

Supporting Information
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Triflic acid mediated cyclization of *N*-phenethyl and *N*-(3-indolyloethyl) unsymmetrical succinimides: regio and diastereoselective synthesis of substituted pyrroloisoquinolinones and indolizinoindolones.

Jayaraman Selvakumar, Selvaraj Mangalaraj, Kamsali Murali Mohan Achari, Krishna Mukund
and Chinnsamy Ramaraj Ramanathan*

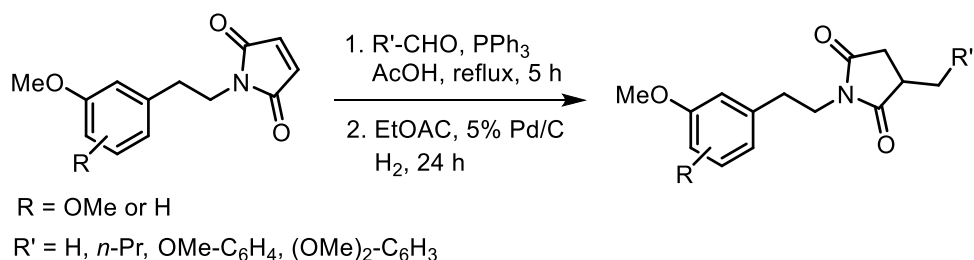
Department of Chemistry, Pondicherry University, Puducherry – 605 014, India.

E-mail: crnath.che@pondiuni.edu.in

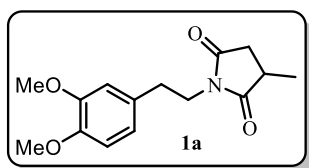
1. General experimental conditions:

Melting points were measured with an EZ Melt (Stanford Research Systems, USA). Infrared spectra were recorded on a Thermo Nicolet 6700 FT-IR spectrophotometer by using potassium bromide thin films. The IR data are reported in absorption frequencies (cm^{-1}). HRMS were measured by micromass Q-TOF (ESI-HRMS). The ^1H and ^{13}C NMR spectroscopic data were recorded with a Bruker AVANCE 400 spectrometer. All NMR spectra were recorded at room temperature, and either CDCl_3 or $\text{DMSO}-d_6$ was used as the solvent with TMS as an internal standard. The chemical shifts are expressed in δ (ppm) downfield from the signal of the TMS. Coupling constant (J) values are given in Hz. The multiplicities of the NMR signals are reported as: s (sing-let), d (doublet), t (triplet), q (quartet), qn (quintet), m (multiplet), dd (doublet of doublet), ddd (doublet of doublet of doublet), dt (doublet of triplet), td (triplet of doublet), or br. s (broad singlet). X-ray crystal data were collected on an Oxford Diffraction Xcalibur diffractometer with Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$). The empirical absorption correction by using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm, was applied. The structure solution and refinement were performed with SHELX-97.¹ All solvents that were used for the reactions were distilled according to standard procedures.² Dry dichloromethane was obtained by distillation over calcium hydride. The substituted phenethylamines were prepared from the corresponding aldehydes by three simple synthetic steps that involved a nitroaldol condensation and a reduction of the double bond and nitro group.³ The reagents TfOH, phenethylamine, and 3,4-dimethoxyphenethylamine from Aldrich were used without further purification. Column chromatography was performed on Merck silica gel (100–200 mesh). The developed TLC plates (Merck 60 F 254 precoated silica plates) were visualized by using phosphomolybdic acid stain, permanganate stain, and UV light.

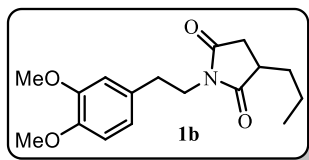
2. Preparation of unsymmetrical phenethyl-succinimides⁴



Phenethylmaleimide (2 mmol) was dissolved in AcOH (1 mL) in a 50 mL round bottom flask with stirring. Triphenylphosphine (540 mg, 2 mmol) and aldehyde (2 mmol) were added and the mixture was refluxed for 5 h. The crude reaction mixture was diluted with water (20 mL) and extracted with ethyl acetate (2 x 20 mL). The organic layer was washed with brine and dried over anhydrous Na₂SO₄. The solvent was evaporated and the purification of the crude product by silica gel column chromatography using ethyl acetate 30% and hexane 70% as eluent yields the methaleneimide as colorless solid (small quantity of triphenylphosphine oxide also come usually). A mixture of methylene imide and 5% Pd on activated charcoal (100 mg) in ethyl acetate (100 mL) was stirred well under hydrogen atmosphere at room temperature for 24 h, the mixture was filtered through a pad of celite and removal of solvent by rotary evaporation under reduced pressure furnished a crude 2-alkyl succinimides **1a-h**. The crude product was purified through silica gel column chromatography using ethyl acetate and hexane (30:70) as eluent.

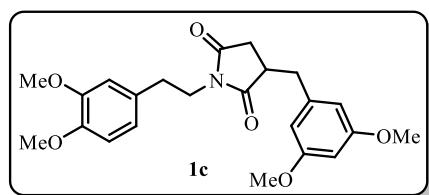


Following the general procedure, the *N*-[2-(3,4-dimethoxyphenyl)ethyl]-maleimide (522 mg, 2 mmol) and paraformaldehyde (300 mg, 2 mmol) furnished **1a** as colorless solid, 399 mg, 72% yield, mp. 89-90 °C, IR (KBr, cm⁻¹): 3001, 2971, 2939, 1770, 1698, 1589, 1517, 1405, 1267, 1233, 1154, 1028, 759; ¹H NMR (400 MHz, CDCl₃): δ 6.78-6.76 (m, 1H), 6.73 (dd, *J* = 6.1, 1.9 Hz, 2H), 3.86 (s, 3H), 3.84 (s, 3H), 3.73-3.69 (m, 2H), 2.87-2.77 (m, 3H), 2.76-2.71 (m, 1H), 2.22 (dd, *J* = 17.6, 4.1 Hz, 1H), 1.26 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 180.5, 176.4, 149.0, 147.9, 130.3, 121.0, 112.2, 111.3, 56.0, 39.9, 36.5, 34.7, 33.12, 16.9.



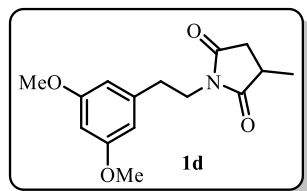
Following the general procedure, the *N*-[2-(3,4-dimethoxyphenyl)ethyl]-maleimide (522 mg, 2 mmol) and propanal

(580 mg, 10 mmol) furnished **1b** as colorless semisolid, 464 mg, 76% yield, IR (KBr, cm^{-1}): 3001, 2937, 2865, 1773, 1693, 1591, 1518, 1464, 1401, 1235, 1153, 1028, 808, 765; ^1H NMR (400 MHz, CDCl_3): δ 6.78 (m, 1H), 6.73-6.72 (m, 2H), 3.86 (s, 3H), 3.83 (s, 3H), 3.73-3.67 (m, 2H), 2.84-2.80 (m, 2H), 2.78-2.67 (m, 2H), 2.30-2.26 (m, 1H), 1.79-1.77 (m, 1H), 1.34-1.30 (m, 3H), 0.91 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 179.8, 176.5, 148.8, 147.7, 130.1, 120.9, 112.0, 111.1, 55.8, 39.7, 39.5, 34.2, 33.4, 32.9, 19.86, 13.71.



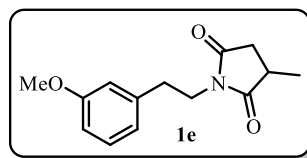
Following the general procedure, the *N*-[2-(3,4-dimethoxyphenyl)ethyl]-maleimide (522 mg, 2 mmol) and 3,5-dimethoxybenzaldehyde (332 mg, 2 mmol) furnished **1c** as colorless semisolid. 694 mg, 84% yield, IR (KBr, cm^{-1}):

2998, 2939, 2837, 1773, 1701, 1596, 1515, 1463, 1401, 1350, 1205, 1066, 1027, 837; ^1H NMR (400 MHz, CDCl_3): δ 6.79-6.77 (m, 1H), 6.73-6.71 (m, 2H), 6.33 (t, $J = 2.4$ Hz, 1H), 6.29 (d, $J = 2.4$ Hz, 2H), 3.88 (s, 3H), 3.85 (s, 3H), 3.76 (s, 6H), 3.72-3.68 (m, 2H), 3.12 (dd, $J = 13.6, 4.4$ Hz, 1H), 3.04 (septet, $J = 4.4$ Hz, 1H), 2.81-2.77 (m, 2H), 2.69-2.60 (m, 2H), 2.40 (dd, $J = 18.4, 4.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 179.0, 176.1, 161.0, 148.9, 147.8, 139.4, 130.2, 120.87, 112.0, 111.2, 107.0, 98.6, 55.89, 55.87, 55.2, 41.0, 39.8, 36.7, 33.2, 33.0, 14.1.



Following the general procedure, the *N*-[2-(3,5-dimethoxyphenyl)ethyl]-maleimide (522 mg, 2 mmol) and paraformaldehyde (300 mg, 2 mmol) furnished **1d** as colorless semisolid. 432 mg, 78% yield, IR (KBr, cm^{-1}): 2936, 2871, 2836,

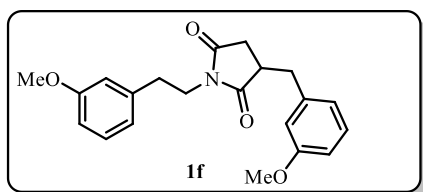
1775, 1713, 1597, 1464, 1403, 1358, 1205, 1154, 1055, 1007, 833; ^1H NMR (400 MHz, CDCl_3): δ 6.35 (d, $J = 2.2$ Hz, 2H), 6.31 (d, $J = 2.2$ Hz, 1H), 3.76 (s, 6H), 3.74-3.71 (m, 2H), 2.89-2.80 (m, 3H), 2.25 (dd, $J = 17.6, 4.0$ Hz, 1H), 1.27 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 180.5, 176.3, 161.0, 140.1, 106.9, 99.0, 55.4, 39.7, 36.5, 34.7, 33.8, 16.9.



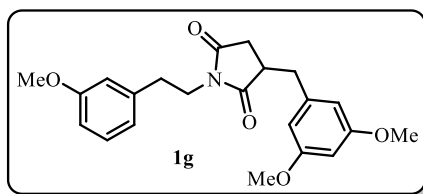
Following the general procedure, the *N*-[2-(3-methoxyphenyl)ethyl]-maleimide (462 mg, 2 mmol) and paraformaldehyde (300 mg, 2 mmol) furnished *N*-[2-(3-methoxyphenyl)ethyl]-3-methylsuccinimide

1e as colorless semisolid. 410 mg, 83% yield, IR (KBr, cm^{-1}): 2966, 2940, 2834, 1775, 1701,

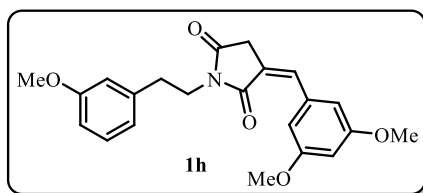
1602, 1490, 1402, 1261, 1157, 1042, 1006, 784, 696; ^1H NMR (400 MHz, CDCl_3): δ 7.19 (t, $J = 7.6$ Hz, 1H), 6.80-6.74 (m, 3H), 3.79 (s, 3H), 3.74 (t, $J = 7.6$ Hz, 2H), 2.86 (t, $J = 7.6$ Hz, 2H), 2.81-2.74 (m, 2H), 2.23 (dd, $J = 17.6, 4.1$ Hz, 1H), 1.27 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 180.3, 176.2, 159.7, 139.2, 129.4, 121.2, 114.4, 112.2, 55.1, 39.6, 36.3, 34.5, 33.4, 16.7.



Following the general procedure, the *N*-[2-(3-methoxyphenyl)ethyl]-maleimide (462 mg, 2 mmol) and 3-methoxybenzaldehyde (272 mg, 2 mmol) furnished *N*-[2-(3-methoxyphenyl)ethyl]-3-(3-methoxybenzyl)succinimide **1f** as colorless semisolid. 572 mg, 81% yield, IR (KBr, cm^{-1}): 2998, 2941, 2835, 1774, 1701, 1601, 1490, 1436, 1262, 1155, 1042, 779; ^1H NMR (400 MHz, CDCl_3): δ 7.20 (td, $J = 7.8, 3.4$ Hz, 2H), 6.78-6.73 (m, 4H), 6.71-6.69 (m, 1H), 3.79 (s, 3H), 3.78 (s, 3H), 3.73-3.69 (m, 2H), 3.15 (dd, $J = 13.7, 4.5$ Hz, 1H), 3.05 (septet, $J = 4.5$ Hz, 1H), 2.83-2.78 (m, 2H), 2.70 (dd, $J = 13.7, 9.0$ Hz, 1H), 2.63 (dd, $J = 18.3, 9.0$ Hz, 1H), 2.39 (dd, $J = 18.3, 4.6$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 179.0, 176.0, 159.8, 159.7, 139.2, 138.7, 129.8, 129.5, 121.2, 114.8, 114.4, 112.2, 55.1, 41.1, 39.7, 36.6, 33.4, 33.2.



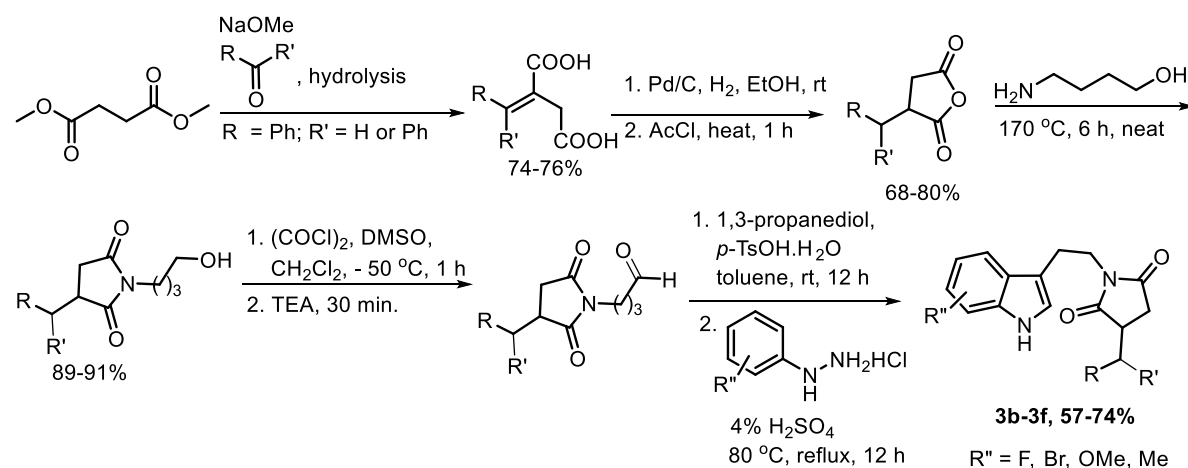
Following the general procedure, the *N*-[2-(3-methoxyphenyl)ethyl]-maleimide (462 mg, 2 mmol) and 3,5-dimethoxybenzaldehyde (332 mg, 2 mmol) furnished **1g** as colorless semisolid. 659 mg, 86% yield, IR (KBr, cm^{-1}): 3002, 2940, 2834, 1773, 1702, 1596, 1457, 1401, 1261, 1205, 115, 1050, 837, 696; ^1H NMR (400 MHz, CDCl_3): δ 7.20 (t, $J = 7.7$ Hz, 1H), 6.78-6.74 (m, 3H), 6.33 (t, $J = 2.2$ Hz, 1H), 6.29 (d, $J = 2.2$ Hz, 2H), 3.79 (s, 3H), 3.76 (s, 6H), 3.71 (t, $J = 8.0$ Hz, 2H), 3.11 (dd, $J = 13.6, 4.4$ Hz, 1H), 3.04 (septet, $J = 4.5$ Hz, 1H), 2.87-2.76 (m, 2H), 2.68-2.60 (m, 2H), 2.40 (dd, $J = 18.4, 4.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 179.0, 176.1, 161.0, 159.7, 139.5, 139.3, 129.5, 121.2, 114.5, 112.2, 107.1, 98.7, 55.3, 55.2, 41.0, 39.7, 36.8, 33.5, 33.2.



Following the general procedure, the *N*-[2-(3-methoxyphenyl)ethyl]-maleimide (462 mg, 2 mmol) and 3,5-

dimethoxybenzaldehyde (332 mg, 2 mmol). Purification of the crude product by silica gel column chromatography using ethyl acetate 30% and hexane 70% furnished small quantity of **1h** as colorless solid (Remaining was a mixture of benzylidenimine and triphenylphosphineoxide). mp. 140-141 °C, IR (KBr, cm^{-1}): 3008, 2945, 2840, 1763, 1701, 1655, 1590, 1399, 1299, 1161, 1072, 842, 778; ^1H NMR (400 MHz, CDCl_3): δ 7.52 (t, $J = 2.4$ Hz, 1H), 7.23-7.18 (m, 1H), 6.85-6.83 (m, 1H), 6.8-6.7 (m, 2H), 6.1 (d, $J = 2.0$ Hz, 2H), 6.52 (t, $J = 2.0$ Hz, 1H), 3.89-3.85 (m, 2H), 3.82 (s, 6H), 3.79 (s, 3H), 3.53 (d, $J = 2.4$ Hz, 1H), 2.94-2.91 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 173.8, 170.8, 161.2, 139.5, 135.9, 134.4, 129.7, 124.0, 121.3, 114.4, 112.4, 108.3, 107.2, 102.3, 55.5, 55.3, 40.1, 34.11, 33.9.

3. Preparation of unsymmetrical indolyethyl-succinimides



3. Preparation of 2-alkenylsuccinic acids

Sodium metal (160 mmol) was added carefully to methanol (120 mL) cooled in an ice bath. After sodium dissolution, the dimethyl succinate (188 mmol) in methanol (30 mL) was added to the solution at room temperature. A solution of aldehyde or ketone (94 mmol) in methanol (30 mL) was added dropwise to the solution for 1 h at reflux condition and the refluxing was continued to further 5 h. To this mixture, NaOH (18 g in 90 mL of H_2O) was added and the reaction mixture was refluxed overnight. The methanol from reaction mixture was evaporated under reduced pressure and the resulting residue was diluted with water (45 mL), washed with ethyl acetate (120 mL), then acidified with conc. HCl. The precipitate was

collected by filtration washed with water and dried under vacuum, to afford 2-alkenylsuccinic acid in pure form.

3.2. Preparation of 2-alkylsuccinic acids

2-Alkenylsuccinic acid (67 mmol) was dissolved in ethanol (120 mL) to which the 10% Pd in activated charcoal (700 mg) was added. The reaction mixture was filled with hydrogen gas using hydrogen balloon with 1 atm pressure and stirred. After completion of the reaction, the mixture was filtered through a bed of celite and the ethanol was evaporated and dried to afford 2-alkylsuccinic acids in pure form.

3.3. Preparation of 2-alkylsuccinic anhydrides

2-Alkylsuccinic acid (52 mmol) was dissolved in acetyl chloride (158 mmol) and the mixture was heated in water bath. Once the mixture was dissolved completely, the setup was cooled gradually to room temperature and then ice cold condition. The solid generated 2-alkylsuccinic anhydrides are used in the next step without further purification.

3.4. Preparation of 3-benzyl-1-(4-hydroxybutyl)pyrrolidine-2,5-dione

A mixture of finely powdered 3-benzyl-dihydrofuran-2,5-dione (4.5 g, 24 mmol) and 4-amino-1-butanol (2.13 g, 24 mmol) were heated at 170 °C with vigorous stirring under nitrogen atmosphere. After 6 h the reaction mixture was cooled to 80 °C and it was poured to 35 mL of ice-cold water. The product was extracted with CHCl₃ (2 x 100 mL), and the combined organic layer was washed with 5% NaHCO₃ solution (100 mL), and with water (100 mL). Organic layer was dried over anhydrous Na₂SO₄, filtered and the solvent was removed under vacuum to give the mixture, which was purified through silica gel column chromatography using ethyl acetate : hexane as eluent (2:5) to give 3-benzyl-1-(4-hydroxybutyl)pyrrolidine-2,5-dione as viscous liquid.

3.5 Preparation of 1-(3-(1,3-dioxan-2-yl)propyl)-3-benzylpyrrolidine-2,5-dione

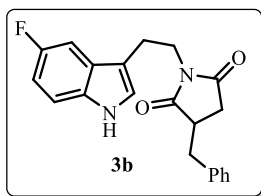
To a well stirred solution of oxalyl chloride (2.1 mL, 23.9 mmol) in 20 mL of CH₂Cl₂, a solution of anhydrous (CH₃)₂SO (3.6 mL, 50.7 mmol) in 20 mL of CH₂Cl₂ was added under nitrogen atmosphere at -50 °C at such a rate that temperature was maintained at -50 °C. Stirring was continued for additional 15 min, then a solution of 3-benzyl-1-(4-hydroxybutyl)pyrrolidine-2,5-dione (5.00 g, 19.1 mmol) in 40 mL of CH₂Cl₂ was added while keeping the temperature at -

50 °C. The reaction mixture was stirred for another 1 h at -50 °C, and triethylamine (14.7 mL, 105.2 mmol) was added. The mixture is allowed to warm to room temperature, and 200 mL of water was added and stirred for 30 min. The organic layer was separated and washed with water, dried over anhydrous Na₂SO₄, filtered and concentrated in vacuo. The crude 4-(3-benzyl-2,5-dioxopyrrolidin-1-yl)butanal was obtained as viscous oil in 99% yield (4.912 g) and was stored under nitrogen atmosphere and used without further purification for the next step.

Propane-1,3-diol (4.1 ml, 56.8 mmol) was added to a solution of 4-(3-benzyl-2,5-dioxopyrrolidin-1-yl)butanal (4.9 g, 18.9 mmol) and *p*-toluenesulphonic acid monohydrate (360 mg, 1.8 mmol) in toluene (200 mL) at room temperature. The solution was stirred for 12 h, then diluted with ethyl acetate (200 mL) and washed with saturated aq. NaHCO₃ (60 mL). The layers were separated and the aqueous layer was extracted with ethyl acetate (4 x 50 mL). The combined organic layers were dried over anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue was purified through column chromatography using silica gel and ethyl acetate : hexane as eluent (1:1) to give 1-(3-(1,3-dioxan-2-yl)propyl)-3-benzylpyrrolidine-2,5-dione as colorless liquid.

3.6. General procedure to prepare unsymmetrical succinimide derivatives of substituted tryptamines

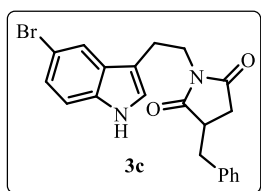
A mixture of substituted phenylhydrazine hydrochloride (1.5 mmol) and 1-(3-(1,3-dioxan-2-yl)propyl)-3-benzylpyrrolidine-2,5-dione (1.5 mmol) in 100 mL of 4% H₂SO₄ was heated at reflux for 12 h. The reaction mixture was cooled to room temperature and treated with saturated aq. NaHCO₃. The tryptamine product was extracted with CH₂Cl₂ (4 x 50 mL). The combined organic layer was dried over anhydrous Na₂SO₄, filtered and the solvent was evaporated under reduced pressure. The residue was purified through neutral alumina column chromatography using ethyl acetate : hexane (1:4) as eluent to give **3b-3g** in pure form.



Following the general procedure, (4-fluorophenyl)hydrazine hydrochloride (258 mg, 1.6 mmol) and 1-(3-(1,3-dioxan-2-yl)propyl)-3-benzylpyrrolidine-2,5-dione (504 g, 1.6 mmol) furnished 3-benzyl-1-(2-(5-fluoro-1*H*-indol-3-yl)ethyl)pyrrolidine-2,5-dione **3b** as viscous liquid.

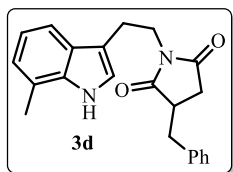
IR (KBr, cm⁻¹): 3352, 2927, 1771, 1694, 1487, 1404, 1261, 1154, 937; ¹H NMR (400 MHz,

CDCl₃): δ 8.13 (br s, 1H), 7.30 (d, J = 2.0 Hz, 1H), 7.28-7.27 (m, 2H), 7.25-7.17 (m, 2H), 7.13-7.11 (m, 2H), 7.06 (s, 1H), 6.94 (td, J = 8.8, 2.4 Hz, 1H), 3.76 (t, J = 7.6 Hz, 2H), 3.12 (dd, J = 13.6, 4.4 Hz, 1H), 3.02 (sep, J = 4.4 Hz, 1H), 2.98-2.88 (m, 2H), 2.66-2.56 (m, 2H), 2.36 (dd, J = 18.4, 4.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 179.17, 176.37, 156.72 (d, J = 230.0 Hz, 1C), 138.10, 132.90, 128.96, 128.47, 127.31 (d, J = 9.0 Hz, 1C), 126.53, 125.22, 112.47 (d, J = 10.0 Hz, 1C), 110.81 (d, J = 4.0 Hz, 1C), 109.13 (d, J = 26.0 Hz, 1C), 102.61 (d, J = 22.0 Hz, 1C), 40.62, 38.50, 35.59, 33.15, 22.86; HRMS-ESI (m/z): [M+H]⁺ Found 351.1491 and calculated 351.1431 for C₂₁H₂₀FN₂O₂⁺.



Following the general procedure, (4-bromophenyl)hydrazine hydrochloride (352 mg, 1.6 mmol) and 1-(3-(1,3-dioxan-2-yl)propyl)-3-benzylpyrrolidine-2,5-dione (500 mg, 1.6 mmol) furnished 3-benzyl-1-(2-(5-bromo-1*H*-indol-3-yl)ethyl)pyrrolidine-2,5-dione **3c** as viscous liquid.

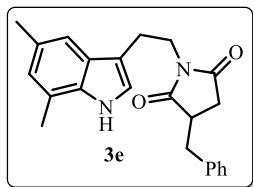
IR (KBr, cm⁻¹): 3431, 2929, 1770, 1698, 1446, 1402, 1159, 798; ¹H NMR (400 MHz, CDCl₃): δ 8.24 (br s, 1H), 7.71 (d, J = 2.4 Hz, 1H), 7.29-7.26 (m, 2H), 7.25-7.18 (m, 3H), 7.12-7.10 (m, 2H), 6.99 (d, J = 2.4 Hz, 1H), 3.75 (t, J = 7.6 Hz, 2H), 3.09 (dd, J = 13.6, 4.4 Hz, 1H), 3.03-2.88 (m, 3H), 2.61-2.53 (m, 2H), 2.33 (dd, J = 18.4, 4.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 179.33, 176.53, 137.31, 134.79, 129.51, 129.07, 128.88, 127.10, 125.00, 123.59, 121.24, 112.87, 112.86, 112.06, 41.30, 39.67, 36.45, 33.39, 23.00; HRMS-ESI (m/z): [M+H]⁺ Found 411.0689 and calculated 411.0630 for C₂₁H₂₀BrN₂O₂⁺.



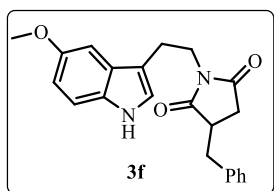
Following the general procedure, *o*-tolylhydrazine hydrochloride (250 mg, 1.6 mmol) and 1-(3-(1,3-dioxan-2-yl)propyl)-3-benzylpyrrolidine-2,5-dione (500 mg, 1.6 mmol) furnished 3-benzyl-1-(2-(7-methyl-1*H*-indol-3-yl)ethyl)pyrrolidine-2,5-dione **3d** as viscous liquid. IR (KBr, cm⁻¹): 3367,

2923, 2854, 1771, 1700, 1496, 1403, 1343, 1160; ¹H NMR (400 MHz, CDCl₃): δ 8.02 (br s, 1H), 7.50 (d, J = 8.0 Hz, 1H), 7.28 (t, J = 1.2 Hz, 1H), 7.26-7.19 (m, 2H), 7.11-7.04 (m, 3H), 7.02 (d, J = 2.4 Hz, 1H), 7.00-6.98 (m, 1H), 3.79-3.76 (m, 2H), 3.09 (dd, J = 13.6, 4.4 Hz, 1H), 3.06-2.92 (m, 3H), 2.62-2.53 (m, 2H), 2.45 (s, 3H), 2.34 (dd, J = 18.4, 4.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 179.33, 176.51, 137.40, 135.85, 129.08, 128.88, 127.22, 127.09, 122.75, 122.02,

120.47, 119.88, 116.54, 112.74, 41.29, 39.67, 36.48, 33.42, 23.42, 16.67; HRMS-ESI (m/z): [M+H]⁺ Found 347.1743 and calculated 347.1681 for C₂₂H₂₃N₂O₂⁺.



Following the general procedure, (2,4-dimethylphenyl)hydrazine hydrochloride (276 mg, 1.6 mmol) and 1-(3-(1,3-dioxan-2-yl)propyl)-3-benzylpyrrolidine-2,5-dione (507 mg, 1.6 mmol) furnished 3-benzyl-1-(2-(5,7-dimethyl-1*H*-indol-3-yl)ethyl)-pyrrolidine-2,5-dione **3e** as viscous liquid. IR (KBr, cm⁻¹): 3346, 2925, 2858, 1772, 1697, 1436, 1406, 1342, 1154; ¹H NMR (400 MHz, CDCl₃): δ 7.84 (br s, 1H), 7.29-7.26 (m, 2H), 7.24-7.20 (m, 2H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.01 (s, 1H), 6.84 (s, 1H), 3.77 (t, *J* = 7.6 Hz, 2H), 3.12 (dd, *J* = 14.4, 4.4 Hz, 1H), 3.03-2.91 (m, 3H), 2.66-2.55 (m, 2H), 2.44 (s, 3H), 2.43 (s, 3H), 2.34 (dd, *J* = 18.4, 4.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 179.32, 176.48, 137.49, 134.25, 129.15, 129.09, 128.92, 127.53, 127.12, 124.61, 122.14, 120.12, 116.14, 112.41, 41.35, 39.75, 36.58, 33.50, 23.46, 21.62, 16.64; HRMS-ESI (m/z): [M+H]⁺ Found 361.1901 and calculated 361.1838 for C₂₃H₂₅N₂O₂⁺.

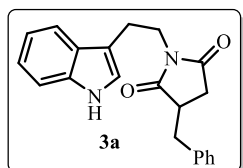


Following the general procedure, (4-methoxyphenyl)hydrazine hydrochloride (276 mg, 1.6 mmol) and 1-(3-(1,3-dioxan-2-yl)propyl)-3-benzylpyrrolidine-2,5-dione (502 mg, 1.6 mmol) furnished 3-benzyl-1-(2-(5-methoxy-1*H*-indol-3-yl)ethyl)pyrrolidine-2,5-dione **3f** as viscous liquid. IR (KBr, cm⁻¹): 3432, 2926, 1760, 1699, 1484, 1445, 1158, 1028; ¹H NMR (400 MHz, CDCl₃): δ 7.95 (br s, 1H), 7.30-7.28 (m, 2H), 7.25-7.20 (m, 2H), 7.13-7.10 (m, 3H), 7.01 (d, *J* = 2.0 Hz, 1H), 6.86 (dd, *J* = 8.8, 2.4 Hz, 1H), 3.90 (s, 3H), 3.78 (t, *J* = 7.6 Hz, 2H), 3.11 (dd, *J* = 13.6, 4.4 Hz, 1H), 3.07-2.90 (m, 3H), 2.63-2.56 (m, 2H), 2.35 (dd, *J* = 18.4, 4.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 179.35, 176.57, 154.25, 137.38, 131.35, 129.10, 128.91, 128.10, 127.12, 123.03, 112.60, 112.08, 112.01, 100.49, 56.01, 41.34, 39.45, 36.55, 33.43, 23.32; HRMS-ESI (m/z): [M+H]⁺ Found 363.1691 and calculated 363.1630 for C₂₂H₂₃N₂O₃⁺.

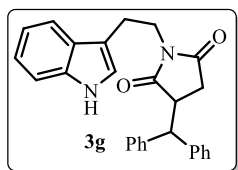
3.7 General procedure to prepare unsymmetrical imide derivatives of tryptamines

A mixture of anhydride (4 mmol) and tryptamine (4 mmol) were heated at 170 °C with vigorous stirring under nitrogen atmosphere. After 6 h the reaction mixture was cooled to 80 °C and it was poured to 100 mL of ice-cold water. The product was extracted with CHCl₃ (4 x 100

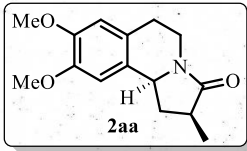
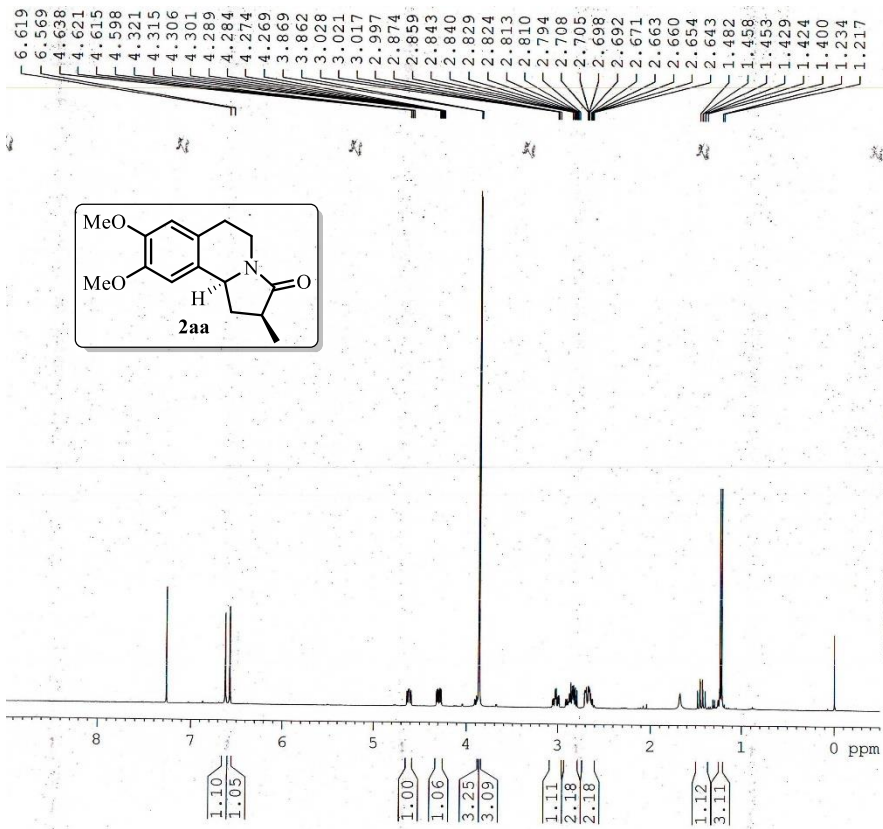
mL), and the combined organic layer was washed with 5% NaHCO₃ solution (3 x 100 mL), and with water (3 x 100 mL). Organic layer was dried over anhydrous Na₂SO₄, filtered and the solvent was removed under vacuum to give the mixture, which was purified through neutral alumina column chromatography using ethyl acetate : hexane as eluent (2:5) to give imides **3a** and **3g** in pure form.



Following the general procedure, 3-benzylidihydrofuran-2,5-dione (0.83 g, 4.3 mmol) and tryptamine (700 mg, 4.3 mmol) furnished 1-(2-(1*H*-indol-3-yl)ethyl)-3-benzylpyrrolidine-2,5-dione **3a** as viscous liquid. 1.06 g, 73% yield, IR (KBr, cm⁻¹): 3382, 2929, 2855, 1770, 1696, 1446, 1349, 1158; ¹H NMR (400 MHz, DMSO-*d*₆): δ 10.86 (br s, 1H), 7.54 (d, *J* = 7.6 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.29 (t, *J* = 8.0 Hz, 2H), 7.23-7.17 (m, 4H), 7.09 (t, *J* = 7.2 Hz, 1H), 7.02 (t, *J* = 7.2 Hz, 1H), 3.61 (t, *J* = 7.6 Hz, 2H), 3.13 (sep, *J* = 4.4 Hz, 1H), 3.01 (dd, *J* = 13.6, 4.4 Hz, 1H), 2.91-2.78 (m, 2H), 2.71-2.60 (m, 2H), 2.33 (dd, *J* = 18.0, 4.4 Hz, 1H); ¹³C NMR (100 MHz, DMSO-*d*₆): δ 179.15, 176.36, 138.09, 136.24, 128.97, 128.44, 127.12, 126.51, 122.98, 121.02, 118.42, 117.95, 111.50, 110.53, 40.60, 38.74, 35.56, 33.14, 22.98; HRMS-ESI (*m/z*): [*M*+*H*]⁺ Found 333.1589 and calculated 333.1525 for C₂₁H₂₁N₂O₂⁺.



Following the general procedure, 3-benzhydryldihydrofuran-2,5-dione (270 mg, 1 mmol) and tryptamine (162 mg, 1 mmol) furnished 1-(2-(1*H*-indol-3-yl)ethyl)-3-benzhydrylpyrrolidine-2,5-dione **3g** as viscous liquid. 306 mg, 74% yield, IR (KBr, cm⁻¹): 3425, 2928, 2826, 1668, 1598, 1442, 1419, 1358, 1306; ¹H NMR (400 MHz, CDCl₃): δ 7.98 (br s, 1H), 7.66 (dd, *J* = 8.0 Hz, 1H), 7.37-7.31 (m, 3H), 7.23-7.13 (m, 8H), 7.06 (d, *J* = 7.2 Hz, 2H), 6.99 (s, 1H), 4.71 (d, *J* = 4.8 Hz, 1H), 3.70 (t, *J* = 8.0 Hz, 2H), 3.60 (p, *J* = 4.8 Hz, 1H), 2.96-2.77 (m, 3H), 2.62 (dd, *J* = 18.0, 4.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 178.81, 176.18, 141.68, 140.06, 136.28, 129.18, 128.80, 128.78, 128.22, 127.56, 127.44, 127.05, 122.30, 122.15, 119.69, 118.93, 112.38, 111.28, 50.75, 43.94, 39.47, 32.43, 23.29; HRMS-ESI (*m/z*): [*M*+*H*]⁺ Found 409.1898 and calculated 409.1838 for C₂₇H₂₅N₂O₂⁺.

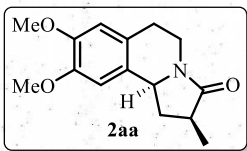
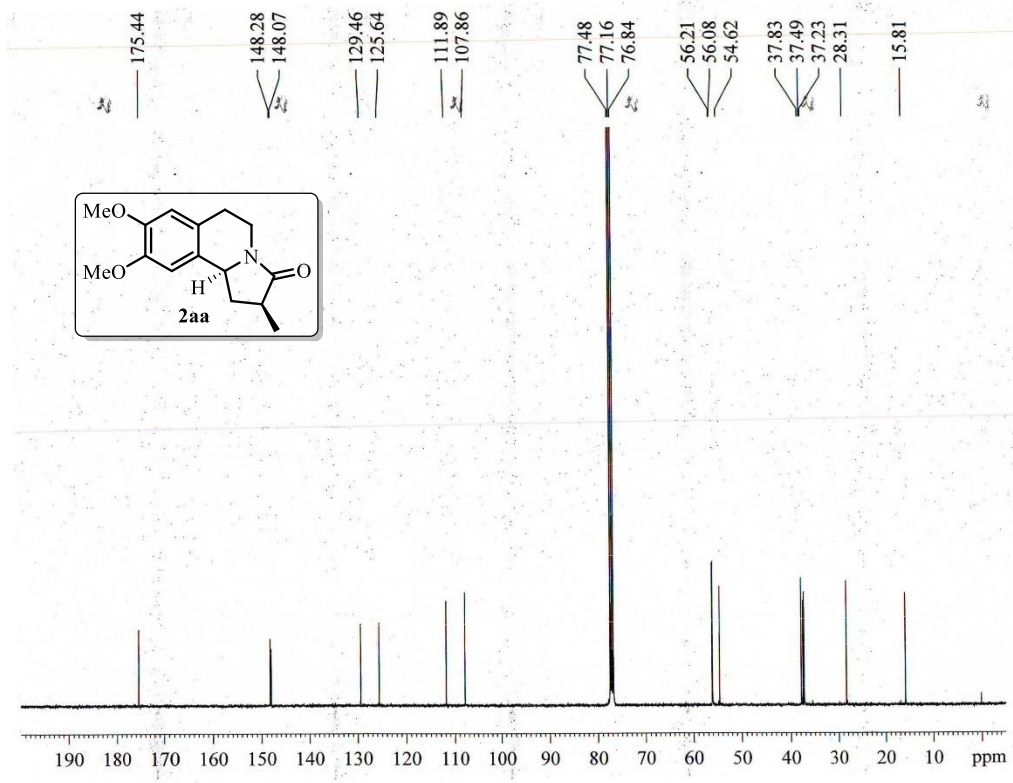


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 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
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 TE 297.7 K
 D1 1.00000000 sec
 TDO 1

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F2 - Processing parameters
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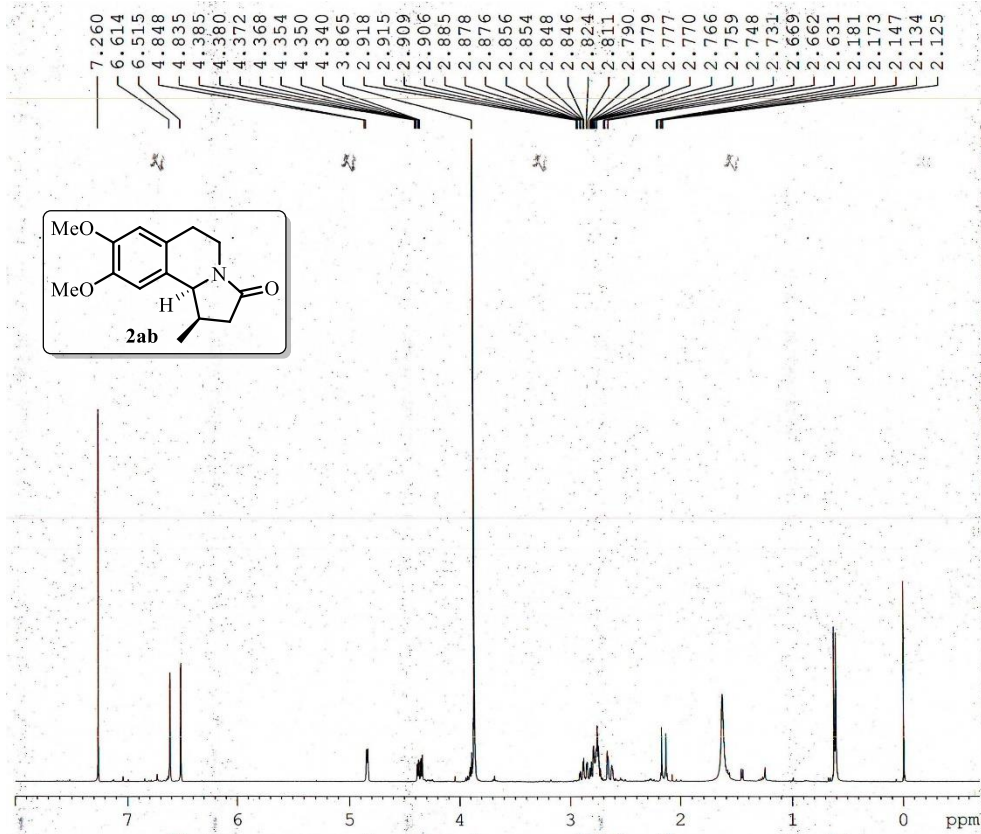
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 AQ 1.3631988 sec
 RG 25.4
 DW 20.800 usec
 DE 6.00 usec
 TE 298.5 K
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 DELTA 1.89999998 sec
 TDO 1

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 P1 9.15 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
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 NUC2 1H
 PCPD2 90.00 usec
 PL12 14.90 dB
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F2 - Processing parameters
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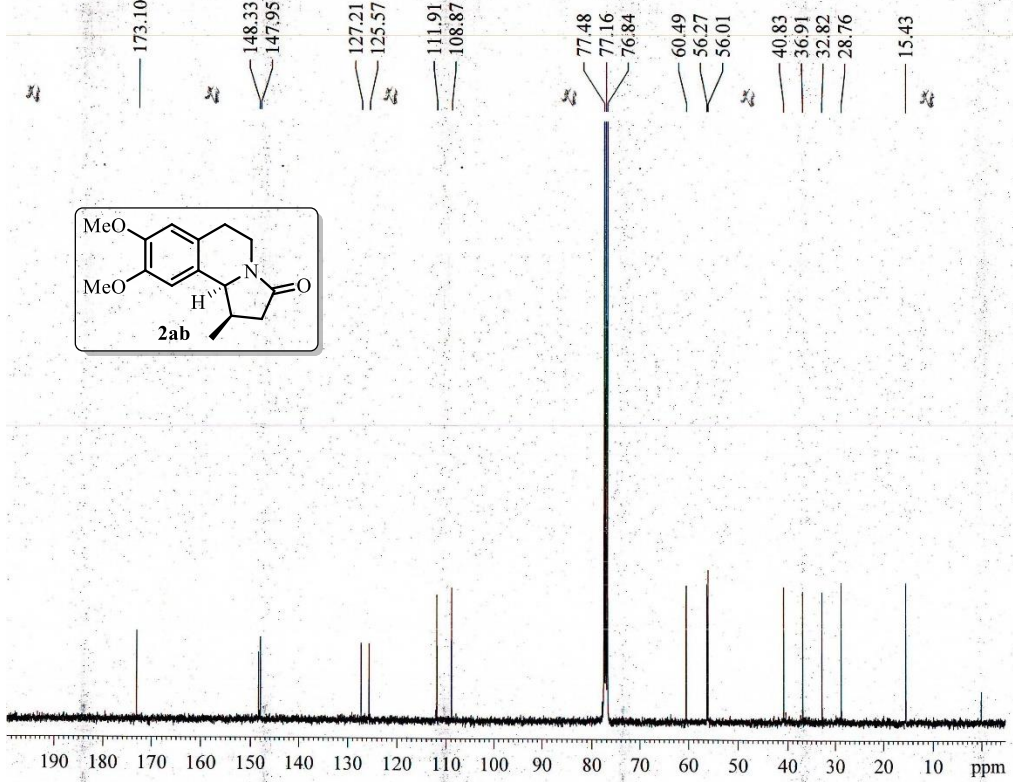


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 PULPROG zg30
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 SOLVENT CDC13
 NS 16
 DS 2
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 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 203
 DW 60.800 usec
 DE 6.00 usec
 TE 297.8 K
 D1 1.0000000 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 1H
 P1 11.42 usec
 PL1 -3.00 dB
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F2 - Processing parameters
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 WDW EM
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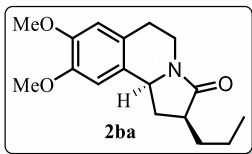
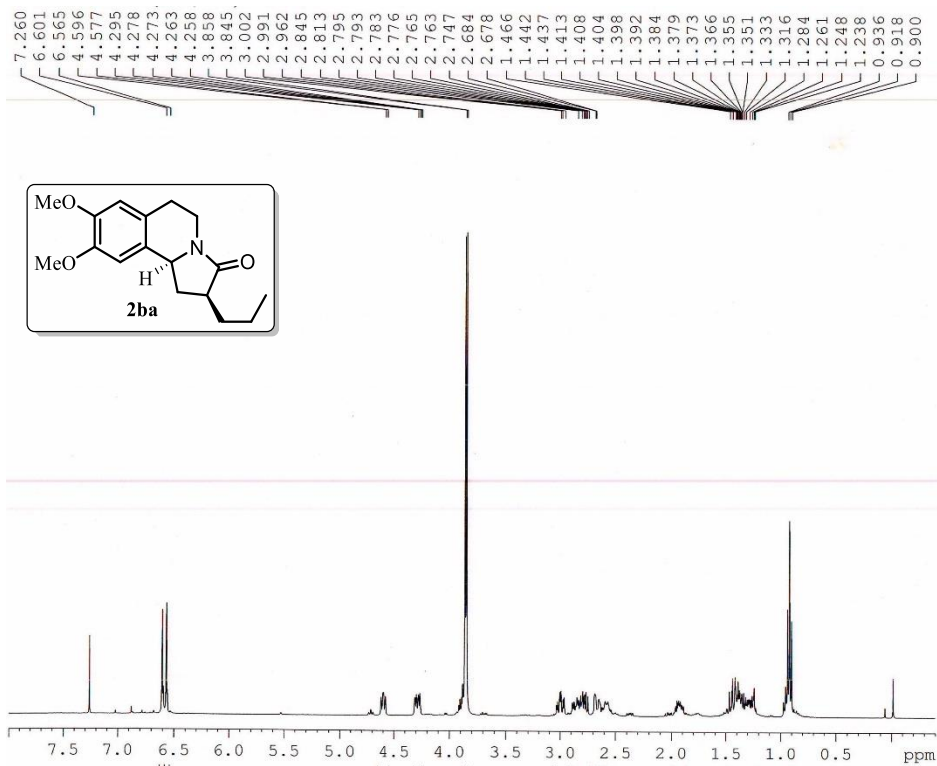
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 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 36
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 DE 6.00 usec
 TE 298.7 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 TDO 1

==== CHANNEL f1 =====
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 P1 9.15 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

==== CHANNEL f2 =====
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 NUC2 1H
 PCPD2 90.00 usec
 PL12 14.90 dB
 PL13 14.90 dB
 PL2 -3.00 dB
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F2 - Processing parameters
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 PC 1.40

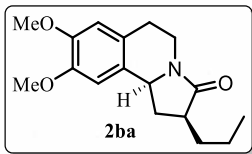
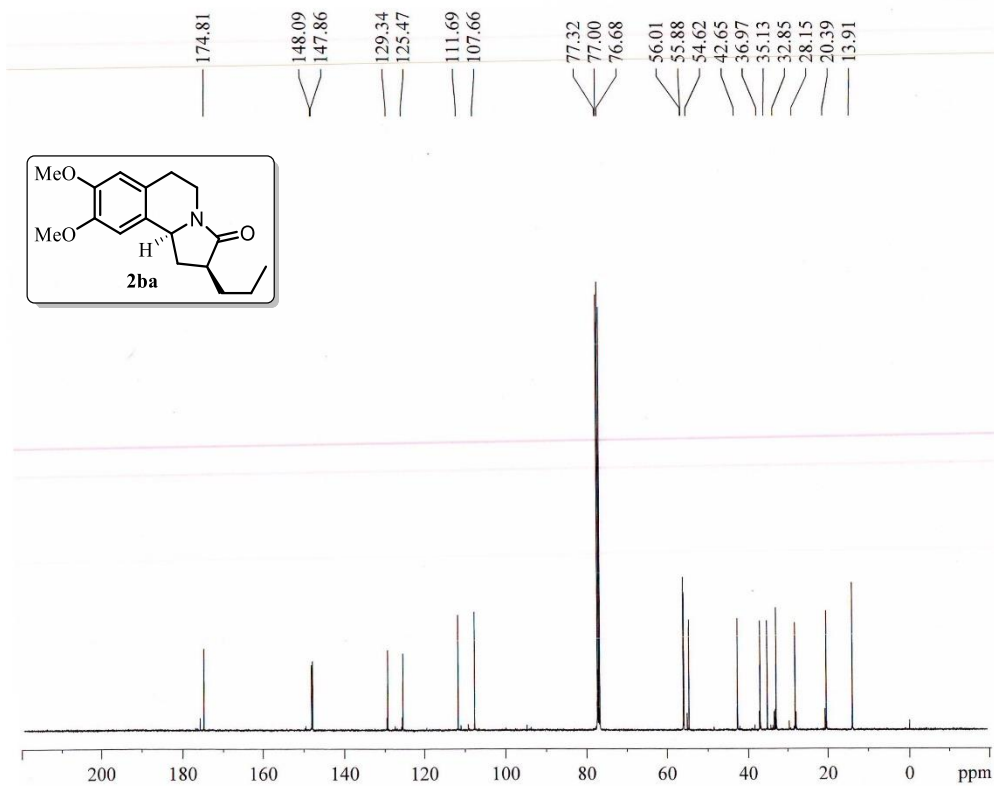


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 PULPROG zg30
 TD 65536
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 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 80.6
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 TE 296.8 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
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 PL1 -3.00 dB
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F2 - Processing parameters
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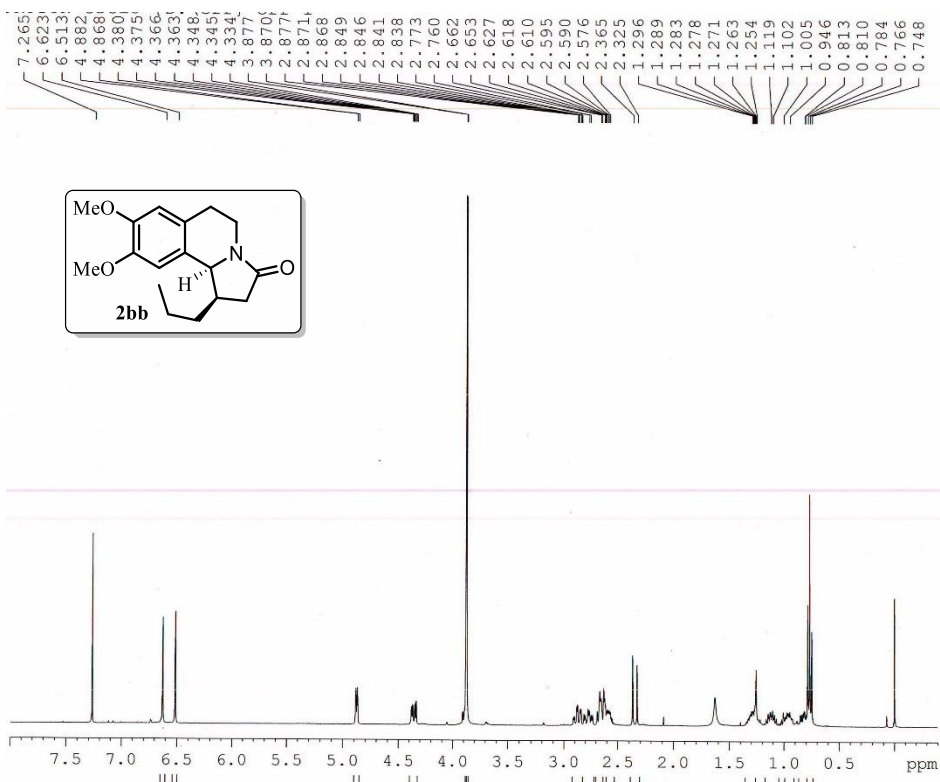
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 NS 5000
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 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 32
 DW 20.800 usec
 DE 6.00 usec
 TE 297.7 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.15 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 FCFD2 90.00 usec
 PL12 14.90 dB
 PL13 14.90 dB
 PL2 -3.00 dB
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F2 - Processing parameters
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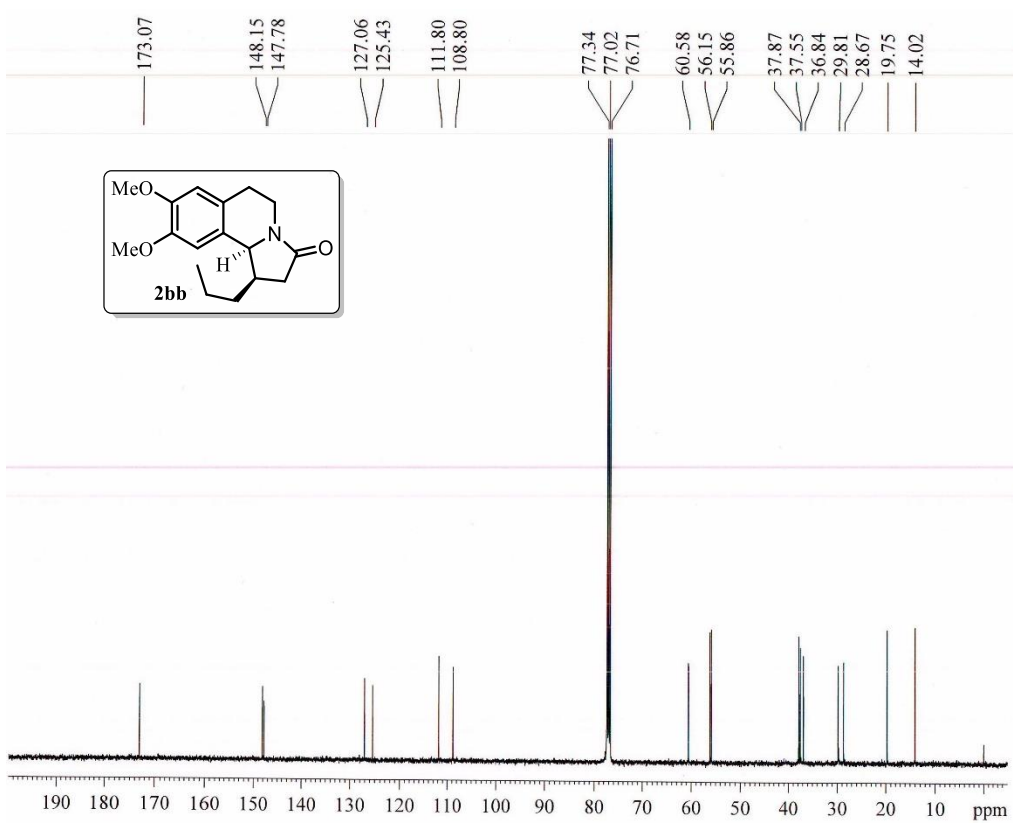


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 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 203
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 DE 6.00 usec
 TE 296.3 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
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 P1 11.42 usec
 PL1 -3.00 dB
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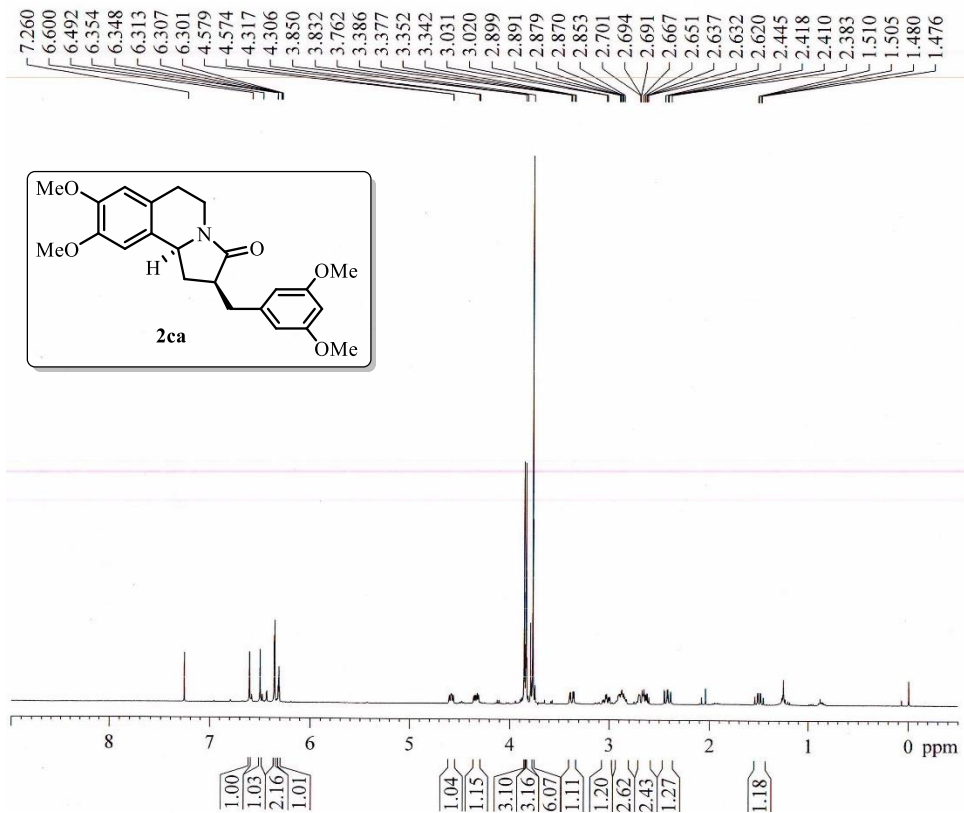
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 TD 65536
 SOLVENT CDCl3
 NS 7000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 32
 DW 20.800 usec
 DE 6.00 usec
 TE 298.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
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 P1 9.15 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 FCPD2 90.00 usec
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 PL2 -3.00 dB
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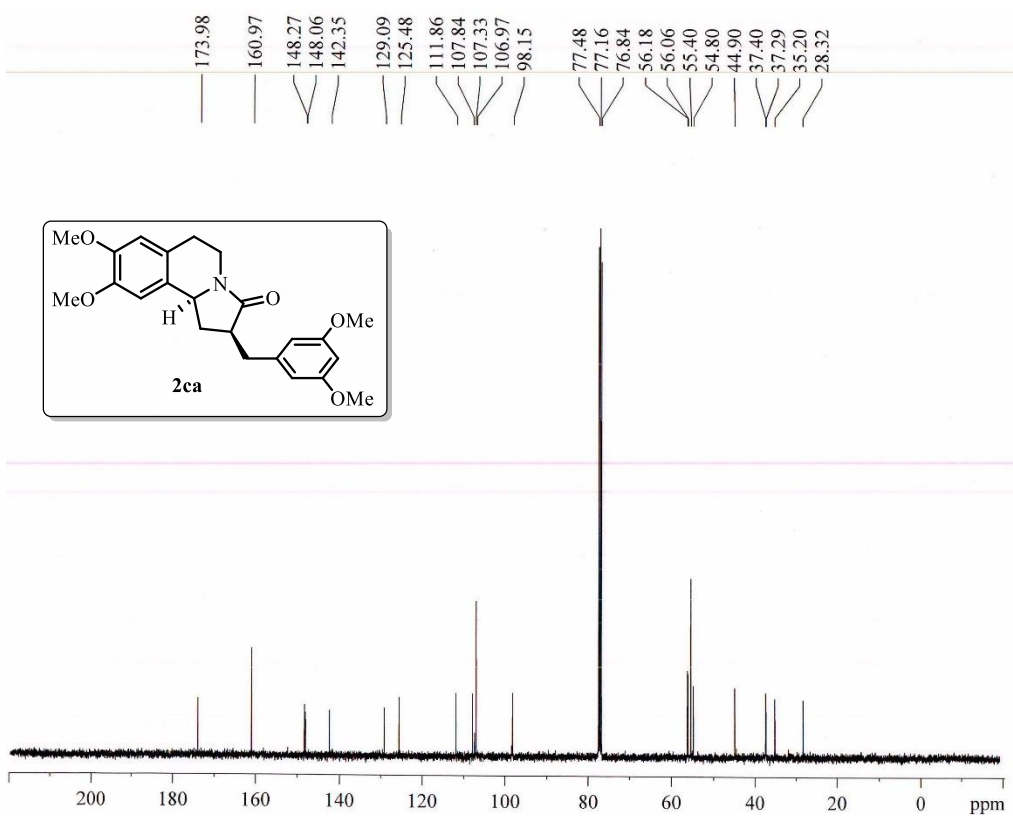
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NS       4
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PL1     -3.00 dB
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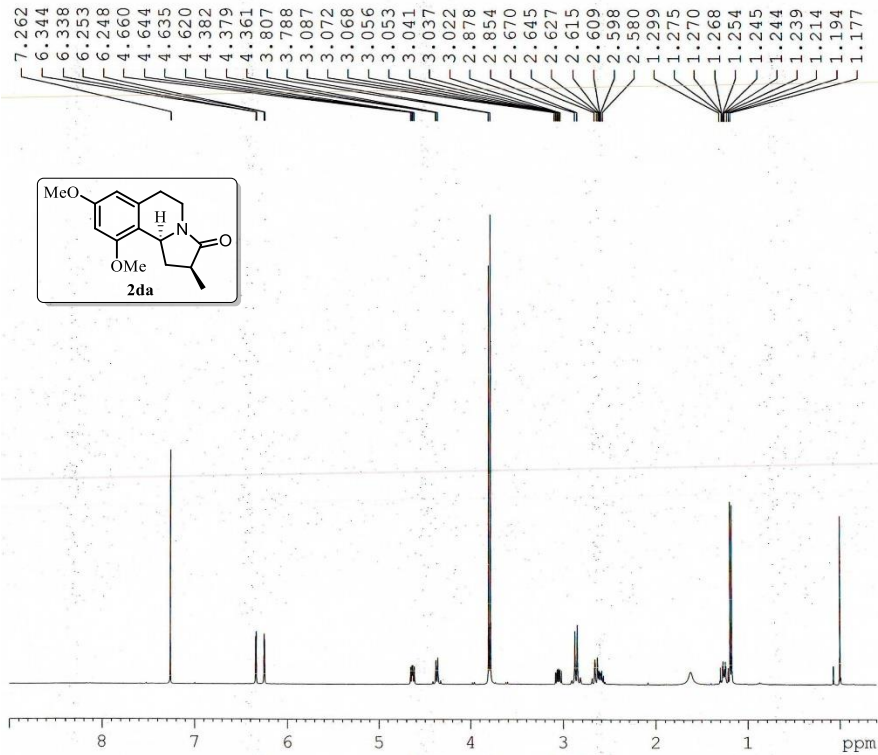
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===== CHANNEL f1 =====
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PL1      0.00 dB
SFO1    100.6228298 MHz

===== CHANNEL f2 =====
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PCPD2    90.00 usec
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PL2      -3.00 dB
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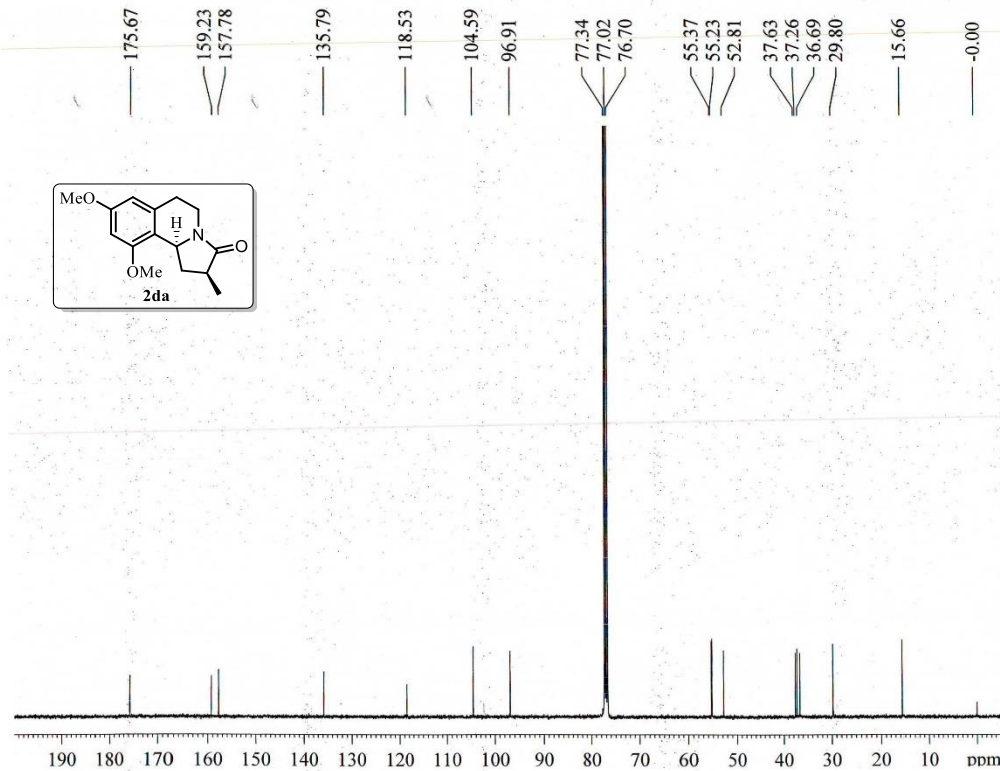


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 PROCNO 1

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 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 2
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 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 228
 DW 60.800 usec
 DE 6.00 usec
 TE 295.7 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 =====
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 P1 11.42 usec
 PL1 -3.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
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 SF 400.1300018 MHz
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Current Data Parameters
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 EXPNO 2
 PROCNO 1

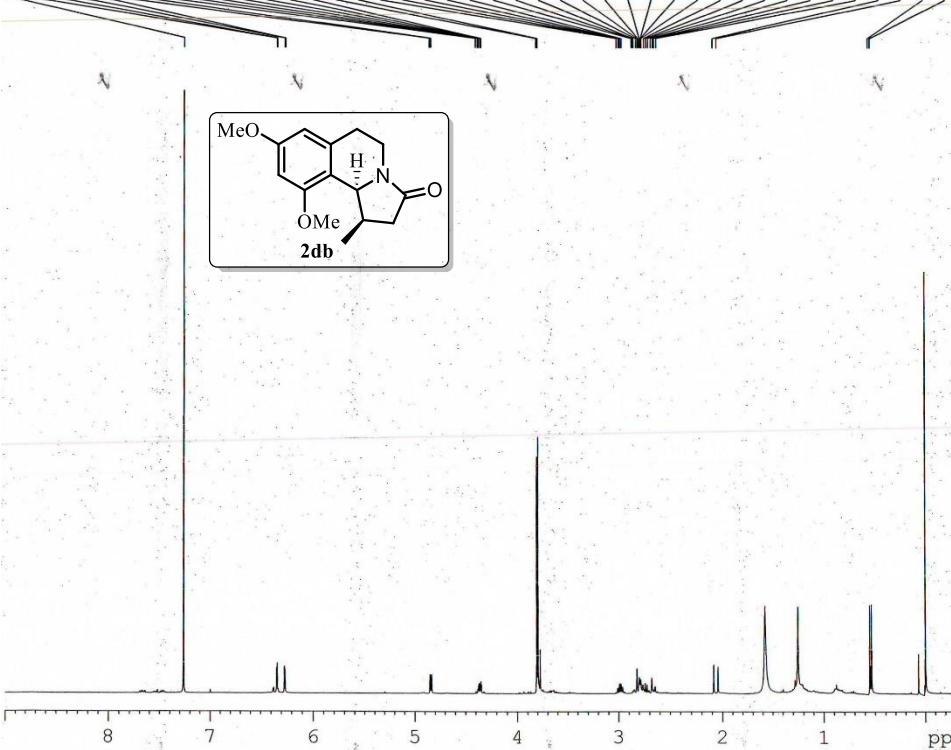
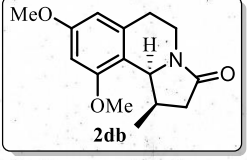
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 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 25.4
 DW 20.800 usec
 DE 6.00 usec
 TE 296.8 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

==== CHANNEL f1 =====
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 P1 9.15 usec
 PL1 0.00 dB
 SF01 100.6228298 MHz

==== CHANNEL f2 =====
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 PL13 14.90 dB
 PL2 -3.00 dB
 SF02 400.1316005 MHz

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 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

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4.352
4.348
3.810
3.797
3.011
2.993
2.979
2.975
2.961
2.865
2.854
2.847
2.822
2.807
2.798
2.785
2.776
2.773
2.766
2.744
2.725
2.705
2.679
2.659
2.646
2.625
2.045
0.552
0.535



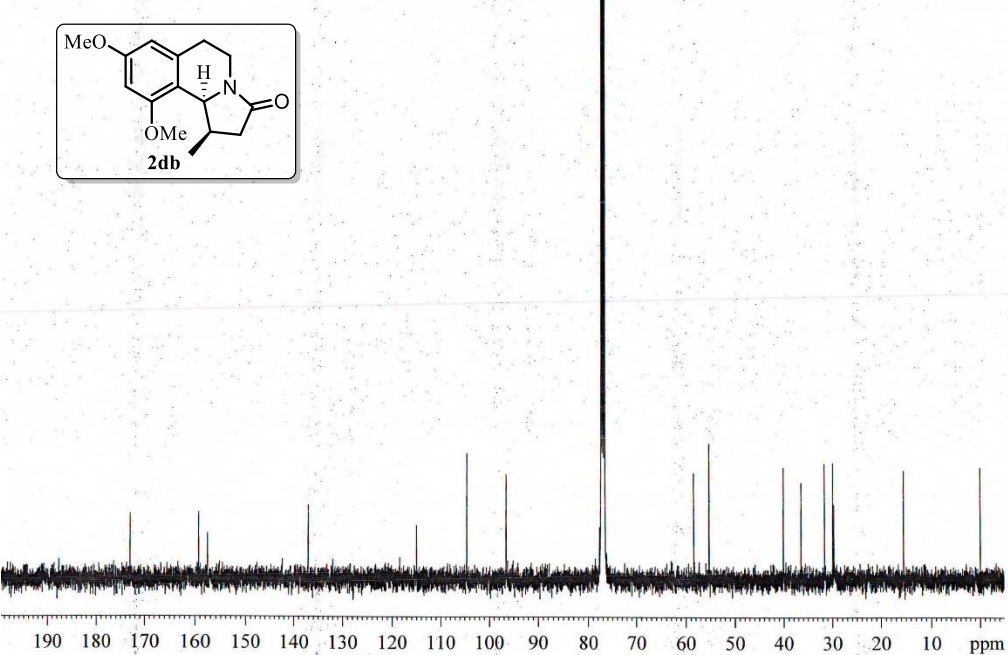
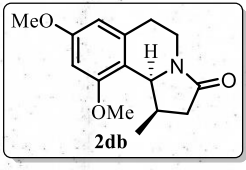
Current Data Parameters
NAME JS-3,5-MECY-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120624
Time 23.01
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 228
DW 60.800 usec
DE 6.00 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 11.42 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300025 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

173.06
159.31
157.60
137.07
115.01
104.59
96.53
77.31
76.99
76.67
58.43
55.30
55.26
40.12
36.45
31.62
29.92
29.68
15.48
-0.03



Current Data Parameters
NAME JS-3,5-MECY-2
EXPNO 2
PROCNO 1

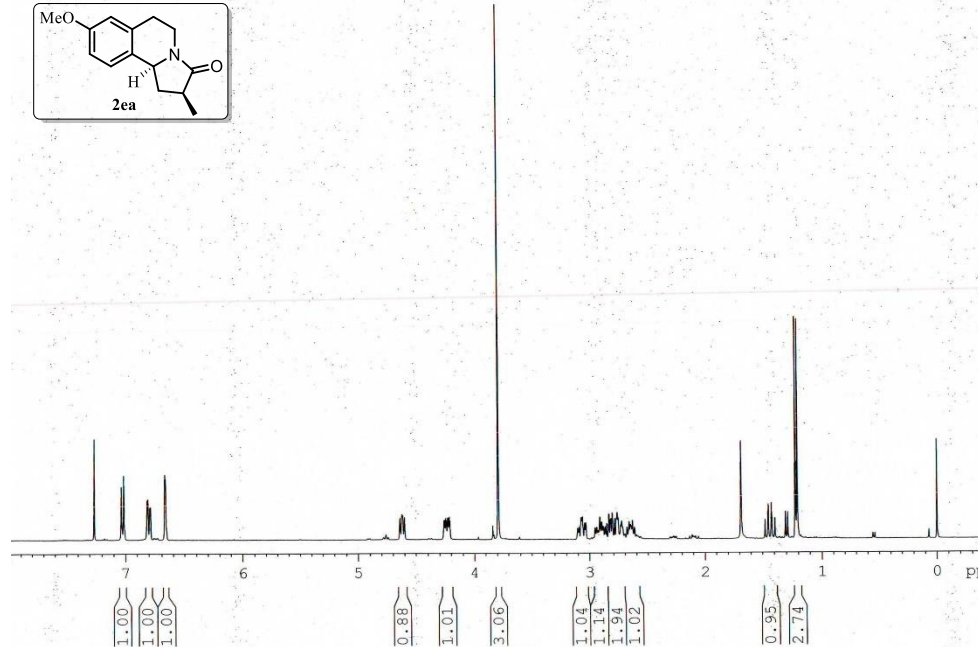
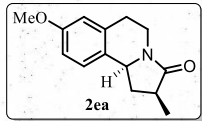
F2 - Acquisition Parameters
Date_ 20120625
Time 8.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 10000
DS 4
SWH 24038.463 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 32
DW 20.800 usec
DE 6.00 usec
TE 295.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.15 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 14.90 dB
PL13 14.90 dB
PL2 -3.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

7.2600
7.0346
7.0134
6.8139
6.8080
6.7927
6.7867
6.6718
6.6658
4.6402
4.6234
4.6168
4.6001
4.2621
4.2551
4.2470
4.2403
4.2301
4.2233
4.2149
4.2085
3.7889
3.0739
3.0708
3.0661
3.0629
3.0596
3.0422
3.0395
3.0310
2.9111
2.8966
2.8845
2.8845
2.8557
2.8386
2.8255
2.8202
2.8091
2.7903
2.7755
2.7680
2.7583
2.7562
2.7285
2.7285
2.6633
2.6518
2.6459
2.6353
1.4747
1.4500
1.4459
1.4213
1.4167
1.3925
1.2227
1.2051



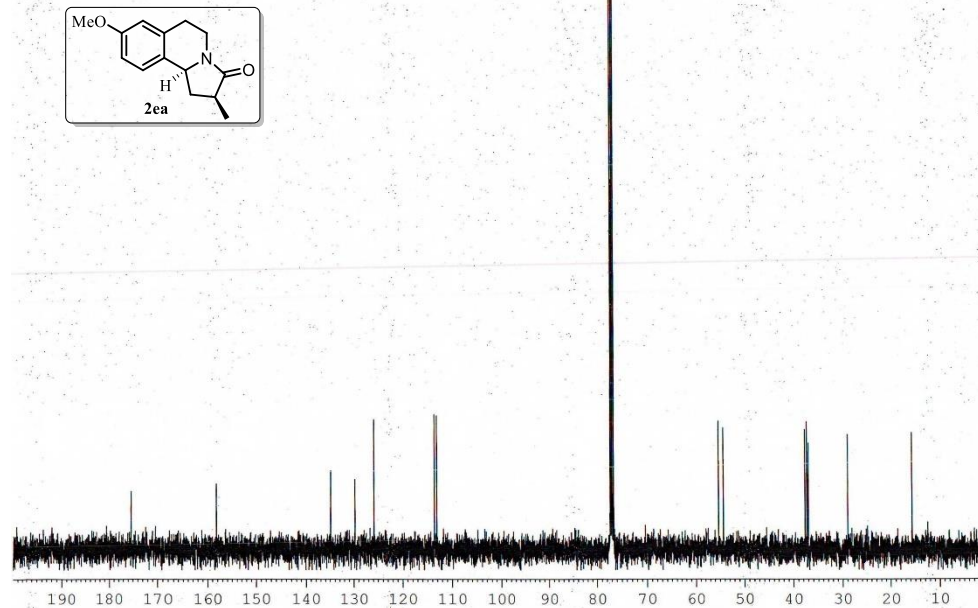
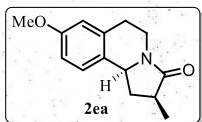
Current Data Parameters
NAME JS-PV-17-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120221
Time 15.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 406
DW 60.800 usec
DE 6.00 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 -0.90 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

175.39
158.27
134.89
129.80
125.88
113.61
113.12
77.32
77.01
76.69
55.28
54.31
37.65
37.28
36.98
28.91
15.66



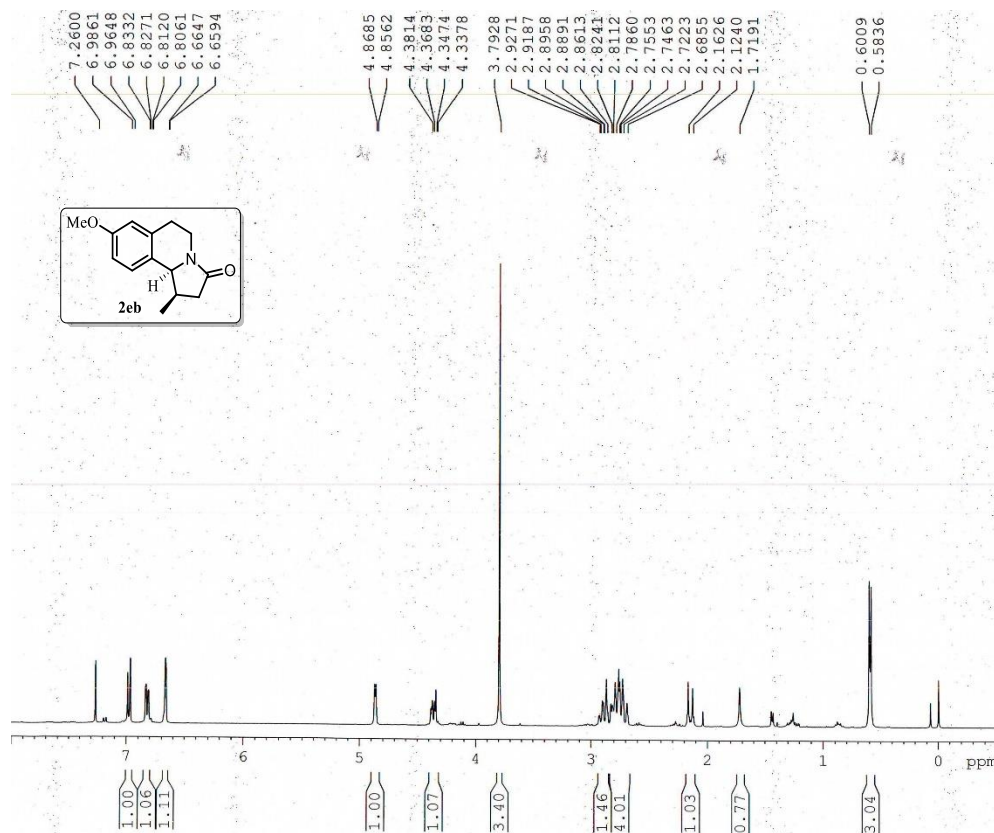
Current Data Parameters
NAME JS-PV-17-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120221
Time 15.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 175
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.60 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 15.60 dB
PL13 15.60 dB
PL2 -0.90 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

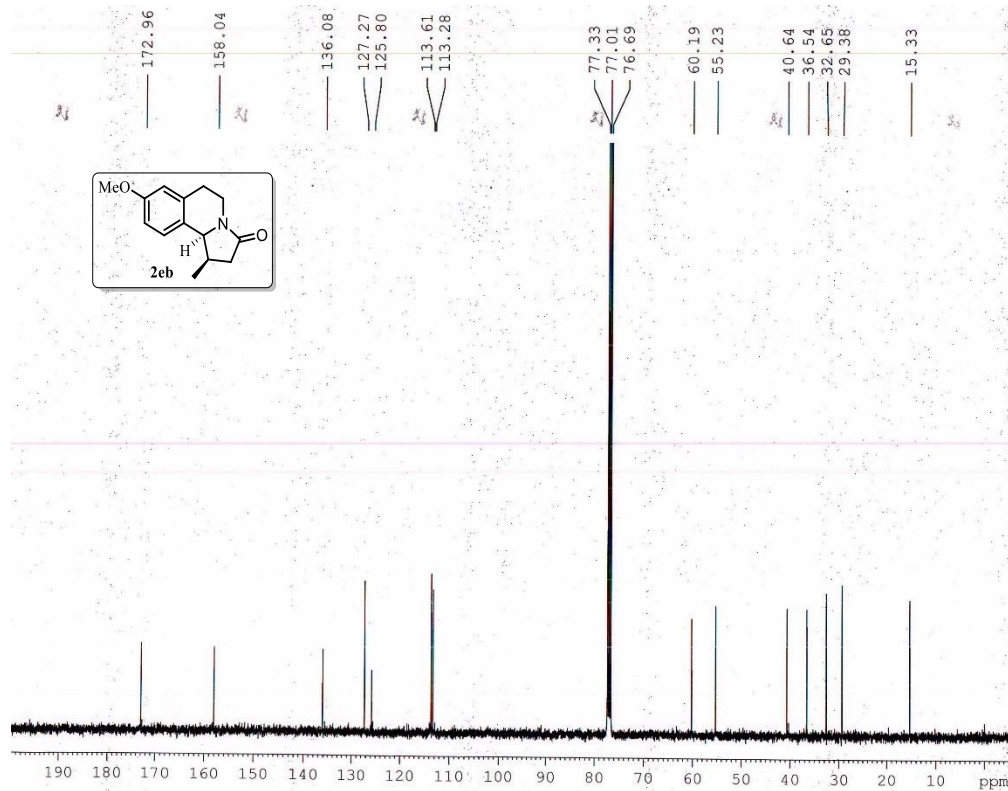


Current Data Parameters
 NAME JS-PV-17-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters %
 Date 20120221
 Time 12.16
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125493 Hz
 AQ 3.9846387 sec
 RG 256
 DW 60.800 usec
 DE 6.00 usec
 TE 297.6 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 -0.90 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300035 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



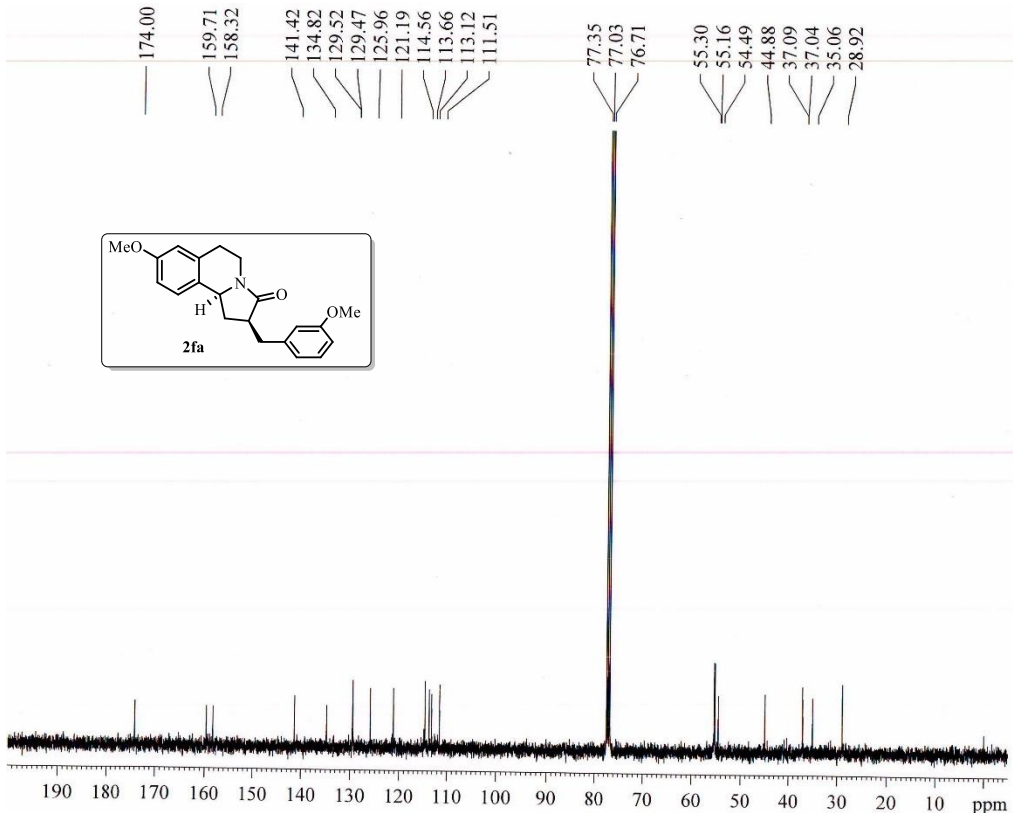
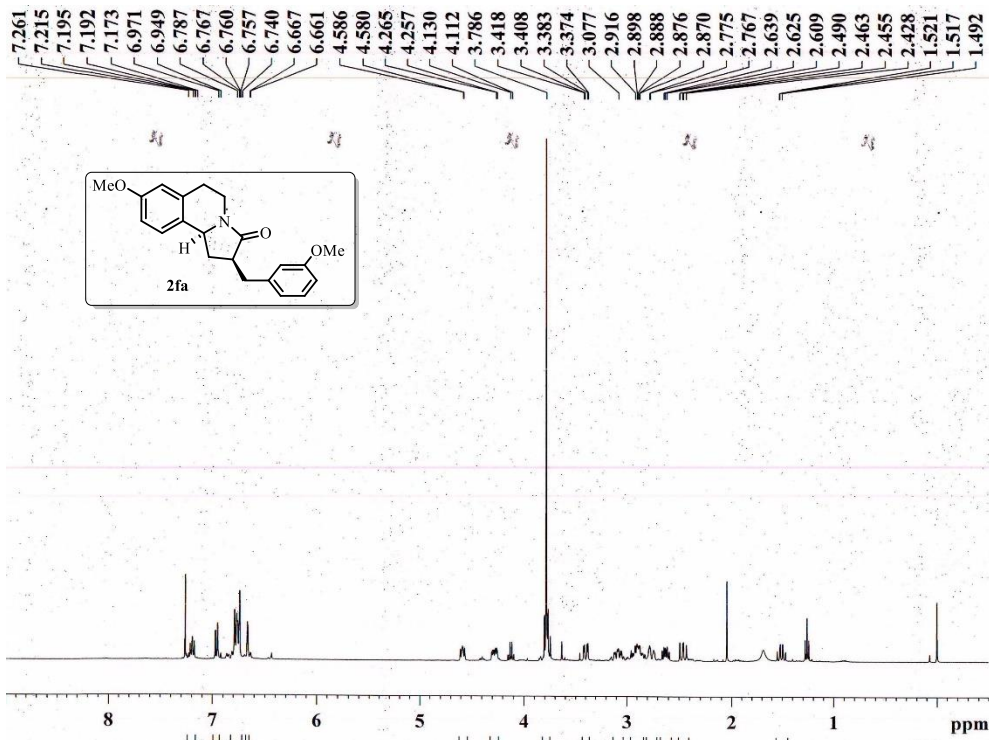
Current Data Parameters
 NAME JS-PV-17-2
 EXPNO 3
 PROCNO 1

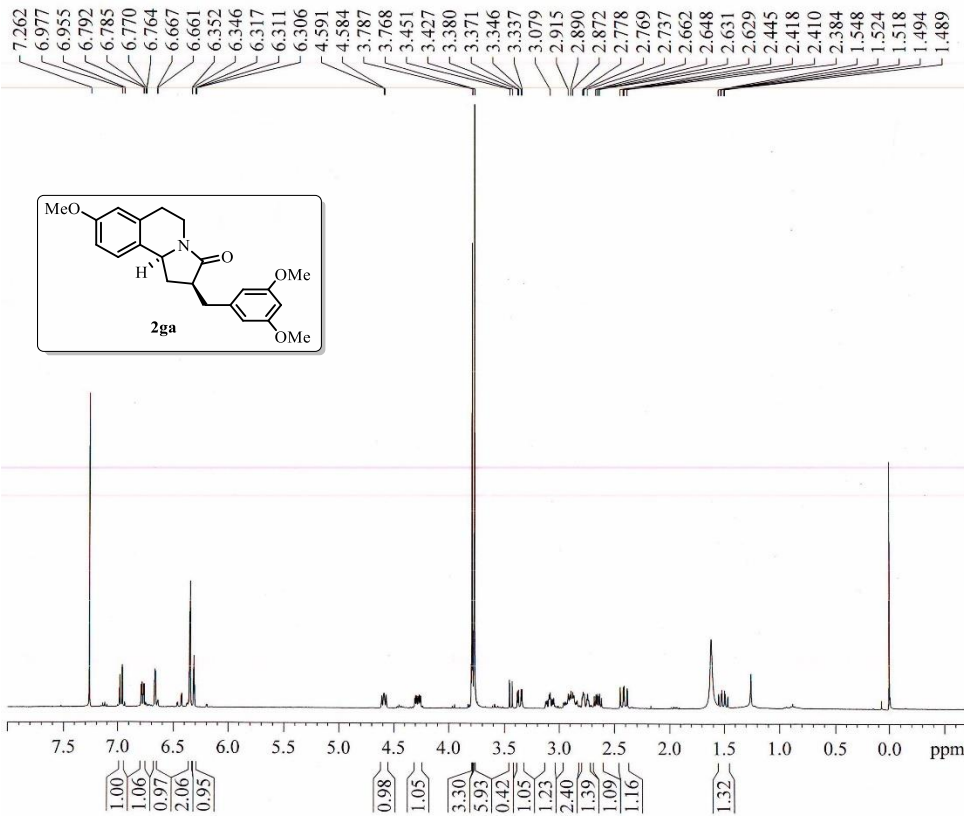
F2 - Acquisition Parameters
 Date 20120221
 Time 18.30
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 1150
 DW 20.800 usec
 DE 6.00 usec
 TE 298.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 -0.60 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL12 15.60 dB
 PL13 15.60 dB
 PL2 -0.90 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



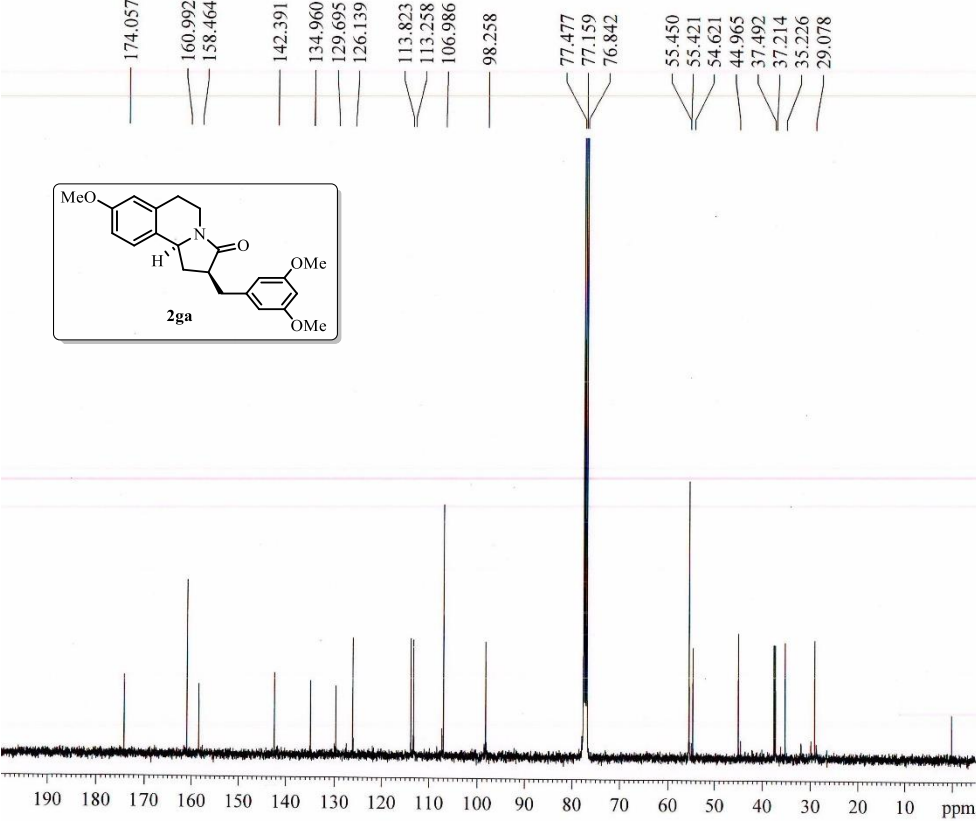


Current Data Parameters
 NAME JS-335BNCY-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120806
 Time 16.59
 INSTRUM spect
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 6536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 228
 DW 60.800 usec
 DE 6.00 usec
 TE 296.2 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.42 usec
 PL1 -3.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300025 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



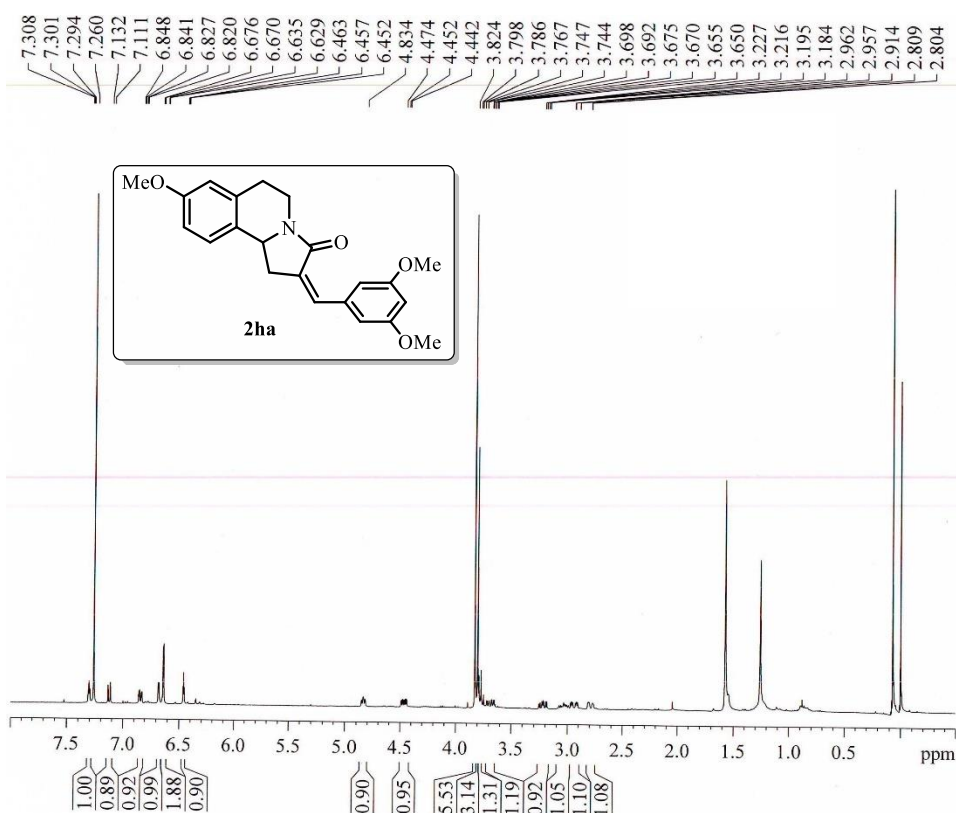
Current Data Parameters
 NAME JS-335BNCY-2
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120807
 Time 5.21
 INSTRUM spect
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 6536
 SOLVENT CDCl3
 NS 10000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 32
 DW 20.800 usec
 DE 6.00 usec
 TE 297.9 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.15 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL12 14.90 dB
 PL13 14.90 dB
 PL2 -3.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SE 100.6127520 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



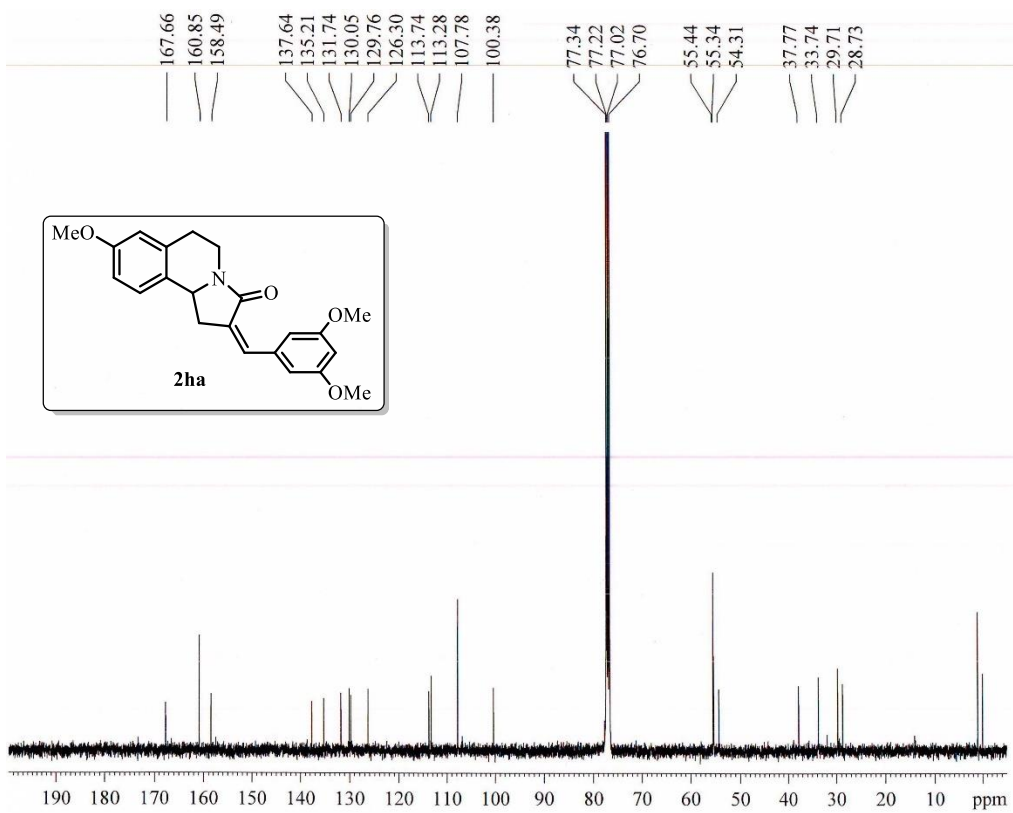
```

Current Data Parameters
NAME      JS-335BNCY-1B
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20120729
Time     19.46
INSTRUM  spect
PROBHD   5 mm DUL 13C-1
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        32
DS        2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        203
DW        60.800 usec
DE        6.00 usec
TE        297.3 K
D1        1.00000000 sec
TDO       1

===== CHANNEL f1 =====
NUC1      1H
P1        11.42 usec
PL1       -3.00 dB
SFO1      400.1324710 MHz

F2 - Processing parameters
SI        32768
SF        400.1300032 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



```

Current Data Parameters
NAME      JS-335BNCY-1B
EXPNO    3
PROCNO   1

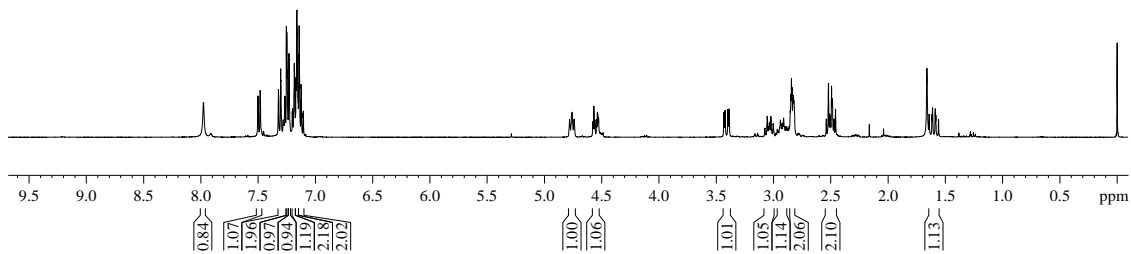
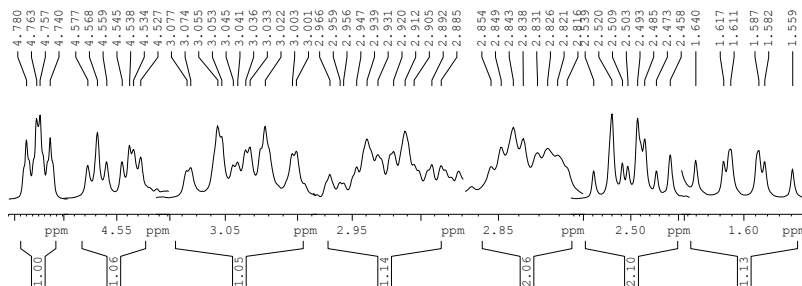
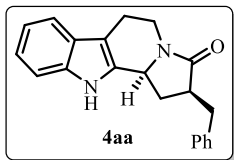
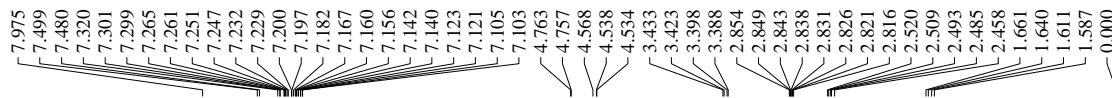
F2 - Acquisition Parameters
Date_    20120730
Time     0.27
INSTRUM  spect
PROBHD   5 mm DUL 13C-1
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        9825
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        32
DW        20.800 usec
DE        6.00 usec
TE        297.7 K
D1        2.00000000 sec
d11       0.03000000 sec
DELTA    1.89999998 sec
TDO       1

===== CHANNEL f1 =====
NUC1      13C
P1        9.15 usec
PL1       0.00 dB
SFO1      100.6228298 MHz

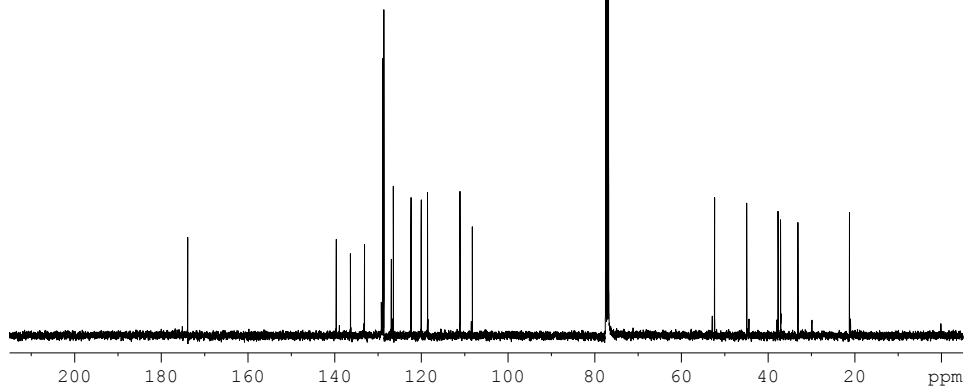
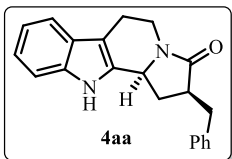
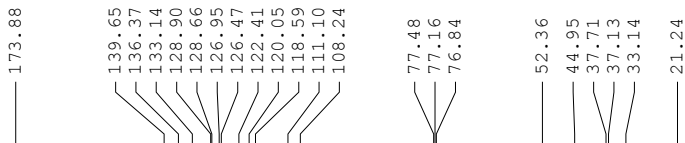
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    90.00 usec
PL12     14.90 dB
PL13     14.90 dB
PL2      -3.00 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        100.6127660 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

PROTON CDC13 {D:\CRR} KOPAL 1



C13CPD CDC13 {D:\HSP} KOPAL 1



Current Data Parameters
 NAME SMR-I-SI-1
 EXPNO 2
 PROCNO 1

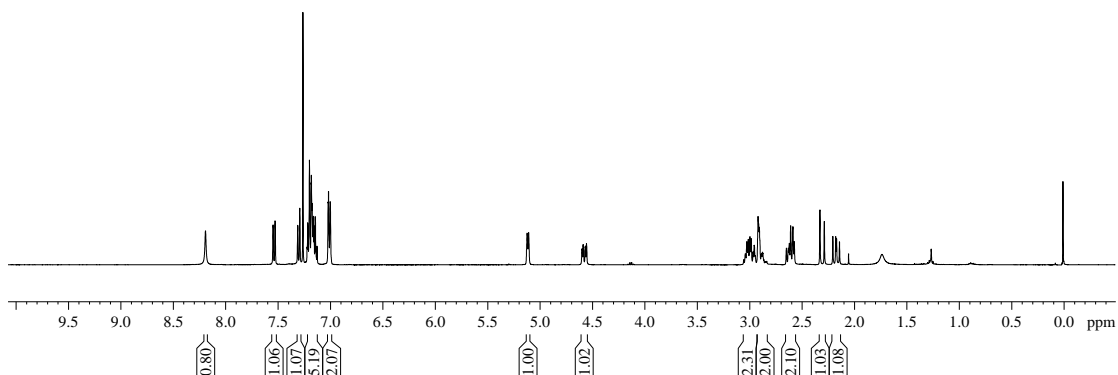
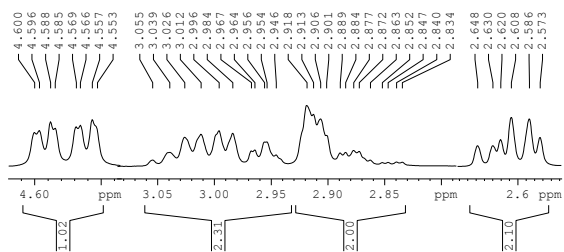
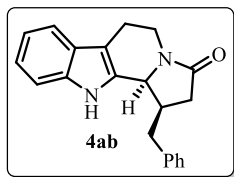
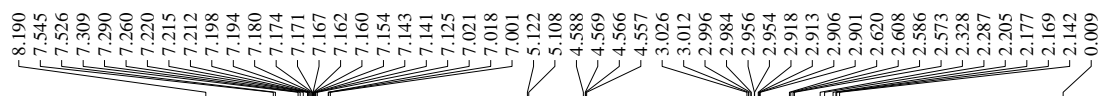
F2 - Acquisition Parameters
 Date_ 20140817
 Time 20.35
 INSTRUM spect
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1500
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 36
 DW 20.800 usec
 DE 6.00 usec
 TE 295.7 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.15 usec
 PL1 0.00 dB
 SFO1 100.628298 MHz

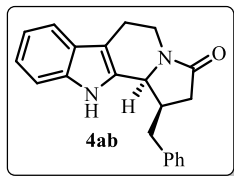
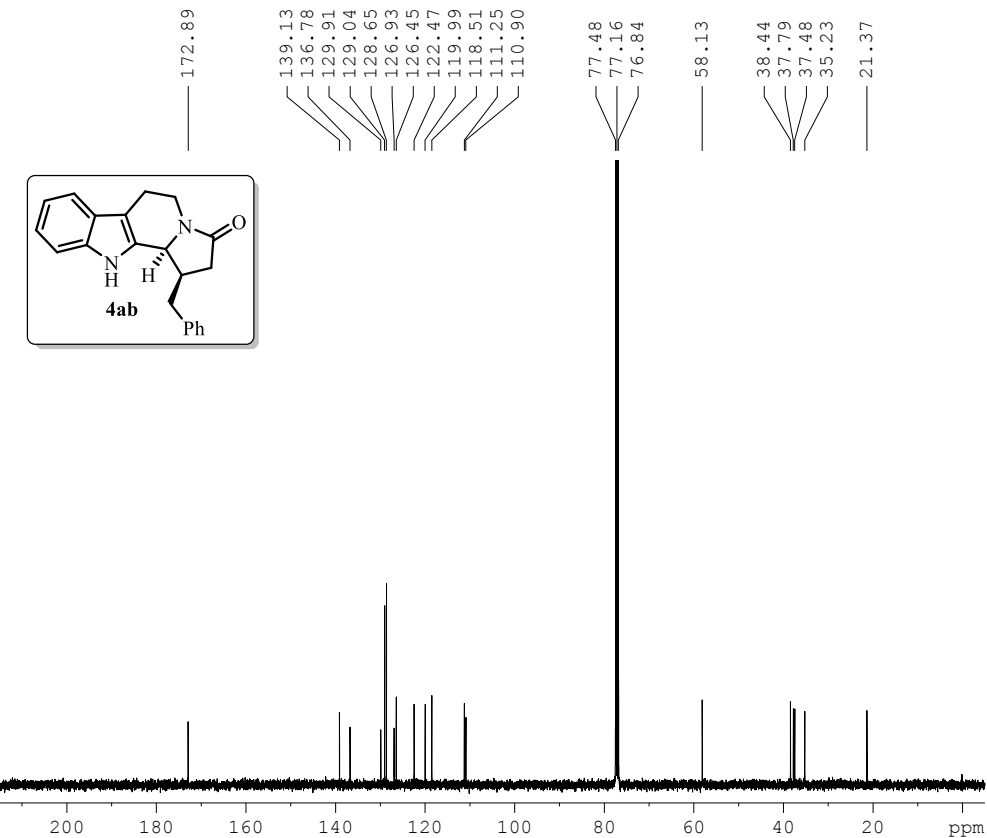
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL12 14.90 dB
 PL13 14.90 dB
 PL2 -3.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127560 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

PROTON CDC13 {D:\CRR} KOPAL 1



C13CPD CDC13 {D:\CRR} KOPAL 1



Current Data Parameters
NAME SMR-I-384-3 B
EXPNO 2
PROCNO 1

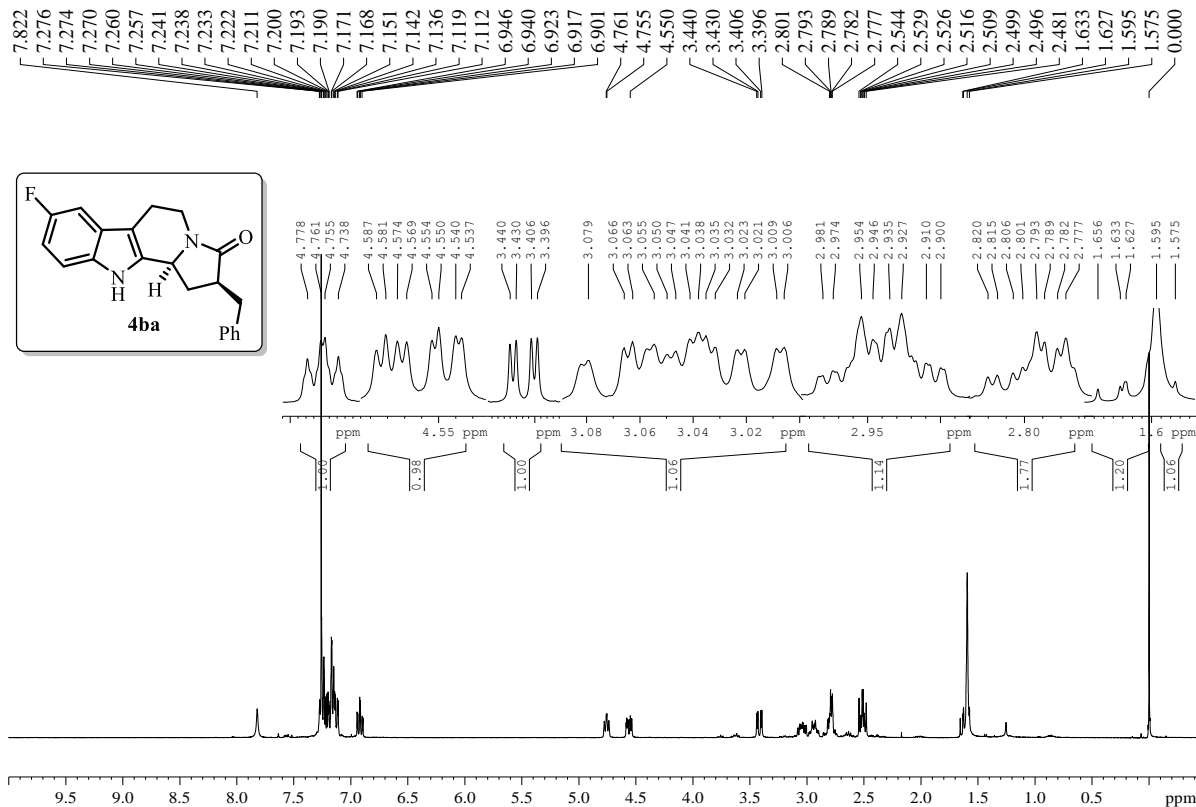
F2 - Acquisition Parameters
Date_ 20130729
Time_ 18.09
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 645
DW 20.800 usec
DE 6.00 usec
TE 299.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.89999998 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.60 dB
SF01 100.6228298 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 14.96 dB
PL13 15.60 dB
PL2 -0.90 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

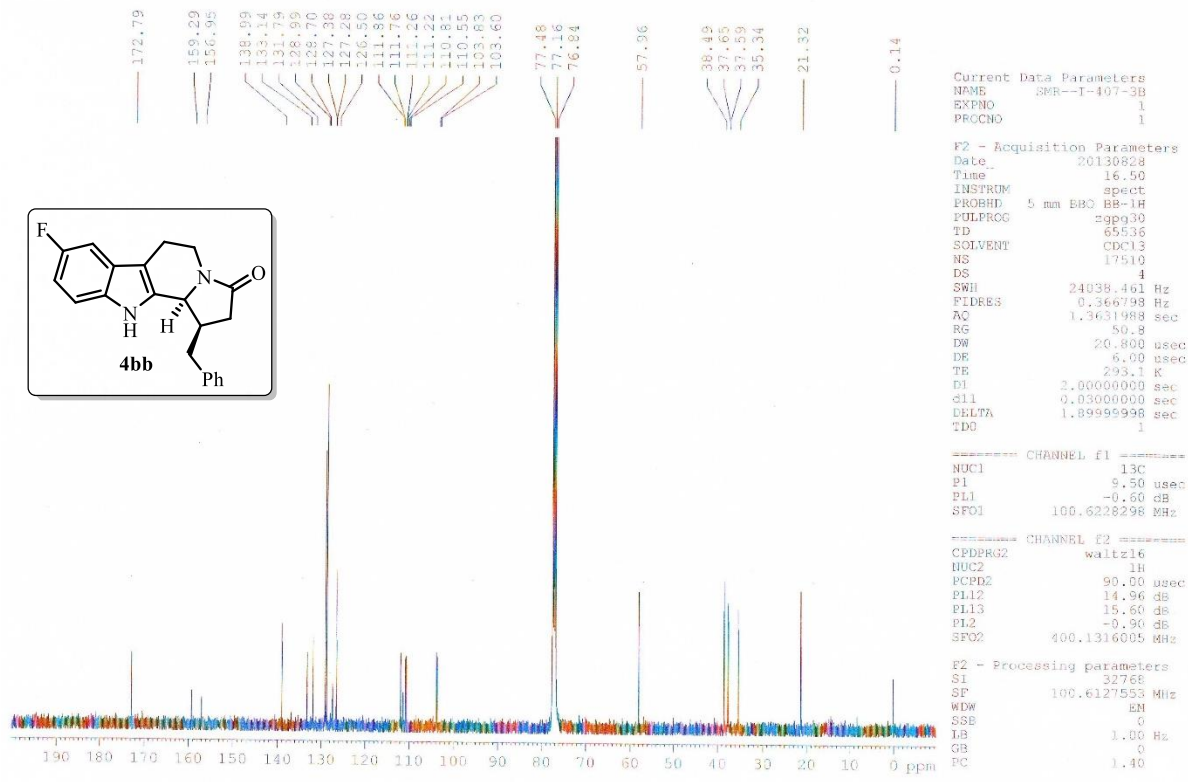
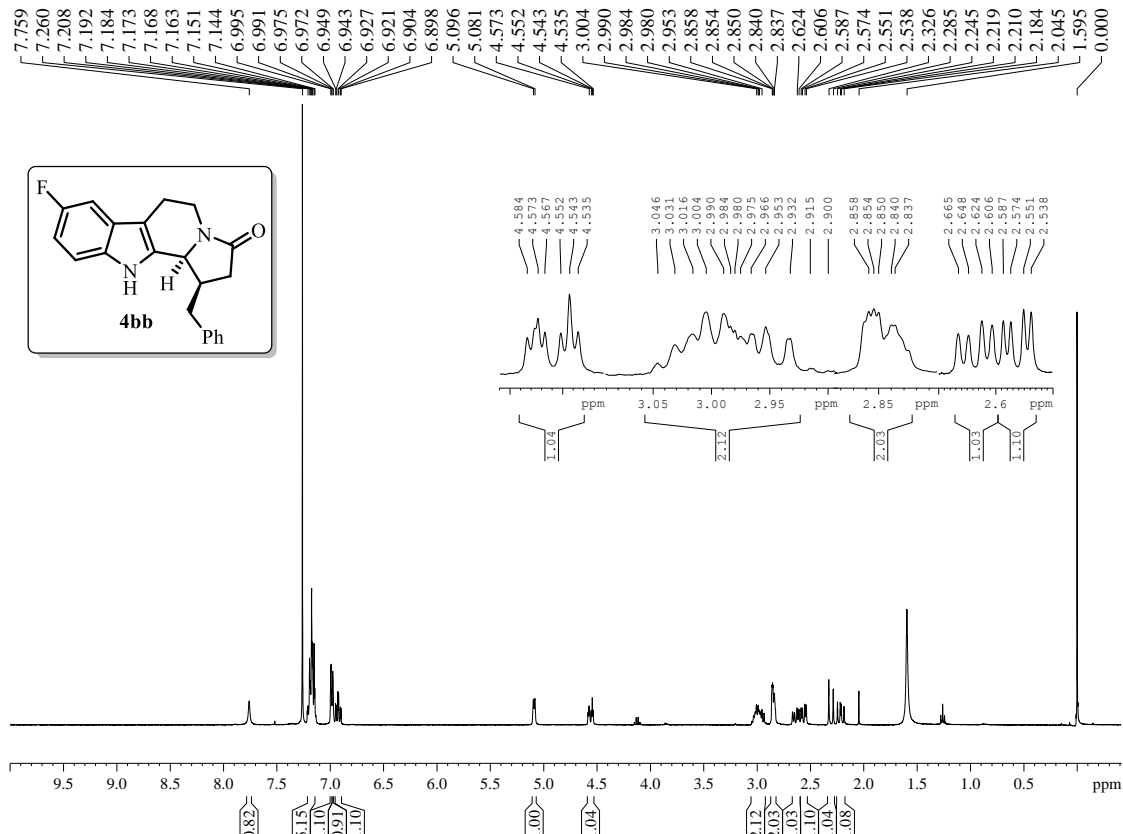
PROTON CDC13 {D:\CRR} KOPAL 1



C13CPD CDC13 {D:\CRR} KOPAL 1



PROTON CDC13 {D:\CRR} KOPAL 1



Current Data Parameters
 NAME SMR-I-407-3B
 EXPNO 1
 PROCNO 1

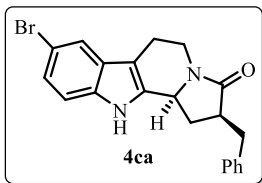
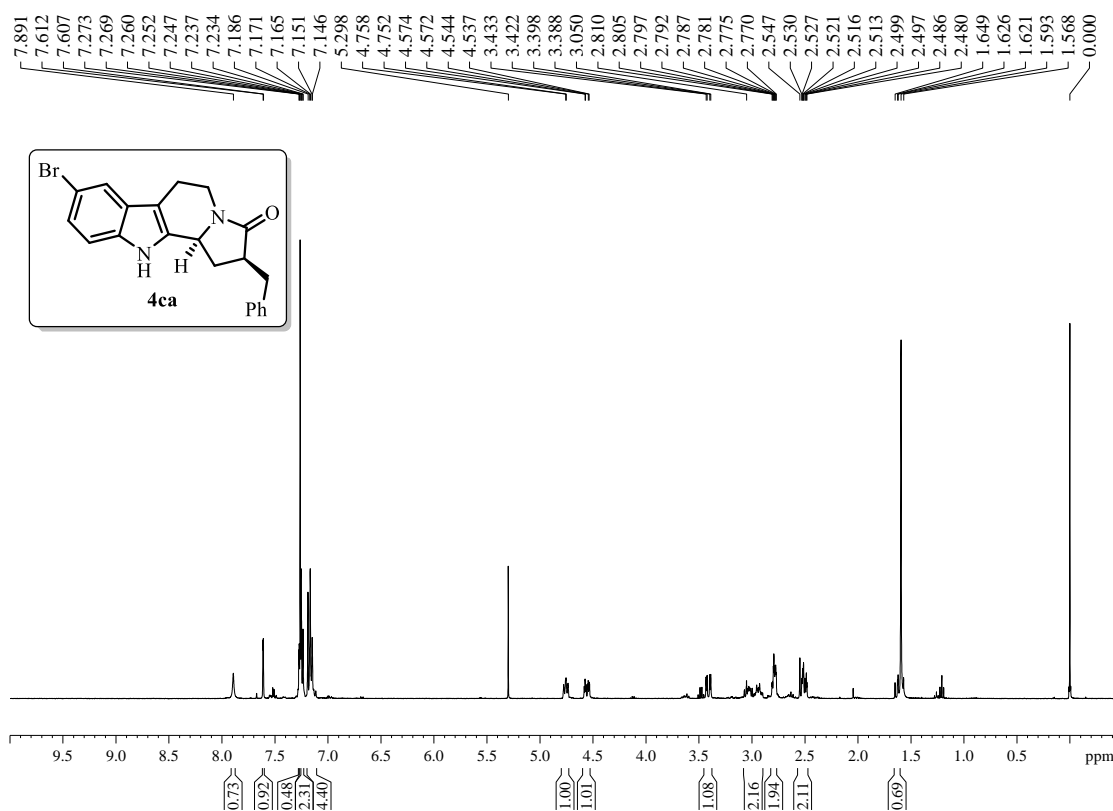
F2 - Acquisition Parameters
 Date 20130323
 Time 16.50
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 17510
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 50.8
 DW 20.800 usec
 DE 6.00 usec
 TE 293.1 K
 FI 2.0000000 sec
 cfl 0.0300000 sec
 DELTA 1.8999998 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 5.50 usec
 PL1 -0.60 dB
 SFO1 100.6228298 MHz

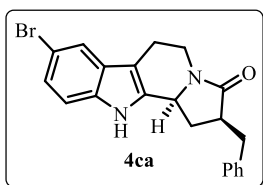
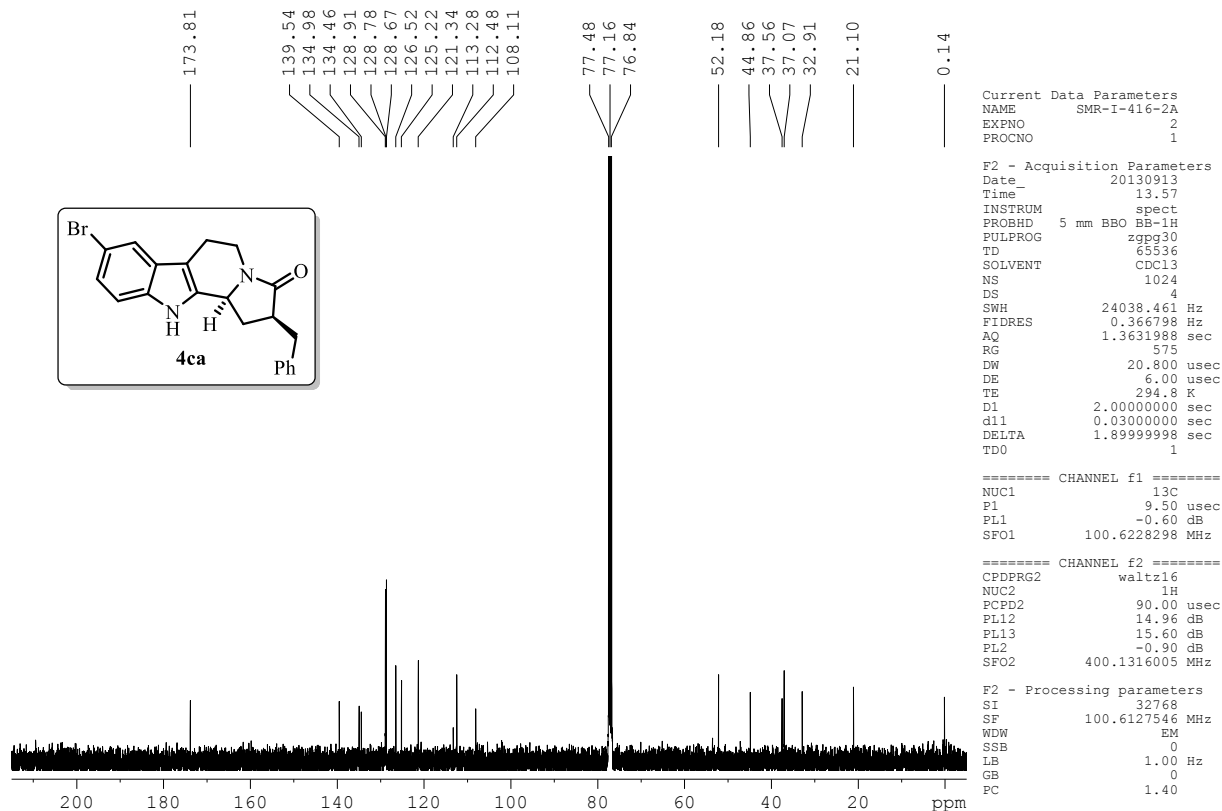
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL12 14.96 dB
 PL13 15.60 dB
 PL2 -0.90 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127553 MHz
 WDW EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

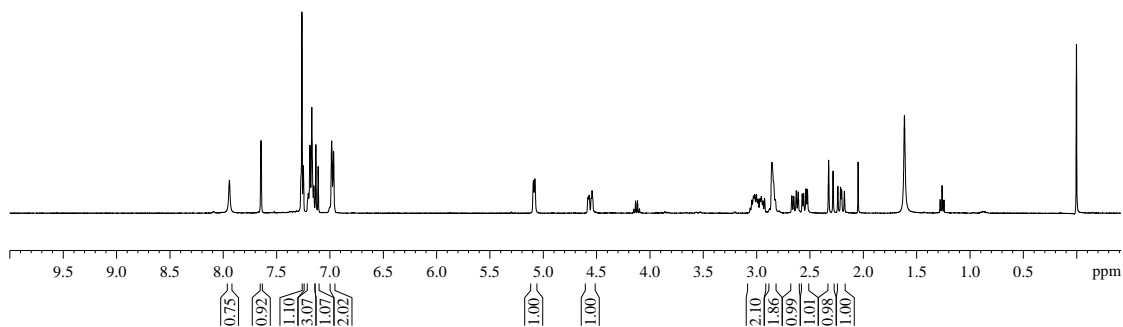
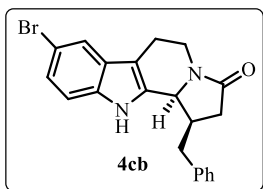
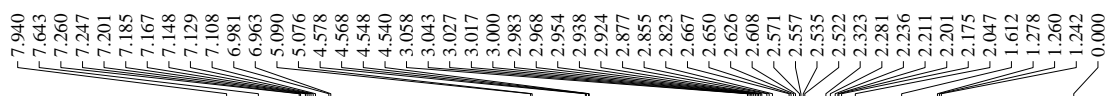
PROTON CDC13 {D:\CRR} KOPAL 1



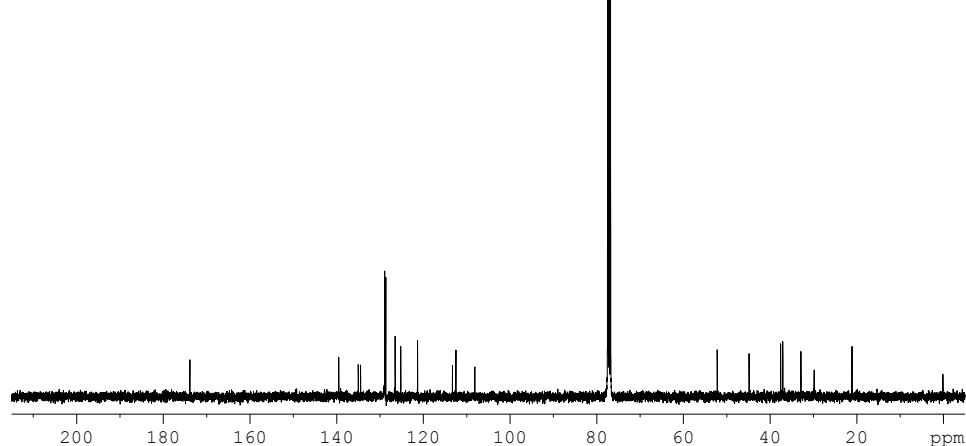
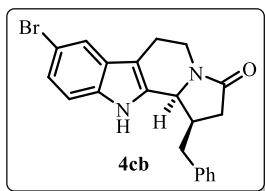
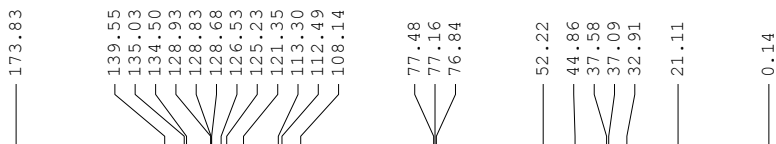
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PROTON CDC13 {D:\CRR} KOPAL 1



C13CPD CDC13 {D:\CRR} KOPAL 1



Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130918
Time 20.11
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 812
DW 20.800 usec
DE 6.00 usec
TE 300.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1

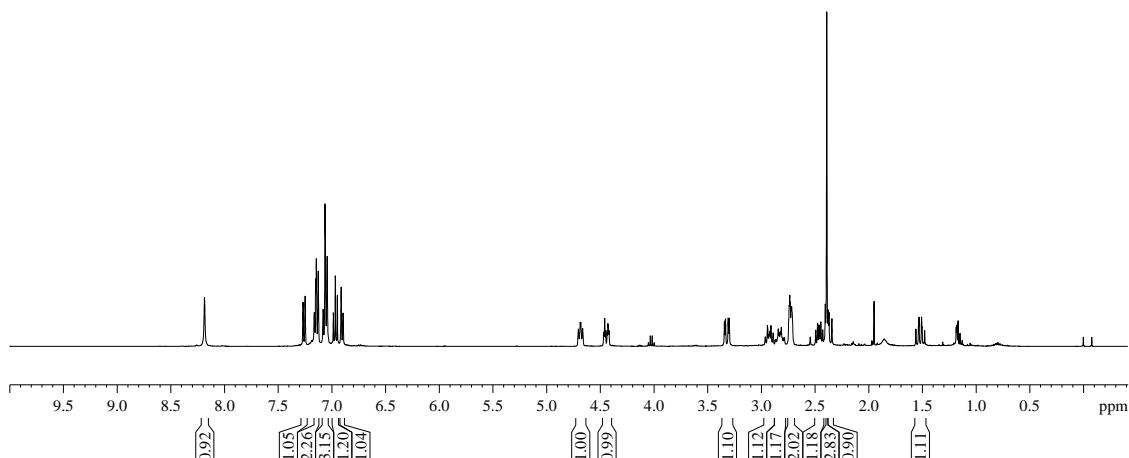
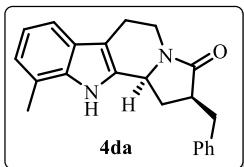
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NUC1 13C
P1 9.50 usec
PL1 -0.60 dB
SFO1 100.6228298 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 14.96 dB
PL13 15.60 dB
PL2 -0.90 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127524 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

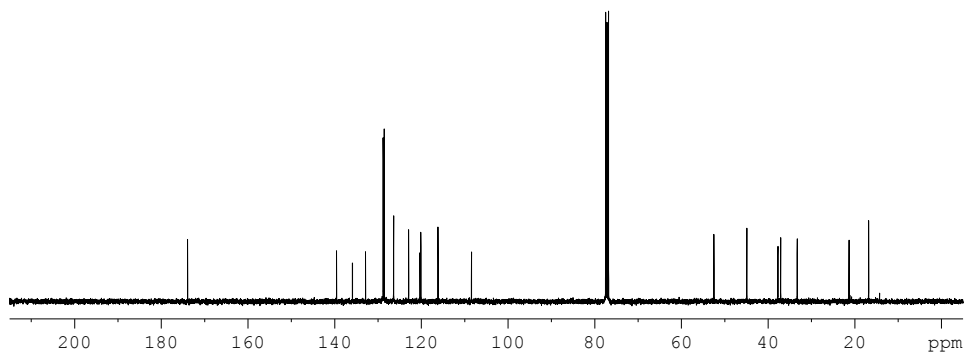
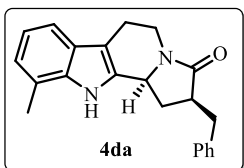
PROTON CDC13 {D:\CRR} KOPAL 1

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7.267
7.247
7.162
7.150
7.143
7.125
7.080
7.077
7.062
7.059
7.042
6.985
6.966
6.947
6.912
6.894
4.703
4.685
4.680
4.663
4.458
4.427
3.342
3.332
3.307
3.297
2.941
2.909
2.841
2.813
2.739
2.734
2.729
2.721
2.716
2.475
2.462
2.457
2.445
2.427
2.403
2.390
2.376
2.369
2.342
1.559
1.535
1.530
1.506
1.501



C13CPD CDC13 {D:\CRR} KOPAL 1

173.95
139.59
135.91
132.90
128.83
128.60
126.44
126.41
122.95
120.33
120.14
116.17
108.45
77.48
77.16
76.84
52.51
44.91
37.75
37.11
33.29
21.33
16.81



Current Data Parameters
NAME SMR-I-411-2A
EXPNO 1
PROCNO 1

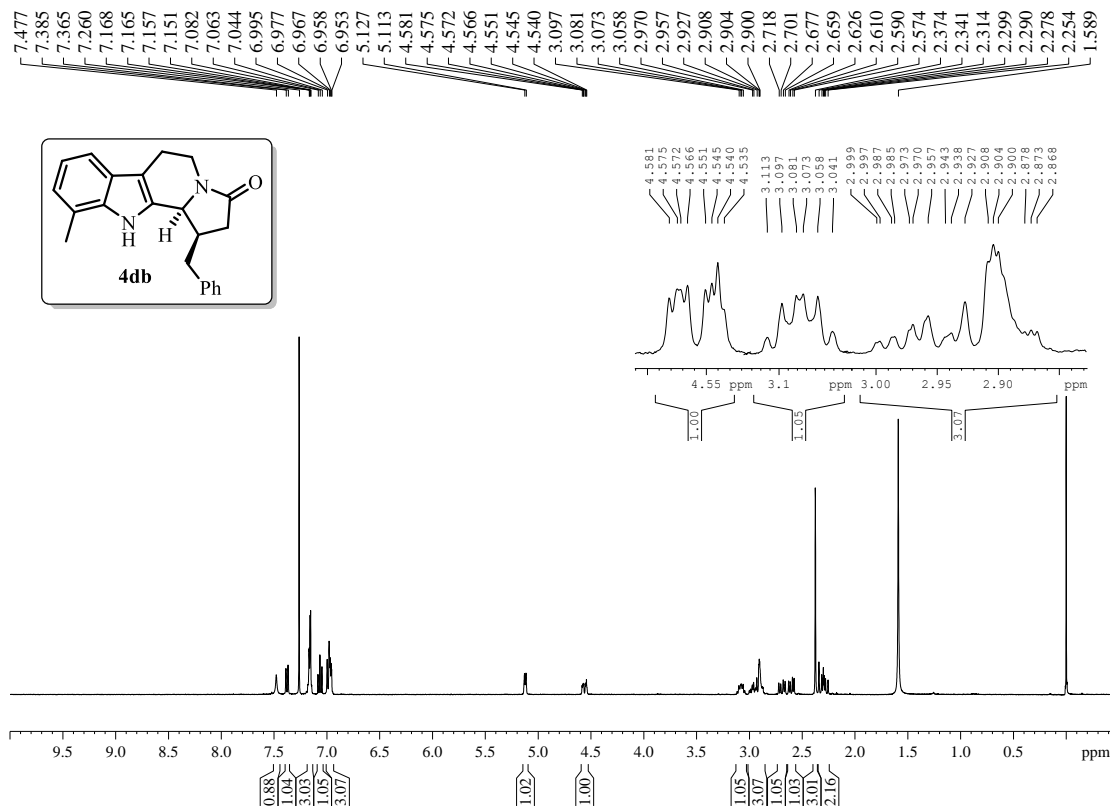
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Date_ 20130830
Time 17.16
INSTRUM spect
PROBHD 5 mm BBO BB-1H
FULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 278
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 50.8
DW 20.800 usec
DE 6.00 usec
TE 296.9 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999999 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.60 dB
SFO1 100.6228298 MHz

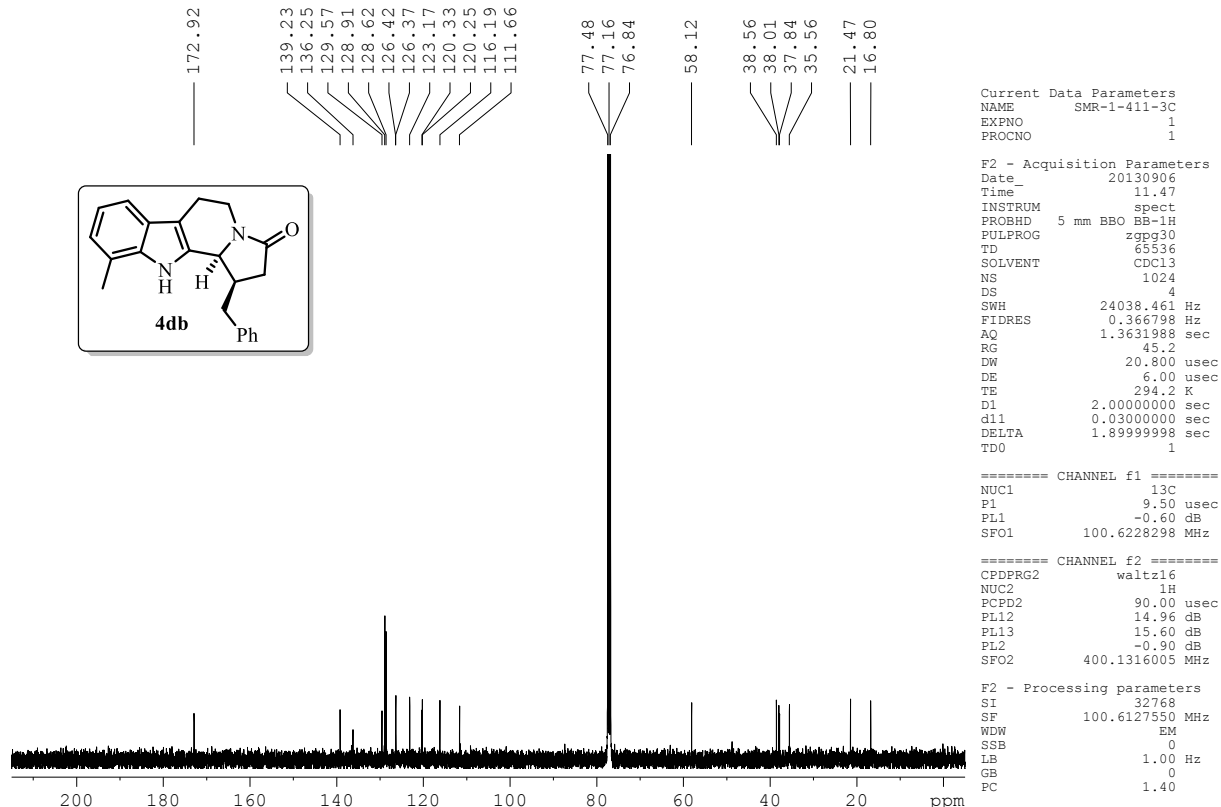
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 14.96 dB
PL13 15.60 dB
PL2 -0.90 dB
SFO2 400.1316005 MHz

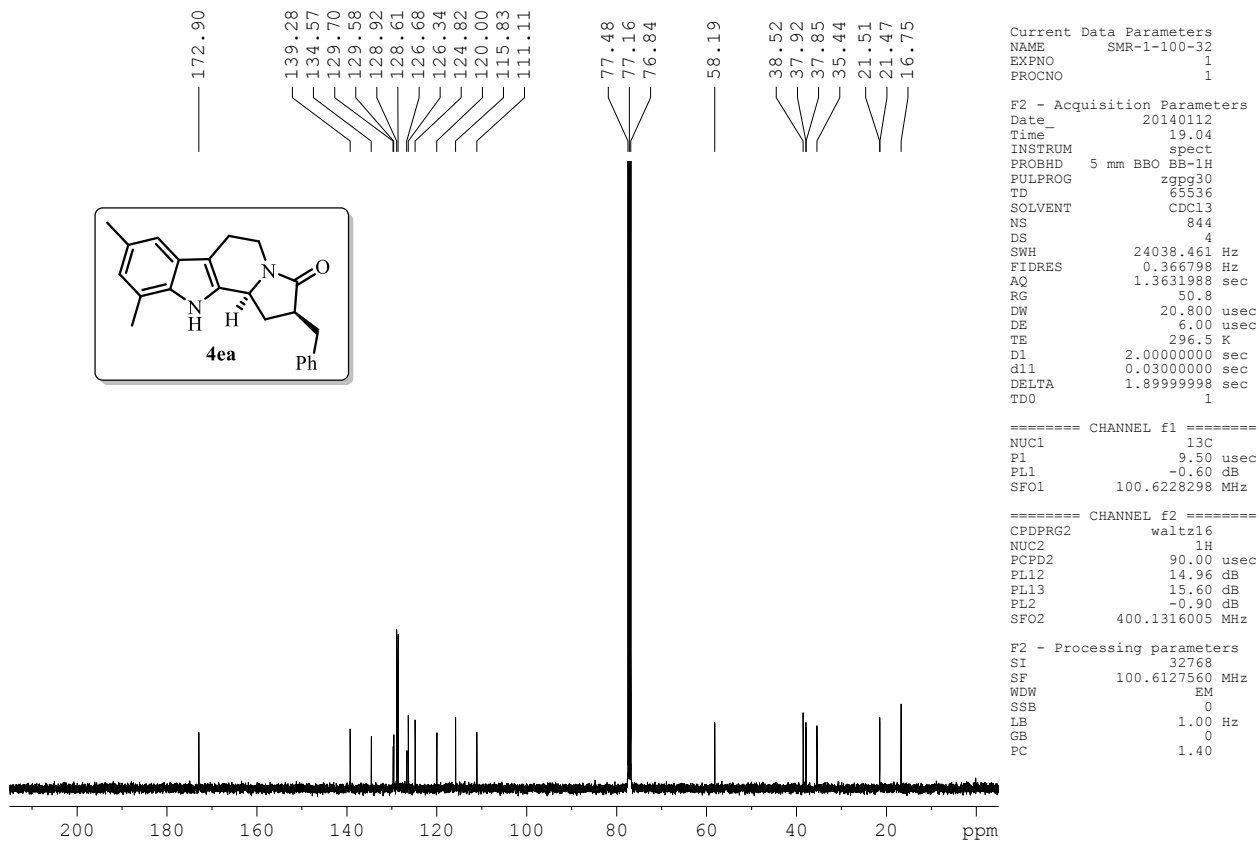
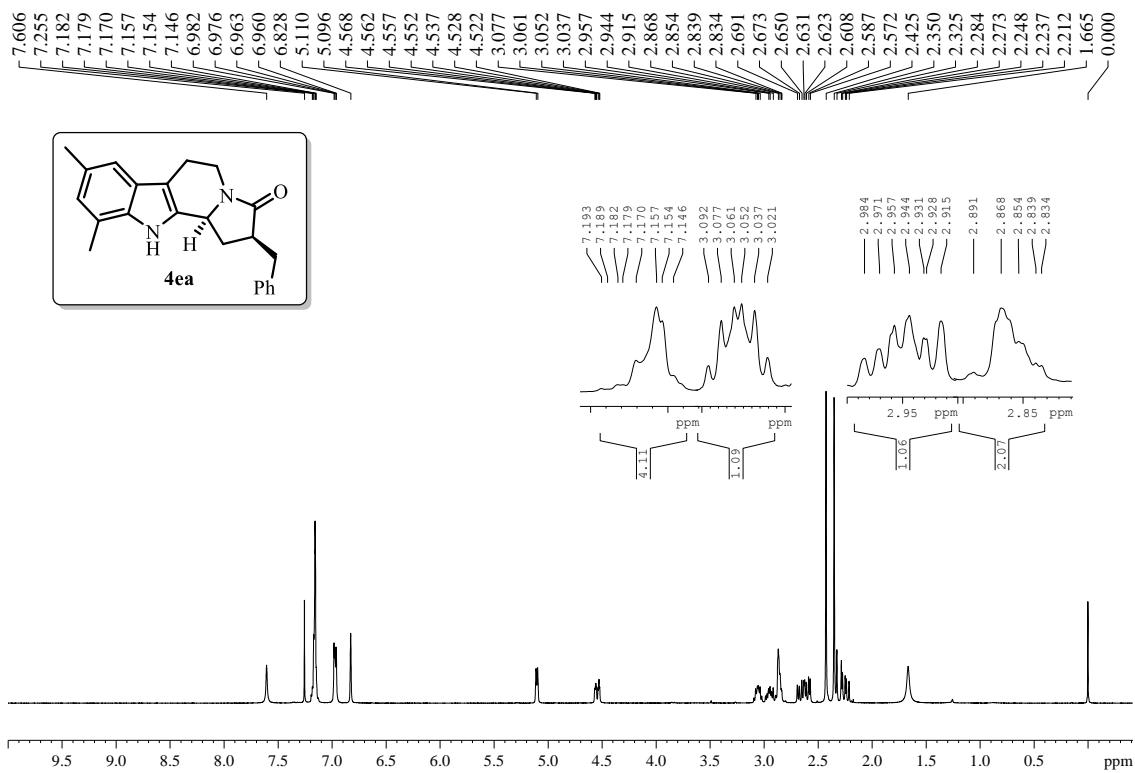
F2 - Processing parameters
SI 32768
SF 100.6127630 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

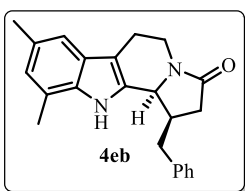
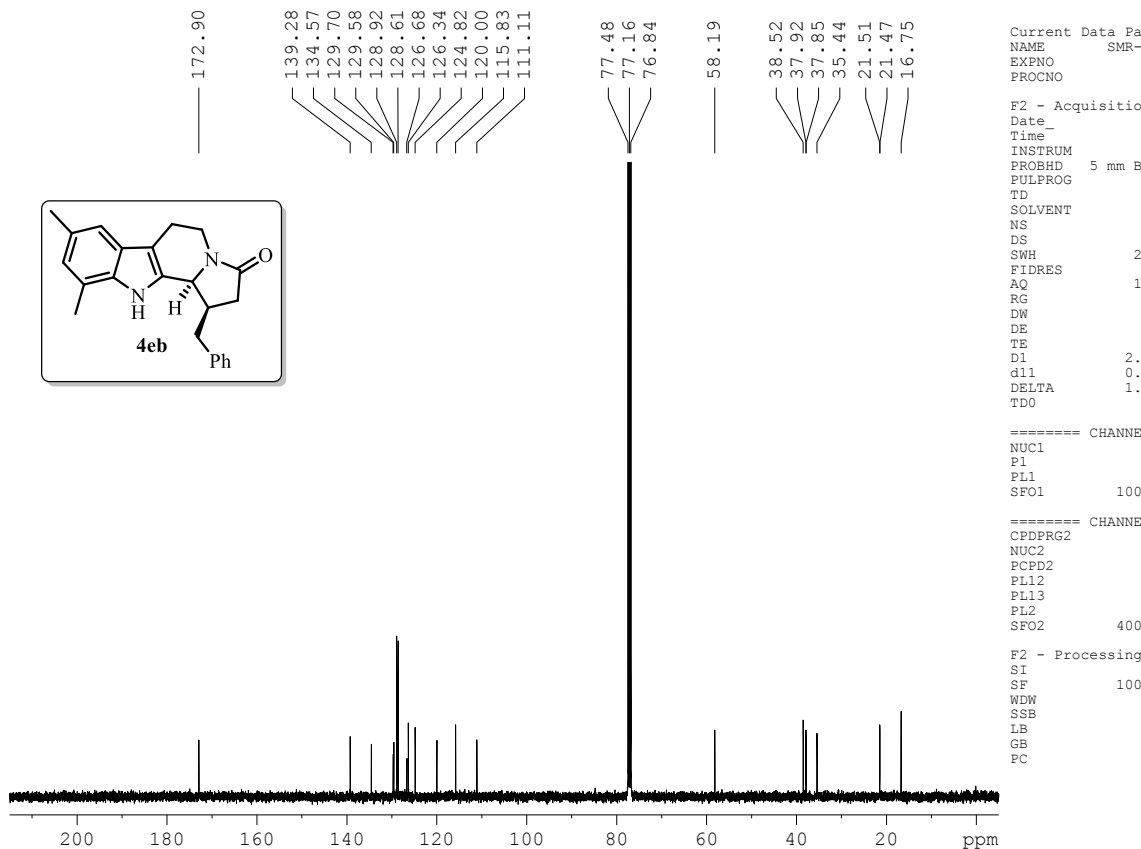
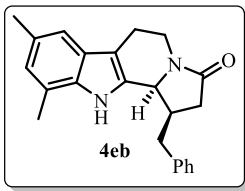
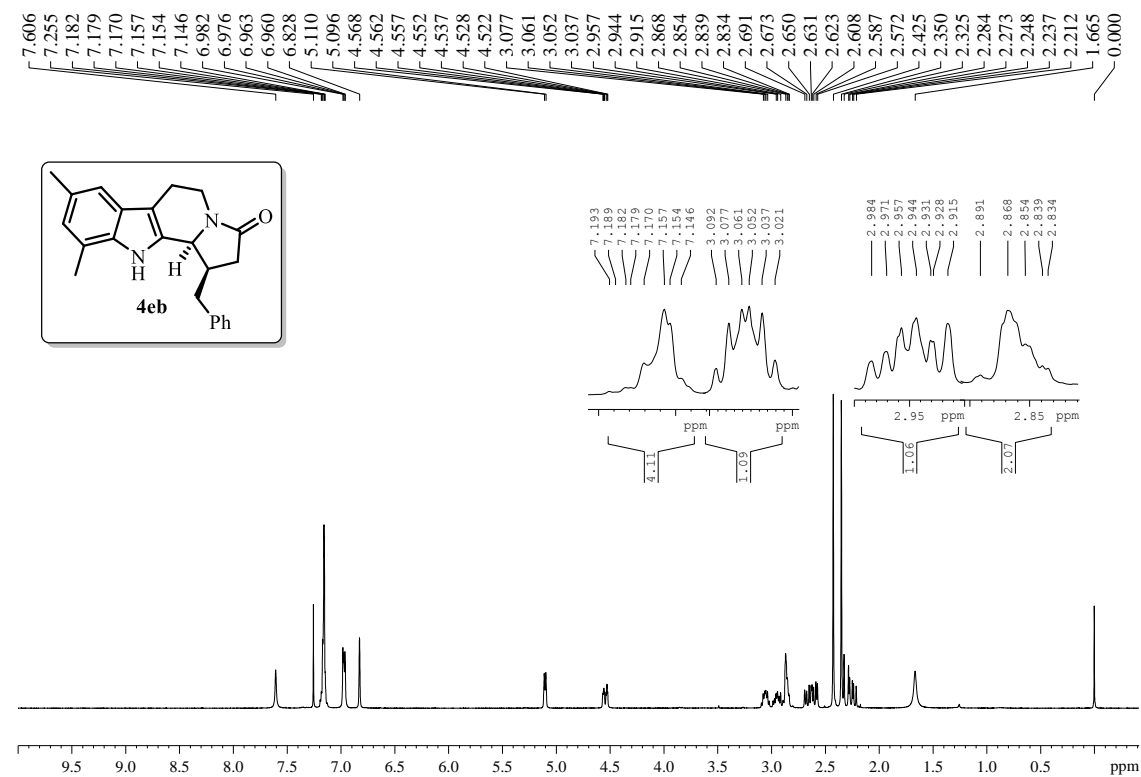
PROTON CDC13 {D:\CRR} KOPAL 1



C13CPD CDC13 {D:\CRR} KOPAL 1







Current Data Parameters
 NAME SMR-1-100-32
 EXPNO 1
 PROCNO 1

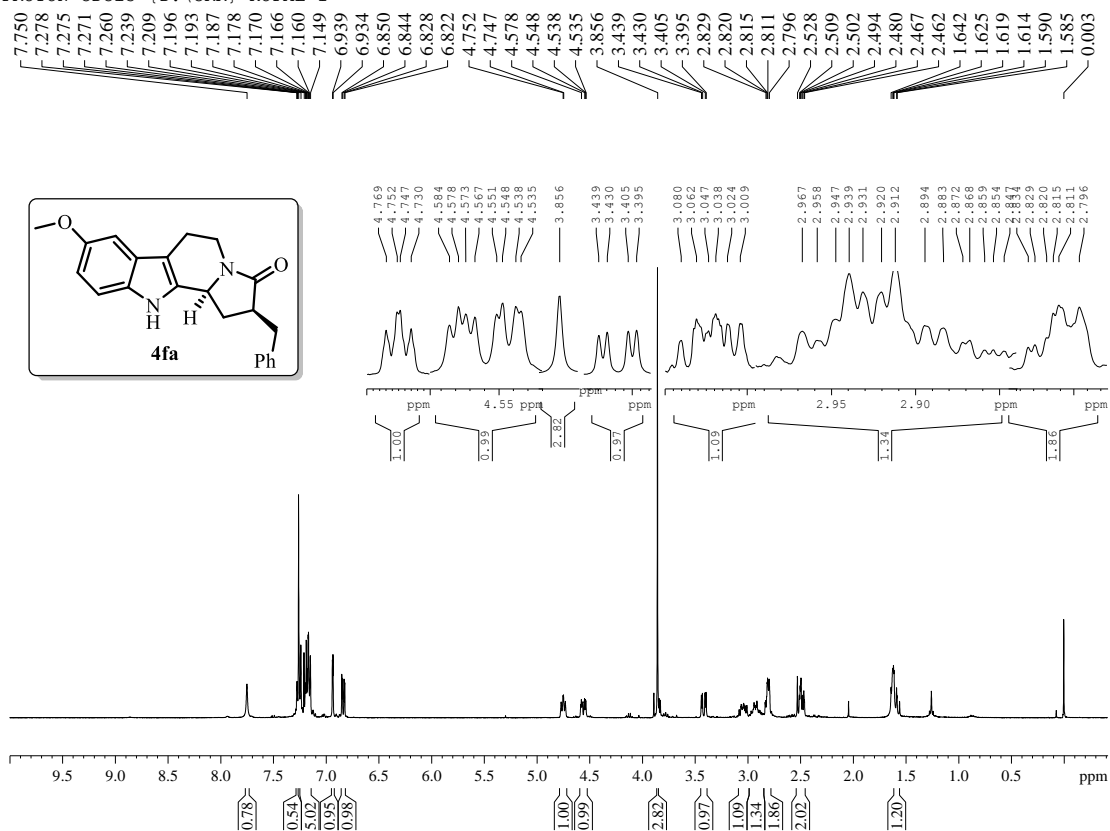
F2 - Acquisition Parameters
 Date_ 20140112
 Time 19.04
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 844
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 50.8
 DW 20.800 usec
 DE 6.00 usec
 TE 296.5 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999999 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 -0.60 dB
 SFO1 100.6228298 MHz

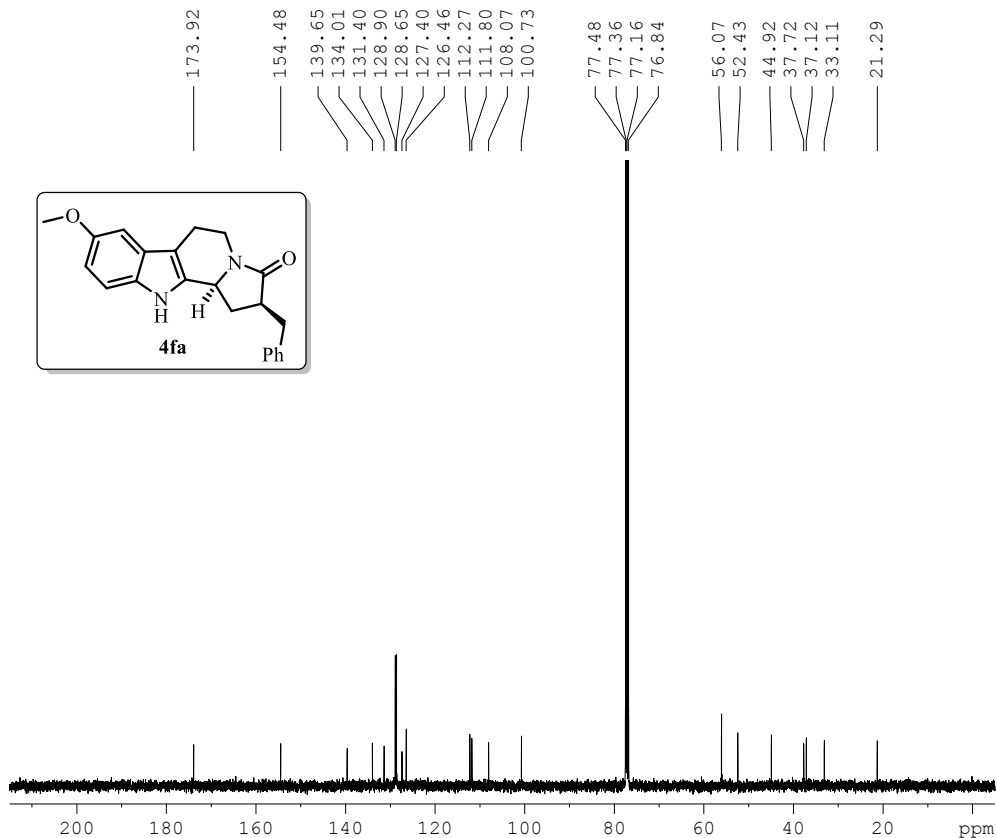
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 FCPD2 90.00 usec
 PL12 14.96 dB
 PL13 15.60 dB
 PL2 -0.90 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127560 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 FC 1.40

PROTON CDC13 {D:\CRR} KOPAL 1



C13CPD CDC13 {D:\CRR} KOPAL 1



Current Data Parameters
 NAME SMR-I-413-2A
 EXPNO 2
 PROCNO 1

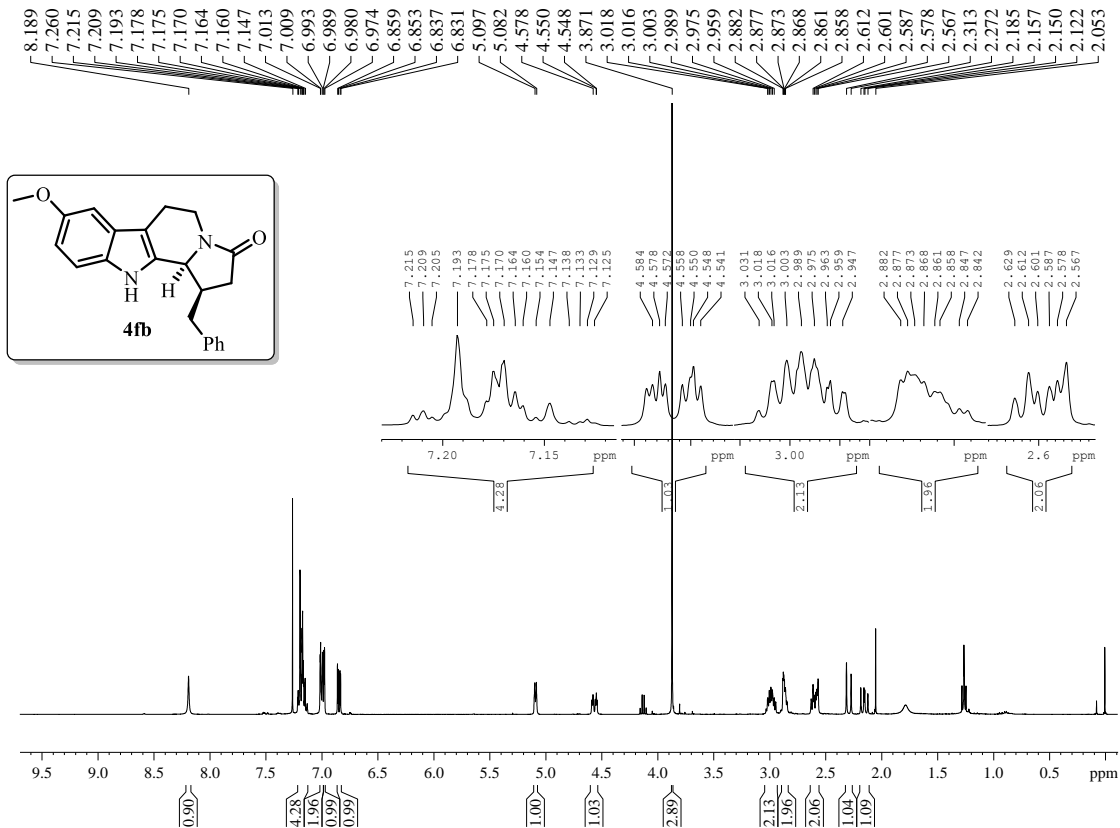
F2 - Acquisition Parameters
 Date_ 20130911
 Time_ 12.35
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 724
 DW 20.800 usec
 DE 6.00 usec
 TE 297.6 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 -0.60 dB
 SFO1 100.6228298 MHz

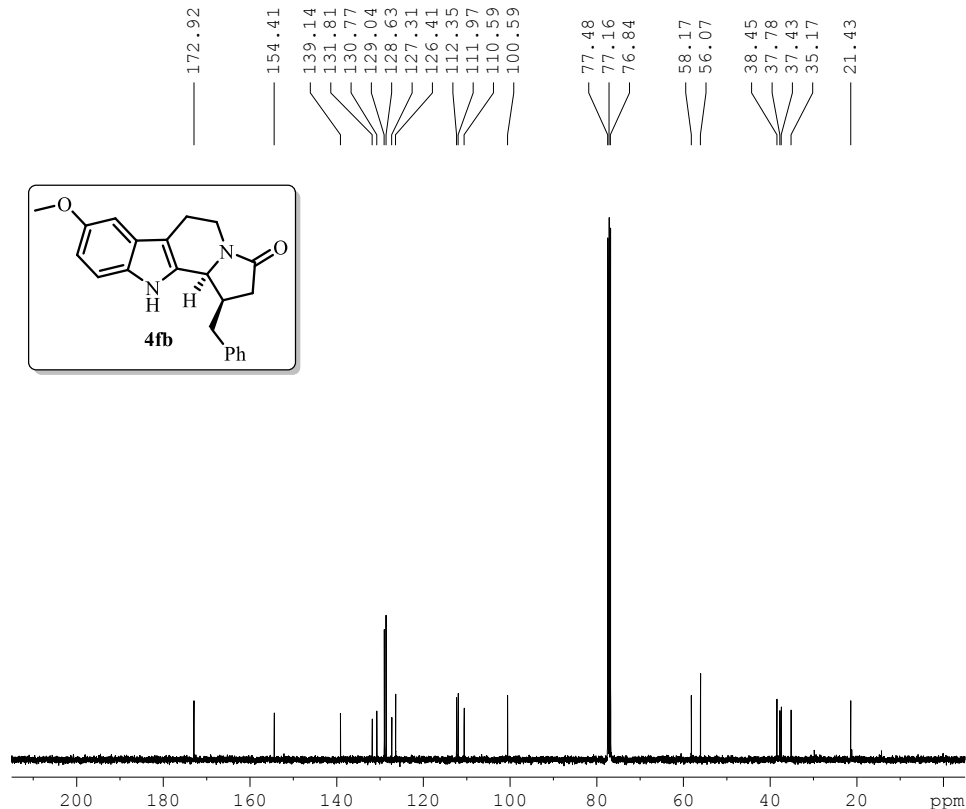
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL12 14.96 dB
 PL13 15.60 dB
 PL2 -0.90 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127556 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

PROTON CDC13 {D:\CRR} KOPAL 1



C13CPD CDC13 {D:\CRR} KOPAL 1



Current Data Parameters
 NAME SMR-1-396-3A
 EXPNO 1
 PROCNO 1

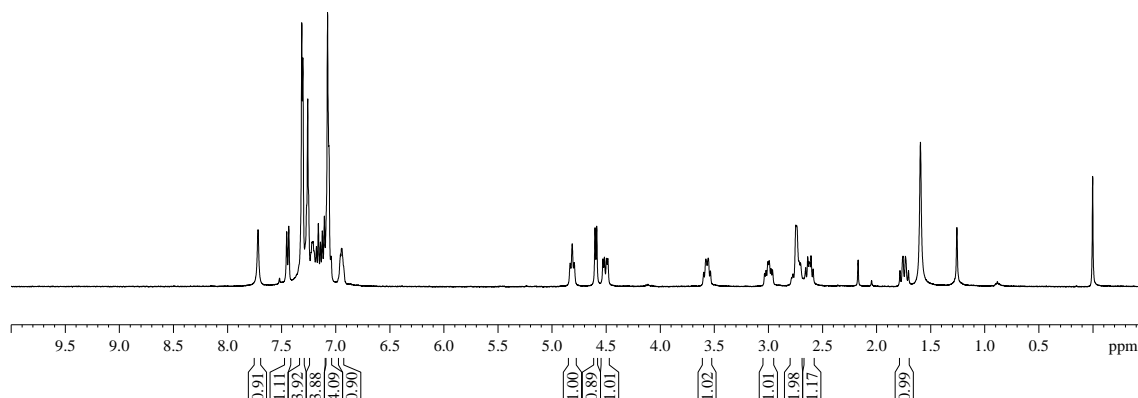
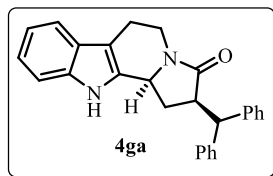
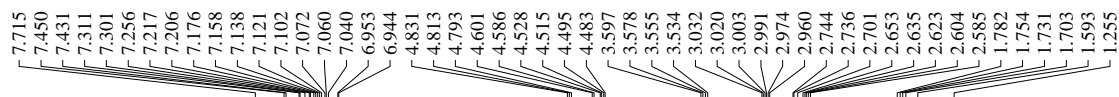
F2 - Acquisition Parameters
 Date_ 20130724
 Time 15.26
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 50.8
 DW 20.800 usec
 DE 6.00 usec
 TE 298.1 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999999 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 -0.60 dB
 SFO1 100.6228298 MHz

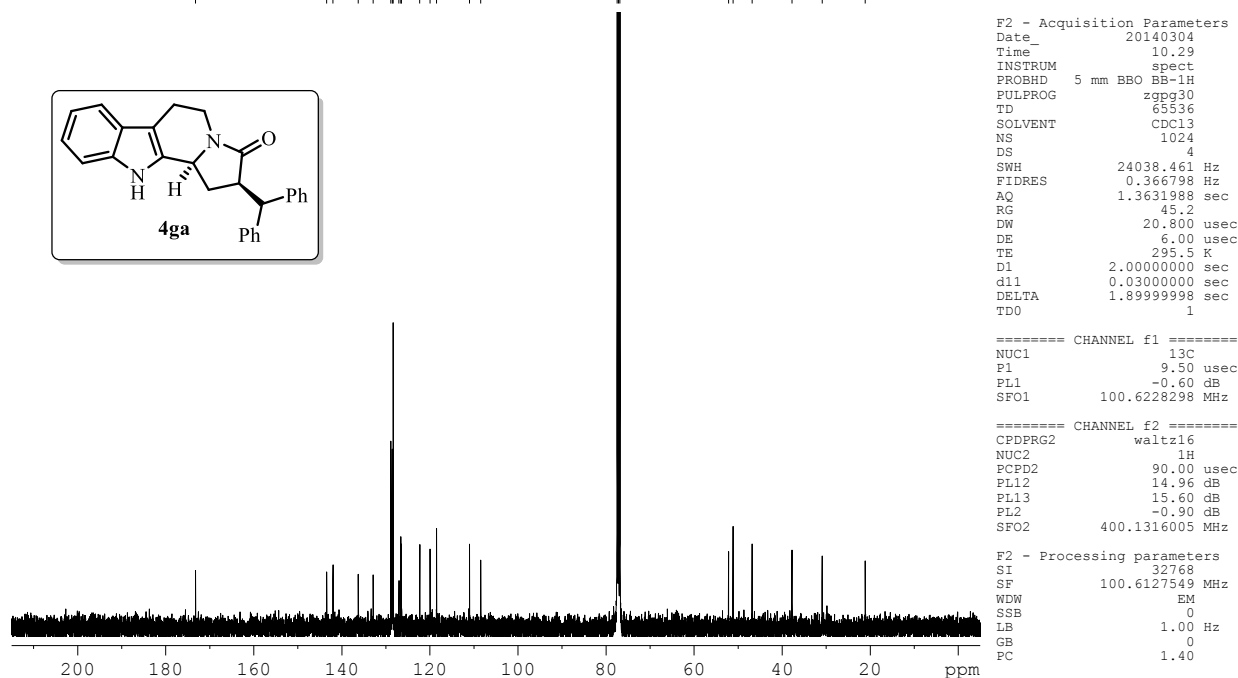
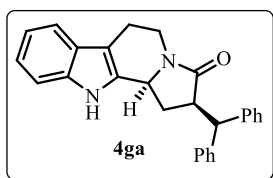
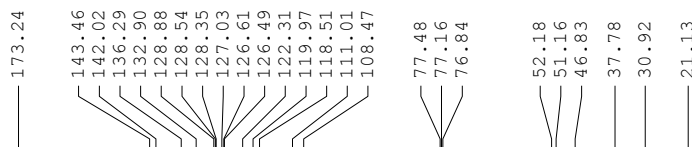
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL12 14.96 dB
 PL13 15.60 dB
 PL2 -0.90 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127568 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 FC 1.40

PROTON CDC13 {D:\CRR} KOPAL 1

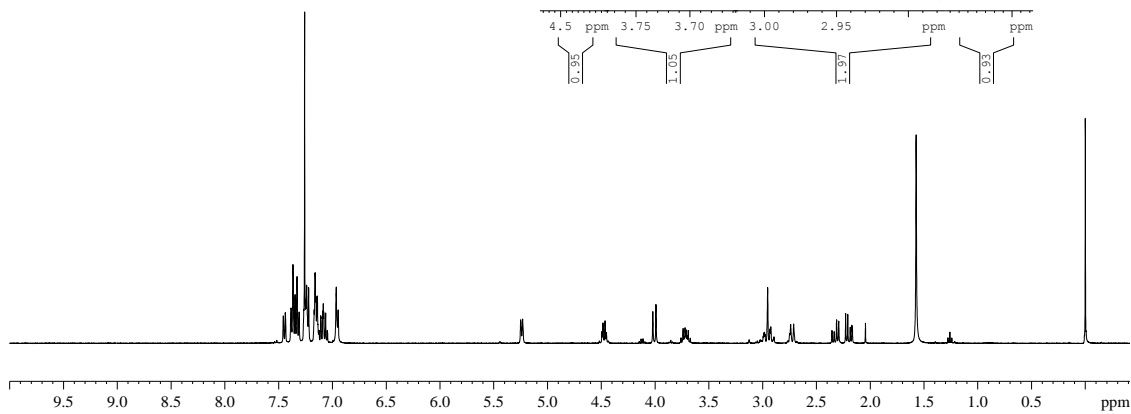
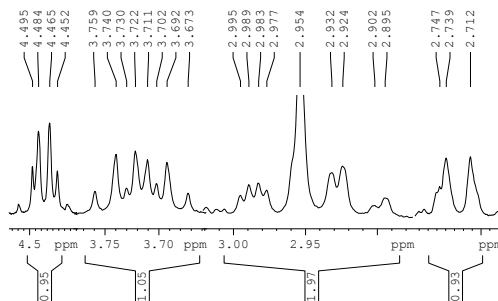
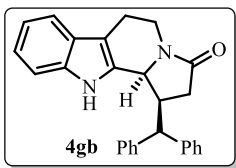


C13CPD CDC13 {D:\CRR} KOPAL 1



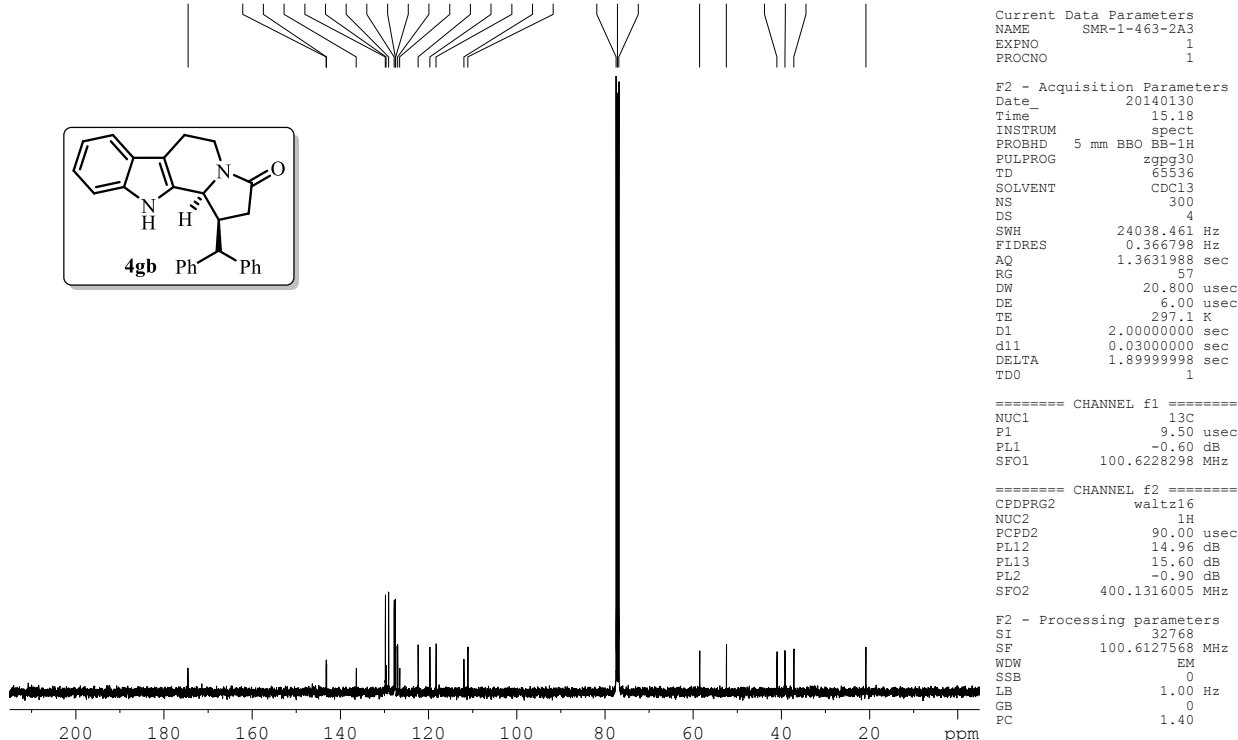
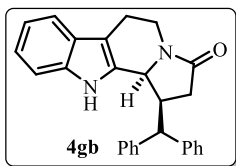
PROTON CDC13 {D:\CRR} KOPAL 1

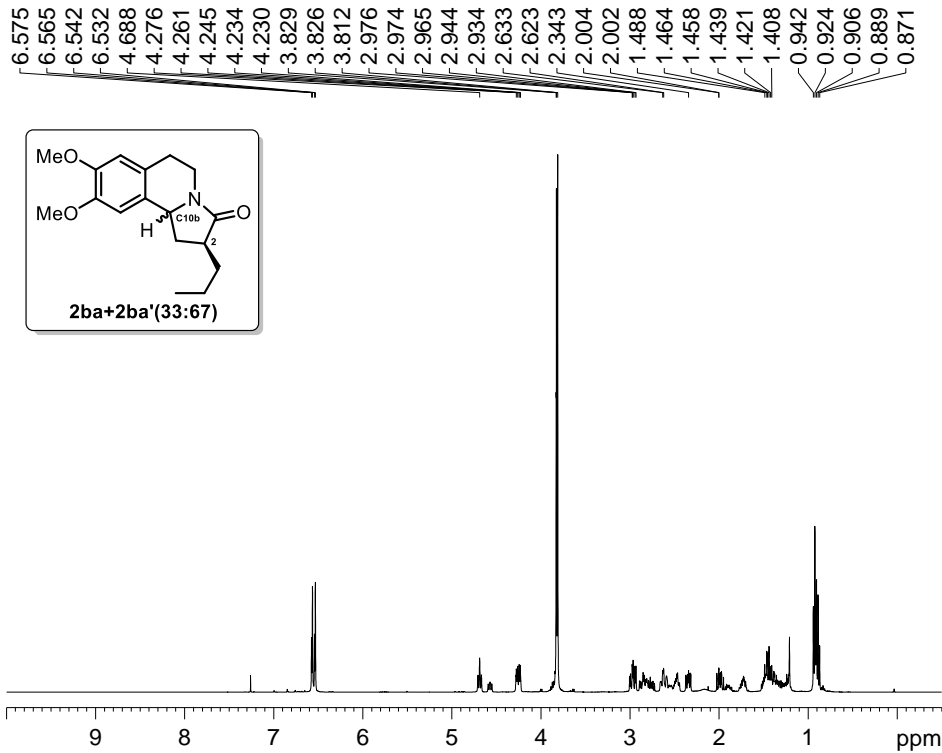
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C13CPD CDC13 {D:\CRR} KOPAL 1

174.53 143.23 143.14 136.39 129.81 129.62 129.07 127.80 127.55 127.45 127.01 126.54 122.38 119.71 118.30 111.99 111.09 77.48 77.16 76.85 58.52 52.46 41.00 39.20 37.17 20.84



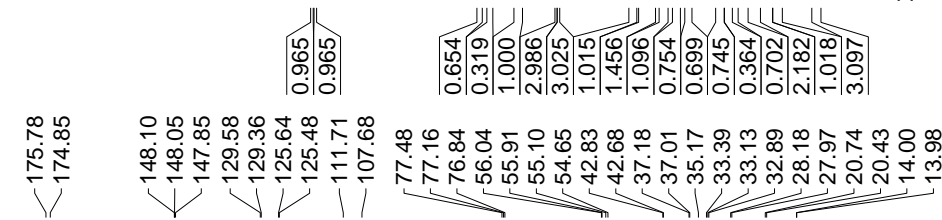


Current Data Parameters
NAME Spectra
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160713
Time 17.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 57
DW 60.800 usec
DE 6.00 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.42 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300048 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



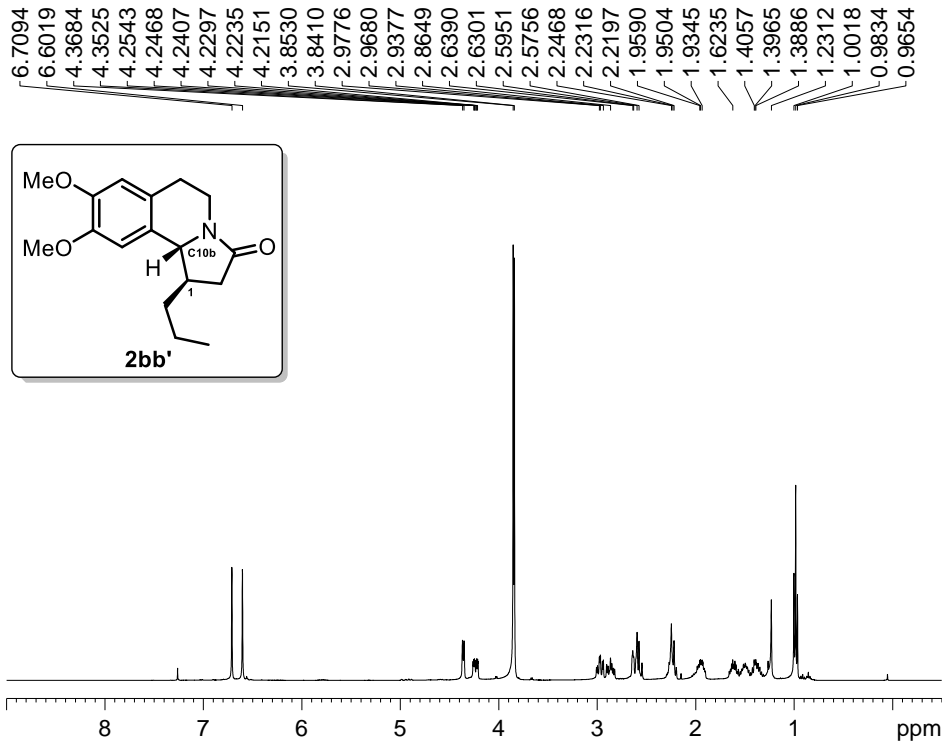
Current Data Parameters
NAME 12.1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160713
Time 17.31
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 211
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 32
DW 20.800 usec
DE 6.00 usec
TE 295.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.15 usec
PL1 0 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 14.90 dB
PL13 14.90 dB
PL2 -3.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127662 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

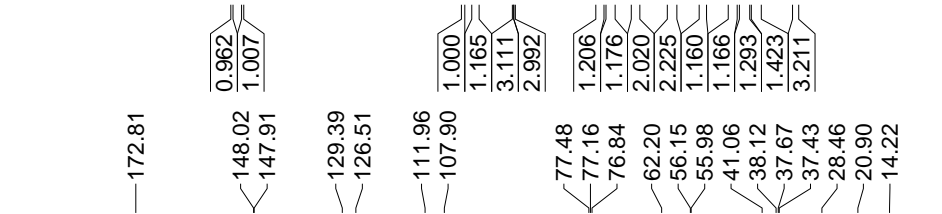


Current Data Parameters
NAME MMA-CL- 12-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160714
Time 17.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.00 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.42 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME MMA-CL- 12-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160714
Time 18.37
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 36
DW 20.800 usec
DE 6.00 usec
TE 293.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.15 usec
PL1 0 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL12 14.90 dB
PL13 14.90 dB
PL2 -3.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127605 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

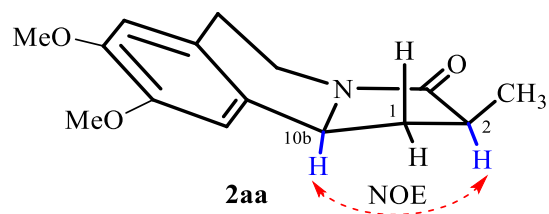
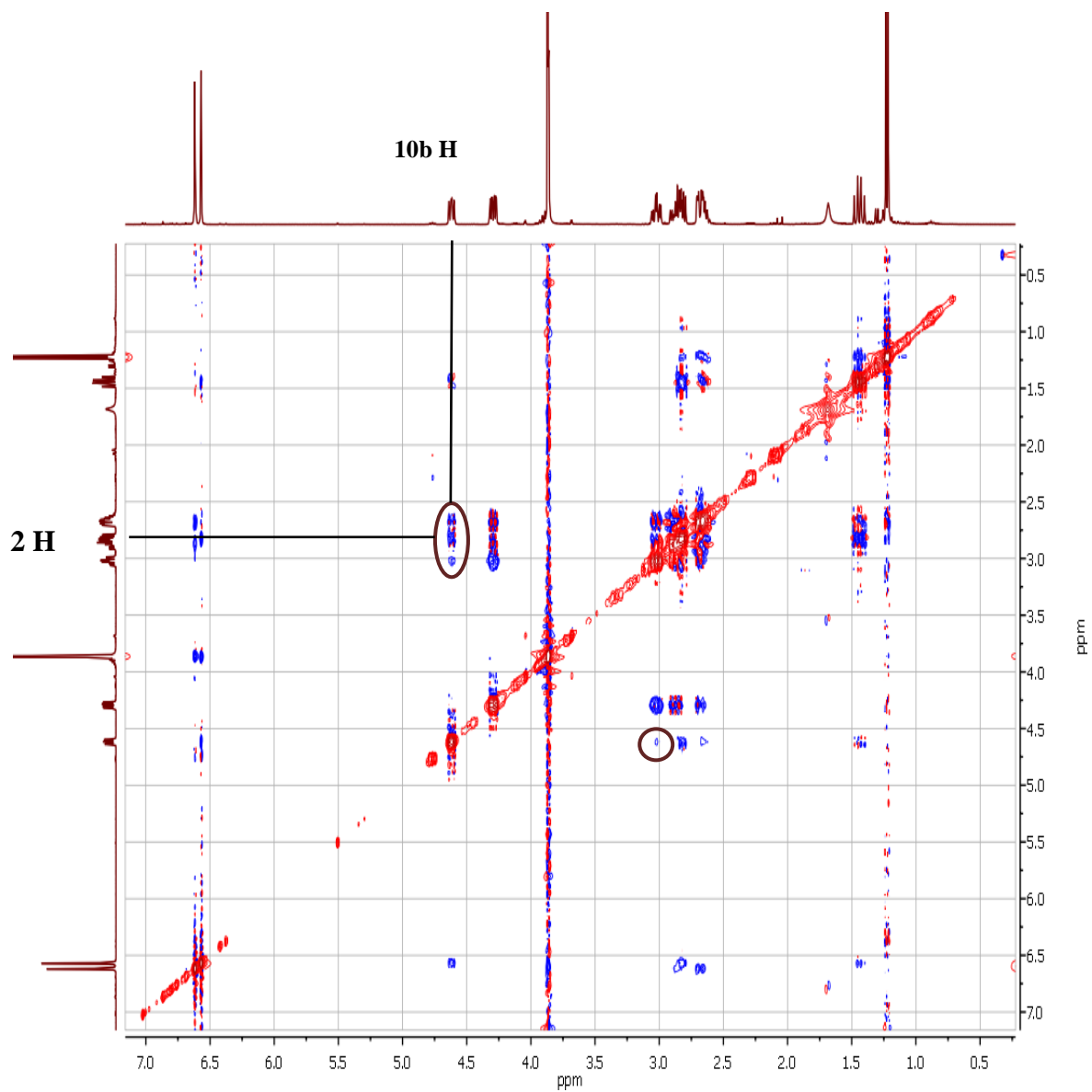


Figure 1 2D-NOESY spectrum showing cross peaks between C10bH-C2H in **2aa**.

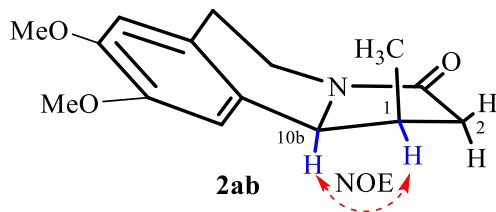
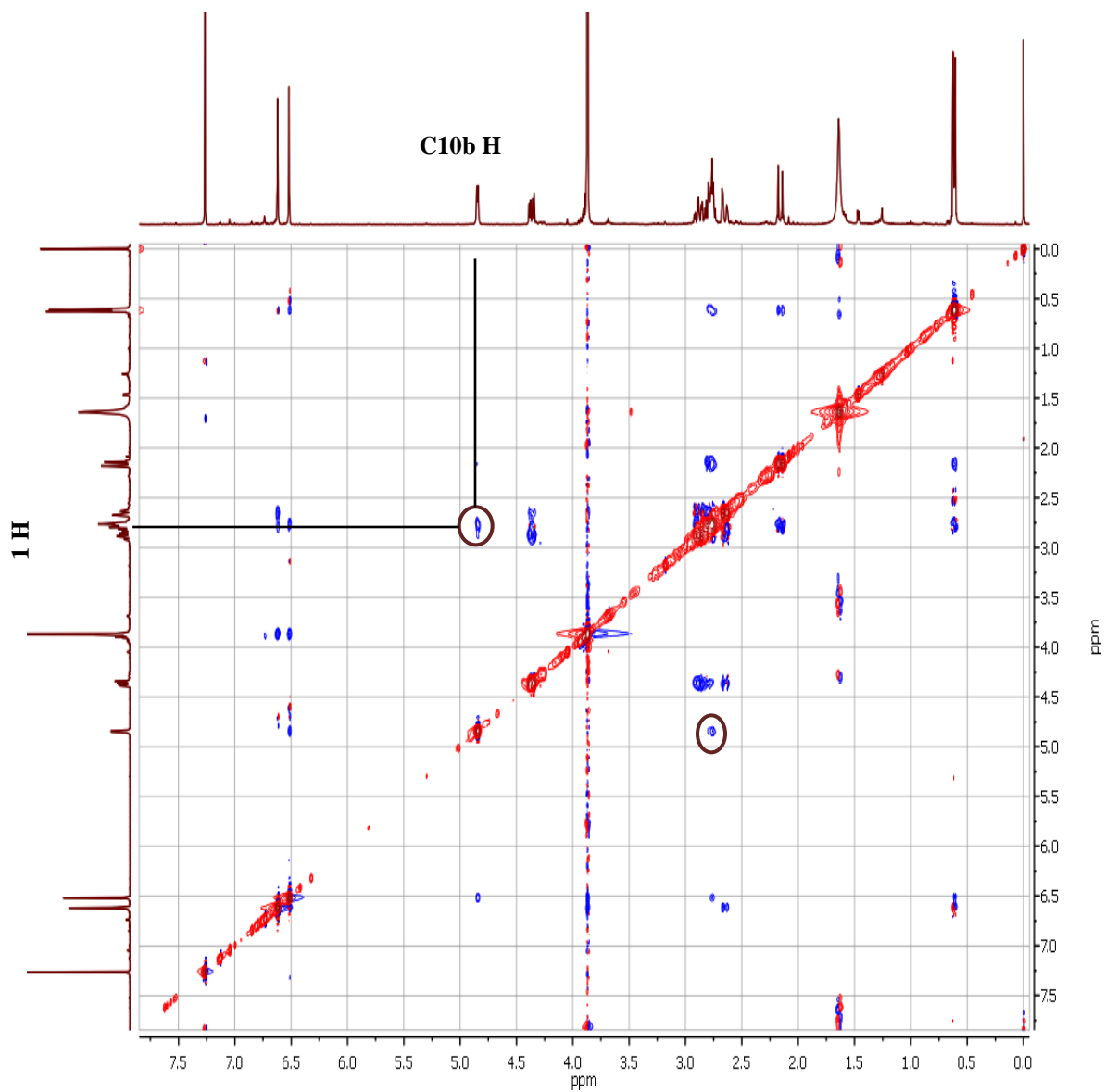


Figure 2 2D-NOESY spectrum showing cross peaks between C10bH-C1H in **2ab**.

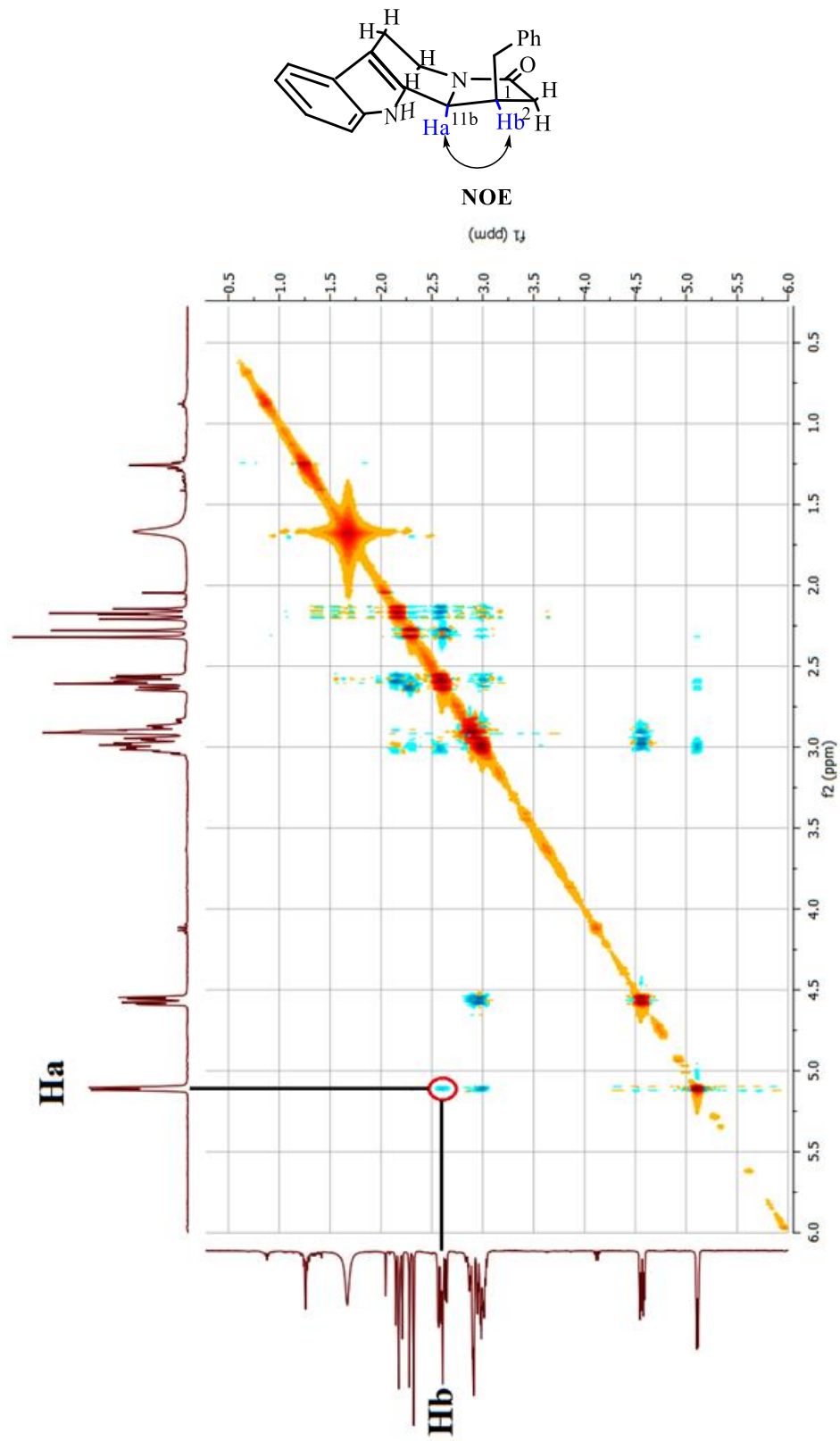


Figure 3 2D NOESY spectrum of **4ab**.

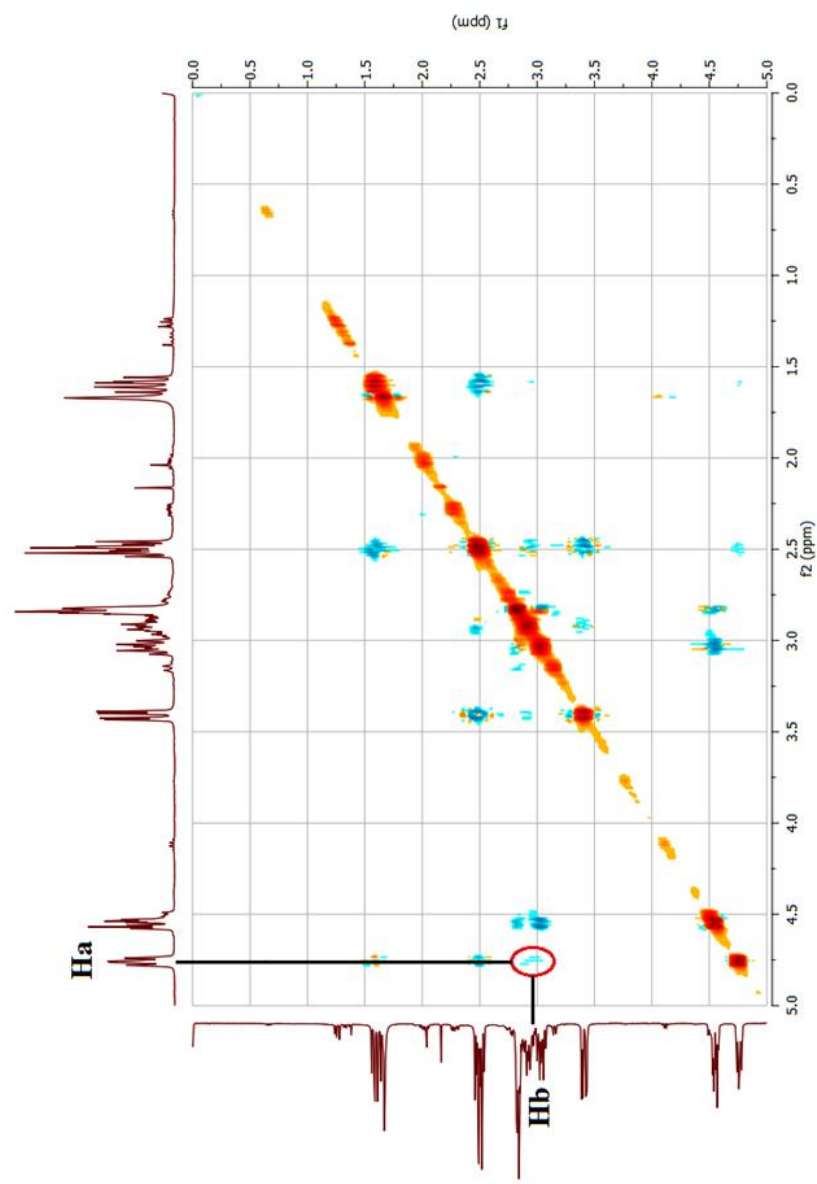
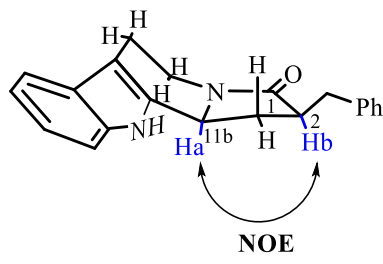


Figure 4 2D NOESY spectrum of 4aa.

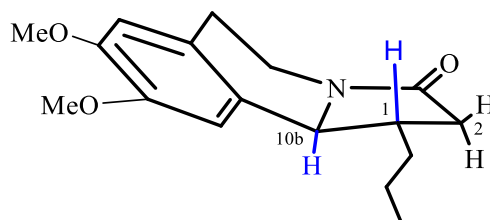
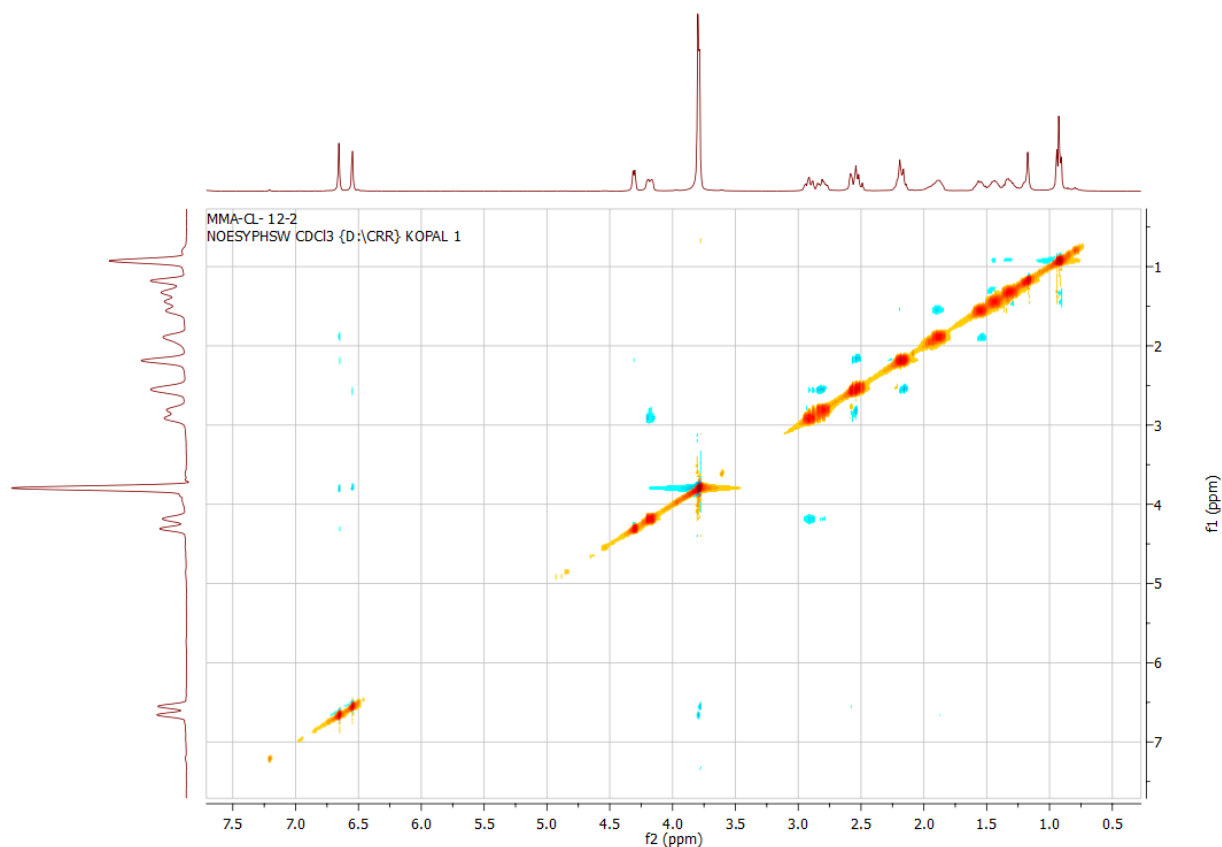


Figure 5 2D NOESY spectrum of **2bb'**.

1. G. M. Sheldrick, *SHELX-97, Programs for the Solution and Refinement of Crystal Structures*, University of Göttingen, Germany, **1997**.
2. W. L. F. Armarego, C. L. L. Chai, *Purification of Laboratory Chemicals*, 6th ed., Elsevier, UK, **2009**.
3. Merchant, J. R.; Mountwala, A. J. *J. Org. Chem.* **1958**, *23*, 1774–1775. (b) Sinhababu, A. K.; Borchardt, R. T.; *Tetrahedron Lett.* **1983**, *24*, 227–230. (c) Trost, B. M.; Yeh, V. S. C.; Ito, H.; Bremeyer, N. *Org. Lett.* **2002**, *4*, 2621–2623. (d) Pei, Z.; Li, L.; Geldern, T. W.; Longenecker, K.; Pireh, D.; Stewart, K.D.; Backes, B. J.; Lai, C.; Lubben, T. H.;

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