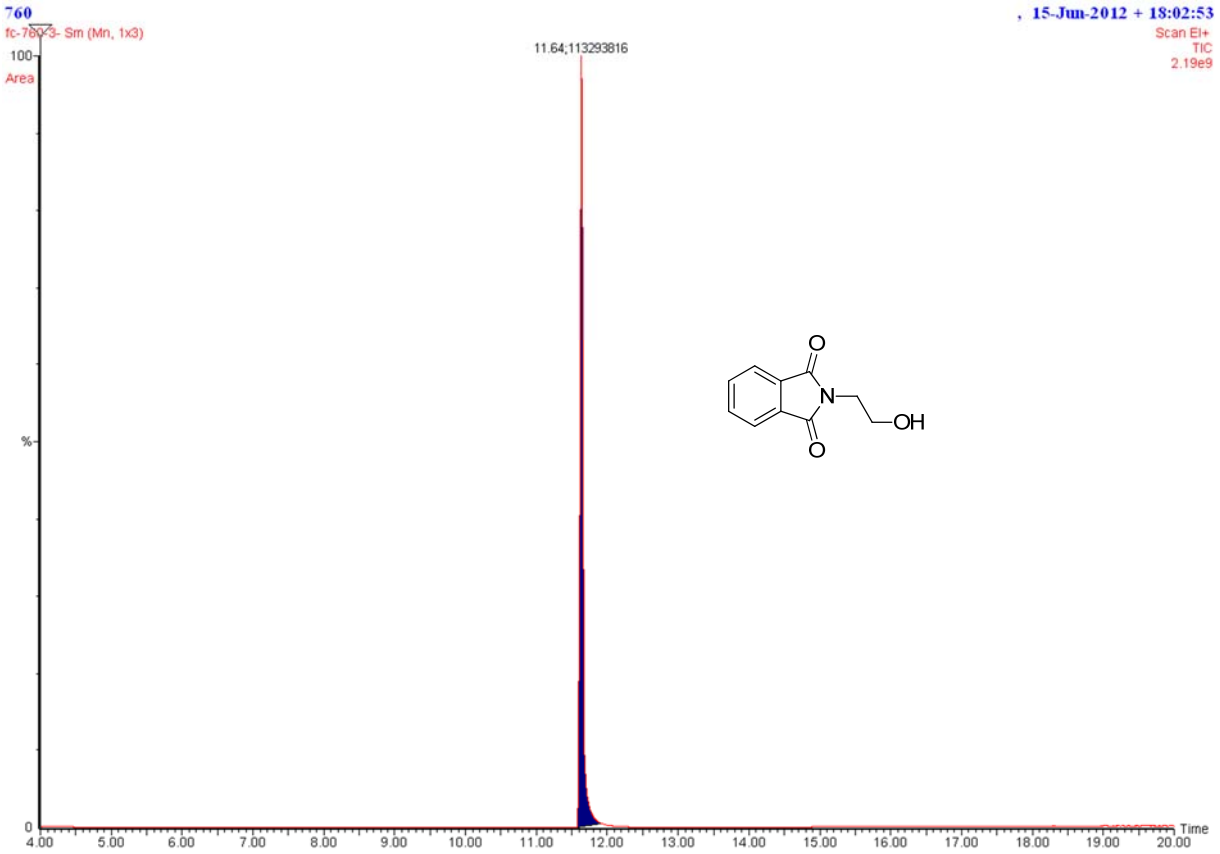
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a23**

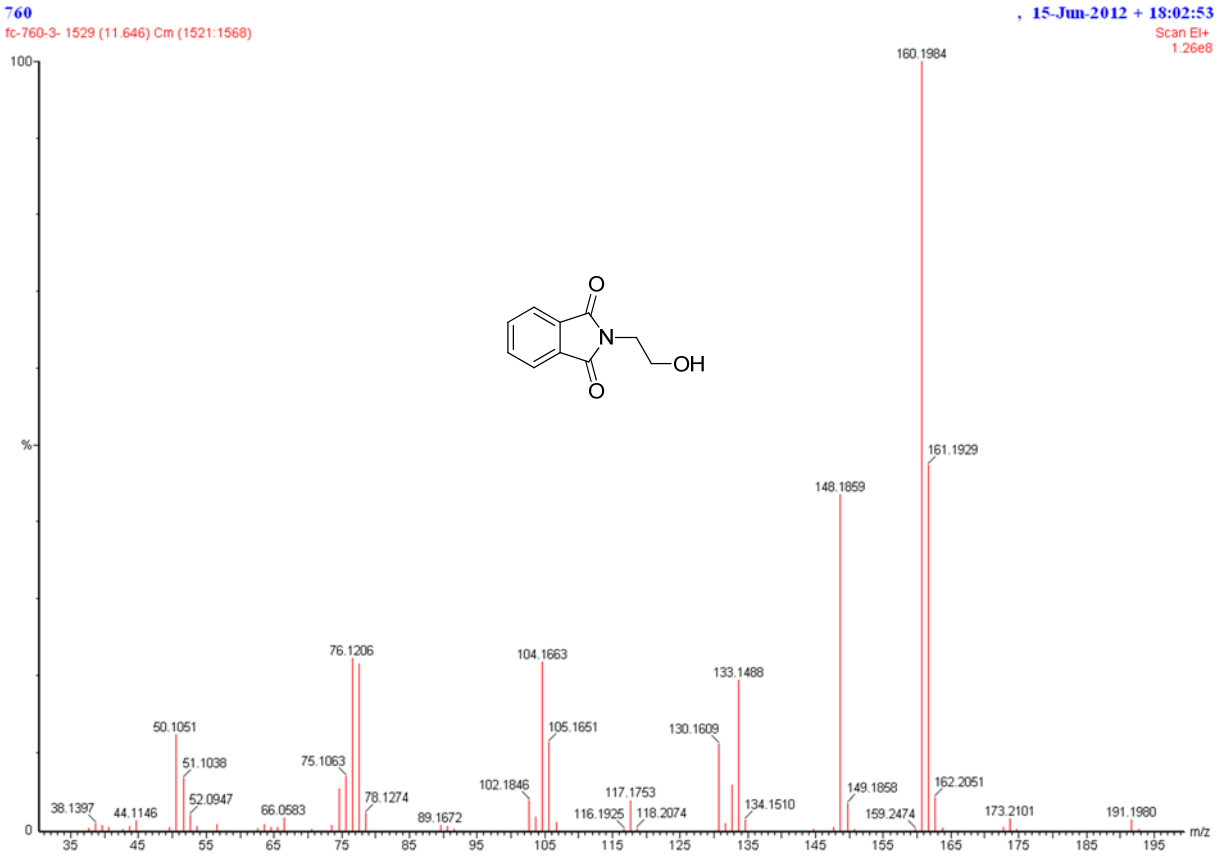

864
fc-864- Sm (Mn, 1x3)
, 26-Sep-2012 + 17:16:26
Scan EI+
TIC
3.48e8
100
Area
%
0
12.31;12683898
12.11
3351
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N
O
O

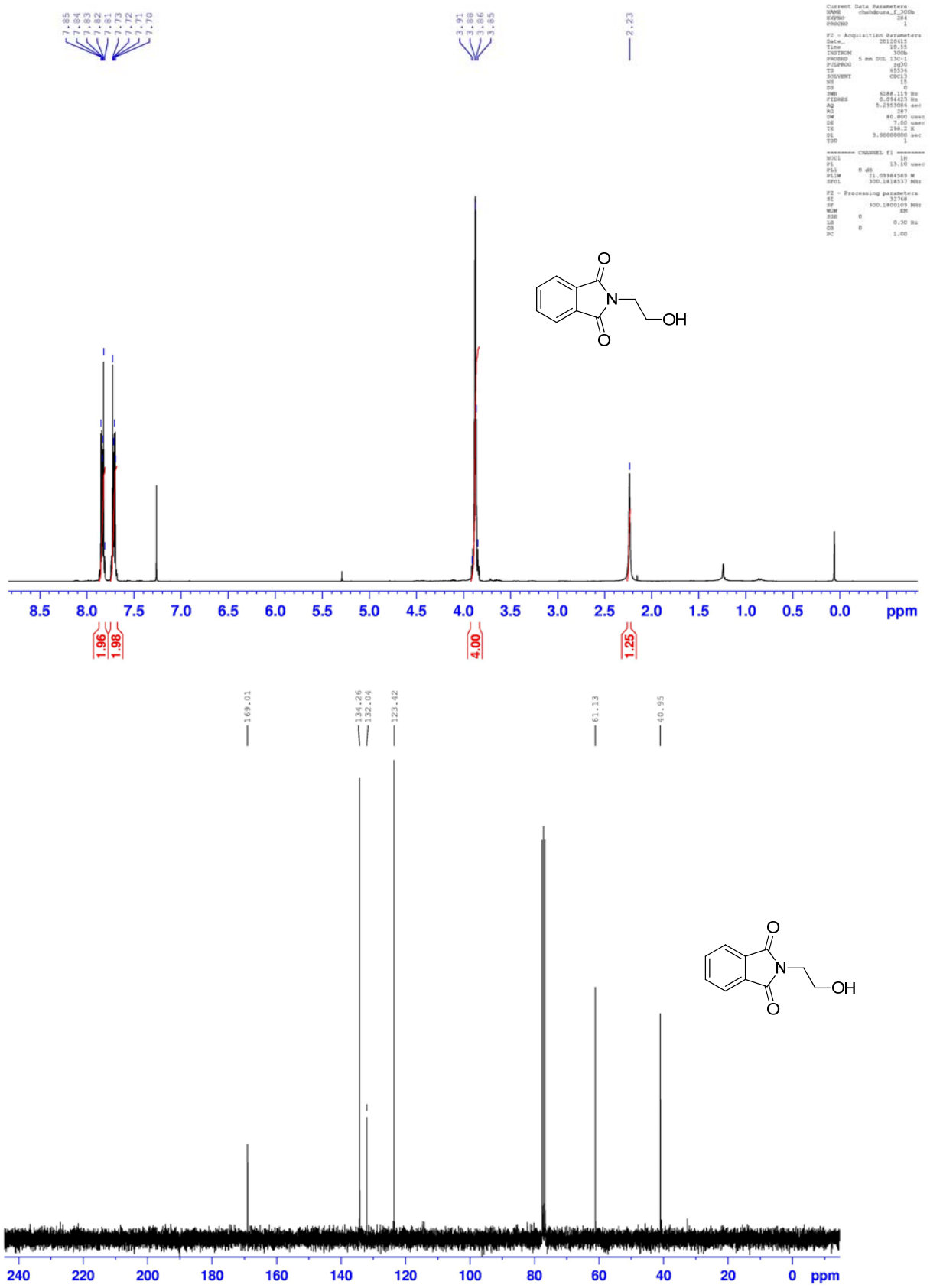

864
fc-864- 1663 (12.317) Cm (1660:1664)
, 26-Sep-2012 + 17:16:26
Scan EI+
4.62e7
100
%
0
227.0490
104.0287
76.0218
105.0184
198.0702
77.0124
199.0027
49.9786
80.9846
133.0007
170.0700
115.0549
130.0127
39.0864
75.0160
94.0370
143.0299
228.0660
171.0582
116.0678
182.0680
38.0584
65.9769
90.0559
117.0269
154.0946
160.0463
40.0418
37.0675
54.0062
122.0087
183.0841
200.0609
225.9246
229.0534
210.0282
32
42
52
62
72
82
92
102
112
122
132
142
152
162
172
182
192
202
212
222
232
m/z
O
N
O
O



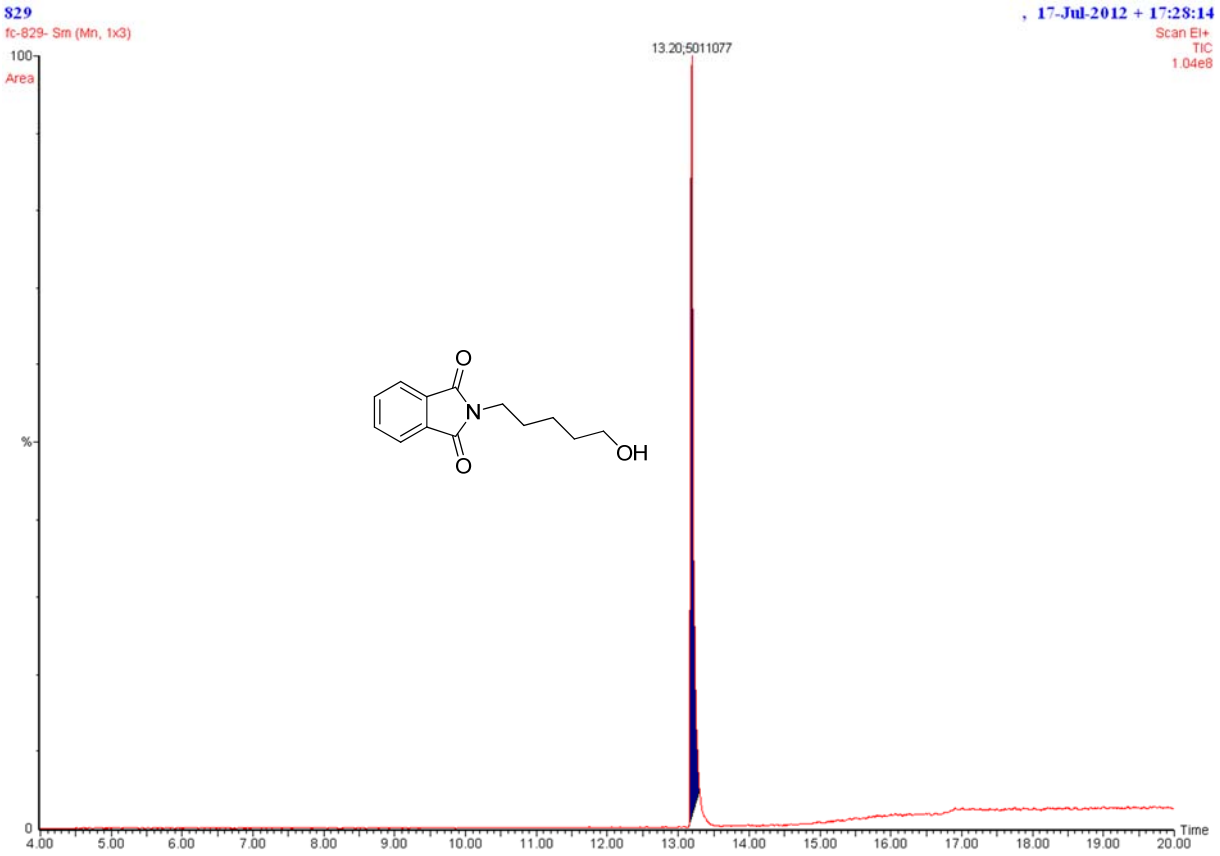
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a24**

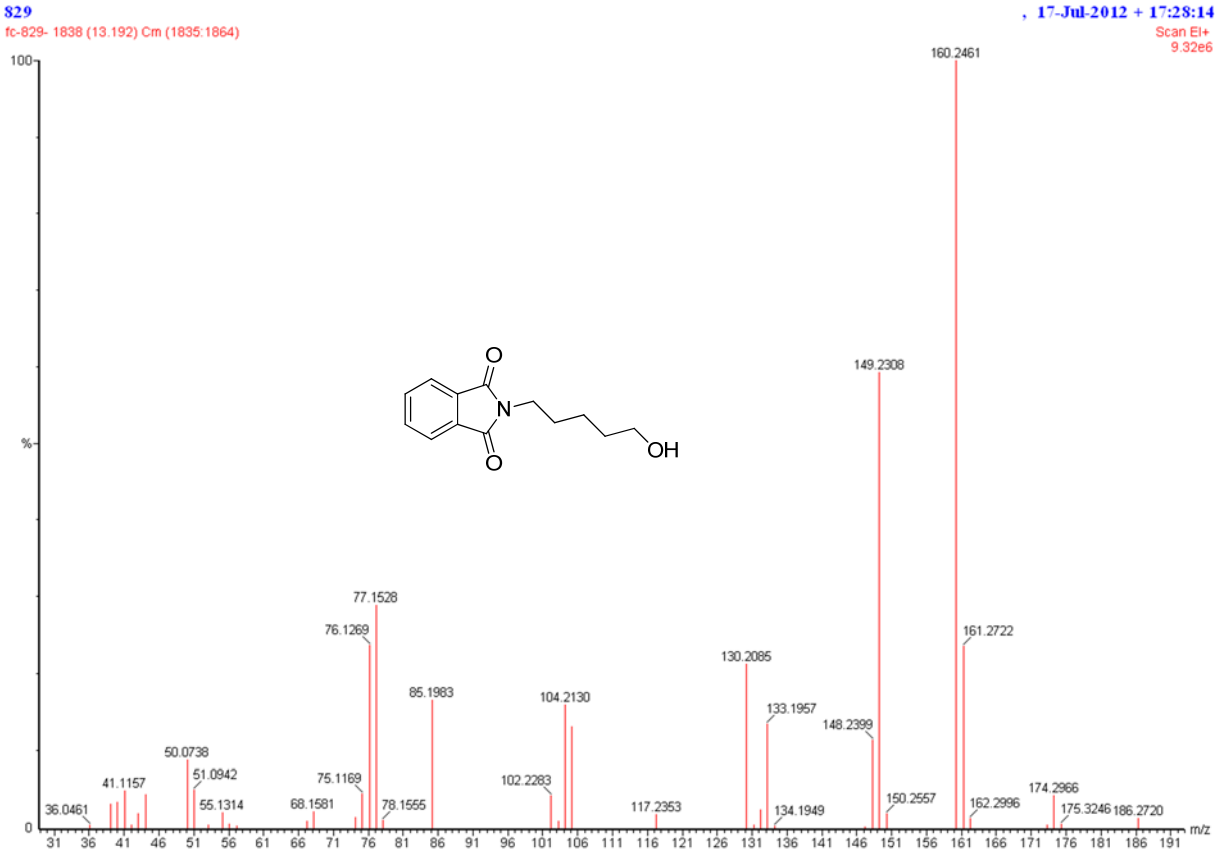

760
fc-760-3- Sm (Mn, 1x3)
, 15-Jun-2012 + 18:02:53
Scan EI+
TIC
2.19e9
100
Area
%
0
11.64;113293816
O
N
OH
O
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

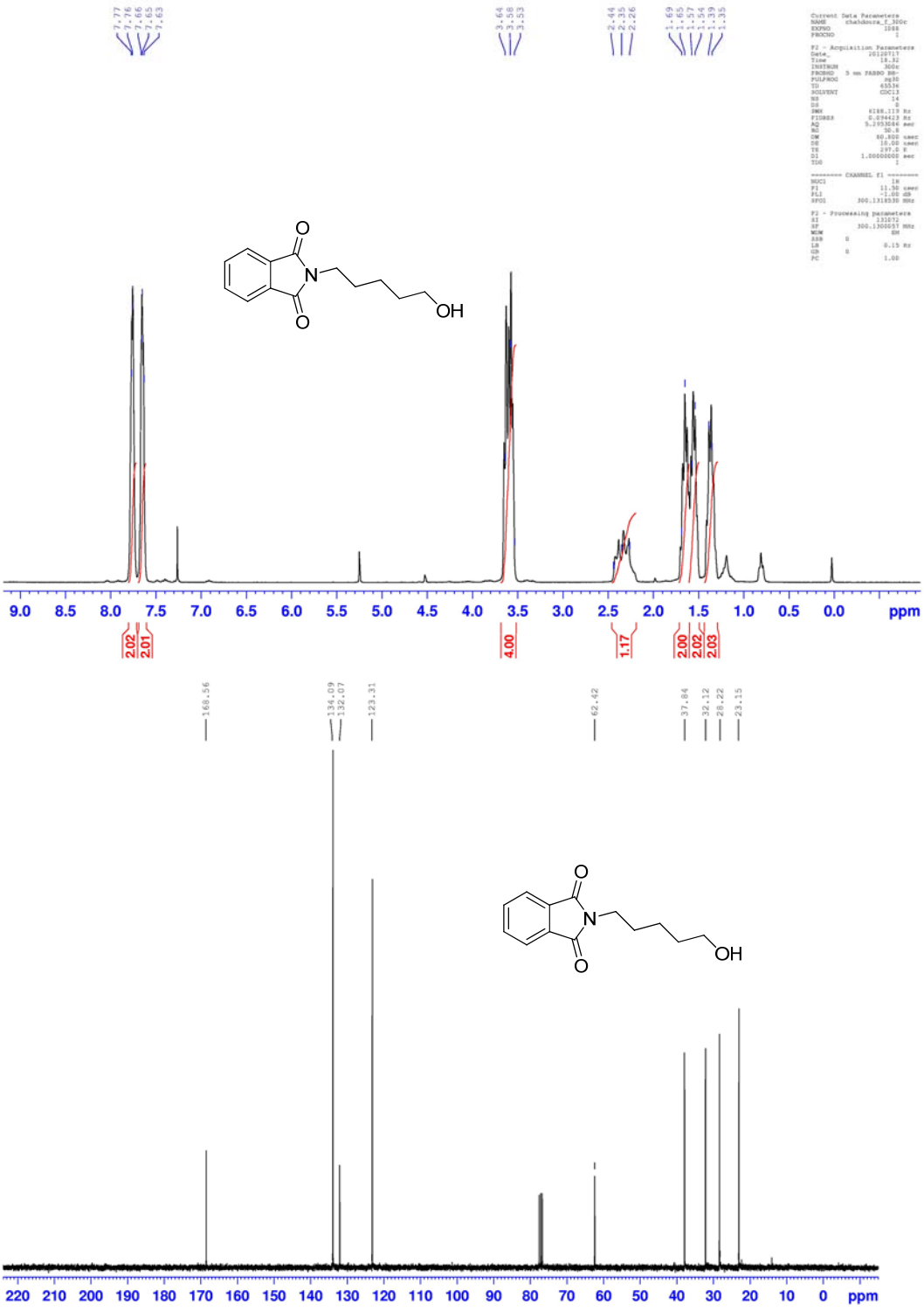

760
fc-760-3- 1529 (11.646) Cm (1521:1568)
, 15-Jun-2012 + 18:02:53
Scan EI+
1.26e8
100
%
0
O
N
OH
O
160.1984
161.1929
148.1859
76.1206
104.1663
133.1488
50.1051
105.1651
130.1609
51.1038
75.1063
102.1846
117.1753
149.1858
162.2051
52.0947
78.1274
116.1925
118.2074
134.1510
159.2474
173.2101
191.1980
38.1397
44.1146
66.0583
89.1672
35
45
55
65
75
85
95
105
115
125
135
145
155
165
175
185
195
m/z



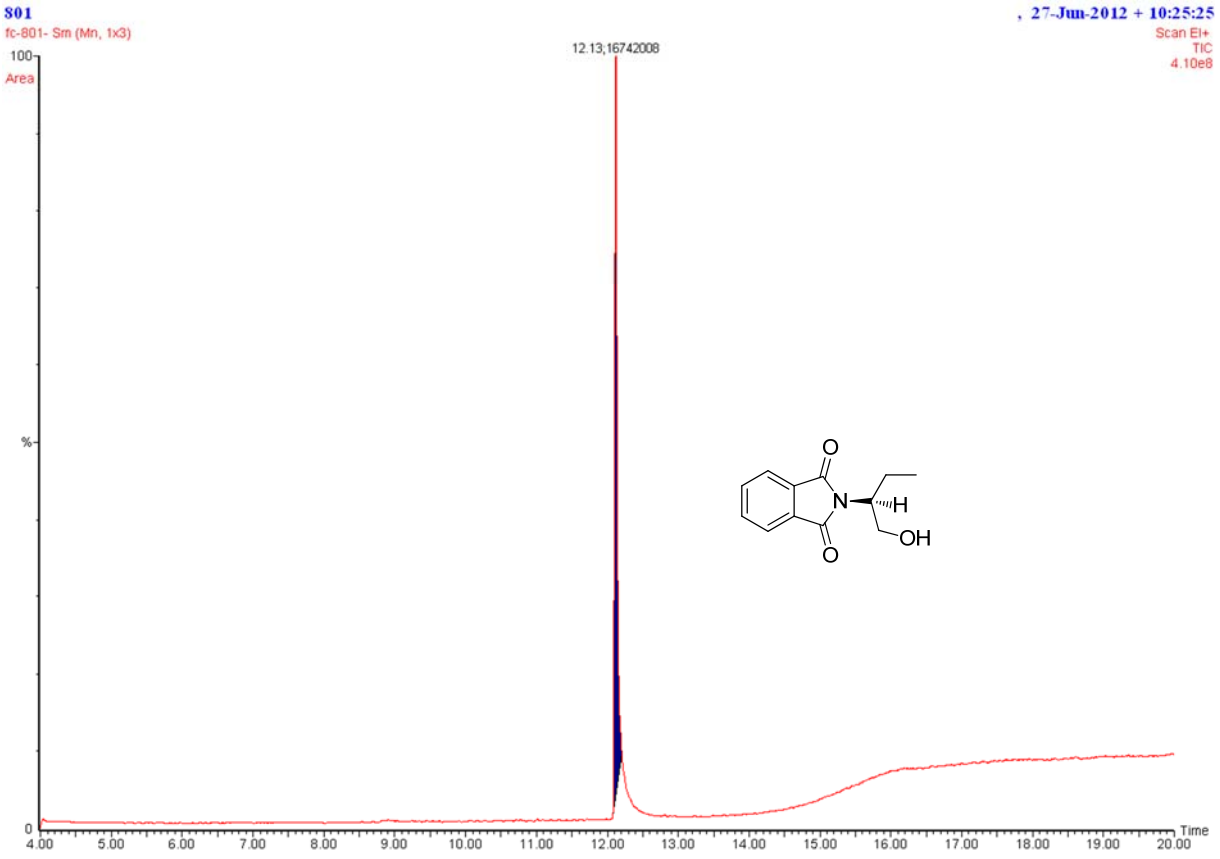
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a25**

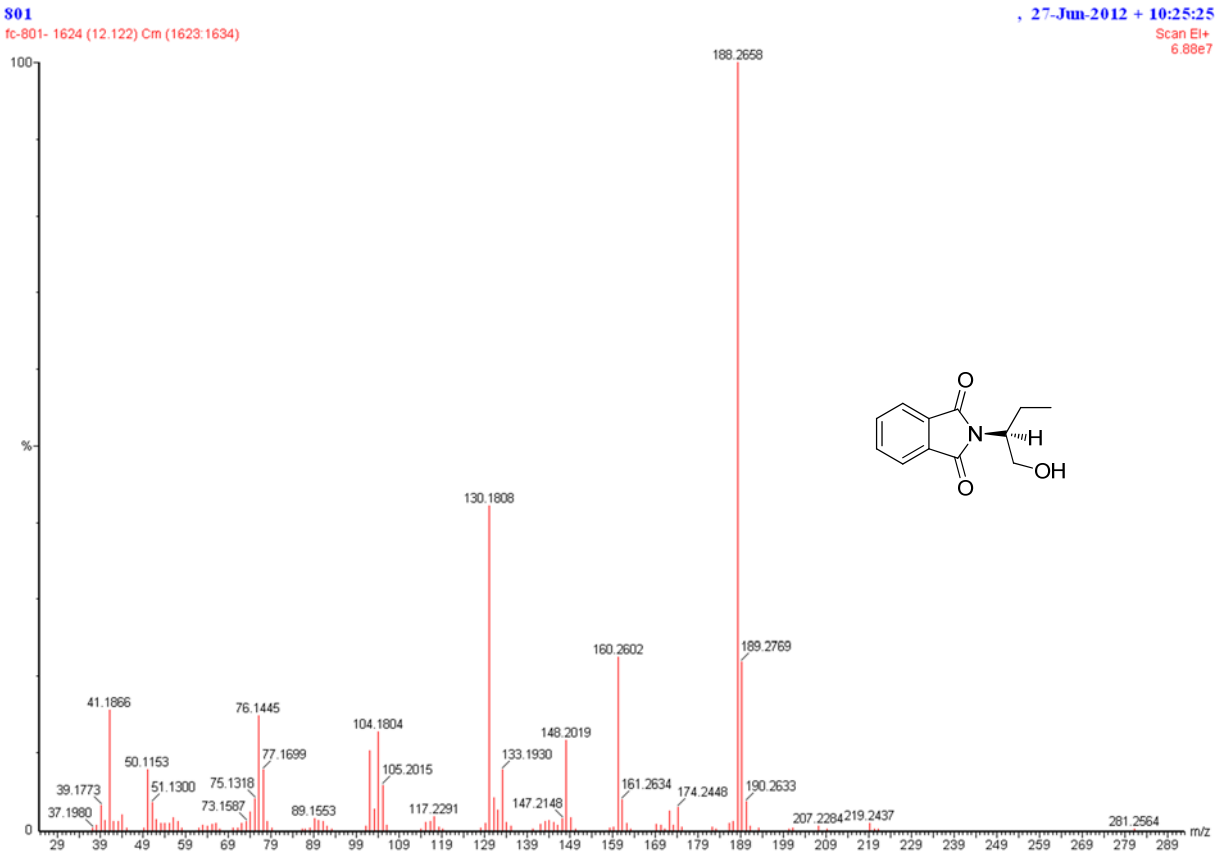

829
fc-829- Sm (Mn, 1x3)
, 17-Jul-2012 + 17:28:14
Scan EI+
TIC
1.04e8
Area
13.20;5011077
O
N
OH
O
%
Time

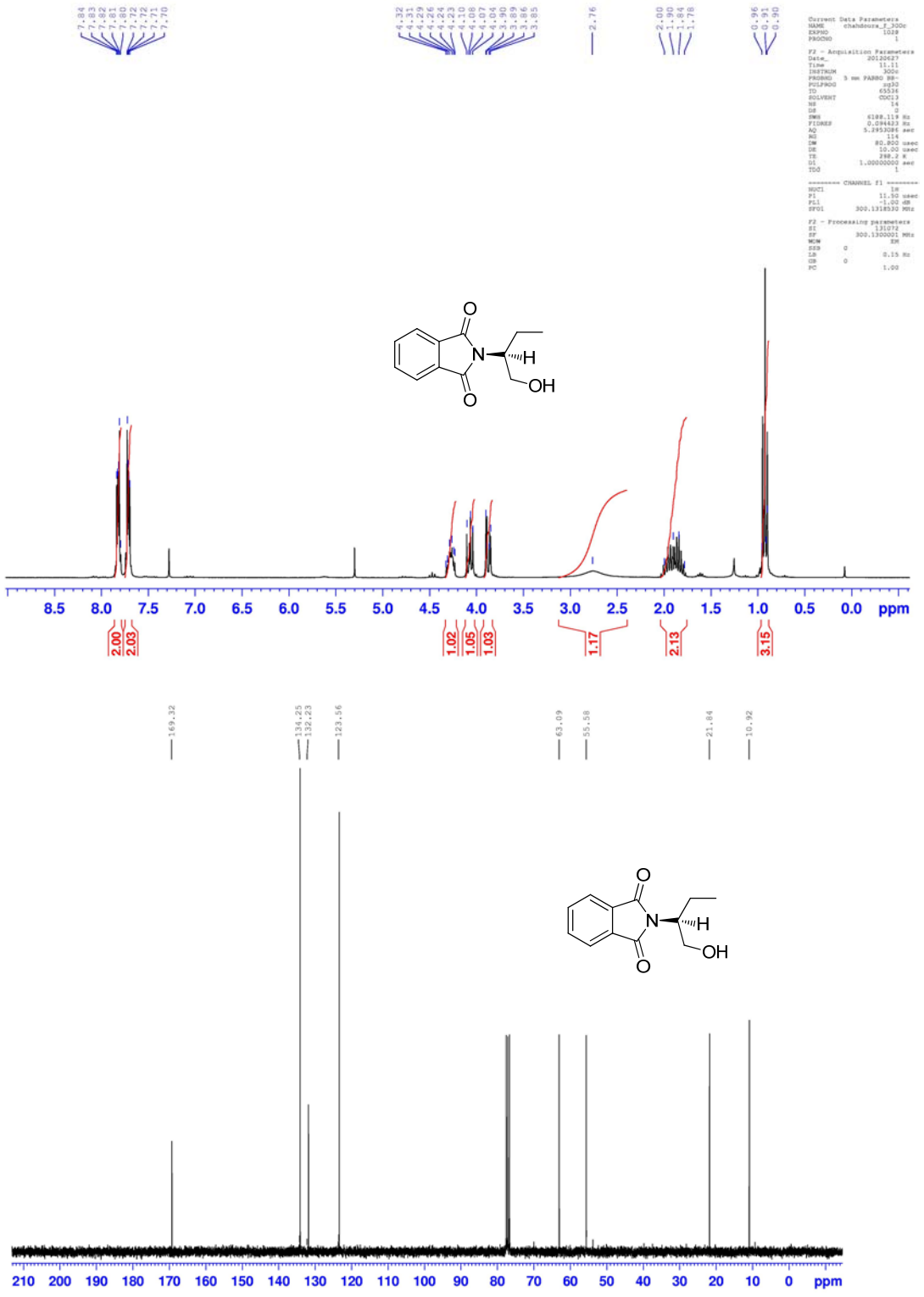

829
fc-829- 1838 (13.192) Cm (1835:1864)
, 17-Jul-2012 + 17:28:14
Scan EI+
9.32e6
160.2461
149.2308
O
N
OH
O
77.1528
76.1269
161.2722
130.2085
85.1983
104.2130
133.1957
148.2399
50.0738
51.0942
41.1157
75.1169
102.2283
174.2966
36.0461
55.1314
68.1581
78.1555
117.2353
134.1949
150.2557
162.2996
175.3246
186.2720
%
m/z



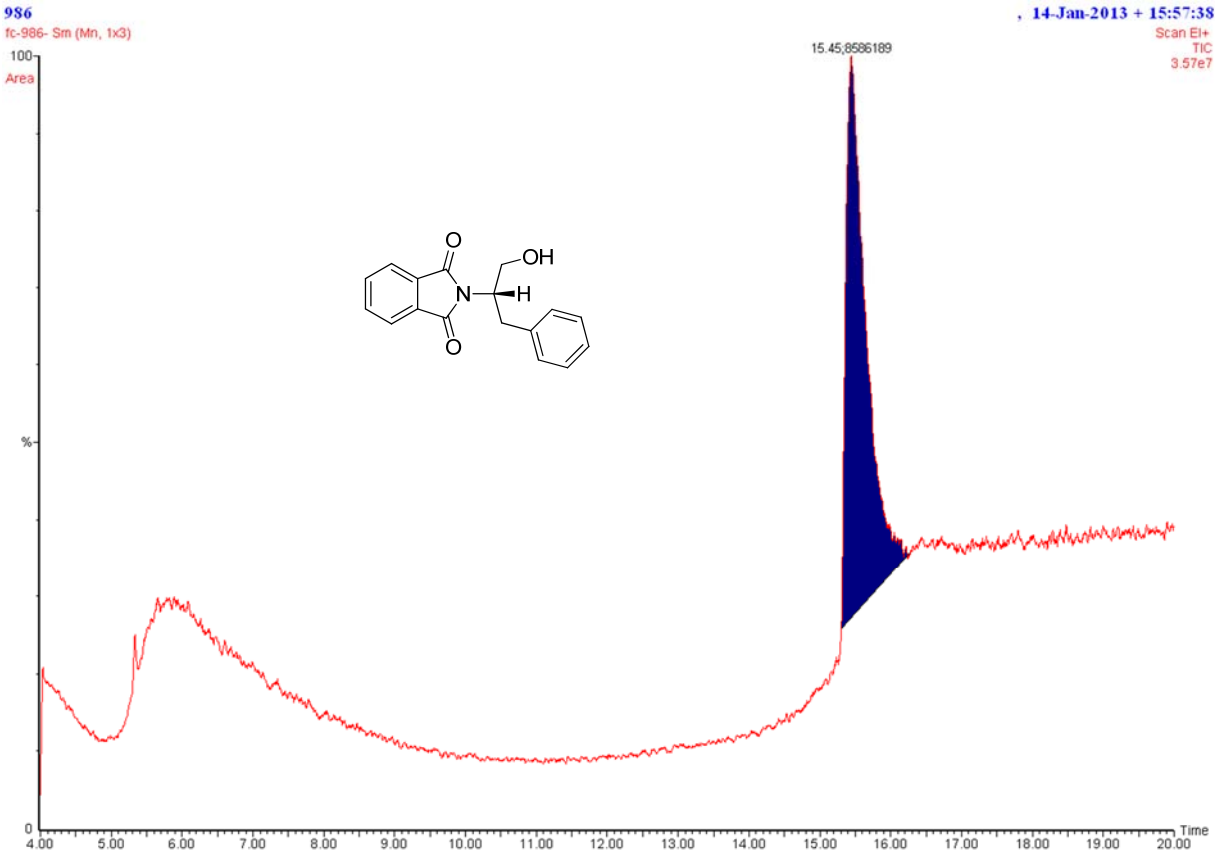
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a26**

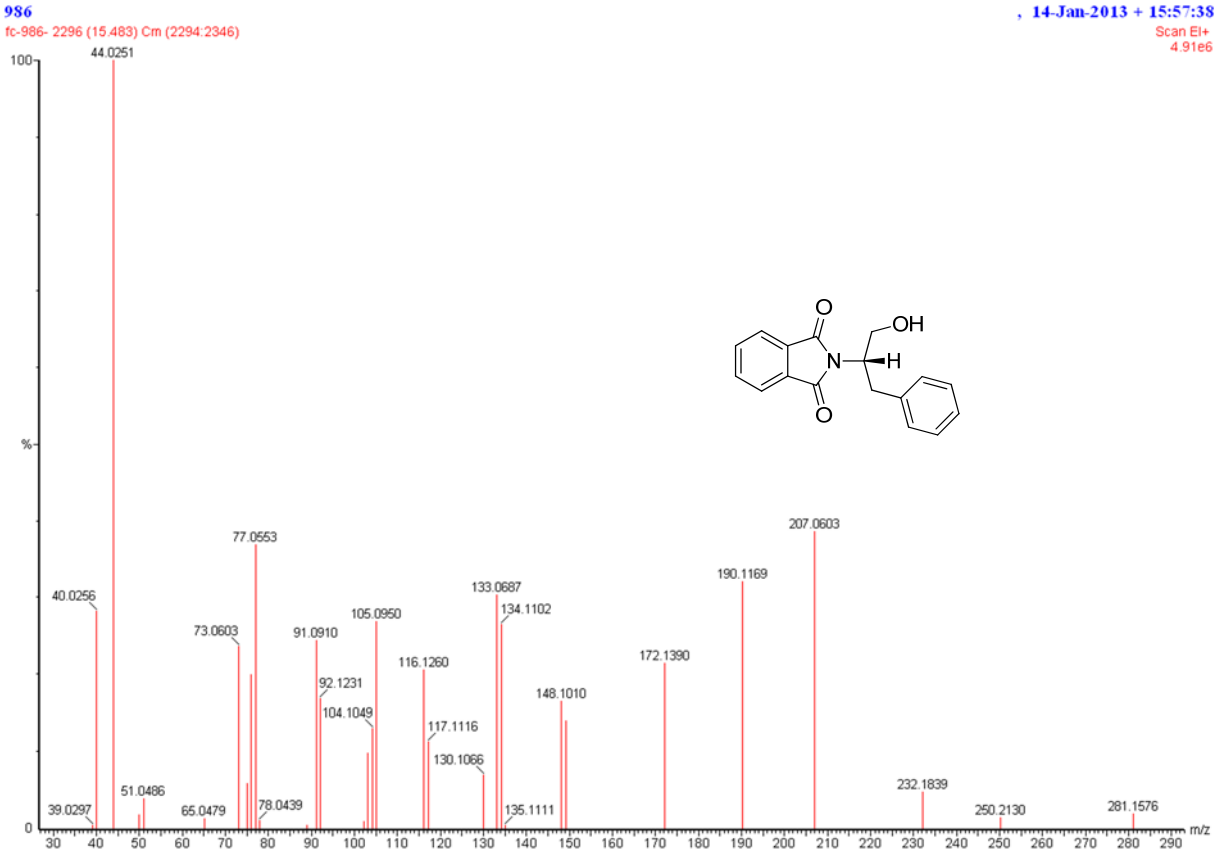

801
fc-801- Sm (Mn, 1x3)
, 27-Jun-2012 + 10:25:25
Scan EI+
TIC
4.10e8
100
Area
%
0
12.13;16742008
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N
H
OH
O

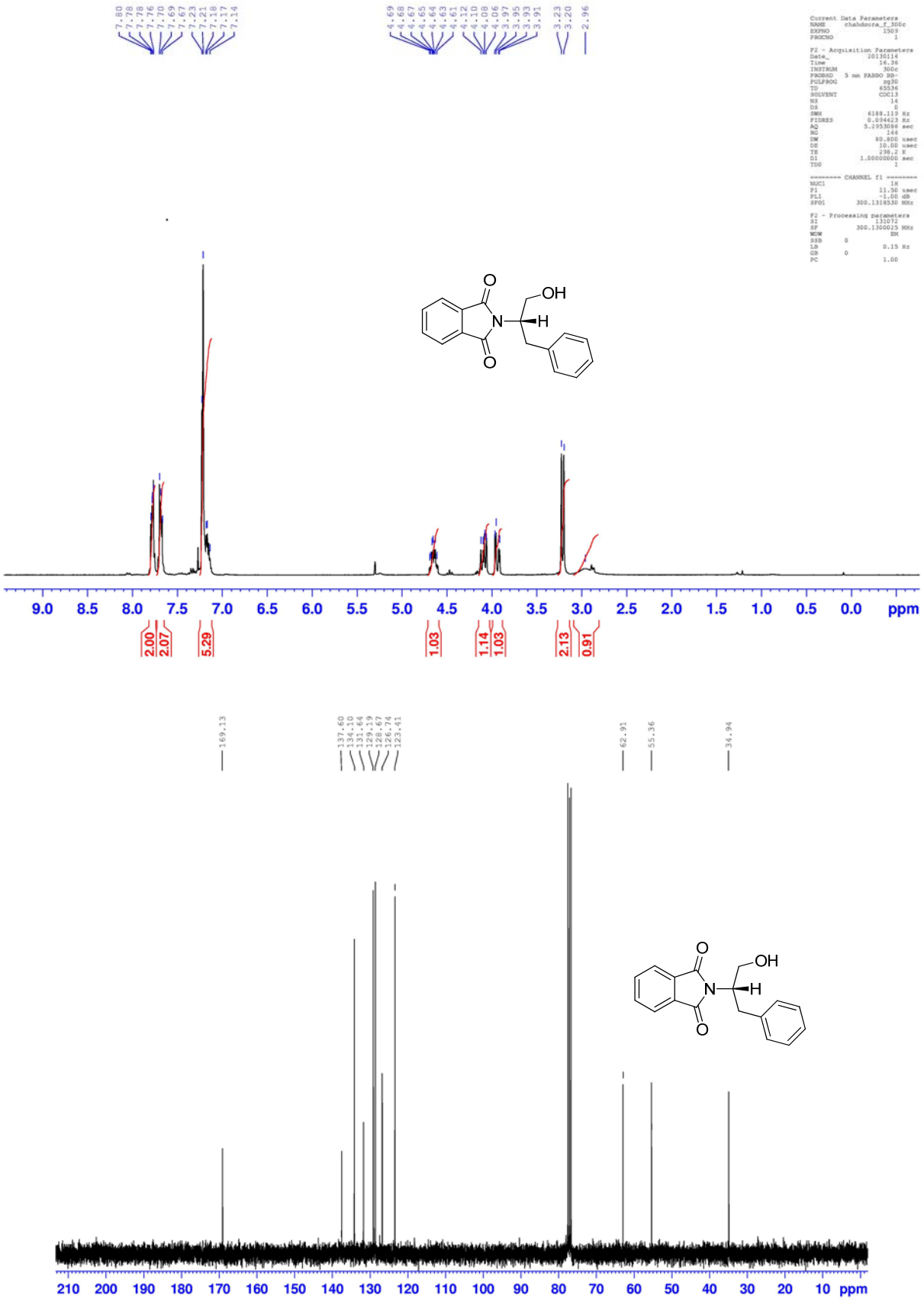

801
fc-801- 1624 (12.122) Cm (1623:1634)
, 27-Jun-2012 + 10:25:25
Scan EI+
6.88e7
100
%
0
188.2658
130.1808
160.2602
189.2769
41.1866
76.1445
104.1804
148.2019
50.1153
77.1699
133.1930
105.2015
39.1773
51.1300
75.1318
161.2634
174.2448
190.2633
37.1980
73.1587
89.1553
117.2291
147.2148
207.2284
219.2437
281.2564
29
39
49
59
69
79
89
99
109
119
129
139
149
159
169
179
189
199
209
219
229
239
249
259
269
279
289
m/z
O
N
H
OH
O



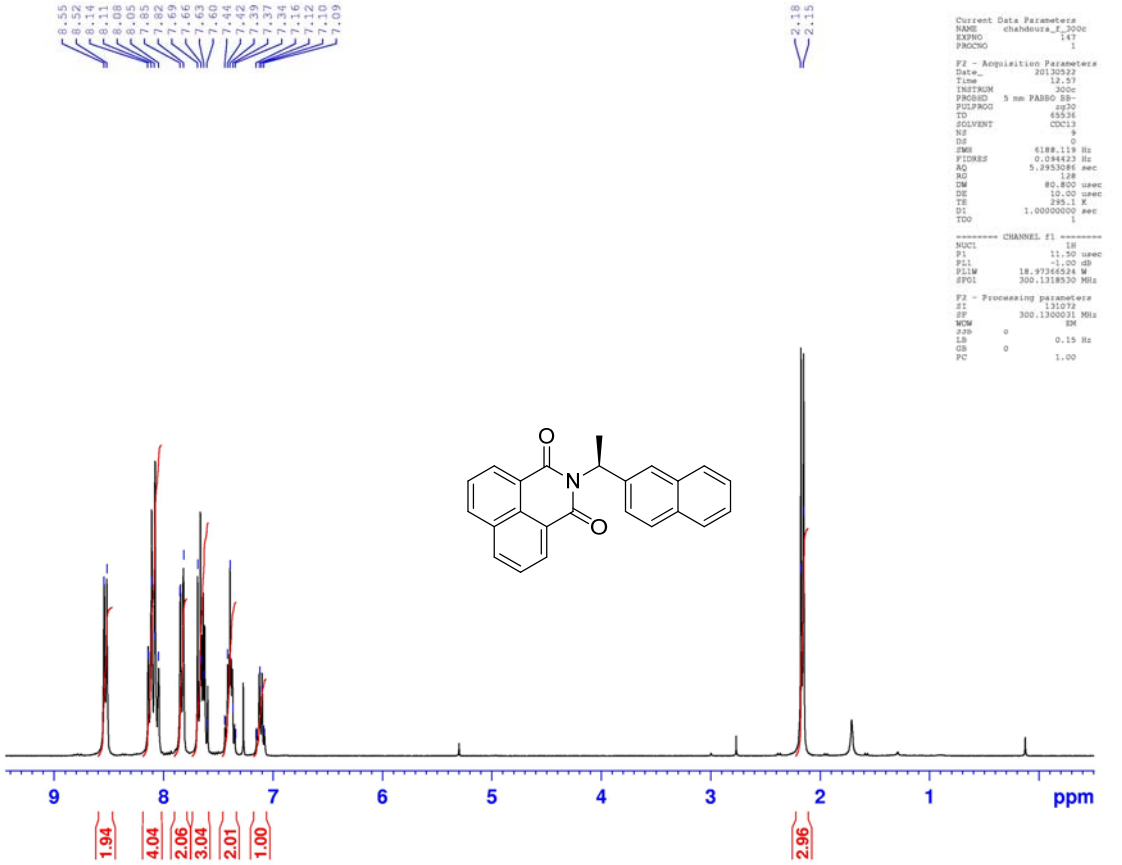
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a27**

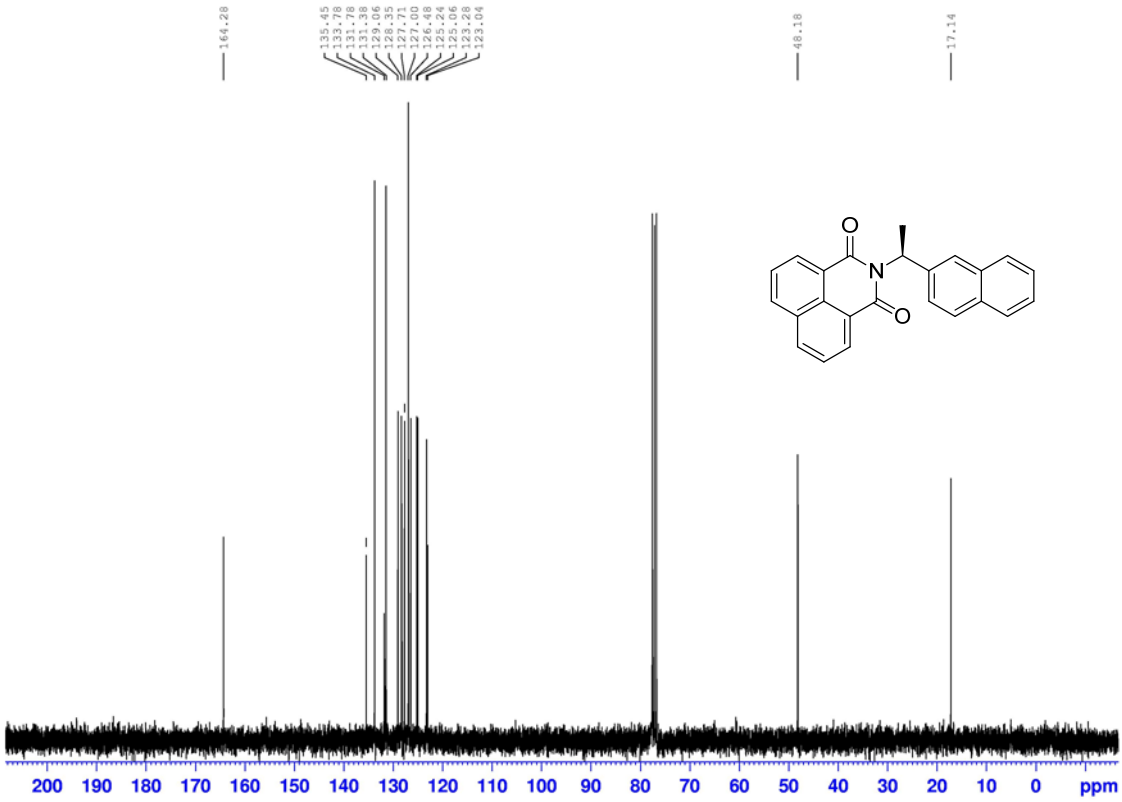

986
fc-986- Sm (Mn, 1x3)
, 14-Jan-2013 + 15:57:38
Scan EI+
TIC
3.57e7
15.45,8586189
100
Area
%
0
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
OH
N
H
O

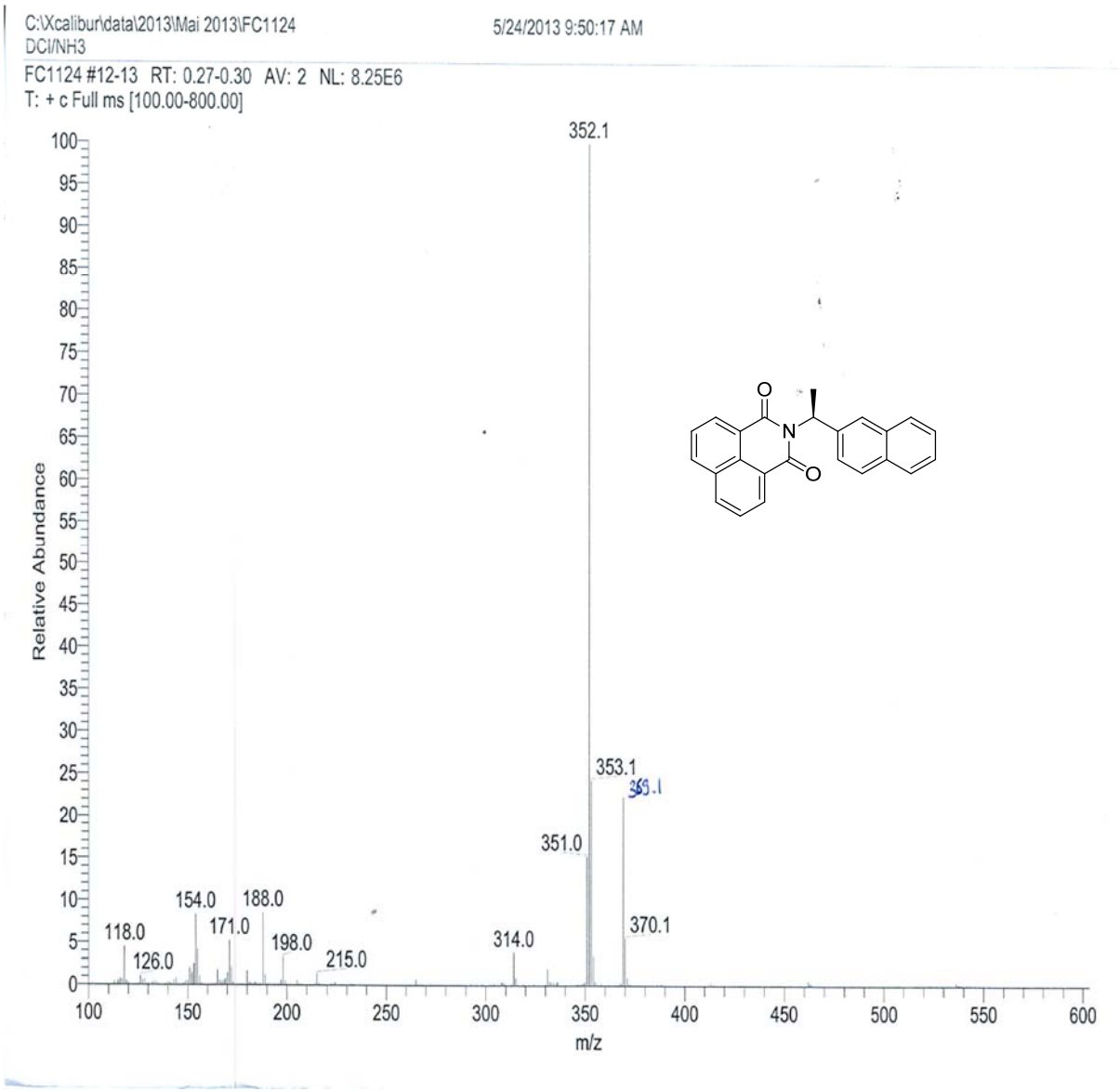

986
fc-986- 2296 (15.483) Cm (2294:2346)
, 14-Jan-2013 + 15:57:38
Scan EI+
4.91e6
100
%
0
44.0251
77.0553
40.0256
73.0603
91.0910
105.0950
133.0687
134.1102
116.1260
92.1231
104.1049
148.1010
172.1390
190.1169
207.0603
117.1116
130.1066
51.0486
39.0297
65.0479
78.0439
135.1111
232.1839
250.2130
281.1576
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
280
290
m/z
O
OH
N
H
O



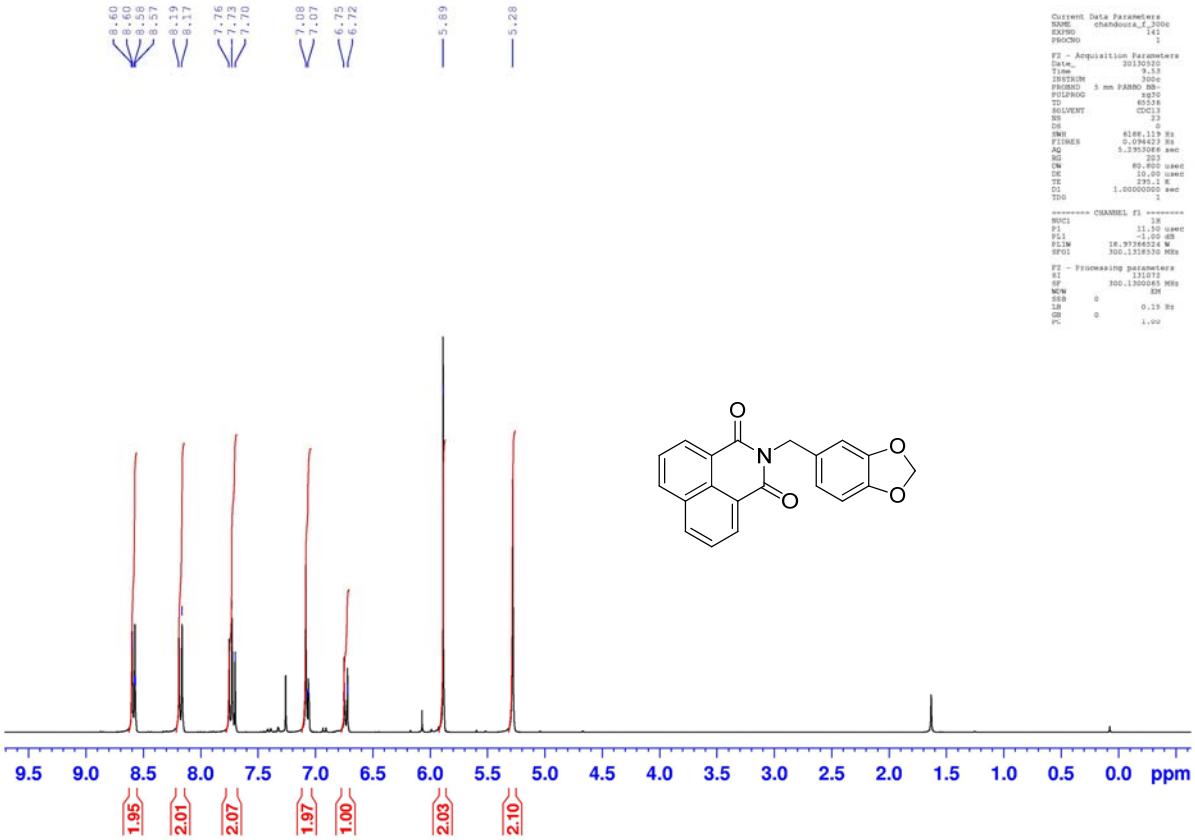
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a28**

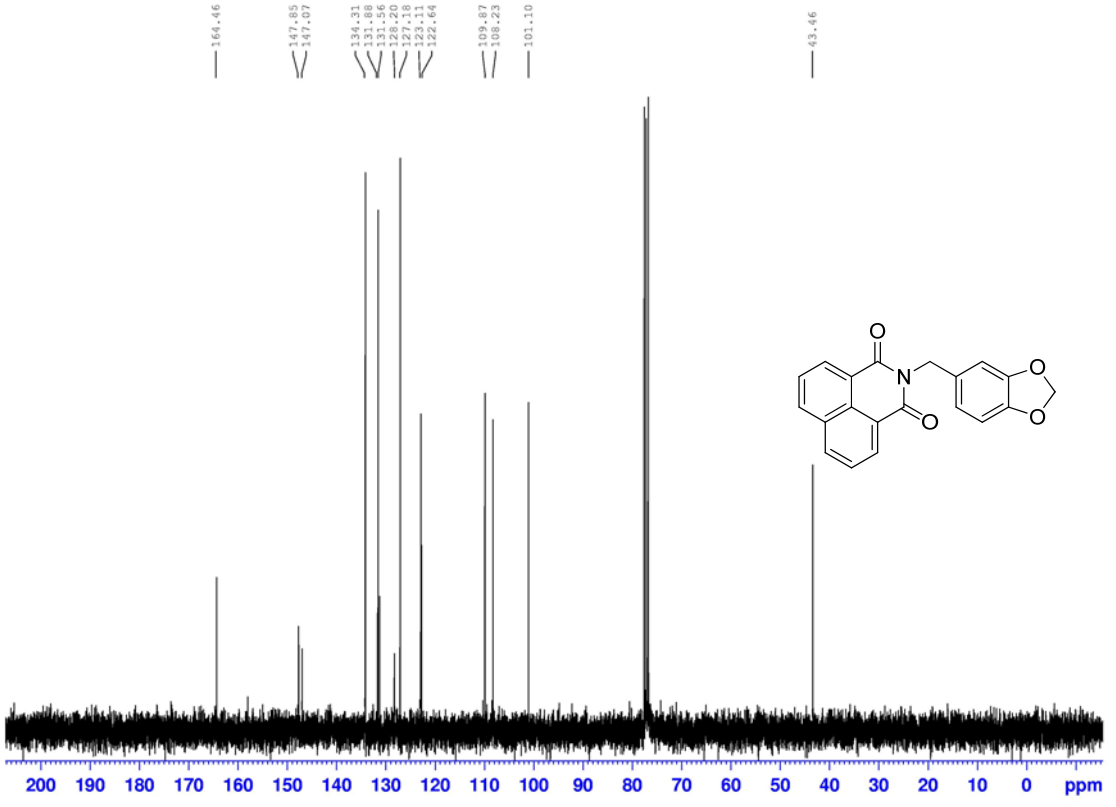

8.55
8.52
8.14
8.11
8.08
8.05
7.85
7.82
7.69
7.66
7.63
7.60
7.44
7.42
7.39
7.37
7.34
7.16
7.12
7.10
7.09
2.18
2.15
Current Data Parameters
NAME chahdeura_f_300c
EXPNO 147
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130522
Time 12.57
INSTRUM 300c
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953086 sec
RG 128
DW 80.800 usec
DE 10.00 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -1.00 dB
PL1W 18.97366524 W
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 131072
SF 300.1300031 MHz
WDW EM
SSB 0
LB 0.15 Hz
GB 0
PC 1.00
O
N
O
9
8
7
6
5
4
3
2
1
ppm
1.94
4.04
2.06
3.04
2.01
1.00
2.96

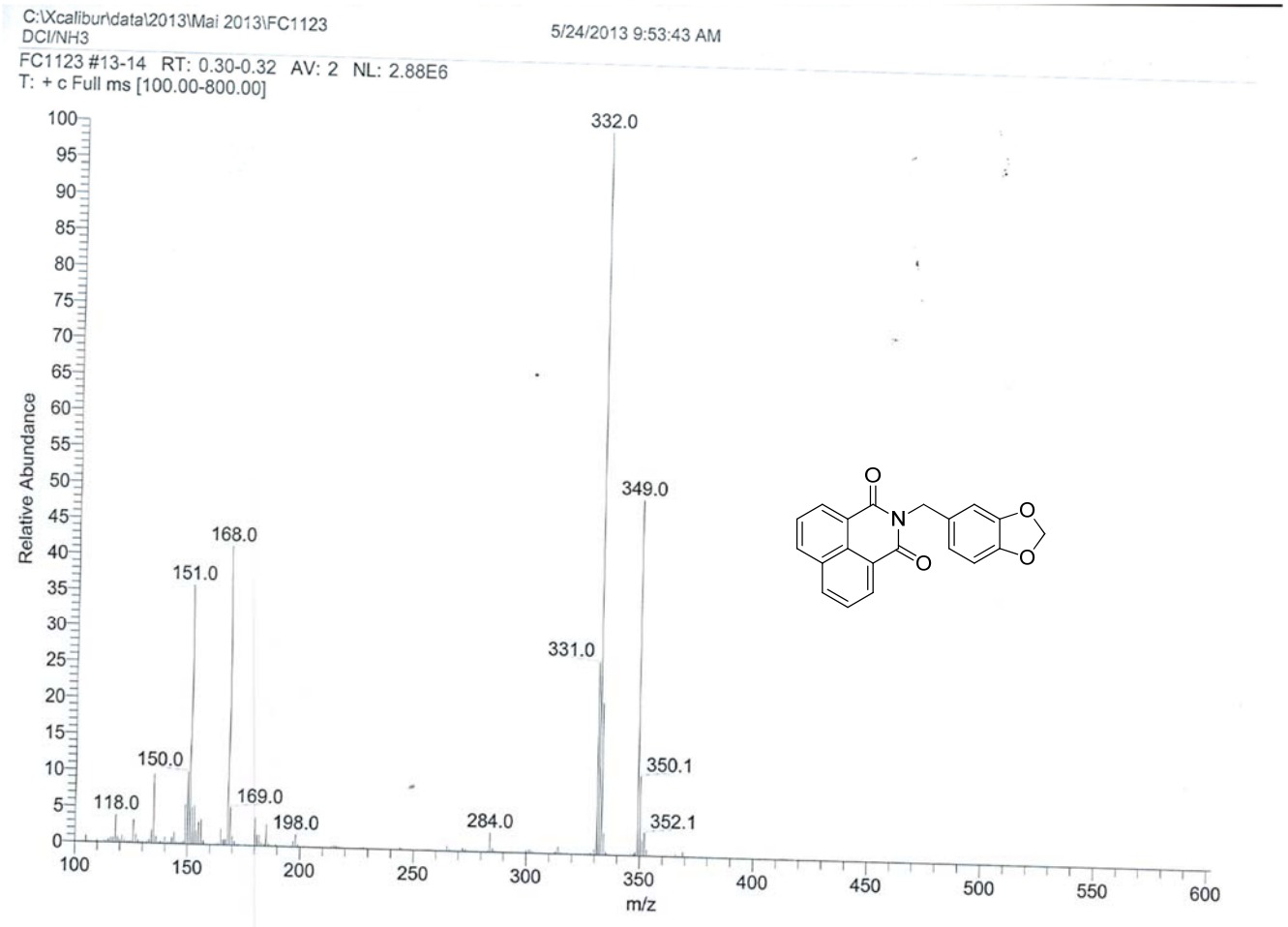

164.28
135.45
133.78
131.78
131.38
129.06
128.35
127.71
127.00
126.68
126.24
125.06
123.28
123.04
48.18
17.14
O
N
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



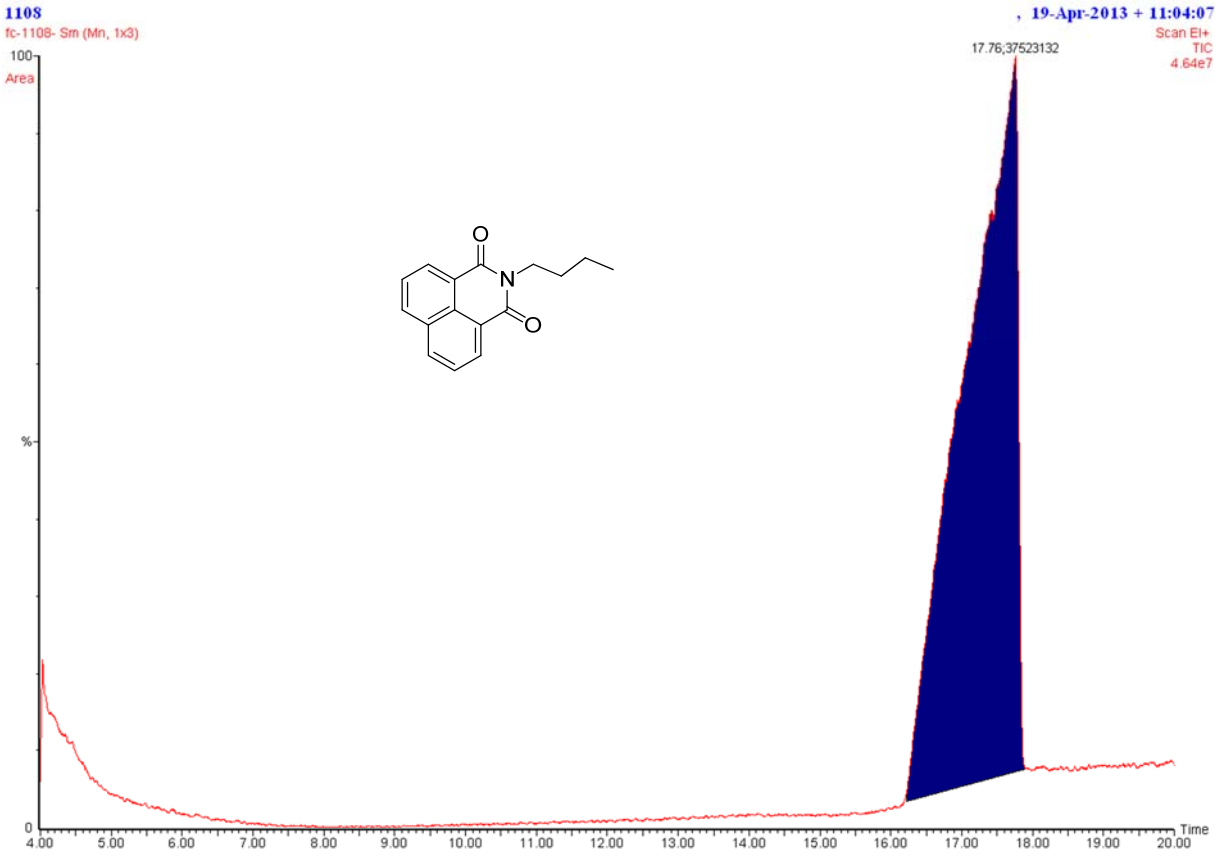
^{1}H NMR (300 MHz) and ^{13}C {^{1}H} NMR (75 MHz) (top) spectra in $CDCl_3$, and DCI(NH_3)-MS (bottom) for **g6**

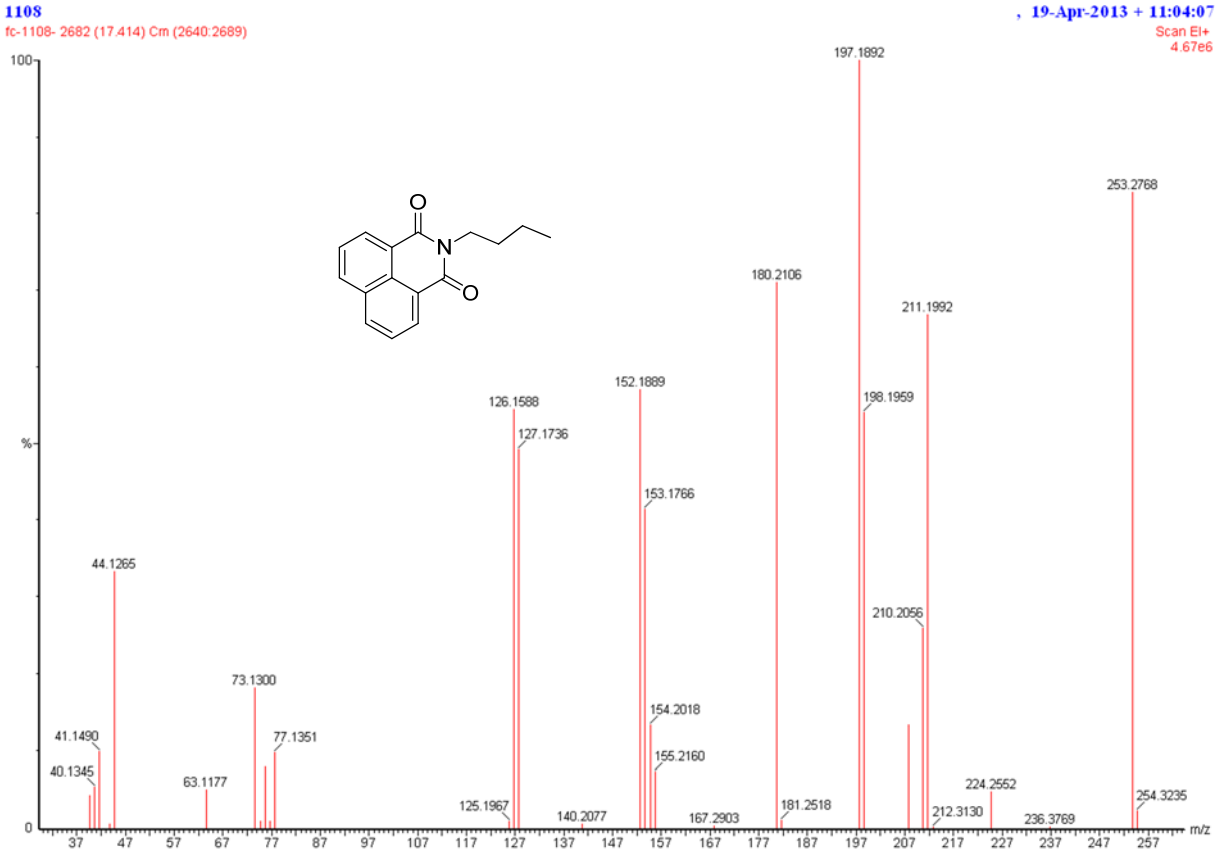

8.60
8.60
8.58
8.57
8.19
8.17
7.76
7.73
7.70
7.08
7.07
6.75
6.72
5.89
5.28
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.95
2.01
2.07
1.97
1.00
2.03
2.10

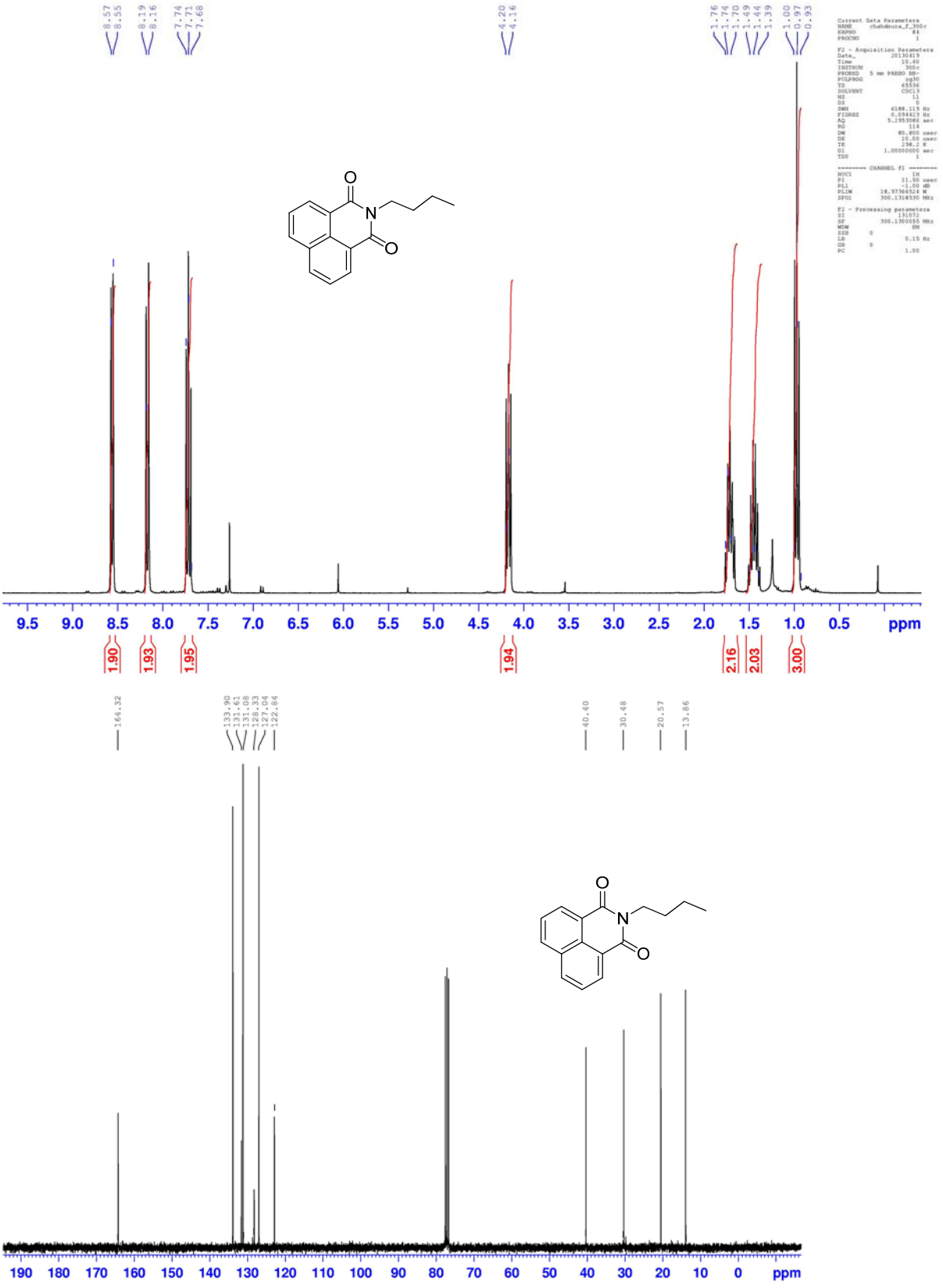

164.46
147.85
147.07
134.31
131.88
131.56
128.20
127.18
123.11
122.64
109.87
108.23
101.10
43.46
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



^{1}H NMR (300 MHz) and ^{13}C {^{1}H} NMR (75 MHz) (top) spectra in $CDCl_3$, and DCI(NH_3)-MS (bottom) for **g8**

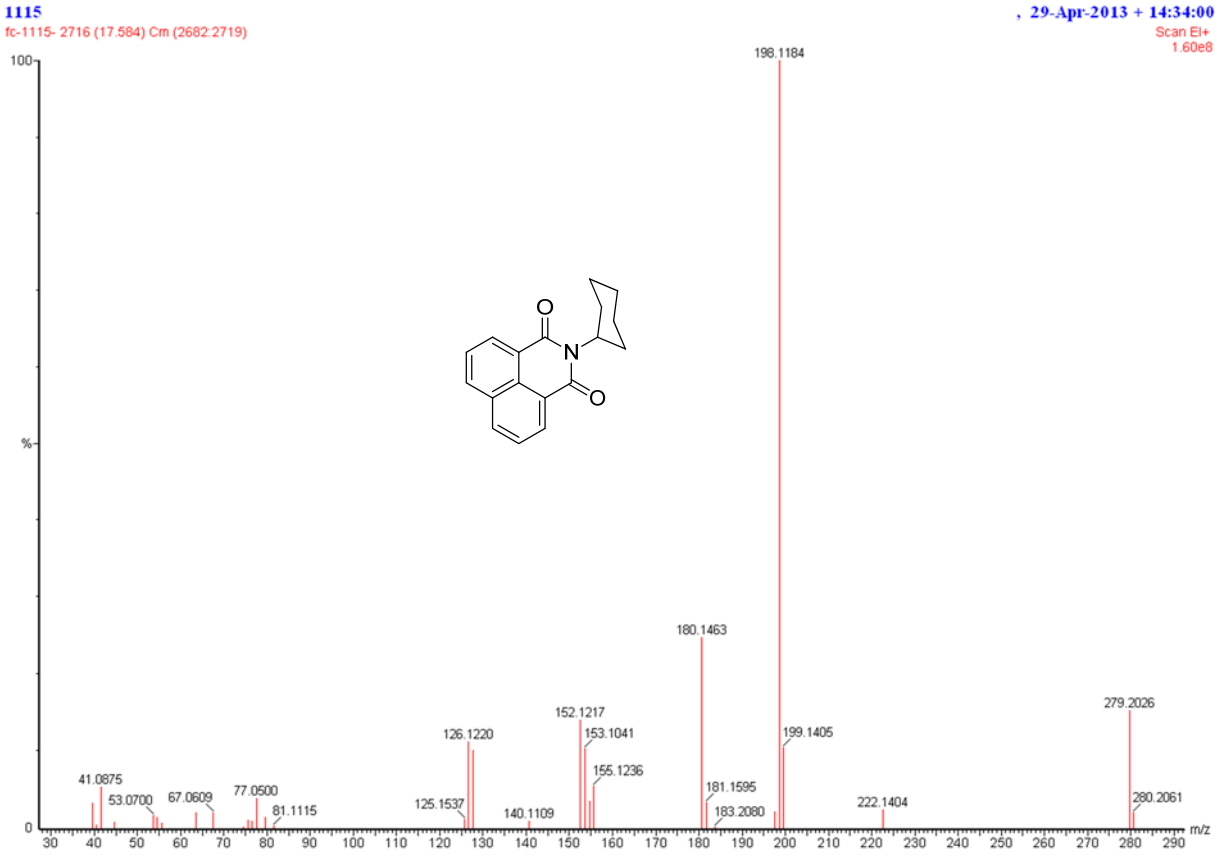

1108
fc-1108- Sm (Mn, 1x3)
, 19-Apr-2013 + 11:04:07
Scan EI+
TIC
4.64e7
17.76;37523132
100
Area
%
0
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

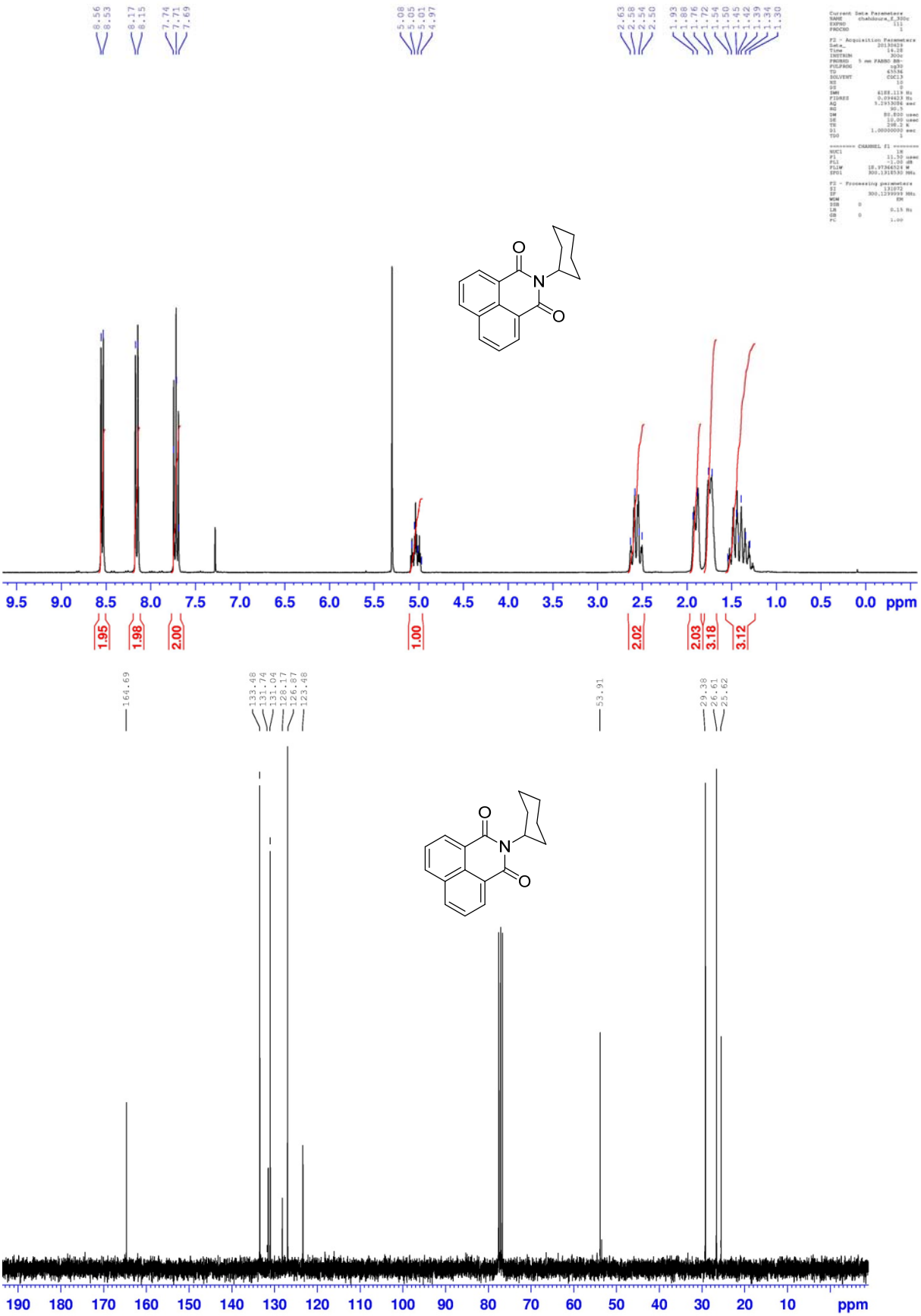

1108
fc-1108- 2682 (17.414) Cm (2640:2689)
, 19-Apr-2013 + 11:04:07
Scan EI+
4.67e6
100
%
0
197.1892
253.2768
180.2106
211.1992
152.1889
126.1588
198.1959
127.1736
153.1766
44.1265
210.2056
73.1300
154.2018
41.1490
77.1351
155.2160
40.1345
63.1177
224.2552
254.3235
125.1967
140.2077
167.2903
181.2518
212.3130
236.3769
37
47
57
67
77
87
97
107
117
127
137
147
157
167
177
187
197
207
217
227
237
247
257
m/z



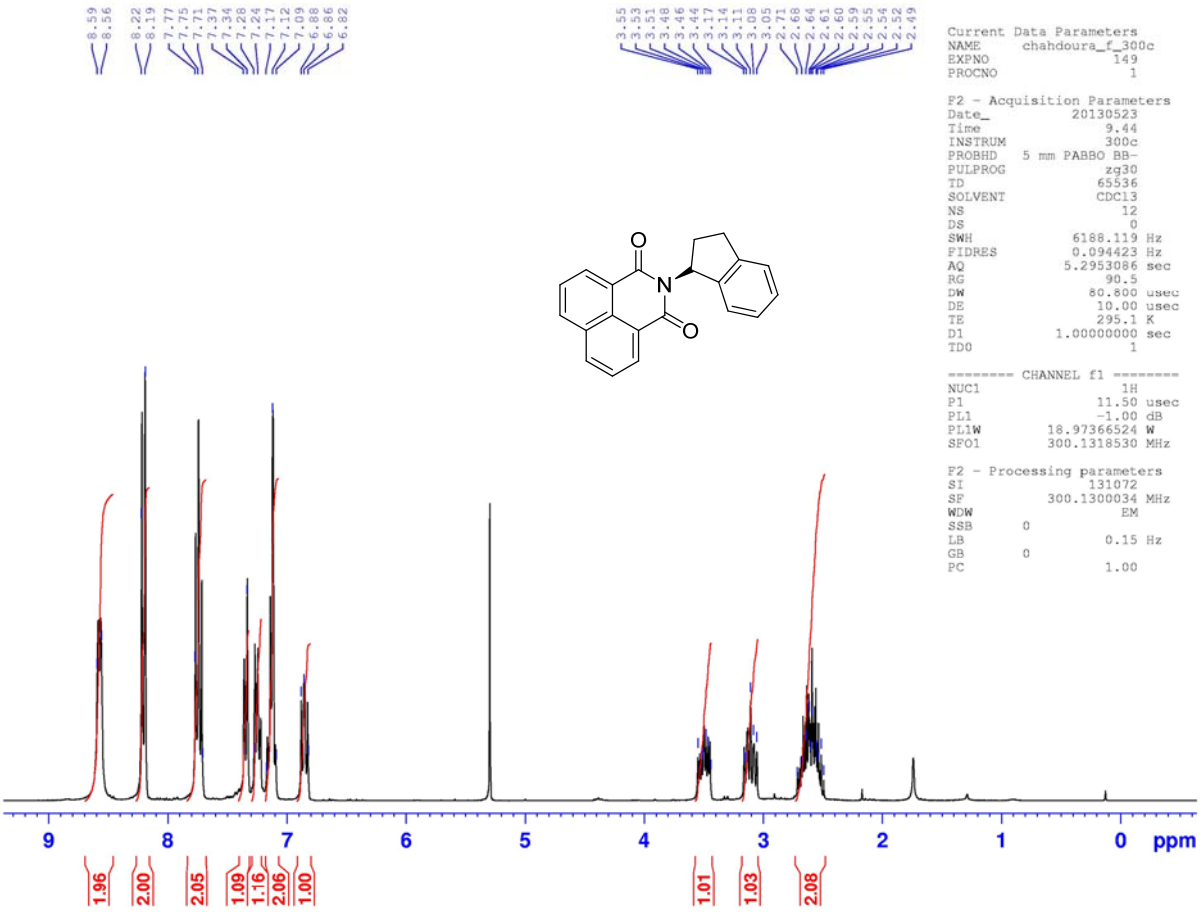
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **g9**

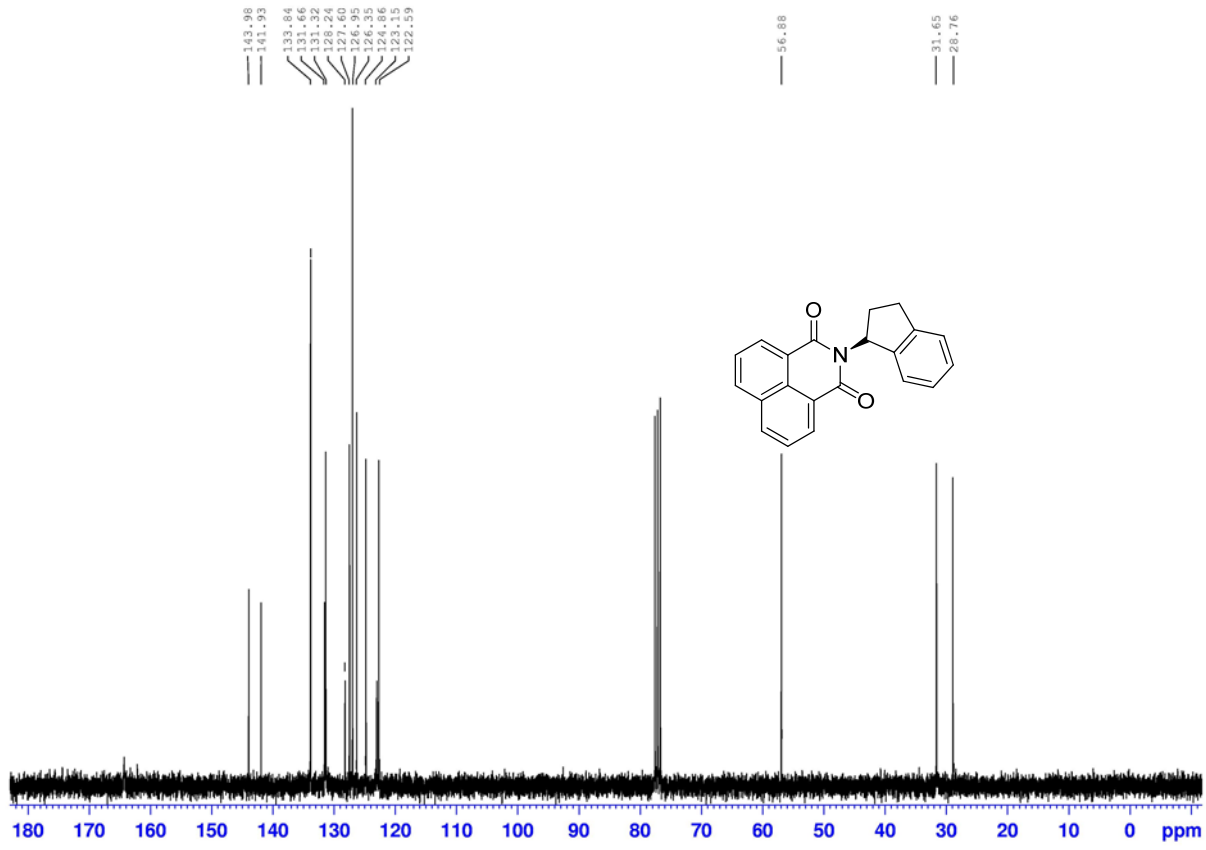

1115
fc-1115- Sm (Mn, 1x3)
, 29-Apr-2013 + 14:34:00
Scan EI+
TIC
4.60e8
17.66;152696160
100
Area
%
0
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N
O

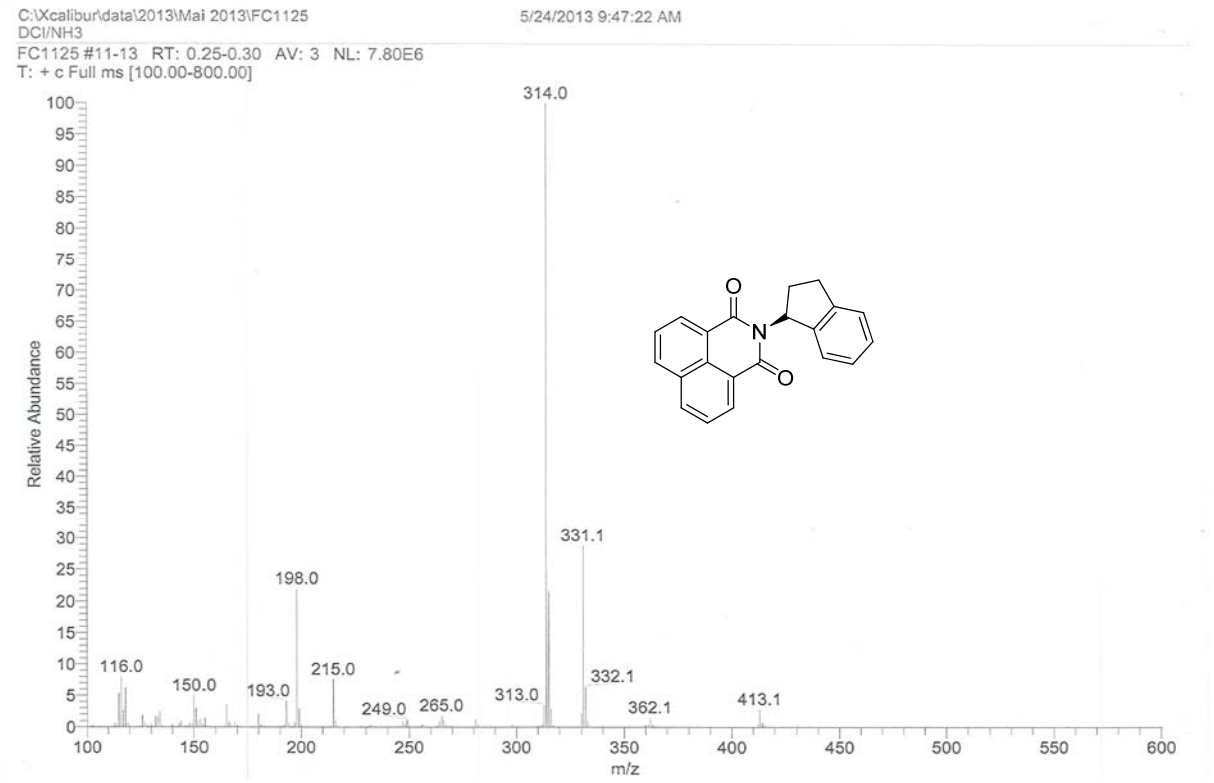

1115
fc-1115- 2716 (17.584) Cm (2682:2719)
, 29-Apr-2013 + 14:34:00
Scan EI+
1.60e8
100
%
0
198.1184
180.1463
279.2026
152.1217
153.1041
126.1220
199.1405
155.1236
41.0875
53.0700
67.0609
77.0500
81.1115
125.1537
140.1109
181.1595
183.2080
222.1404
280.2061
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
280
290
m/z
O
N
O



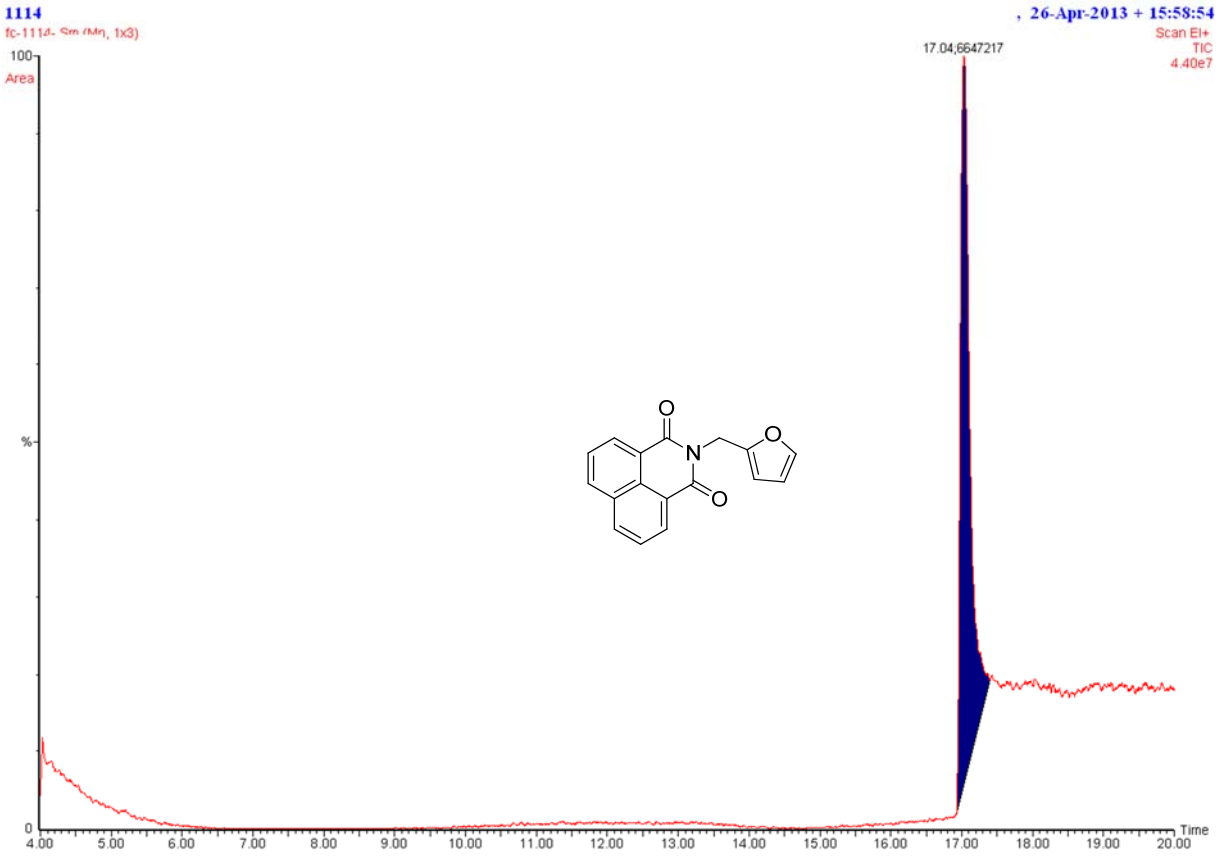
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **g12**

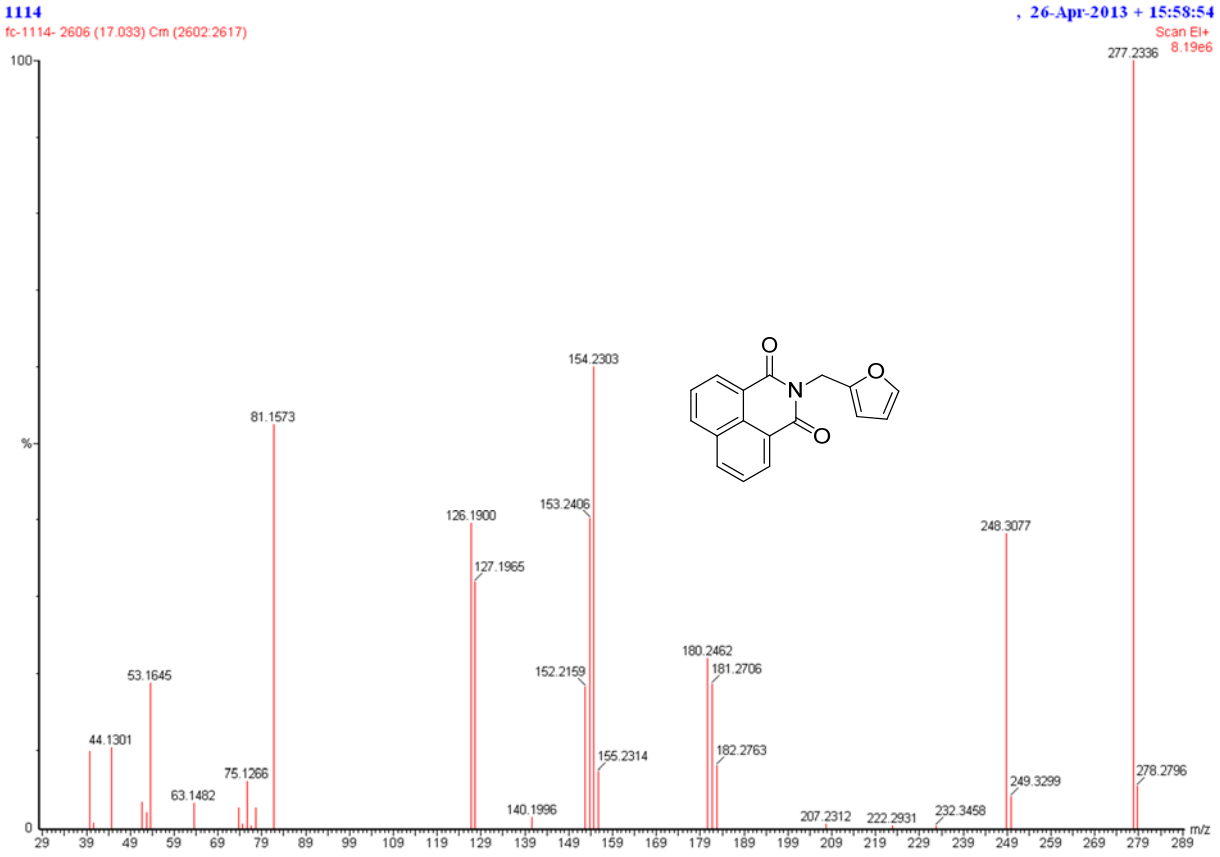

8.59
8.56
8.22
8.19
7.77
7.75
7.71
7.37
7.34
7.28
7.24
7.17
7.12
7.09
6.88
6.86
6.82
3.55
3.53
3.51
3.48
3.46
3.44
3.17
3.14
3.11
3.08
3.05
2.71
2.68
2.64
2.61
2.60
2.59
2.55
2.54
2.52
2.49
Current Data Parameters
NAME chahdoura_f_300c
EXPNO 149
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130523
Time 9.44
INSTRUM 300c
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953086 sec
RG 90.5
DW 80.800 usec
DE 10.00 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -1.00 dB
PL1W 18.97366524 W
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 131072
SF 300.1300034 MHz
WDW EM
SSB 0
LB 0.15 Hz
GB 0
PC 1.00
O
N
O
9
8
7
6
5
4
3
2
1
0
ppm
1.96
2.00
2.05
1.09
1.16
2.06
1.00
1.01
1.03
2.08

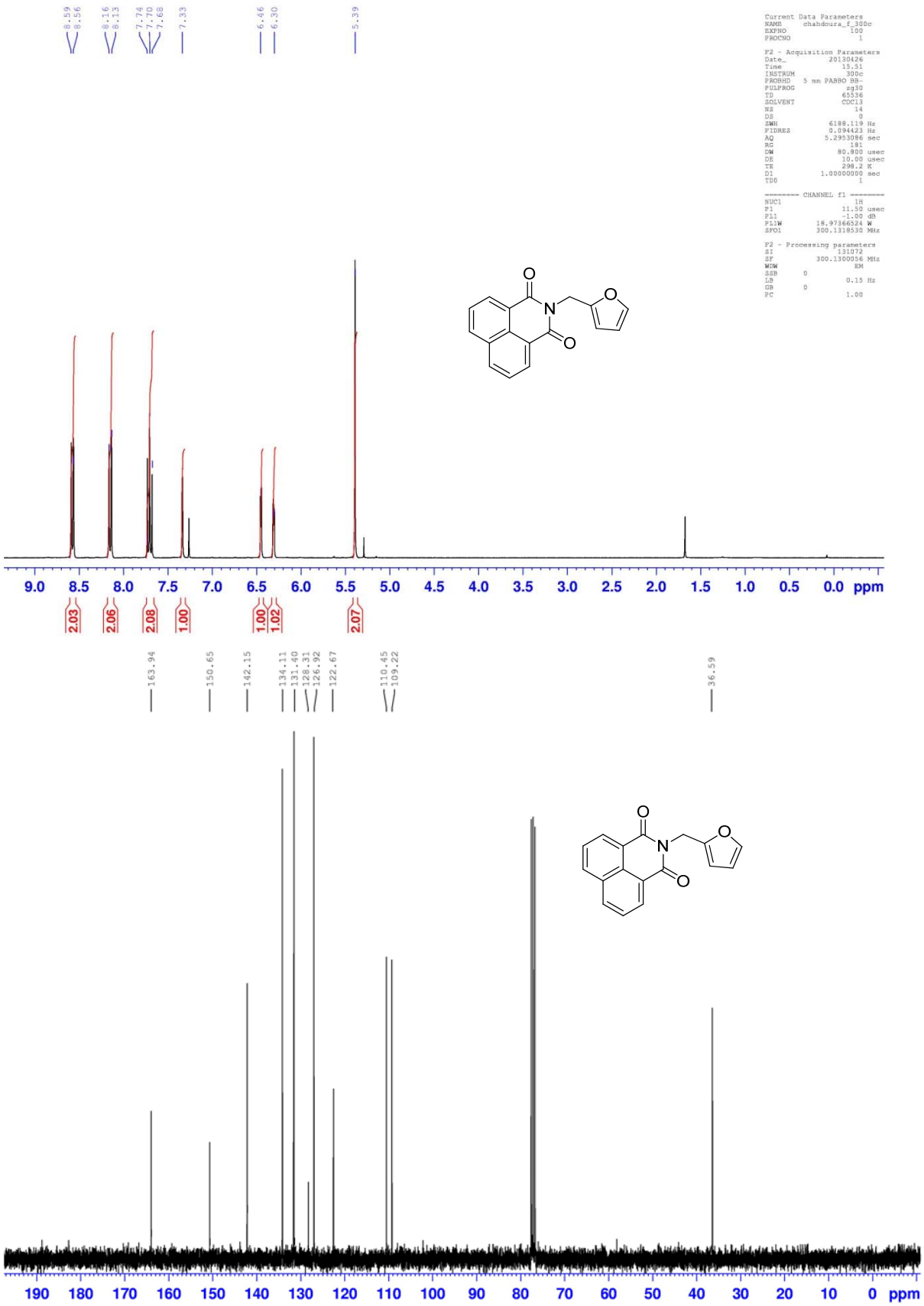

143.98
141.93
133.84
131.66
131.32
128.24
127.60
126.95
126.35
124.86
123.15
122.59
56.88
31.65
28.76
O
N
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



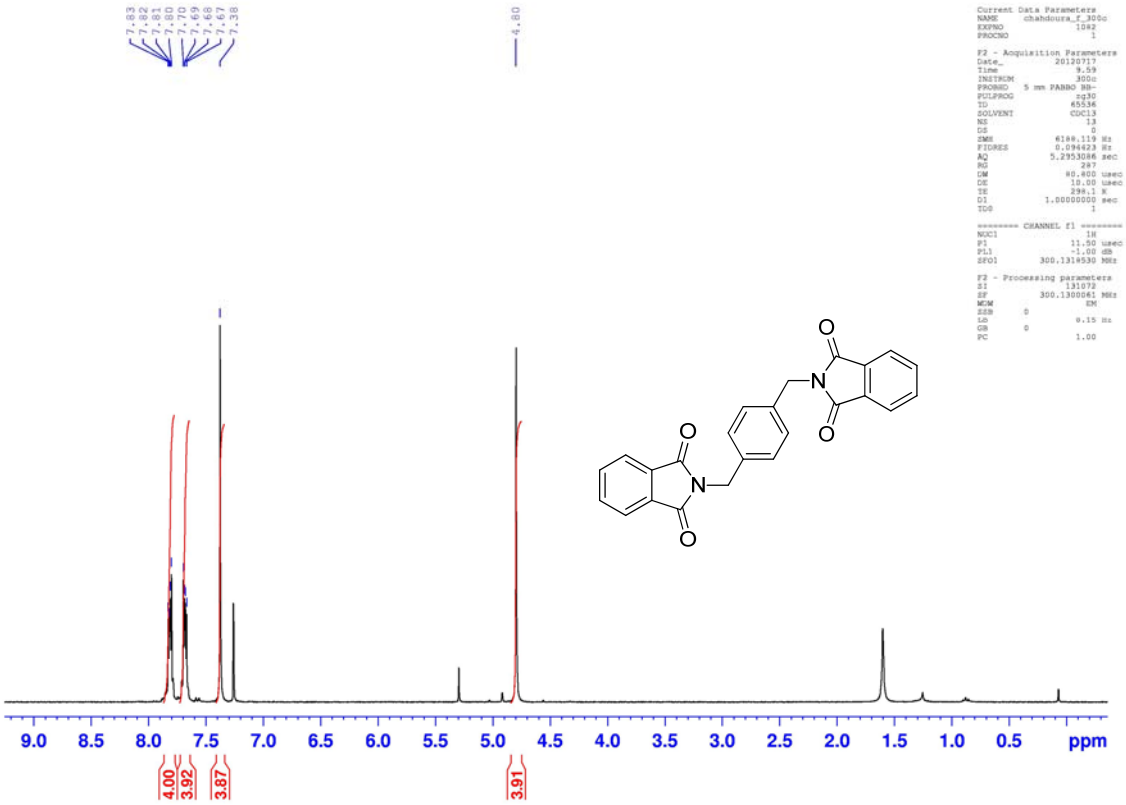
^{1}H NMR (300 MHz) and ^{13}C {^{1}H} NMR (75 MHz) (top) spectra in $CDCl_3$, and DCI(NH_3)-MS (bottom) for **g16**

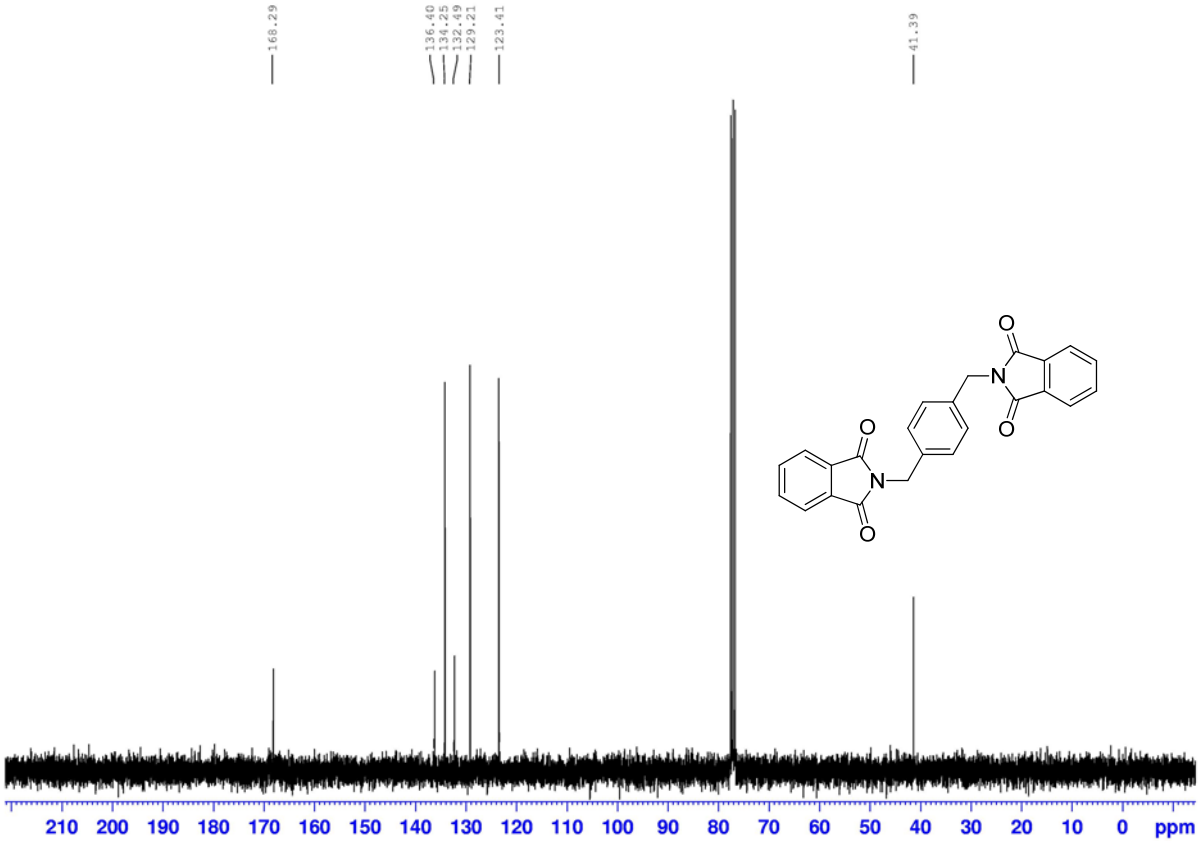

1114
, 26-Apr-2013 + 15:58:54
Scan EI+
TIC
4.40e7
17.04,6647217
Area
%
Time

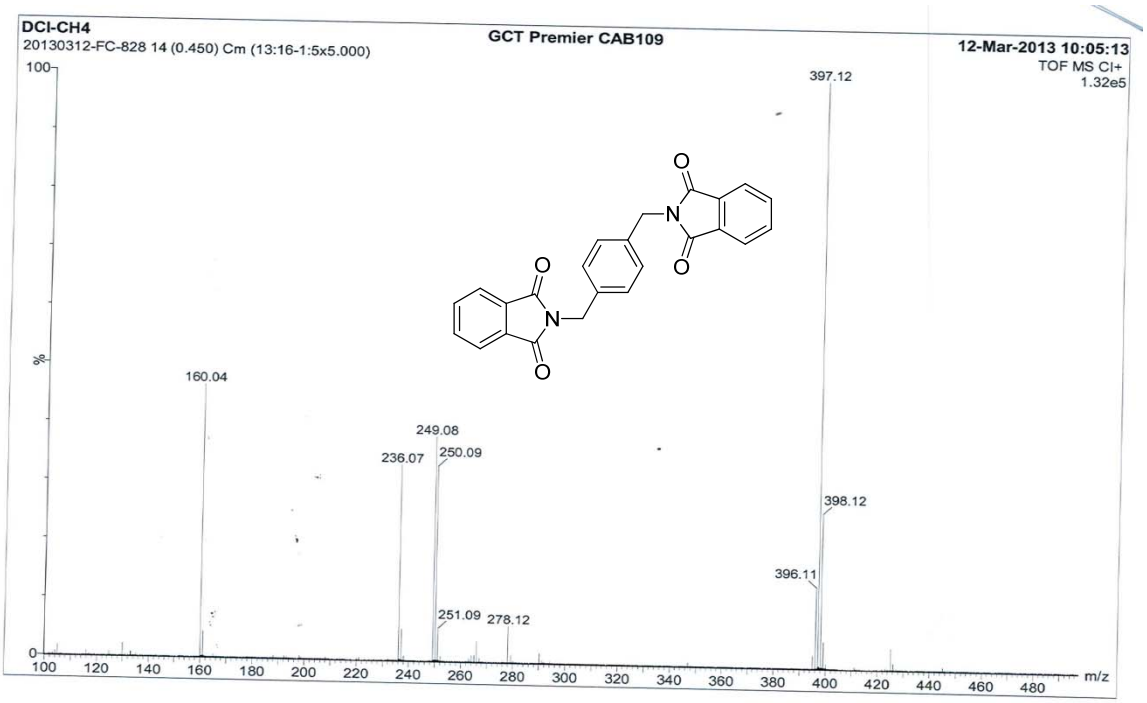

1114
fc-1114- 2606 (17.033) Cm (2602:2617)
, 26-Apr-2013 + 15:58:54
Scan EI+
8.19e6
277.2336
154.2303
81.1573
153.2406
126.1900
248.3077
127.1965
180.2462
181.2706
53.1645
152.2159
44.1301
155.2314
182.2763
75.1266
278.2796
63.1482
249.3299
140.1996
207.2312
222.2931
232.3458
%
m/z



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **g24**

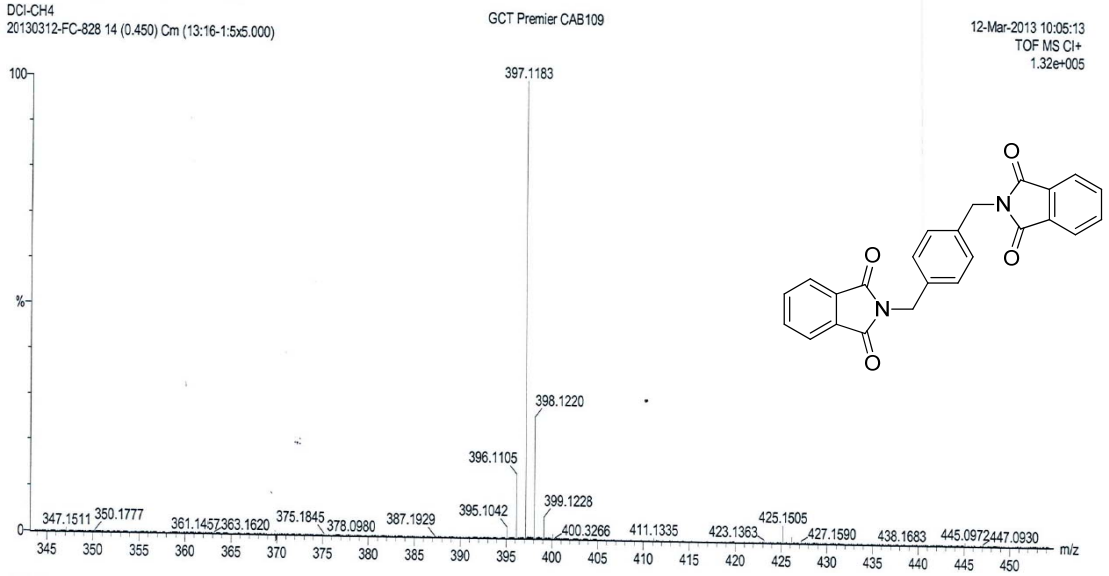

7.83
7.82
7.81
7.80
7.70
7.69
7.68
7.67
7.38
4.80
Current Data Parameters
NAME chahdoura_f_300c
EXPNO 1042
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120717
Time 9.59
INSTRUM 300c
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953086 sec
RG 287
DW 80.800 usec
DE 10.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -1.00 dB
SFO1 300.1319530 MHz
F2 - Processing parameters
SI 131072
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.15 Hz
GB 0
PC 1.00
O
N
O
O
N
O
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.00
3.92
3.87
3.91


168.29
136.40
134.25
132.49
129.21
123.41
41.39
O
N
O
O
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



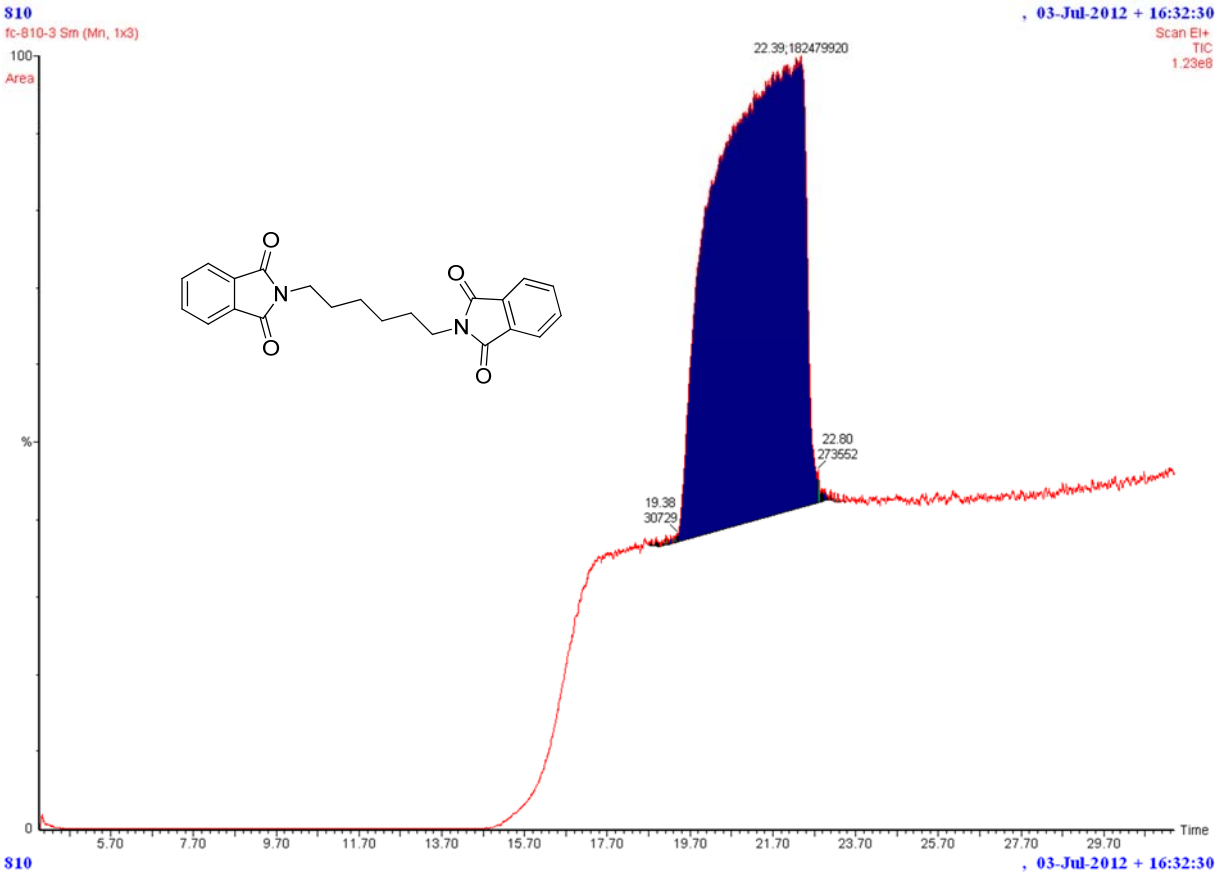
Single Mass Analysis
Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

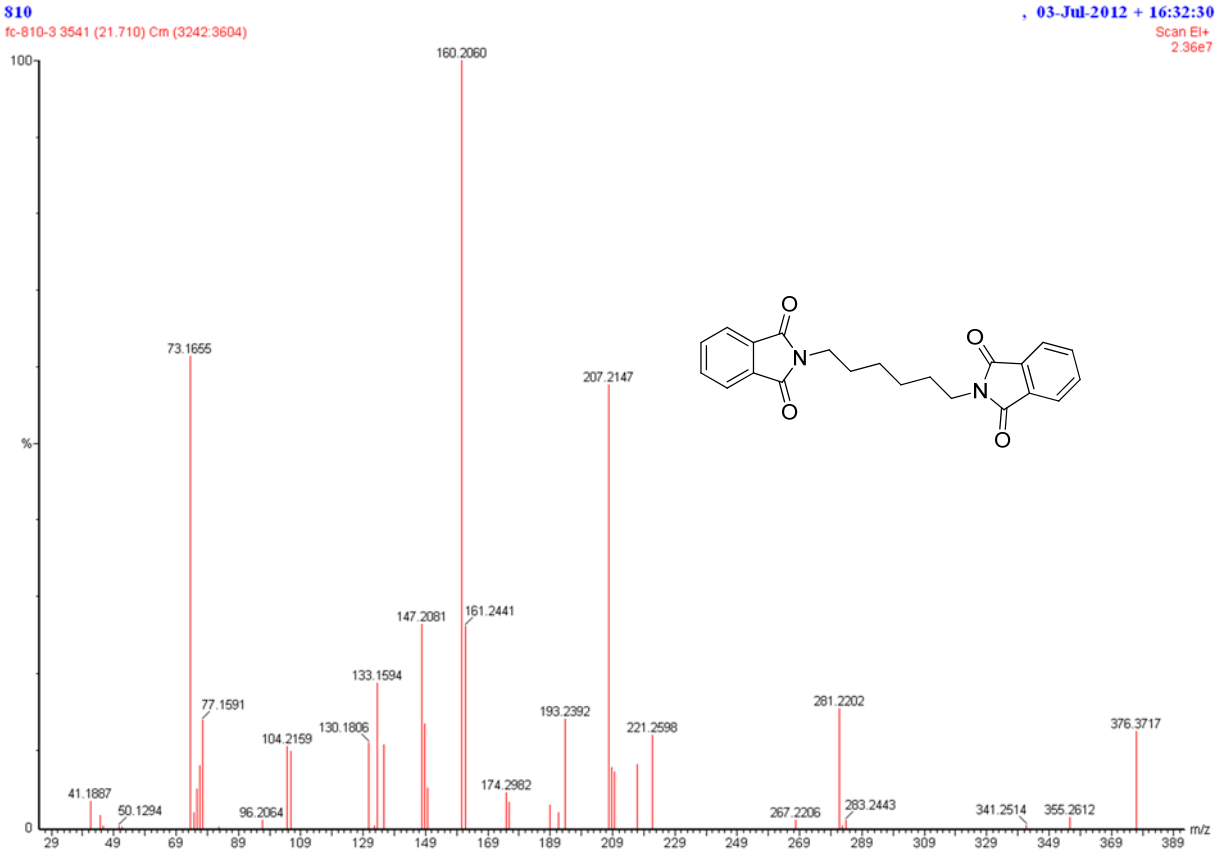
Monoisotopic Mass, Odd and Even Electron Ions
619 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-60 N: 0-15 O: 0-8

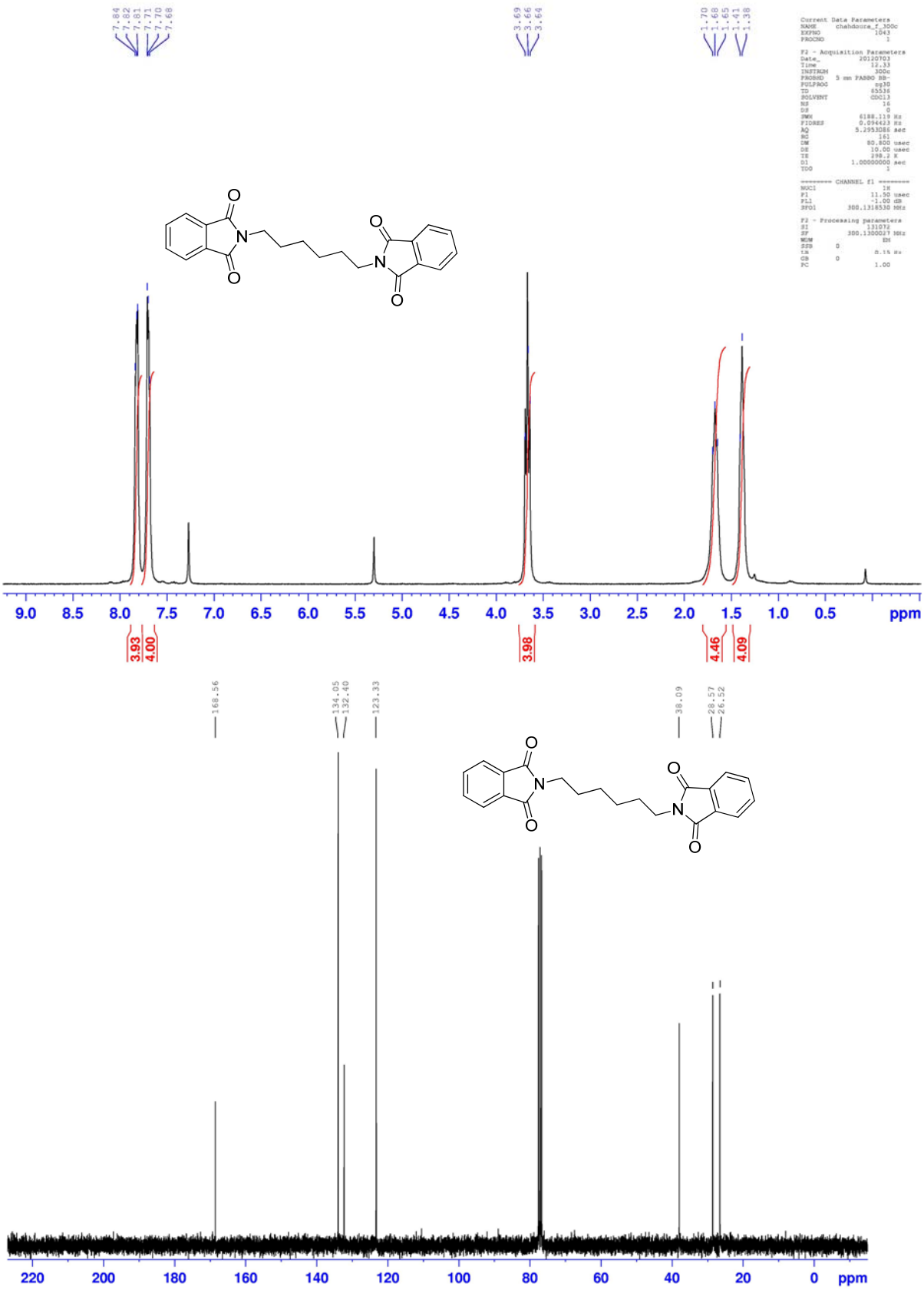


Minimum:				-1.5		
Maximum:		1.3	3.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
397.1183	397.1175	0.8	2.0	18.0	41.6	C22 H15 N5 O3
	397.1188	-0.5	-1.3	17.5	45.1	C24 H17 N2 O4
	397.1193	-1.0	-2.5	10.5	3396.0	C9 H13 N14 O5

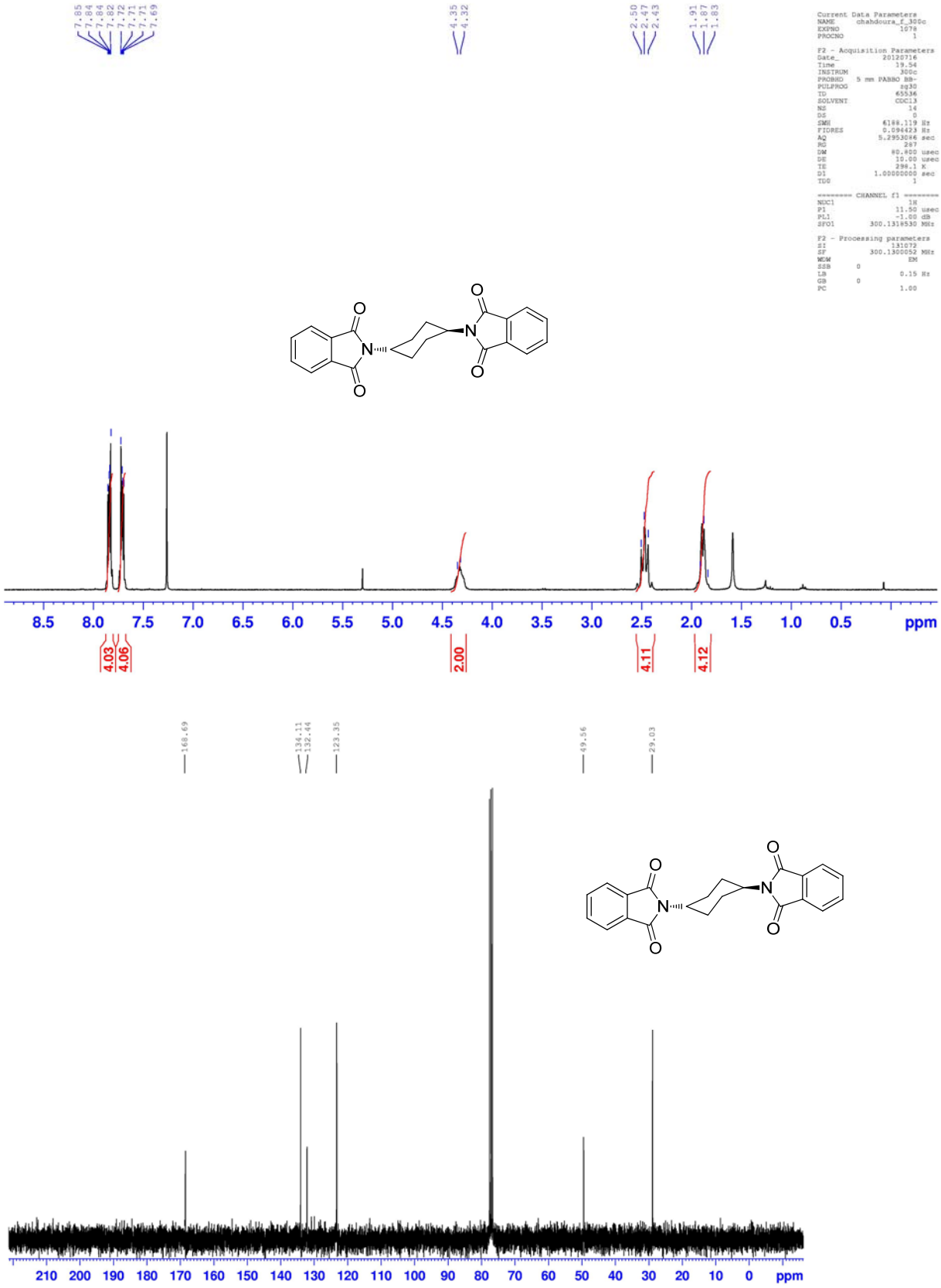
^{1}H NMR (300 MHz) and ^{13}C {^{1}H} NMR (75 MHz) (top) spectra in $CDCl_3$, and low and high resolution DCI(CH_4)-MS (bottom) for **a29**


810
fc-810-3 Sm (Mn, 1x3)
, 03-Jul-2012 + 16:32:30
Scan EI+
TIC
1.23e8
Area
100
%
0
22.39;182479920
22.80
273552
19.38
30729
5.70
7.70
9.70
11.70
13.70
15.70
17.70
19.70
21.70
23.70
25.70
27.70
29.70
Time

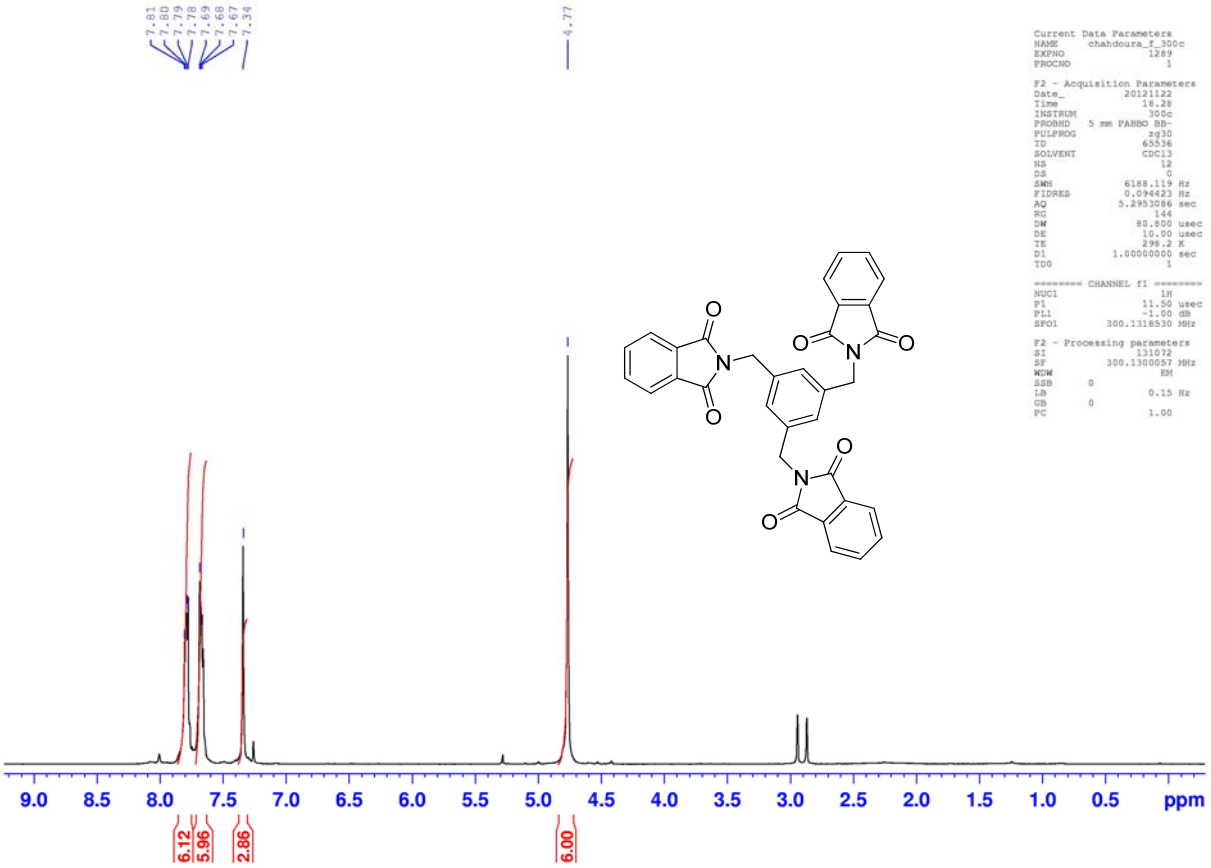

810
fc-810-3 3541 (21.710) Cm (3242:3604)
, 03-Jul-2012 + 16:32:30
Scan EI+
2.36e7
100
%
0
160.2060
73.1655
207.2147
147.2081
161.2441
133.1594
77.1591
193.2392
281.2202
376.3717
104.2159
130.1806
221.2598
41.1887
50.1294
96.2064
174.2982
267.2206
283.2443
341.2514
355.2612
29
49
69
89
109
129
149
169
189
209
229
249
269
289
309
329
349
369
389
m/z



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a30**



^{1}H NMR (300 MHz) (top)) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a31**

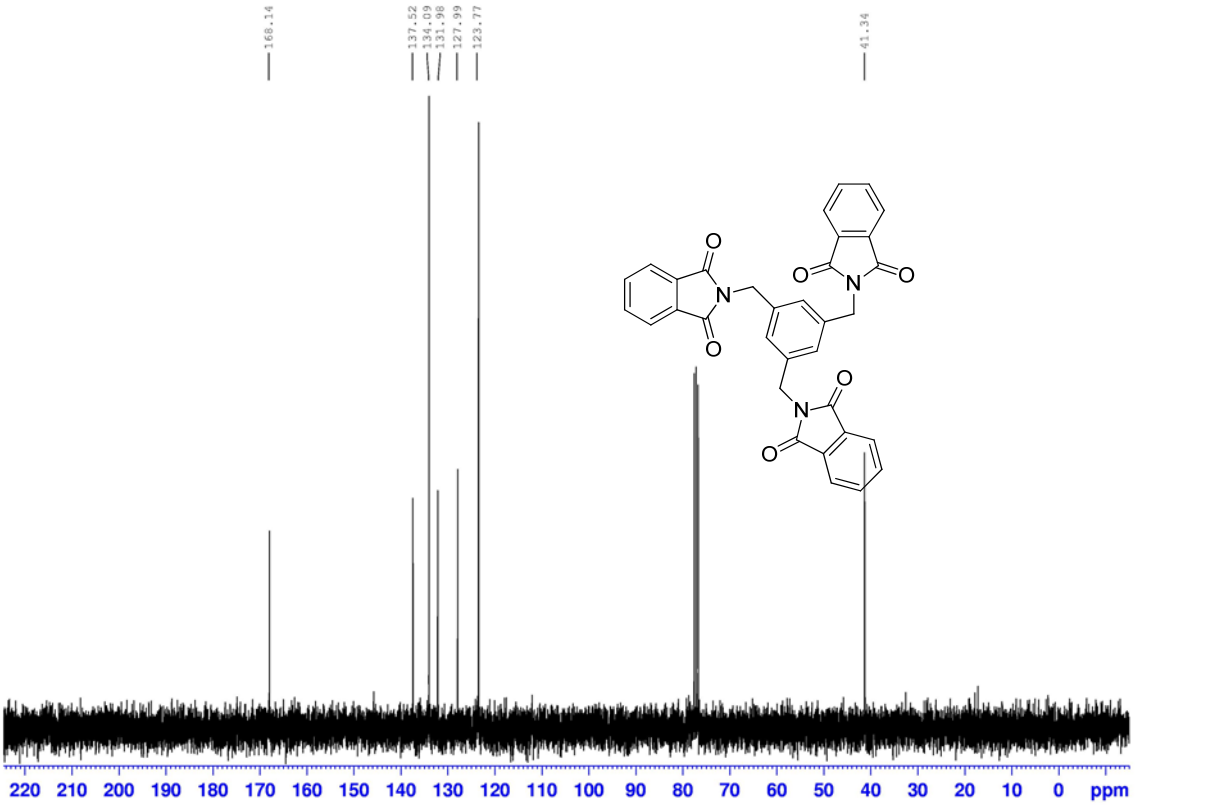


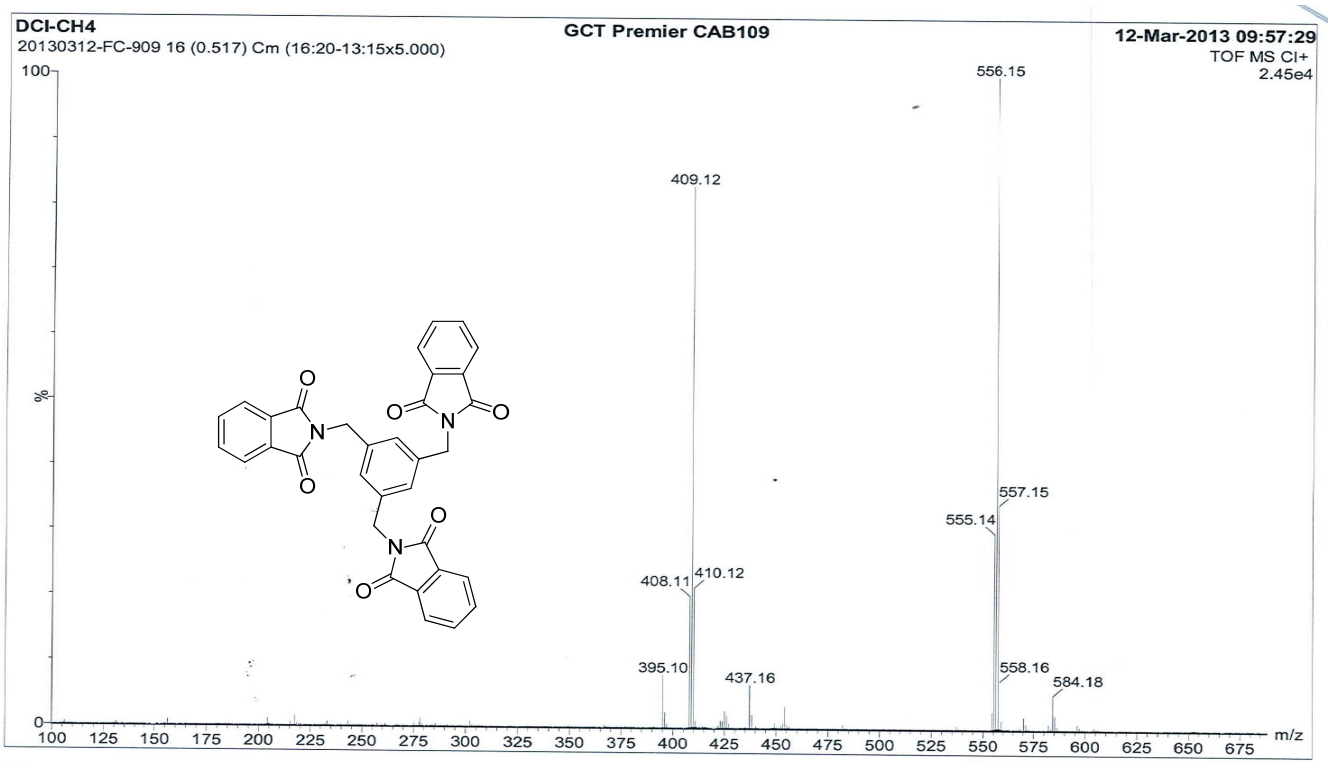

```
Current Data Parameters
NAME      chahdoura_f_300c
EXPNO                 1289
PROCNO                   1

F2 - Acquisition Parameters
Date_              20121122
Time                  18.28
INSTRUM                300c
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      12
DS                       0
SWH                6188.119 Hz
FIDRES             0.094423 Hz
AQ                 5.2953086 sec
RG                      144
DW                   80.800 usec
DE                   10.00 usec
TE                  298.2 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                     1H
P1                    11.50 usec
PL1                   -1.00 dB
SFO1           300.1318530 MHz

F2 - Processing parameters
SI                    131072
SF             300.1300057 MHz
WDW                      EM
SSB          0
LB                     0.15 Hz
GB           0
PC                     1.00
```

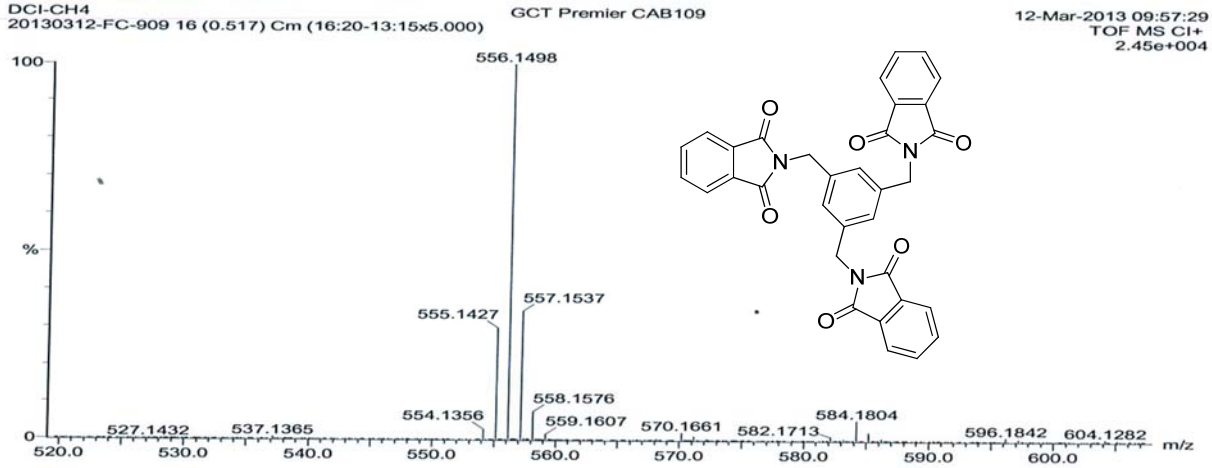




Elemental Composition Report **Page 1**

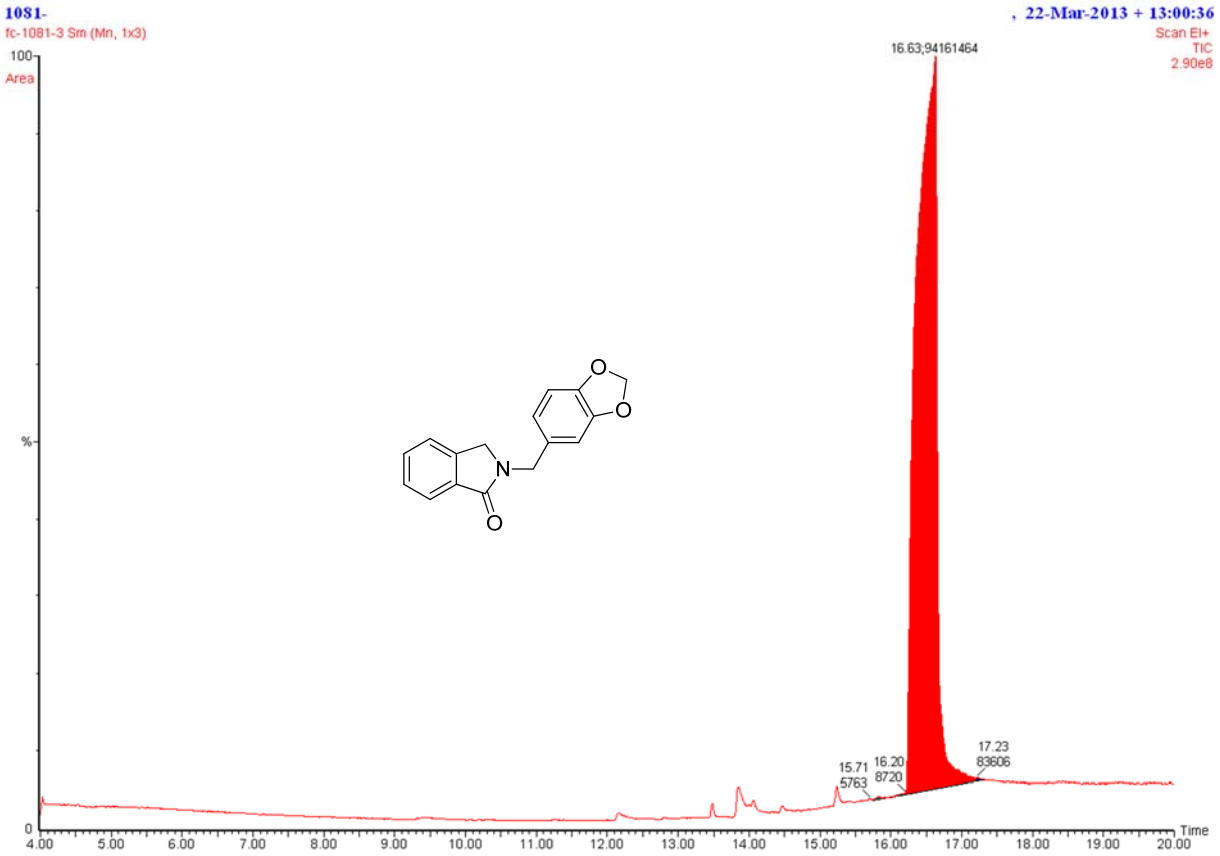
Single Mass Analysis
Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

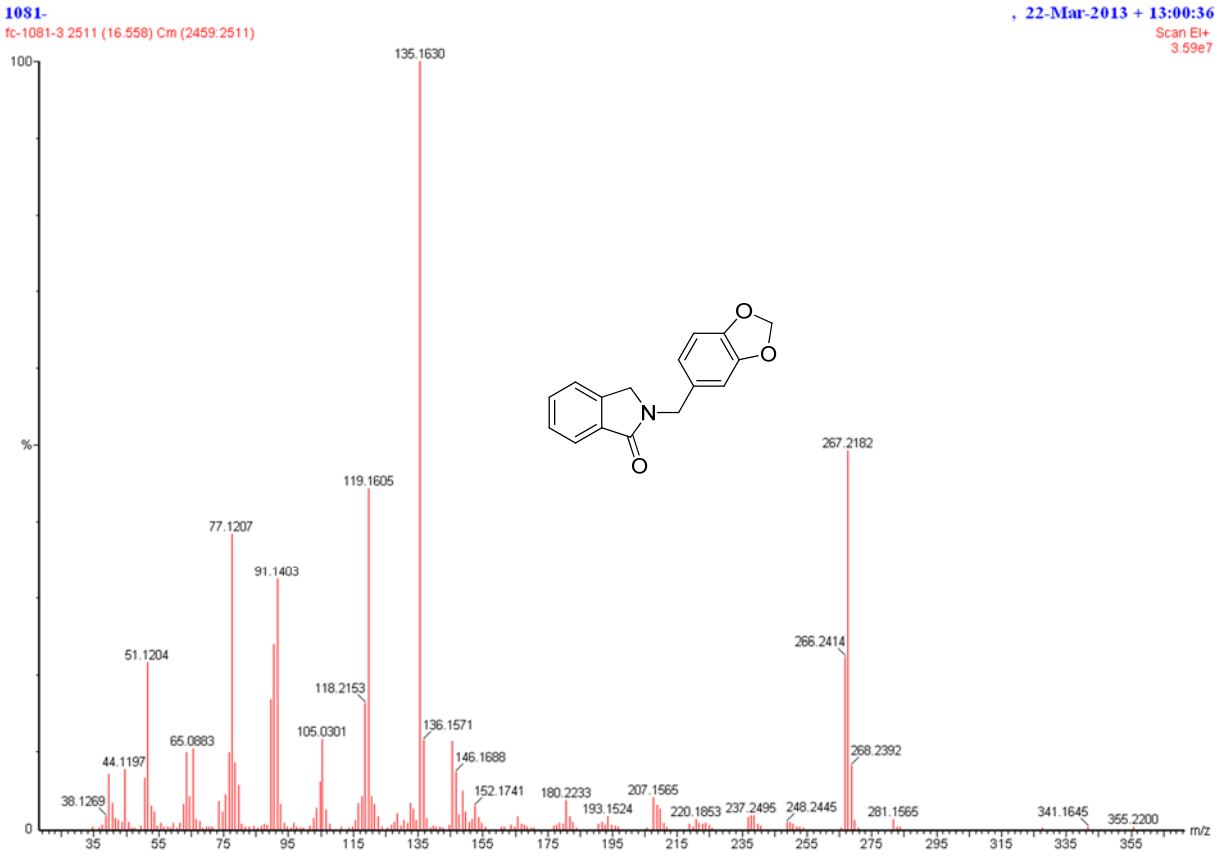
Monoisotopic Mass, Odd and Even Electron Ions
744 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-60 N: 0-15 O: 0-8

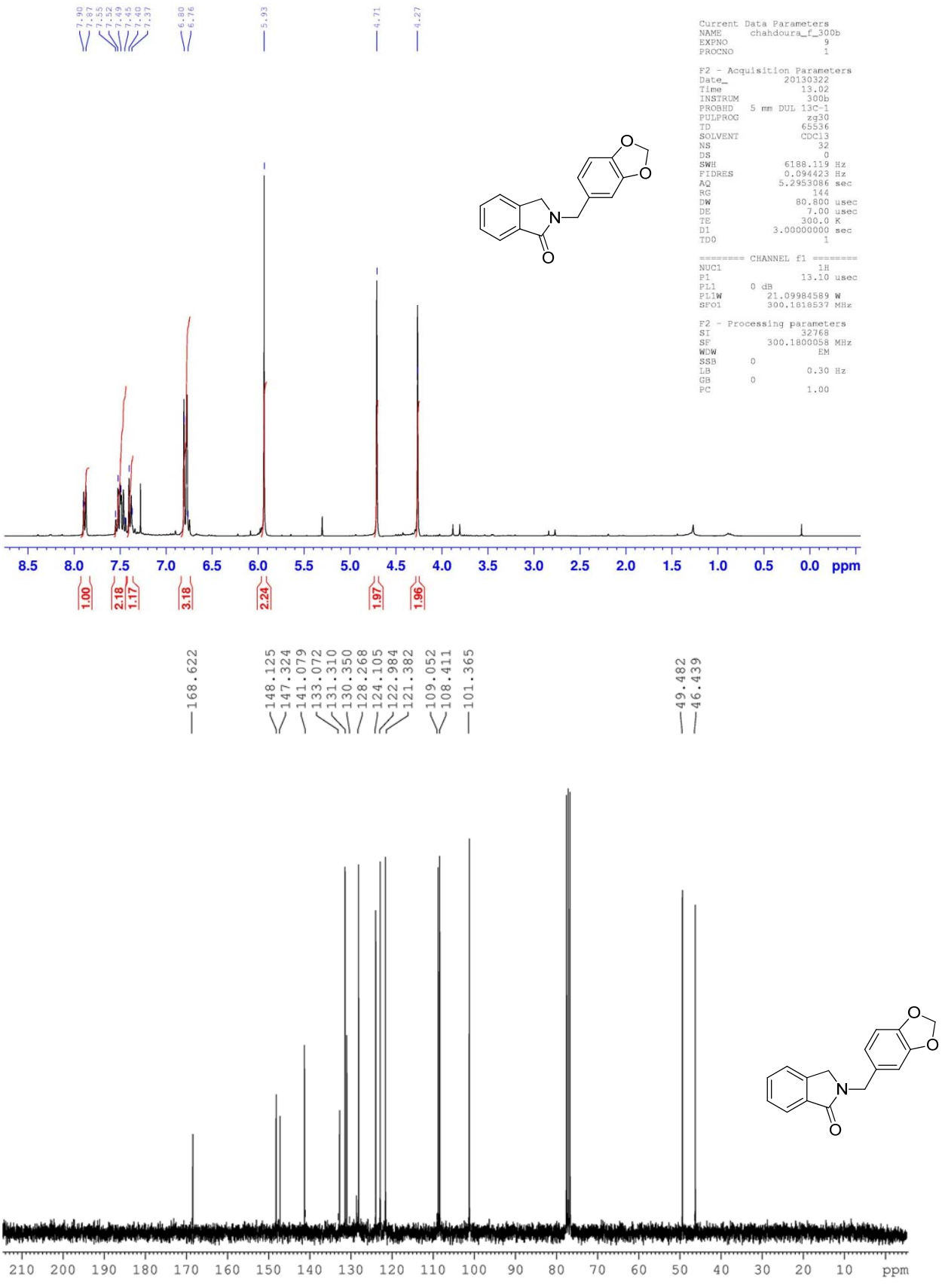


Minimum:				-1.5		
Maximum:		1.3	3.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
556.1498	556.1482	1.6	2.9	25.5	11.1	C29 H18 N9 O4
	556.1495	0.3	0.5	25.0	23.2	C31 H20 N6 O5
	556.1495	0.3	0.5	30.5	45.7	C30 H14 N13
	556.1509	-1.1	-2.0	24.5	52.2	C33 H22 N3 O6
	556.1509	-1.1	-2.0	30.0	70.3	C32 H16 N10 O
	556.1514	-1.6	-2.9	17.5	293.7	C18 H18 N15 O7

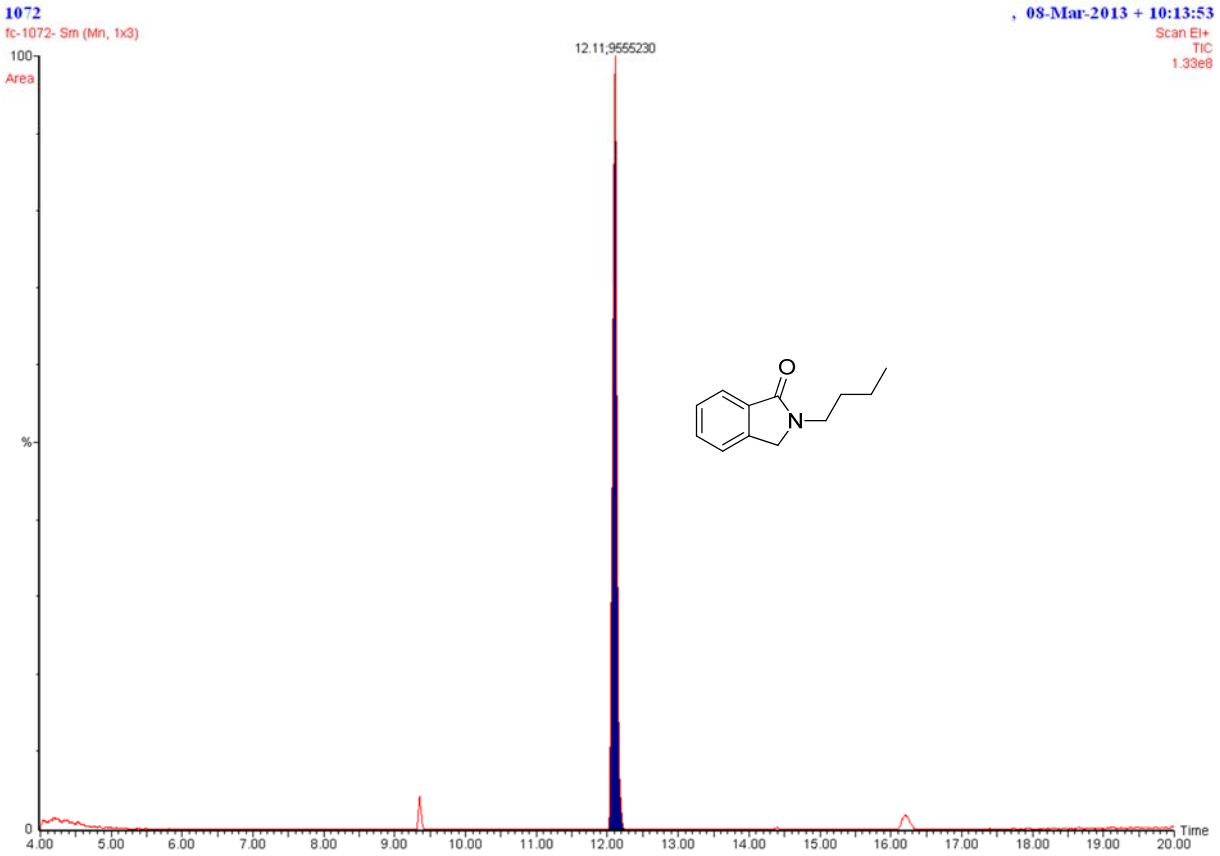
^{1}H NMR (300 MHz) and ^{13}C {^{1}H} NMR (75 MHz) (top) spectra in $CDCl_3$, and low and high resolution DCI(CH_4)-MS (bottom) for **a32**

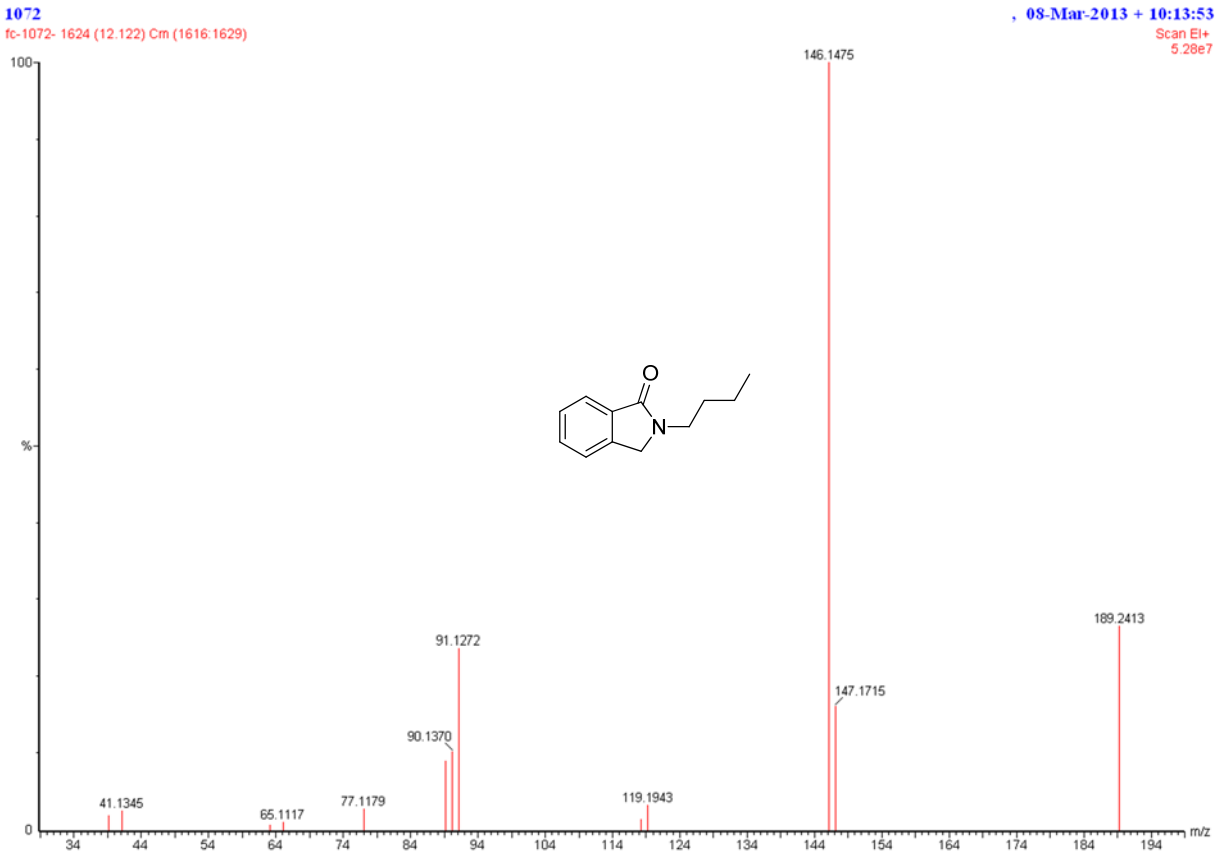

1081-
fc-1081-3 Sm (Mn, 1x3)
, 22-Mar-2013 + 13:00:36
Scan EI+
TIC
2.90e8
100
Area
%
0
16.63;94161464
15.71
5763
16.20
8720
17.23
83606
O
O
N
O
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

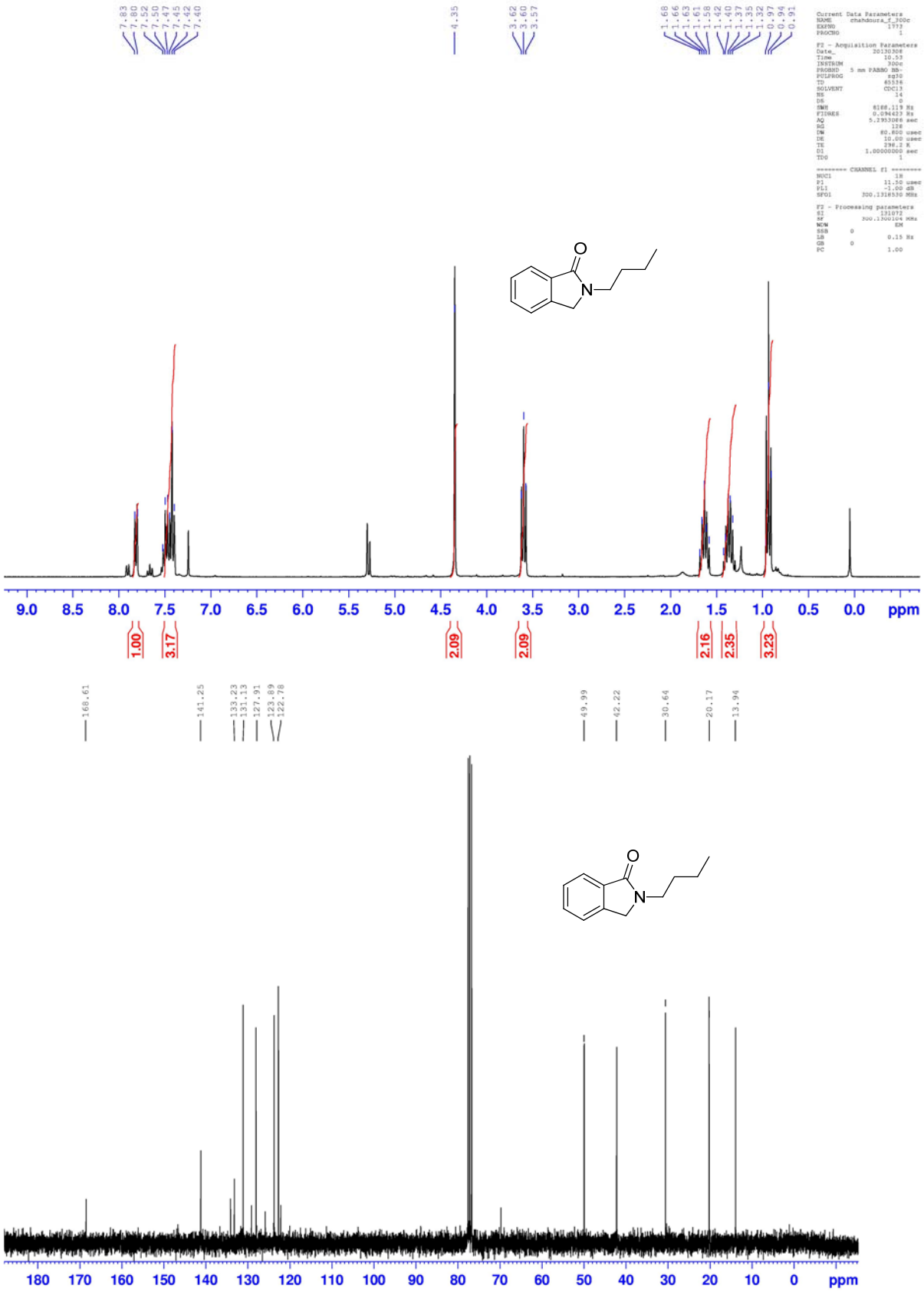

1081-
fc-1081-3 2511 (16.558) Cm (2459:2511)
, 22-Mar-2013 + 13:00:36
Scan EI+
3.59e7
100
%
0
135.1630
267.2182
119.1605
77.1207
91.1403
266.2414
51.1204
118.2153
136.1571
105.0301
65.0883
146.1688
268.2392
44.1197
38.1269
152.1741
180.2233
207.1565
193.1524
220.1853
237.2495
248.2445
281.1565
341.1645
355.2200
O
O
N
O
35
55
75
95
115
135
155
175
195
215
235
255
275
295
315
335
355
m/z



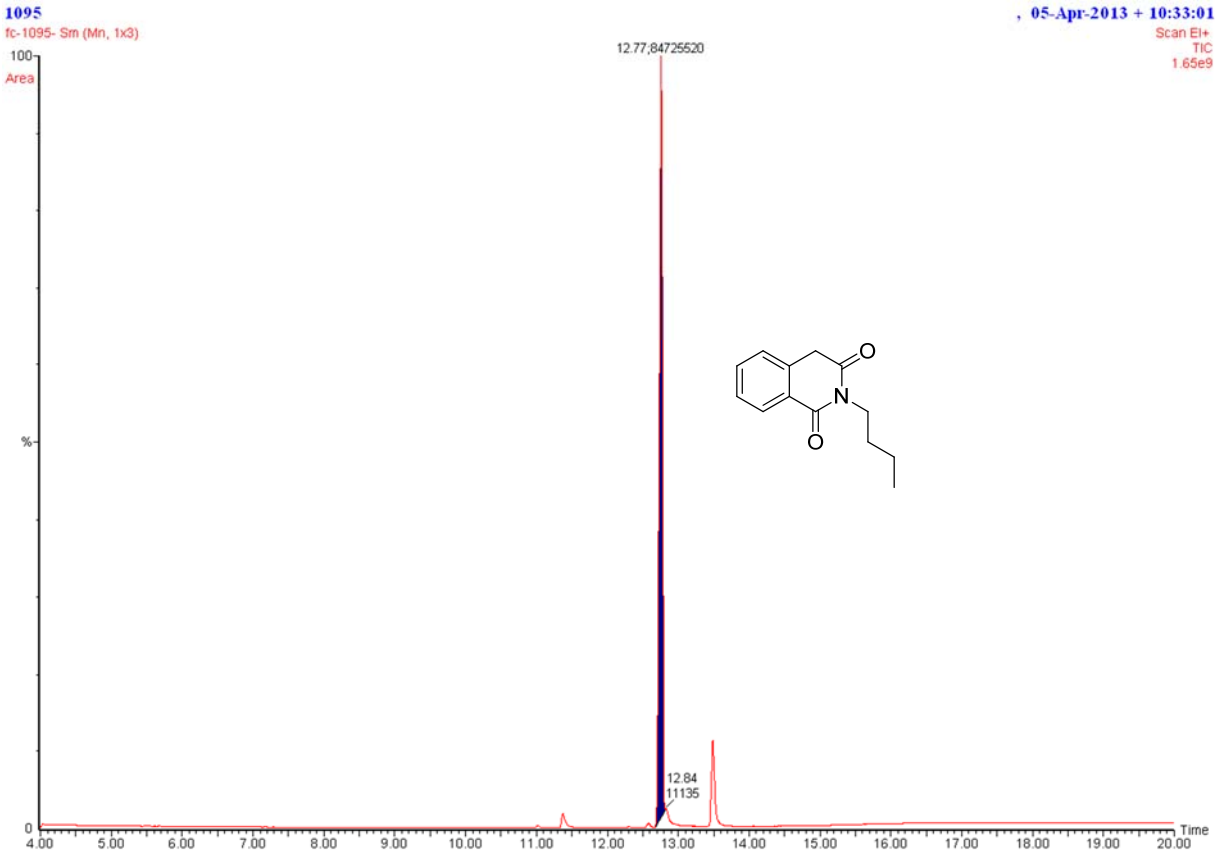
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **h8**

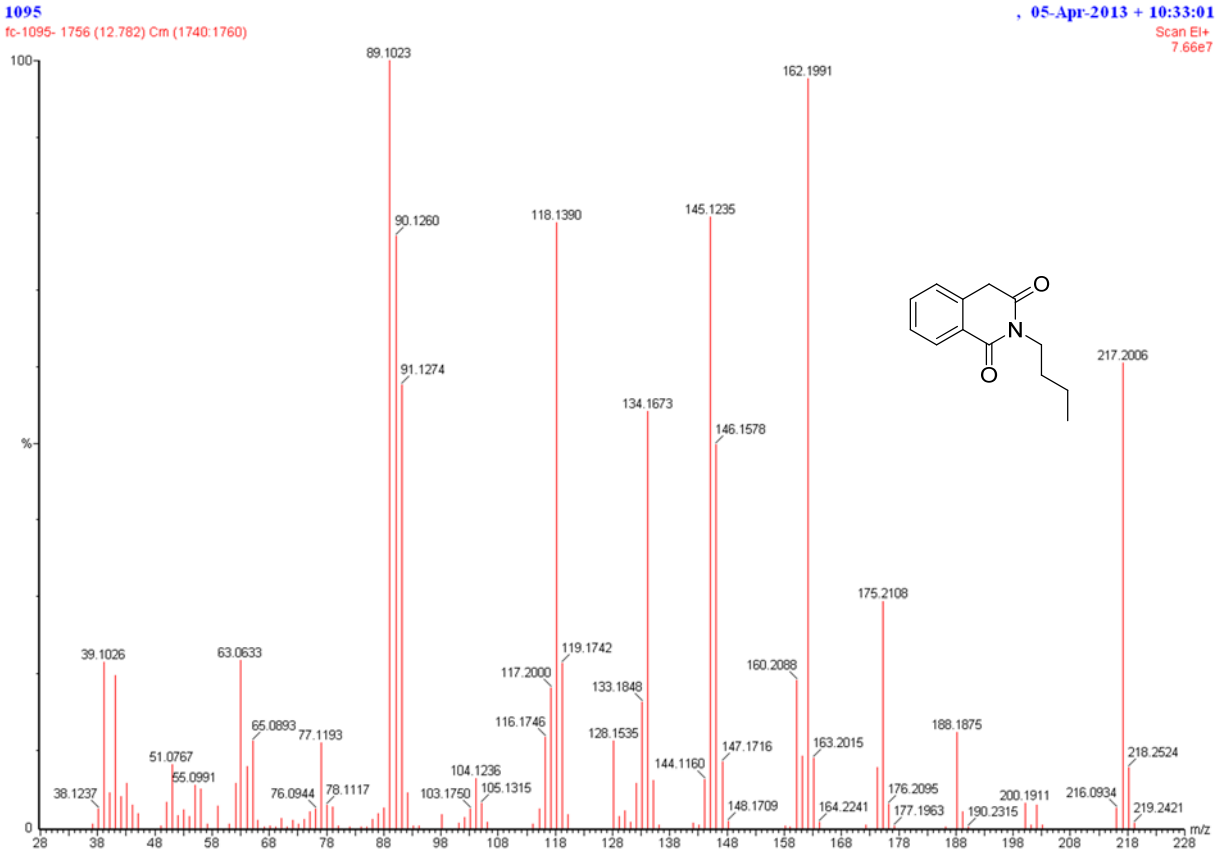

1072
fc-1072- Sm (Mn, 1x3)
, 08-Mar-2013 + 10:13:53
Scan EI+
TIC
1.33e8
12.11;9555230
Area
%
Time
O
N

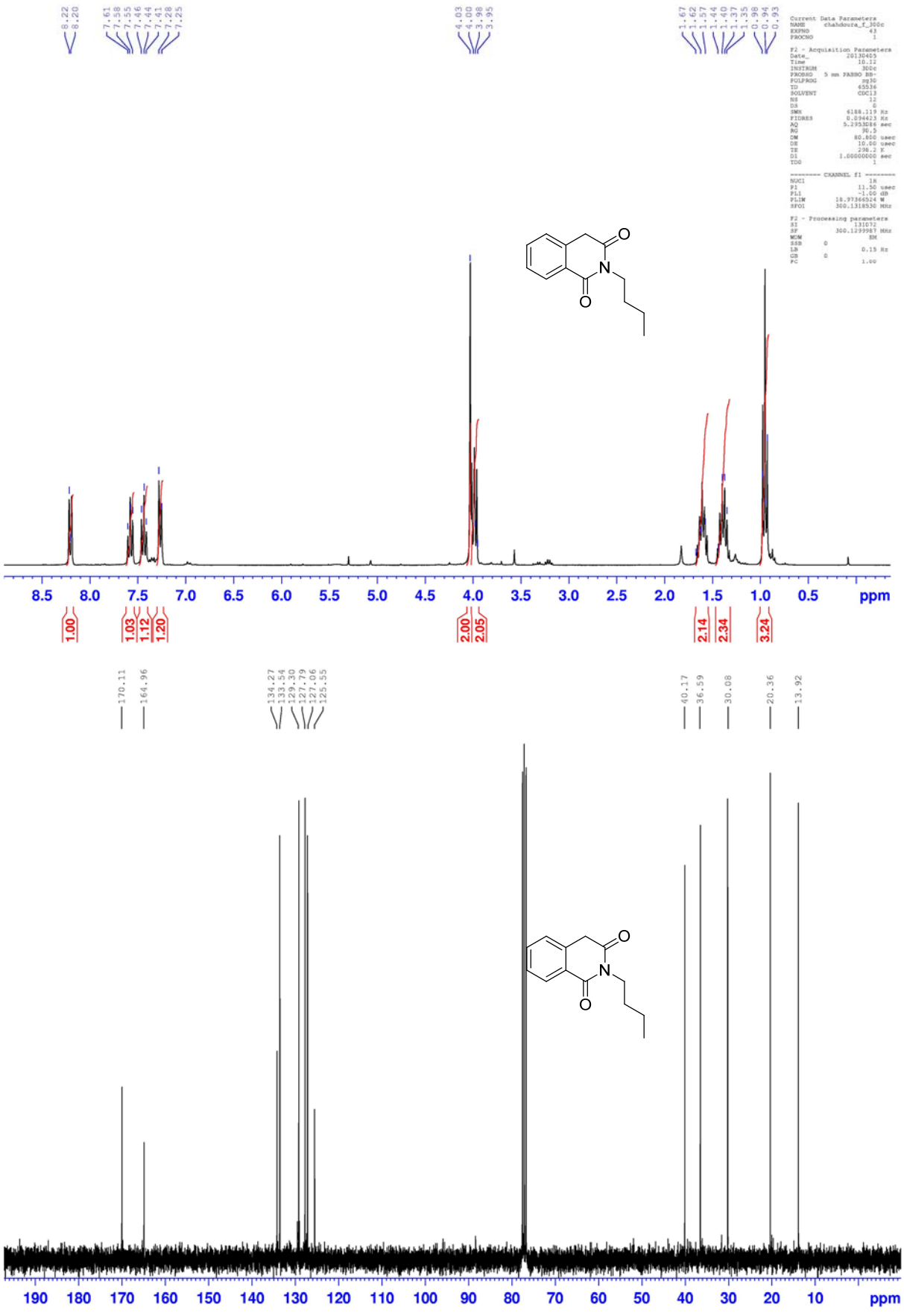

1072
fc-1072- 1624 (12.122) Cm (1616:1629)
, 08-Mar-2013 + 10:13:53
Scan EI+
5.28e7
146.1475
189.2413
91.1272
147.1715
90.1370
41.1345
65.1117
77.1179
119.1943
%
m/z
O
N



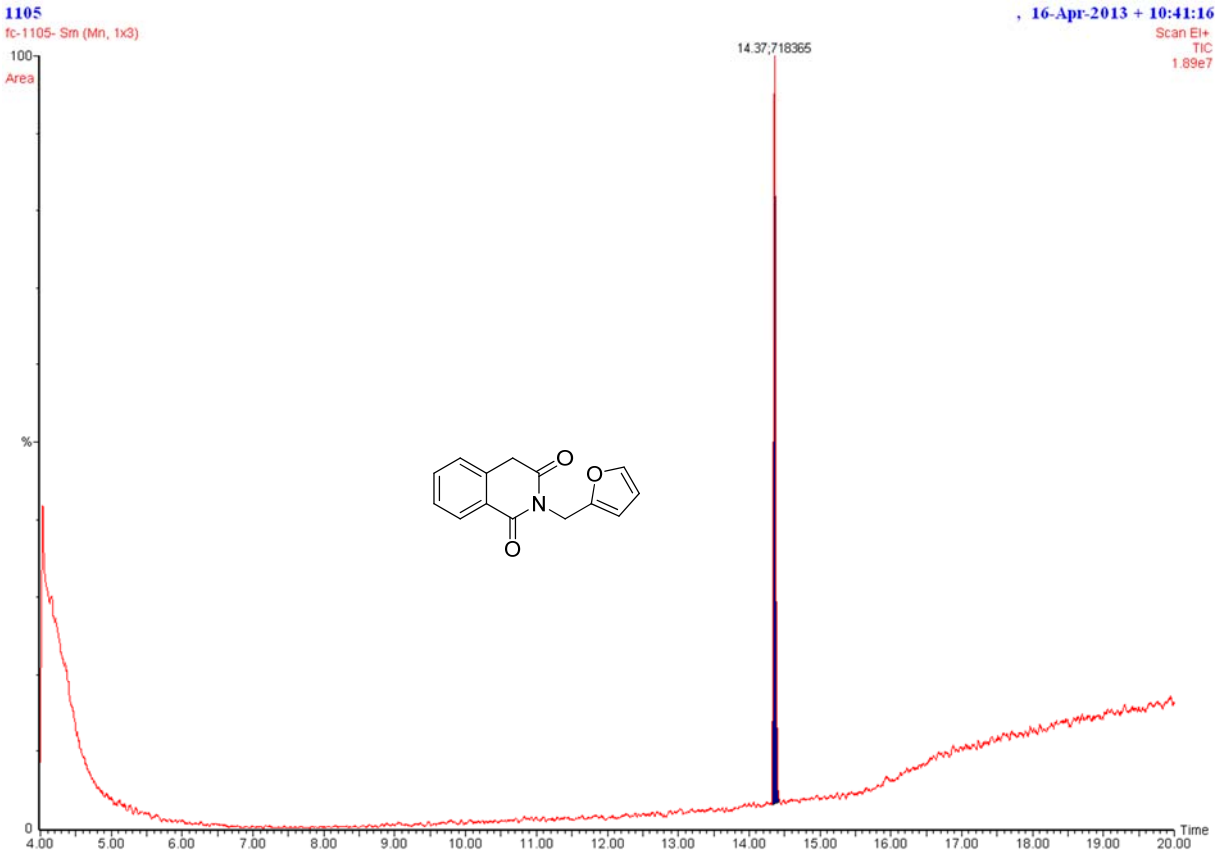
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **i9**

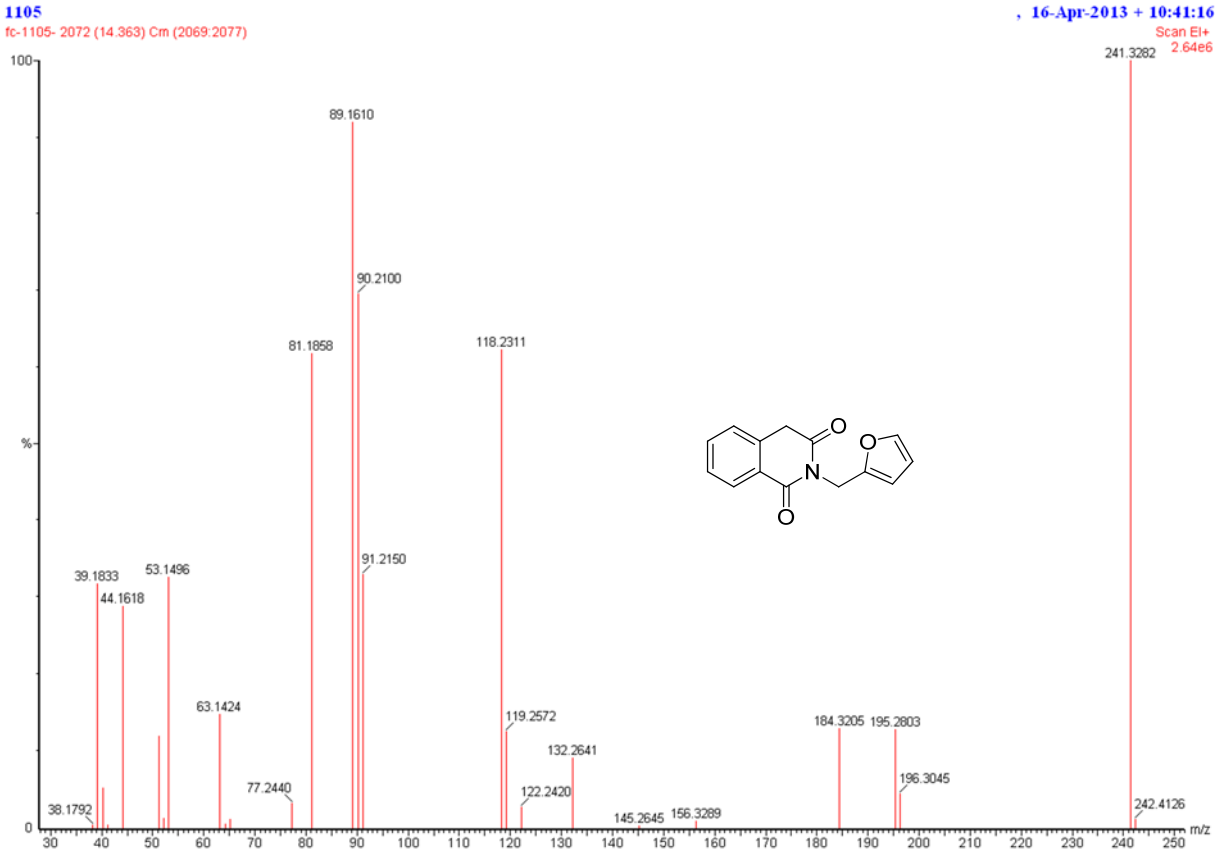

1095
fc-1095- Sm (Mn, 1x3)
, 05-Apr-2013 + 10:33:01
Scan EI+
TIC
1.65e9
100
Area
%
0
12.77,84725520
12.84
11135
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N
O

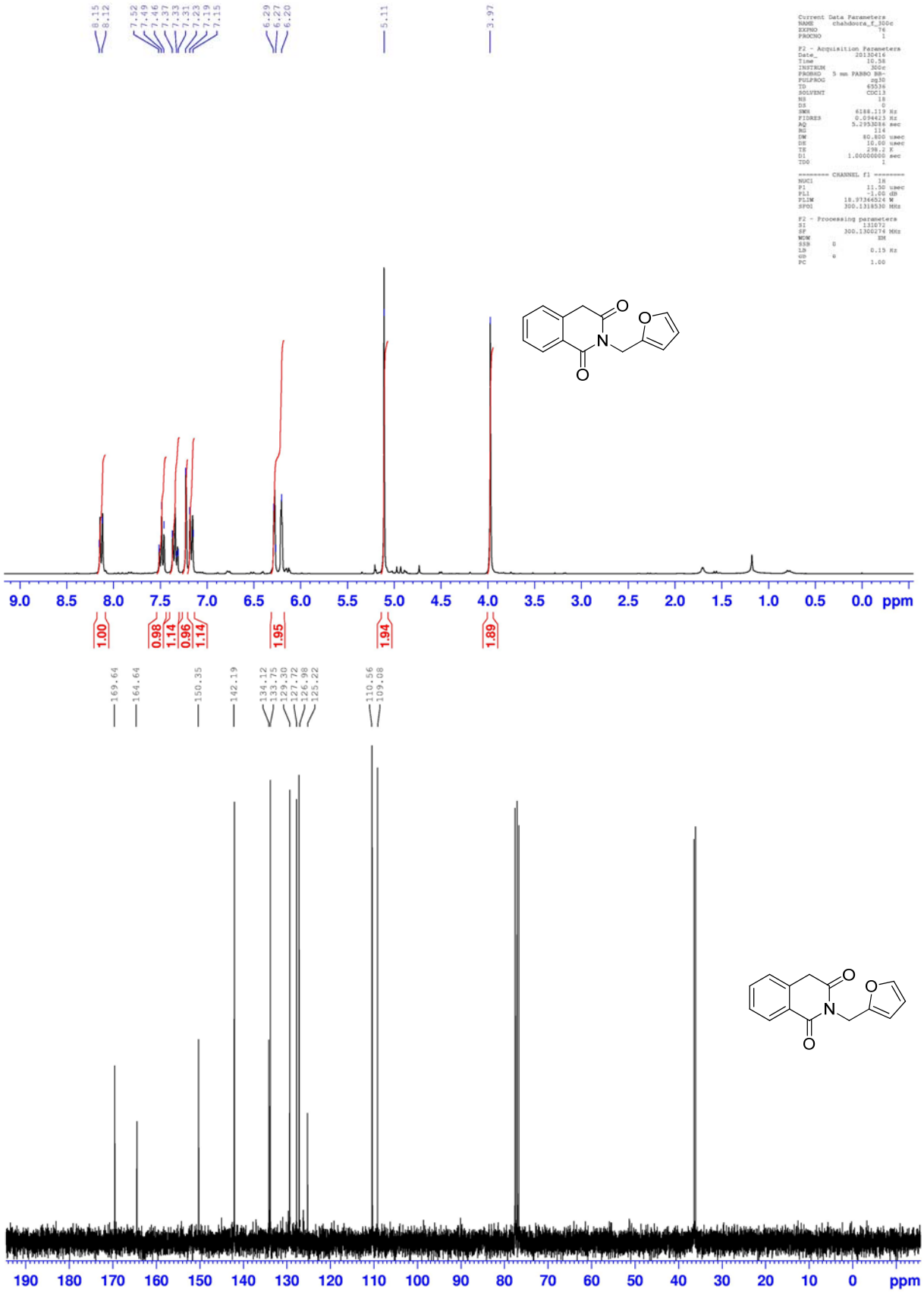

1095
fc-1095- 1756 (12.782) Cm (1740:1760)
, 05-Apr-2013 + 10:33:01
Scan EI+
7.66e7
100
%
0
89.1023
162.1991
90.1260
118.1390
145.1235
91.1274
134.1673
146.1578
217.2006
175.2108
39.1026
63.0633
119.1742
117.2000
160.2088
133.1848
116.1746
65.0693
77.1193
128.1535
188.1875
147.1716
163.2015
51.0767
55.0991
104.1236
144.1160
218.2524
38.1237
76.0944
78.1117
103.1750
105.1315
176.2095
200.1911
216.0934
148.1709
164.2241
177.1963
190.2315
219.2421
28
38
48
58
68
78
88
98
108
118
128
138
148
158
168
178
188
198
208
218
228
m/z
O
N
O



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **j9**

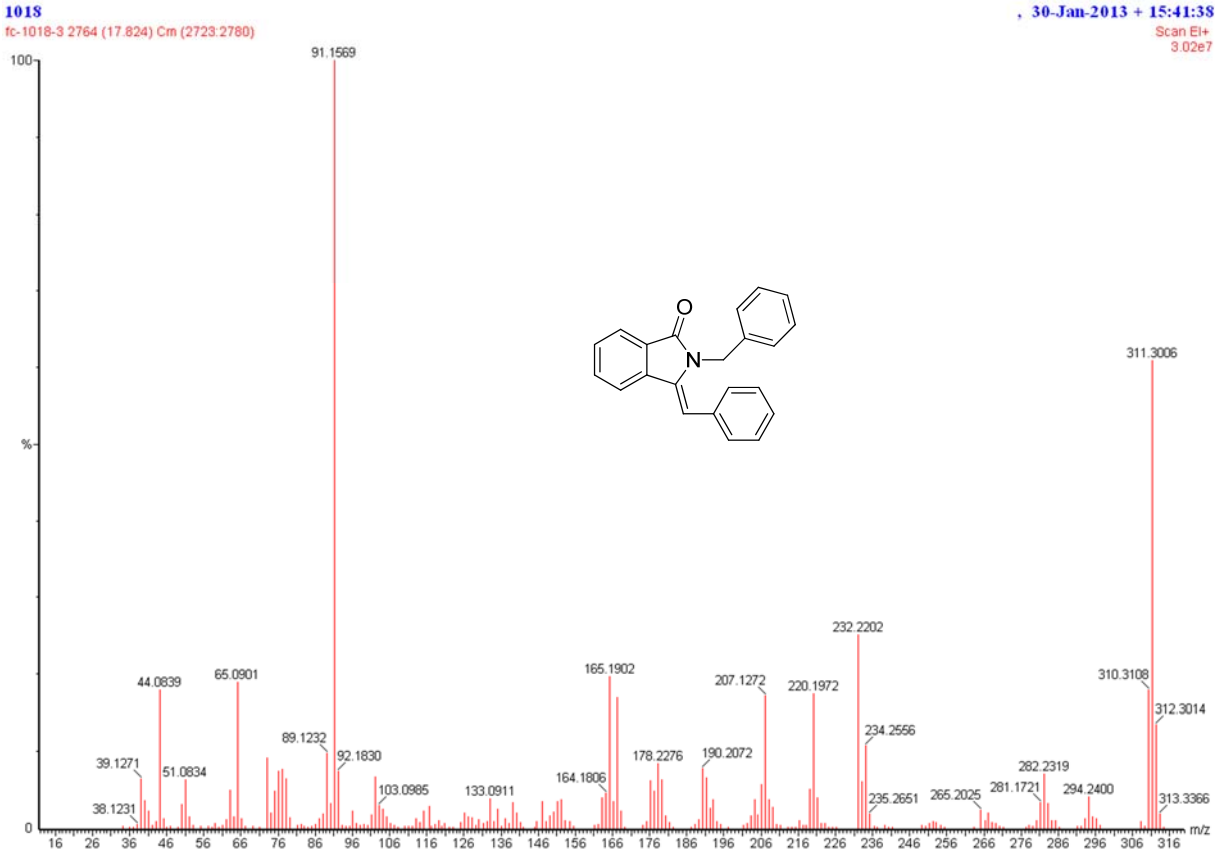

1105
fc-1105- Sm (Mn, 1x3)
, 16-Apr-2013 + 10:41:16
Scan EI+
TIC
1.89e7
Area
14.37;718365
%
Time


1105
fc-1105- 2072 (14.363) Cm (2069:2077)
, 16-Apr-2013 + 10:41:16
Scan EI+
2.64e6
%
m/z
241.3282
89.1610
90.2100
81.1858
118.2311
91.2150
39.1833
53.1496
44.1618
63.1424
119.2572
132.2641
184.3205
195.2803
196.3045
77.2440
122.2420
242.4126
38.1792
145.2645
156.3289



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **k24**


1018
, 30-Jan-2013 + 15:41:38
fc-1018-3 Sm (Mn, 1x3)
Scan EI+
TIC
2.69e8
Area
100
%
0
17.82,52895408
O
N
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time


1018
, 30-Jan-2013 + 15:41:38
fc-1018-3 2764 (17.824) Cm (2723:2780)
Scan EI+
3.02e7
100
%
0
91.1569
311.3006
232.2202
165.1902
44.0839
65.0901
207.1272
220.1972
310.3108
312.3014
89.1232
92.1830
234.2556
39.1271
51.0834
178.2276
190.2072
282.2319
164.1806
103.0985
133.0911
281.1721
294.2400
313.3366
38.1231
235.2651
265.2025
O
N
16
26
36
46
56
66
76
86
96
106
116
126
136
146
156
166
176
186
196
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216
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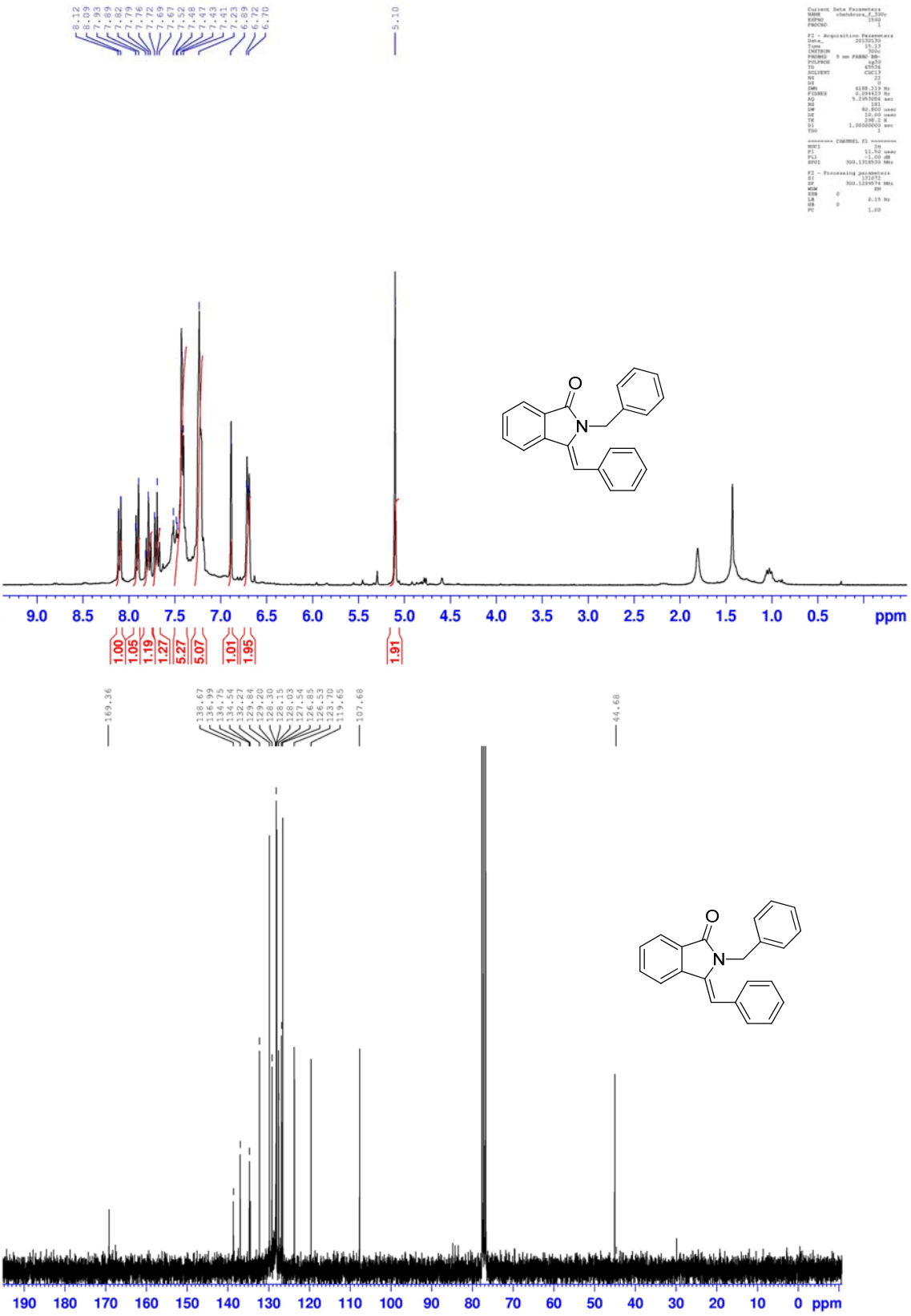
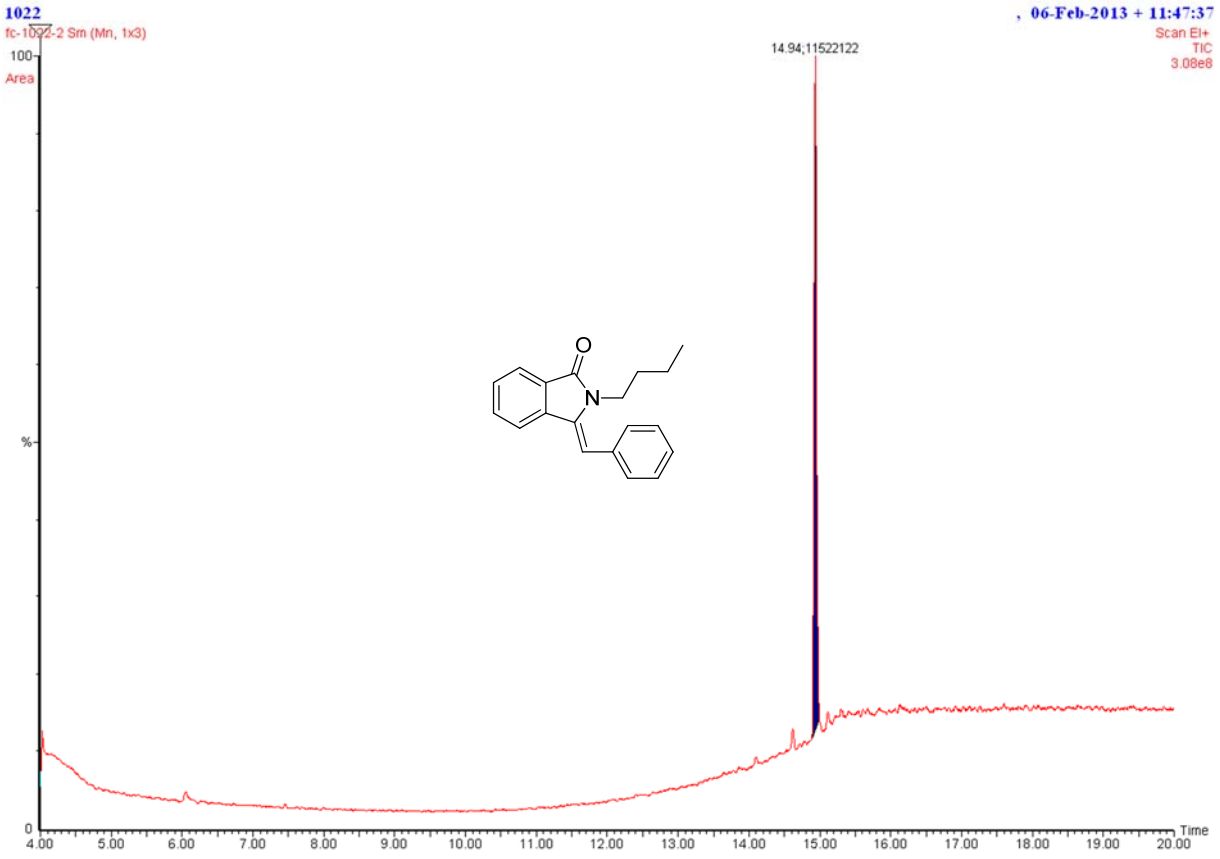
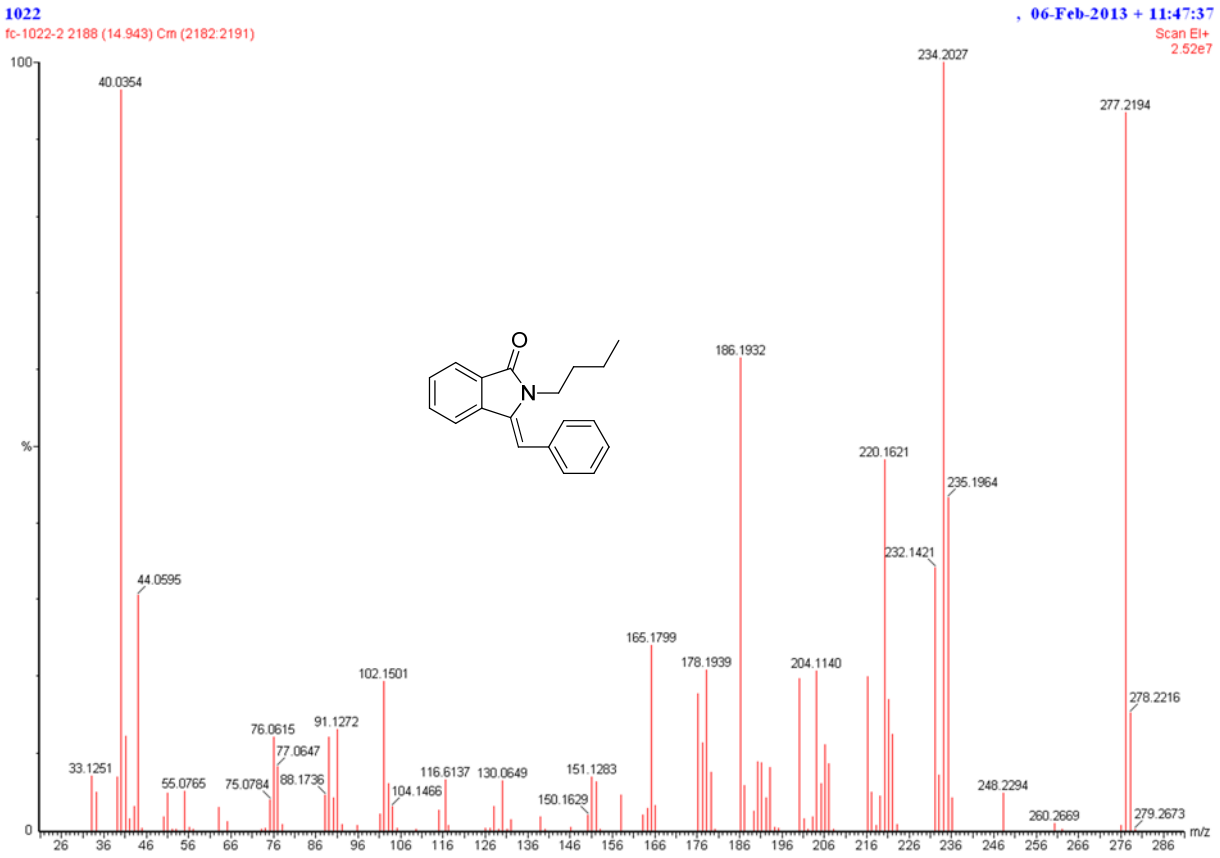
236
246
256
266
276
286
296
306
316
m/z



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **b33**


1022
fc-1022-2 Sm (Mn, 1x3)
, 06-Feb-2013 + 11:47:37
Scan EI+
TIC
3.08e8
Area
100
%
0
14.94;11522122
O
N
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time


1022
fc-1022-2 2188 (14.943) Cm (2182:2191)
, 06-Feb-2013 + 11:47:37
Scan EI+
2.52e7
100
%
0
O
N
40.0354
44.0595
33.1251
55.0765
75.0784
76.0615
77.0647
88.1736
91.1272
102.1501
104.1466
116.6137
130.0649
150.1629
151.1283
165.1799
178.1939
186.1932
204.1140
220.1621
232.1421
234.2027
235.1964
248.2294
260.2669
277.2194
278.2216
279.2673
26
36
46
56
66
76
86
96
106
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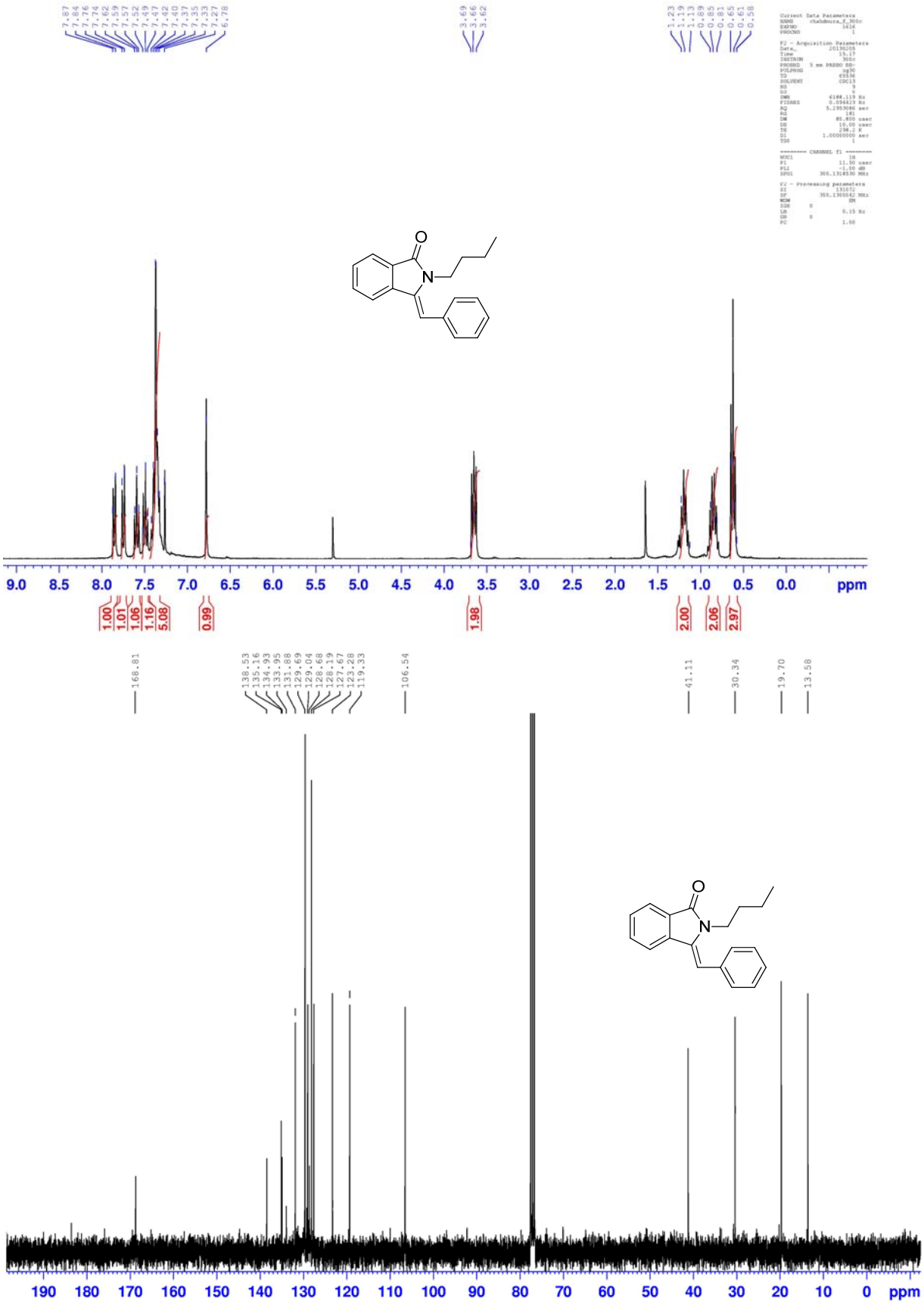
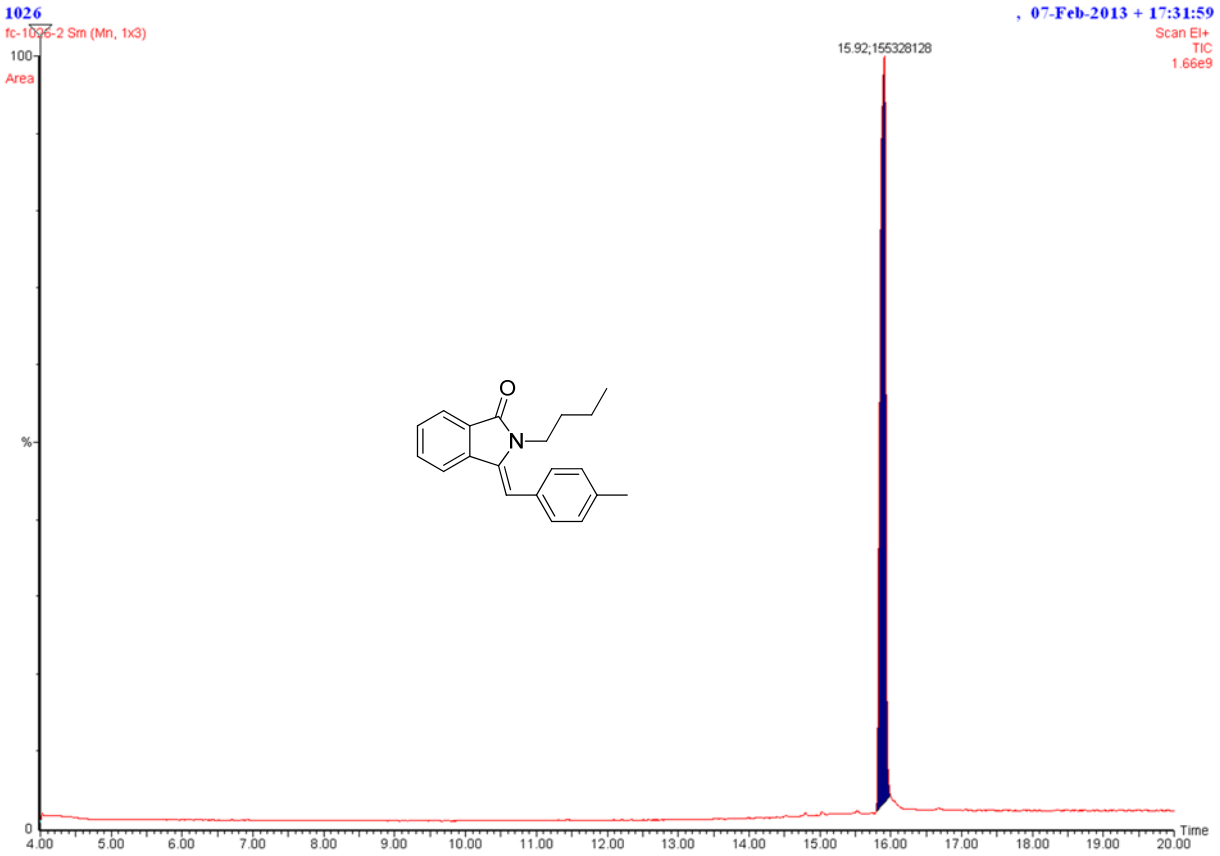
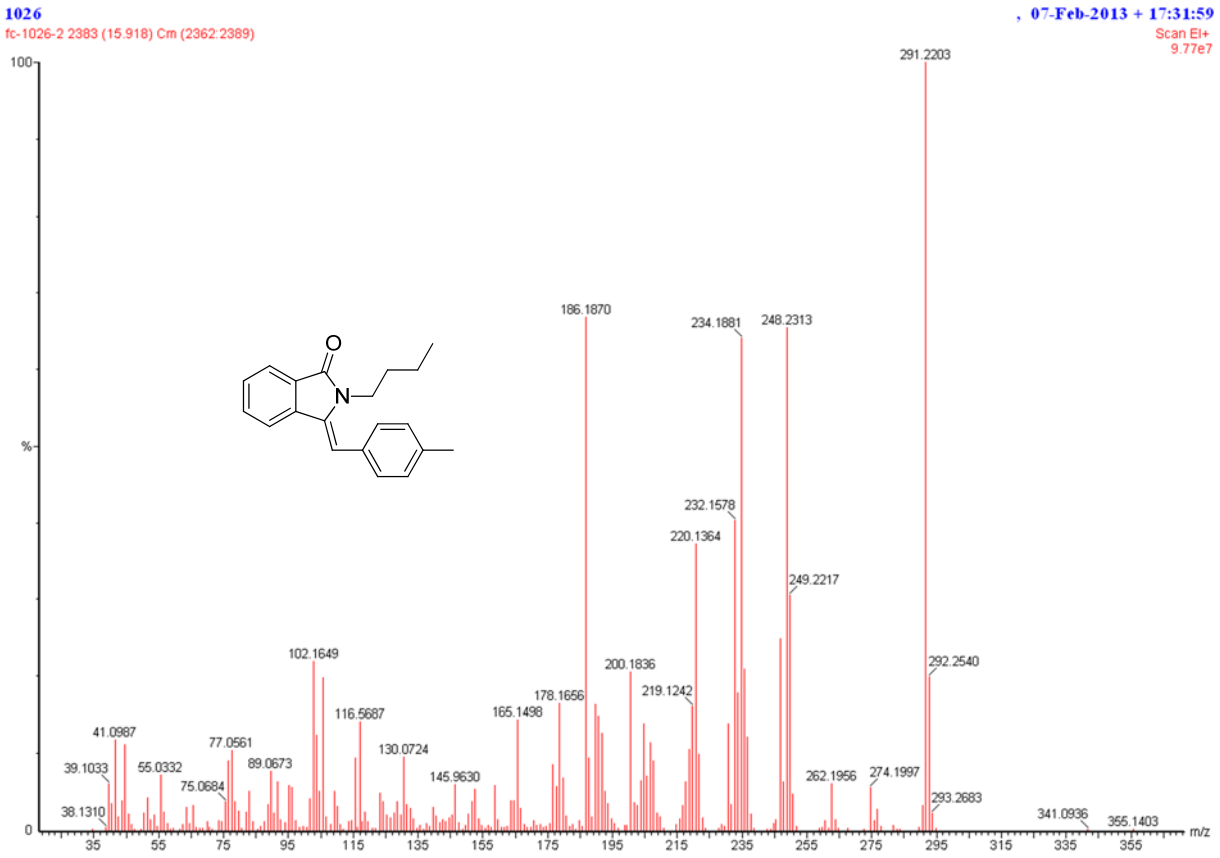
216
226
236
246
256
266
276
286
m/z



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **I33**


1026
, 07-Feb-2013 + 17:31:59
fc-1026-2 Sm (Mn, 1x3)
Scan EI+
TIC
1.66e9
100
Area
%
0
15.92;155328128
O
N
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time


1026
, 07-Feb-2013 + 17:31:59
fc-1026-2 2383 (15.918) Cm (2362:2389)
Scan EI+
9.77e7
100
%
0
O
N
291.2203
186.1870
234.1881
248.2313
232.1578
220.1364
249.2217
102.1649
200.1836
292.2540
219.1242
178.1656
116.5687
165.1498
130.0724
41.0987
77.0561
89.0673
39.1033
55.0332
145.9630
75.0684
262.1956
274.1997
293.2683
38.1310
341.0936
355.1403
35
55
75
95
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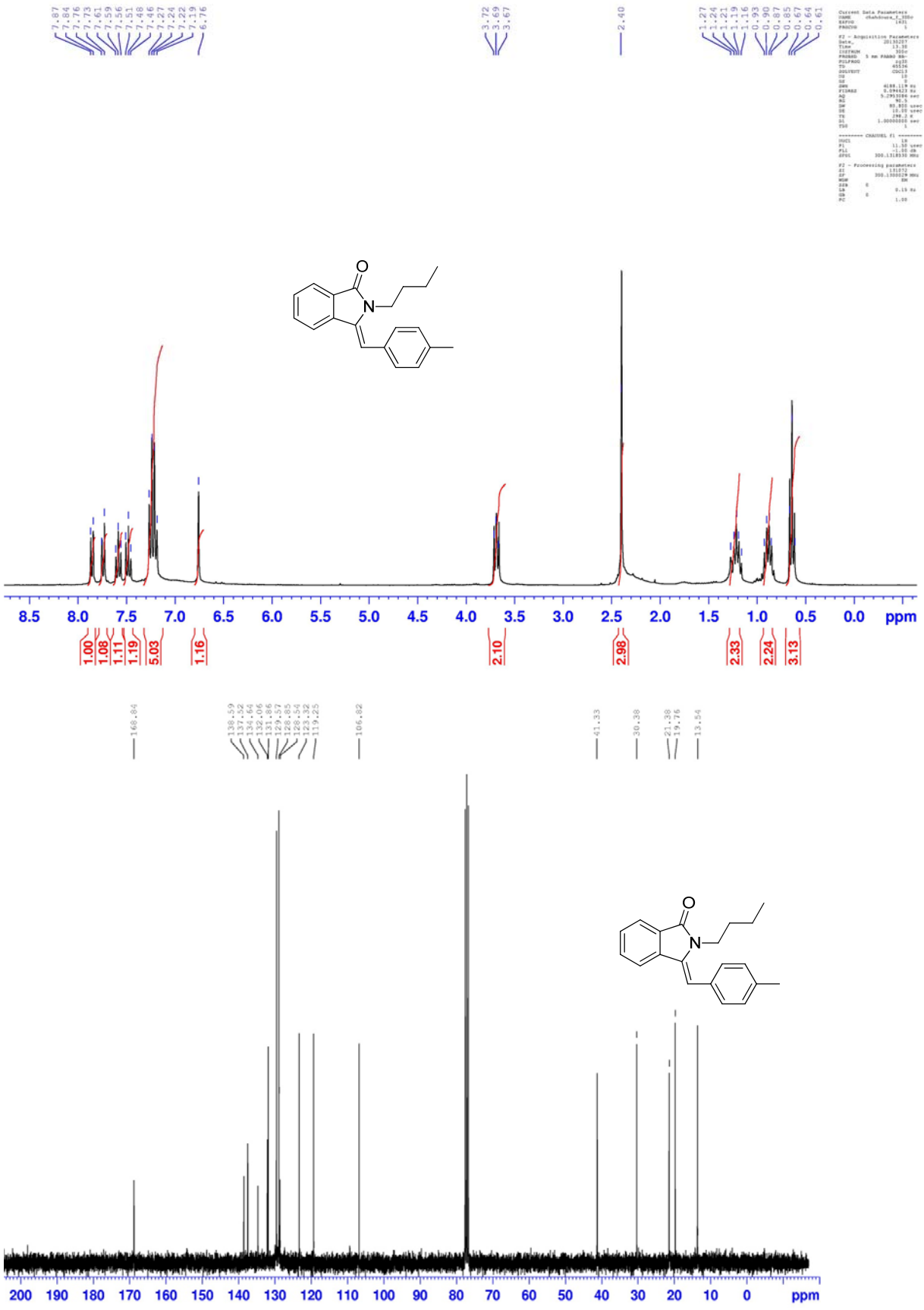
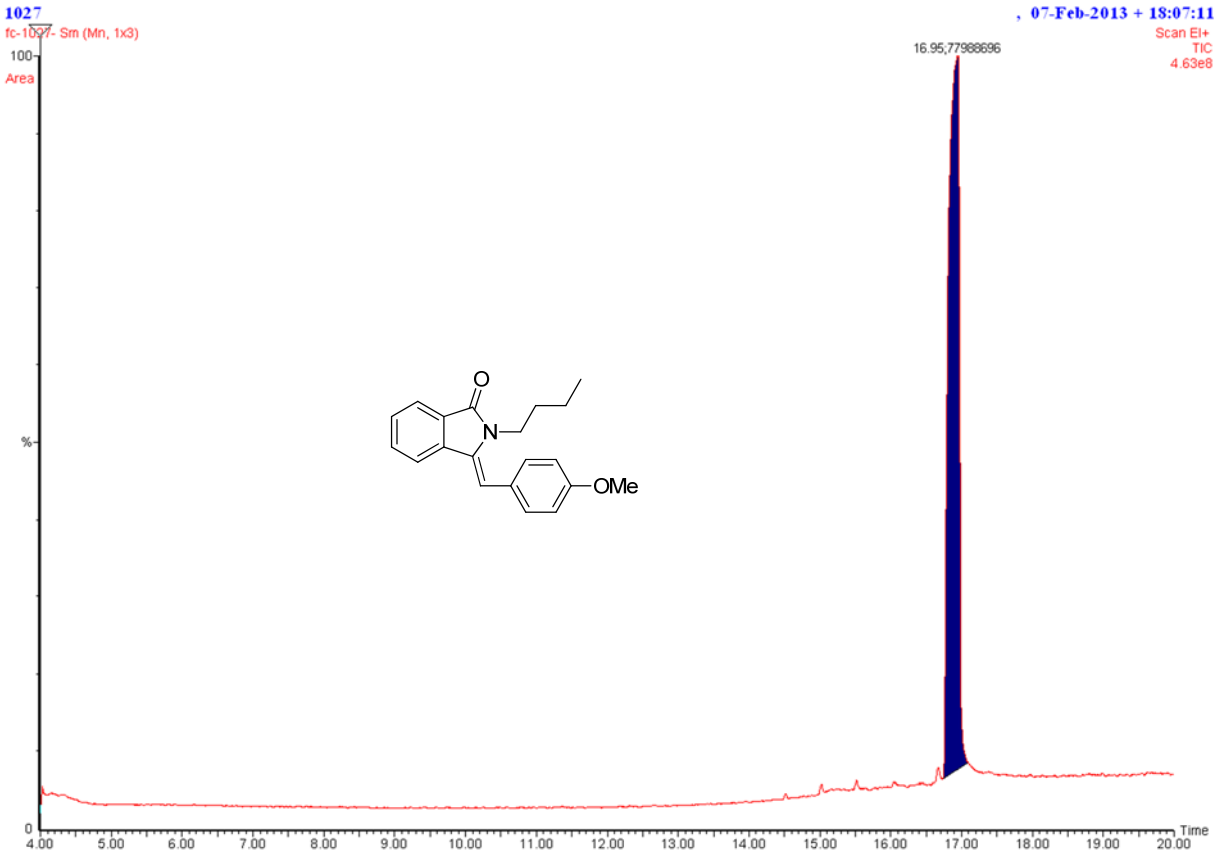
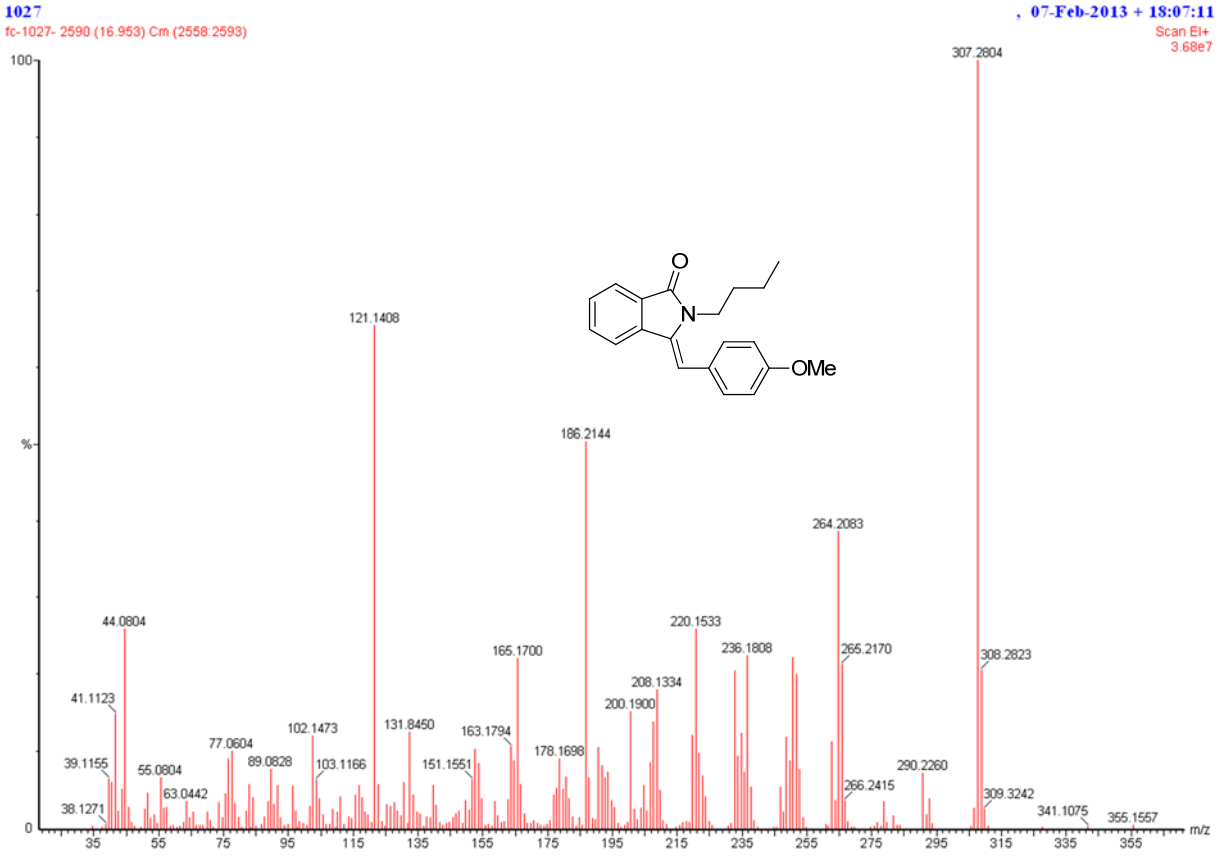
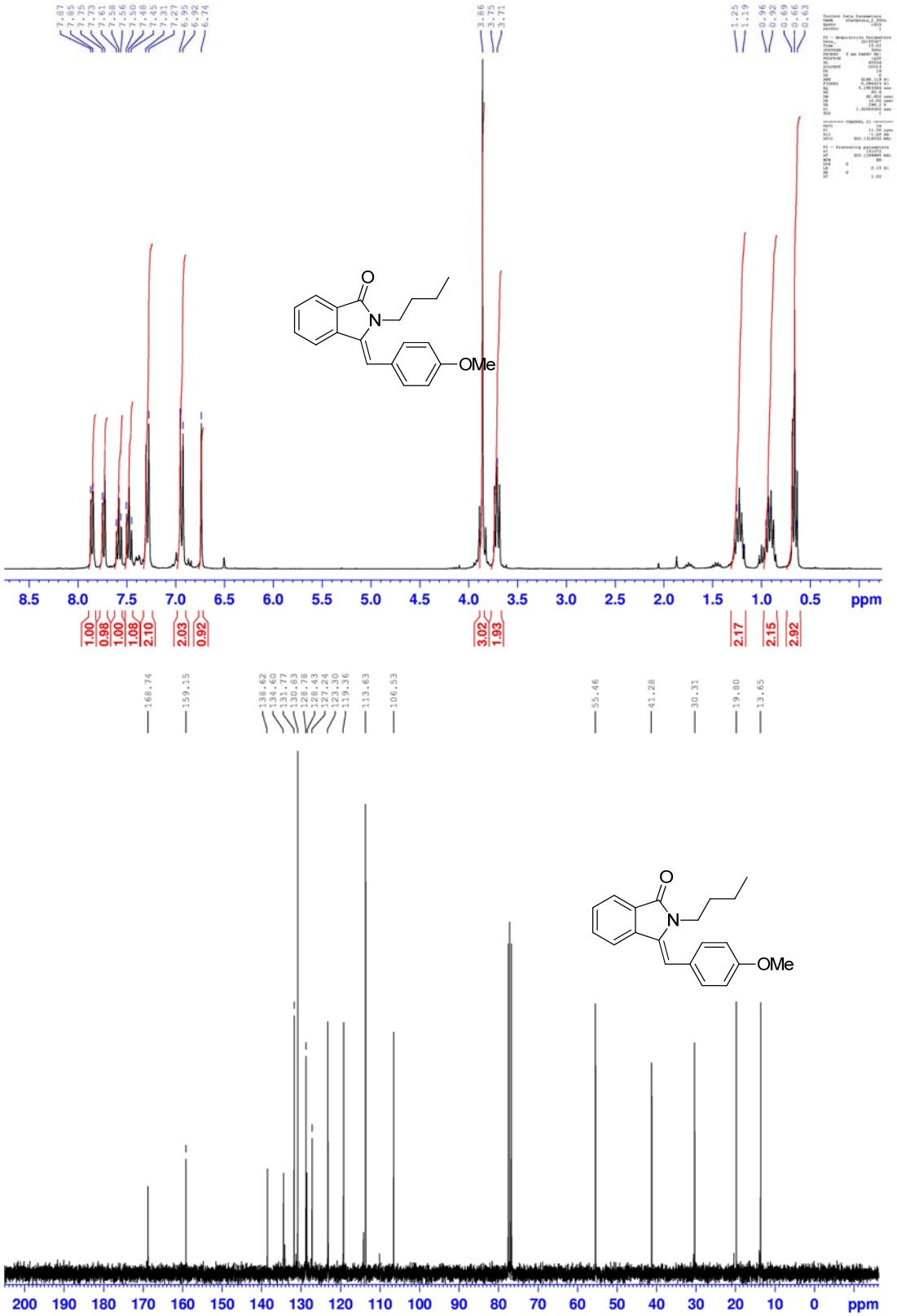
215
235
255
275
295
315
335
355
m/z



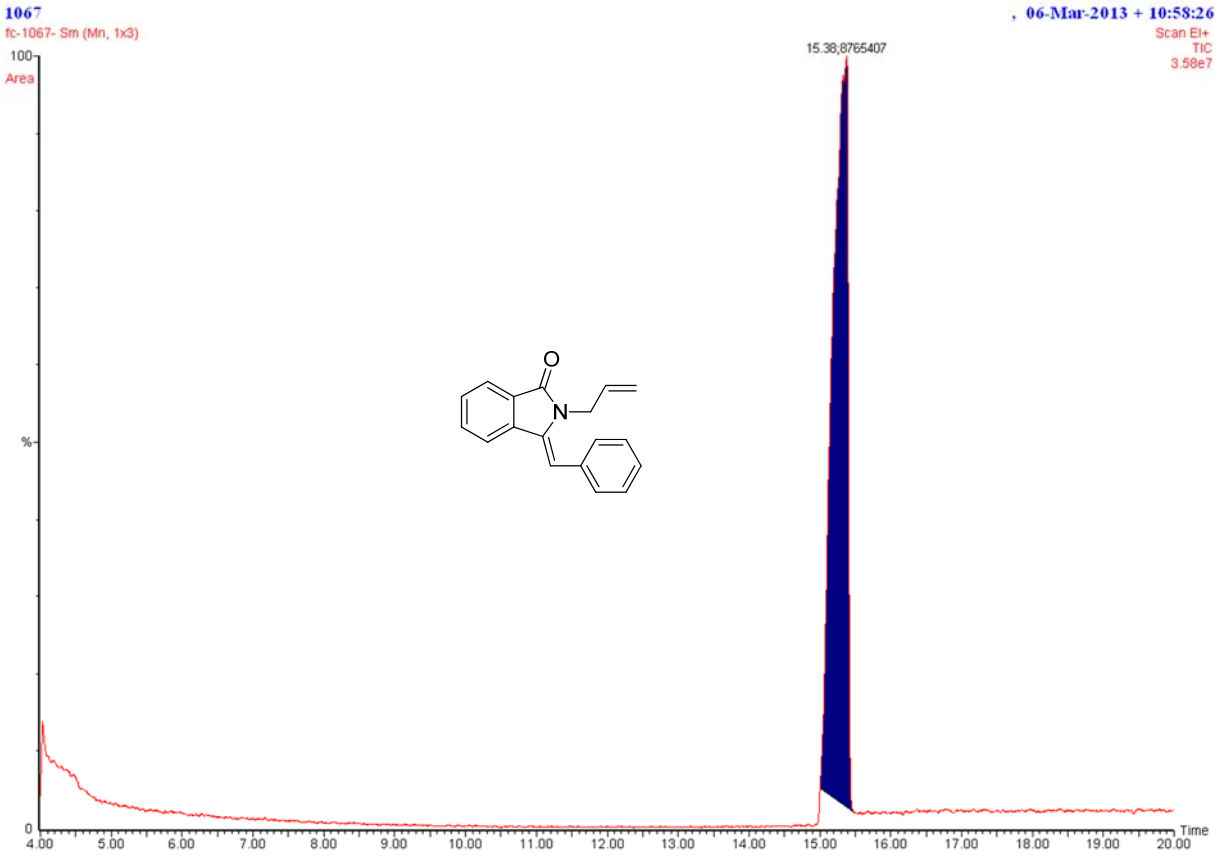
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **l34**

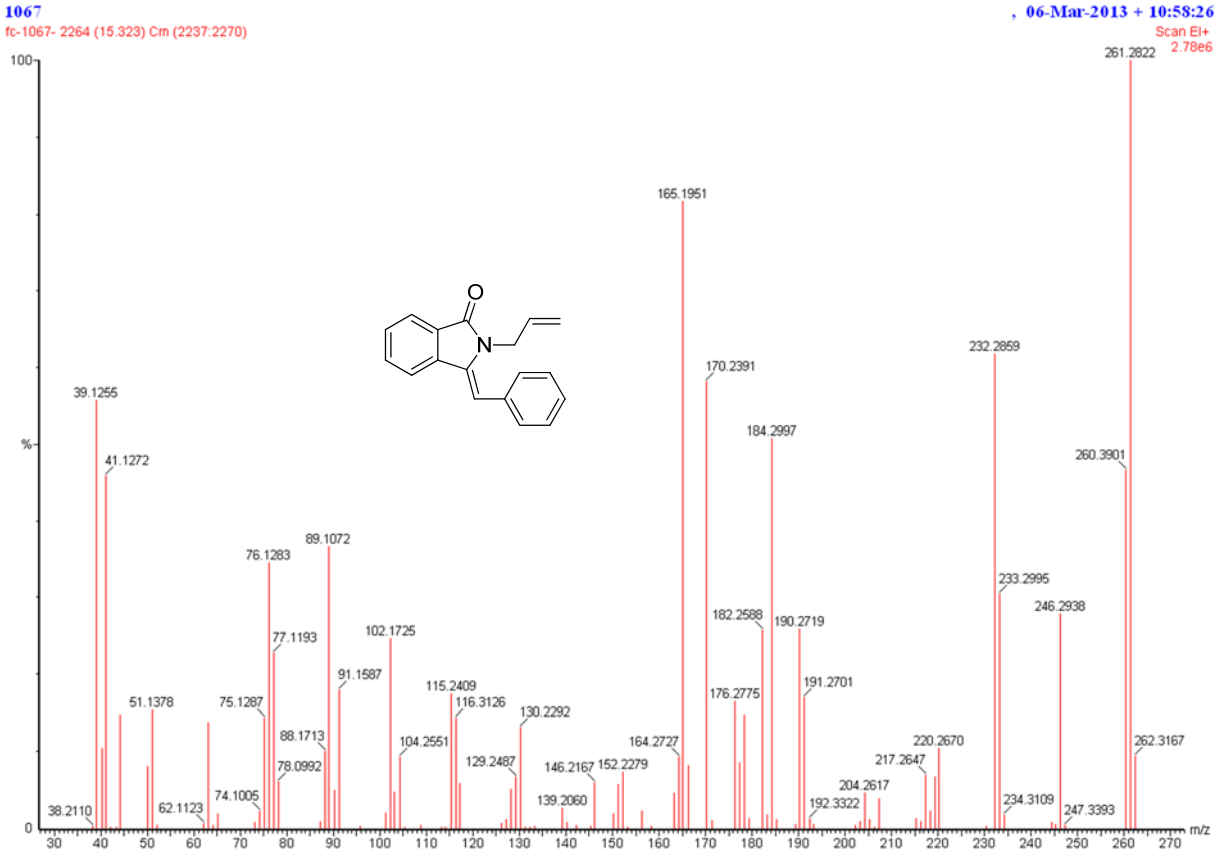

1027
fc-1027- Sm (Mn, 1x3)
, 07-Feb-2013 + 18:07:11
Scan EI+
TIC
4.63e8
Area
16.95,77988696
O
N
OMe
%
Time

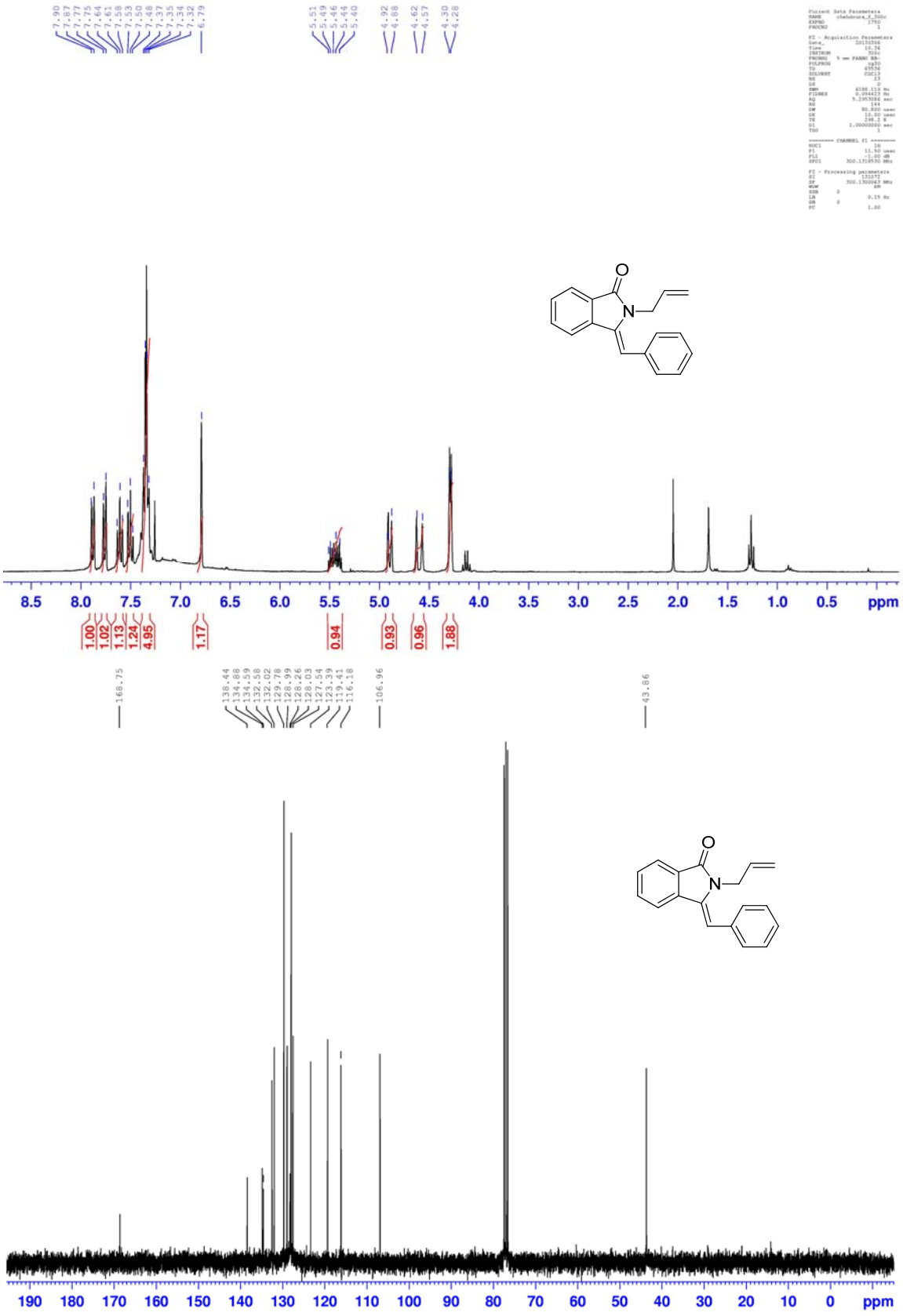

1027
fc-1027- 2590 (16.953) Cm (2558:2593)
, 07-Feb-2013 + 18:07:11
Scan EI+
3.68e7
307.2804
121.1408
186.2144
264.2083
220.1533
44.0804
165.1700
236.1808
265.2170
308.2823
208.1334
41.1123
200.1900
131.8450
102.1473
163.1794
77.0604
178.1698
290.2260
89.0828
39.1155
55.0804
103.1166
151.1551
266.2415
309.3242
63.0442
38.1271
341.1075
355.1557
O
N
OMe
%
m/z



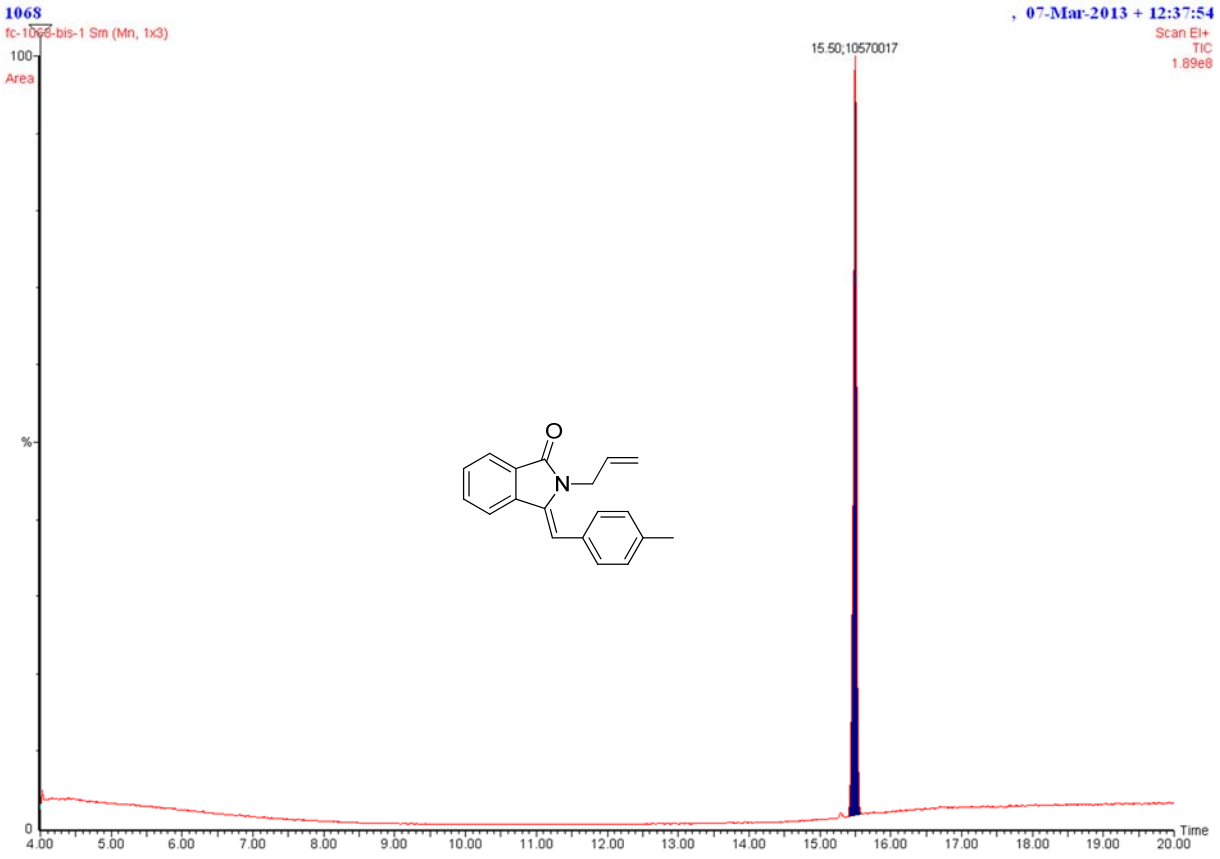
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **l35**

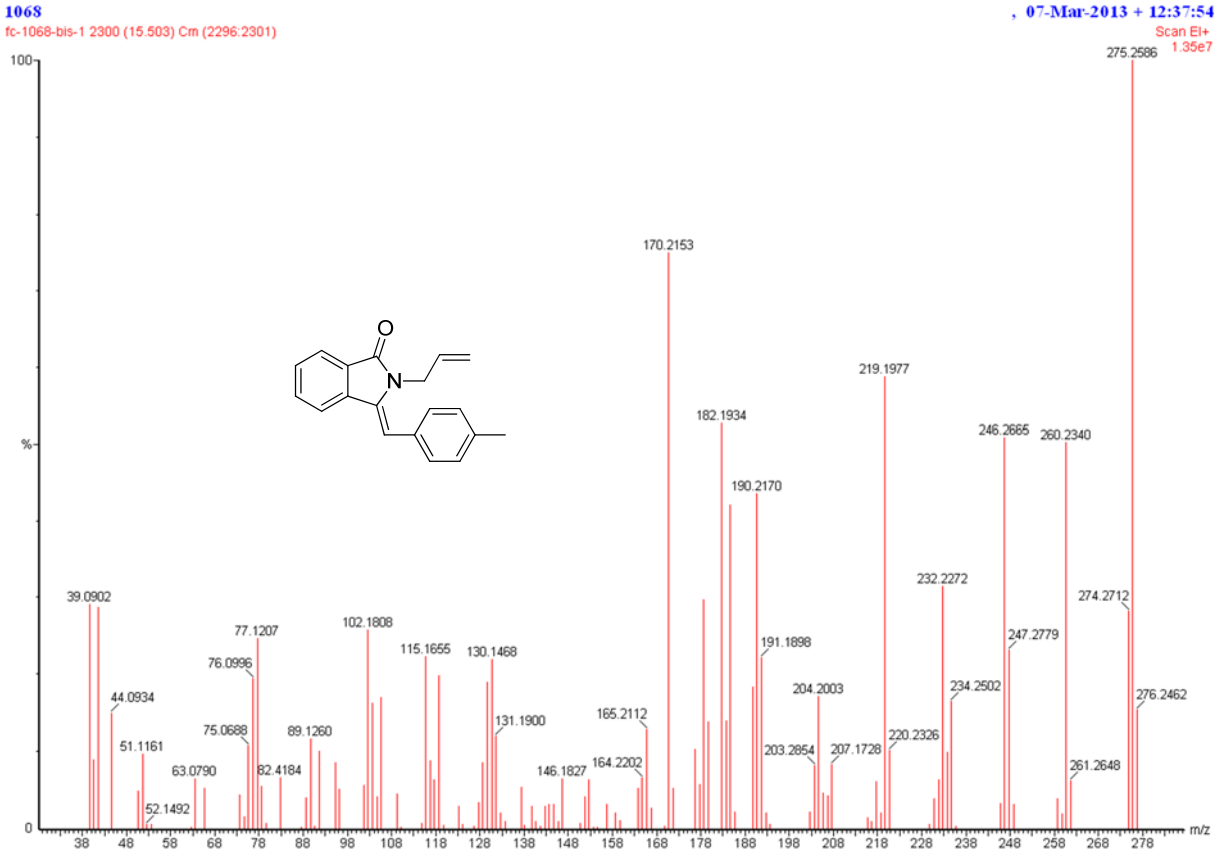

1067
fc-1067- Sm (Mn, 1x3)
, 06-Mar-2013 + 10:58:26
Scan EI+
TIC
3.58e7
15.38;8765407
100
Area
%
0
Time
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
O
N

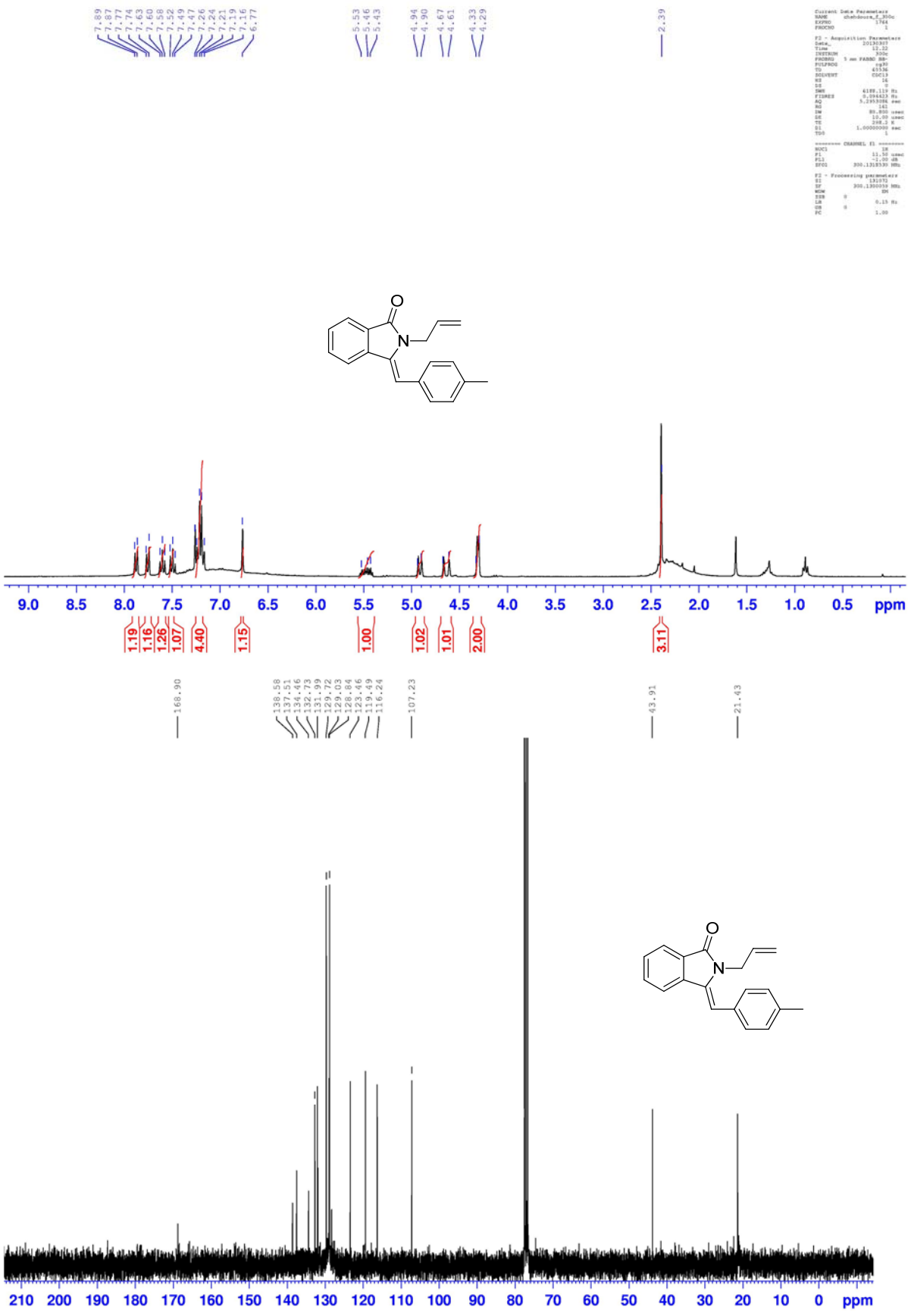

1067
fc-1067- 2264 (15.323) Cm (2237:2270)
, 06-Mar-2013 + 10:58:26
Scan EI+
2.78e6
100
%
0
m/z
261.2822
165.1951
232.2859
170.2391
39.1255
184.2997
260.3901
41.1272
89.1072
76.1283
233.2995
246.2938
182.2588
190.2719
102.1725
77.1193
191.2701
91.1587
115.2409
116.3126
176.2775
51.1378
75.1287
130.2292
262.3167
88.1713
104.2551
164.2727
220.2670
129.2487
78.0992
146.2167
152.2279
217.2647
204.2617
74.1005
139.2060
234.3109
38.2110
62.1123
192.3322
247.3393
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
O
N



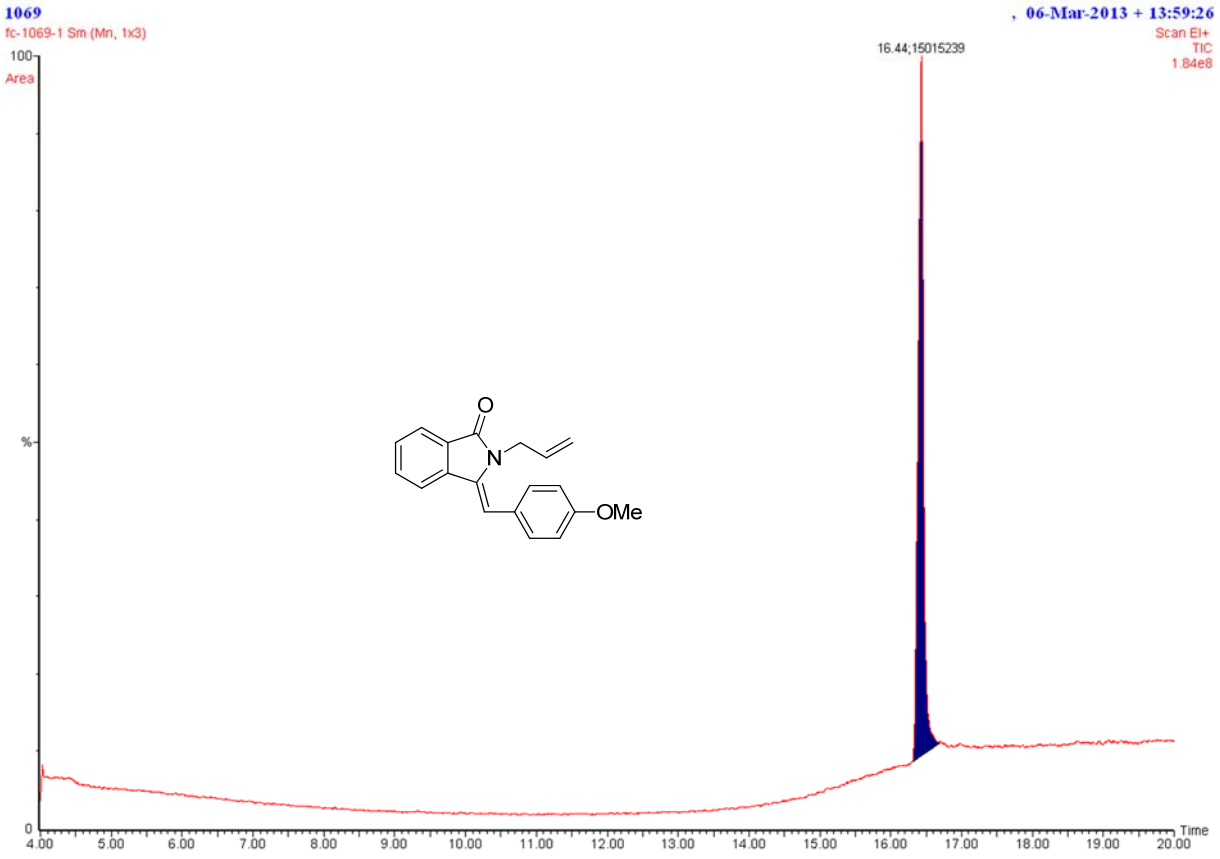
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **m33**

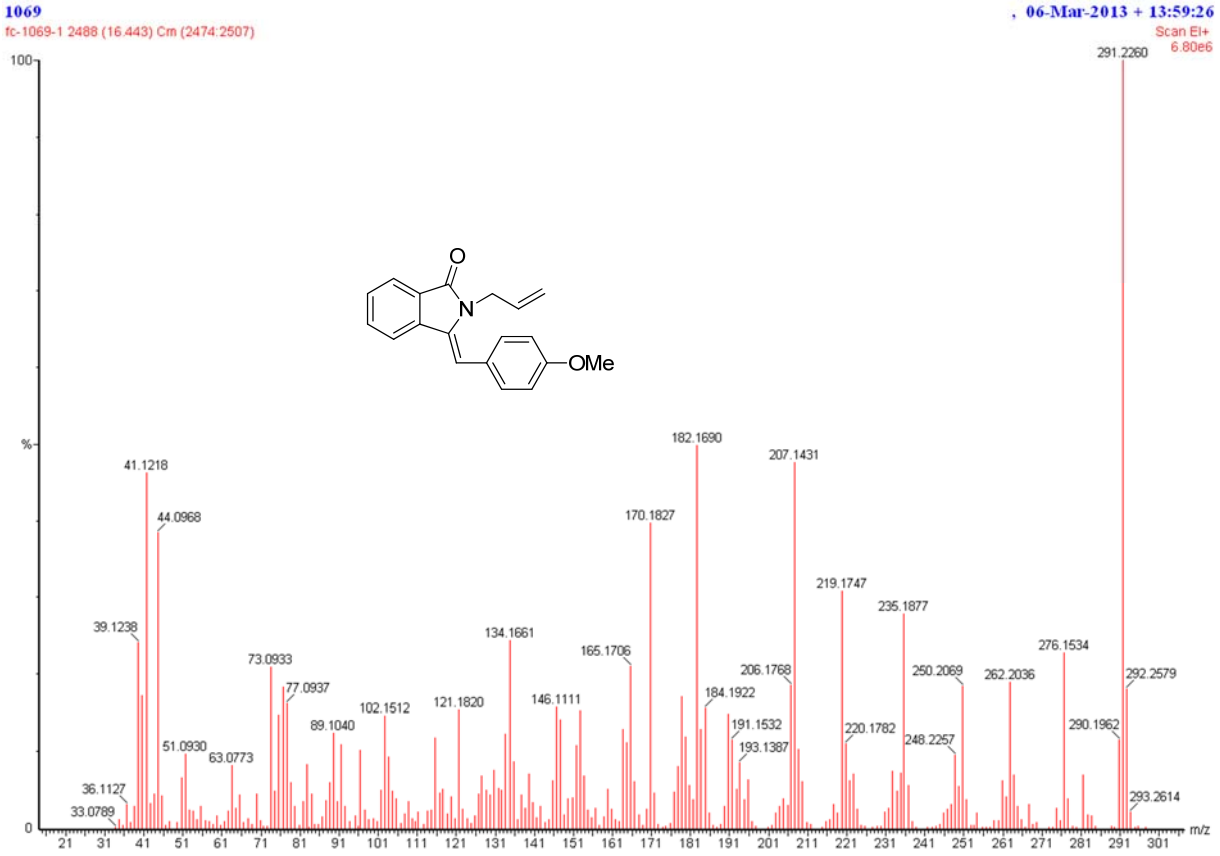

1068
, 07-Mar-2013 + 12:37:54
fc-1068-bis-1 Sm (Mn, 1x3)
Scan EI+
TIC
1.89e8
100
Area
%
0
15.50,10570017
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N

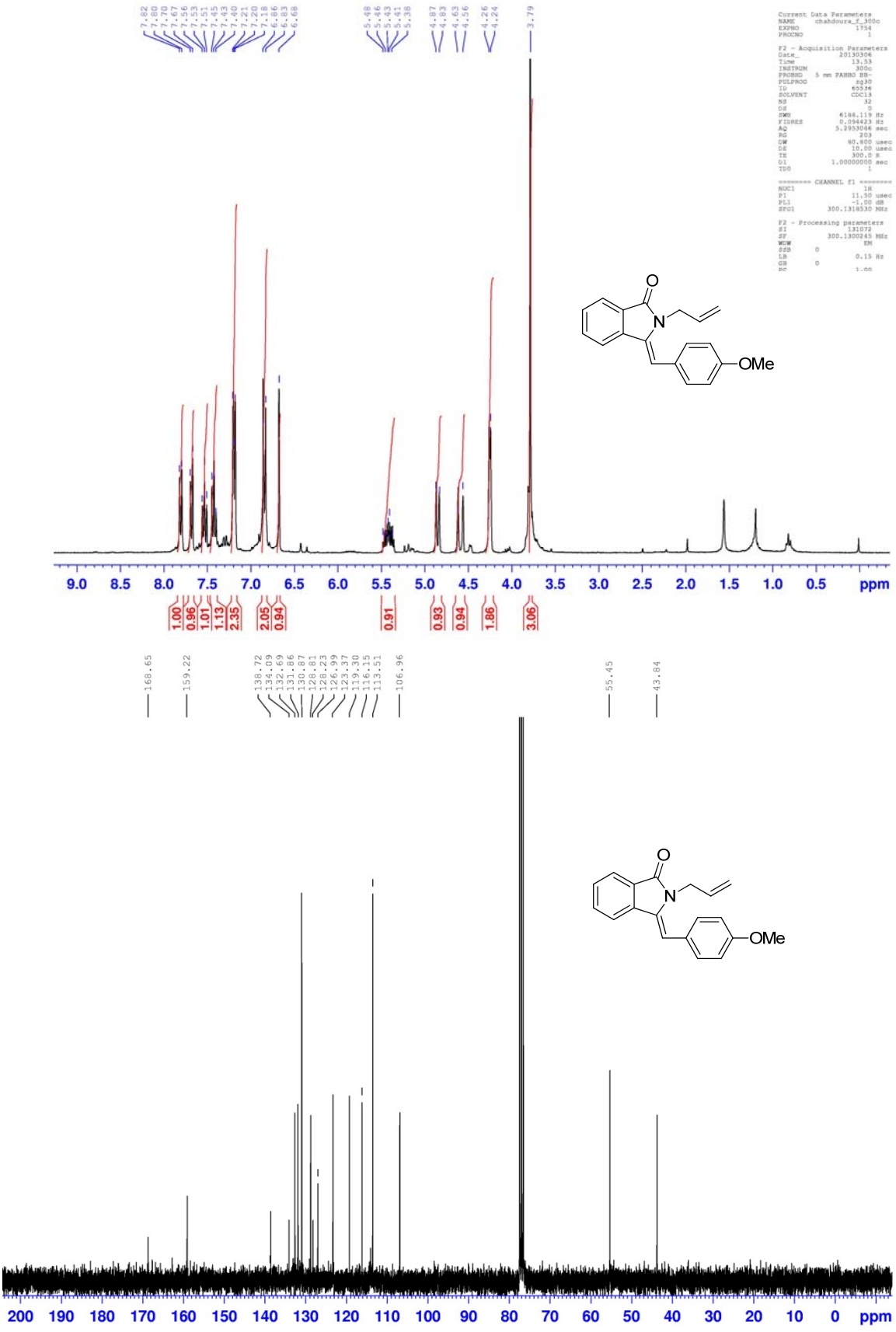

1068
, 07-Mar-2013 + 12:37:54
fc-1068-bis-1 2300 (15.503) Cm (2296:2301)
Scan EI+
1.35e7
100
%
0
275.2586
170.2153
219.1977
182.1934
246.2665
260.2340
190.2170
232.2272
274.2712
191.1898
247.2779
204.2003
234.2502
276.2462
39.0902
77.1207
102.1808
115.1655
130.1468
76.0996
44.0934
131.1900
165.2112
75.0688
89.1260
220.2326
203.2854
207.1728
51.1161
146.1827
164.2202
261.2648
63.0790
82.4184
52.1492
38
48
58
68
78
88
98
108
118
128
138
148
158
168
178
188
198
208
218
228
238
248
258
268
278
m/z
O
N



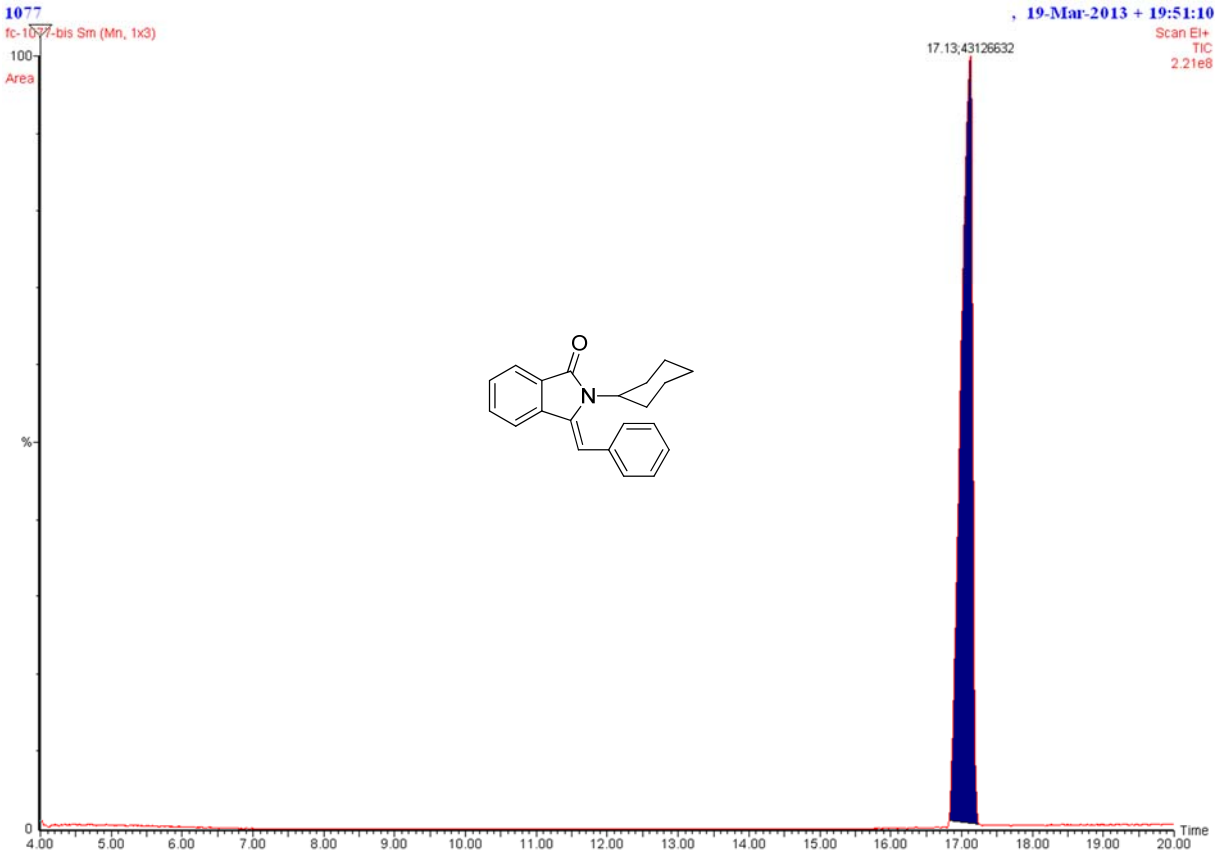
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **m34**

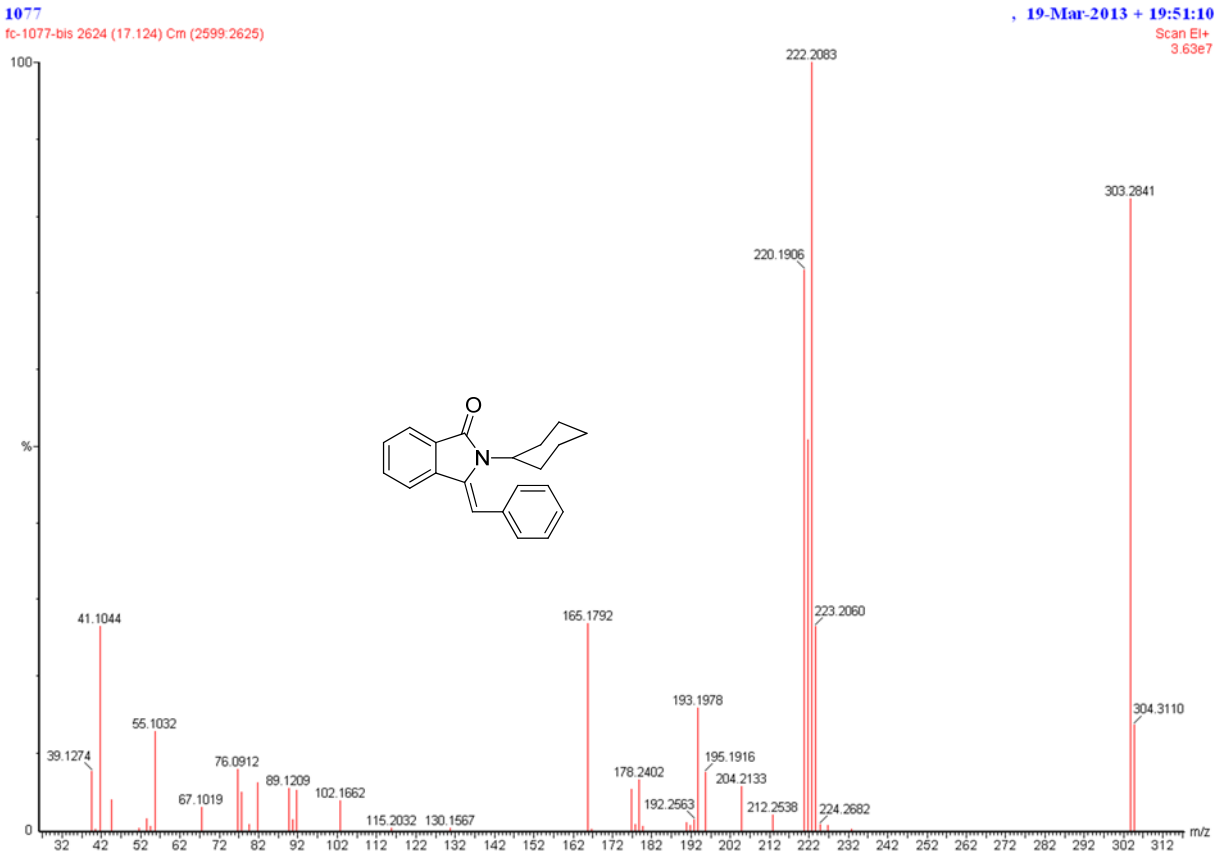

1069
fc-1069-1 Sm (Mn, 1x3)
, 06-Mar-2013 + 13:59:26
Scan EI+
TIC
1.84e8
16.44;15015239
Area
%
Time
OMe

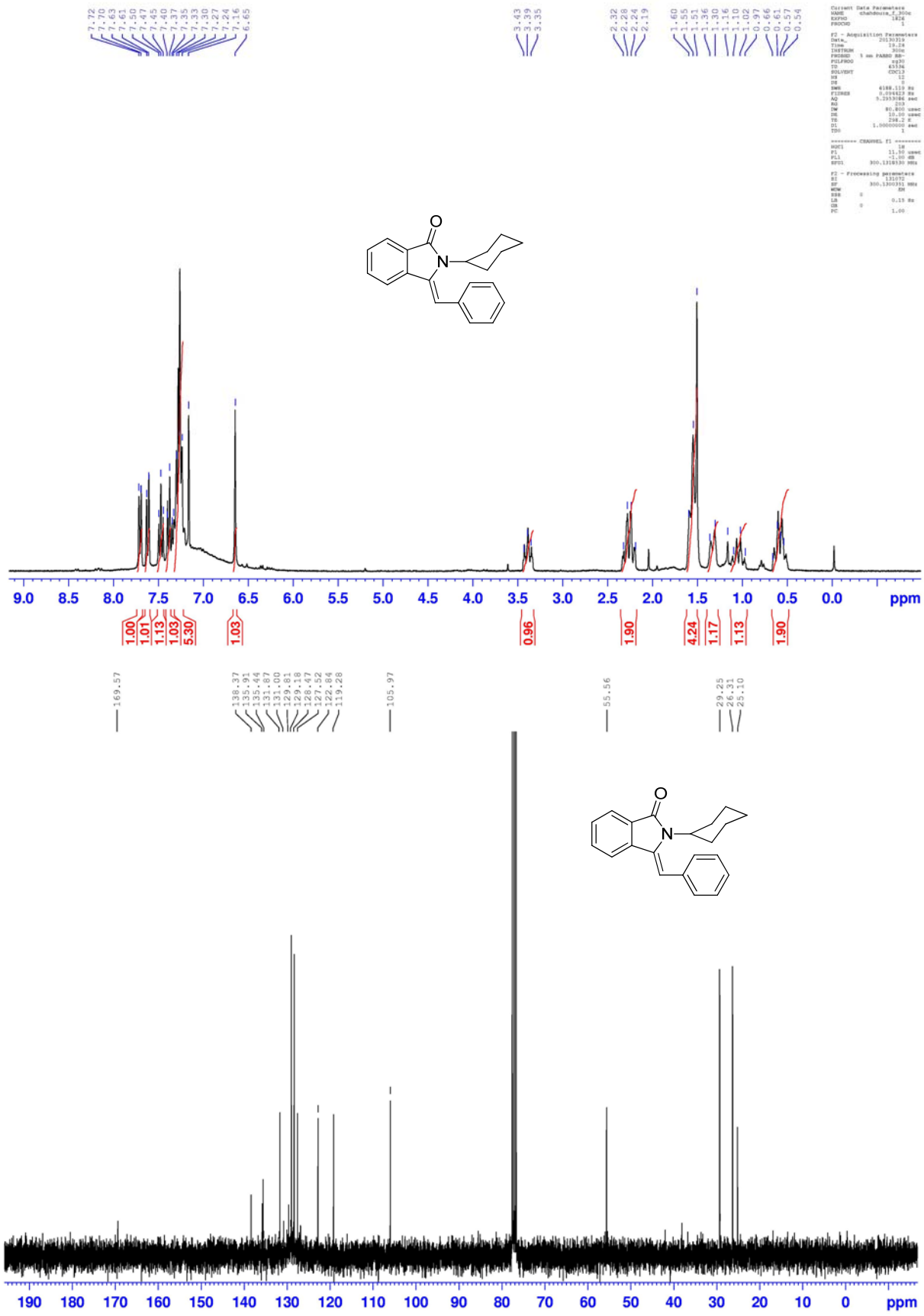

1069
fc-1069-1 2488 (16.443) Cm (2474:2507)
, 06-Mar-2013 + 13:59:26
Scan EI+
6.80e6
%
m/z
OMe
291.2260
182.1690
207.1431
41.1218
44.0968
170.1827
219.1747
235.1877
39.1238
134.1661
165.1706
276.1534
73.0933
77.0937
206.1768
250.2069
262.2036
292.2579
102.1512
121.1820
146.1111
184.1922
89.1040
191.1532
220.1782
290.1962
51.0930
63.0773
193.1387
248.2257
36.1127
33.0789
293.2614



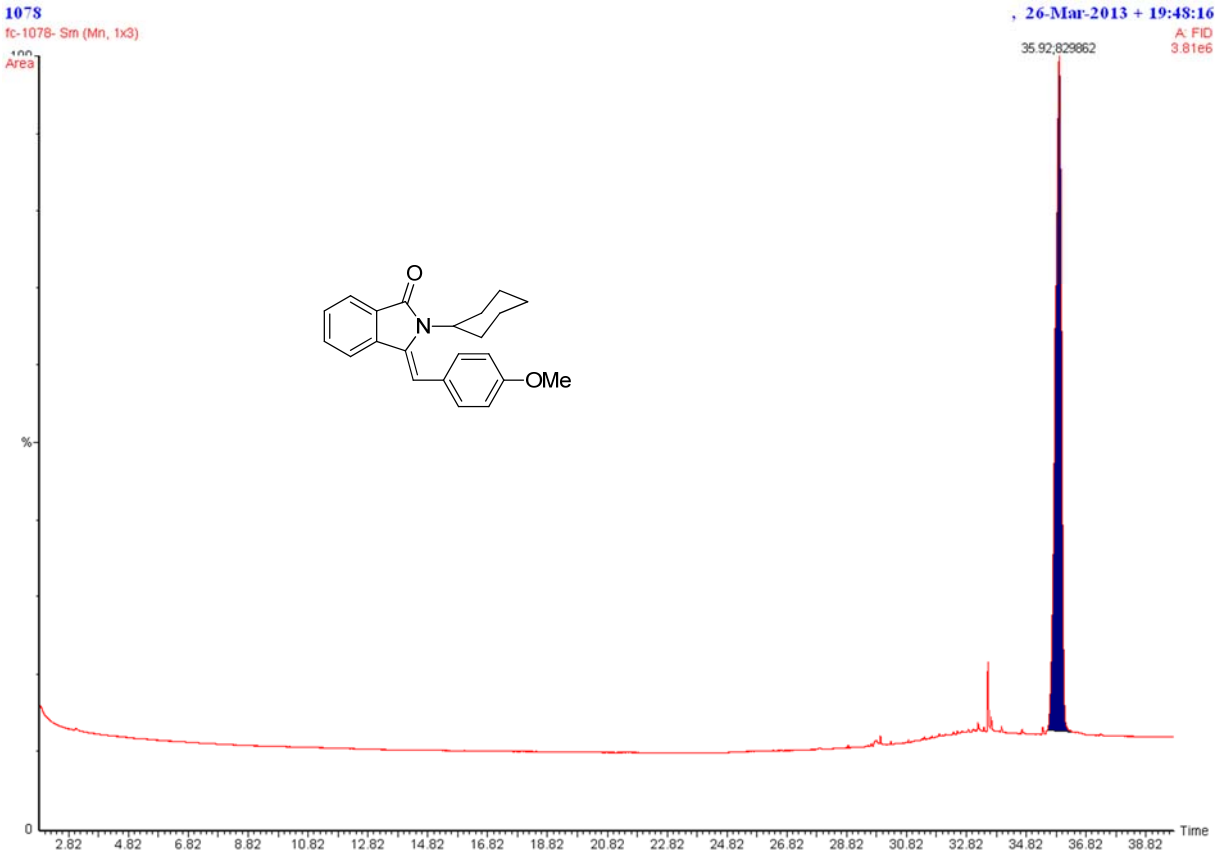
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **m35**

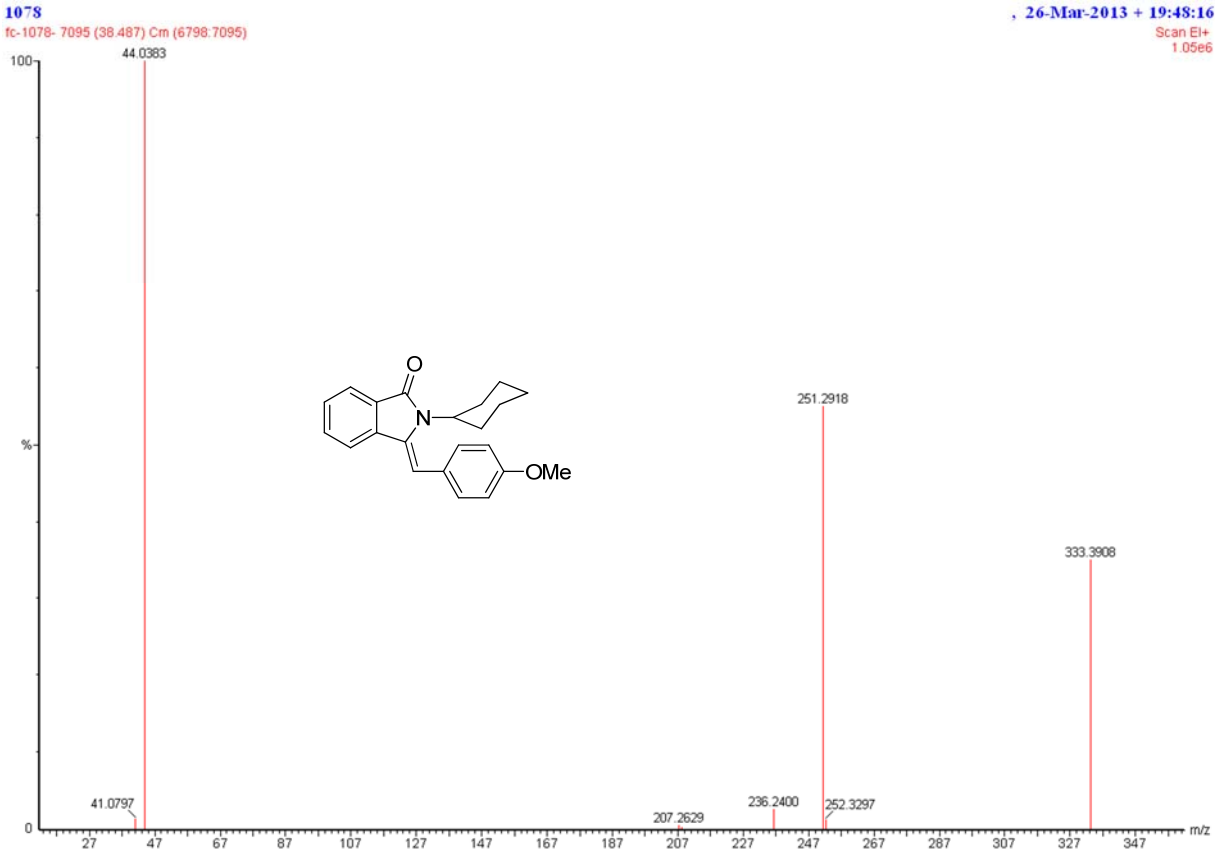

1077
fc-1077-bis Sm (Mn, 1x3)
, 19-Mar-2013 + 19:51:10
Scan EI+
TIC
2.21e8
17.13;43126632
100
Area
%
0
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N

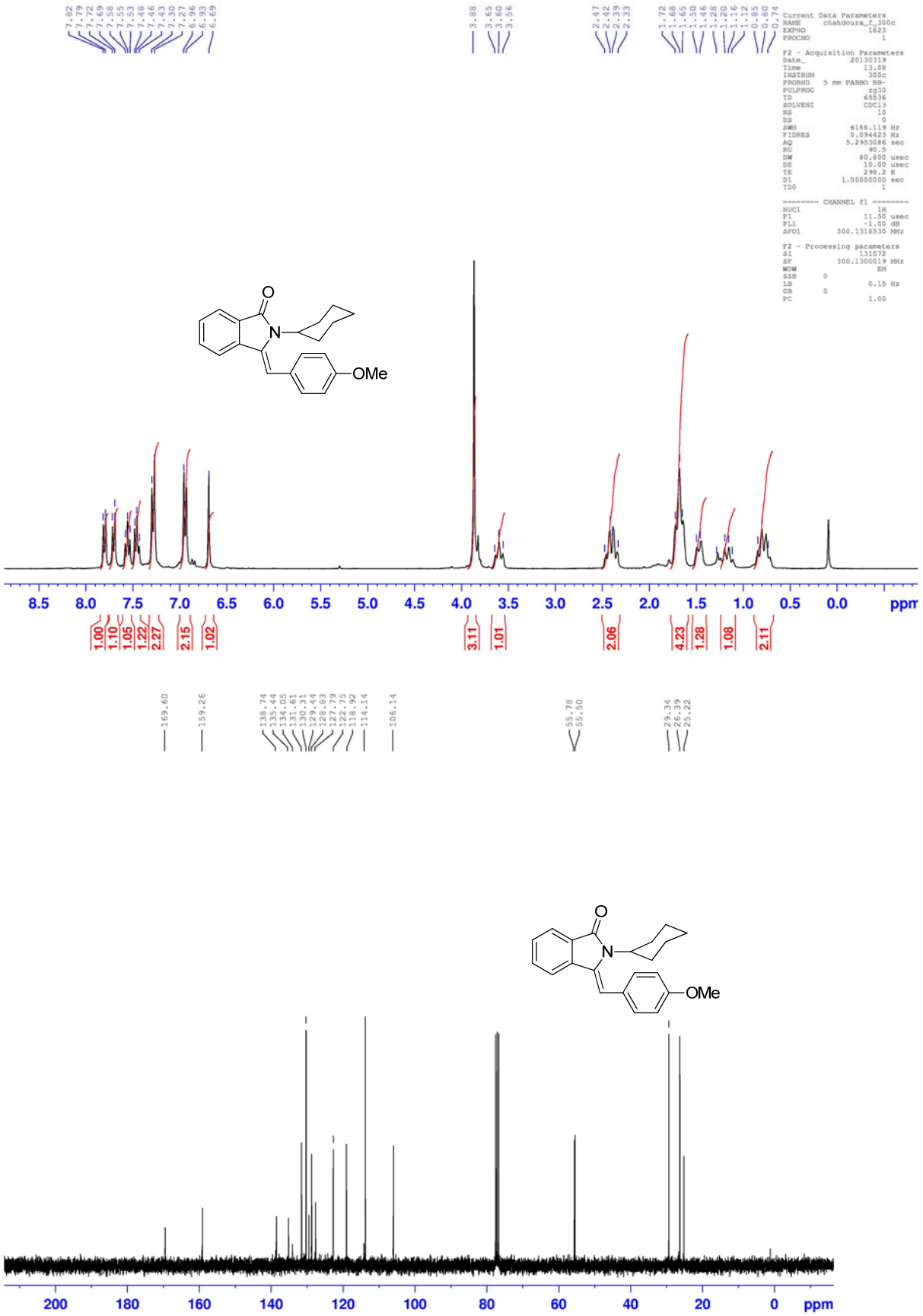

1077
fc-1077-bis 2624 (17.124) Cm (2599:2625)
, 19-Mar-2013 + 19:51:10
Scan EI+
3.63e7
100
%
0
222.2083
303.2841
220.1906
223.2060
304.3110
165.1792
41.1044
55.1032
193.1978
195.1916
39.1274
76.0912
89.1209
178.2402
204.2133
67.1019
102.1662
192.2563
212.2538
224.2682
115.2032
130.1567
32
42
52
62
72
82
92
102
112
122
132
142
152
162
172
182
192
202
212
222
232
242
252
262
272
282
292
302
312
m/z
O
N



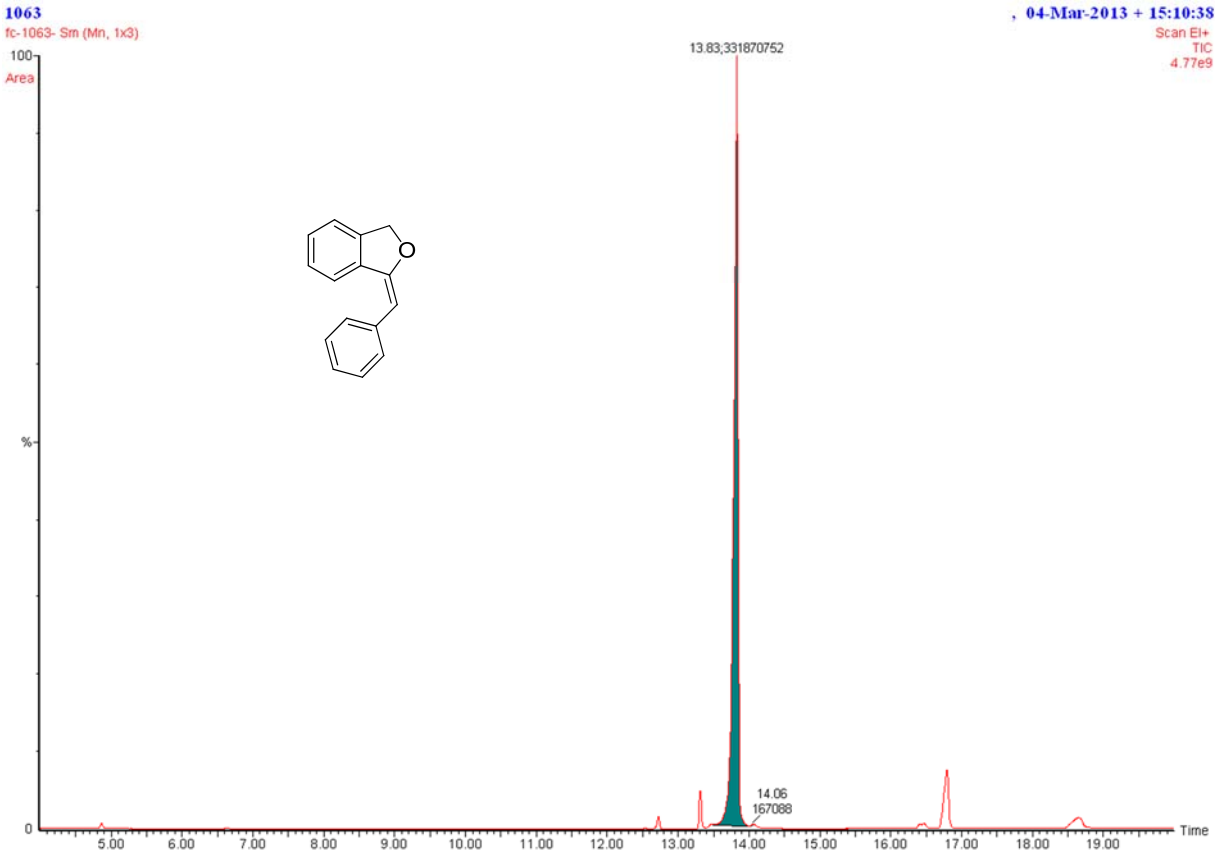
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **n33**

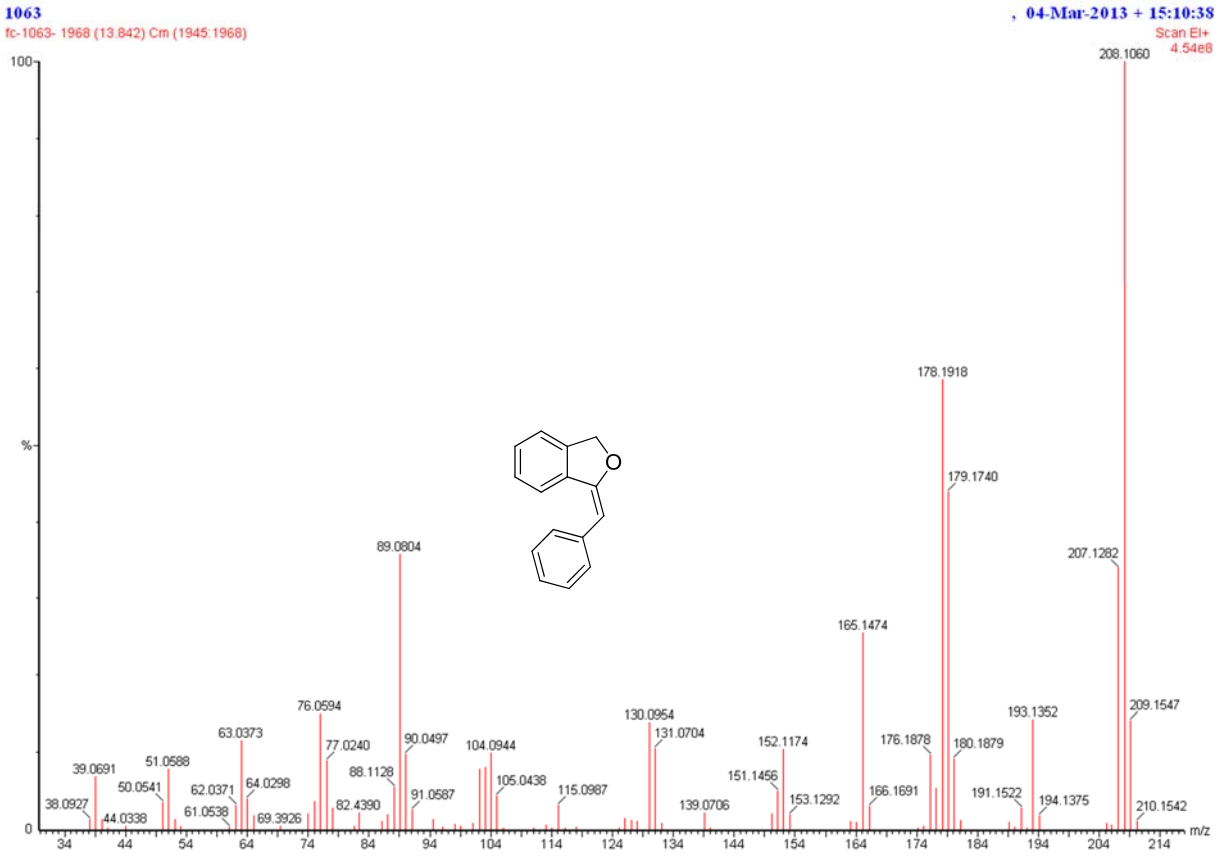

1078
fc-1078- Sm (Mn, 1x3)
, 26-Mar-2013 + 19:48:16
A: FID
3.81e6
35.92;829862
Area
%
0
Time
2.82
4.82
6.82
8.82
10.82
12.82
14.82
16.82
18.82
20.82
22.82
24.82
26.82
28.82
30.82
32.82
34.82
36.82
38.82
O
N
OMe

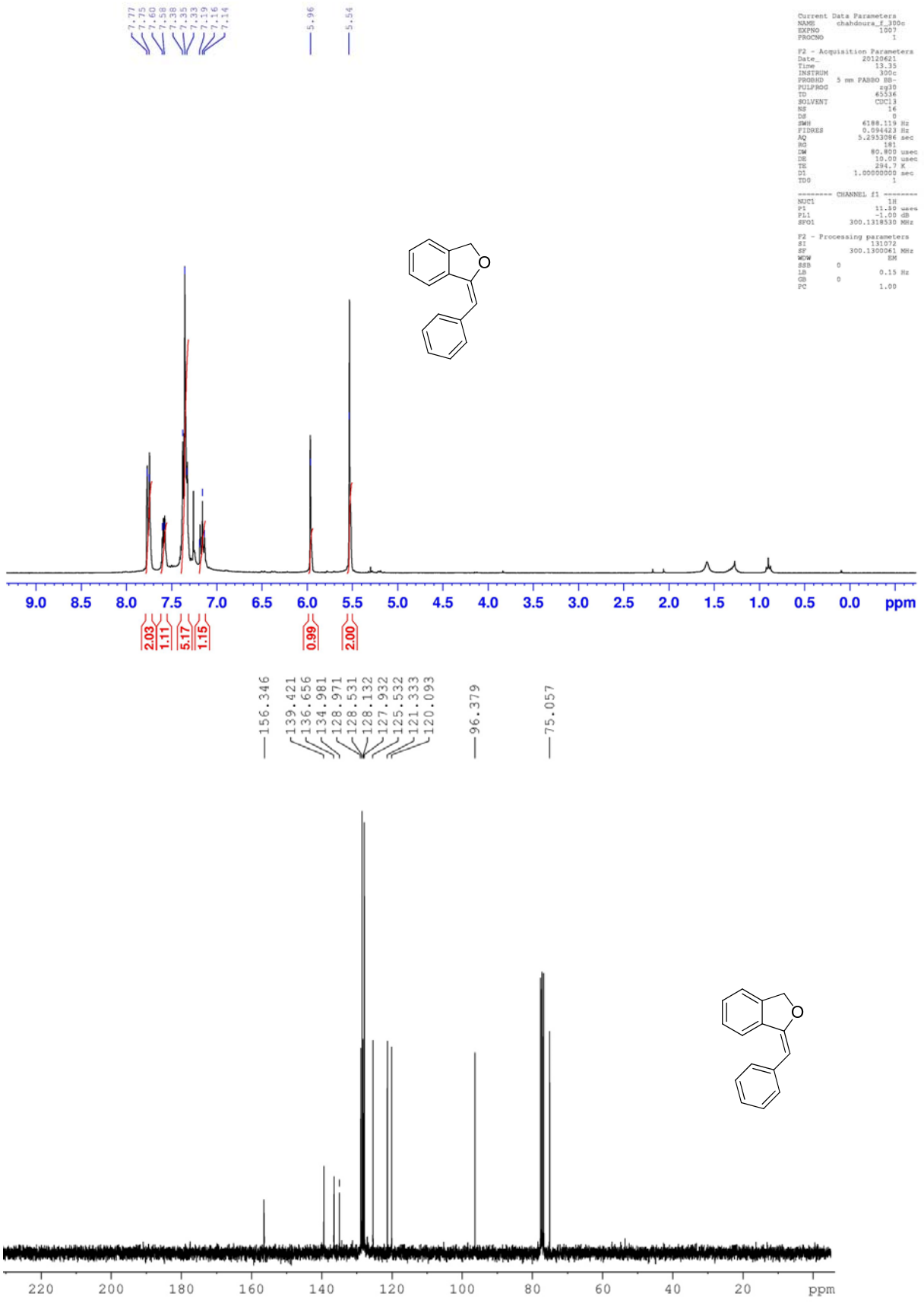

1078
fc-1078- 7095 (38.487) Cm (6798:7095)
, 26-Mar-2013 + 19:48:16
Scan EI+
1.05e6
100
%
0
44.0383
41.0797
207.2629
236.2400
251.2918
252.3297
333.3908
m/z
27
47
67
87
107
127
147
167
187
207
227
247
267
287
307
327
347
O
N
OMe



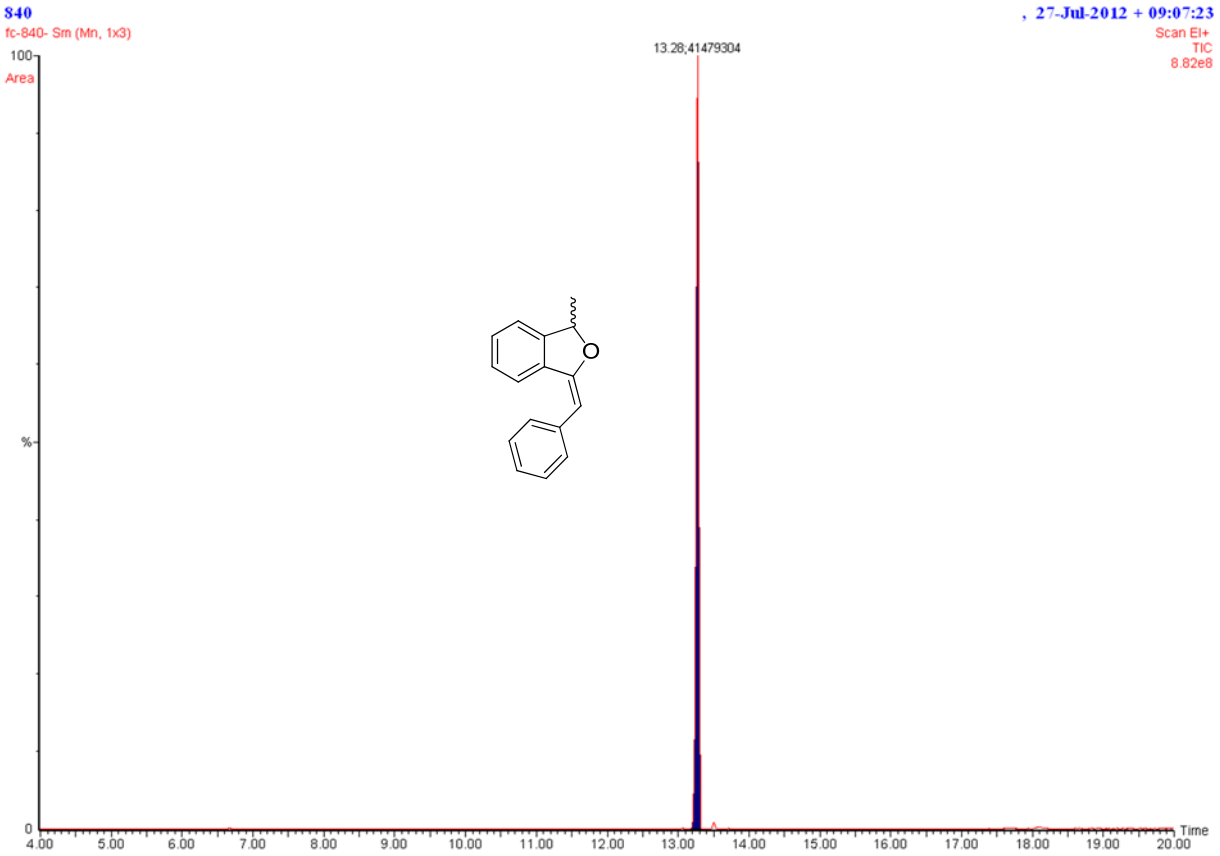
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **n35**

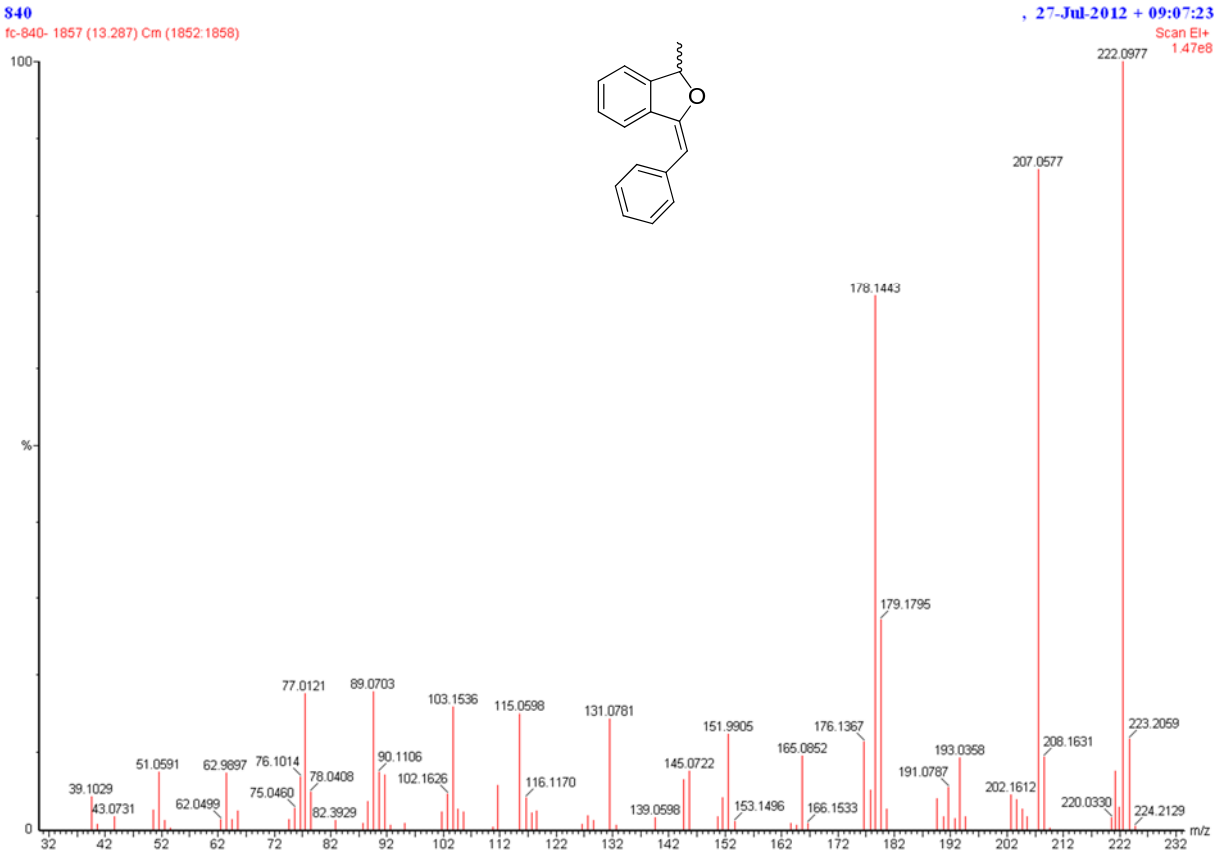

1063
fc-1063- Sm (Mn, 1x3)
, 04-Mar-2013 + 15:10:38
Scan EI+
TIC
4.77e9
100
Area
%
0
13.83;331870752
14.06
167088
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
Time
O

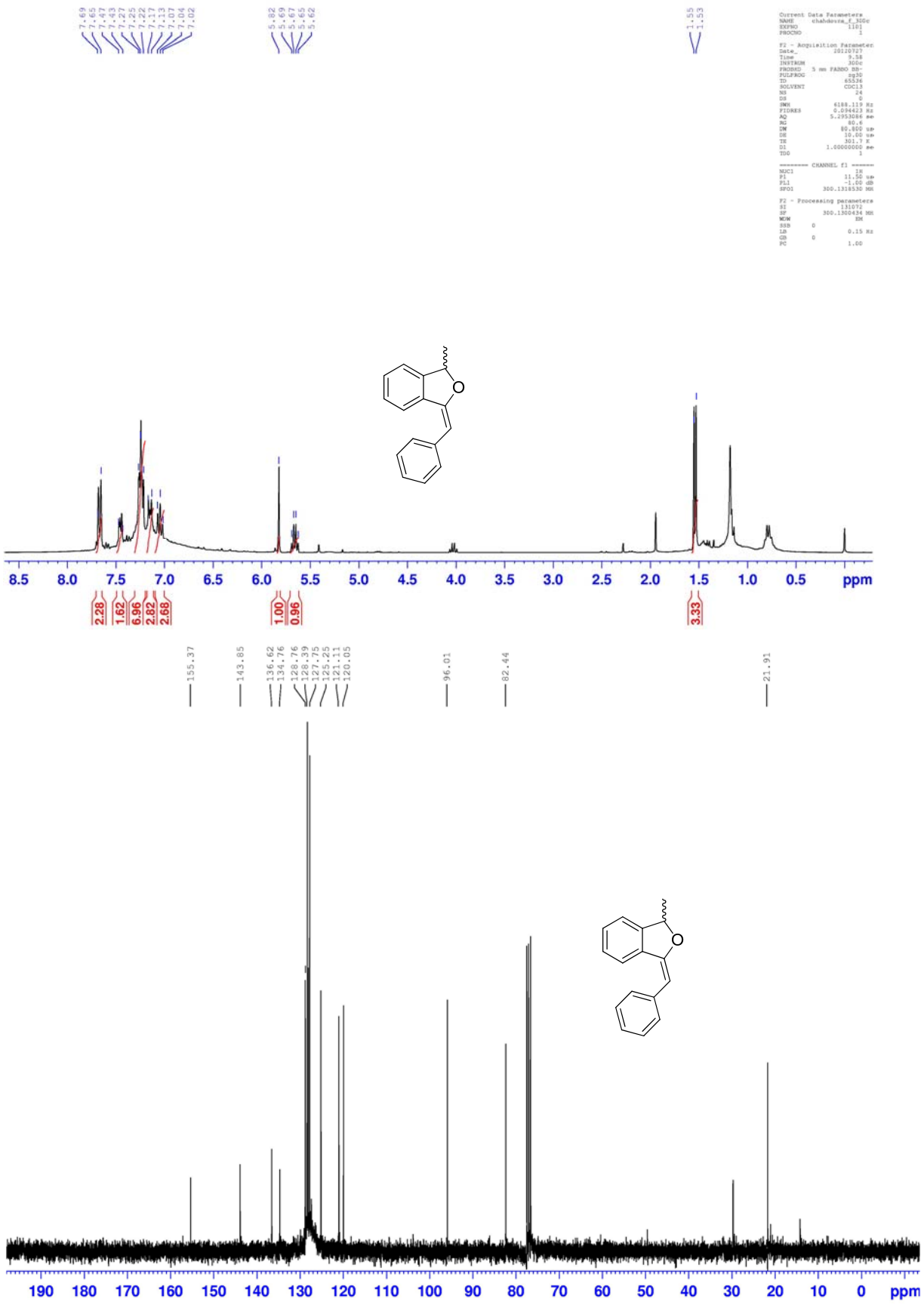

1063
fc-1063- 1968 (13.842) Cm (1945:1968)
, 04-Mar-2013 + 15:10:38
Scan EI+
4.54e8
100
%
0
208.1060
178.1918
179.1740
207.1282
165.1474
89.0804
209.1547
193.1352
130.0954
131.0704
152.1174
176.1878
180.1879
76.0594
63.0373
77.0240
90.0497
104.0944
88.1128
105.0438
91.0587
115.0987
39.0691
51.0588
50.0541
62.0371
64.0298
38.0927
44.0338
61.0538
69.3926
82.4390
139.0706
151.1456
153.1292
166.1691
191.1522
194.1375
210.1542
34
44
54
64
74
84
94
104
114
124
134
144
154
164
174
184
194
204
214
m/z
O



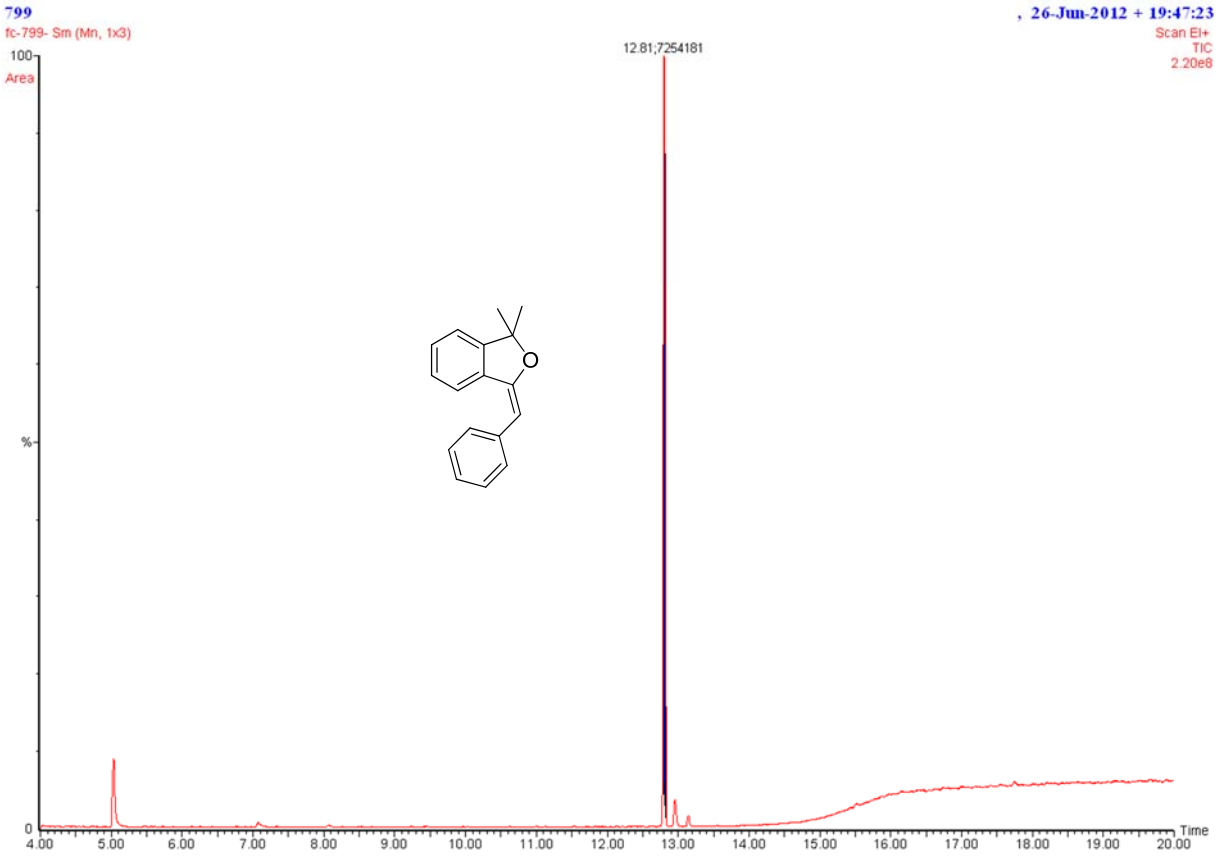
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **o33**

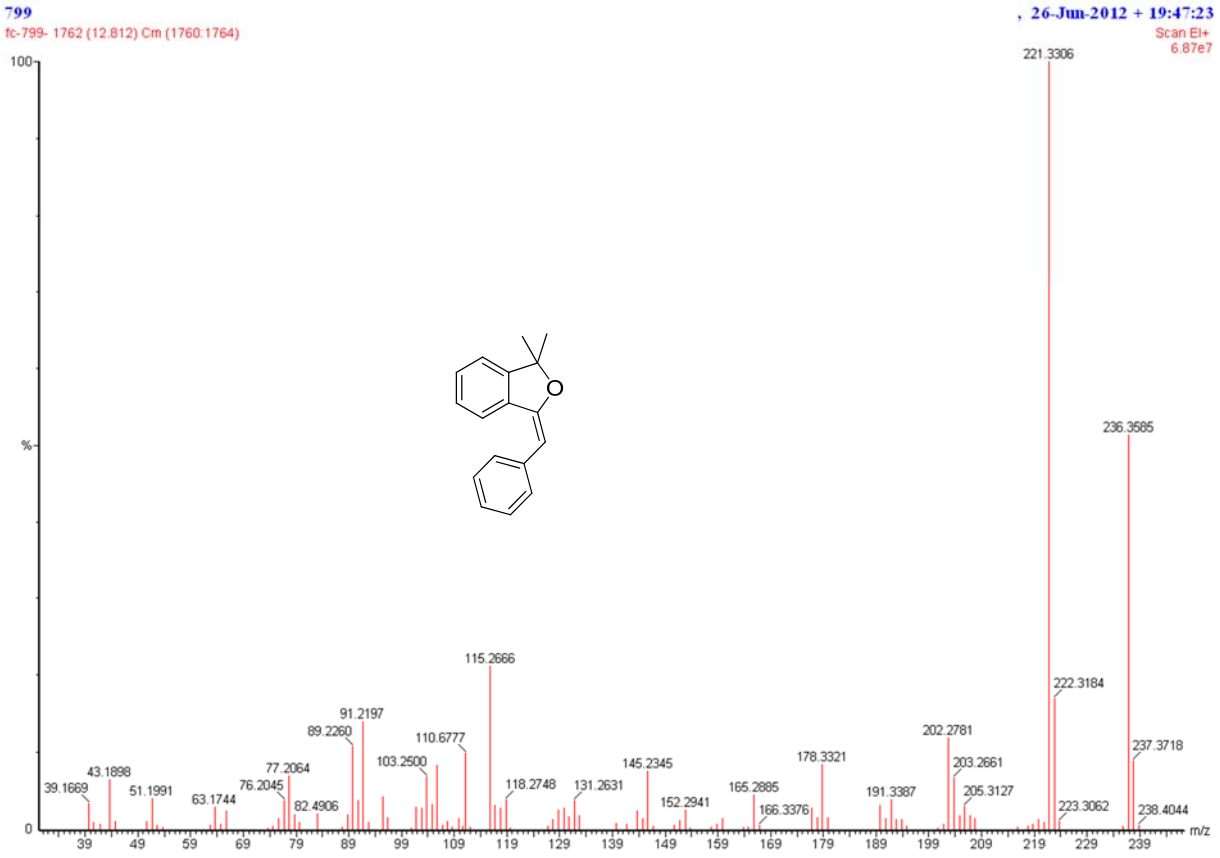

840
fc-840- Sm (Mn, 1x3)
, 27-Jul-2012 + 09:07:23
Scan EI+
TIC
8.82e8
Area
13.28;41479304
%
Time

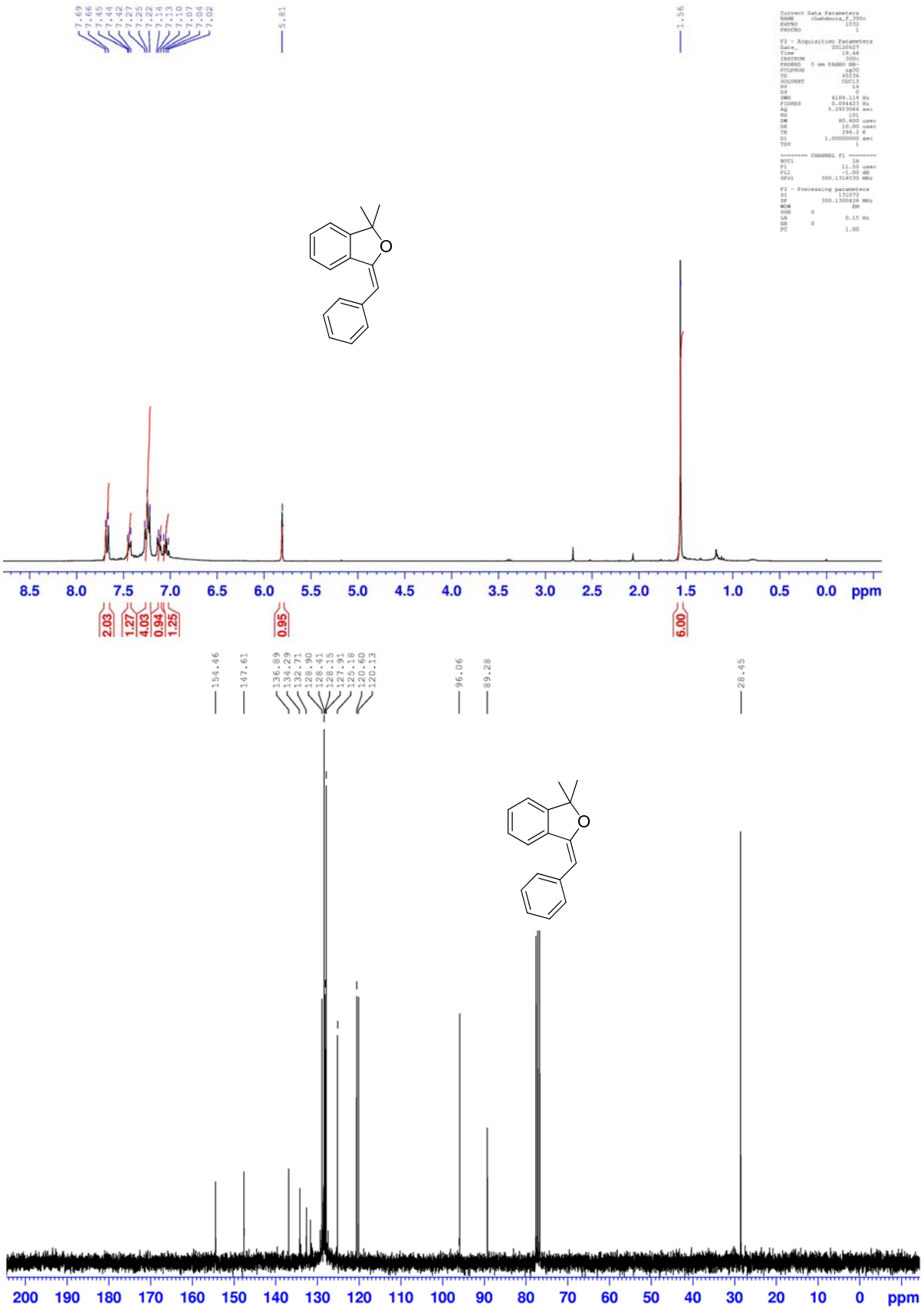

840
fc-840- 1857 (13.287) Cm (1852:1858)
, 27-Jul-2012 + 09:07:23
Scan EI+
1.47e8
%
222.0977
207.0577
178.1443
179.1795
77.0121
89.0703
103.1536
115.0598
131.0781
151.9905
176.1367
165.0852
145.0722
193.0358
208.1631
223.2059
191.0787
202.1612
220.0330
224.2129
51.0591
62.9897
76.1014
78.0408
90.1106
102.1626
116.1170
39.1029
43.0731
62.0499
75.0460
82.3929
139.0598
153.1496
166.1533
m/z



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **p33**


799
fc-799- Sm (Mn, 1x3)
, 26-Jun-2012 + 19:47:23
Scan EI+
TIC
2.20e8
100
Area
%
0
12.81;7254181
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time


799
fc-799- 1762 (12.812) Cm (1760:1764)
, 26-Jun-2012 + 19:47:23
Scan EI+
6.87e7
100
%
0
221.3306
236.3585
115.2666
222.3184
91.2197
89.2260
110.6777
202.2781
237.3718
103.2500
178.3321
203.2661
43.1898
77.2064
145.2345
39.1669
51.1991
76.2045
118.2748
131.2631
165.2885
191.3387
205.3127
63.1744
82.4906
152.2941
166.3376
223.3062
238.4044
39
49
59
69
79
89
99
109
119
129
139
149
159
169
179
189
199
209
219
229
239
m/z



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **q33**

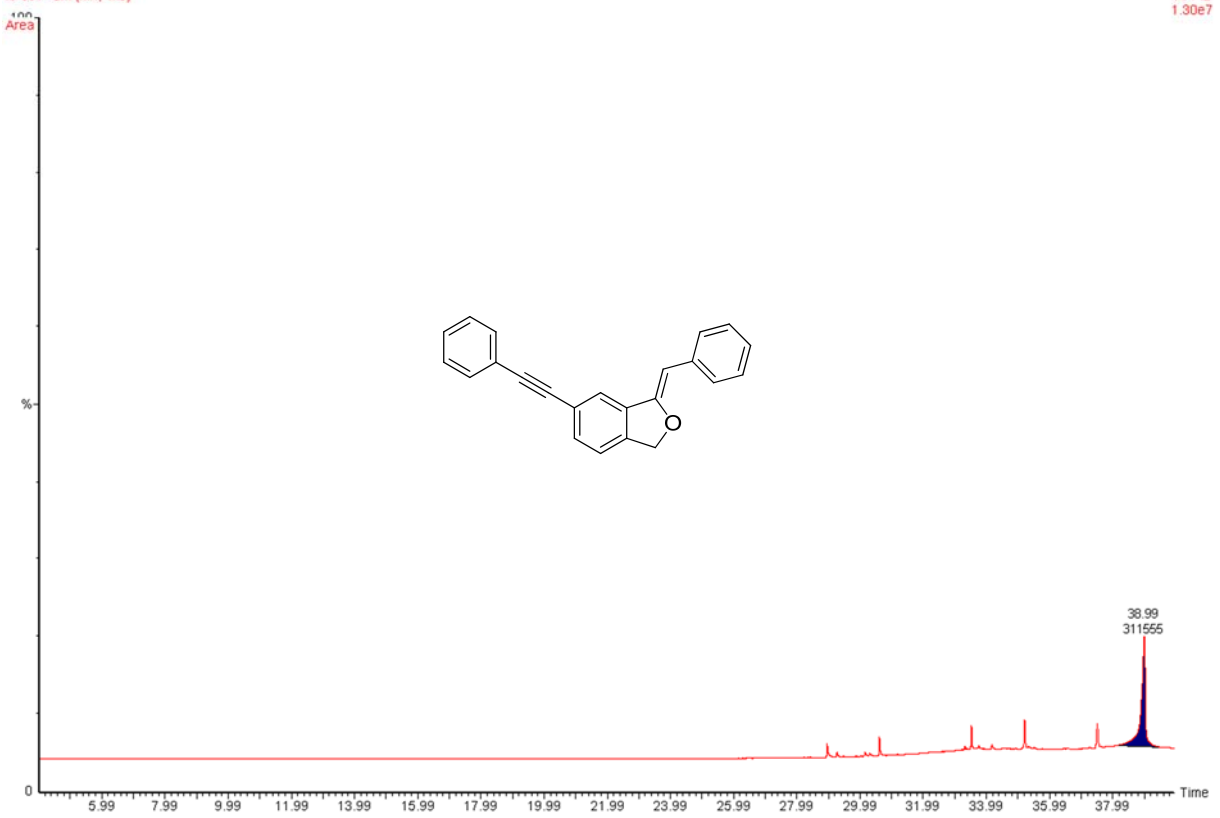
997

fc-997- Sm (Mn, 1x3)

, 18-Jan-2013 + 15:36:44

A: FID

1.30e7



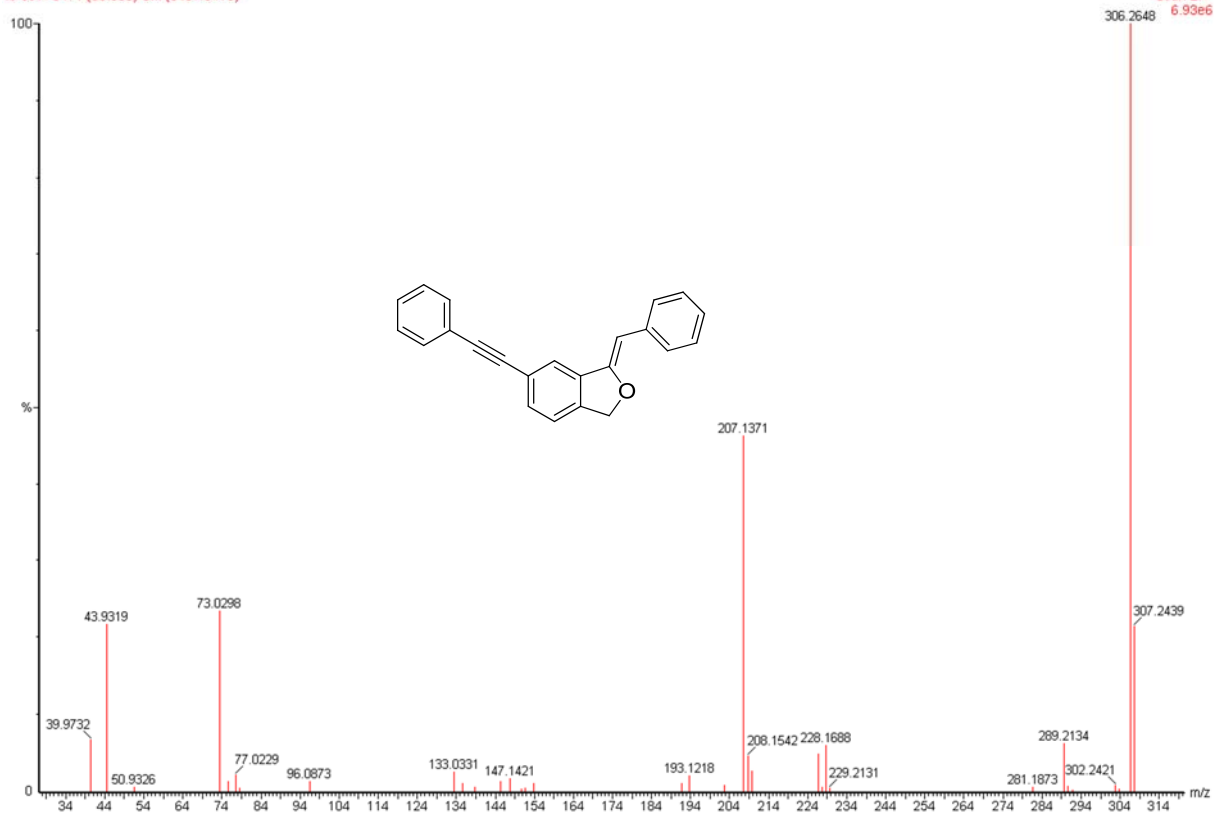
997

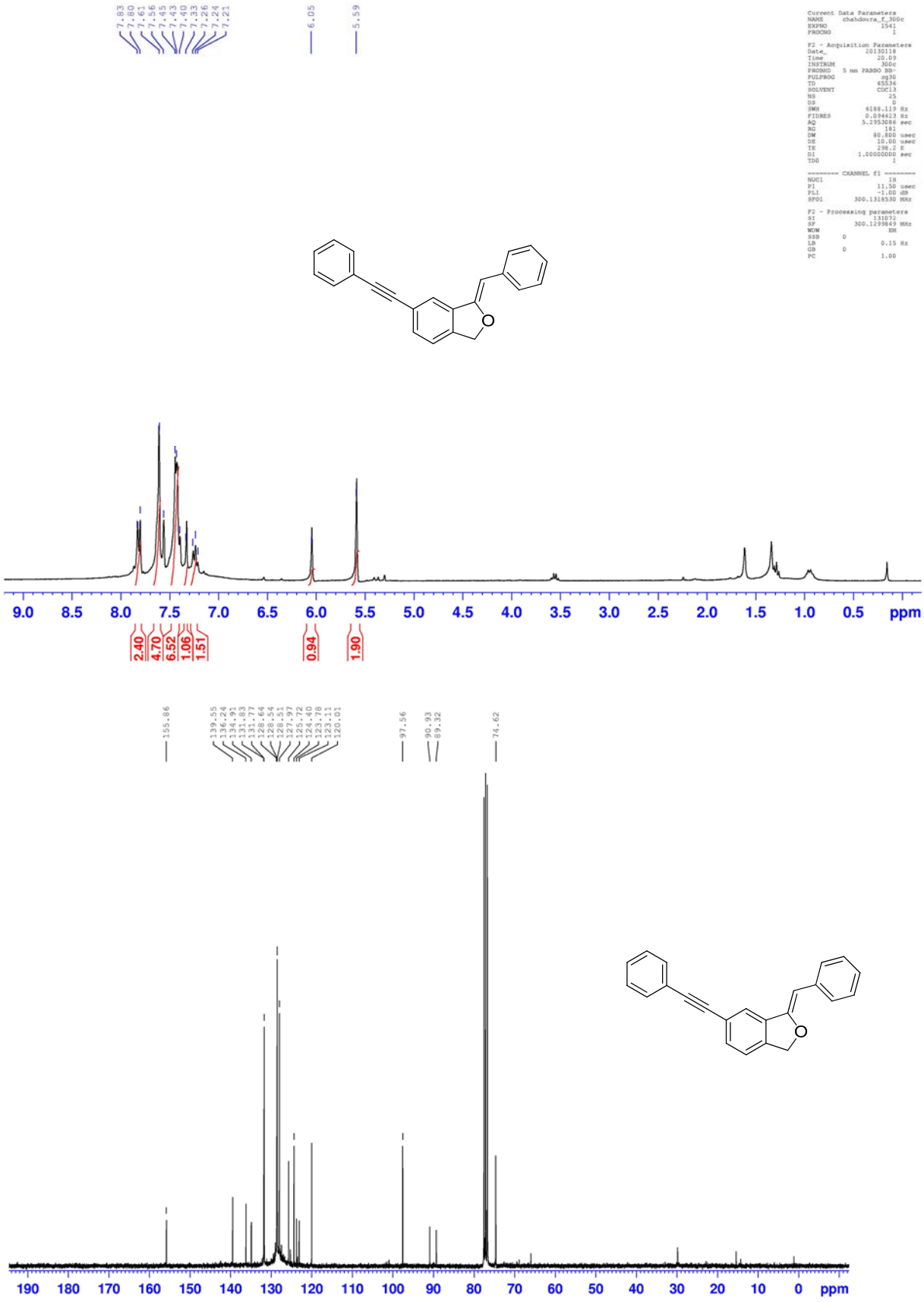
fc-997- 6471 (35.366) Cm (6467:6476)

, 18-Jan-2013 + 15:36:44

Scan EI+

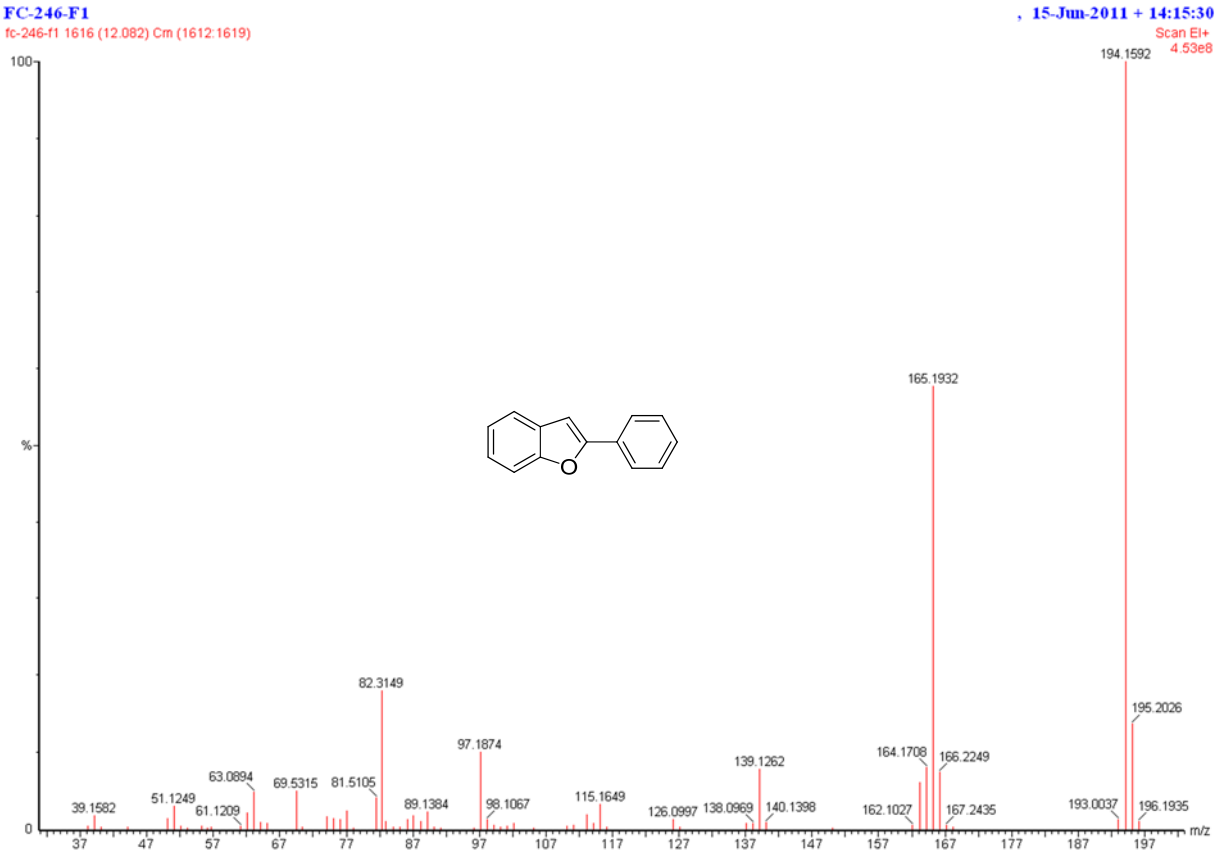
6.93e6

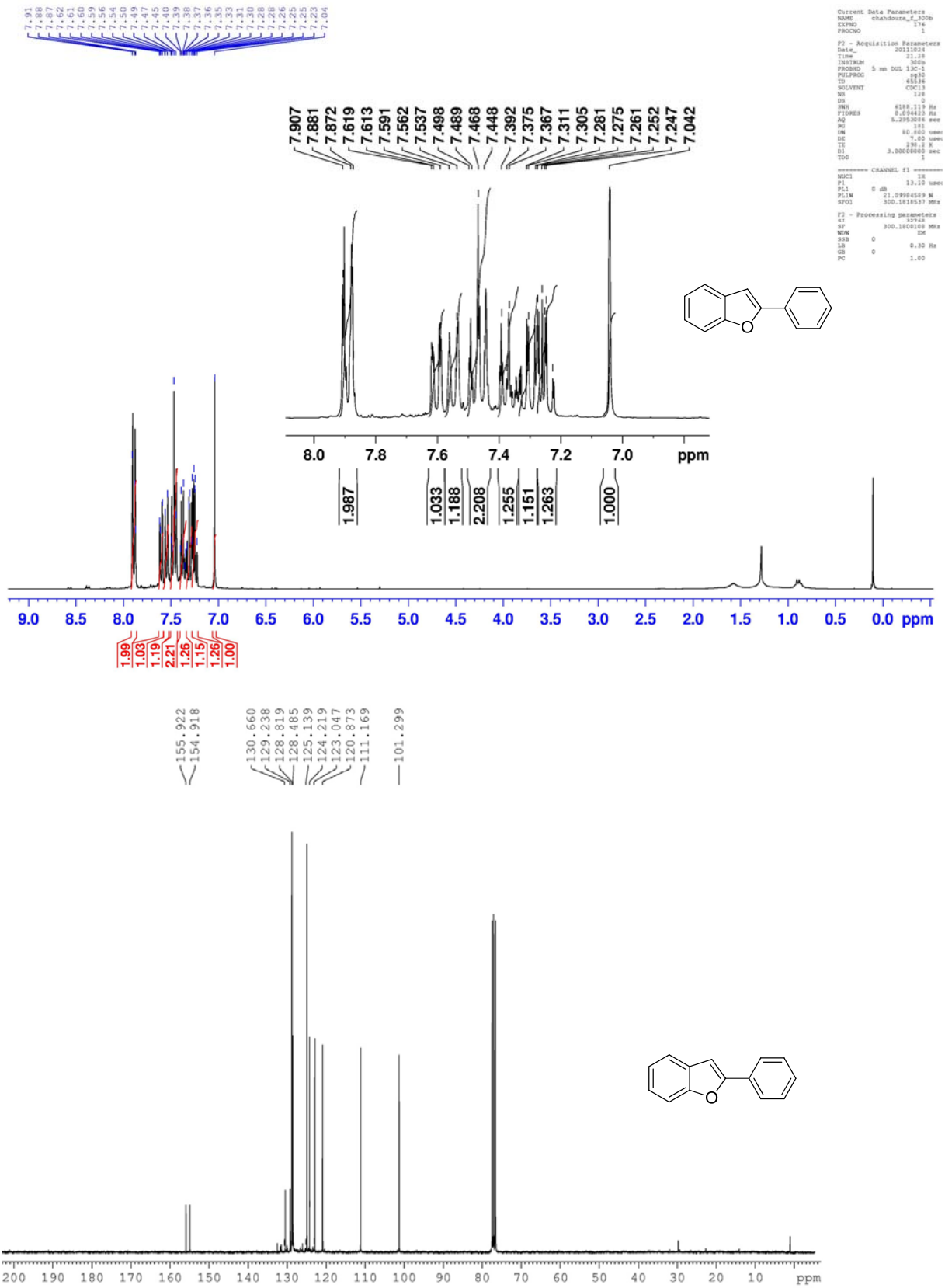




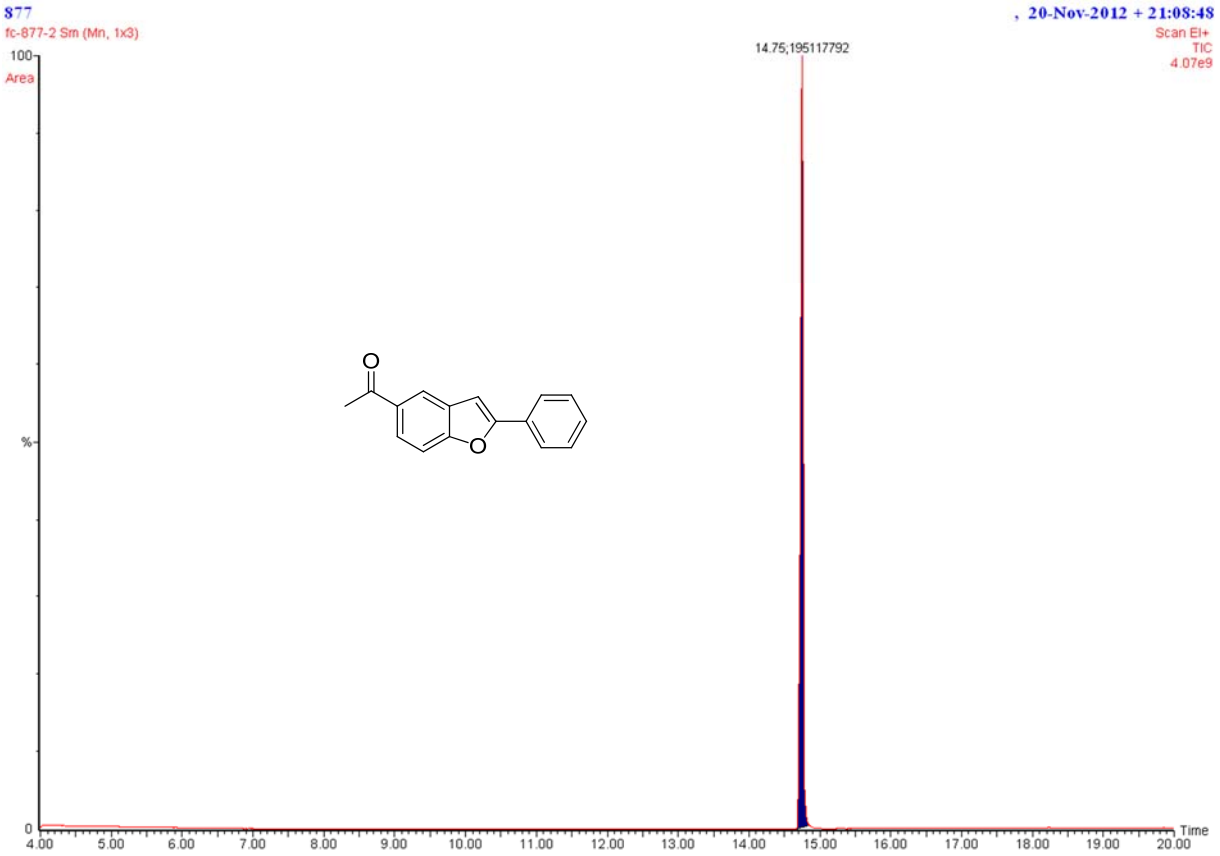
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **r33**

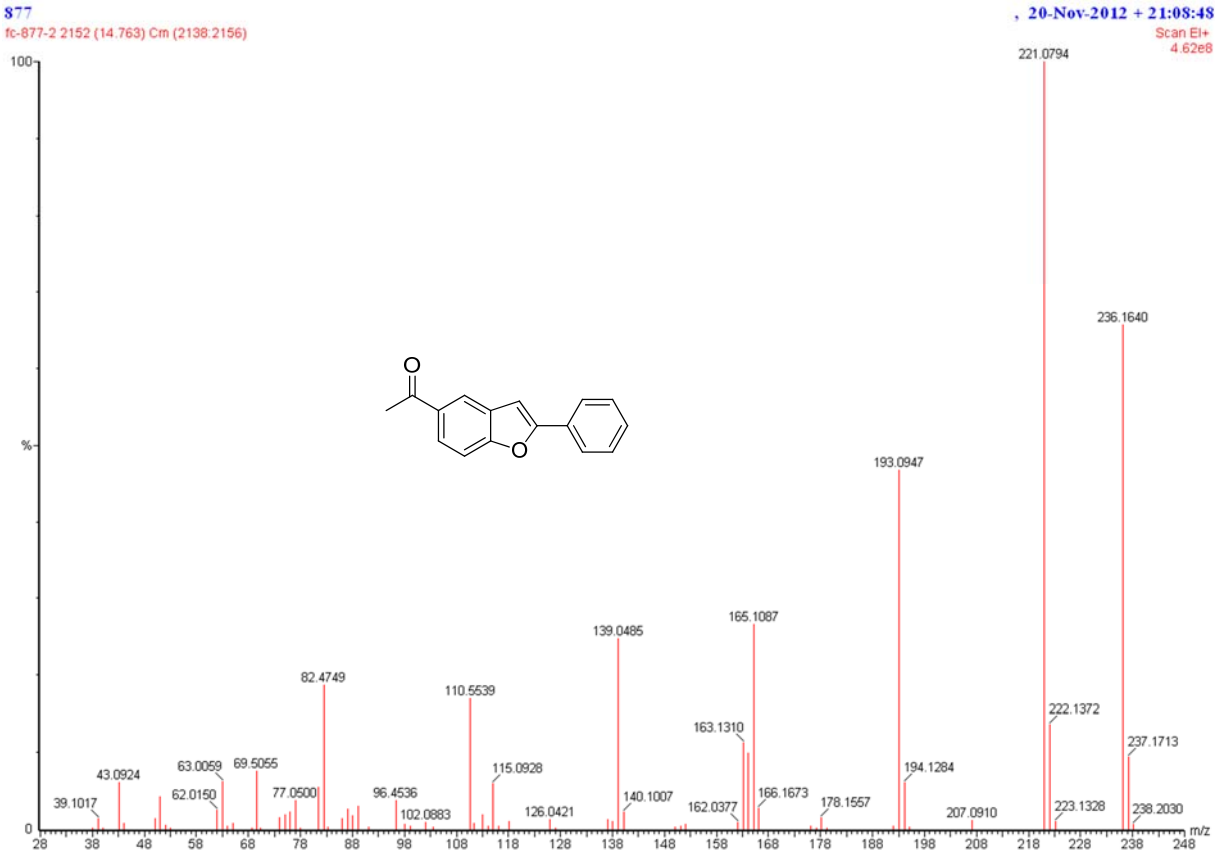

FC-246-F1
fc-246-f1 Sm (Mn, 1x3)
, 15-Jun-2011 + 14:15:30
Scan EI+
TIC
1.48e9
12.08;52500748
100
Area
%
0
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
Time

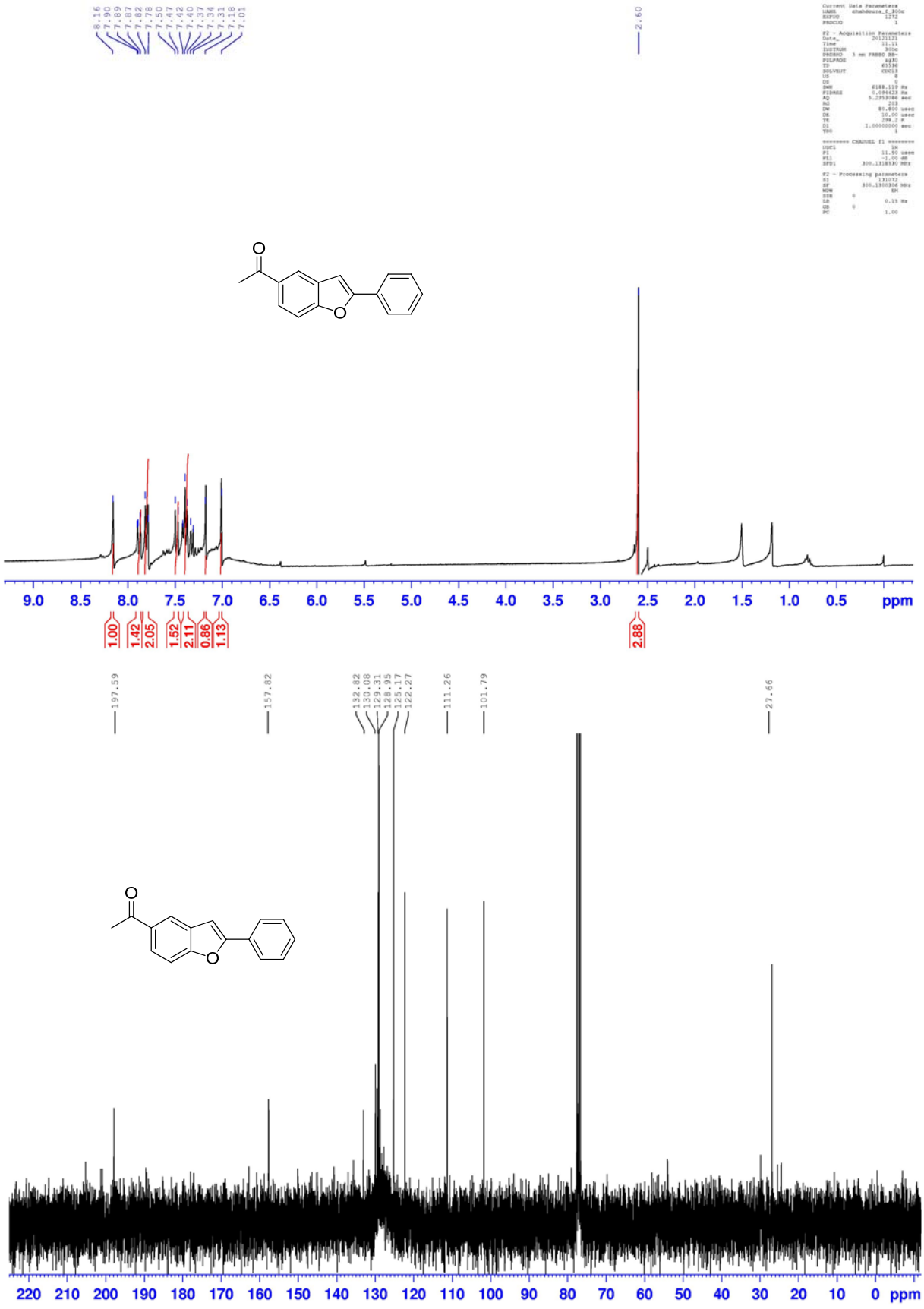

FC-246-F1
fc-246-f1 1616 (12.082) Cm (1612:1619)
, 15-Jun-2011 + 14:15:30
Scan EI+
4.53e8
194.1592
165.1932
82.3149
195.2026
97.1874
139.1262
164.1708
166.2249
63.0894
69.5315
81.5105
39.1582
51.1249
61.1209
89.1384
98.1067
115.1649
126.0997
138.0969
140.1398
162.1027
167.2435
193.0037
196.1935
100
%
0
37
47
57
67
77
87
97
107
117
127
137
147
157
167
177
187
197
m/z



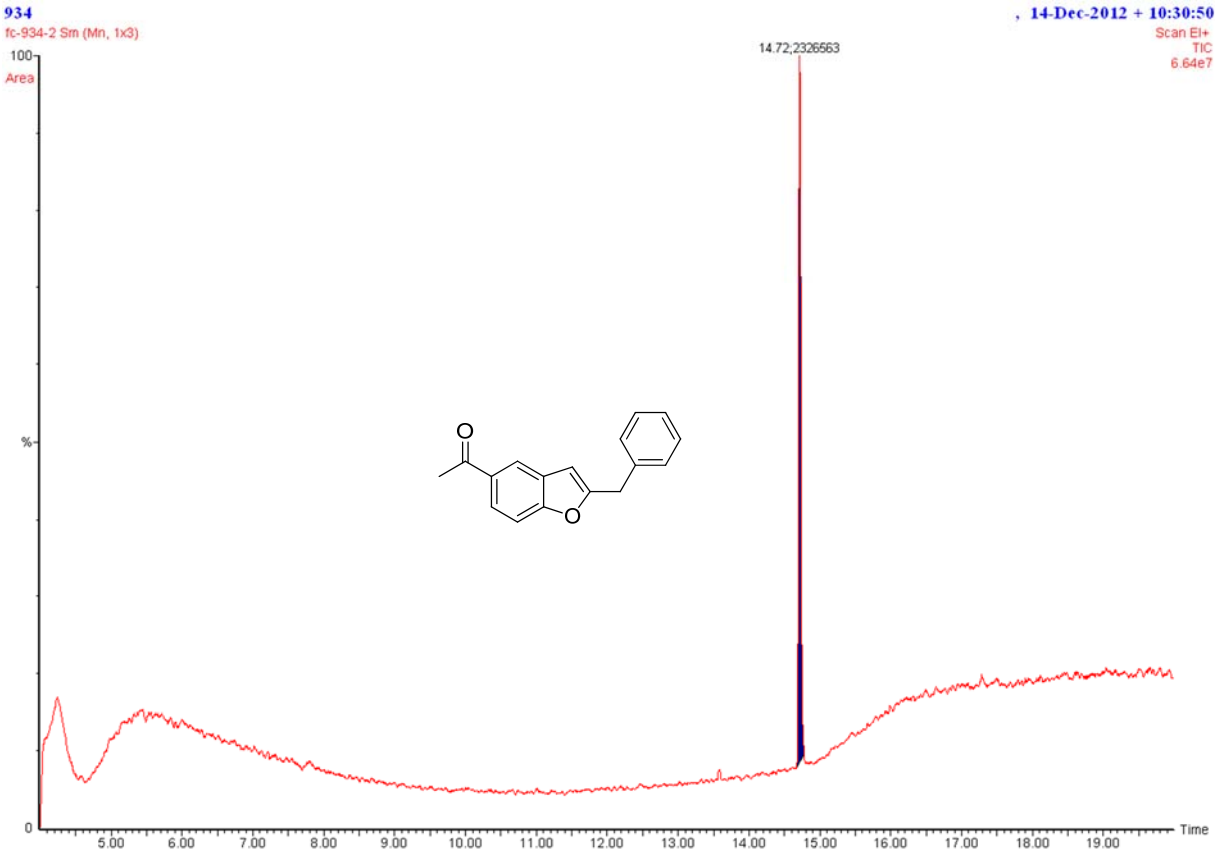
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **s33**

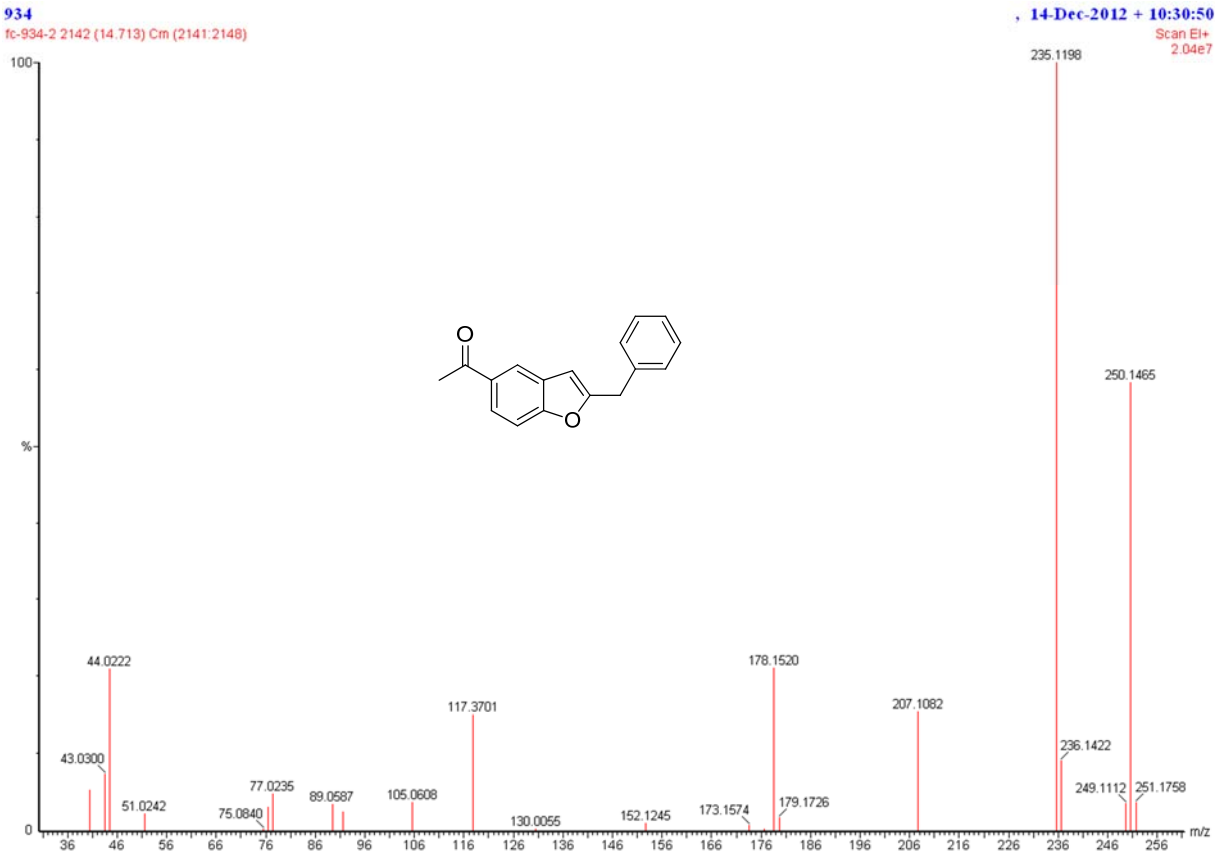

877
fc-877-2 Sm (Mn, 1x3)
, 20-Nov-2012 + 21:08:48
Scan EI+
TIC
4.07e9
14.75;195117792
100
Area
%
0
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

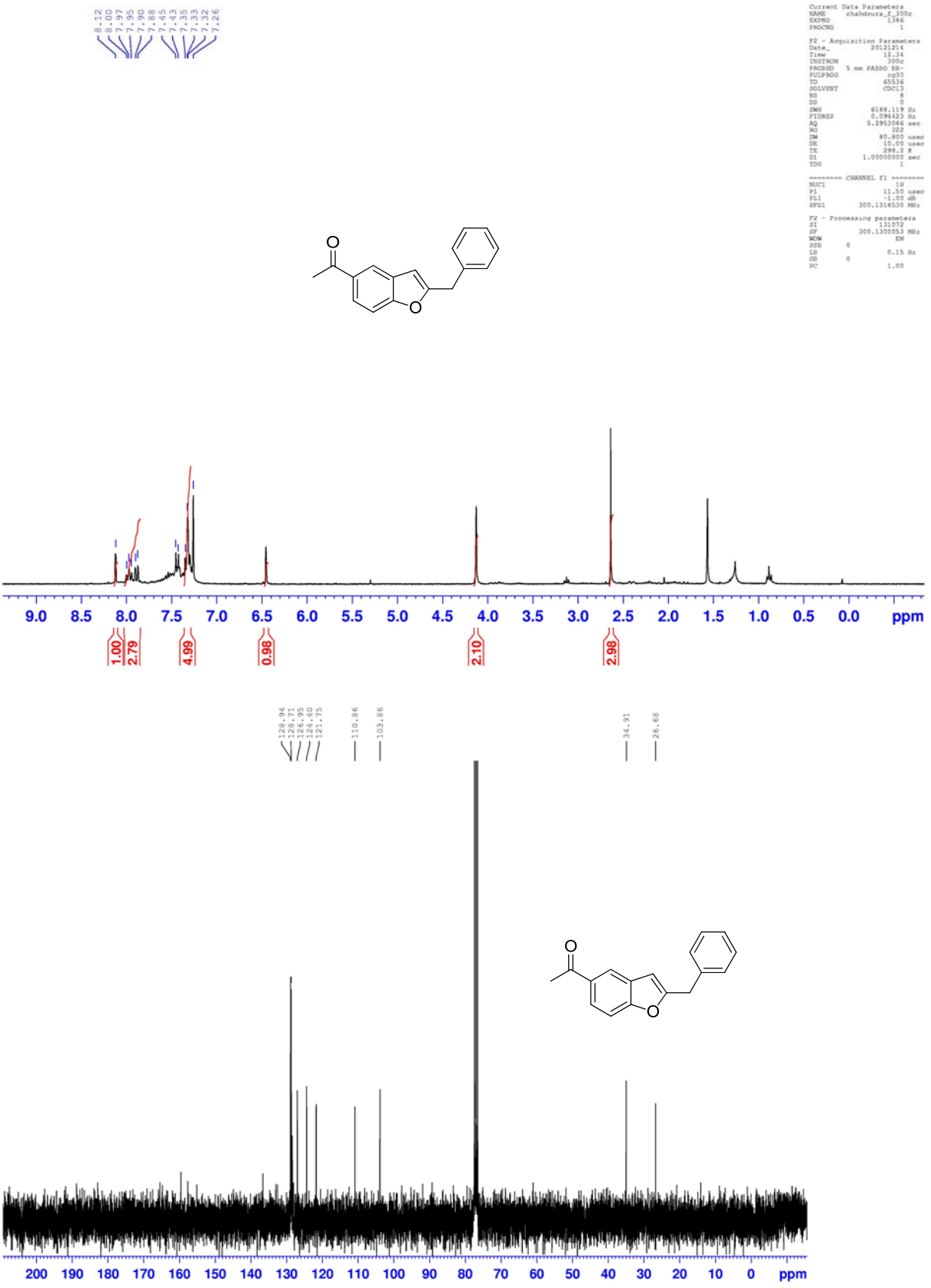

877
fc-877-2 2152 (14.763) Cm (2138:2156)
, 20-Nov-2012 + 21:08:48
Scan EI+
4.62e8
100
%
0
221.0794
236.1640
193.0947
139.0485
165.1087
82.4749
110.5539
222.1372
163.1310
237.1713
43.0924
63.0059
69.5055
115.0928
194.1284
62.0150
77.0500
96.4536
140.1007
166.1673
178.1557
39.1017
102.0883
126.0421
162.0377
207.0910
223.1328
238.2030
28
38
48
58
68
78
88
98
108
118
128
138
148
158
168
178
188
198
208
218
228
238
248
m/z



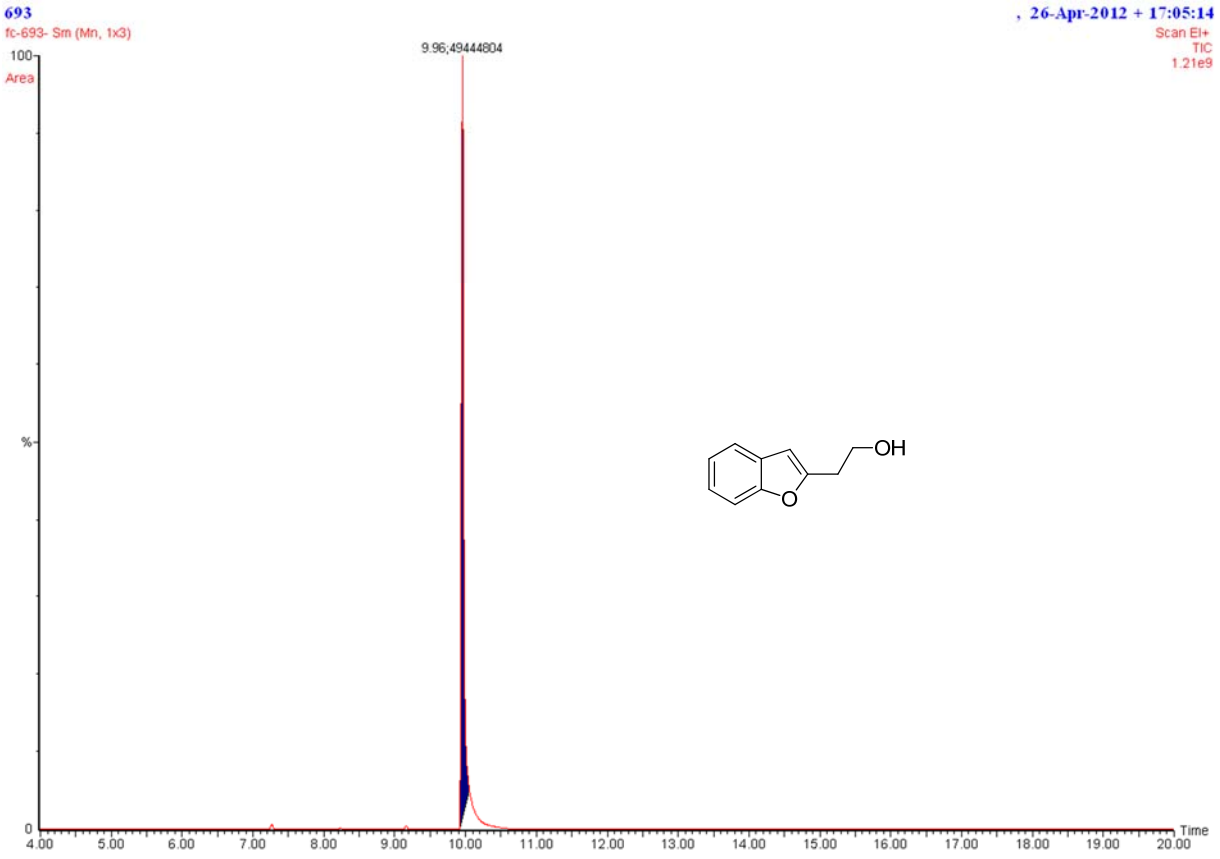
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **t33**

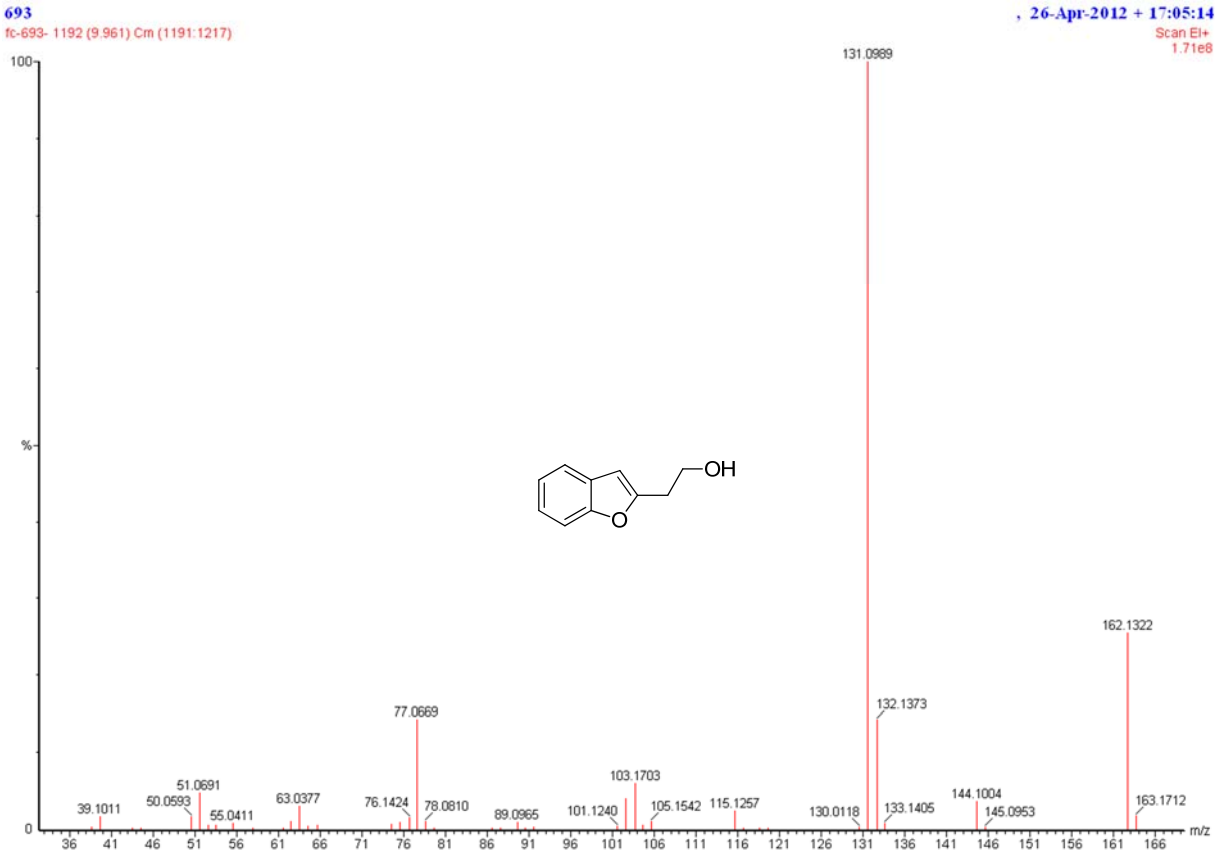

934
fc-934-2 Sm (Mn, 1x3)
, 14-Dec-2012 + 10:30:50
Scan EI+
TIC
6.64e7
100
Area
%
0
14.72;2326563
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
Time

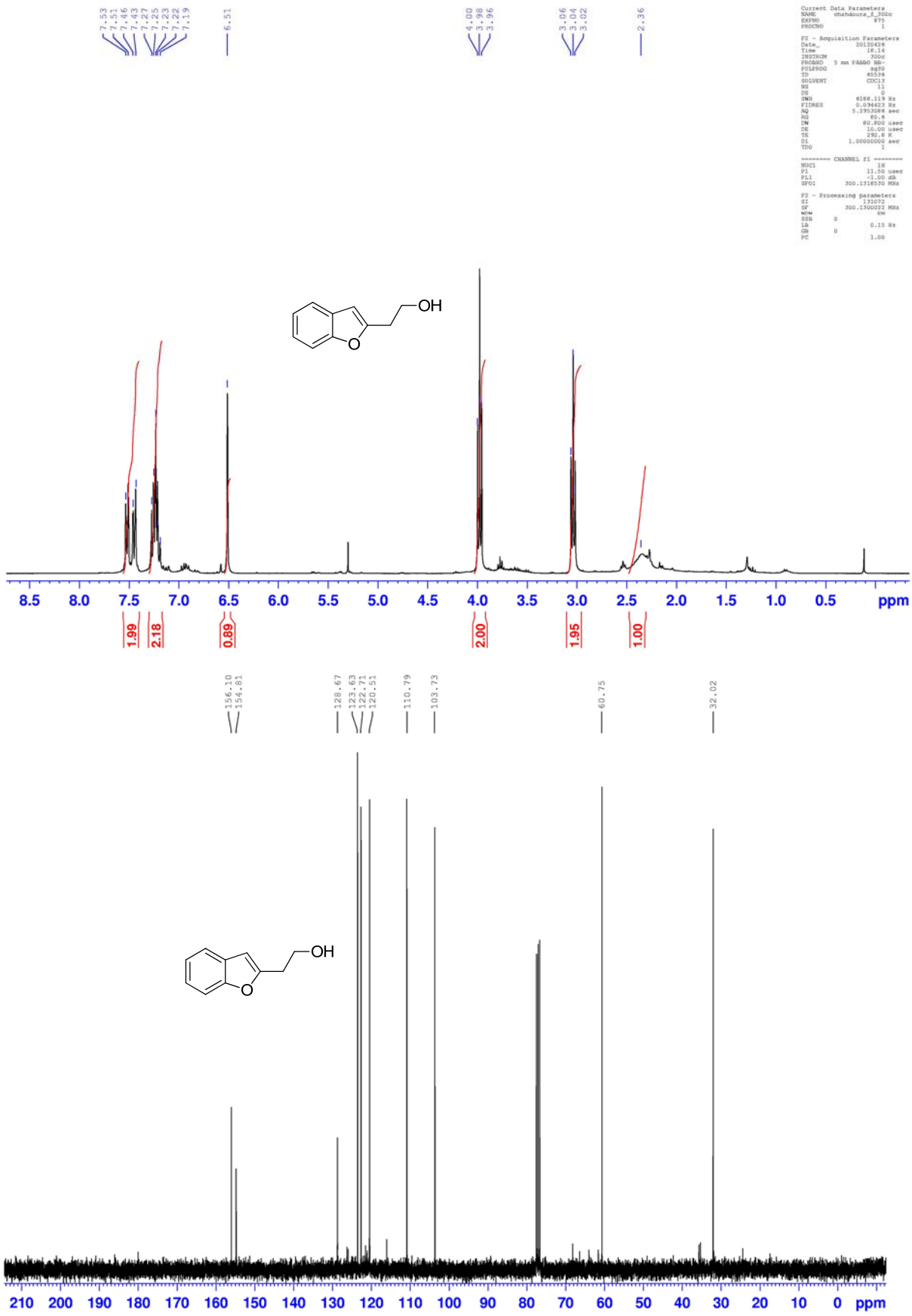

934
fc-934-2 2142 (14.713) Cm (2141:2148)
, 14-Dec-2012 + 10:30:50
Scan EI+
2.04e7
100
%
0
235.1198
250.1465
44.0222
178.1520
117.3701
207.1082
43.0300
236.1422
77.0235
89.0587
105.0608
51.0242
75.0840
249.1112
251.1758
130.0055
152.1245
173.1574
179.1726
36
46
56
66
76
86
96
106
116
126
136
146
156
166
176
186
196
206
216
226
236
246
256
m/z



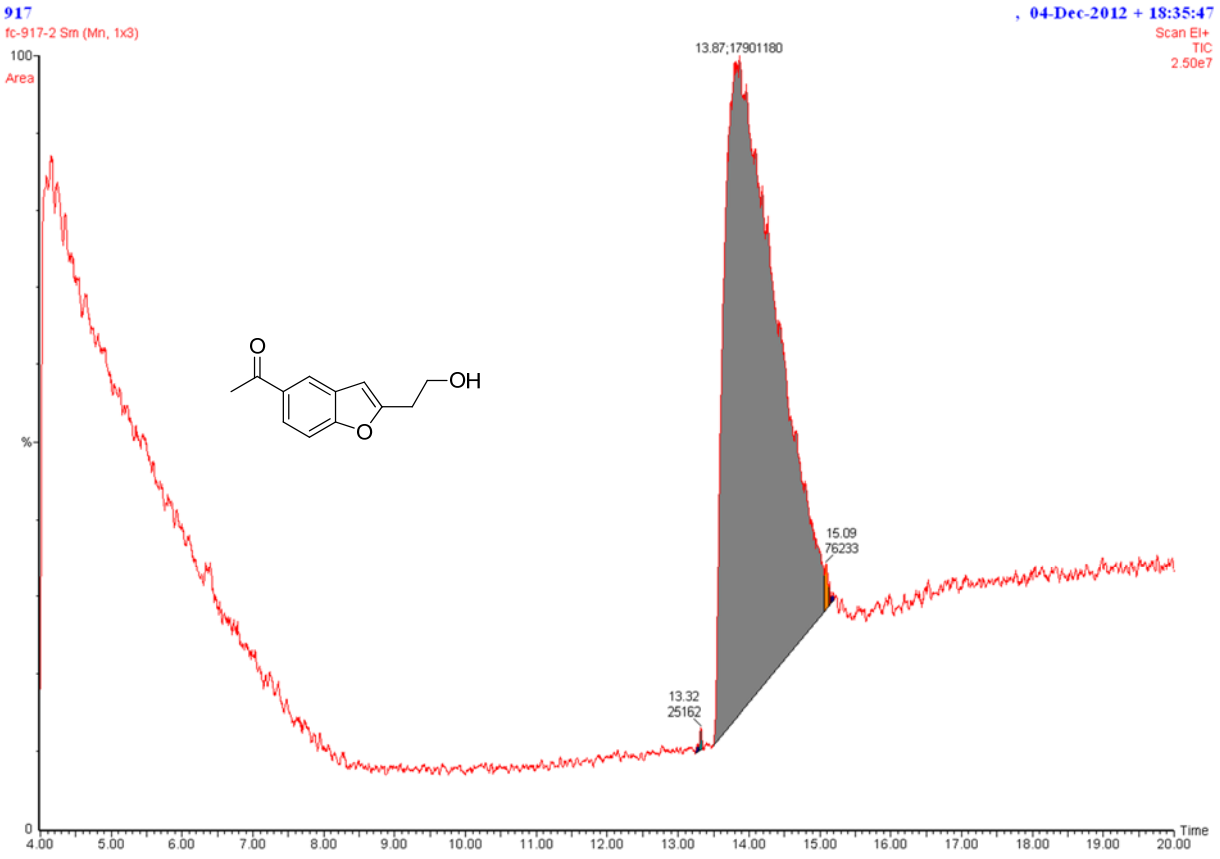
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **t34**

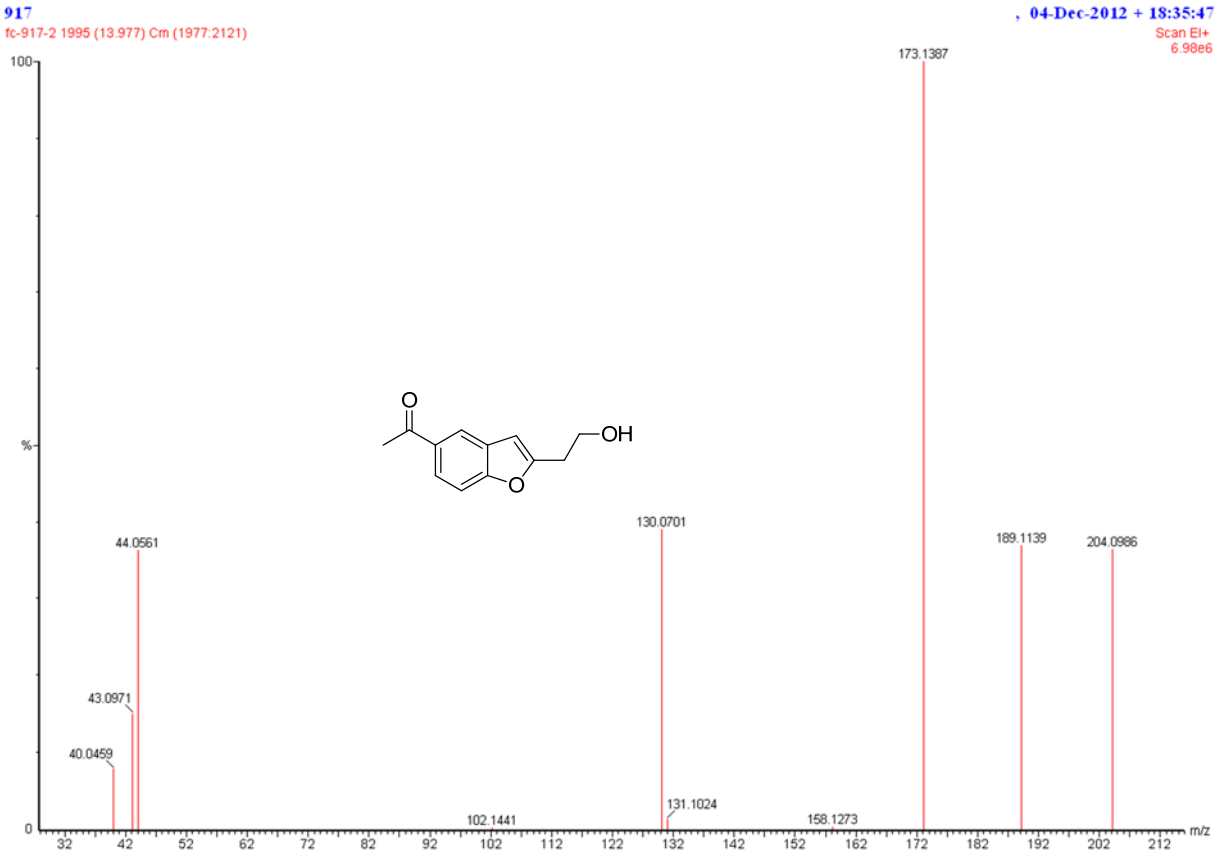

693
fc-693- Sm (Mn, 1x3)
, 26-Apr-2012 + 17:05:14
Scan EI+
TIC
1.21e9
9.96;49444804
100
Area
%
0
OH
O
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00
Time

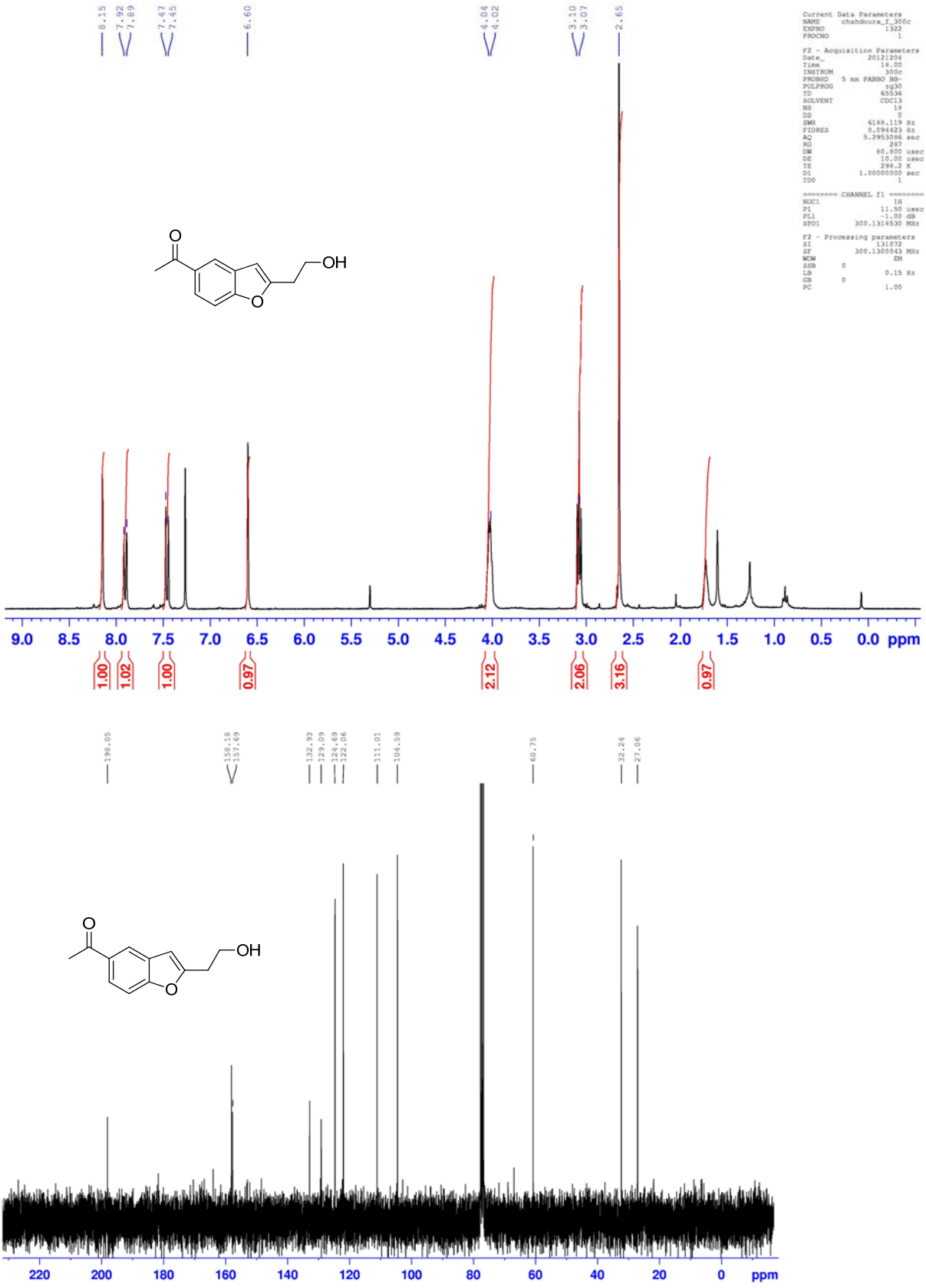

693
fc-693- 1192 (9.961) Cm (1191:1217)
, 26-Apr-2012 + 17:05:14
Scan EI+
1.71e8
100
%
0
131.0969
OH
O
162.1322
132.1373
77.0669
103.1703
51.0691
39.1011
50.0593
55.0411
63.0377
76.1424
78.0810
89.0965
101.1240
105.1542
115.1257
130.0118
133.1405
144.1004
145.0953
163.1712
36 41 46 51 56 61 66 71 76 81 86 91 96 101 106 111 116 121 126 131 136 141 146 151 156 161 166
m/z



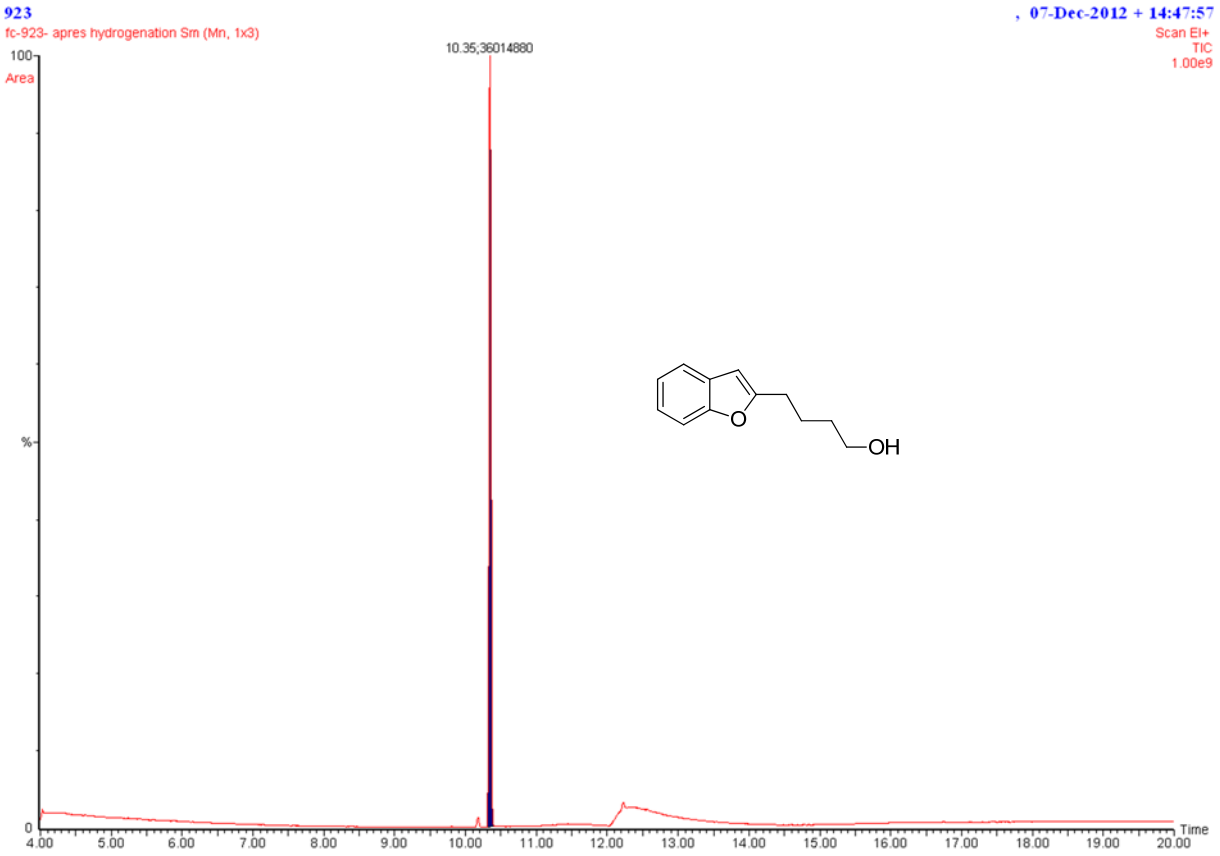
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **s35**

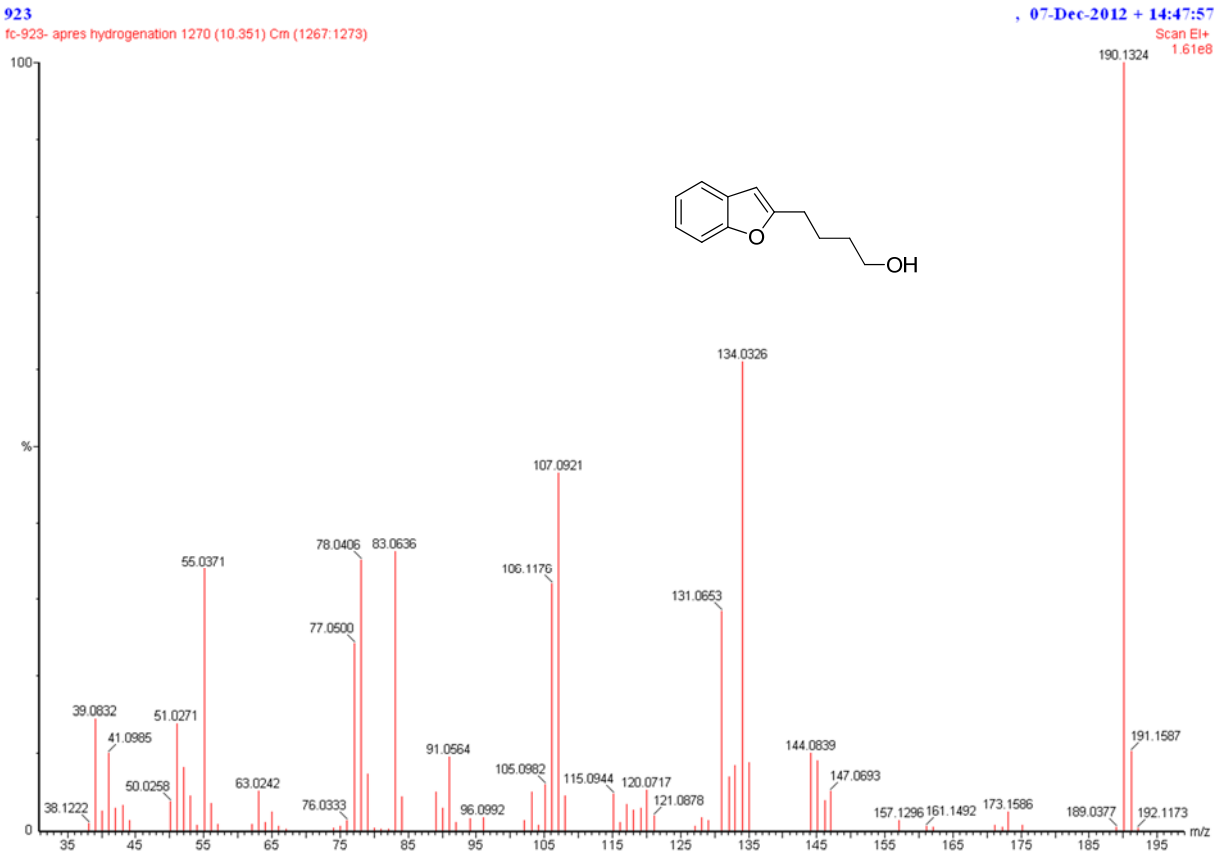

917
fc-917-2 Sm (Mn, 1x3)
, 04-Dec-2012 + 18:35:47
Scan EI+
TIC
2.50e7
100
Area
%
0
13.87;17901180
15.09
76233
13.32
25162
O
OH
O
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

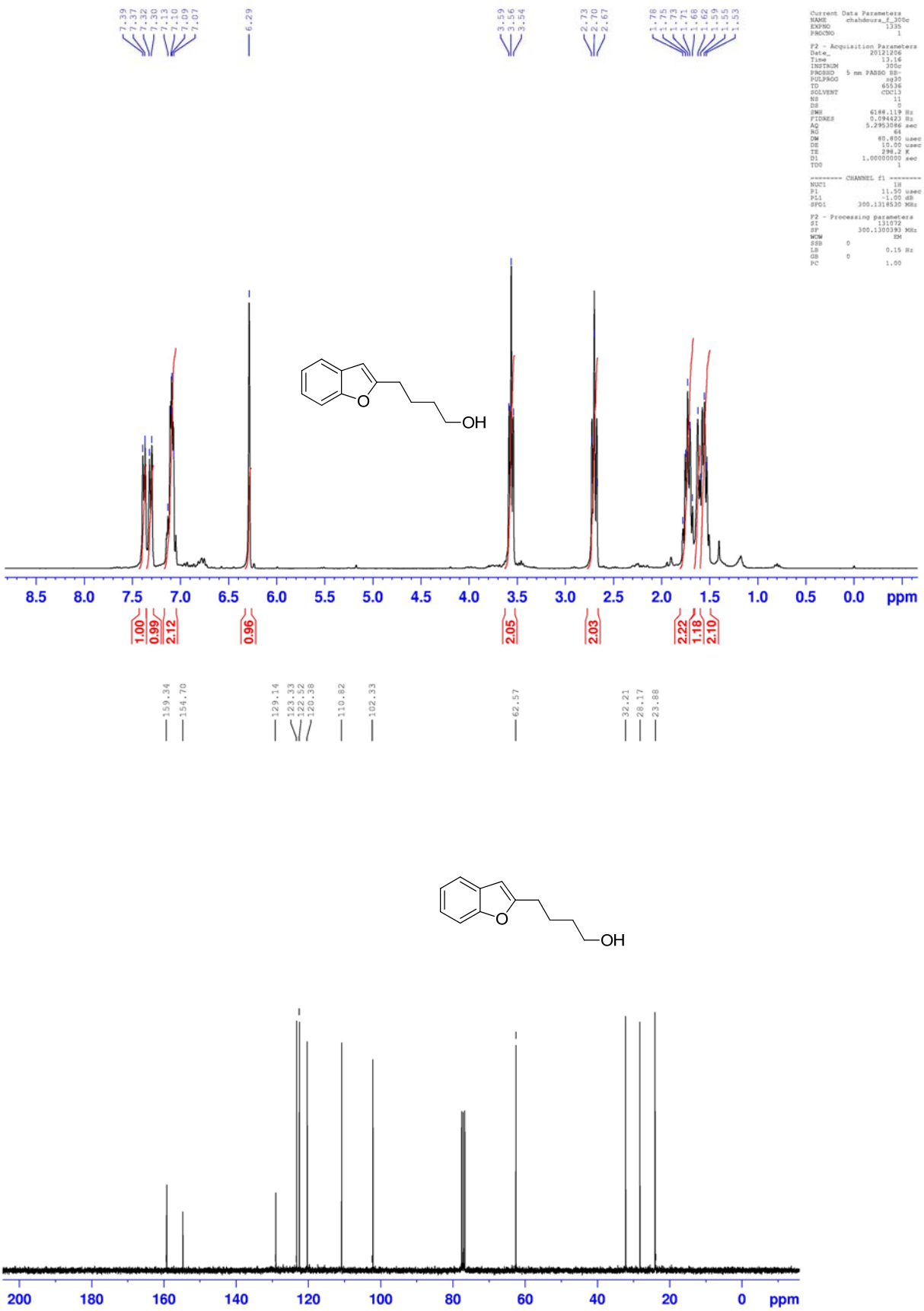

917
fc-917-2 1995 (13.977) Cm (1977:2121)
, 04-Dec-2012 + 18:35:47
Scan EI+
6.98e6
100
%
0
173.1387
130.0701
44.0561
189.1139
204.0986
43.0971
40.0459
102.1441
131.1024
158.1273
O
OH
O
32
42
52
62
72
82
92
102
112
122
132
142
152
162
172
182
192
202
212
m/z



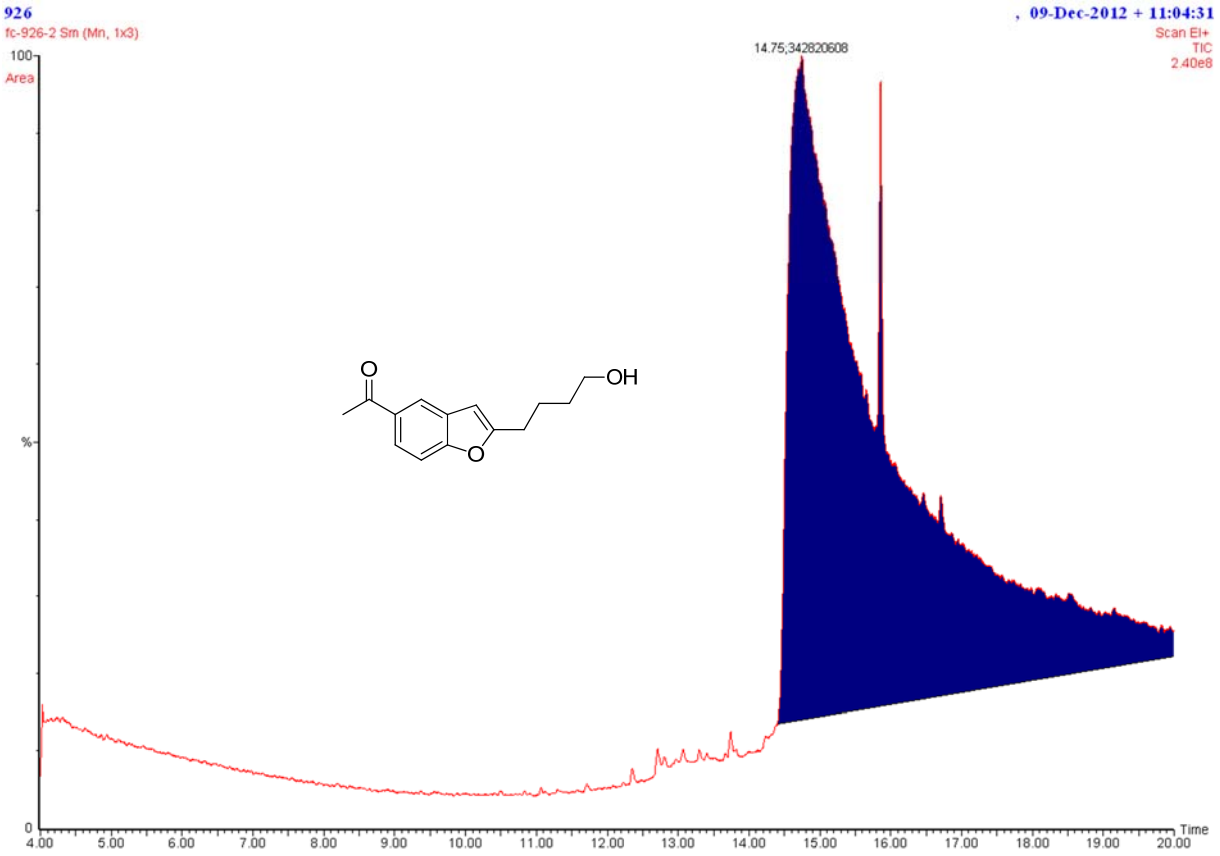
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **t35**

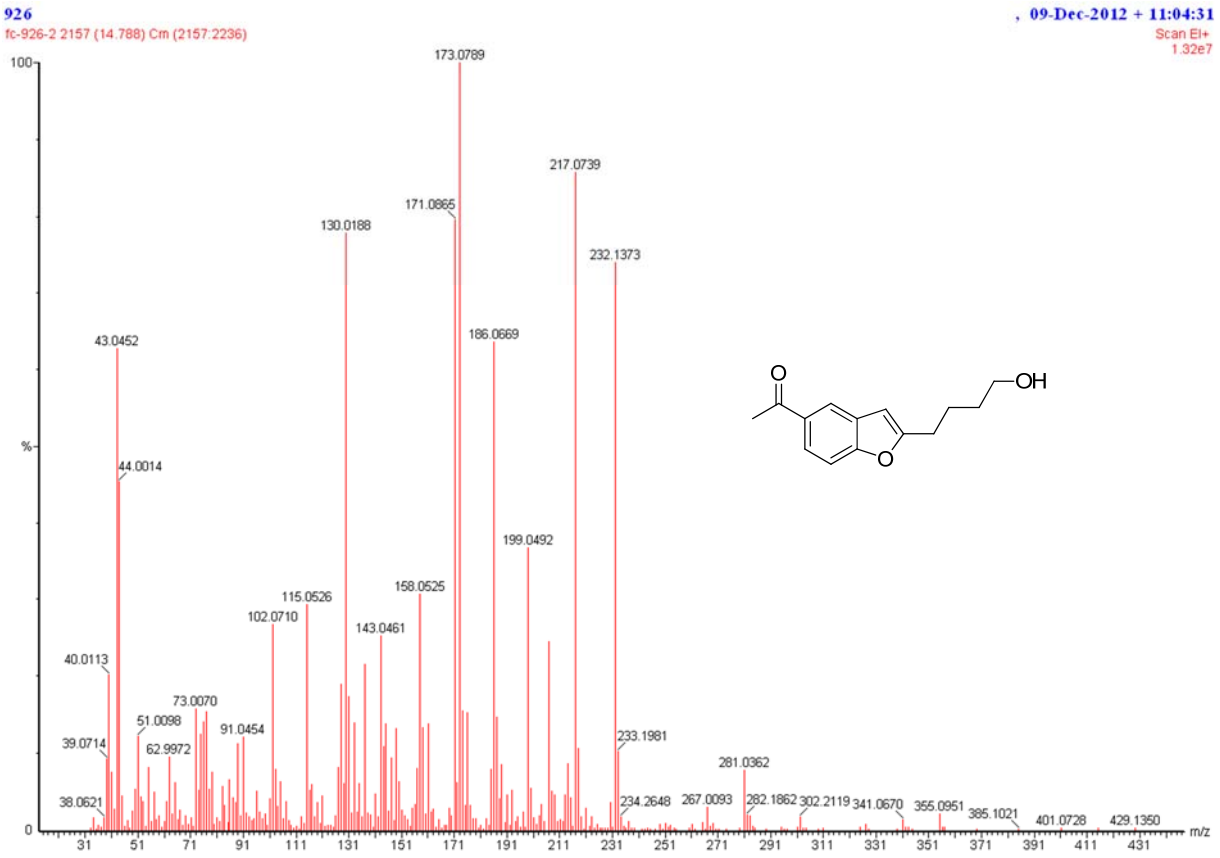

923
fc-923- apres hydrogenation Sm (Mn, 1x3)
, 07-Dec-2012 + 14:47:57
Scan EI+
TIC
1.00e9
100
Area
%
0
10.35;36014880
O
OH
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

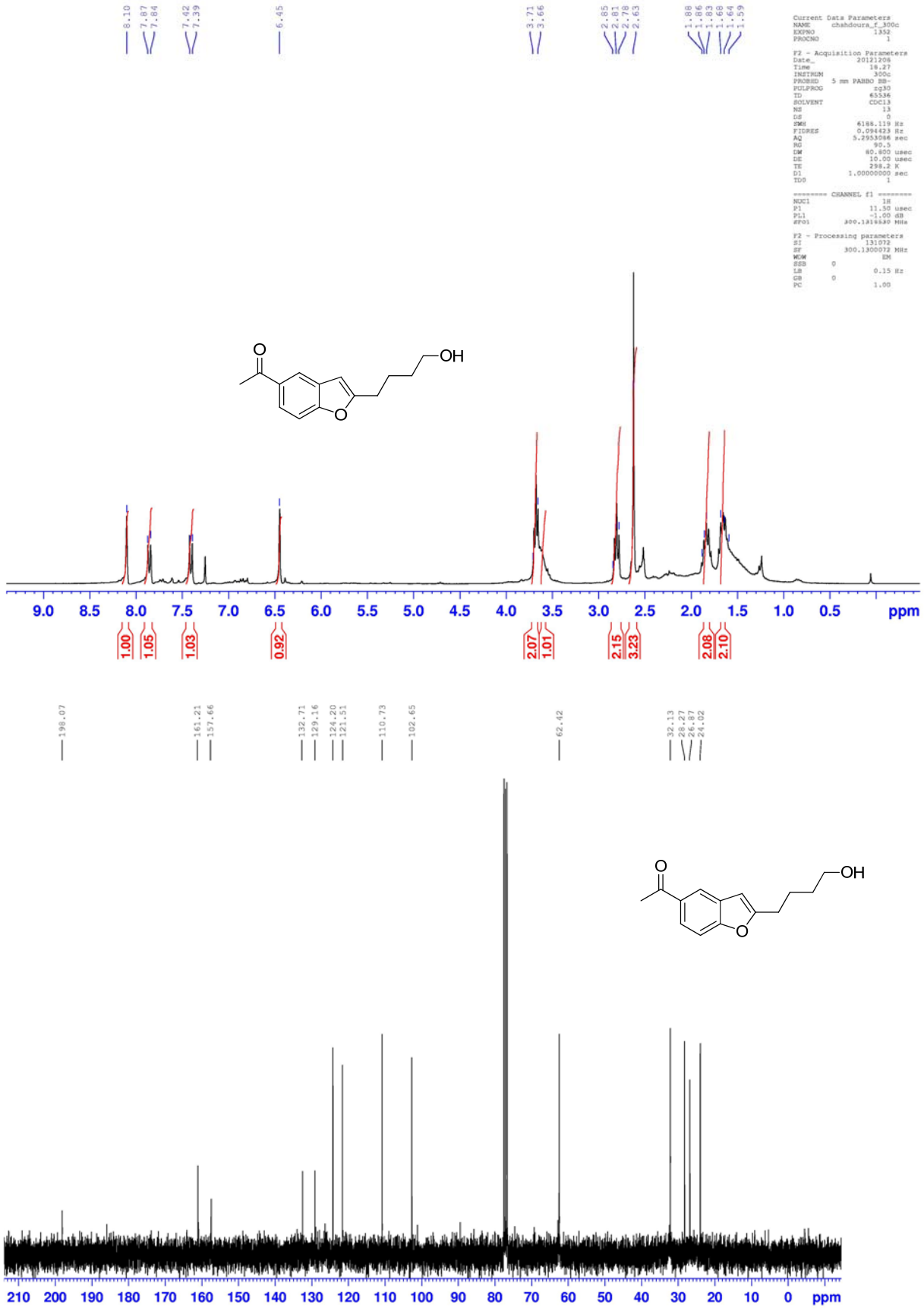

923
fc-923- apres hydrogenation 1270 (10.351) Cm (1267:1273)
, 07-Dec-2012 + 14:47:57
Scan EI+
1.61e8
100
%
0
190.1324
O
OH
134.0326
107.0921
78.0406
83.0636
55.0371
106.1176
131.0653
77.0500
39.0832
51.0271
41.0985
91.0564
144.0839
191.1587
105.0982
115.0944
120.0717
147.0693
50.0258
63.0242
121.0878
38.1222
76.0333
96.0992
157.1296
161.1492
173.1586
189.0377
192.1173
35
45
55
65
75
85
95
105
115
125
135
145
155
165
175
185
195
m/z



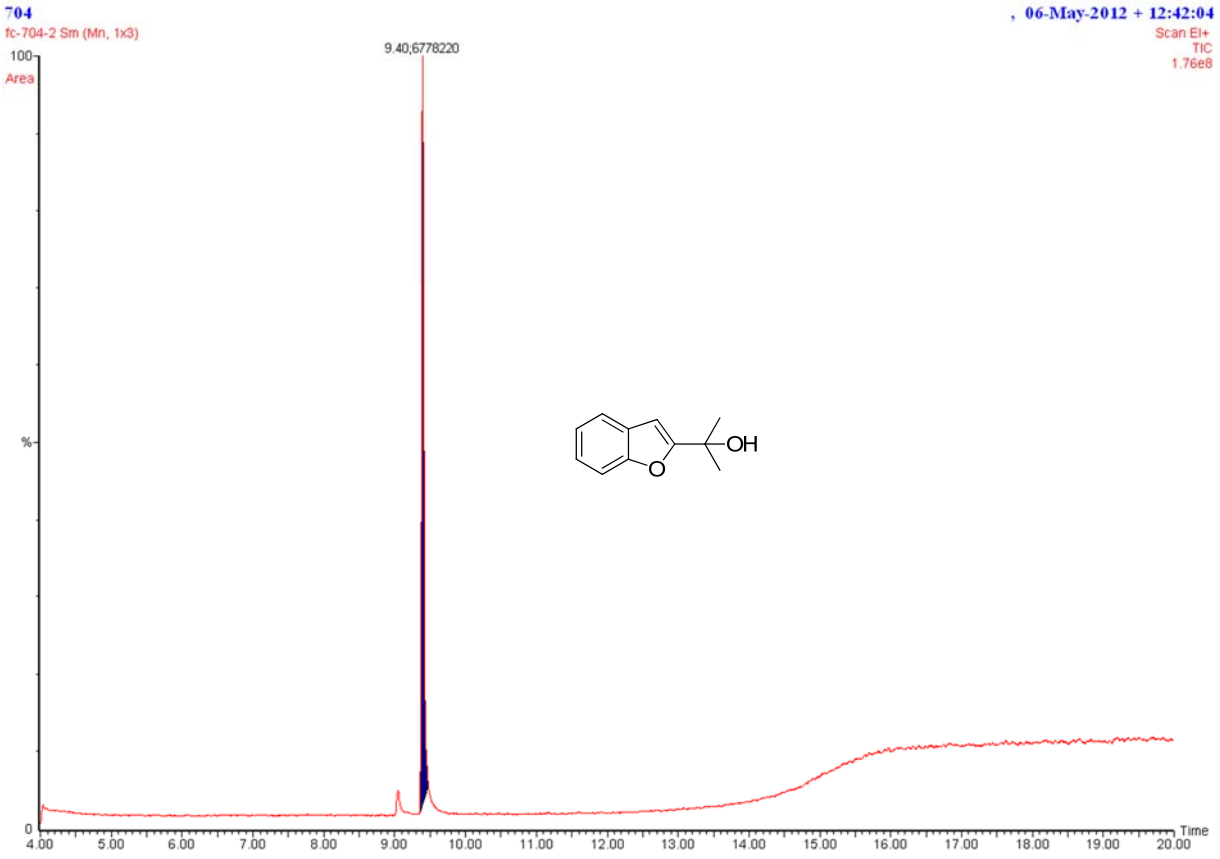
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **s36**

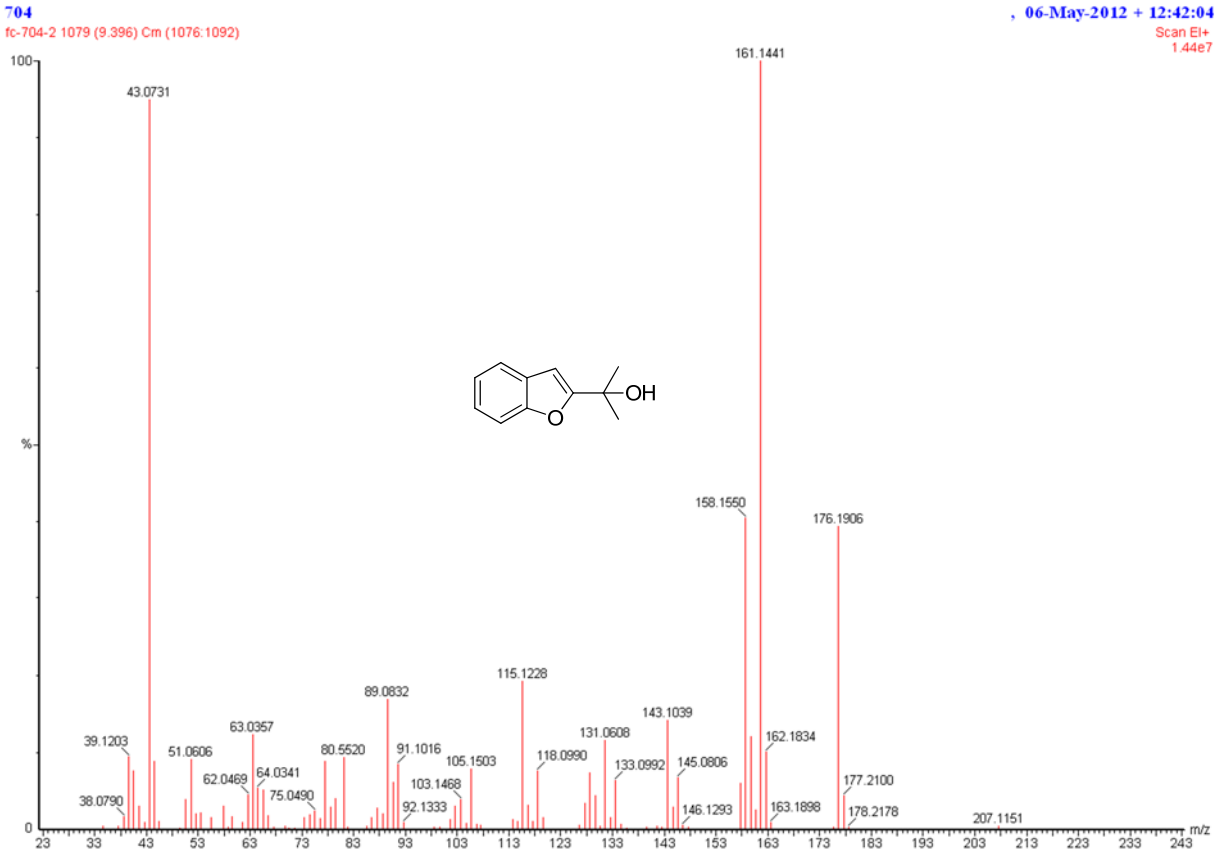

926
fc-926-2 Sm (Mn, 1x3)
, 09-Dec-2012 + 11:04:31
Scan EI+
TIC
2.40e8
100
Area
%
0
14.75;342820608
O
OH
O
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

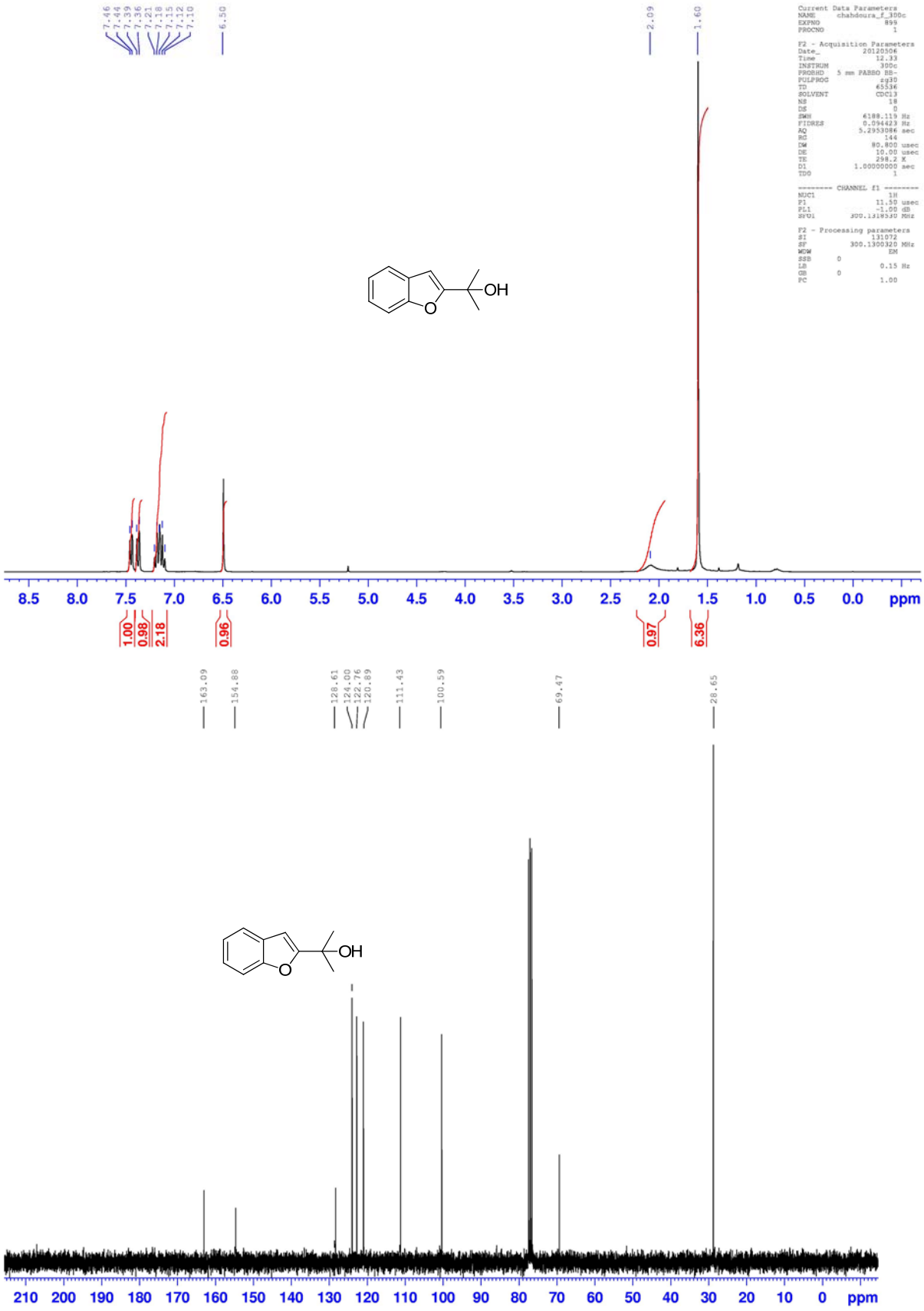

926
fc-926-2 2157 (14.788) Cm (2157:2236)
, 09-Dec-2012 + 11:04:31
Scan EI+
1.32e7
100
%
0
173.0789
217.0739
171.0865
130.0188
232.1373
186.0669
43.0452
44.0014
199.0492
158.0525
115.0526
102.0710
143.0461
40.0113
73.0070
51.0098
91.0454
39.0714
62.9972
233.1981
281.0362
38.0621
234.2648
267.0093
282.1862
302.2119
341.0670
355.0951
385.1021
401.0728
429.1350
O
OH
O
31
51
71
91
111
131
151
171
191
211
231
251
271
291
311
331
351
371
391
411
431
m/z



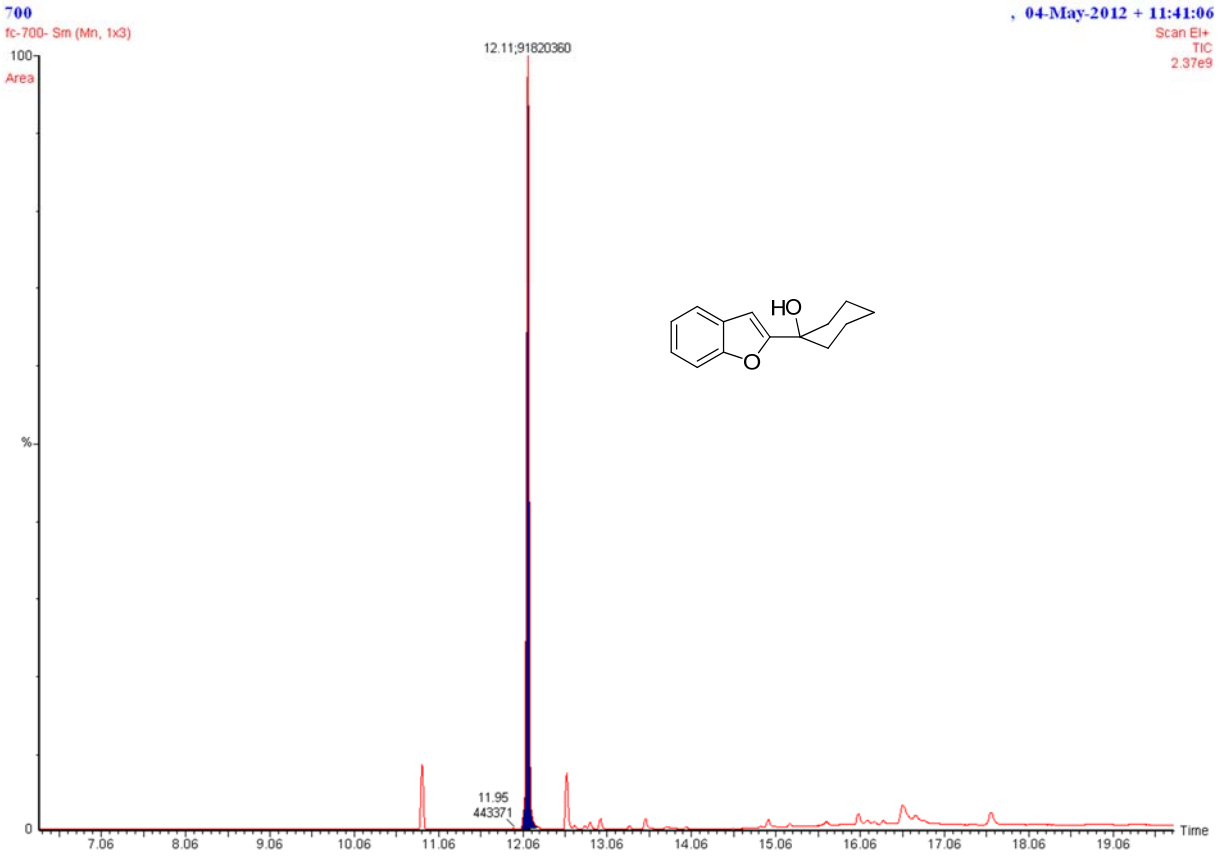
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **t36**

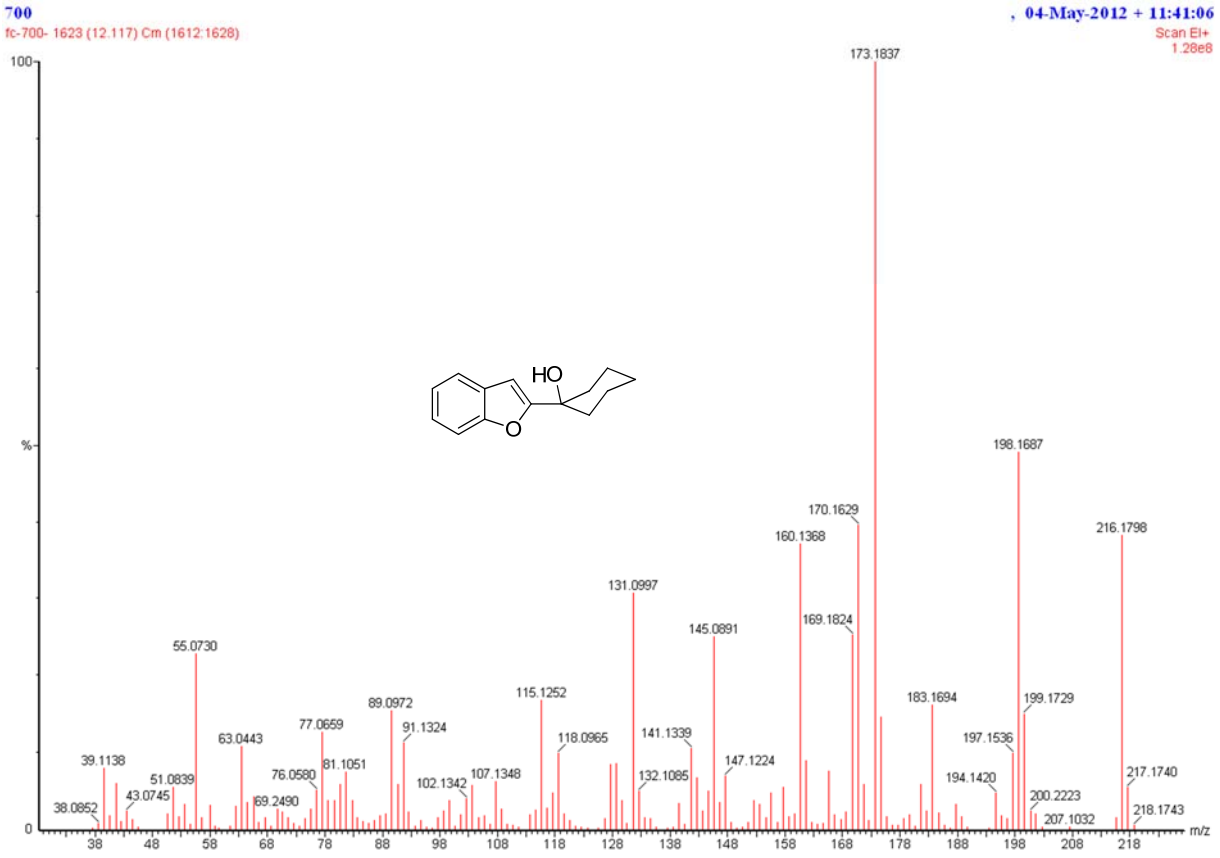

704
fc-704-2 Sm (Mn, 1x3)
, 06-May-2012 + 12:42:04
Scan EI+
TIC
1.76e8
100
Area
%
0
9.40;6778220
OH
O
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

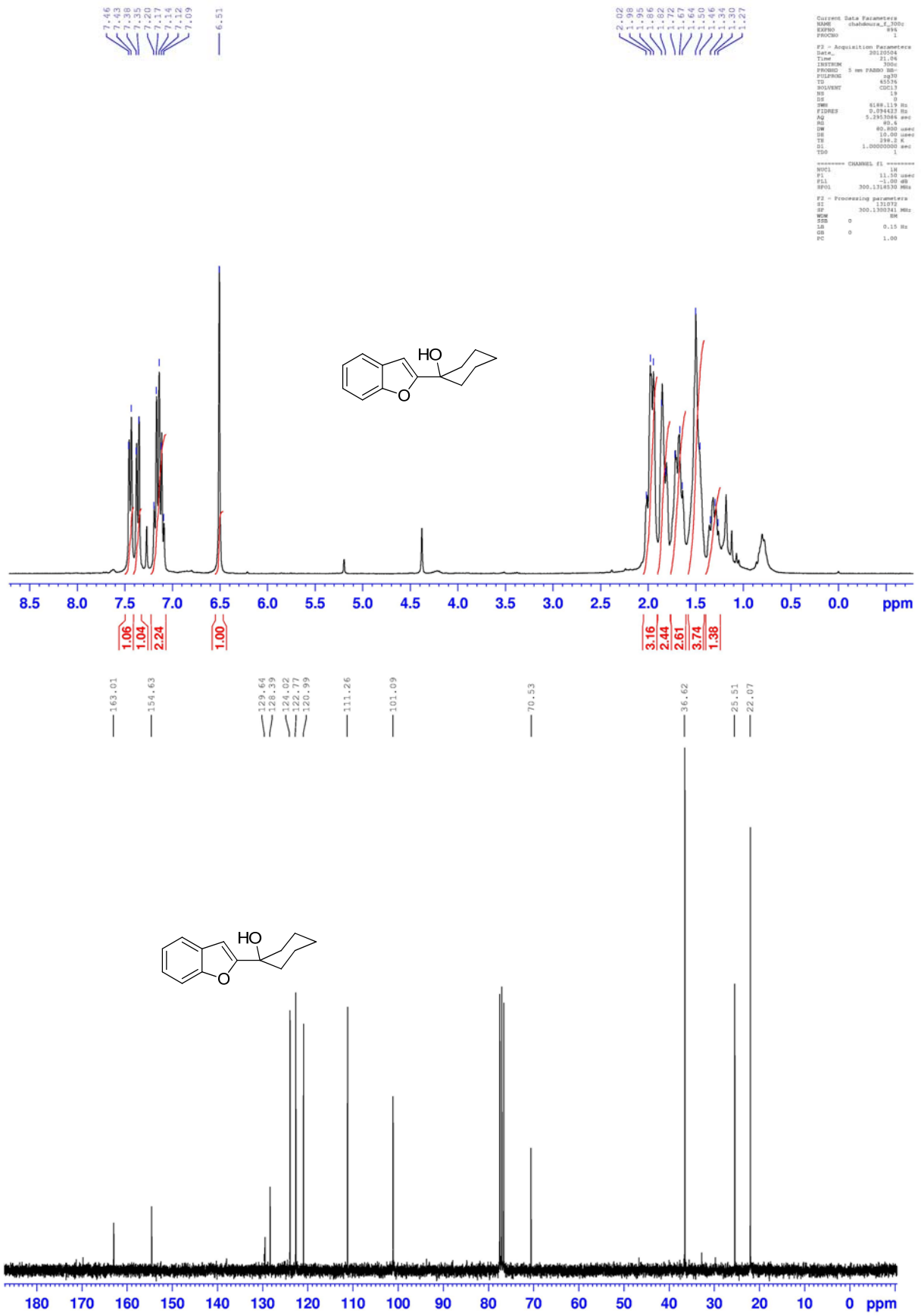

704
fc-704-2 1079 (9.396) Cm (1076:1092)
, 06-May-2012 + 12:42:04
Scan EI+
1.44e7
100
%
0
43.0731
161.1441
158.1550
176.1906
115.1228
89.0832
143.1039
131.0608
162.1834
39.1203
63.0357
51.0606
80.5520
91.1016
105.1503
118.0990
133.0992
145.0806
62.0469
64.0341
103.1468
177.2100
38.0790
75.0490
92.1333
146.1293
163.1898
178.2178
207.1151
OH
O
23
33
43
53
63
73
83
93
103
113
123
133
143
153
163
173
183
193
203
213
223
233
243
m/z



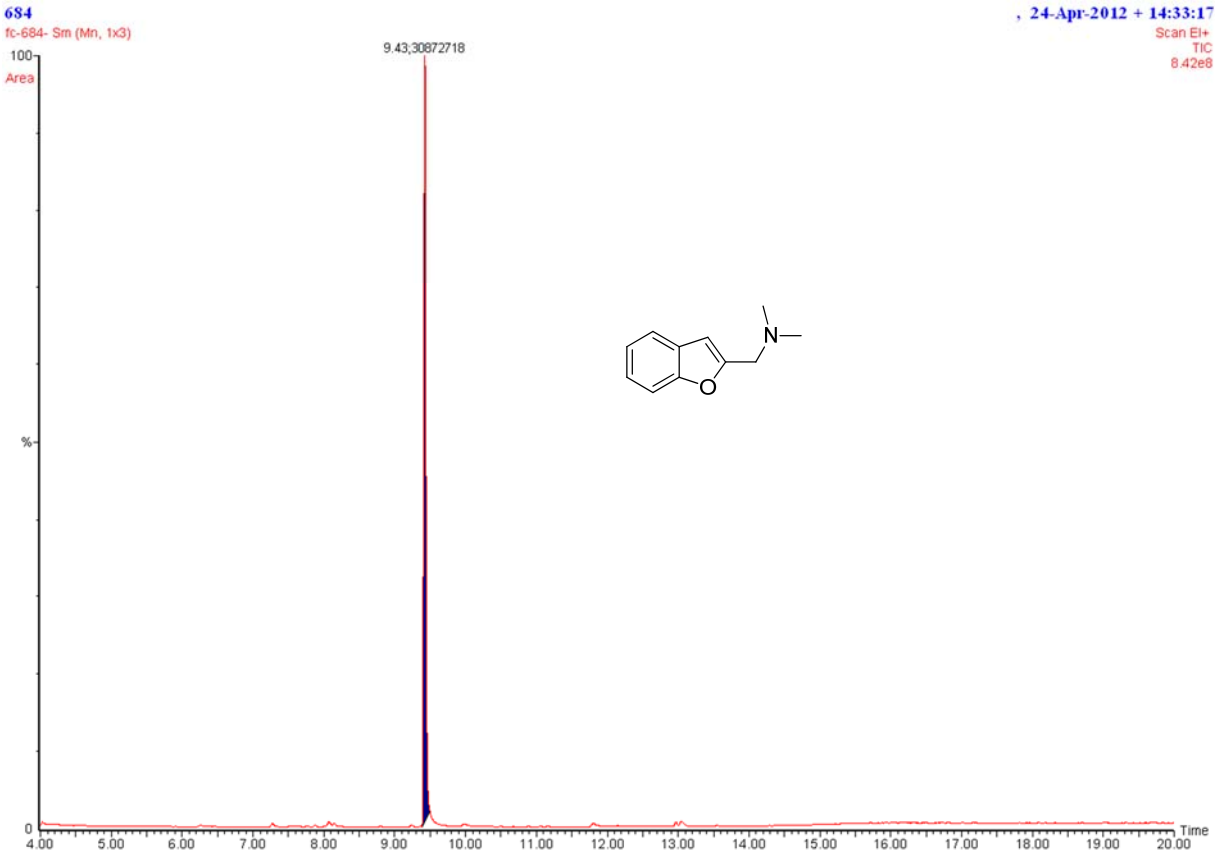
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **s37**

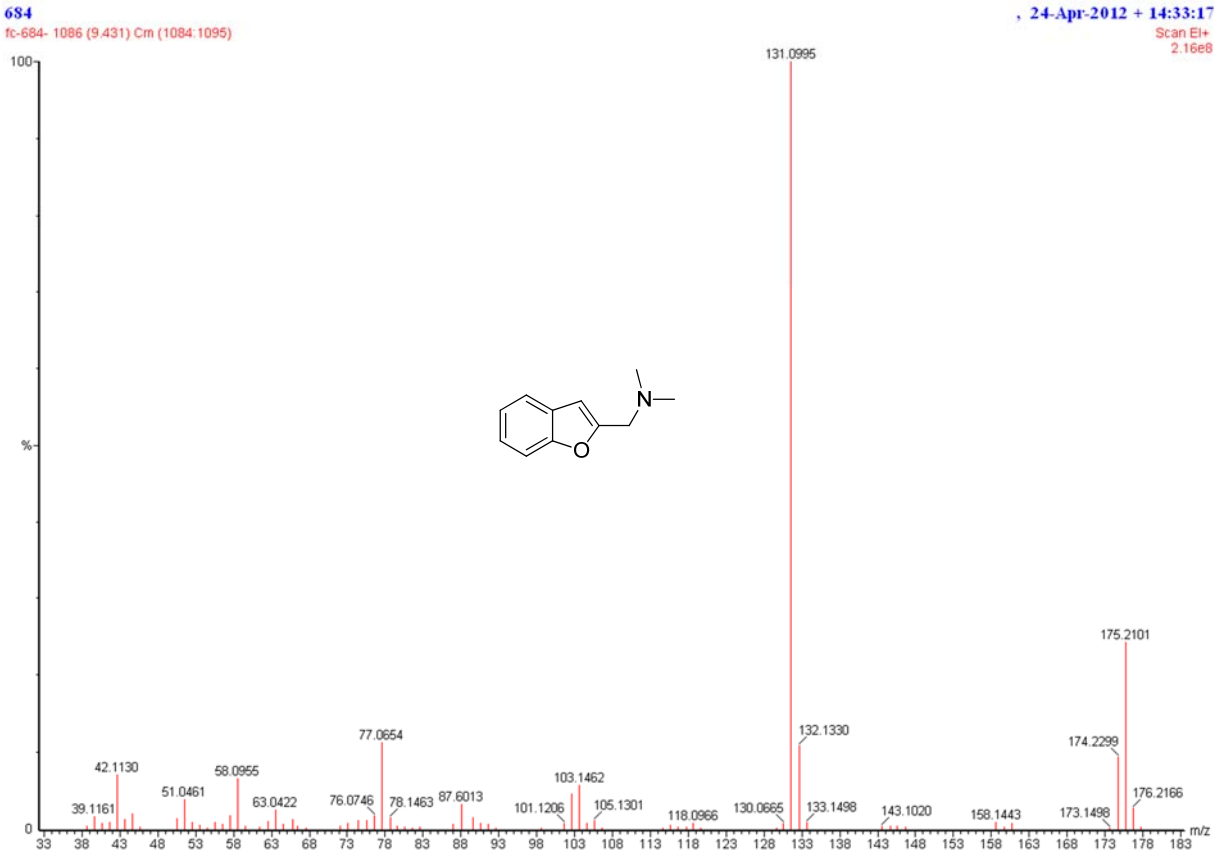

700
fc-700- Sm (Mn, 1x3)
, 04-May-2012 + 11:41:06
Scan EI+
TIC
2.37e9
100
Area
%
0
12.11;91820360
11.95
443371
HO
O
7.06
8.06
9.06
10.06
11.06
12.06
13.06
14.06
15.06
16.06
17.06
18.06
19.06
Time

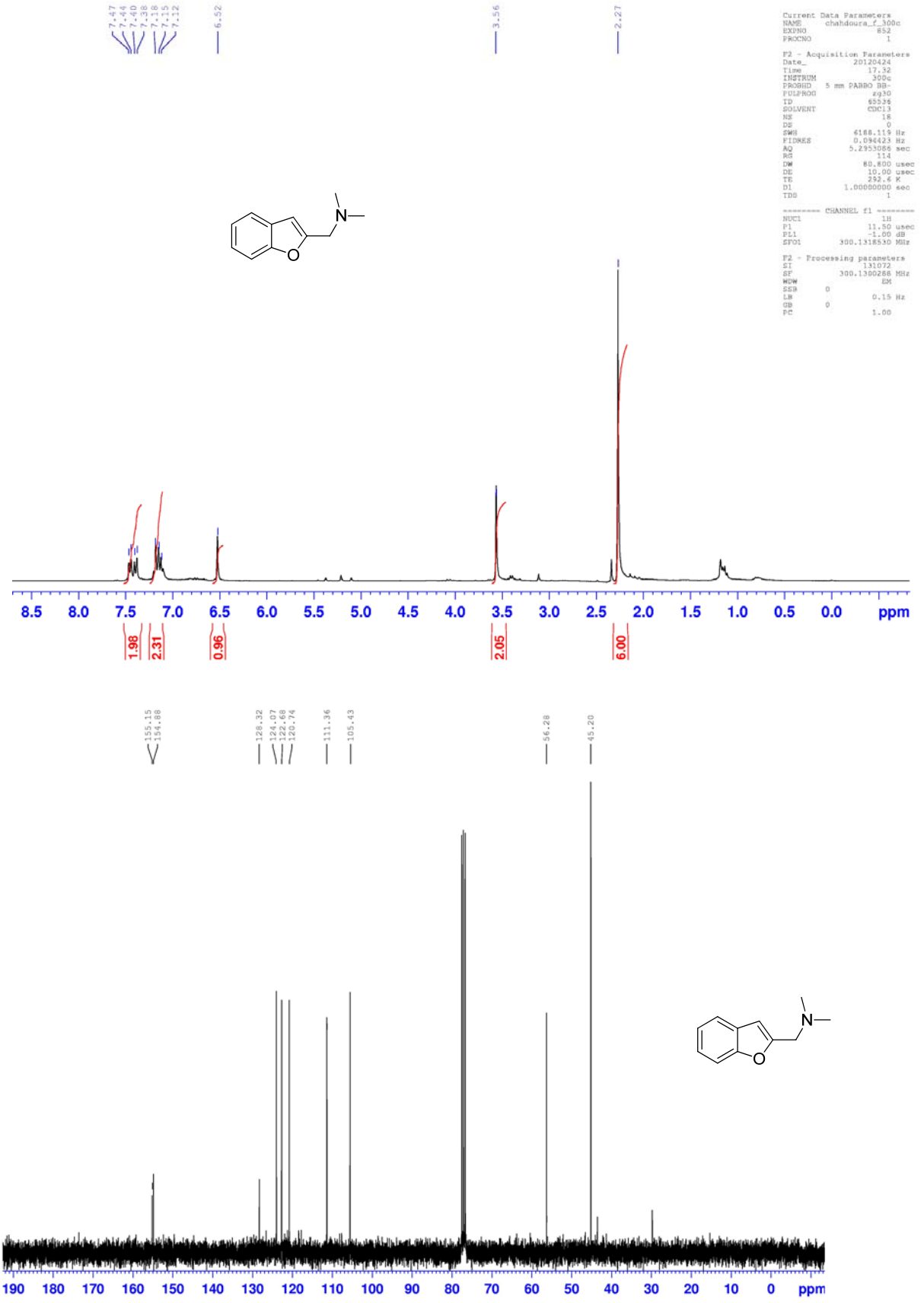

700
fc-700- 1623 (12.117) Cm (1612:1628)
, 04-May-2012 + 11:41:06
Scan EI+
1.28e8
100
%
0
HO
O
173.1837
198.1687
170.1629
160.1368
216.1798
131.0997
169.1824
145.0891
55.0730
115.1252
89.0972
183.1694
199.1729
91.1324
77.0659
118.0965
141.1339
197.1536
63.0443
39.1138
147.1224
81.1051
76.0580
107.1348
132.1085
194.1420
217.1740
51.0839
102.1342
43.0745
69.2490
200.2223
38.0852
207.1032
218.1743
38
48
58
68
78
88
98
108
118
128
138
148
158
168
178
188
198
208
218
m/z



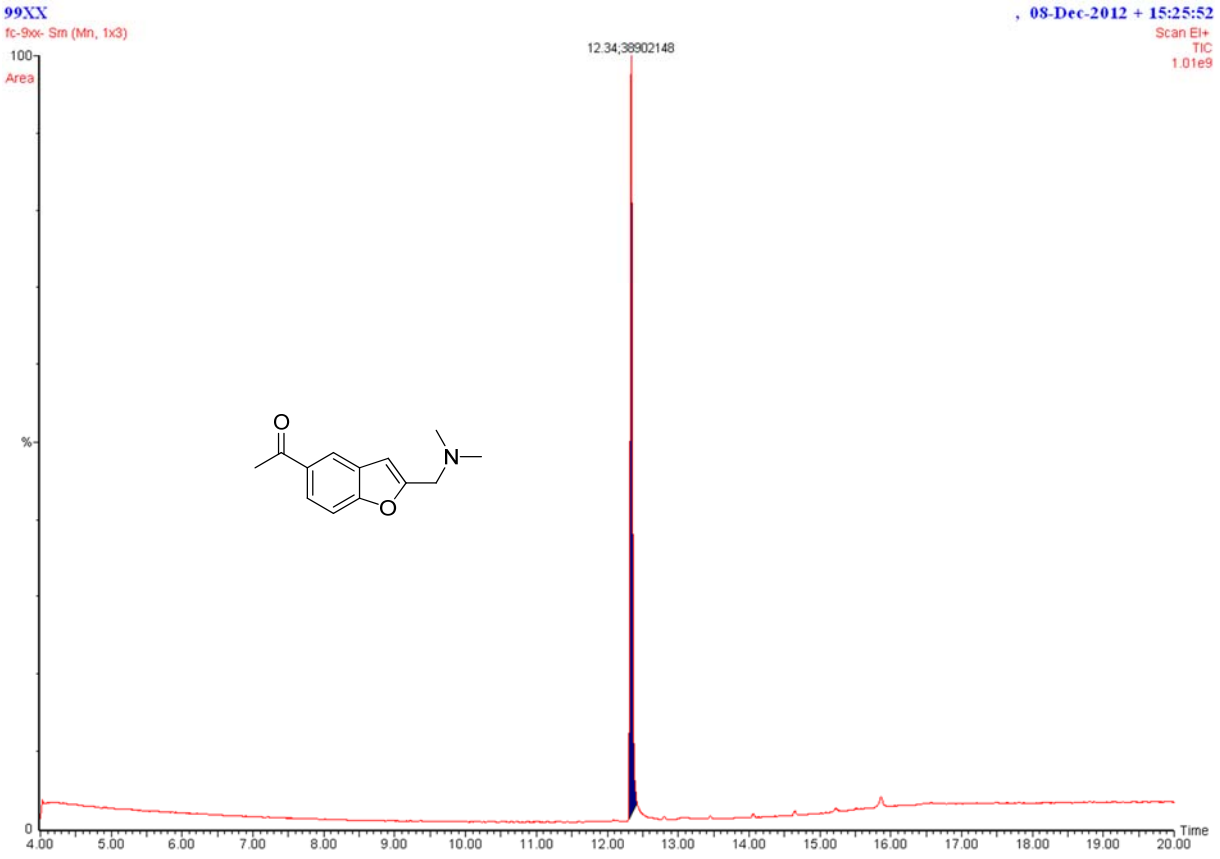
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **s38**

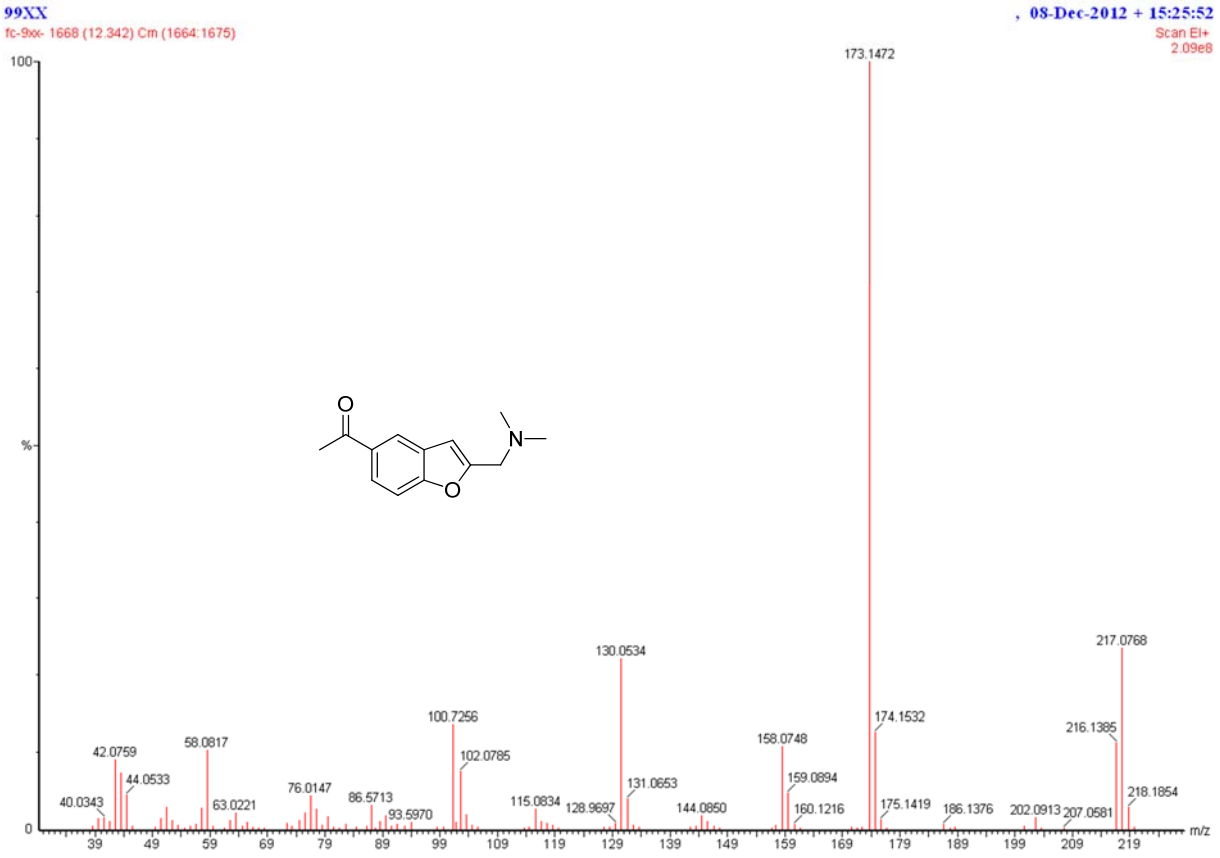

684
fc-684- Sm (Mn, 1x3)
, 24-Apr-2012 + 14:33:17
Scan EI+
TIC
8.42e8
9.43;30872718
100
Area
%
0
Time
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
N
O

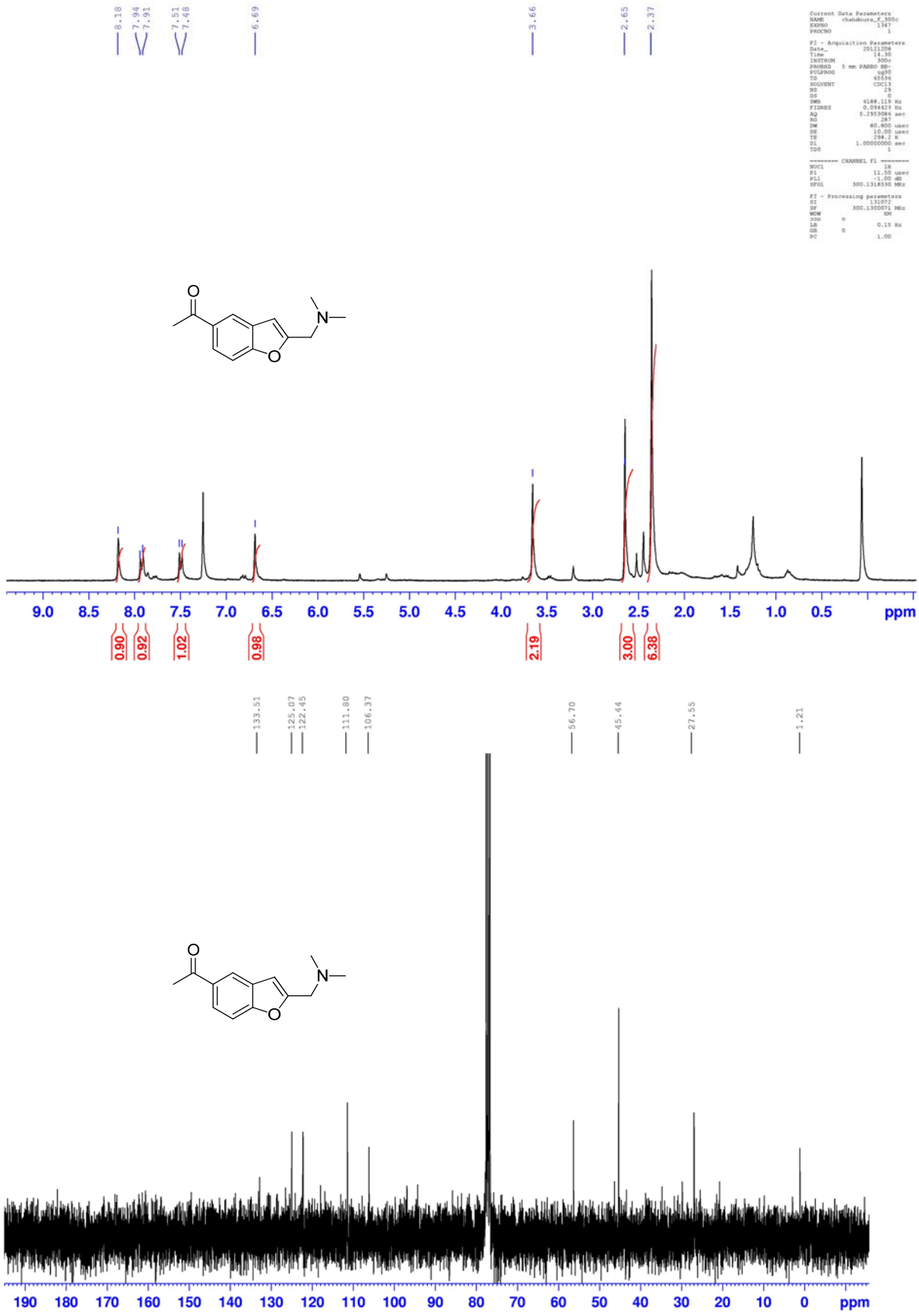

684
fc-684- 1086 (9.431) Cm (1084:1095)
, 24-Apr-2012 + 14:33:17
Scan EI+
2.16e8
131.0995
100
%
0
m/z
N
O
175.2101
132.1330
174.2299
77.0654
42.1130
58.0955
176.2166
103.1462
51.0461
39.1161
63.0422
76.0746
78.1463
87.6013
101.1206
105.1301
118.0966
130.0665
133.1498
143.1020
158.1443
173.1498
33
38
43
48
53
58
63
68
73
78
83
88
93
98
103
108
113
118
123
128
133
138
143
148
153
158
163
168
173
178
183



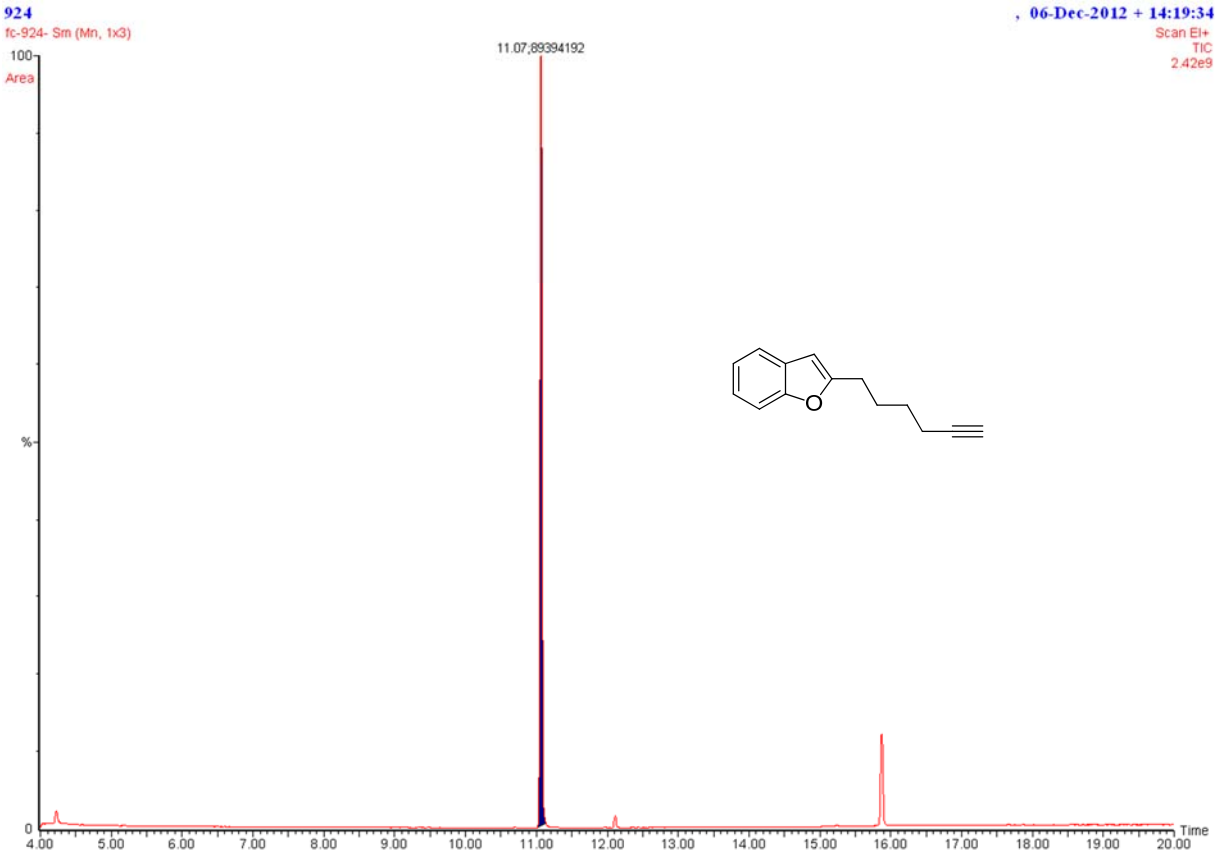
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **s39**

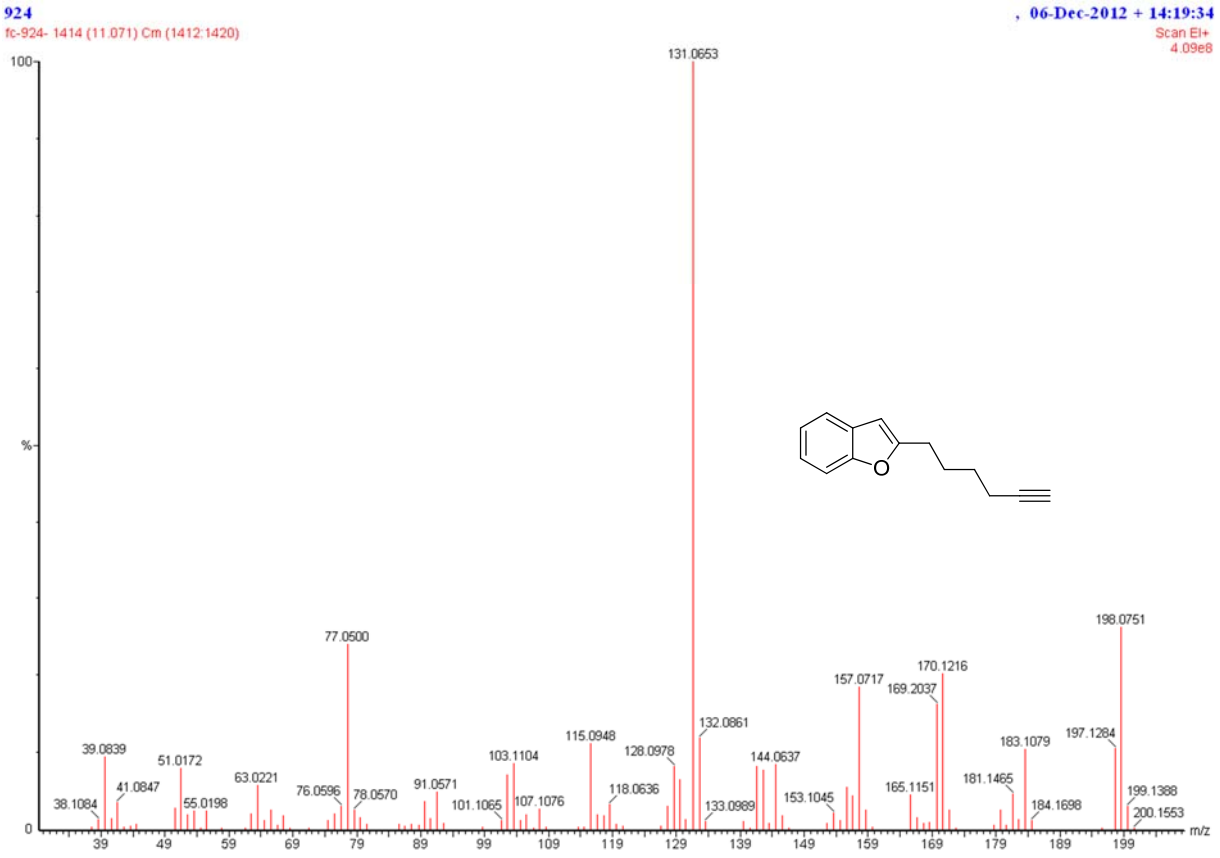

99XX
fc-9xx- Sm (Mn, 1x3)
, 08-Dec-2012 + 15:25:52
Scan EI+
TIC
1.01e9
12.34;38902148
100
Area
%
0
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N
O

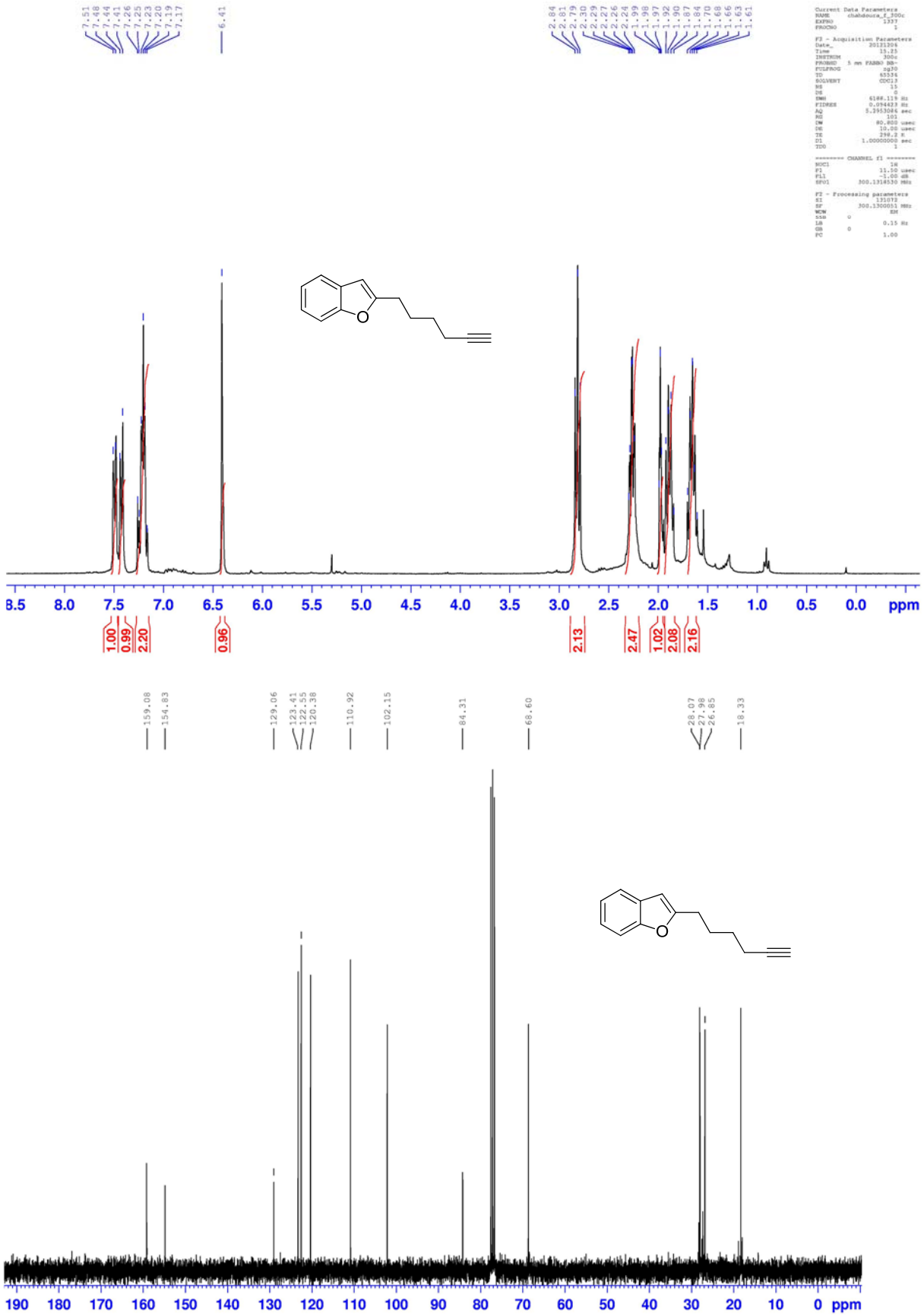

99XX
fc-9xx- 1668 (12.342) Cm (1664:1675)
, 08-Dec-2012 + 15:25:52
Scan EI+
2.09e8
173.1472
100
%
0
130.0534
217.0768
100.7256
174.1532
216.1385
58.0817
158.0748
42.0759
102.0785
159.0894
44.0533
131.0653
218.1854
76.0147
160.1216
40.0343
63.0221
86.5713
175.1419
186.1376
202.0913
207.0581
93.5970
115.0834
128.9697
144.0850
39
49
59
69
79
89
99
109
119
129
139
149
159
169
179
189
199
209
219
m/z
O
N
O



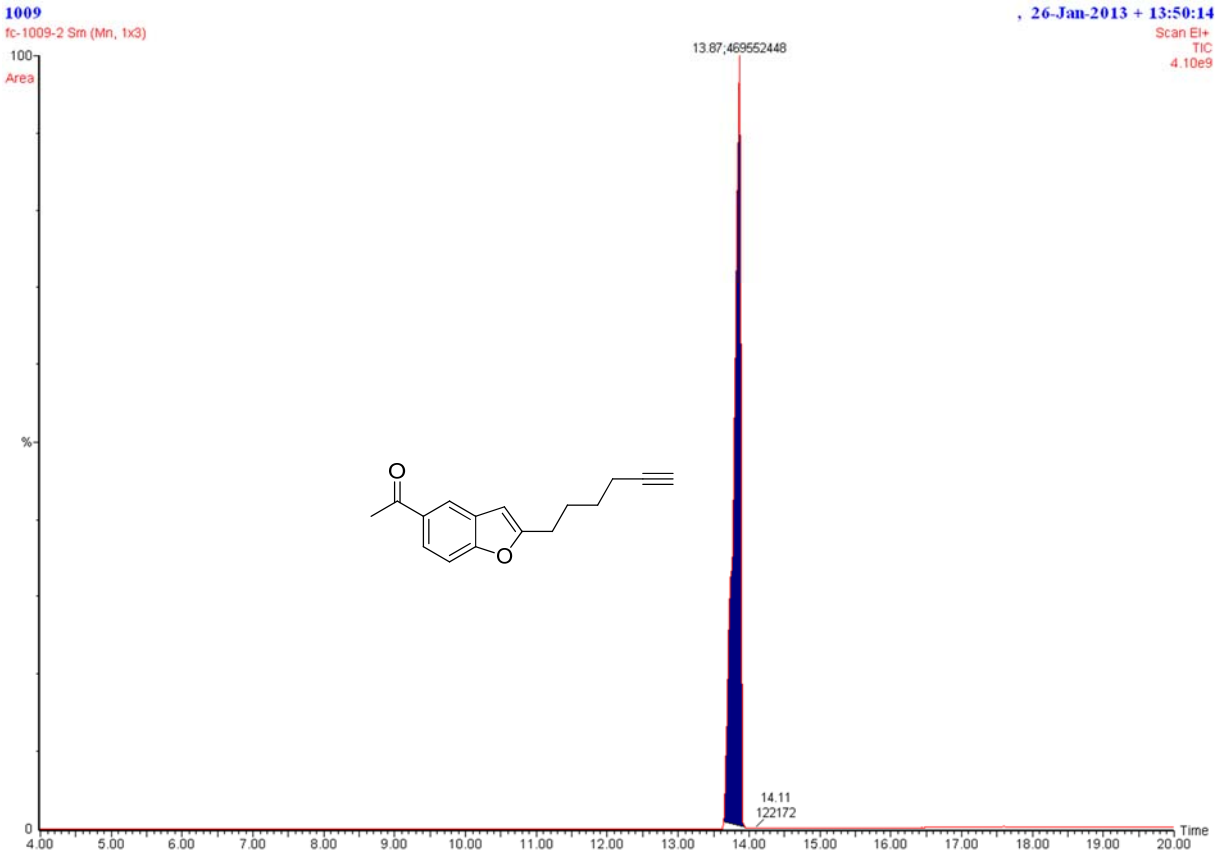
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **t39**

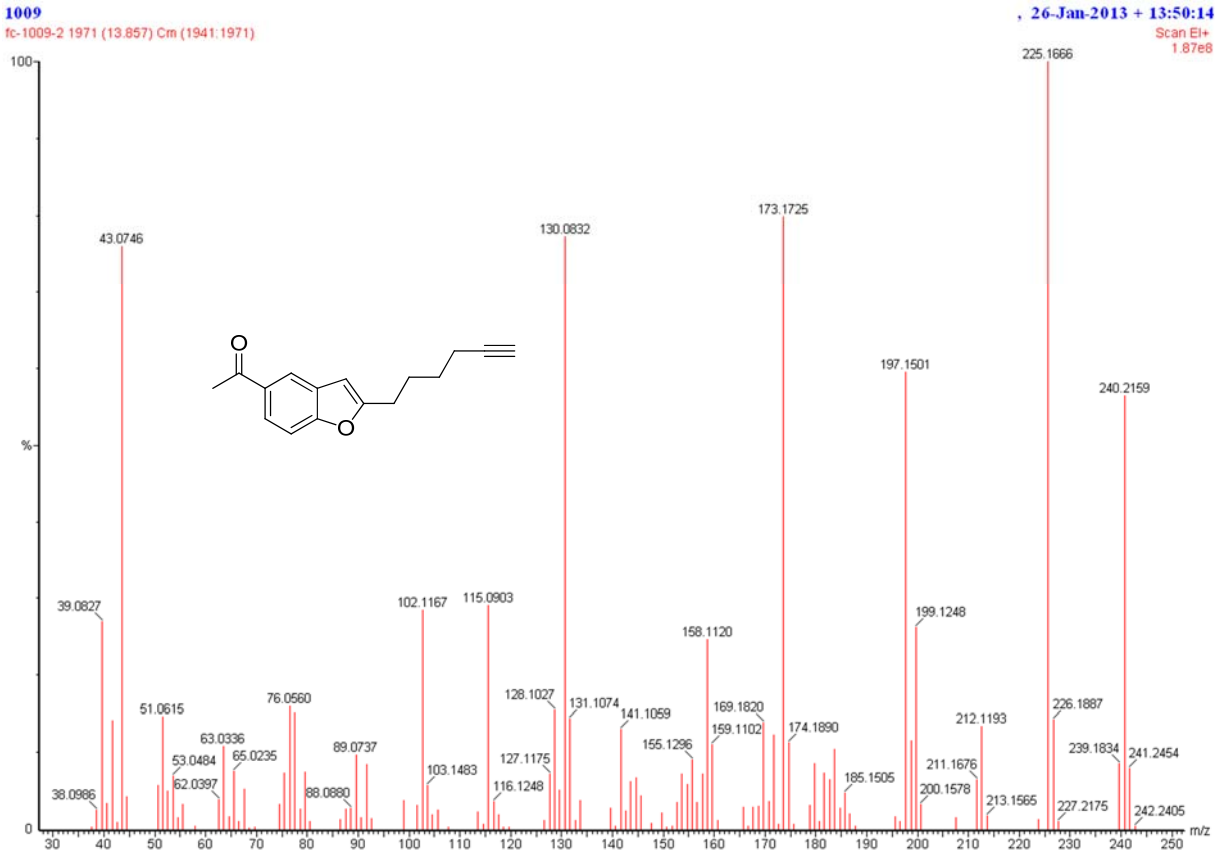

924
fc-924- Sm (Mn, 1x3)
, 06-Dec-2012 + 14:19:34
Scan EI+
TIC
2.42e9
11.07;89394192
100
Area
%
0
Time
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00

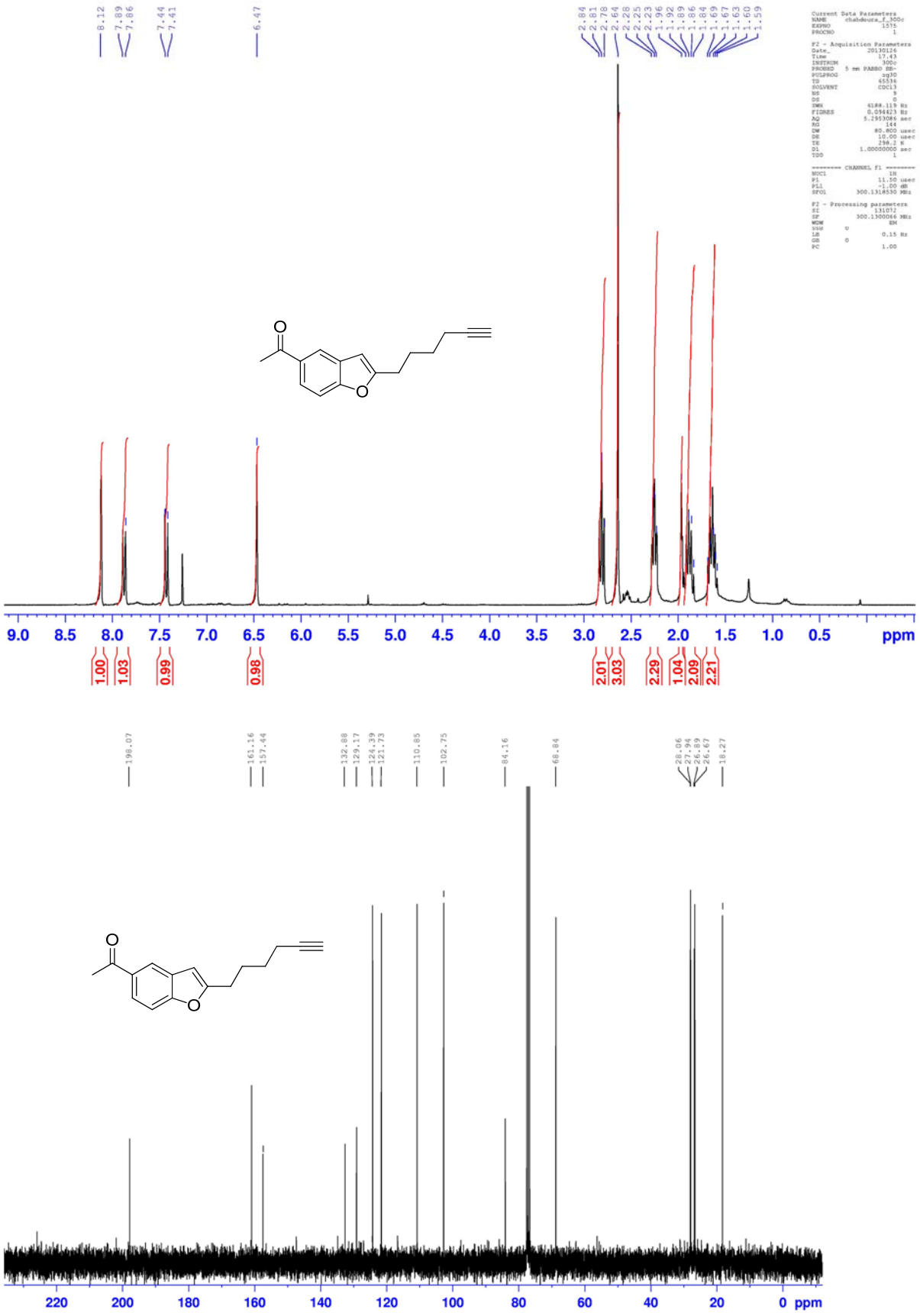

924
fc-924- 1414 (11.071) Cm (1412:1420)
, 06-Dec-2012 + 14:19:34
Scan EI+
4.09e8
100
%
0
m/z
131.0653
77.0500
198.0751
170.1216
157.0717
169.2037
132.0861
115.0948
128.0978
144.0637
183.1079
197.1284
39.0839
51.0172
103.1104
63.0221
41.0847
76.0596
78.0570
91.0571
118.0636
165.1151
181.1465
199.1388
38.1084
55.0198
101.1065
107.1076
133.0989
153.1045
184.1698
200.1553
39
49
59
69
79
89
99
109
119
129
139
149
159
169
179
189
199



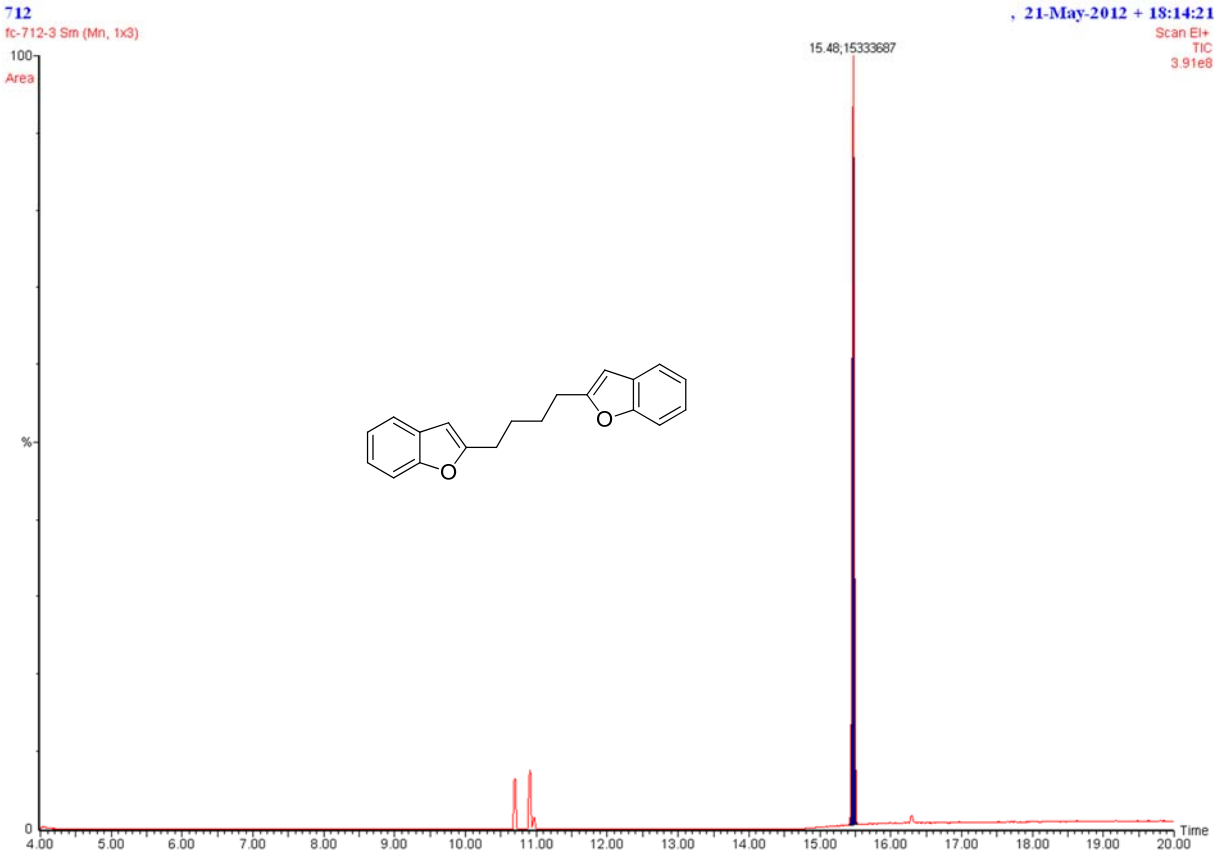
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **s40**

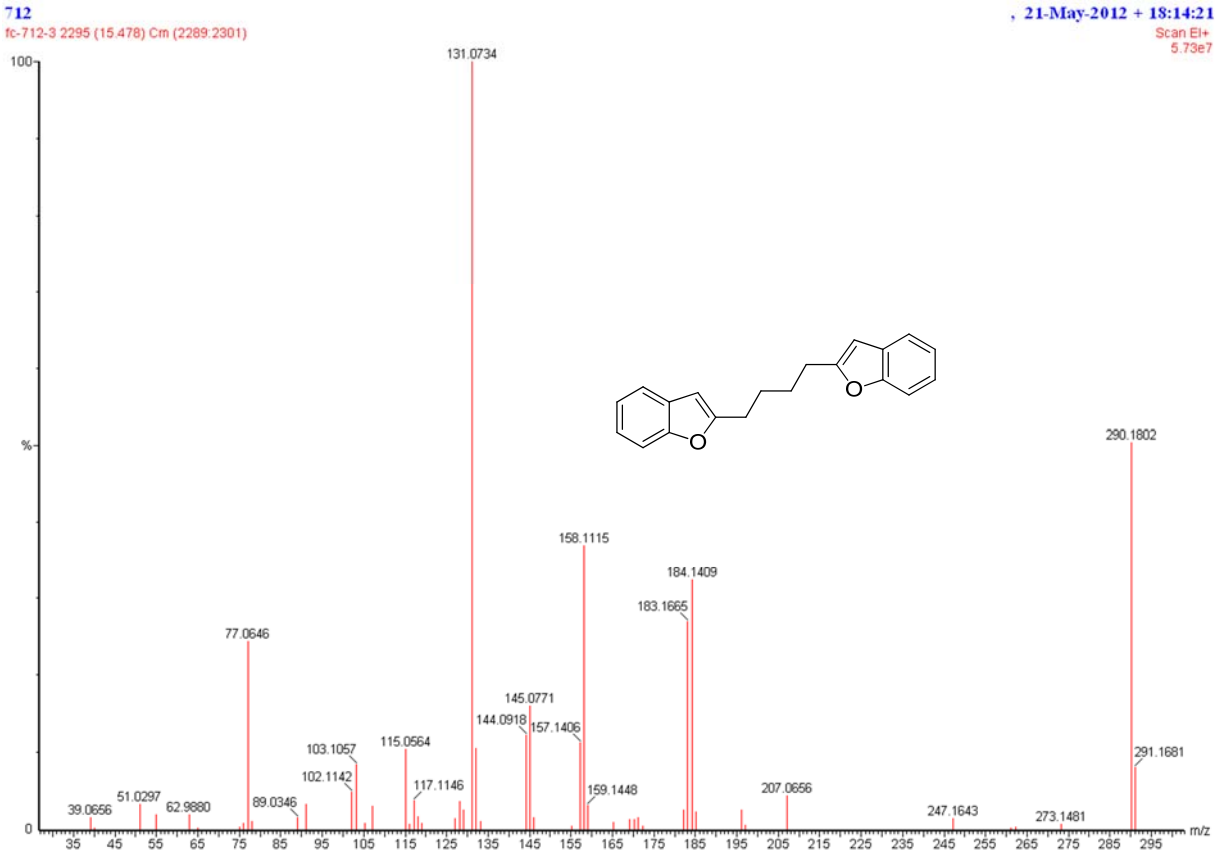

1009
fc-1009-2 Sm (Mn, 1x3)
, 26-Jan-2013 + 13:50:14
Scan EI+
TIC
4.10e9
100
Area
%
0
13.87;469552448
14.11
122172
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

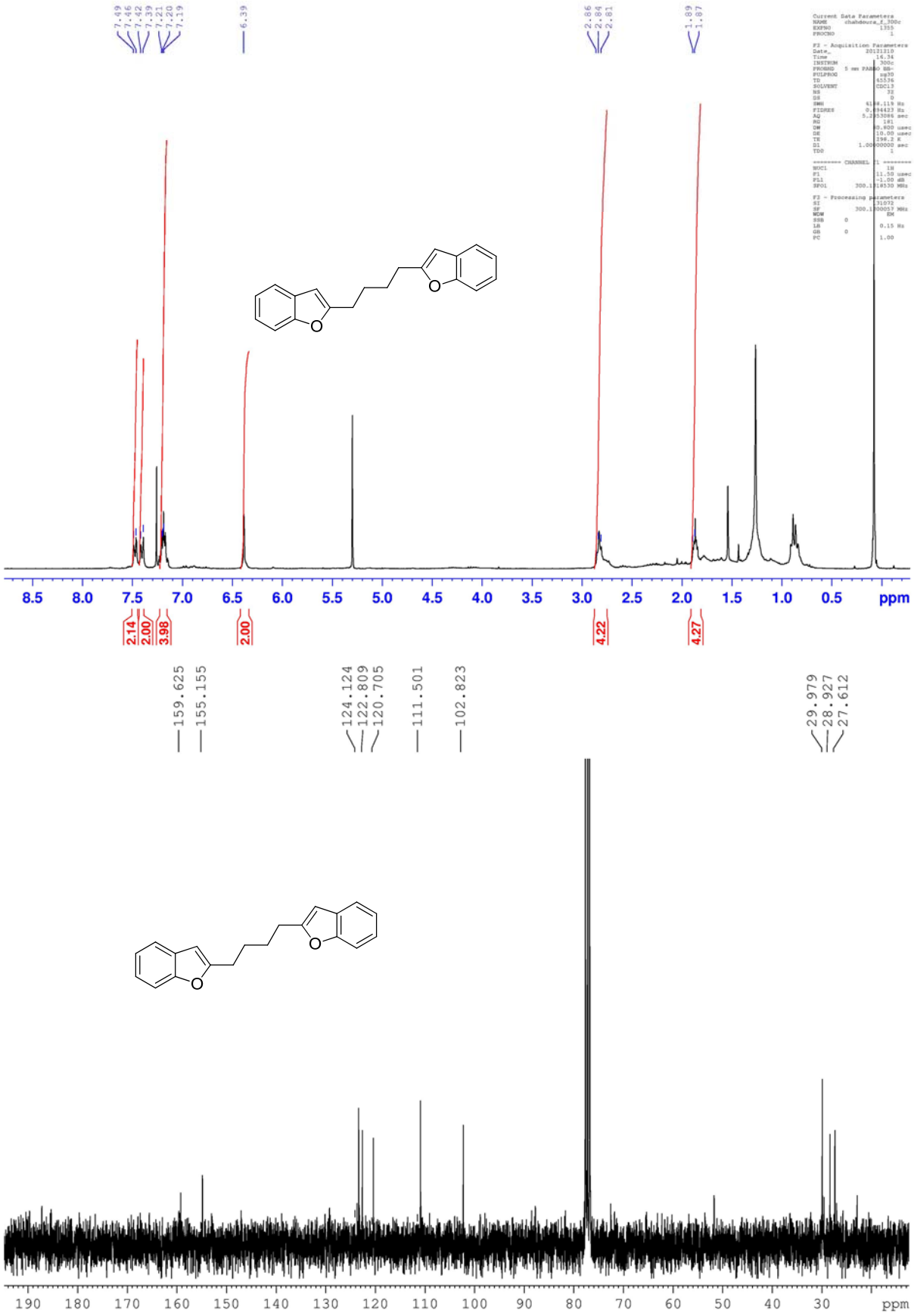

1009
fc-1009-2 1971 (13.857) Cm (1941:1971)
, 26-Jan-2013 + 13:50:14
Scan EI+
1.87e8
100
%
0
225.1666
173.1725
130.0832
43.0746
197.1501
240.2159
199.1248
158.1120
102.1167
115.0903
39.0827
128.1027
131.1074
141.1059
169.1820
159.1102
174.1890
212.1193
226.1887
76.0560
51.0615
63.0336
155.1296
89.0737
53.0484
65.0235
127.1175
103.1483
239.1834
241.2454
211.1676
185.1505
62.0397
116.1248
200.1578
213.1565
227.2175
38.0986
88.0880
242.2405
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
m/z



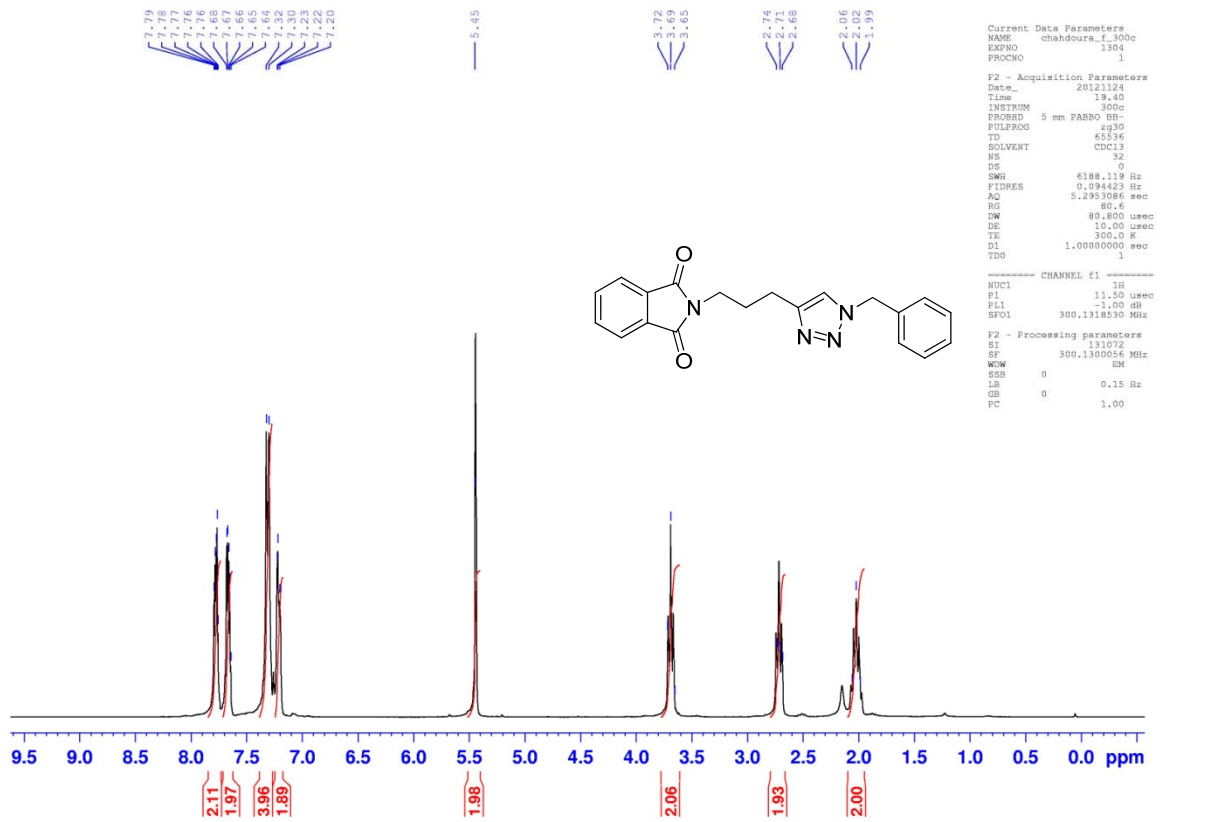
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **t40**

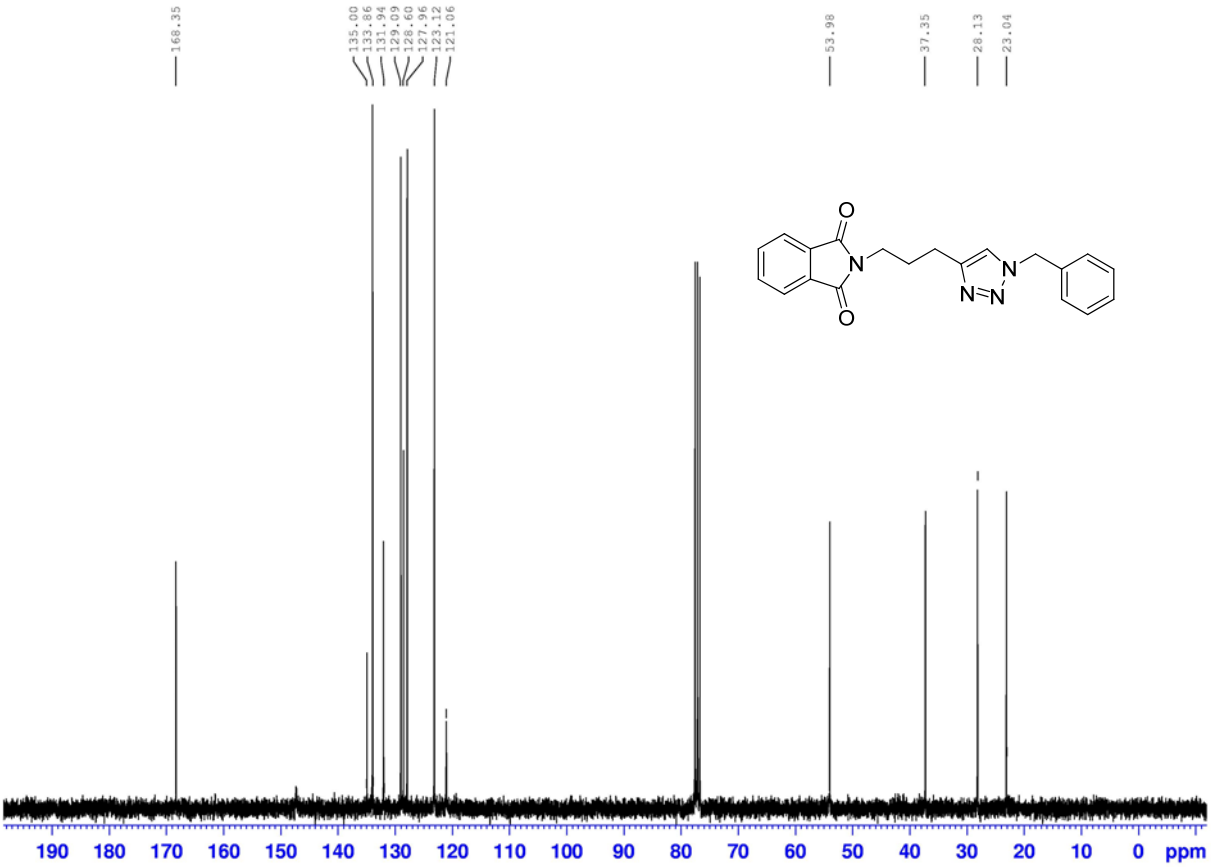

712
fc-712-3 Sm (Mn, 1x3)
, 21-May-2012 + 18:14:21
Scan EI+
TIC
3.91e8
Area
100
%
0
15.48;15333687
Time
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
O
O

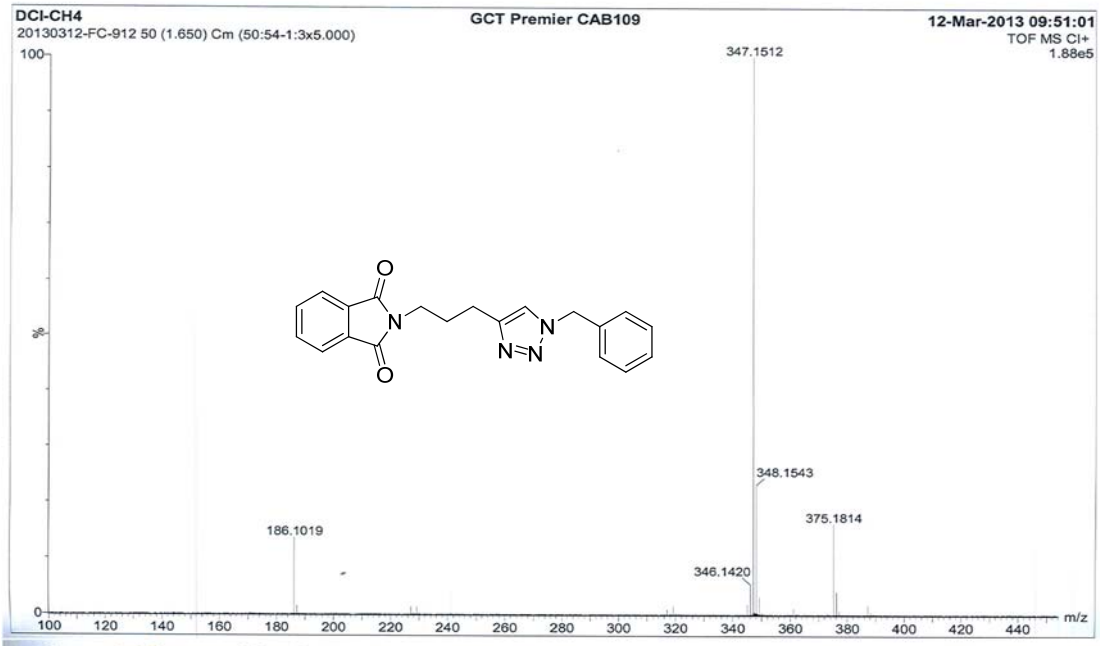

712
fc-712-3 2295 (15.478) Cm (2289:2301)
, 21-May-2012 + 18:14:21
Scan EI+
5.73e7
100
%
0
131.0734
290.1802
158.1115
184.1409
183.1665
77.0646
145.0771
144.0918
157.1406
115.0564
103.1057
102.1142
117.1146
159.1448
207.0656
291.1681
247.1643
273.1481
39.0656
51.0297
62.9880
89.0346
m/z
35
45
55
65
75
85
95
105
115
125
135
145
155
165
175
185
195
205
215
225
235
245
255
265
275
285
295
O
O



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **s40s**


7.79
7.78
7.77
7.76
7.76
7.68
7.67
7.66
7.65
7.64
7.32
7.30
7.23
7.22
7.20
5.45
3.72
3.69
3.65
2.74
2.71
2.68
2.06
2.02
1.99
Current Data Parameters
NAME chahdoura_f_300c
EXPNO 1304
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121124
Time 19.40
INSTRUM 300c
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2933086 sec
RG 80.6
DW 80.800 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -1.00 dB
SFO1 300.1318530 MHz
F2 - Processing parameters
SI 131072
SF 300.1300056 MHz
WDW EM
SSB 0
LB 0.15 Hz
GB 0
PC 1.00
O
N
O
N
N=N
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.11
1.97
3.96
1.89
1.98
2.06
1.93
2.00

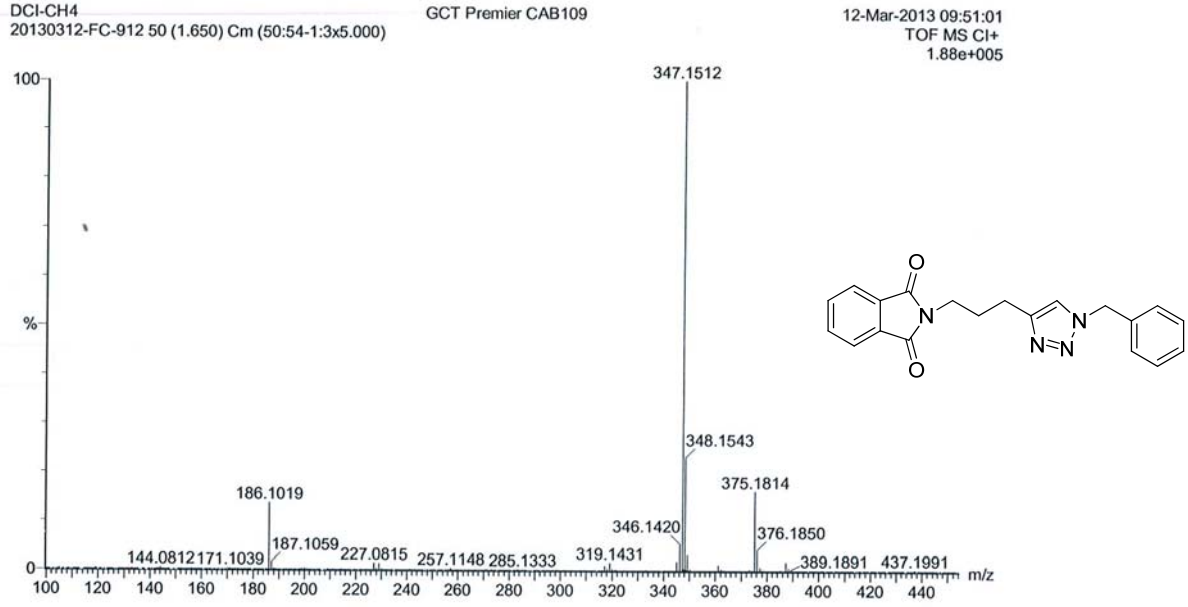

168.35
135.00
133.86
131.94
129.09
128.60
127.96
123.12
121.06
53.98
37.35
28.13
23.04
O
N
O
N
N=N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



Elemental Composition Report **Page 1**

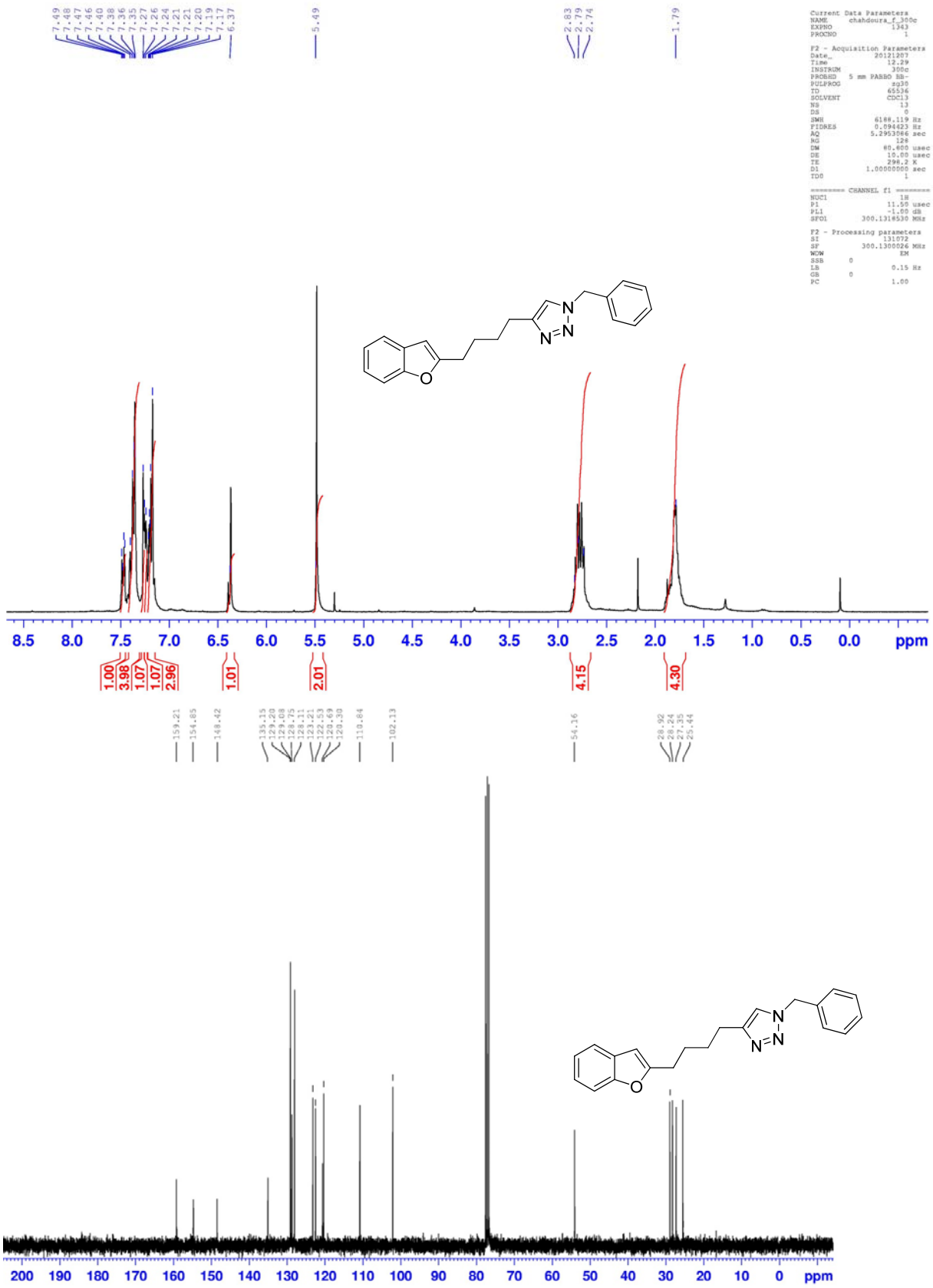
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
453 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-100 N: 0-10 O: 0-10

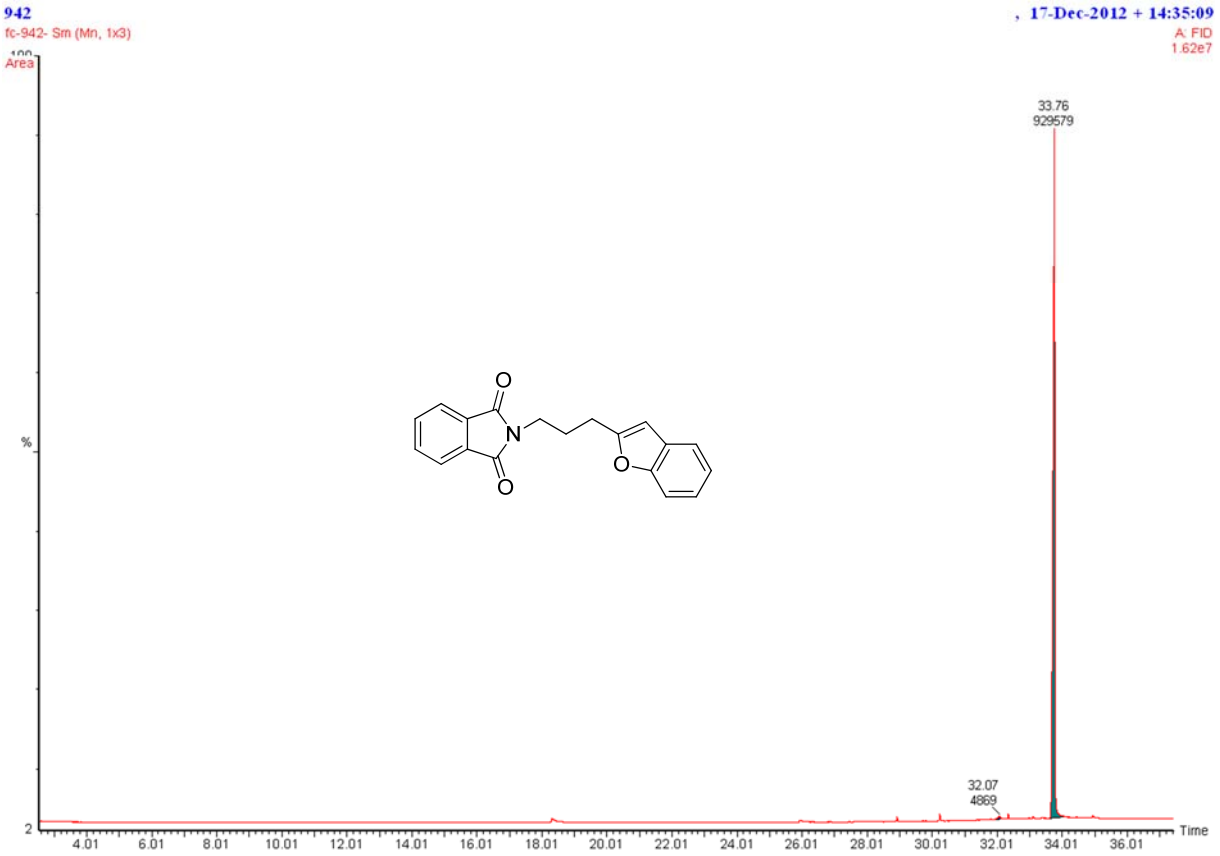


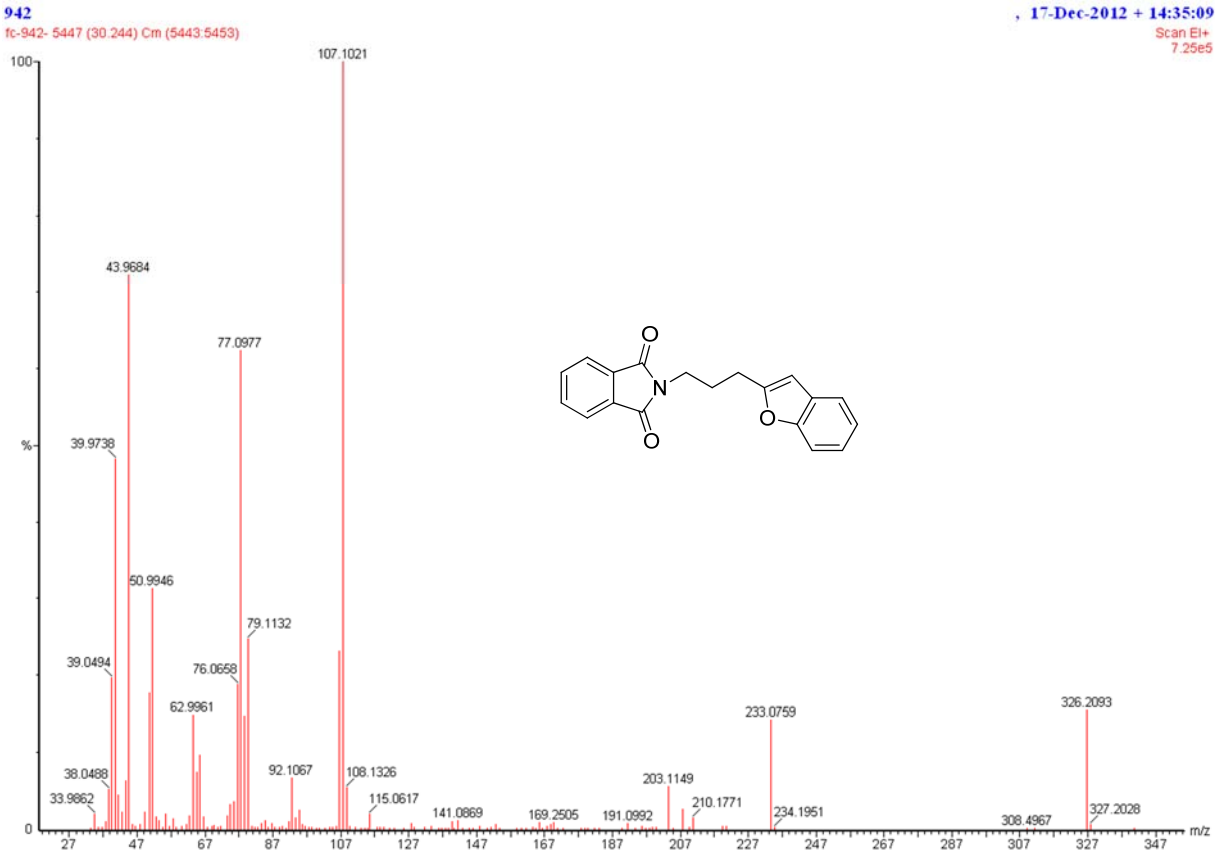
Minimum:				-1.5		
Maximum:		1.3	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
347.1512	347.1508	0.4	1.2	13.5	32.4	C20 H19 N4 O2
	347.1521	-0.9	-2.6	13.0	215.9	C22 H21 N O3
	347.1527	-1.5	-4.3	0.5	4995.1	C8 H23 N6 O9
	347.1513	-0.1	-0.3	1.0	6289.8	C6 H21 N9 O8

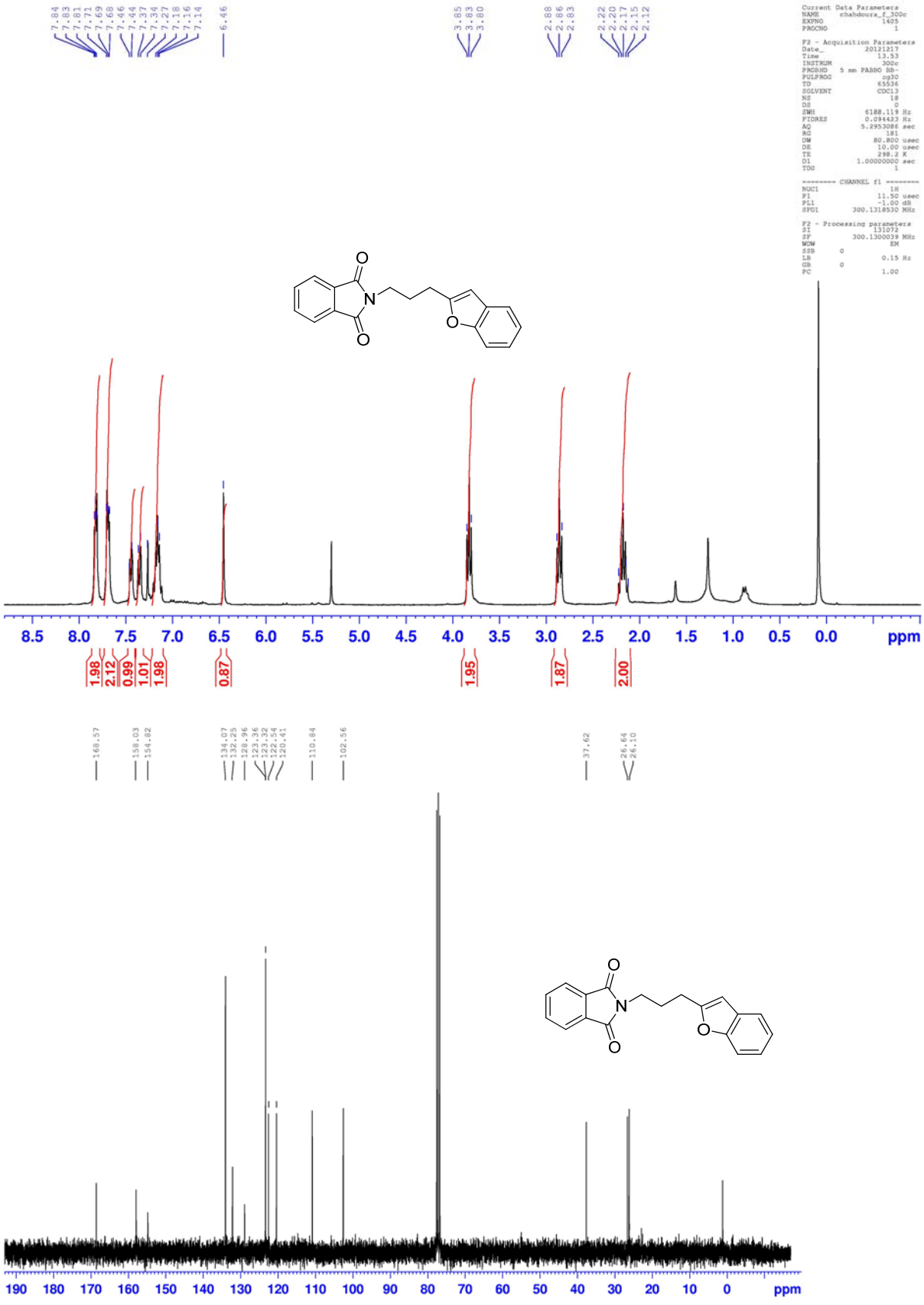
^{1}H NMR (300 MHz) (top) and ^{13}C {^{1}H} NMR (75 MHz) (middle) spectra in $CDCl_3$, low and high resolution DCI (CH_4) spectre and for **s17u**



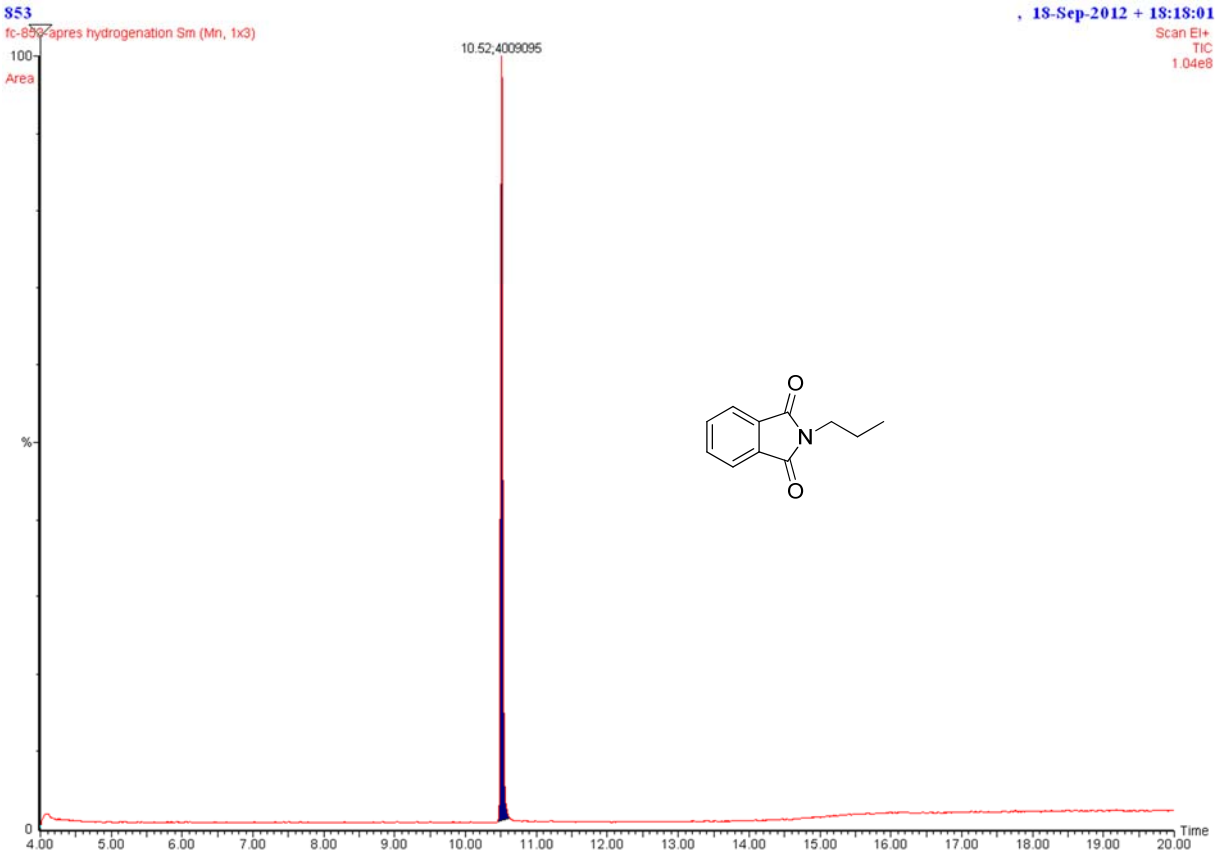
^{1}H NMR (300 MHz) (top) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **s40u**

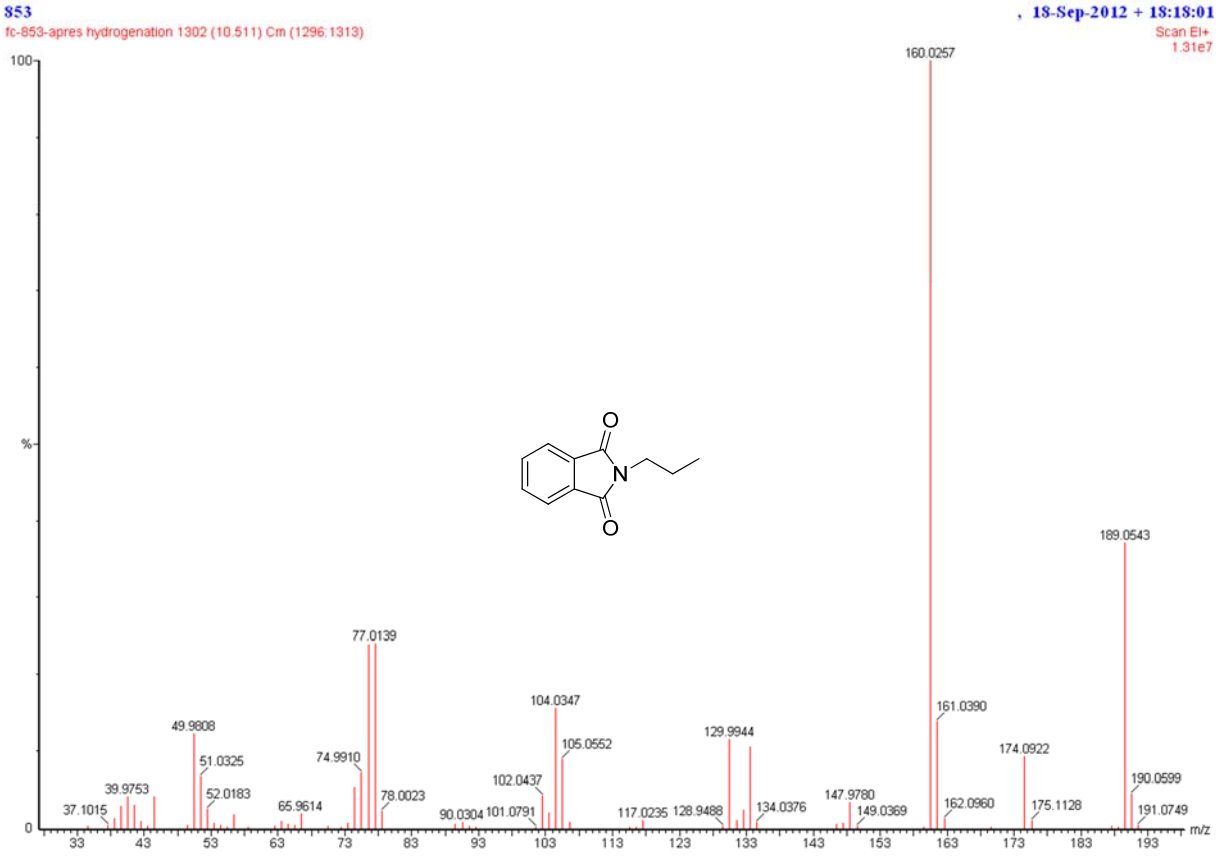

942
fc-942- Sm (Mn, 1x3)
, 17-Dec-2012 + 14:35:09
A: FID
1.62e7
Area
%
33.76
929579
32.07
4869
Time
4.01
6.01
8.01
10.01
12.01
14.01
16.01
18.01
20.01
22.01
24.01
26.01
28.01
30.01
32.01
34.01
36.01

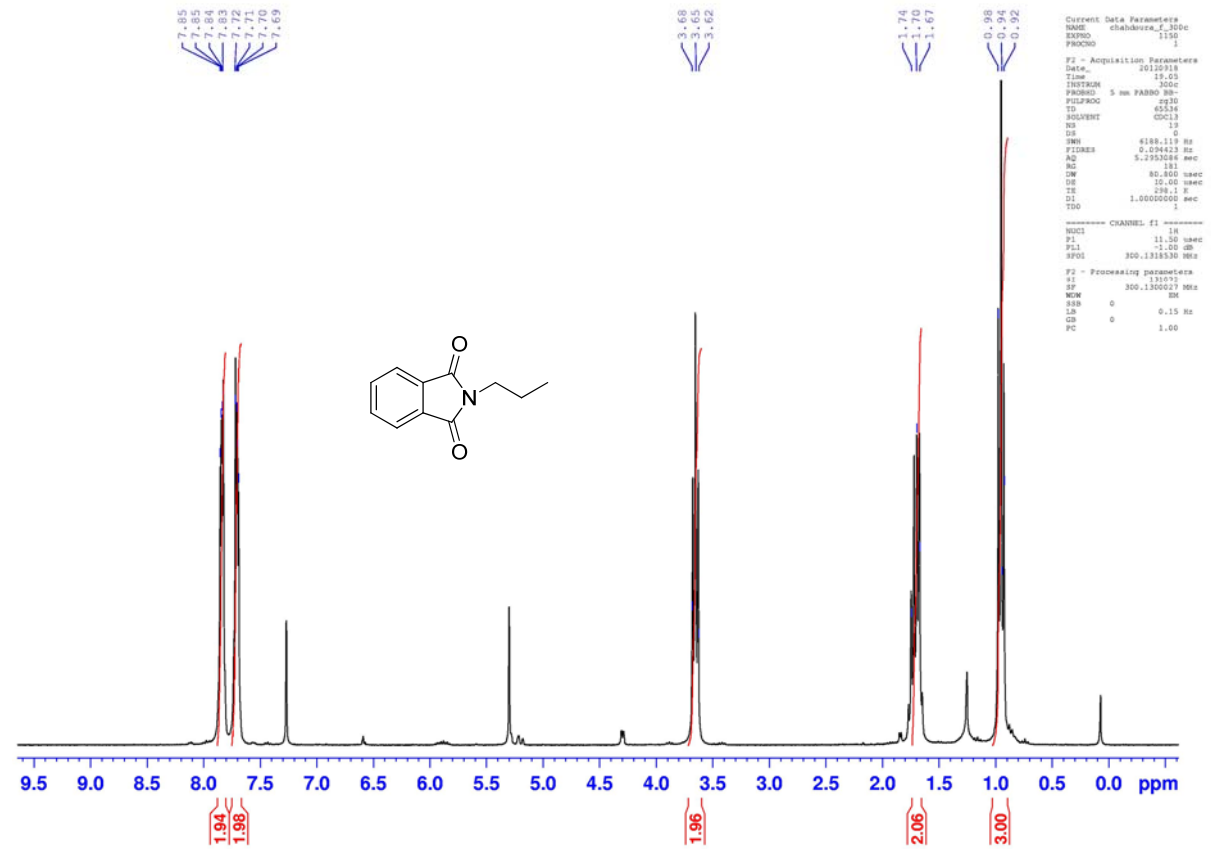

942
fc-942- 5447 (30.244) Cm (5443:5453)
, 17-Dec-2012 + 14:35:09
Scan EI+
7.25e5
100
%
0
107.1021
43.9684
77.0977
39.9738
50.9946
79.1132
39.0494
76.0658
62.9961
233.0759
326.2093
38.0488
92.1067
108.1326
203.1149
33.9862
115.0617
210.1771
141.0869
169.2505
191.0992
234.1951
308.4967
327.2028
m/z
27
47
67
87
107
127
147
167
187
207
227
247
267
287
307
327
347

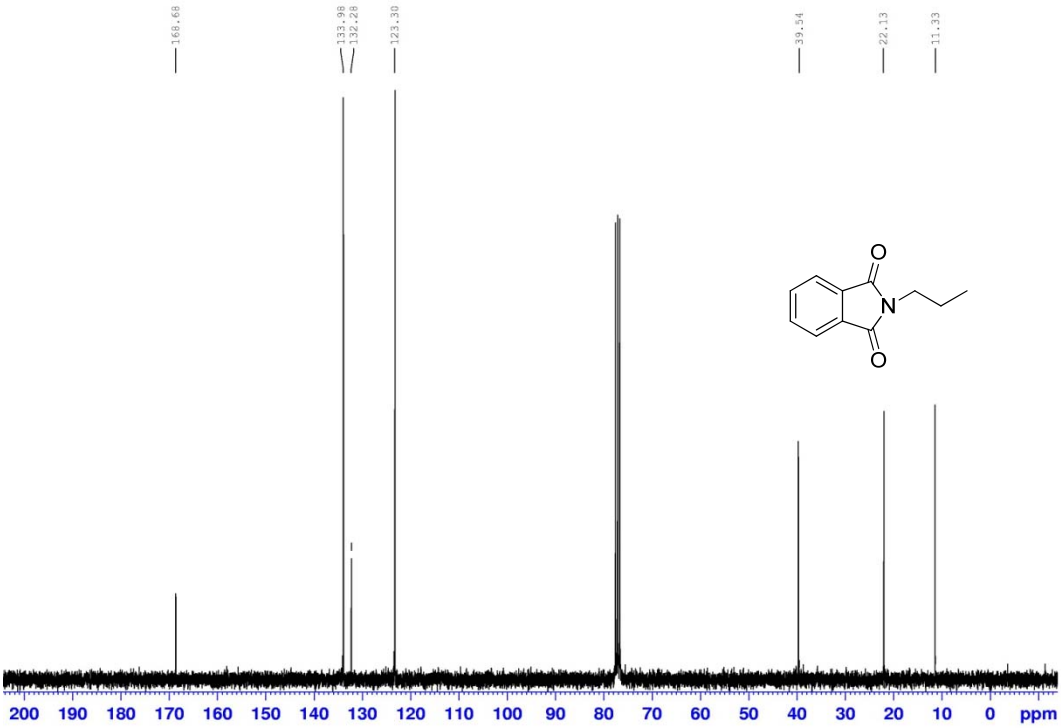


GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a17s**

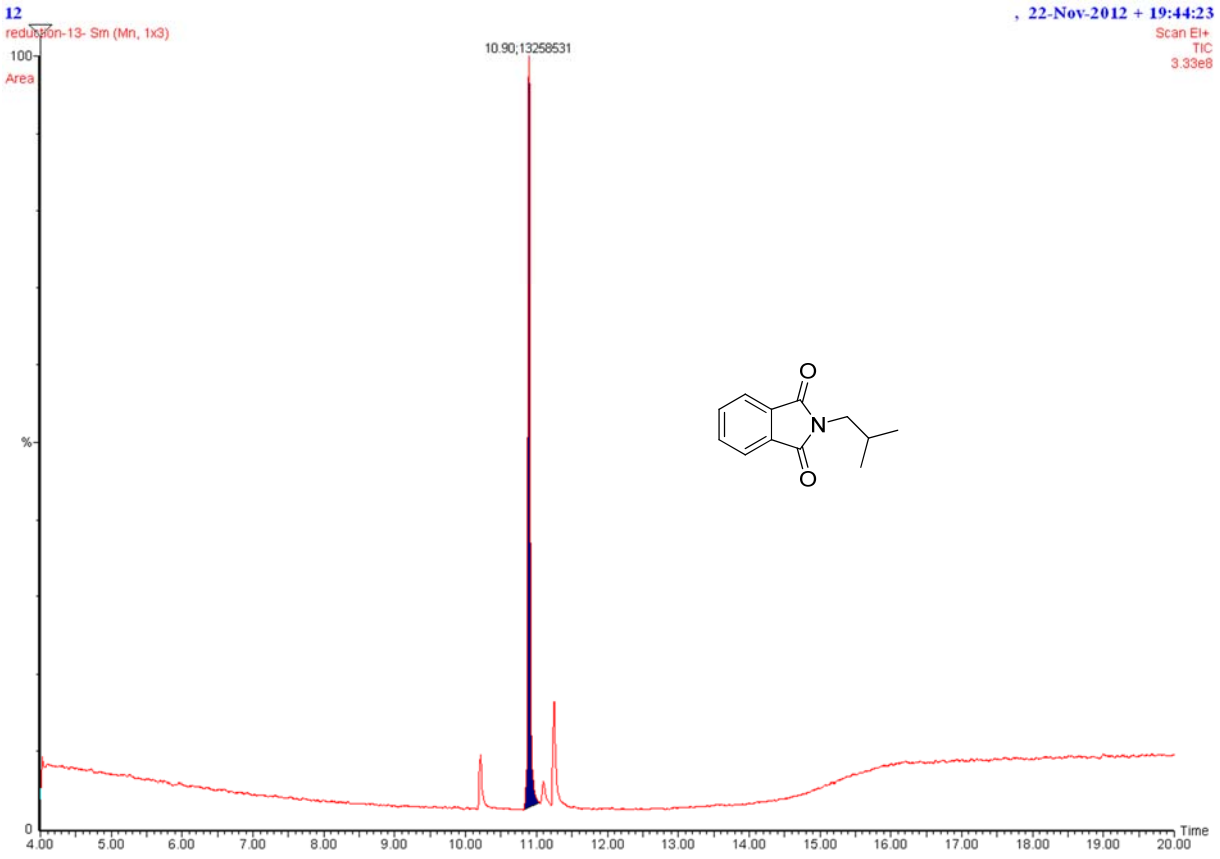

853
fc-853-apres hydrogenation Sm (Mn, 1x3)
, 18-Sep-2012 + 18:18:01
Scan EI+
TIC
1.04e8
100
Area
%
0
10.52,4009095
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O
N
O

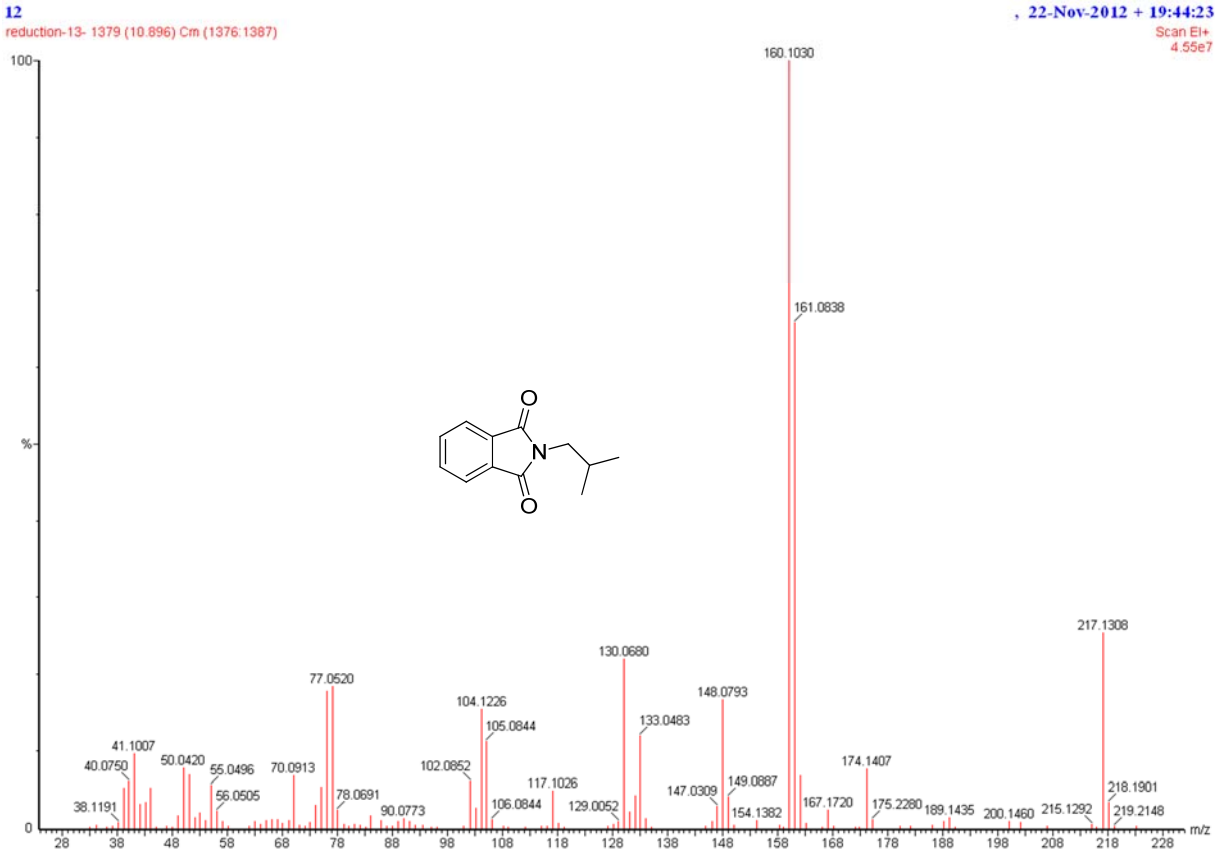

853
fc-853-apres hydrogenation 1302 (10.511) Cm (1296:1313)
, 18-Sep-2012 + 18:18:01
Scan EI+
1.31e7
100
%
0
160.0257
189.0543
77.0139
104.0347
161.0390
49.9808
129.9944
105.0552
174.0922
51.0325
74.9910
190.0599
39.9753
102.0437
52.0183
78.0023
147.9780
37.1015
65.9614
90.0304
101.0791
117.0235
128.9488
134.0376
149.0369
162.0960
175.1128
191.0749
33
43
53
63
73
83
93
103
113
123
133
143
153
163
173
183
193
m/z
O
N
O

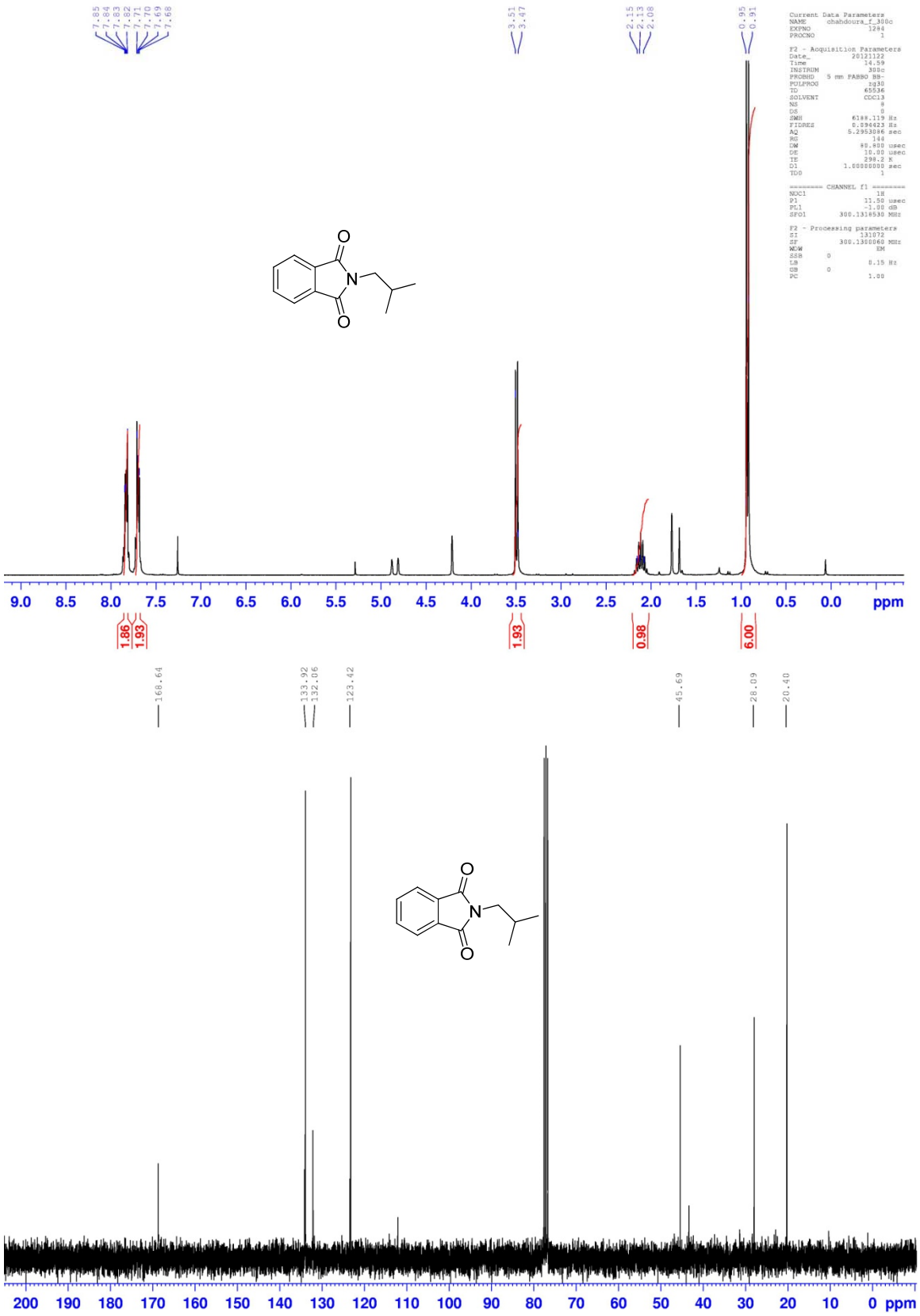




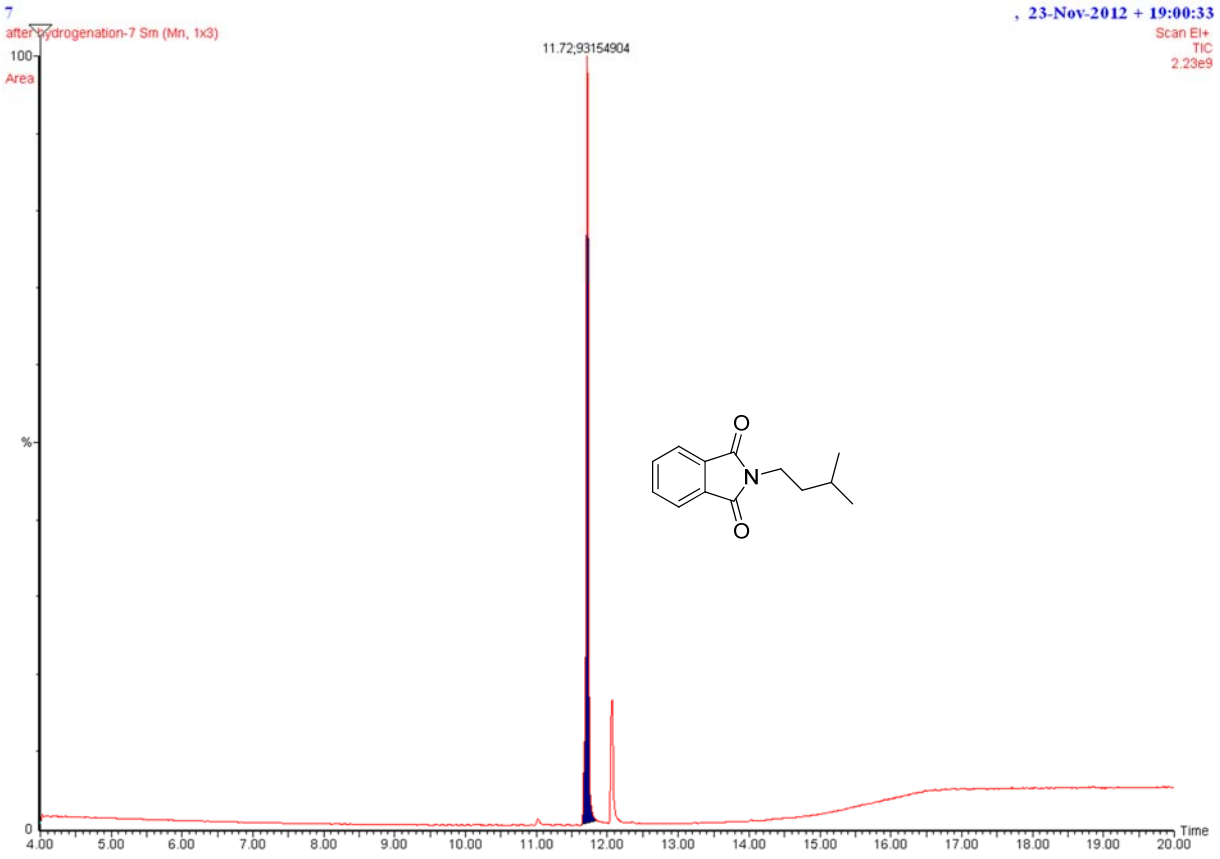
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a18H**

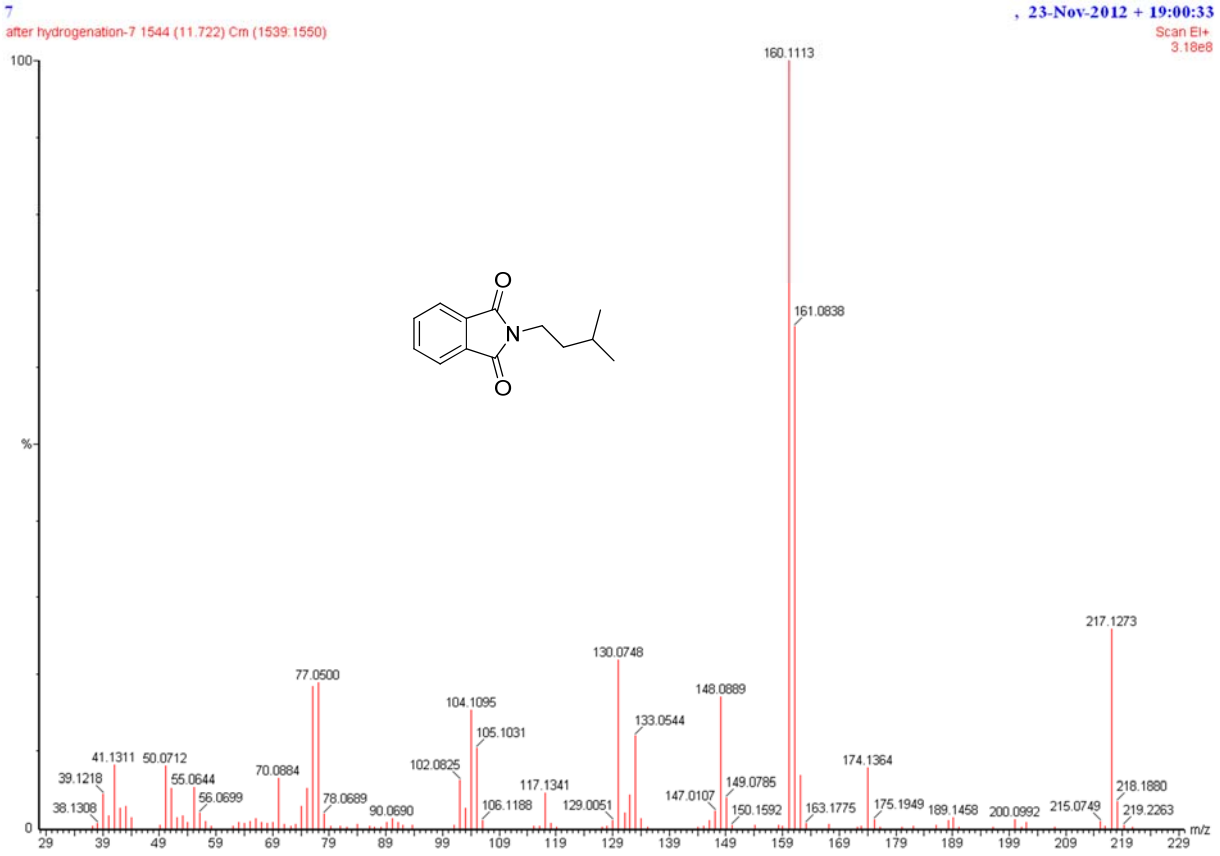

12
reduction-13- Sm (Mn, 1x3)
, 22-Nov-2012 + 19:44:23
Scan EI+
TIC
3.33e8
Area
100
%
0
10.90;13258531
O
N
O
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

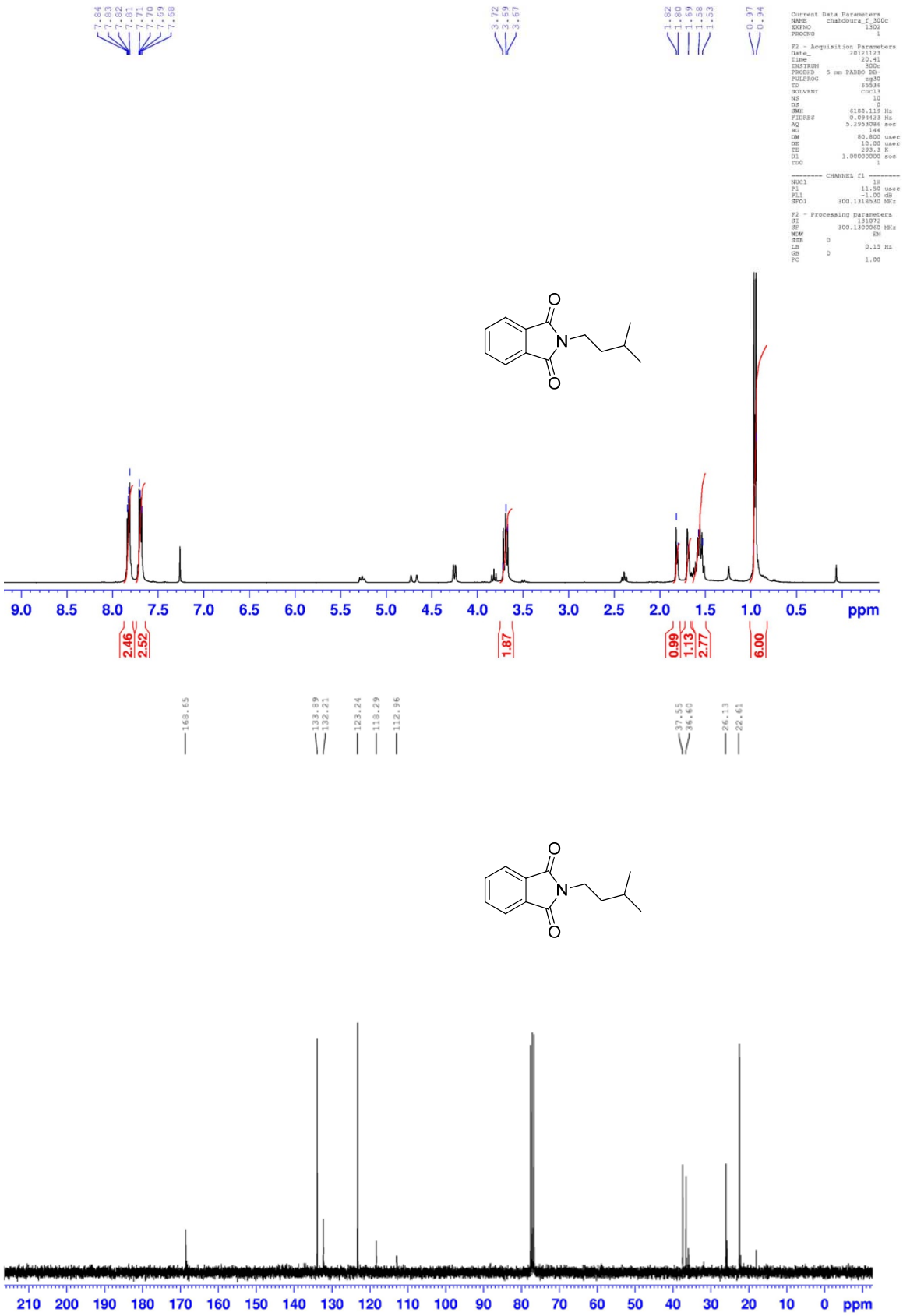

12
reduction-13- 1379 (10.896) Cm (1376:1387)
, 22-Nov-2012 + 19:44:23
Scan EI+
4.55e7
100
%
0
O
N
O
160.1030
161.0838
217.1308
130.0680
77.0520
104.1226
105.0844
148.0793
133.0483
41.1007
40.0750
50.0420
55.0496
70.0913
102.0852
174.1407
117.1026
147.0309
149.0887
218.1901
38.1191
56.0505
78.0691
90.0773
106.0844
129.0052
154.1382
167.1720
175.2280
189.1435
200.1460
215.1292
219.2148
28
38
48
58
68
78
88
98
108
118
128
138
148
158
168
178
188
198
208
218
228
m/z



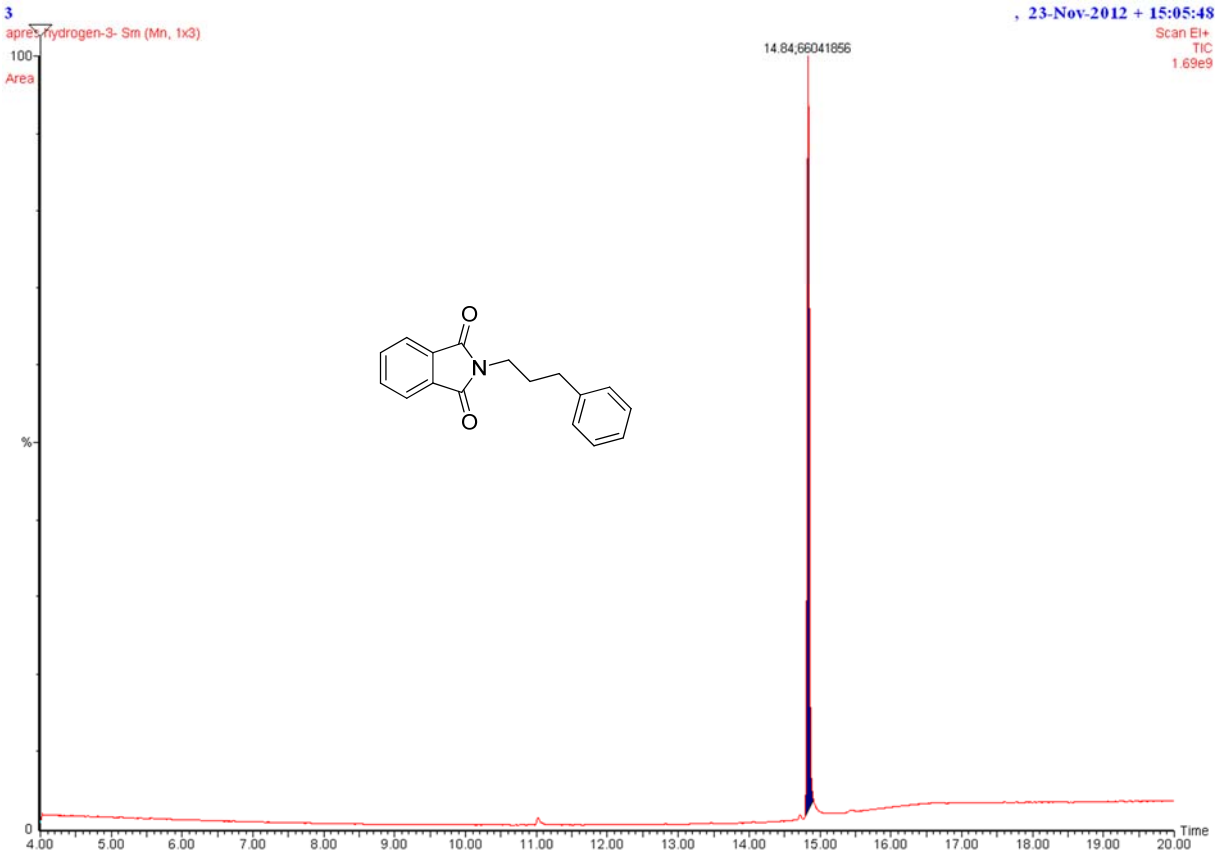
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a19H**

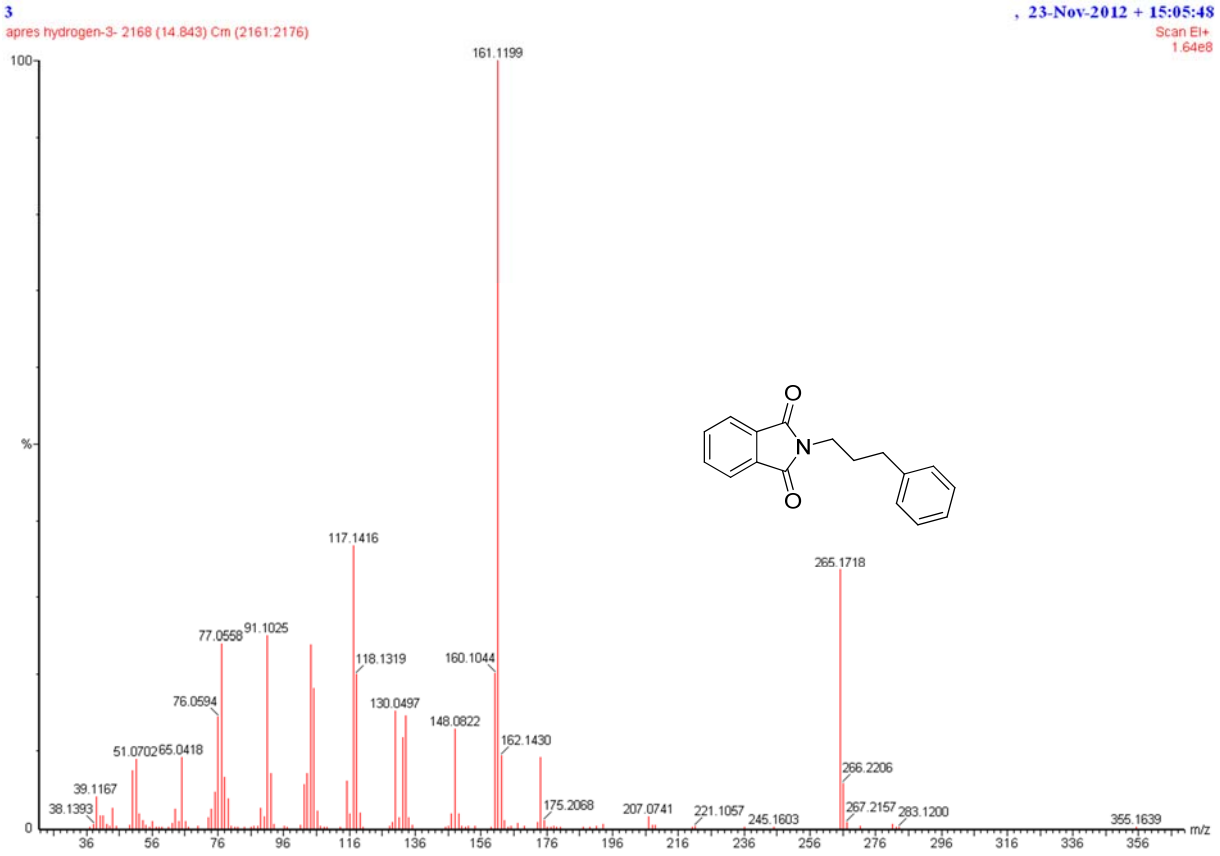

7
after hydrogenation-7 Sm (Mn, 1x3)
, 23-Nov-2012 + 19:00:33
Scan EI+
TIC
2.23e9
100
Area
%
0
11.72;93154904
O
N
O
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

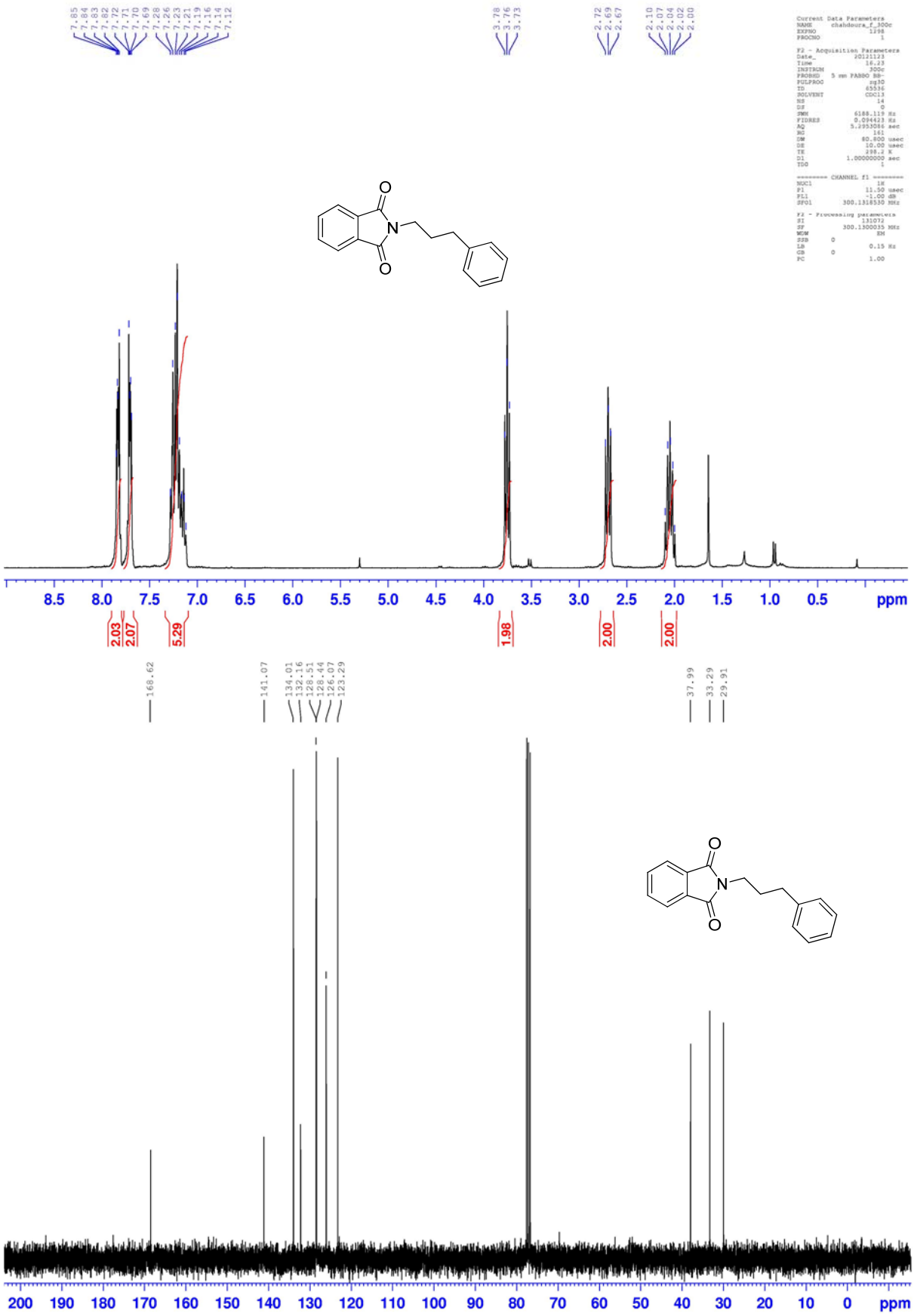

7
after hydrogenation-7 1544 (11.722) Cm (1539:1550)
, 23-Nov-2012 + 19:00:33
Scan EI+
3.18e8
100
%
0
160.1113
161.0838
217.1273
130.0748
148.0889
77.0500
104.1095
105.1031
133.0544
41.1311
50.0712
39.1218
55.0644
70.0884
102.0825
174.1364
56.0699
78.0689
117.1341
149.0785
218.1880
38.1308
90.0690
106.1188
129.0051
147.0107
150.1592
163.1775
175.1949
189.1458
200.0992
215.0749
219.2263
O
N
O
29
39
49
59
69
79
89
99
109
119
129
139
149
159
169
179
189
199
209
219
229
m/z



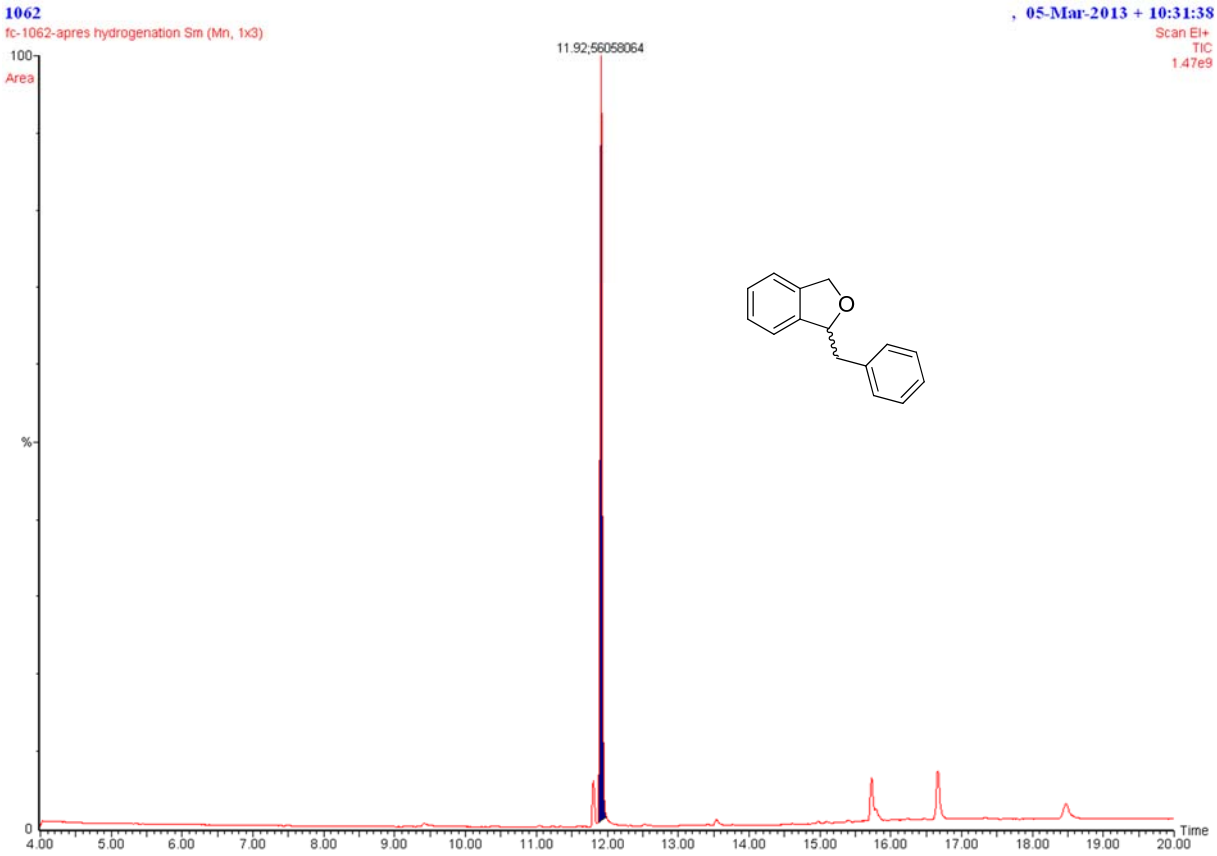
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a20H**

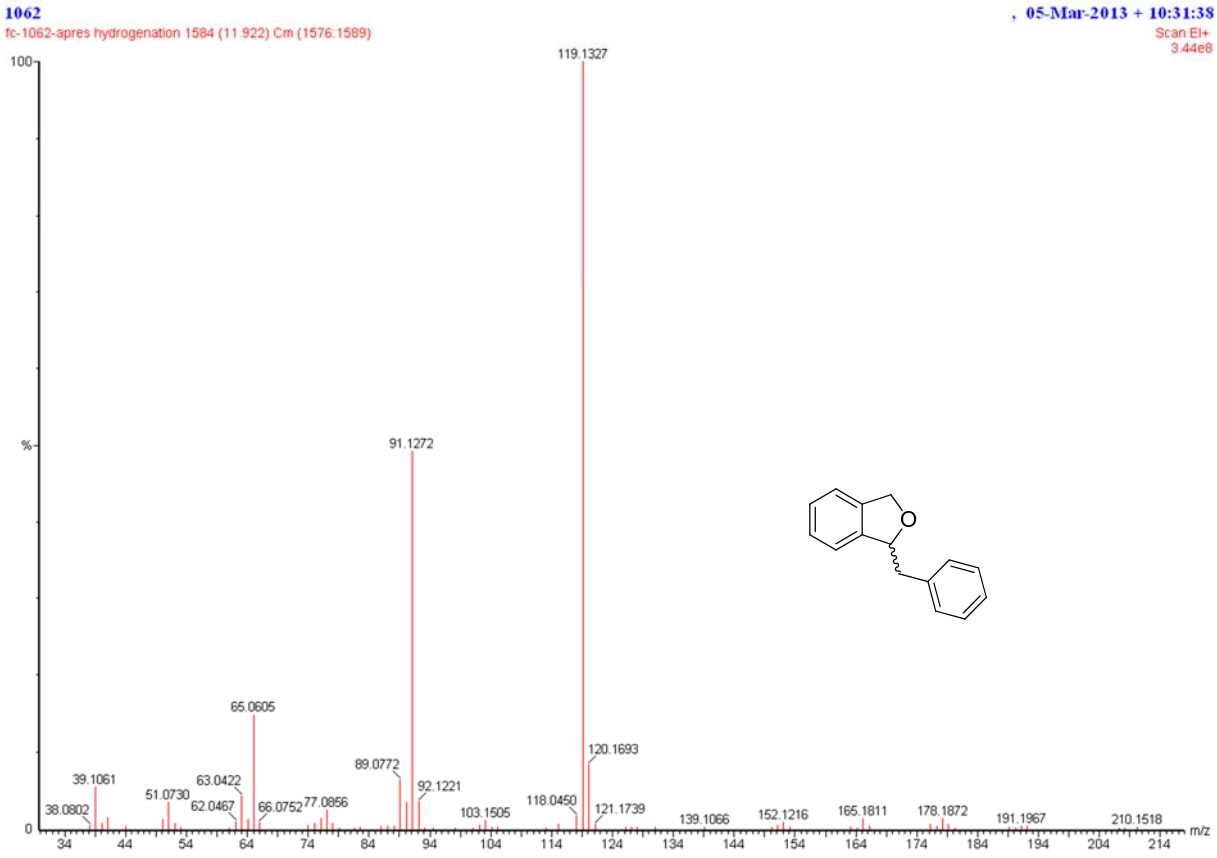

3
, 23-Nov-2012 + 15:05:48
apres hydrogen-3- Sm (Mn, 1x3)
Scan EI+
TIC
1.69e9
100
Area
14.84;66041856
%
0
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

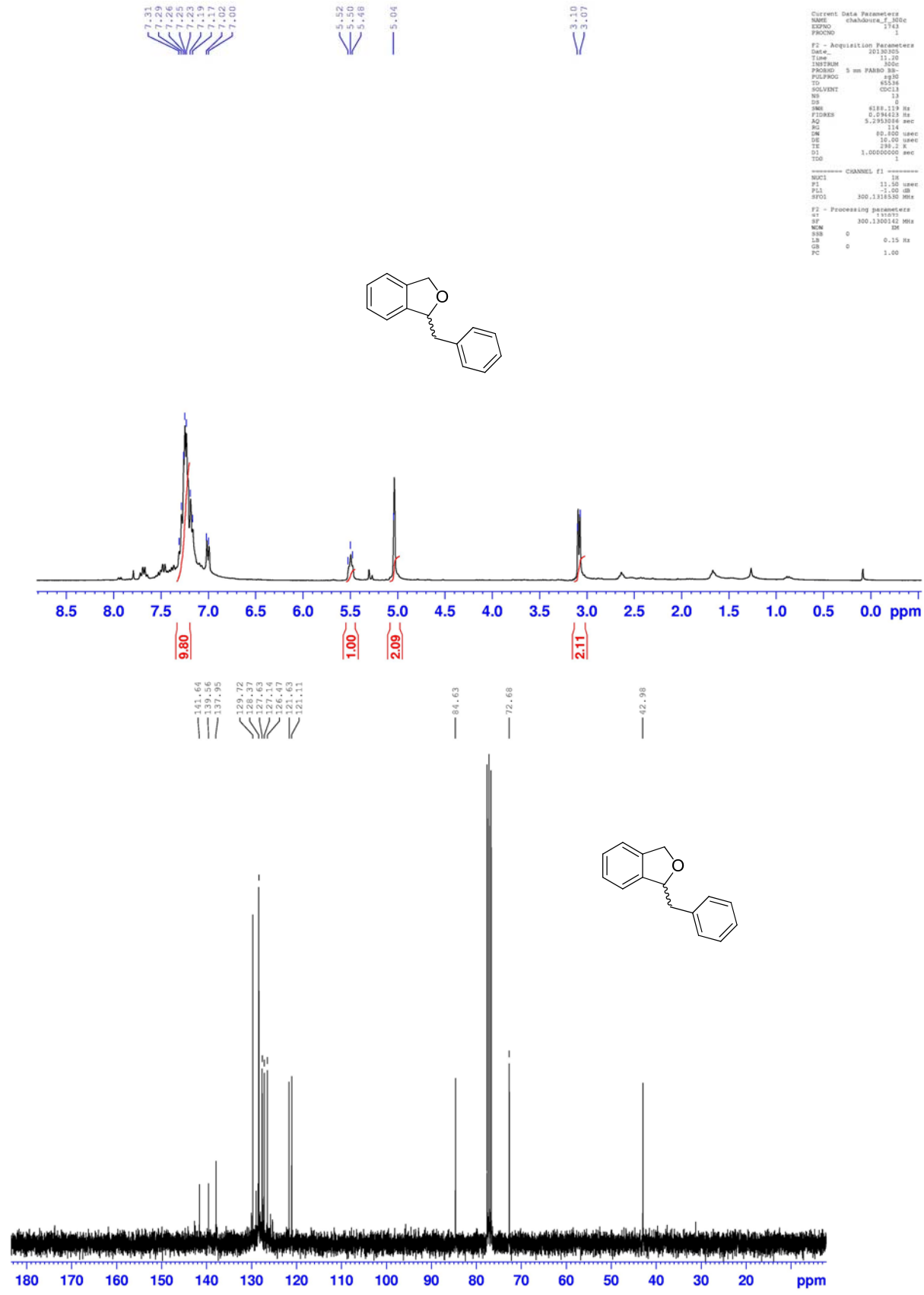

3
, 23-Nov-2012 + 15:05:48
apres hydrogen-3- 2168 (14.843) Cm (2161:2176)
Scan EI+
1.64e8
100
%
0
161.1199
117.1416
265.1718
77.0558
91.1025
118.1319
160.1044
76.0594
130.0497
148.0822
162.1430
51.0702
65.0418
39.1167
38.1393
266.2206
267.2157
283.1200
175.2068
207.0741
221.1057
245.1603
355.1639
36
56
76
96
116
136
156
176
196
216
236
256
276
296
316
336
356
m/z



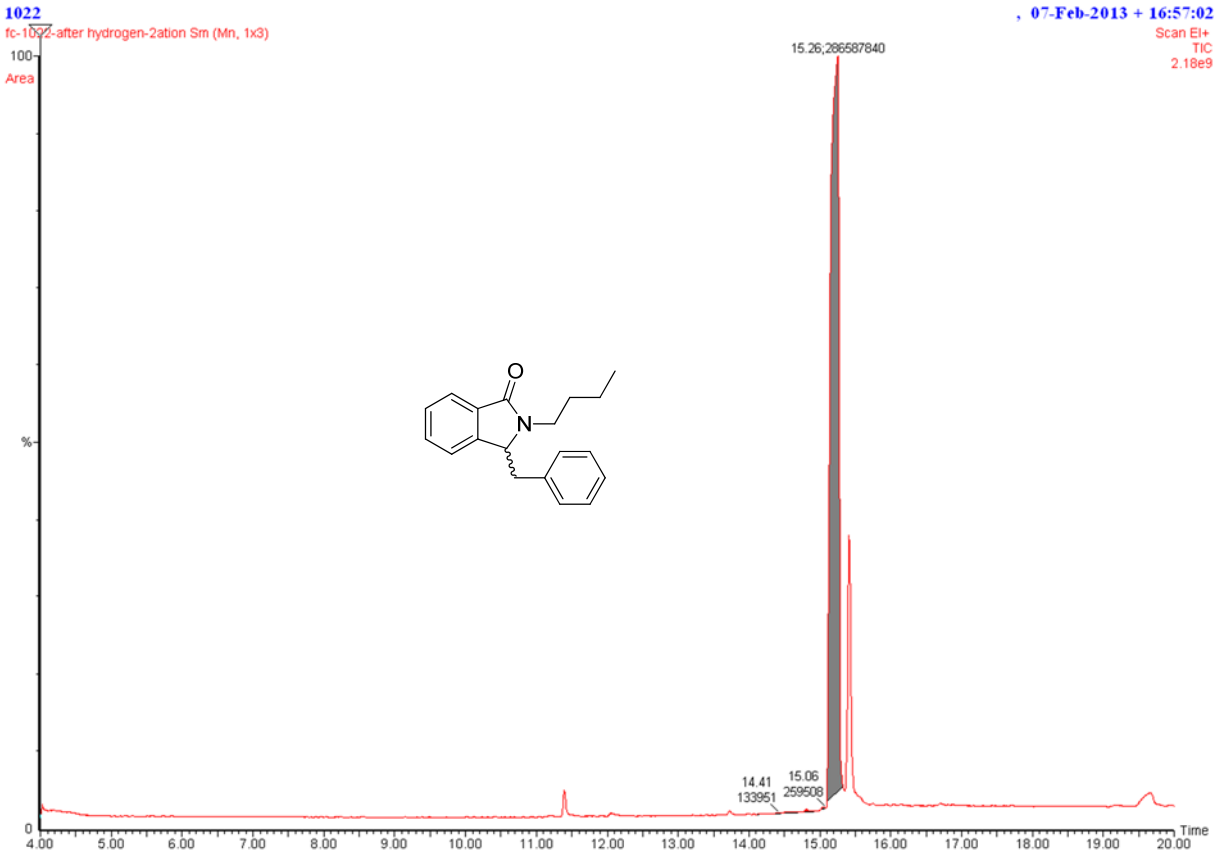
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **a21H**

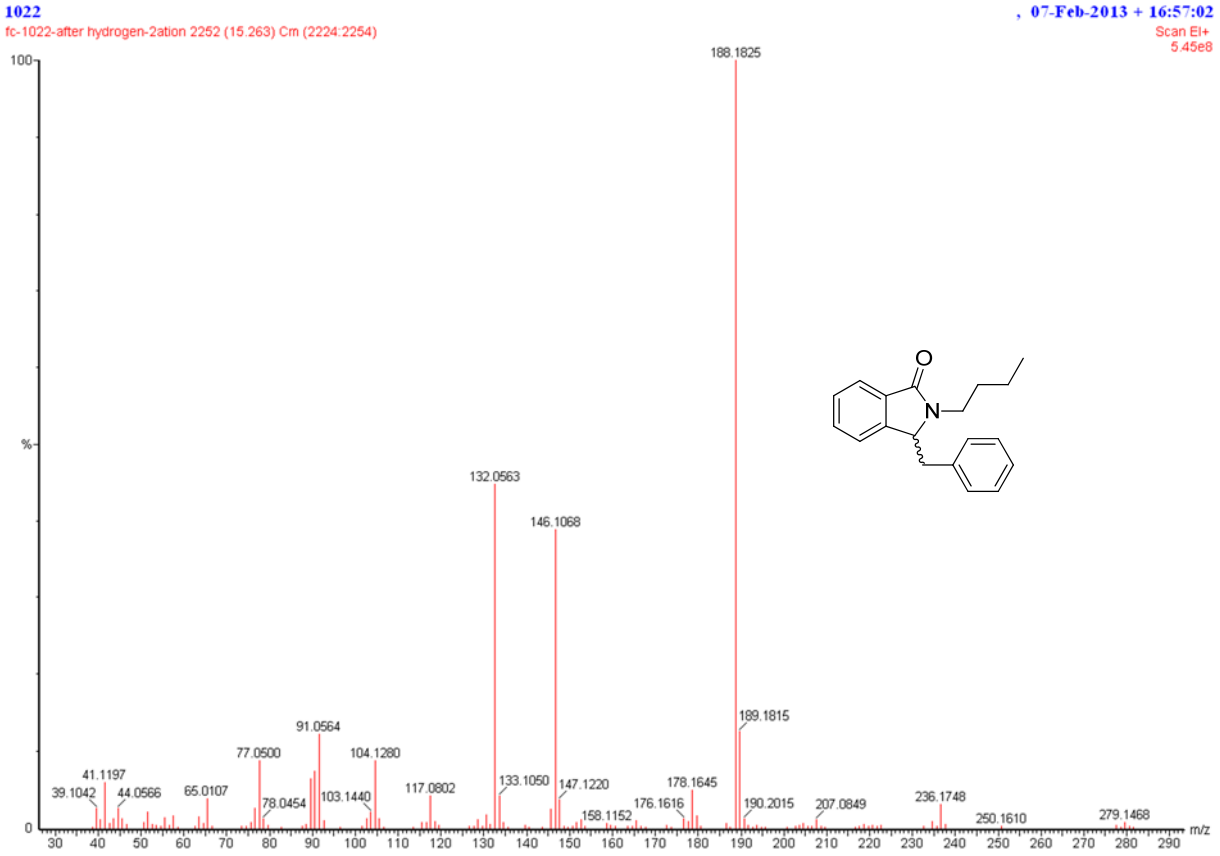

1062
, 05-Mar-2013 + 10:31:38
fc-1062-apres hydrogenation Sm (Mn, 1x3)
Scan EI+
TIC
1.47e9
11.92;56058064
100
Area
%
0
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time
O

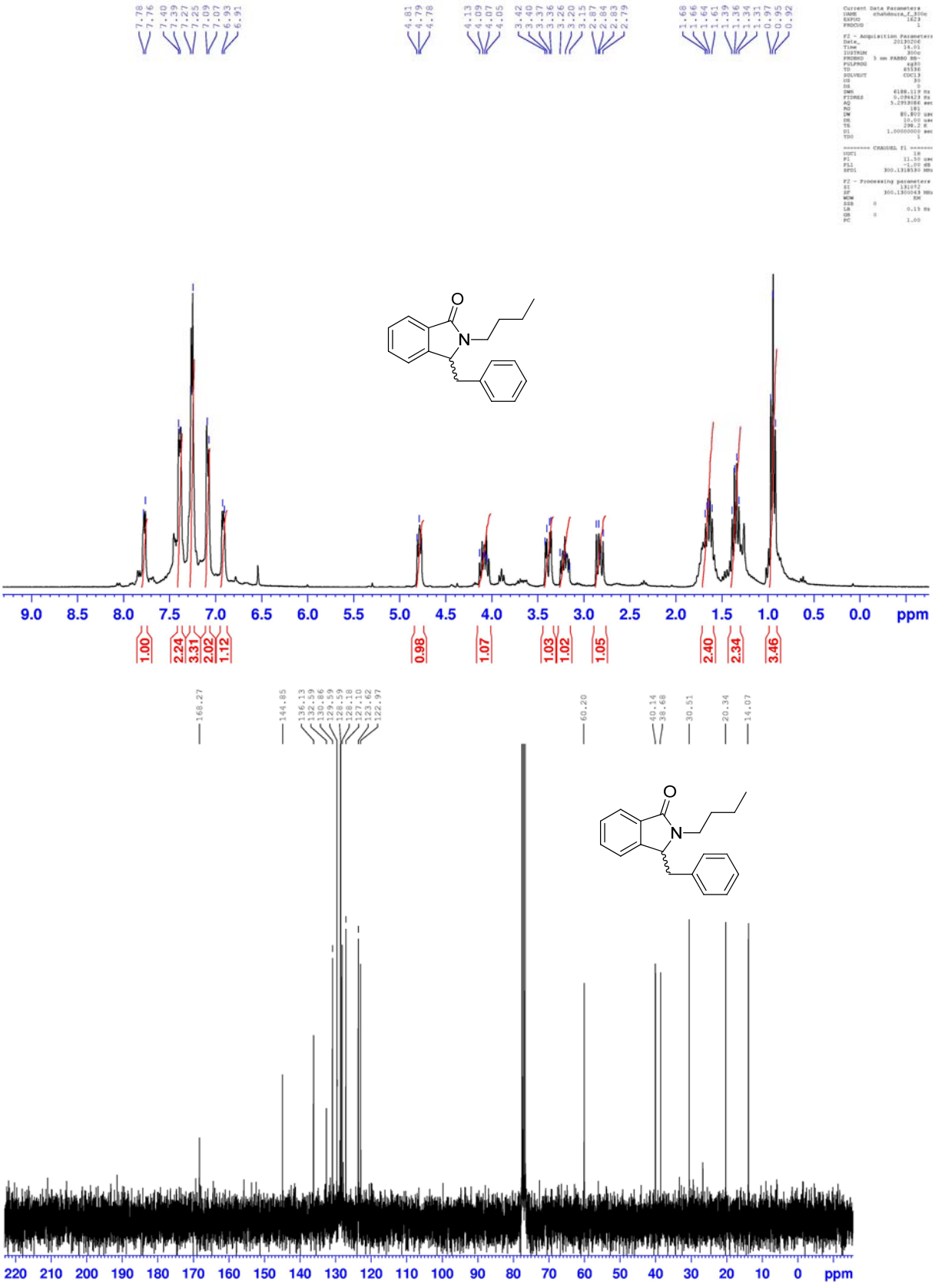

1062
, 05-Mar-2013 + 10:31:38
fc-1062-apres hydrogenation 1584 (11.922) Cm (1576:1589)
Scan EI+
3.44e8
119.1327
100
%
0
91.1272
65.0605
89.0772
120.1693
39.1061
63.0422
92.1221
51.0730
62.0467
66.0752
77.0856
103.1505
118.0450
121.1739
38.0802
139.1066
152.1216
165.1811
178.1872
191.1967
210.1518
34
44
54
64
74
84
94
104
114
124
134
144
154
164
174
184
194
204
214
m/z
O



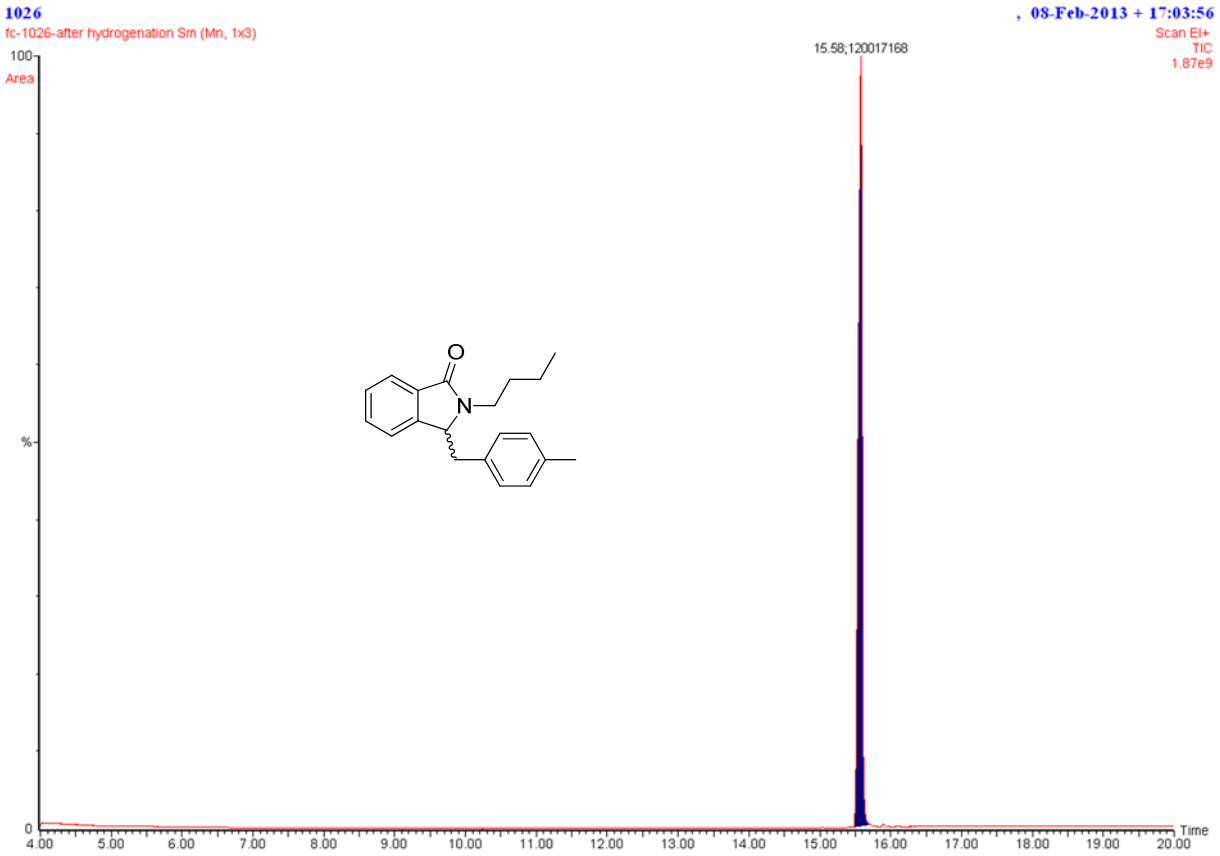
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **o33H**

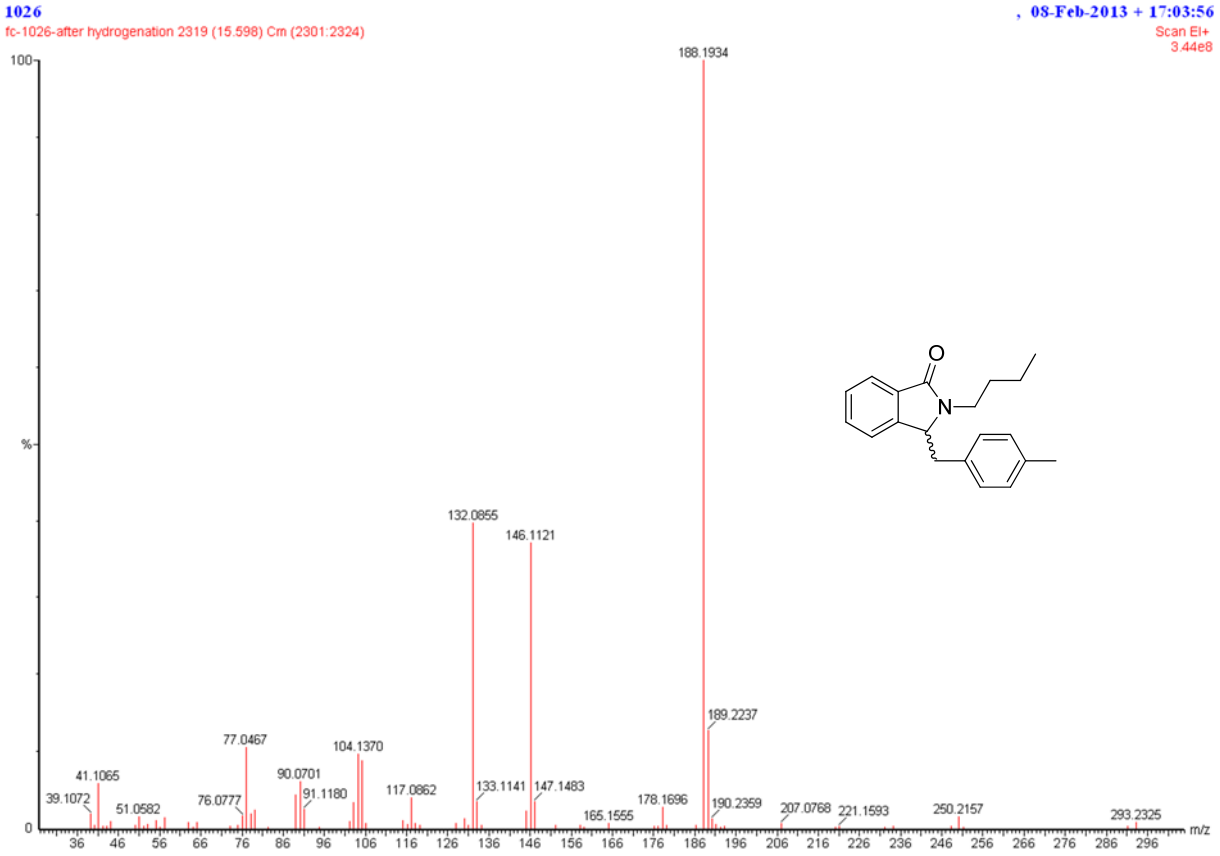

1022
, 07-Feb-2013 + 16:57:02
fc-1022-after hydrogen-2ation Sm (Mn, 1x3)
Scan EI+
TIC
2.18e9
100
Area
%
0
15.26;286587840
14.41
133951
15.06
259508
O
N
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

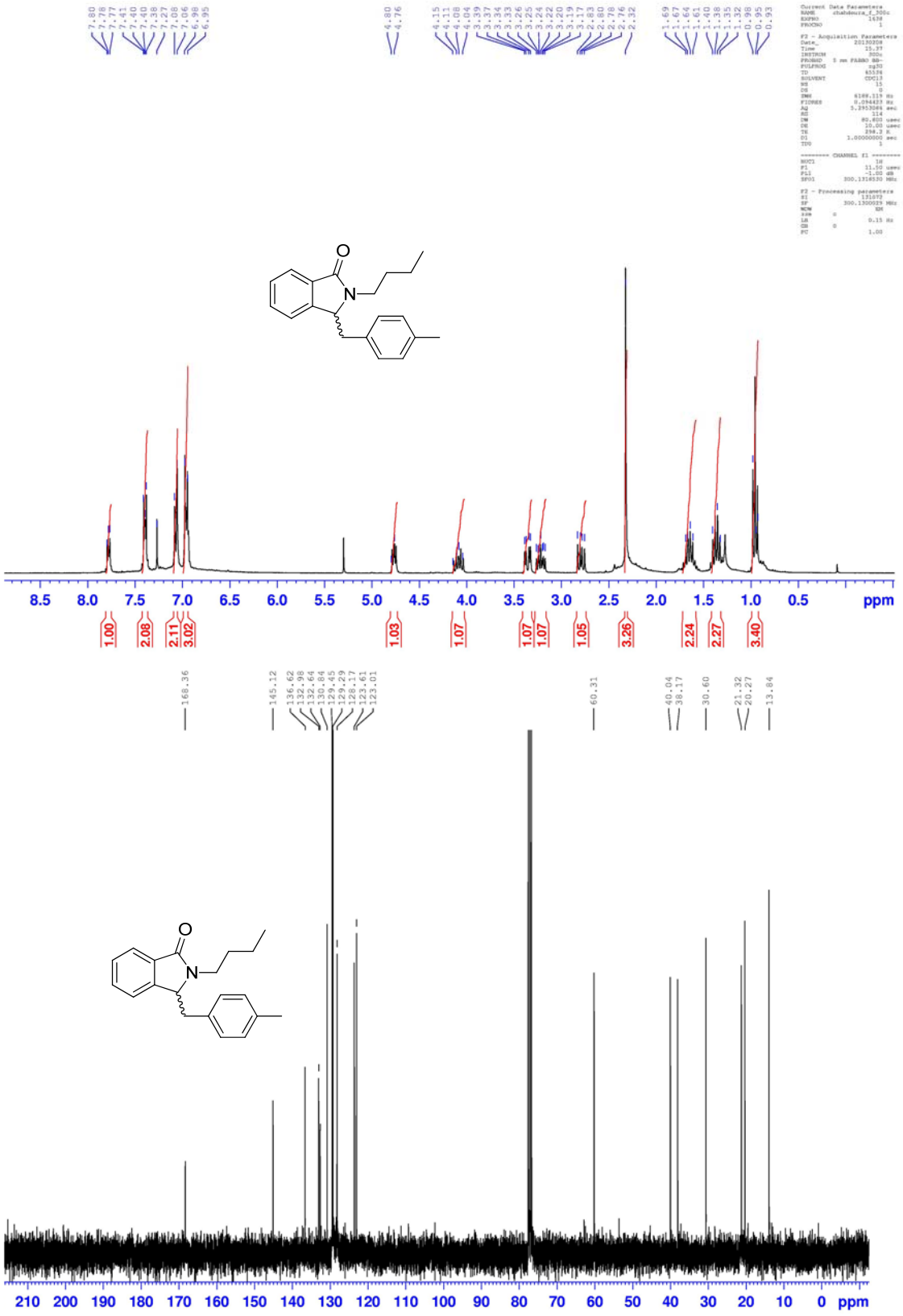

1022
, 07-Feb-2013 + 16:57:02
fc-1022-after hydrogen-2ation 2252 (15.263) Cm (2224:2254)
Scan EI+
5.45e8
100
%
0
188.1825
132.0563
146.1068
189.1815
91.0564
77.0500
104.1280
41.1197
39.1042
44.0566
65.0107
78.0454
103.1440
117.0802
133.1050
147.1220
158.1152
176.1616
178.1645
190.2015
207.0849
236.1748
250.1610
279.1468
O
N
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
280
290
m/z



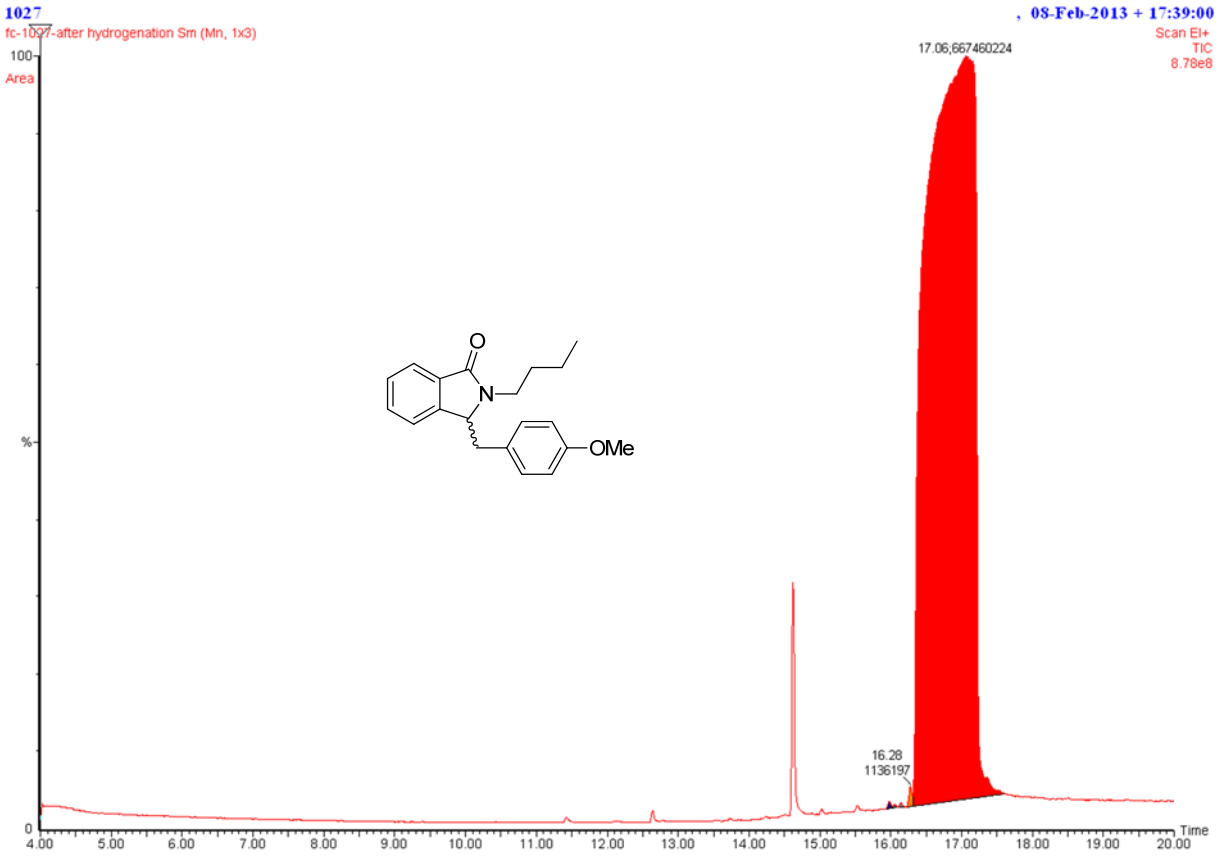
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **l33H**

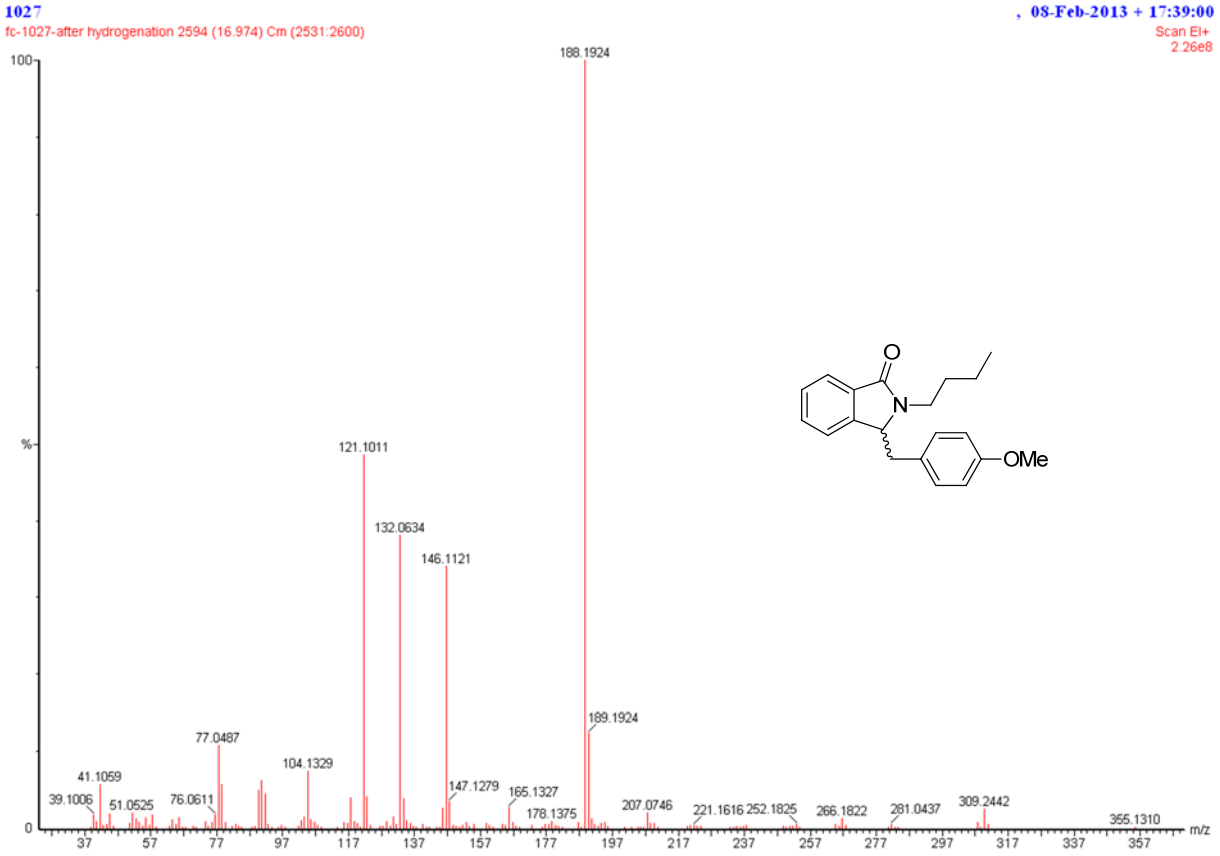

1026
, 08-Feb-2013 + 17:03:56
fc-1026-after hydrogenation Sm (Mn, 1x3)
Scan EI+
TIC
1.87e9
15.58;120017168
100
Area
%
0
Time
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
O
N

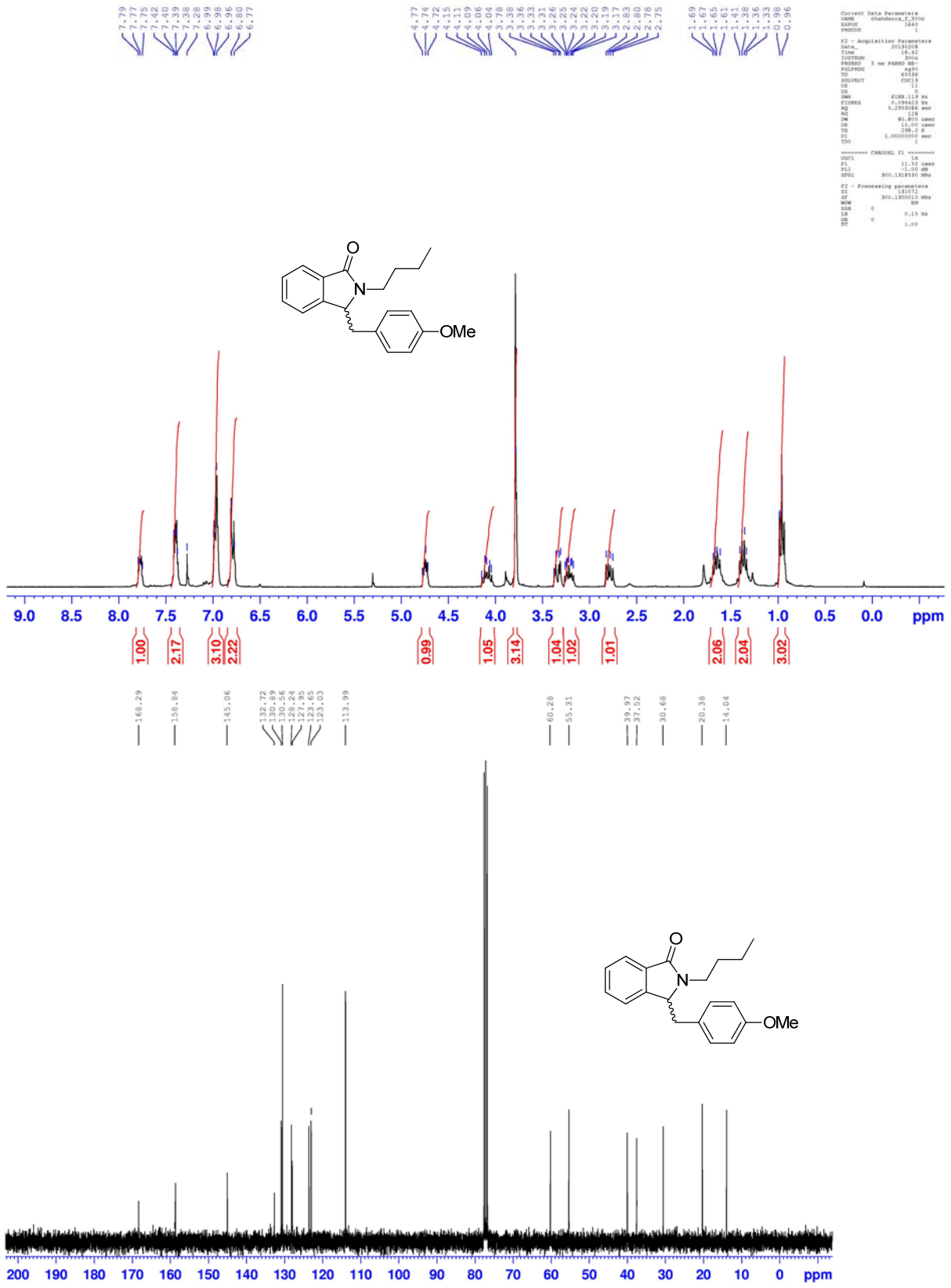

1026
, 08-Feb-2013 + 17:03:56
fc-1026-after hydrogenation 2319 (15.598) Cm (2301:2324)
Scan EI+
3.44e8
188.1934
100
%
0
132.0855
146.1121
189.2237
77.0467
104.1370
41.1065
90.0701
39.1072
91.1180
51.0582
76.0777
117.0862
133.1141
147.1483
165.1555
178.1696
190.2359
207.0768
221.1593
250.2157
293.2325
m/z
36
46
56
66
76
86
96
106
116
126
136
146
156
166
176
186
196
206
216
226
236
246
256
266
276
286
296
O
N



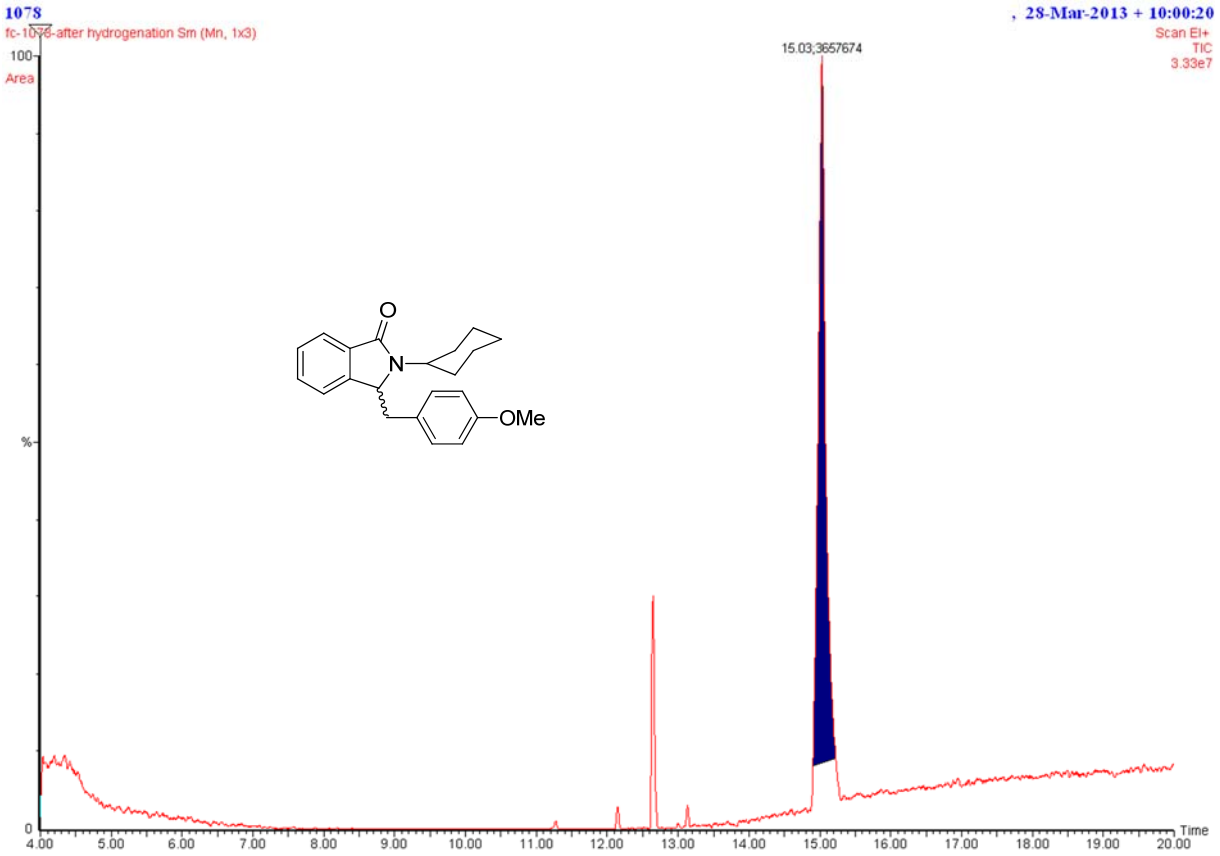
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **l34H**

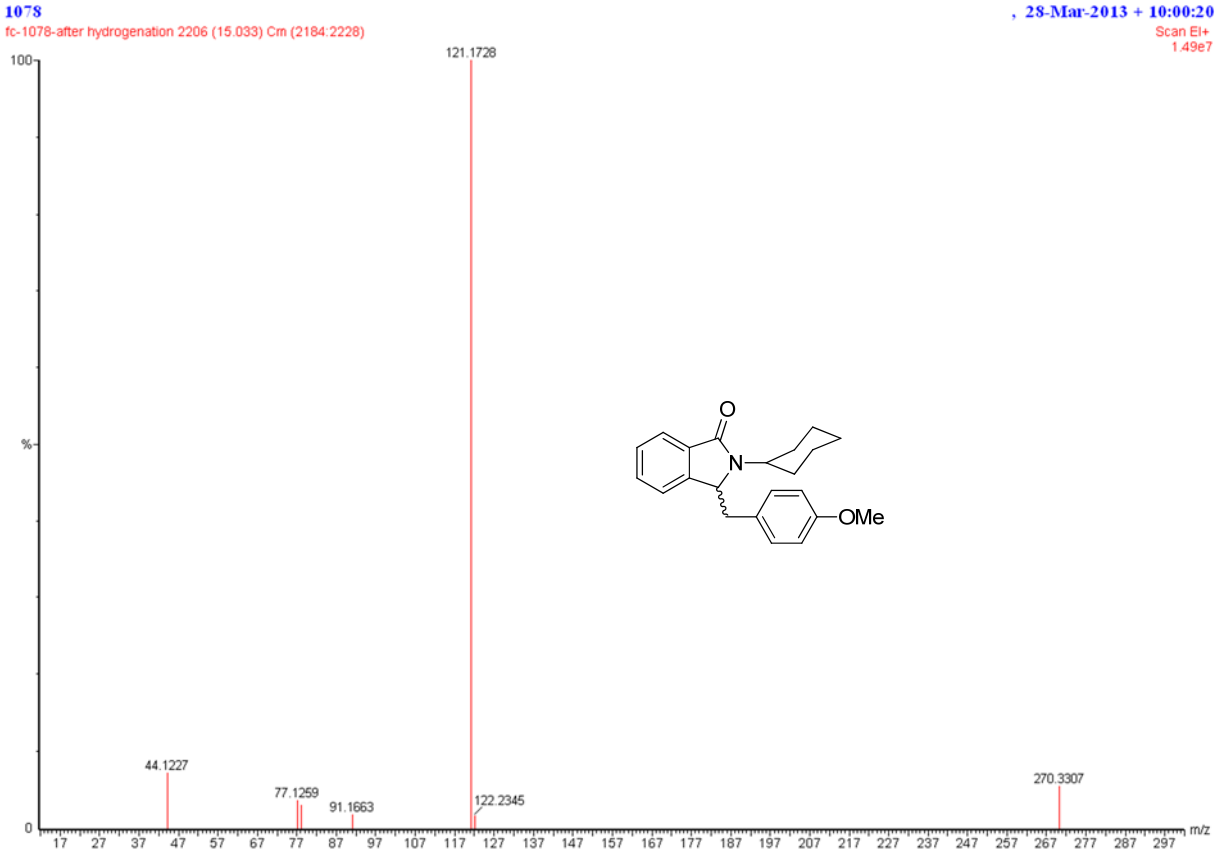

1027
fc-1027-after hydrogenation Sm (Mn, 1x3)
, 08-Feb-2013 + 17:39:00
Scan EI+
TIC
8.78e8
Area
100
%
0
17.06;667460224
16.28
1136197
O
N
OMe
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

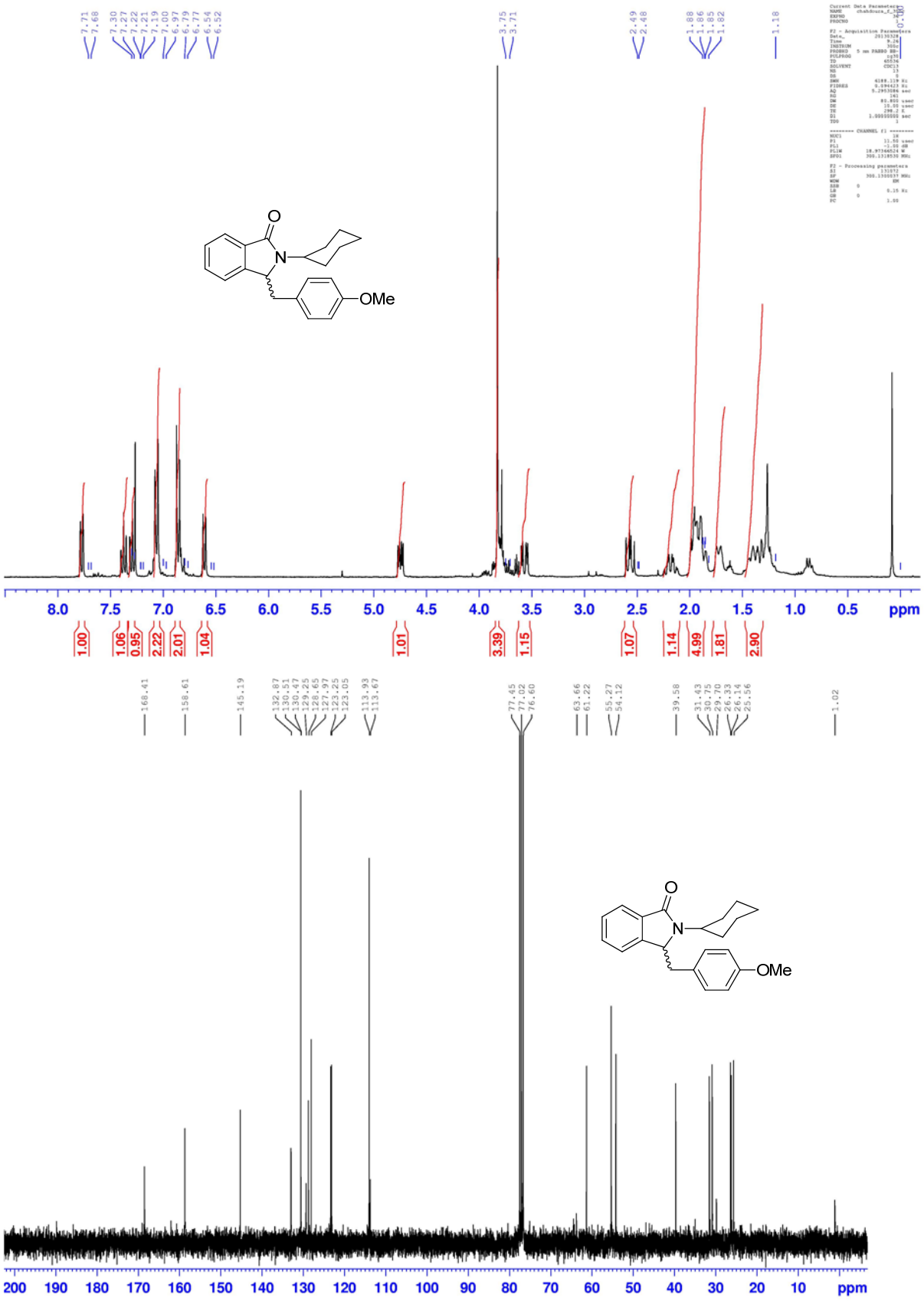

1027
fc-1027-after hydrogenation 2594 (16.974) Cm (2531:2600)
, 08-Feb-2013 + 17:39:00
Scan EI+
2.26e8
100
%
0
188.1924
121.1011
132.0634
146.1121
189.1924
77.0487
104.1329
41.1059
39.1006
51.0525
76.0611
147.1279
165.1327
178.1375
207.0746
221.1616
252.1825
266.1822
281.0437
309.2442
355.1310
O
N
OMe
37
57
77
97
117
137
157
177
197
217
237
257
277
297
317
337
357
m/z



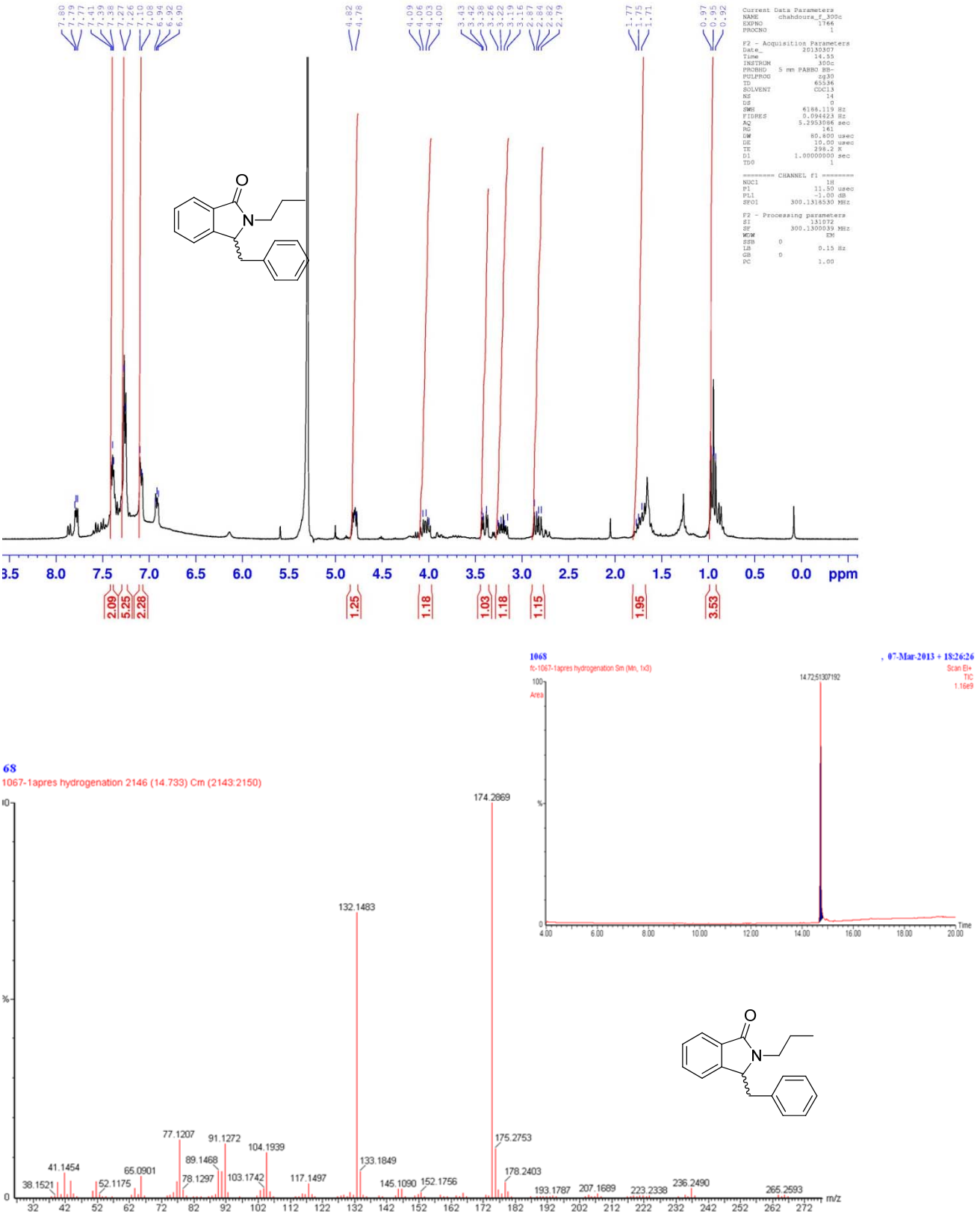
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **l35H**


1078
fc-1078-after hydrogenation Sm (Mn, 1x3)
, 28-Mar-2013 + 10:00:20
Scan EI+
TIC
3.33e7
100
Area
%
0
15.03;3657674
O
N
OMe
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

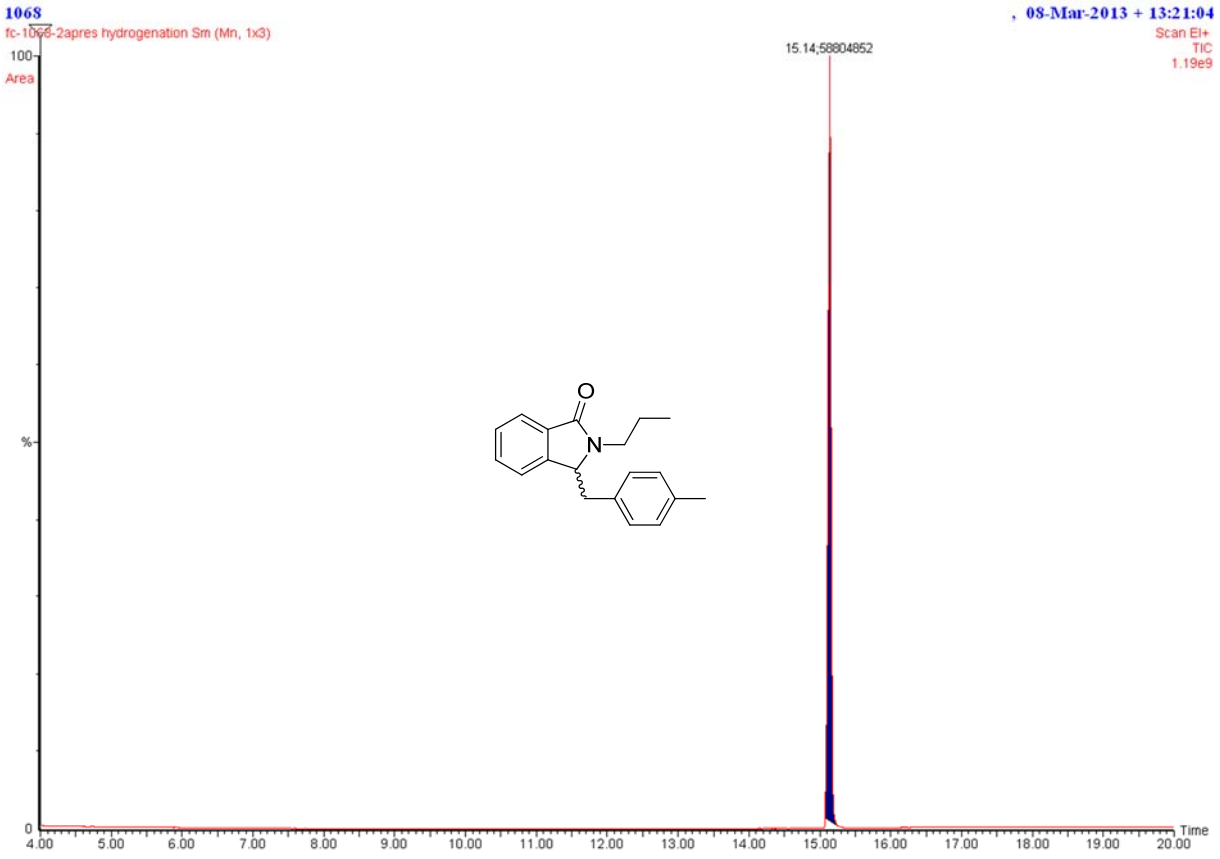

1078
fc-1078-after hydrogenation 2206 (15.033) Cm (2184:2228)
, 28-Mar-2013 + 10:00:20
Scan EI+
1.49e7
100
%
0
121.1728
44.1227
77.1259
91.1663
122.2345
270.3307
O
N
OMe
17
27
37
47
57
67
77
87
97
107
117
127
137
147
157
167
177
187
197
207
217
227
237
247
257
267
277
287
297
m/z

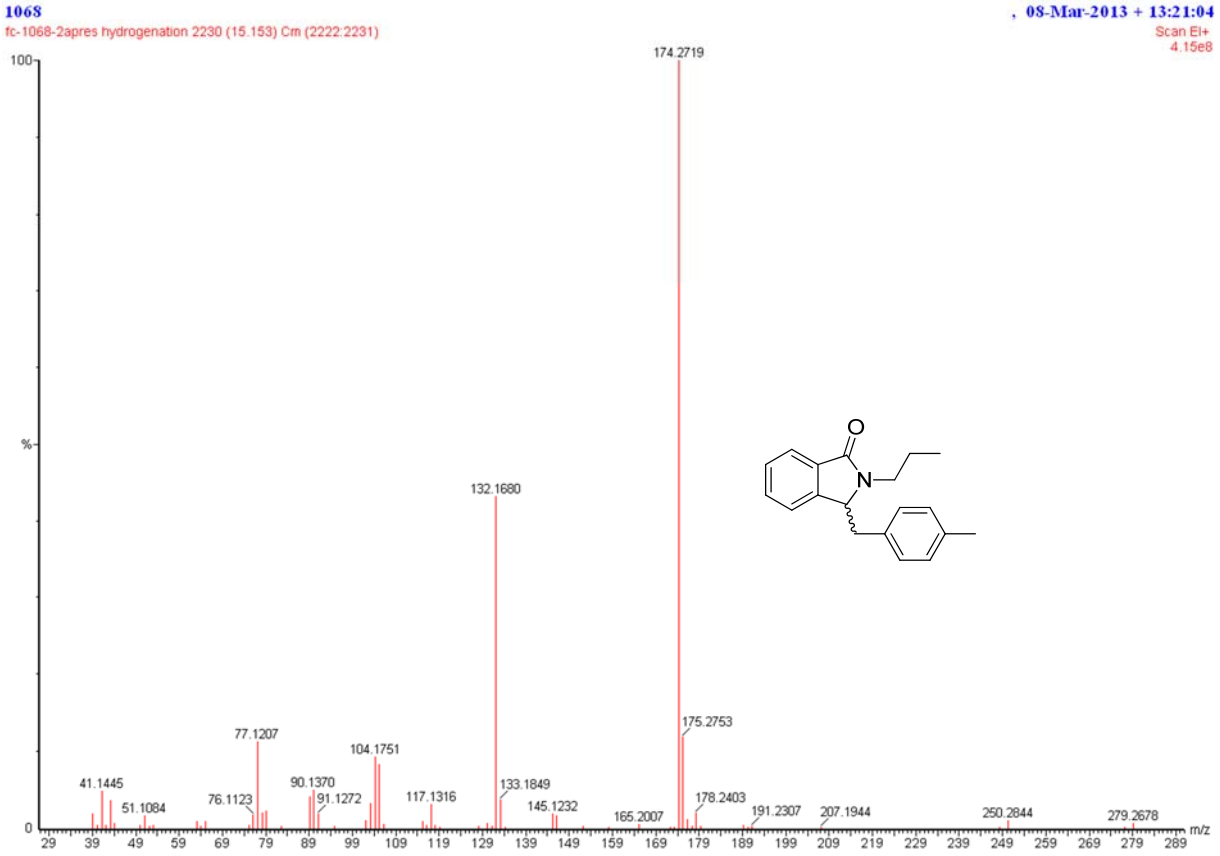


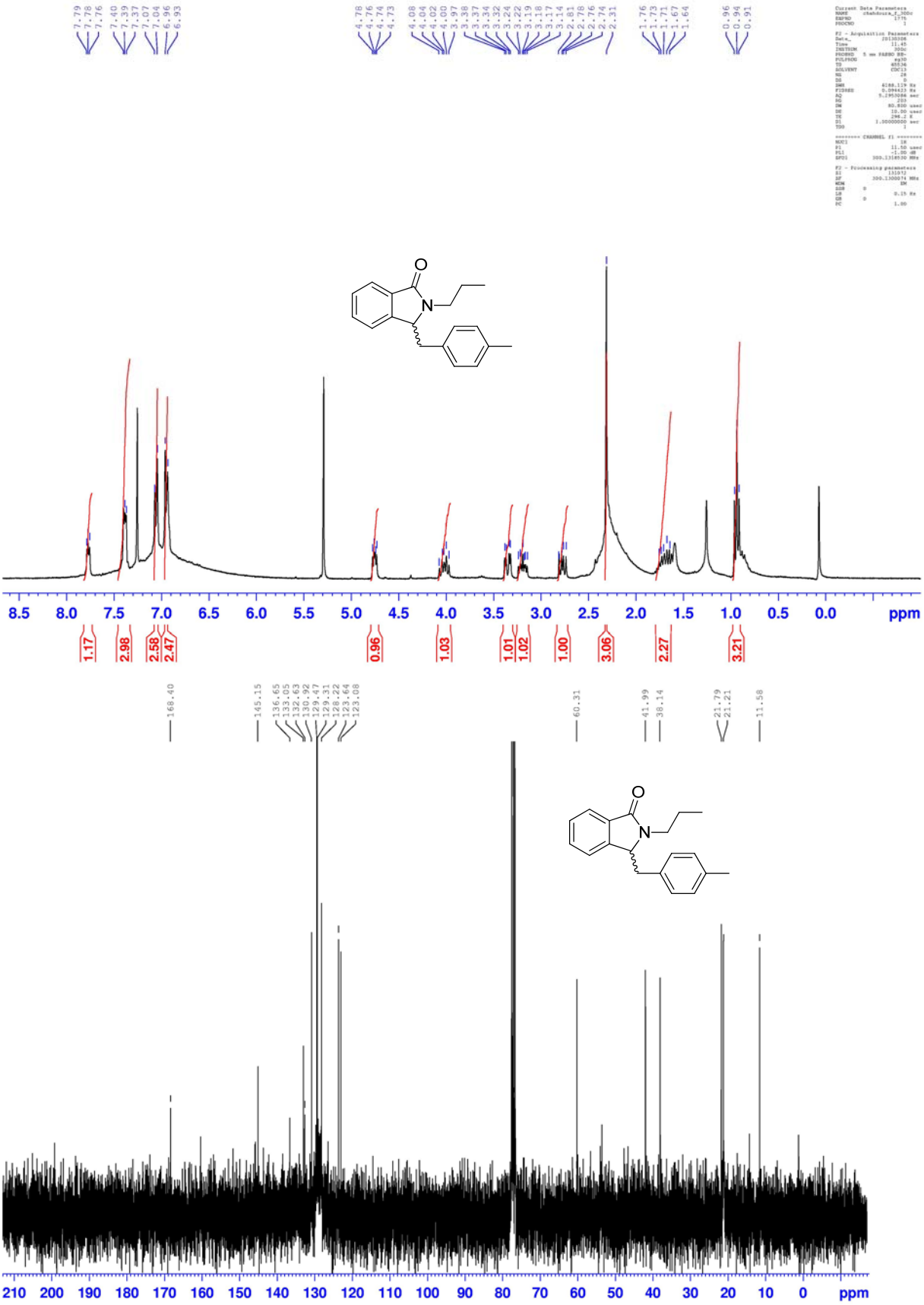
GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **n35H**



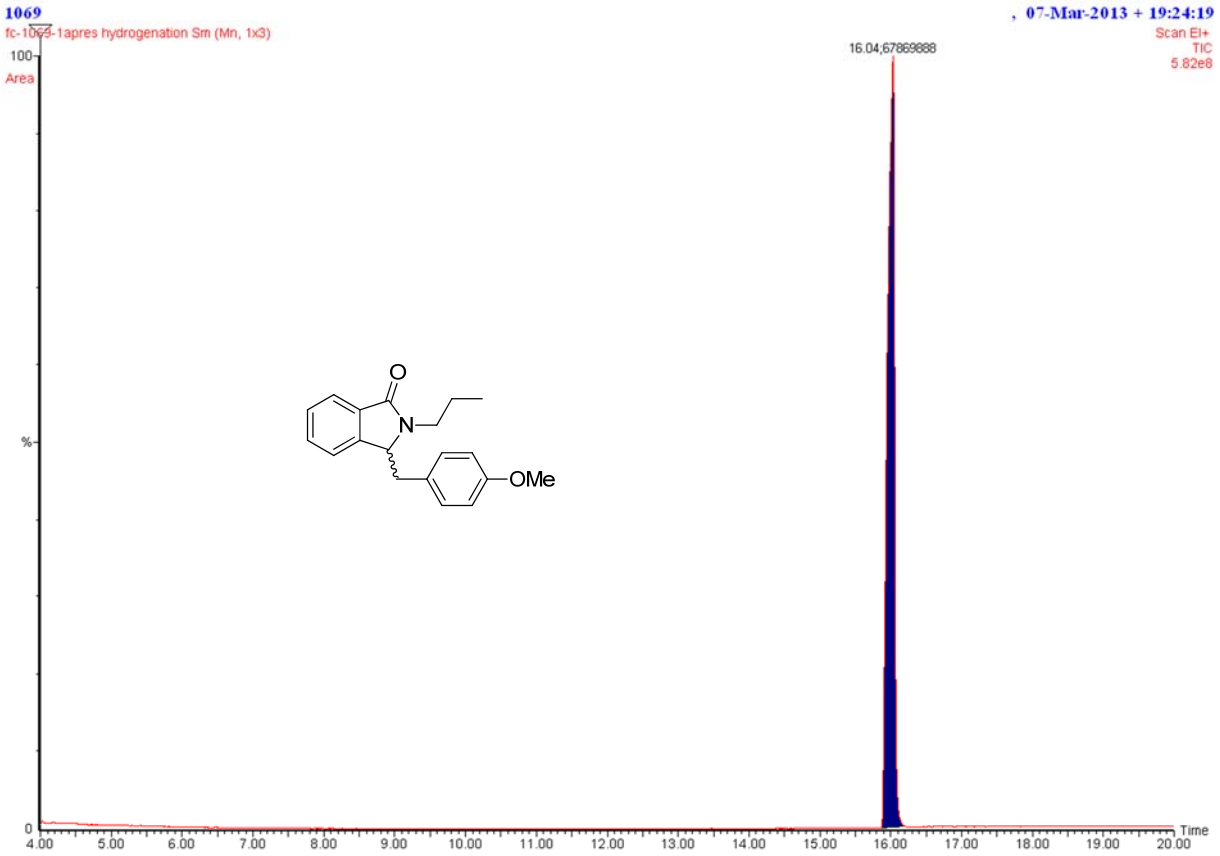
^{1}H NMR (300 MHz, $CDCl_3$) (top) and GC (FID and MS) spectra (bottom) for **m33H**

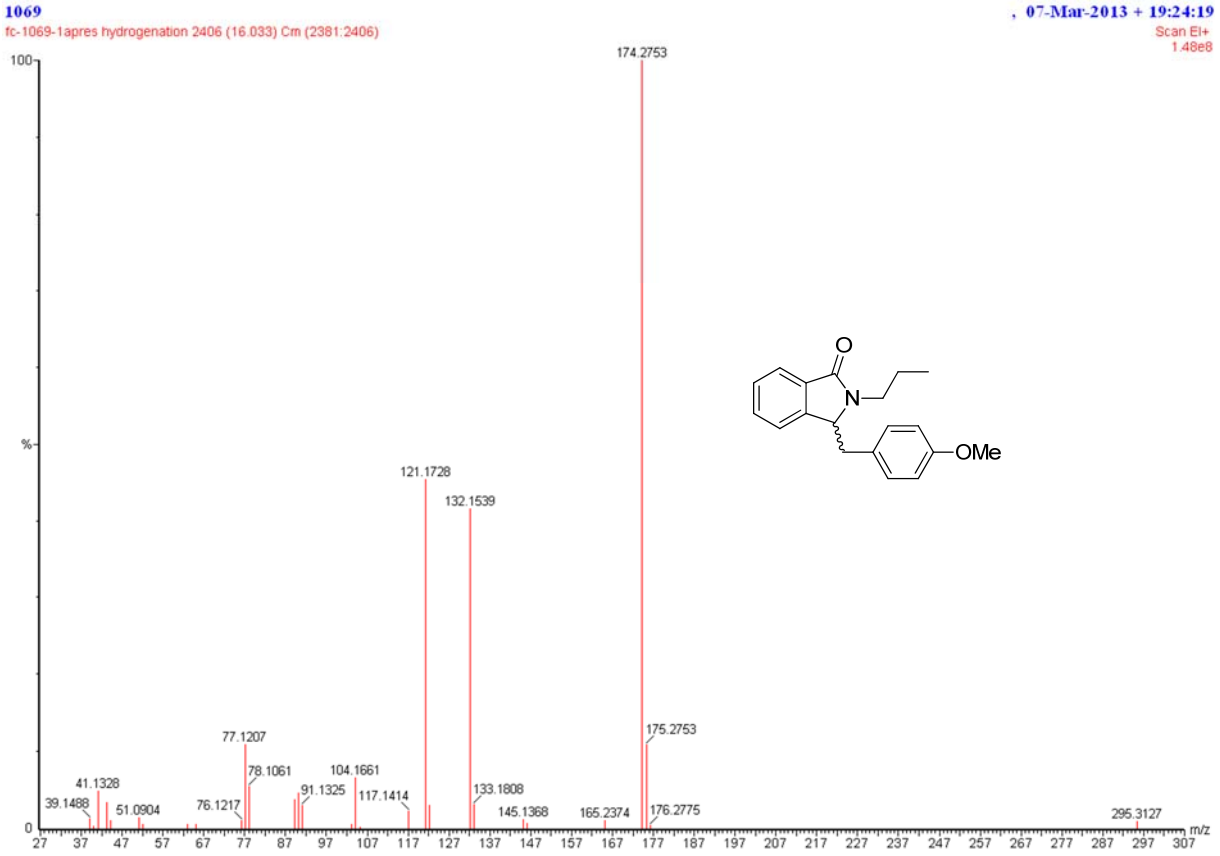

1068
, 08-Mar-2013 + 13:21:04
fc-1068-2apres hydrogenation Sm (Mn, 1x3)
Scan EI+
TIC
1.19e9
100
Area
%
0
15.14;58804852
O
N
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
16.00
17.00
18.00
19.00
20.00
Time

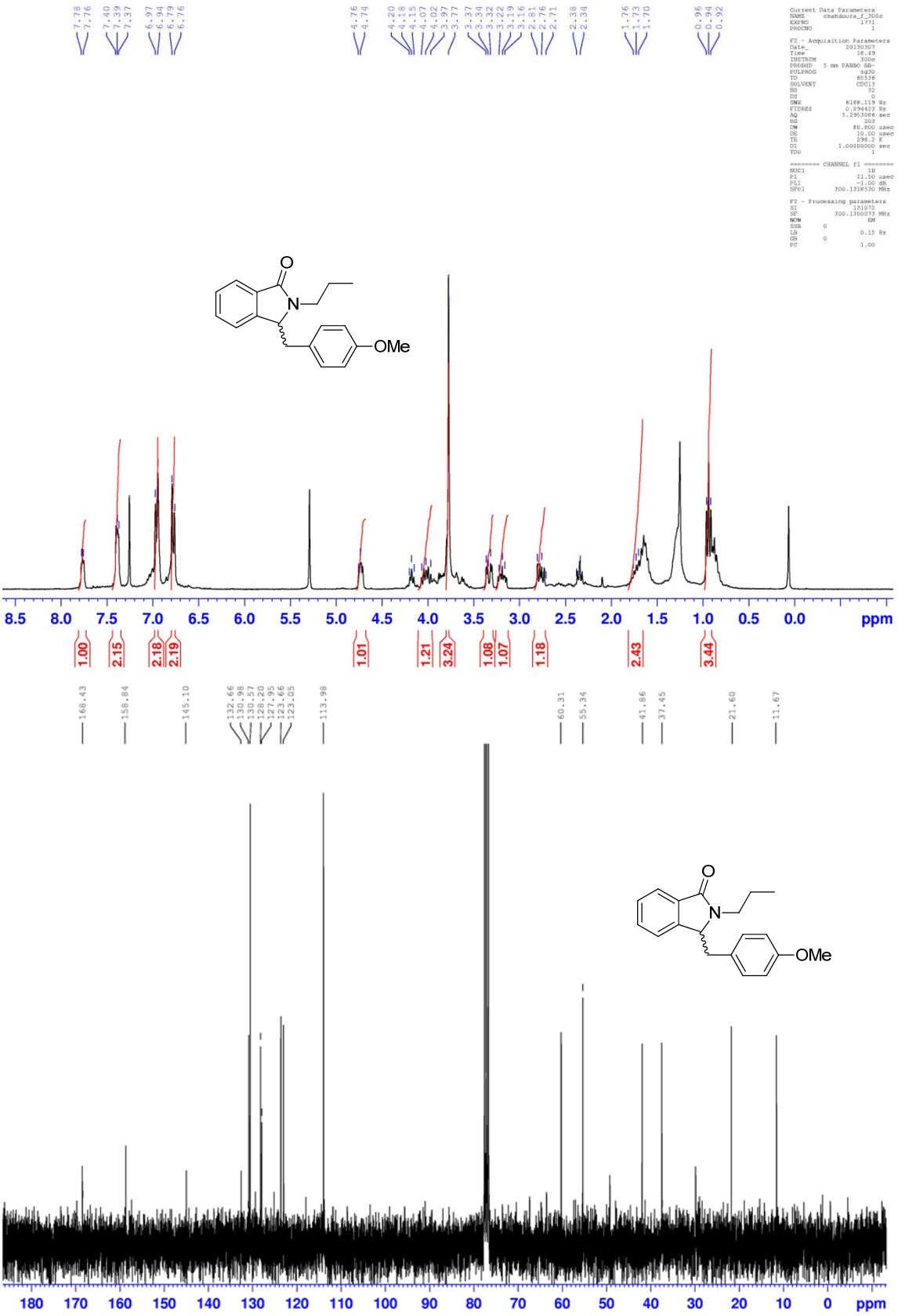

1068
, 08-Mar-2013 + 13:21:04
fc-1068-2apres hydrogenation 2230 (15.153) Cm (2222:2231)
Scan EI+
4.15e8
100
%
0
174.2719
132.1680
175.2753
77.1207
104.1751
41.1445
51.1084
76.1123
90.1370
91.1272
117.1316
133.1849
145.1232
165.2007
178.2403
191.2307
207.1944
250.2844
279.2678
O
N
29
39
49
59
69
79
89
99
109
119
129
139
149
159
169
179
189
199
209
219
229
239
249
259
269
279
289
m/z



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C $\{^{1}H\}$ NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **m34H**


1069
fc-1069-1apres hydrogenation Sm (Mn, 1x3)
, 07-Mar-2013 + 19:24:19
Scan EI+
TIC
5.82e8
Area
16.04;67869888
O
N
OMe
%
Time


1069
fc-1069-1apres hydrogenation 2406 (16.033) Cm (2381:2406)
, 07-Mar-2013 + 19:24:19
Scan EI+
1.48e8
174.2753
O
N
OMe
121.1728
132.1539
77.1207
78.1061
104.1661
91.1325
117.1414
133.1808
175.2753
41.1328
39.1488
51.0904
76.1217
145.1368
165.2374
176.2775
295.3127
%
m/z



GC (FID and MS) spectra (top), ^{1}H NMR (300 MHz) (middle) and ^{13}C {^{1}H} NMR (75 MHz) (bottom) spectra in $CDCl_3$ for **m35H**