

Supporting Information

Asymmetric first total synthesis of Preoxazin, Oxazin-5 and Oxazin-6

Dattatraya H. Dethe,* Alok Ranjan and Vijendra H. Pardeshi

Organic Chemistry Division, National Chemical Laboratory, Pune – 411008, Maharashtra,
India

Tel: + 91-20-25902336, fax: +91-20-25902629

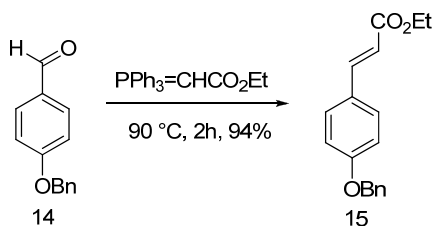
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General Information

All reactions were carried out under nitrogen atmosphere with dry solvents under anhydrous conditions, unless otherwise mentioned. All the chemicals were purchased commercially, and used without further purification. Anhydrous THF and diethyl ether were distilled from sodium-benzophenone and dichloromethane was distilled from calcium hydride. Yields refer to chromatographically pure material, unless otherwise stated. Reaction were monitored by thin-layer chromatography (TLC) carried out on 0.25 mm Merck silica gel plates (60F-254) using UV light as a visualizing agent and p-anisaldehyde or ninhydrine stain, and heat as developing agents. Merck silica gel (particle size 100-200 and 230-400 mesh) was used for flash column chromatography. Optical rotations were measured with JASCO P-2000 digital polarimeter at room temperature using 50 mm cell of 1 ml capacity. Infrared spectra were recorded on a Perkin-Elmer Spectrum One FT-IR spectrometer. Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. NMR spectra were recorded on either a Bruker Avance 200 (^1H : 200 MHz, ^{13}C : 50 MHz), Bruker Avance 400 (^1H : 400 MHz, ^{13}C : 100 MHz), Bruker Avance 500 (^1H : 500 MHz, ^{13}C : 125 MHz). Mass spectrometric data were obtained using QTOF-Micromass-UK. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of a doublet, ddd = doublet of a doublet of a doublet, dt = doublet of a triplet, m = multiplet, br = broad, td = triplet of a doublet.

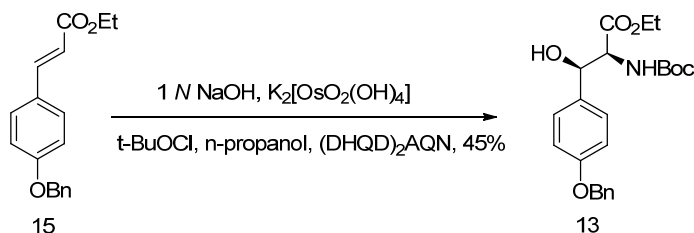
(E)-ethyl 3-(4-(benzyloxy)phenyl)acrylate (15):



To a solution of the aldehyde **14** (8.0 g, 37.7 mmol) in water (150 ml) was added $\text{PPh}_3=\text{CHCO}_2\text{Et}$ salt (19.7 g, 56.5 mmol) and heated it to $90\text{ }^\circ\text{C}$ for 2h. The reaction mixture was cooled to RT and extracted with CH_2Cl_2 (3 x 80 ml), washed with brine and dried over Na_2SO_4 . Evaporation of the solvent and purification of the residue on a silica gel column using EtOAc-petroleum ether (1:9) as eluent furnished the ester **15** (*E/Z*-ratio: 95/5, 10.0 g, 94%) as white solid, $R_f = 0.30$ (EtOAc-petroleum ether, 1:9).

IR (neat): $\nu_{\text{max}}/\text{cm}^{-1}$ 3282, 1682 (C=O), 1632, 1603, 1585, 1515, 1439, 1371, 1279, 1189, 1033, 830; **^1H NMR** (200 MHz, CDCl_3): δ 7.64 (d, J 16.0 Hz, 1H) 7.53-7.29 (m, 7H), 6.97 (td, J 8.7 and 2.0 Hz, 2H), 6.31 (d, J 16.0 Hz, 1H), 5.10 (s, 2H), 4.25 (q, J 14.3 and 7.1 Hz, 2H), 1.33 (t, J 7.1 Hz, 3H); **^{13}C NMR** (50 MHz, CDCl_3): δ 167.3, 160.5, 144.2, 136.5, 129.7, 128.6, 128.1, 127.4, 115.9, 115.2, 70.1, 60.3, 14.3; **HRMS-ESI:** calcd for $\text{C}_{18}\text{H}_{19}\text{O}_3$ ($\text{M}+\text{H}$) 283.1334; found 283.1330.

Ethyl (2*S*,3*R*)-3-(4-(benzyloxy)phenyl)-2-((tert-butoxycarbonyl)amino)-3-hydroxypropionate (13):

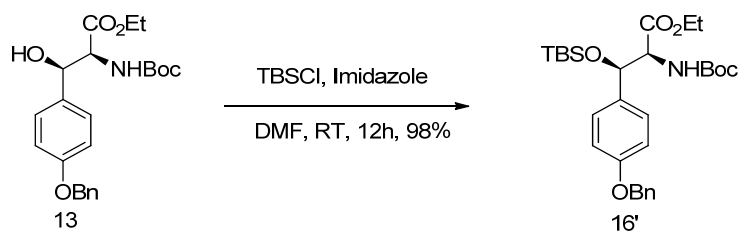


A round bottom flask was charged with sodium hydroxide (30.5 ml of 1 N solution) and diluted with water (45 ml) in a dark fume hood. Part of this alkaline solution (5.00 ml) was transferred into a vial to dissolve $\text{K}_2[\text{OsO}_2(\text{OH})_4]$ (147 mg, 0.4 mmol). With vigorous stirring, *n*-propanol (40.0 ml) and tert-butyl carbamate (3.63 g, 31.0 mmol) were added to the flask, followed by dropwise addition of freshly prepared *t*-butyl hypochlorite (3.46 ml, 3.05 mmol). After five minutes, *n*-propanol solution (35 ml) of $(\text{DHQD})_2\text{AQN}$ (343 mg, 0.4 mmol) and ethyl cinnamate **15** (2.82 g, 1.0 mmol), and the aqueous $\text{K}_2[\text{OsO}_2(\text{OH})_4]$ solution

were added. After 2 h, the solution was quenched with 5 g sodium bisulfite, added water and extracted with ethyl acetate (3 x 50 ml), washed with brine and dried over Na₂SO₄. Evaporation of the solvent and purification of the residue on a flash silica gel column using ethyl acetate-petroleum ether (1:4) furnished the ester **13** (1.61 g, 45%, 92% ee), *R*_f = 0.30 (EtOAc-petroleum ether 1:4).

$[\alpha]_D^{25} = -12.8$ (*c* 4.0, CHCl₃); **IR (neat)**: $\nu_{\max}/\text{cm}^{-1}$ 3421, 2930, 1716, 1690, 1512, 1392, 1244, 1162, 1162, 1025; **¹H NMR** (200 MHz, CDCl₃): δ 7.50-7.20 (m, 7H), 6.94 (td, *J* 8.7 and 2.0 Hz, 2H), 5.35 (d, *J* 8.8 Hz, 1H), 5.12 (t, *J* 3.4 Hz, 1H), 5.04 (s, 2H), 4.45 (br d, *J* 7.4 Hz, 1H), 4.18 (q, *J* 14.3 and 7.2 Hz, 2H), 2.9 (br s, 1H), 1.35 (s, 9H), 1.22 (t, *J* 7.1 Hz, 3H); **¹³C NMR** (50 MHz, CDCl₃): δ 171.0, 158.6, 155.8, 136.9, 132.4, 128.6, 128.0, 127.5, 127.4, 114.7, 80.1, 73.7, 70.0, 61.7, 59.7, 28.2, 14.1; **HRMS-ESI**: calcd for C₂₃H₃₀NO₆ (M+H) 416.2073; found 416.2086.

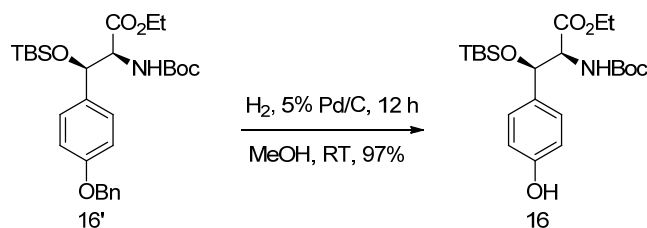
(2S,3R)-ethyl 3-(4-(benzyloxy)phenyl)-2-((tert-butoxycarbonyl)amino)-3-((tert-butyl dimethylsilyl)oxy)propanoate (16'**):**



To a solution of the alcohol **13** (4.0 g, 9.6 mmol) in DMF (25 ml) were added TBSCl (1.9 g, 12.5 mmol), imidazole (0.9 g, 14.4 mmol) and stirred magnetically at RT for 12 h. Water was added to the reaction mixture, extracted with ethyl acetate (3 x 20 ml), washed with brine and dried over Na₂SO₄. Evaporation of the solvent and purification of the residue on a silica gel column using ethyl acetate-petroleum ether (1:9) furnished the TBS ether **16'** (4.9 g, 98%) as colorless oil, *R*_f = 0.40 (EtOAc-petroleum ether 1:9).

$[\alpha]_D^{25} = -16.5$ (*c* 5.4, CHCl₃); **IR (neat)**: $\nu_{\max}/\text{cm}^{-1}$ 2930, 1720, 1643, 1510, 1367, 1250, 830, 778; **¹H NMR** (200 MHz, CDCl₃): δ 7.50-7.20 (m, 7H), 6.94 (dt, *J* 8.7 and 2.0 Hz, 2H), 5.30-5.10 (m, 2H), 5.05 (s, 2H), 4.38 (dd, *J* 7.5 and 2.5 Hz, 1H), 4.30-4.10 (m, 2H), 1.35 (s, 9H), 1.31 (t, *J* 7.1 Hz, 3H), 0.89 (s, 9H), 0.00 (s, 3H), -0.15 (s, 3H); **¹³C NMR** (50 MHz, CDCl₃): δ 170.0, 158.3, 154.9, 137.1, 133.0, 128.6, 127.9, 127.5, 114.3, 79.6, 75.2, 69.9, 61.0, 60.9, 53.5, 28.4, 25.8, 18.2, 14.1, -4.8, -5.3; **HRMS-ESI**: calcd for C₂₉H₄₄NO₆Si (M+H) 530.2938; found 530.2941.

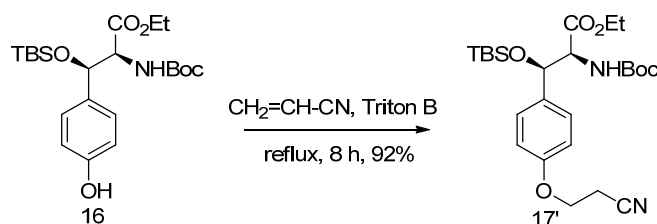
Ethyl (2*S*,3*R*)-2-((tert-butoxycarbonyl)amino)-3-((tert-butyldimethylsilyl)oxy)-3-(4-hydroxyphenyl)propanoate (16**):**



To a solution of the ester **16'** (2 g, 3.8 mmol) in methanol was added 5% Pd/C (40 mg) and shaken in hydrogenation atmosphere at RT for 12 h. The reaction mixture was filtered on celite using ethyl acetate. Evaporation of the solvent and purification of the residue on a silica gel column using ethyl acetate-petroleum ether (1:4) as eluent furnished hydroxy ester **16** (1.6 g, 97%) as a colorless oil, $R_f = 0.30$ (EtOAc-petroleum ether 1:4).

$[\alpha]_D^{25} = -17.8$ (c 2.3, CHCl_3); **IR** (neat): $\nu_{\text{max}}/\text{cm}^{-1}$ 3434, 3020, 2859, 1694, 1616, 1516, 1369, 1169, 1084, 834; **$^1\text{H NMR}$** (400 MHz, CD_3CN): δ 7.22 (d, J 8.5 Hz, 2H), 6.97 (s, 1H), 6.79 (d, J 8.5 Hz, 2H), 5.50 (br d, J 9.3 Hz, 1H), 5.22 (s, 1H), 4.30-4.05 (m, 3H), 1.36 (s, 9H), 1.27 (t, J 7.0 Hz, 3H), 0.89 (s, 9H), 0.01 (s, 3H), -0.15 (s, 3H); **$^{13}\text{C NMR}$** (100 MHz, CD_3CN): δ 172, 157.9, 156.9, 133.7, 129.1, 116.0, 80.4, 75.6, 62.6, 62.3, 28.9, 26.4, 19.0, 14.8, -4.0, -4.8; **HRMS-ESI**: calcd for $\text{C}_{22}\text{H}_{38}\text{NO}_6\text{Si}$ ($\text{M}+\text{H}$) 440.2468; found 440.2467.

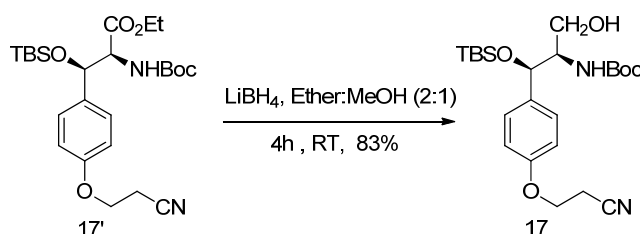
Ethyl (2*S*, 3*R*)-2-((tert-butoxycarbonyl)amino)-3-((tert-butyldimethylsilyl)oxy)-3-(4-(2-cyanoethoxy)phenyl)propanoate (17'**):**



To a solution of the phenol **16** (1.0 g, 2.3 mmol) in acrylonitrile (10 ml) was added catalytic amount of triton B and refluxed for 8 h. Excess acrylonitrile was removed under reduced pressure and the reaction mixture was diluted with water, extracted with CH_2Cl_2 , washed with brine and dried over Na_2SO_4 . Evaporation of the solvent and purification of the residue on silica gel column using ethyl acetate-petroleum ether (1:4) as eluent furnished the cyano ester **17'** (1.0 g, 92%). $R_f = 0.40$ (EtOAc-petroleum ether, 1:4).

$[\alpha]_D^{25} = -15.6$ (*c* 2.0, CHCl_3); **IR** (neat): $\nu_{\text{max}}/\text{cm}^{-1}$ 3447, 3020, 2931, 2240, 1712, 1612, 1510, 1215, 1171, 1086, 757, 669; **$^1\text{H NMR}$** (200 MHz, CDCl_3): δ 7.30 (d, *J* 8.3 Hz, 2H), 6.87 (d, *J* 8.4 Hz, 2H), 5.35-5.00 (m, 2H), 4.40-4.05 (m, 5H), 2.82 (t, *J* 6.3 Hz, 2H), 1.35 (s, 9H), 1.32 (t, *J* 7.2 Hz, 3H), 0.89 (s, 9H), 0.00 (s, 3H), -0.15 (s, 3H); **$^{13}\text{C NMR}$** (50 MHz, CDCl_3): δ 170.5, 157.1, 155.3, 133.9, 127.5, 117.1, 114.1, 79.5, 74.2, 62.5, 61.3, 60.5, 28.1, 25.5, 18.4, 17.9, 13.9, -4.8, -5.7; **HRMS-ESI**: calcd for $\text{C}_{25}\text{H}_{41}\text{N}_2\text{O}_6\text{Si}$ ($\text{M}+\text{H}$) 493.2734; found 493.2727.

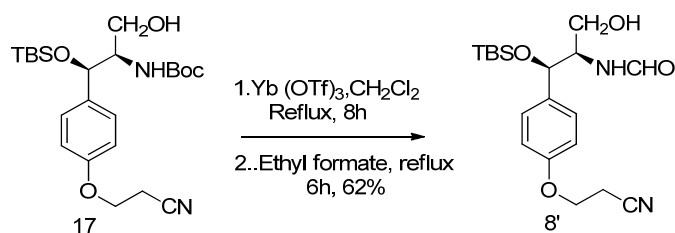
Tert-butyl((1*R*,2*R*)-1-(4-(2-cyanoethoxy)phenyl)-3-hydroxy-1-((tri methyl silyl)oxy)-propan-2-yl)carbamate (17):



To a magnetically stirred solution of the cyano ester **17'** (2 g, 4.1 mmol) in ether/methanol (20:10 ml) was added LiBH_4 (0.45 g, 20.5 mmol) and stirred for 4 h at RT. Solvent was evaporated under reduced pressure, water (15 ml) was added to the residue and extracted with ether (3 x 20 ml). The combined extract was washed with brine (15 ml) and dried over Na_2SO_4 . Evaporation of the solvent and purification of the residue on a silica gel column using ethyl acetate-petroleum ether (1:2) as eluent furnished the alcohol **17** (1.5 g, 83%), *R_f* = 0.30 (EtOAc-petroleum ether, 1:2).

$[\alpha]_D^{25} = -22.2$ (*c* 4.2, CHCl_3); **IR** (neat): $\nu_{\text{max}}/\text{cm}^{-1}$ 3442, 3020, 2931, 2255, 1700, 1510, 1392, 1367, 1216, 1172, 1086, 838; **$^1\text{H NMR}$** (200 MHz, CDCl_3): δ 7.25 (d, *J* 8.6 Hz, 2H), 6.86 (d, *J* 8.6 Hz, 2H), 5.0-4.8 (m, 2H), 4.18 (t, *J* 6.3 Hz, 2H), 3.75-3.50 (m, 3H), 2.83 (t, *J* 6.3 Hz, 2H), 1.37 (s, 9H), 0.90 (s, 9H), 0.06 (s, 3H), -0.16 (s, 3H); **$^{13}\text{C NMR}$** (50 MHz, CDCl_3): δ 156.9, 156.1, 135.0, 127.6, 117.2, 114.2, 79.5, 72.9, 62.7, 62.5, 58.5, 28.2, 25.7, 18.5, 18.0, -4.7, -5.3; **HRMS-ESI**: calcd for $\text{C}_{23}\text{H}_{39}\text{N}_2\text{O}_5\text{Si}$ ($\text{M}+\text{H}$) 451.2628; found 451.2620.

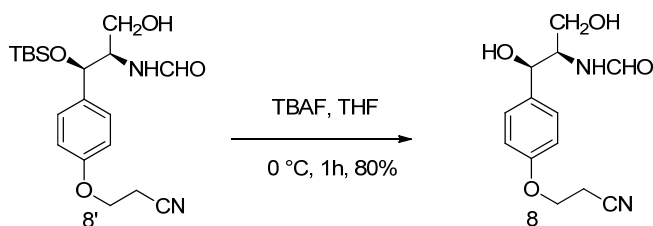
***N*-((1*R*,2*R*)-1-(4-(2-cyanoethoxy)phenyl)-3-hydroxy-1-((trimethylsilyl)oxy)propan-2-yl)formamide (8') :**



To a solution of boc-amine **17** (80.0 mg, 0.18 mmol) in CH₂Cl₂ (8 ml) was added Yb(OTf)₃ (110 mg, 0.18 mmol) and refluxed for 8 h. It was quenched with NaHCO₃ and concentrated on vacuo. The residue was dissolved in ethyl formate (6 ml) and refluxed for 6 h. Evaporation of the solvent and purification of the residue on a silica gel column using MeOH-CH₂Cl₂ (1:19) furnished TBS-protected bursatellin **8'** (41.6 mg, 62%, 2 steps) as colorless oil, *R_f* = 0.30 (MeOH-CH₂Cl₂ 1:19).

[α]_D²⁵ = -15.1 (*c* 4.4, CHCl₃); IR (neat): ν_{max} /cm⁻¹ 3358, 2928, 2856, 2280, 1746, 1672, 1512, 1389, 1245, 1080, 1047, 837; ¹H NMR (500 MHz, CDCl₃, peaks due to major rotamer): δ 8.14 (s, 1H), 7.25 (d, *J* 15.0 Hz, 2H), 6.86 (d, *J* 8.8 Hz, 1H), 6.10 (d, *J* 7.3 Hz, 1H), 4.97 (d, *J* 3.3 Hz, 1H), 4.19 (t, *J* 6.5 Hz, 2H), 4.07-4.01 (m, 1H), 3.68 (d, *J* 5.8 Hz, 2H), 2.84 (t, *J* 6.0 Hz, 2H), 0.91 (s, 9H), 0.08 (s, 3H), -0.14 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 165.0, 161.6, 157.3, 134.7, 127.8, 127.4, 117.2, 114.5, 114.5, 73.5, 72.3, 63.0, 62.6, 60.8, 57.2, 25.8, 25.8, 18.7, 18.2, -4.6, -5.2; HRMS-ESI: calcd for C₁₉H₃₀N₂O₄SiNa (M+Na) 401.1873; found 401.1869.

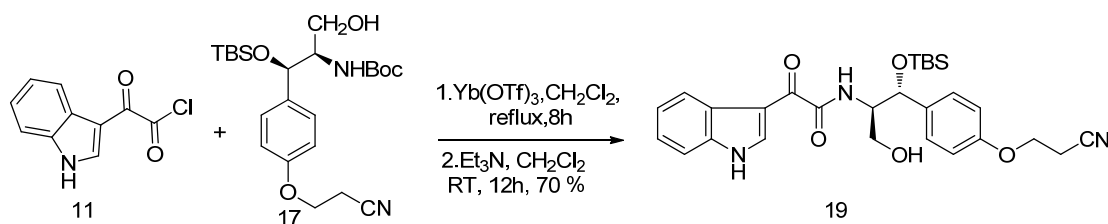
Synthesis of (-)-bursatellin (**8**):



To a magnetically stirred solution of the TBS protected bursatellin **8'** (20 mg, 0.05 mmol) in THF (2 ml) was added 1 M solution of TBAF in THF (0.05 ml, 0.05 mmol) and stirred for 1 h at 0 °C. Solvent was evaporated under reduced pressure, water (3 ml) was added to the residue and extracted with CH₂Cl₂ (3 x 8 ml). The combined extract was washed with brine (6 ml) and dried over Na₂SO₄. Evaporation of the solvent and purification of the residue on a silica gel column using MeOH-CH₂Cl₂ (1:9) as eluent furnished the (-)-bursatellin (**8**) (11.2 mg, 80%), *R_f* = 0.40 (MeOH:CH₂Cl₂ 1:9).

$[\alpha]_D^{25} = -8.6$ (*c* 0.6, MeOH); **IR** (neat): $\nu_{\max}/\text{cm}^{-1}$ 3360, 2977, 2240, 1660, 1450, 1377, 1174, 1089; **¹H NMR** (400 MHz, CD₃OD): δ 7.90 (s, 1H), 7.23 (d, *J* 8.5 Hz, 2H), 6.82 (d, *J* 8.5 Hz, 2H), 4.80 (d, *J* 3.6 Hz, 1H), 4.07 (t, *J* 6.0 Hz, 2H), 4.01-3.96 (m, 1H), 3.54 (dd, *J* 10.8 and 6.2 Hz, 1H), 3.38 (dd, *J* 10.8 and 6.0 Hz, 1H), 2.80 (t, *J* 6.0 Hz, 2H); **¹³C NMR** (100 MHz, CD₃OD): δ 164.0, 159.0, 136.7, 128.6, 119.0, 115.4, 71.9, 64.2, 62.5, 57.1, 19.0;

***N*-((1*R*,2*R*)-1-((*tert*-butyldimethylsilyloxy)-1-(4-(2-cyanoethoxy)phenyl)-3-hydroxy propan-2-yl)-2-(1*H*-indol-3-yl)-2-oxoacetamide (**19**):**



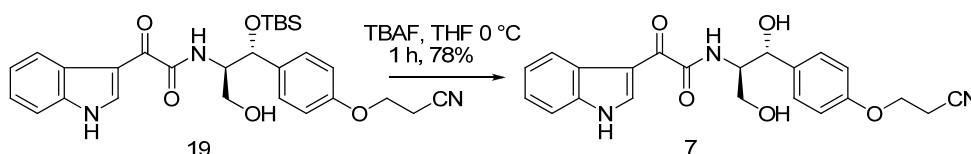
To a solution of indole (1 g, 8.6 mmol) in anhydrous diethyl ether (15 ml) at 0 °C, freshly distilled oxalyl chloride (0.8 ml, 9.5 mmol) was added dropwise. The reaction mixture was stirred at 0 °C for 0.5 h and then allowed to warm to RT. The resulting yellow crystals were collected by filtration, washed with cold anhydrous diethyl ether and dried under vacuum (1.63 g, 15.7 mmol). The yellow solid has been used without further purification.

To a solution of boc-amine **17** (100 mg, 0.2 mmol) in CH₂Cl₂ (10 ml) was added Yb(OTf)₃ (138 mg, 0.2 mmol) and refluxed for 8 h. Aq. NaHCO₃ was added to the reaction mixture and extracted with CH₂Cl₂ (3 x 10 ml). The organic layer was washed with brine and dried over Na₂SO₄. After evaporation of the solvent, the residue was dissolved in CH₂Cl₂, cooled to 0 °C. To the cold solution was added Et₃N (17 mg, 0.16 mmol) followed by 3-indoleglyoxalyl chloride **11** (40 mg, 0.14 mmol) and stirred magnetically at RT for 12 h. Water (10 ml) was added to the reaction mixture and extracted with CH₂Cl₂ (3 x 10 ml). Combined organic layers were washed with brine and dried over anhydrous Na₂SO₄. Evaporation of the solvent and purification of the residue on a silica gel column using MeOH:CH₂Cl₂ (1:19) furnished the TBS-protected preoxazin (**19**) (73 mg, 70%) as yellow solid.

$[\alpha]_D^{25} = -24.0$ (*c* 0.9, CHCl₃); **IR** (neat): $\nu_{\max}/\text{cm}^{-1}$ 3392, 3020, 2930, 2400, 2280, 1635, 1511, 1423, 1215, 1045, 929, 758, 669; **¹H NMR** (400 MHz, CDCl₃): δ 9.58 (s, 1H), 8.71 (dd, *J* 5.1 and 3.4 Hz, 1H), 8.39 (d, *J* 7.5 Hz, 1H), 7.92 (d, *J* 9.0 Hz, 1H), 7.40-7.20 (m, 5H), 6.73 (d, *J* 8.7 Hz, 2H), 5.00 (d, *J* 3.1 Hz, 1H), 4.15-4.05 (m, 1H), 4.02 (t, *J* 6.2 Hz, 2H), 3.85-3.70 (m, 2H), 2.72 (t, *J* 6.2 Hz, 2H), 0.92 (s, 9H), 0.07 (s, 3H), -0.15 (s, 3H); **¹³C NMR** (100 MHz,

CDCl₃): δ 180.3, 163.0, 157.0, 138.5, 135.8, 134.7, 127.4, 126.5, 124.1, 123.3, 122.2, 117.4, 114.2, 112.9, 111.9, 72.73, 62.73, 62.36, 58.1, 25.8, 18.6, 18.1, -4.61, -5.25; **HRMS-ESI**: calcd for C₂₈H₃₆N₃O₅Si (M+H) 522.2424; found 522.2424.

Synthesis of preoxazin-7 (7):

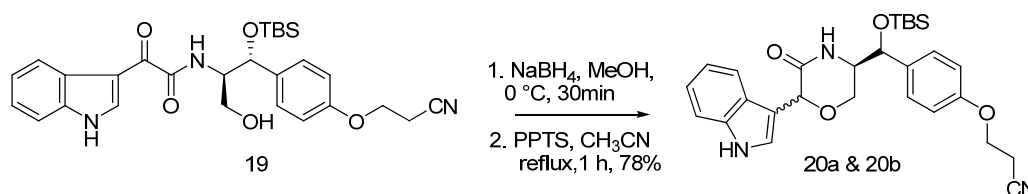


To the solution of **19** (80 mg, 0.2 mmol) in THF (5.0 ml) was added TBAF (1.0 M in THF, 0.2 ml, 0.2 mmol). The resulting mixture was stirred for 1 h at 0 °C, before it was concentrated in vacuo. Water (5 ml) was added to the residue and extracted with ether (3 x 20 ml). The combined extract was washed with brine (15 ml) and dried over Na₂SO₄. Evaporation of the solvent and purification of the residue on a silica gel column using ethyl acetate-petroleum ether (9:1) as eluent furnished the preoxazin-7 (**7**) (48.7 mg, 78 %) as a white solid, *R_f* = 0.30 (EtOAc-petroleum ether, 9:1).

$[\alpha]_D^{25} = -11$ (*c* 0.6, MeOH); **IR** (neat): $\nu_{\max}/\text{cm}^{-1}$ 3480, 3340, 3162, 2977, 2270, 1660, 1622, 1450, 1377, 1174, 1089, 929; **¹H NMR** (400 MHz, CD₃CN): δ 10.12 (br s, 1H), 8.81 (s, 1H), 8.31 (d, *J* 9.1 Hz, 1H), 7.79 (br s, 1H), 7.54 (d, *J* 9.1 Hz, 1H), 7.30 (dd, *J* 9.1 and 7.3 Hz, 2H), 7.29 (dd, *J* 9.1 and 7.3 Hz, 2H), 6.88 (d, *J* 8.9 Hz, 2H), 5.00 (t, *J* 3.6 Hz, 1H), 4.14 (t, *J* 6.2 Hz, 2H), 4.10-4.00 (m, 1H), 3.86 (d, *J* 4.0 Hz, 1H), 3.70 (dd, *J* 11.4 and 5.6 Hz, 1H), 3.63 (dd, *J* 11.4 and 5.6 Hz, 2H), 2.81 (t, *J* 6.2 Hz, 2H); **¹³C NMR** (100 MHz, CD₃CN): δ 181.8, 163.6, 158.4, 139.9, 137.2, 136.5, 128.5, 127.6, 124.9, 124.0, 122.7, 119.3, 115.3, 113.5, 113.3, 72.5, 64.0, 63.1, 57.8, 19.2,

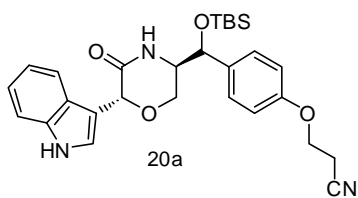
HRMS-(ESI): calcd for C₂₂H₂₁N₃O₅Na (M+Na) 430.1379; found 430.1382.

3-(4-((R)-((3R,6R)-6-(1H-indol-3-yl)-5-oxomorpholin-3-yl)((tert-butyldimethylsilyloxy) methyl)phenoxy)propanenitrile (**20a**) & 3-(4-((R)-((3R,6S)-6-(1H-indol-3-yl)-5-oxomorpholin-3-yl)((tert-butyldimethylsilyloxy) methyl)phenoxy)propanenitrile (**20b**) :



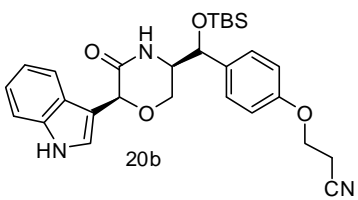
To a cold solution (0 °C) of **19** (104 mg, 0.2 mmol) in MeOH was added NaBH₄ (40 mg, 1 mmol) and stirred vigorously at the same temperature for 30 min. Excess MeOH was removed from reaction mixture under reduced pressure. Added water (10 ml), extracted with ethyl acetate (3 x 8 ml), washed with brine (10 ml) and dried over Na₂SO₄ and concentrated. To the crude alcohol was added acetonitrile (50 ml), catalytic (10 mg) pyridine para-toluene sulfonate (PPTS) and refluxed for 1 h. The reaction mixture was quenched with aq. NaHCO₃ (20 ml), extracted with ethyl acetate (3 x 8 ml), washed with brine and dried over Na₂SO₄. Evaporation of the solvent and purification of the residue using preparative thin layer chromatography furnished the diastereomers **20a** (23 mg) and **20b** (58 mg) (yield 78% for two steps).

TBS-protected oxazin-5 (**20a**):



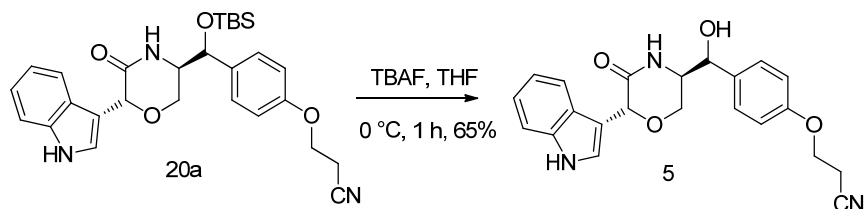
$[\alpha]_D^{25} = +19.1$ (*c* 2.0 CHCl₃); **IR** (neat): $\nu_{\max}/\text{cm}^{-1}$ 3396, 3269, 2928, 2856, 1668 (C=O), 1612, 1512, 1462, 1238, 1105, 858, 839, 780, 744, 618; **¹H NMR** (CDCl₃, 400 MHz): δ 8.35 (br s, 1H), 7.68 (d, *J* 7.8 Hz, 1H), 7.35-7.00 (m, 6H), 6.89 (d, *J* 8.6 Hz, 1H), 6.55 (br s, 1H), 5.51 (s, 1H), 4.63 (d, *J* 8.3 Hz, 1H), 4.18 (t, *J* 6.3 Hz, 2H), 3.70-3.55 (m, 2H), 3.41 (dd, *J* 11.5 and 5.0 Hz, 1H), 2.83 (t, *J* 6.3 Hz, 2H), 1.64 (br s, 1H), 0.91 (s, 9H), 0.10 (s, 3H), -0.19 (s, 3H); **¹³C NMR** (CDCl₃, 100 MHz): δ 169.4, 157.8, 136.4, 133.7, 128.2, 126.1, 124.6, 122.4, 120.0, 119.5, 117.1, 114.7, 111.3, 76.2, 73.8, 62.6, 61.5, 58.6, 25.8, 18.6, 18.1, -4.5, -5.0; **HRMS**: calcd for C₂₈H₃₆N₃O₄Si (M+H) 506.2475; found 506.2486.

Synthesis of TBS-protected oxazin-6 (**20b**):



$[\alpha]_D^{25} = -28.4$ (*c* 4.5, CHCl₃); **IR** (neat): $\nu_{\max}/\text{cm}^{-1}$ 3398, 2927, 2856, 1668 (C=O), 1611, 1511, 1459, 1248, 1097, 859, 838, 779, 743, 617; **¹H NMR** (CDCl₃, 500 MHz): δ 8.65 (br s, 1H), 7.71 (d, *J* 7.6 Hz, 1H), 7.35-7.00 (m, 6H), 6.81 (d, *J* 8.6 Hz, 2H), 6.62 (s, 1H), 5.51 (s, 1H), 4.61 (d, *J* 8.5 Hz, 1H), 4.12 (t, *J* 6.1 Hz, 2H), 3.70-3.50 (m, 2H), 3.50-3.40 (m, 1H), 2.79 (t, *J* 6.1 Hz, 2H), 0.90 (s, 9H), 0.09 (s, 3H), -0.21 (s, 3H). **¹³C NMR** (CDCl₃, 125 MHz): δ 169.7, 157.7, 136.5, 133.5, 128.2, 126.2, 124.9, 122.3, 119.9, 119.4, 117.1, 114.6, 111.5, 110.9, 76.1, 73.8, 62.5, 61.1, 58.6, 25.8, 18.6, 18.1, -4.5, -5.0; **HRMS**: calcd for C₂₈H₃₆N₃O₄Si (M+H) 506.2475; found 506.2463.

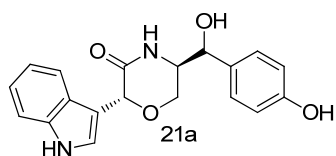
Oxazinin-5 (5) and (2R,5R)-5-((R)-(tert-butyldimethylsilyloxy)(4-hydroxyphenyl)methyl)-2-(1H-indol-3-yl)morpholin-3-one (21a):



To an ice cold (0 °C) solution of the compound **20a** (22 mg, 0.043 mmol) in anhydrous THF (2 ml) was added dropwise 1 M solution of TBAF in THF (0.08 ml, 0.08 mmol) and stirred magnetically for 1 h. The reaction mixture was quenched with aq. NaHCO₃ (5 ml), extracted with ethyl acetate (5 ml x 3), washed with brine and dried over Na₂SO₄. Evaporation of the solvent and purification of the residue using preparative TLC furnished oxazinin-5 (**5**) (11 mg, 65%) and the diol **21a** (2.5 mg, 15%) as white solid.

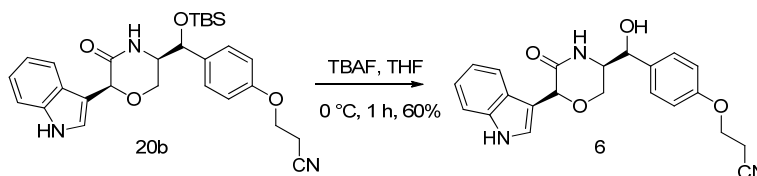
$[\alpha]_D^{25} = 6.9$ (*c* 0.4, MeOH); **IR** (neat): $\nu_{\max}/\text{cm}^{-1}$ 3360, 3302, 2924, 2853, 2257, 1660, 1609, 1513, 1456, 1306, 1240, 1177, 1106, 1049, 838, 749, 618; **¹H NMR** (CDCl₃, 400 MHz): δ 9.29 (br s, 1H), 7.58 (d, *J* 8.1 Hz, 1H), 7.40 (d, *J* 8.3 Hz, 1H), 7.34 (d, *J* 8.5 Hz, 2H), 7.27 (d, *J* 2.7 Hz, 1H), 7.15 (dd, *J* 8.1 and 7.1 Hz, 1H), 7.05 (dd, *J* 8.1 and 7.1 Hz, 1H), 6.96 (d, *J* 8.8 Hz, 1H), 5.29 (s, 1H), 4.58 (dd, *J* 8.1 and 4.2 Hz, 1H), 4.19 (t, *J* 6.1 Hz, 2H), 3.86 (d, *J* 4.1 Hz, 1H), 3.85-3.70 (m, 1H), 3.59 (dd, *J* 12.0 and 3.9 Hz, 1H), 3.50 (dd, *J* 12.0 and 7.6 Hz, 1H), 2.85 (t, *J* 6.1 Hz, 2H); **¹³C NMR** (CDCl₃, 100 MHz): δ 169.2, 158.0, 136.5, 134.1, 128.1, 126.1, 125.3, 121.8, 119.4, 118.2, 114.6, 112.0, 111.5, 73.9, 73.6, 63.1, 63.0, 57.5, 18.1; **HRMS**: calcd for C₂₂H₂₁N₃O₄Na (M+Na) 414.1430; found 414.1418.

Side chain cleaved compound (21a)



$[\alpha]_D^{25} = 5.3$ (*c* 0.6, MeOH); **IR** (neat): $\nu_{\max}/\text{cm}^{-1}$ 3362, 3169, 2923, 2853, 1653, 1612, 1463, 1186, 1108, 967, 838, 745, 618; **¹H NMR** (CDCl₃, 400 MHz): δ 9.30 (br s, 1H), 7.58 (d, *J* 8.0 Hz, 1H), 7.39 (d, *J* 8.0 Hz, 1H), 7.25 (s, 1H), 7.20 (d, *J* 7.3 Hz, 2H), 7.18-7.00 (m, 3H), 6.85-6.75 (m, 2H), 5.28 (s, 1H), 4.49 (dd, *J* 8.1 and 3.9 Hz, 1H), 3.85-3.75 (m, 2H), 3.56 (dd, *J* 12.0 and 3.9 Hz, 1H), 3.47 (dd, *J* 11.7 and 7.8 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz): δ 169.3, 156.9, 136.4, 132.2, 128.0 (2C), 126.2, 125.3, 121.8, 119.4 (2C), 115.2 (2C), 111.9, 74.1, 73.6, 63.2, 57.6; **HRMS**: *m/z* calcd for C₁₉H₁₈N₂O₄Na (M+Na) 361.1164; found 361.1181.

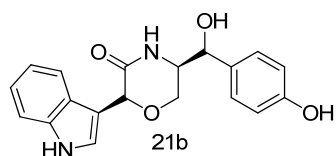
Oxazinin-6 (6) and (2S,5R)-5-((R)-(tert-butyldimethylsilyloxy)(4-hydroxyphenyl)methyl)-2-(1H-indol-3-yl)morpholin-3-one (21b):



To an ice cold (0 °C) solution of the compound **20b** (20 mg, 0.04 mmol) in anhydrous THF (2 ml) was added dropwise 1 M solution of TBAF in THF (0.08 ml, 0.08 mmol) and stirred magnetically for 1 h. The reaction mixture was quenched with aq. NaHCO₃ (5 ml), extracted with ethyl acetate (5 ml x 3), washed with brine and dried over Na₂SO₄. Evaporation of the solvent and purification of the residue using preparative TLC furnished oxazinin-6 (**6**) (9.3 mg, 60%) and the diol **21b** (3.7 mg, 24%) as white solid.

$[\alpha]_D^{25} = 9.00$ (*c* 0.5, MeOH); **IR** (neat): $\nu_{\max}/\text{cm}^{-1}$ 3360 (OH), 3163, 2924, 2857, 2256 (CN), 1661 (C=O), 1618, 1513, 1456, 1306, 1241, 1177, 1107, 1051, 838, 749, 618; **¹H NMR** (CDCl₃, 400 MHz): δ 9.37 (br s, 1H), 7.62 (d, *J* 7.8 Hz, 1H), 7.43 (d, *J* 8.0 Hz, 1H), 7.35-7.26 (m, 2H), 7.25-7.20 (m, 1H), 7.18-7.14 (m, 1H), 7.09-7.05 (m, 1H), 6.92 (d, *J* 8.3 Hz, 2H), 6.86 (br s, 1H), 5.38 (s, 1H), 4.71 (dd, *J* 7.8 and 3.9 Hz, 1H), 4.17 (t, *J* 6.1 Hz, 2H), 3.91 (d, *J* 3.9 Hz, 1H), 3.68-3.52 (m, 3H), 2.84 (t, *J* 6.1 Hz, 2H); **¹³C NMR** (CDCl₃, 100 MHz): δ 170.1, 158.8, 137.3, 135.4, 129.3, 127.3, 126.3, 122.7, 126.3, 122.7, 120.6, 120.2, 119.2, 115.6, 112.7, 112.5, 75.3, 74.5, 64.2, 62.7, 58.2, 19.0.

Side chain cleaved compound (21b)



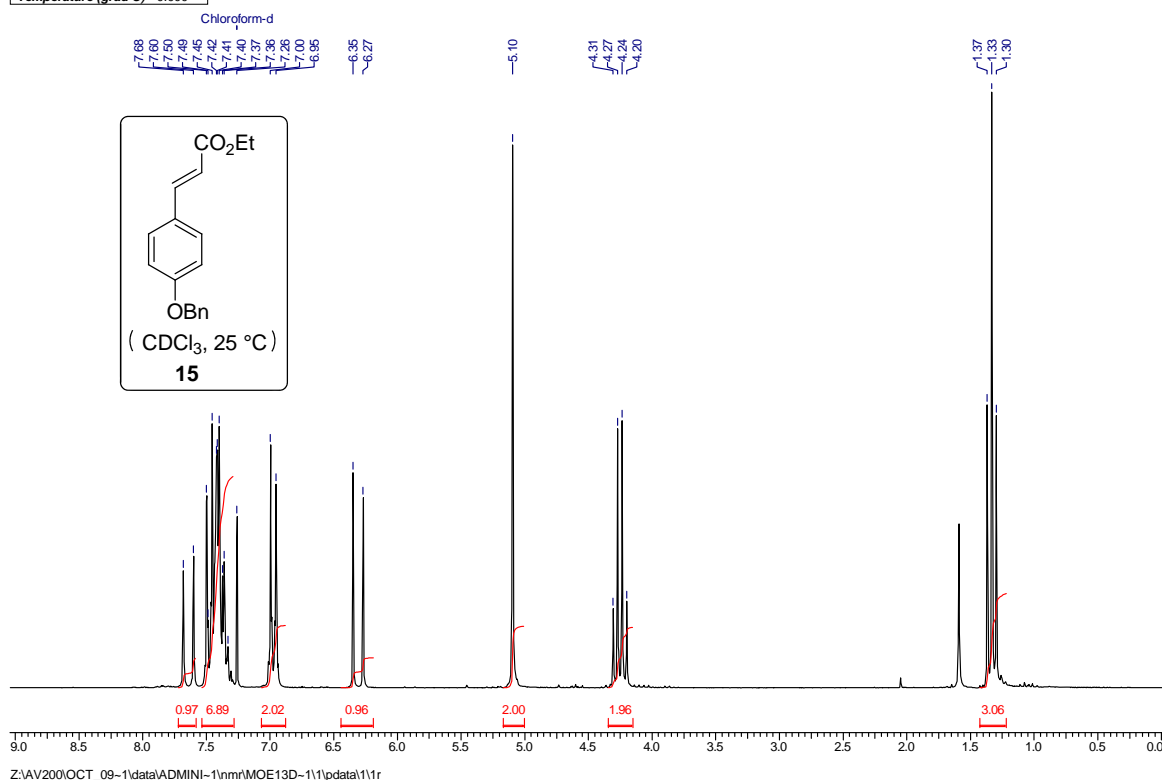
$[\alpha]_D^{25} = 7.00$ (*c* 0.6, MeOH); **IR** (neat): $\nu_{\max}/\text{cm}^{-1}$ 3360 (OH), 2923, 2853, 1652 (C=O), 1613, 1457, 1182, 1108, 966, 838, 745, 618; **¹H NMR** (CDCl₃, 400 MHz): δ 9.38 (br s, 1H), 7.63 (d, *J* 7.6 Hz, 1H), 7.43 (d, *J* 8.1 Hz, 1H), 7.30-7.00 (m, 6H), 6.87 (br s, 1H), 6.85-9.70 (m, 2H), 5.38 (s, 1H), 4.70-4.60 (m, 1H), 3.83 (br s, 1H), 3.65-3.50 (m, 3H).

¹³C NMR (CDCl₃, 100 MHz): δ 170.2, 157.8, 137.5, 133.4, 129.2, 127.5, 126.4, 122.9, 120.5, 120.4, 116.2, 112.7, 112.6, 75.6, 74.5, 62.8, 58.6; **HRMS**: calcd for C₁₉H₁₈N₂O₄Na (M+Na) 361.1164; found 361.1158.

NMR Spectrum :

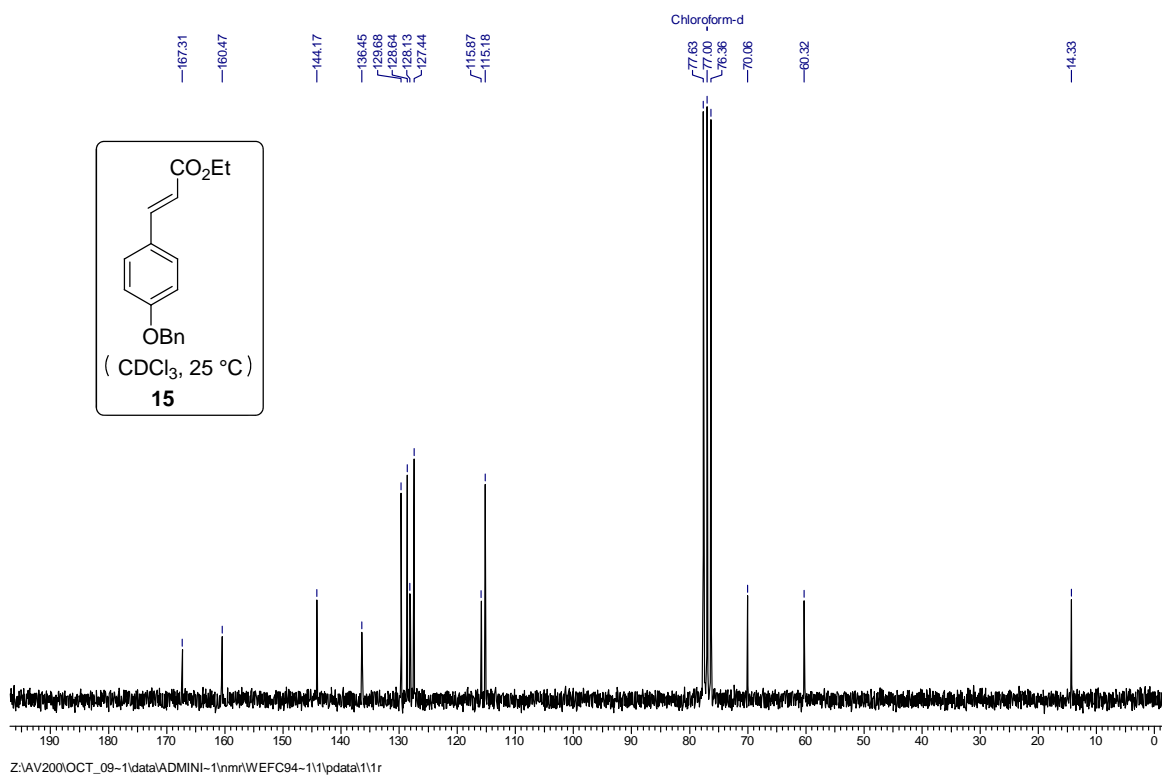
30 May 2011
 Alck

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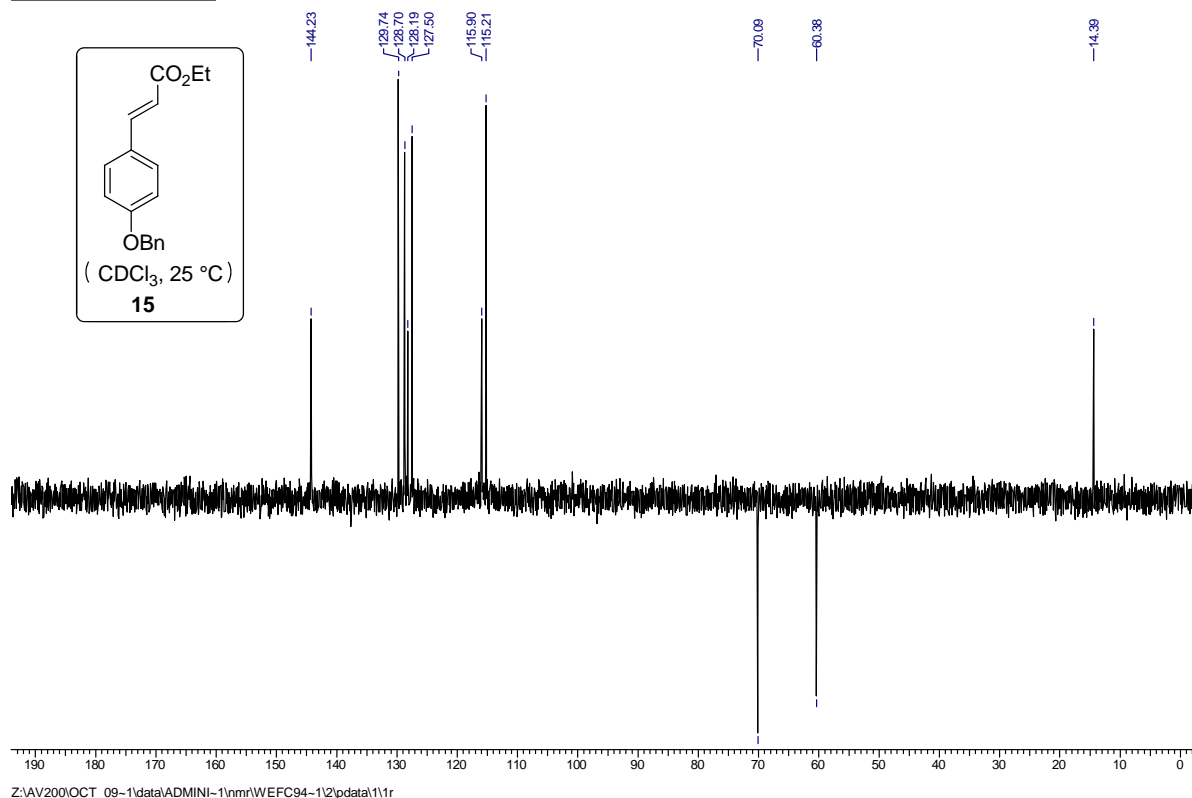
30 May 2011
 Alck

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| Temperature (grad C) | 0.000 | | | | | | |



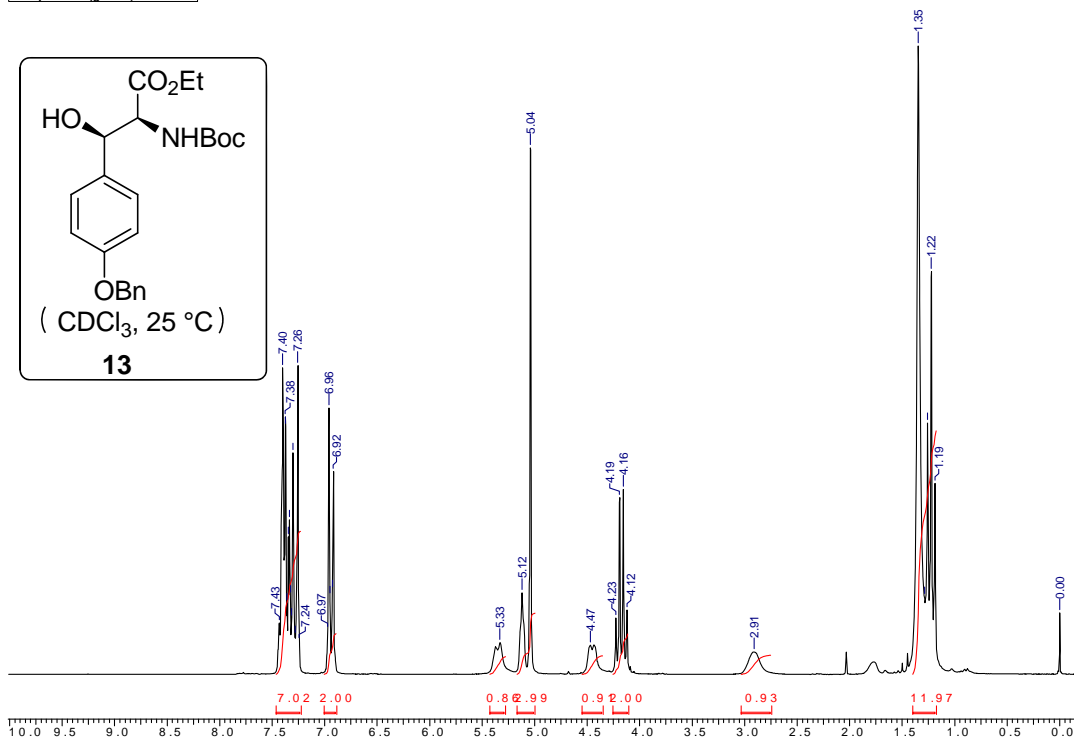
30 May 2011
 Alk

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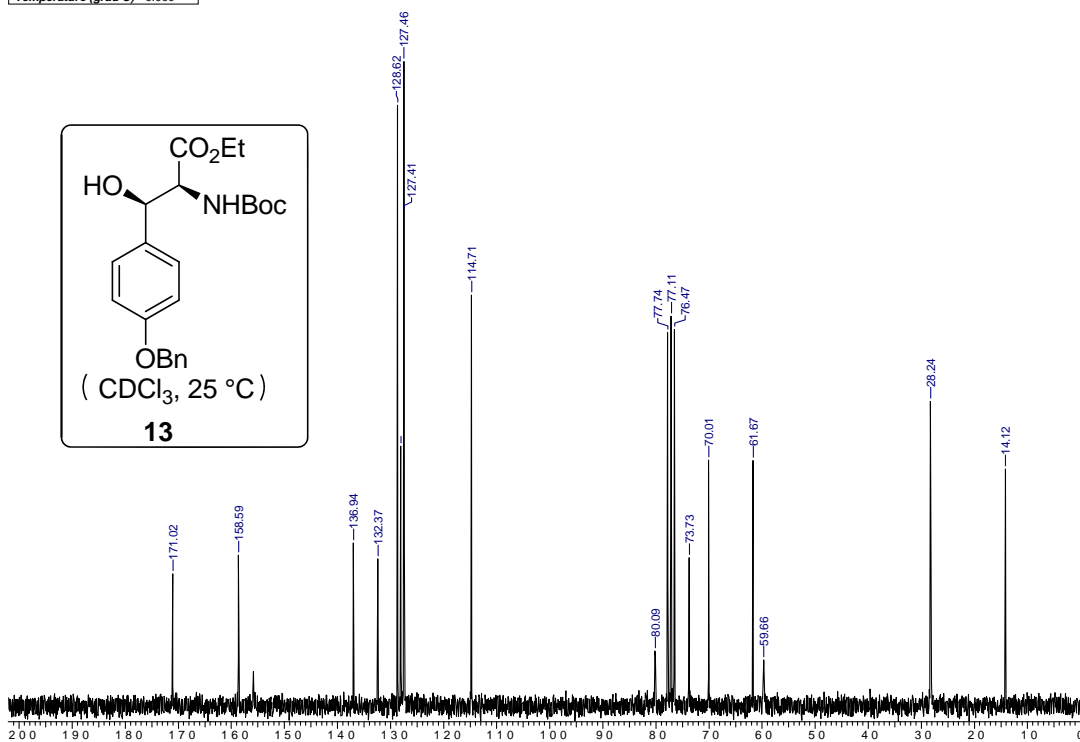
22 Dec 2010

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| | | | | Sweep Width (Hz) | 4139.07 |



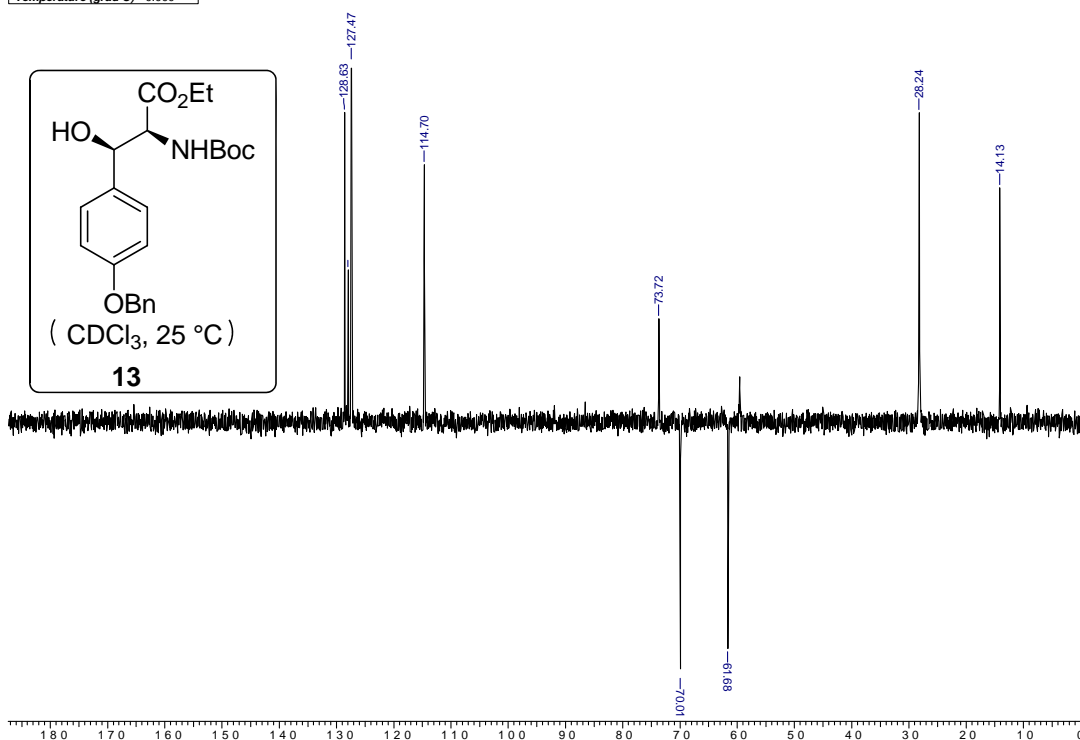
22 Dec 2010

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|------------------------|--------|---------|-----------------|-----------------------|---------------------|
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| Temperature (grad C) | 0.000 | | | Points Count | 32768 |
| | | | | Sweep Width (Hz) | 11990.41 |



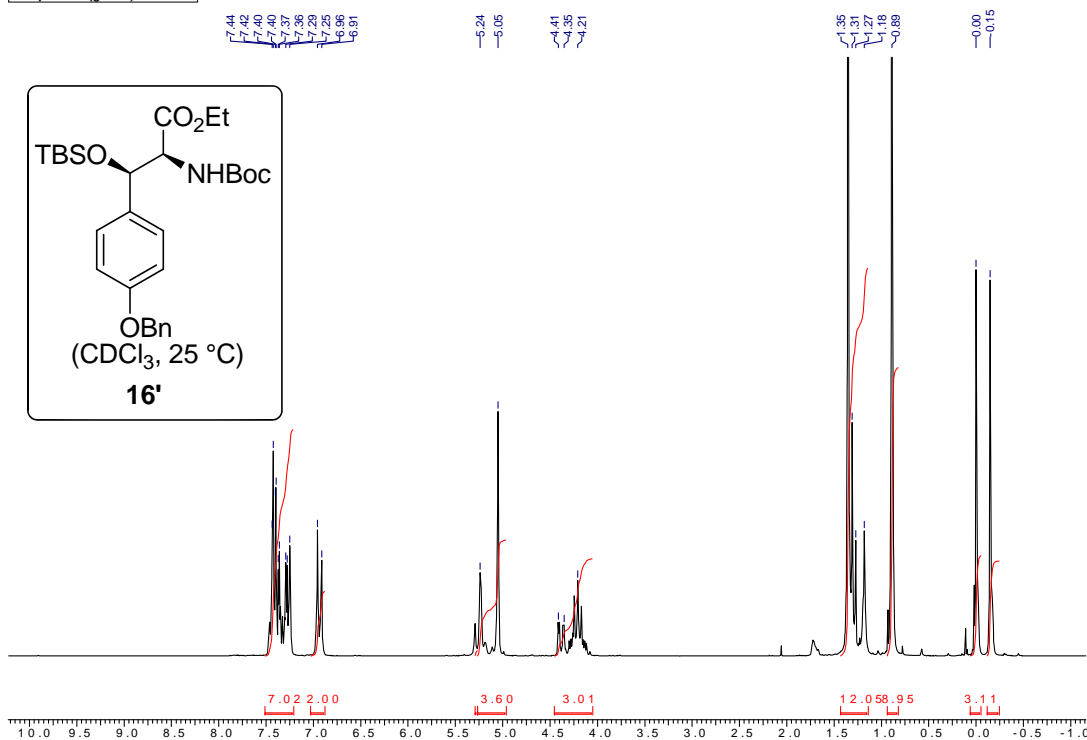
22 Dec 2010

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| | | | | Sweep Width (Hz) | 11990.41 |



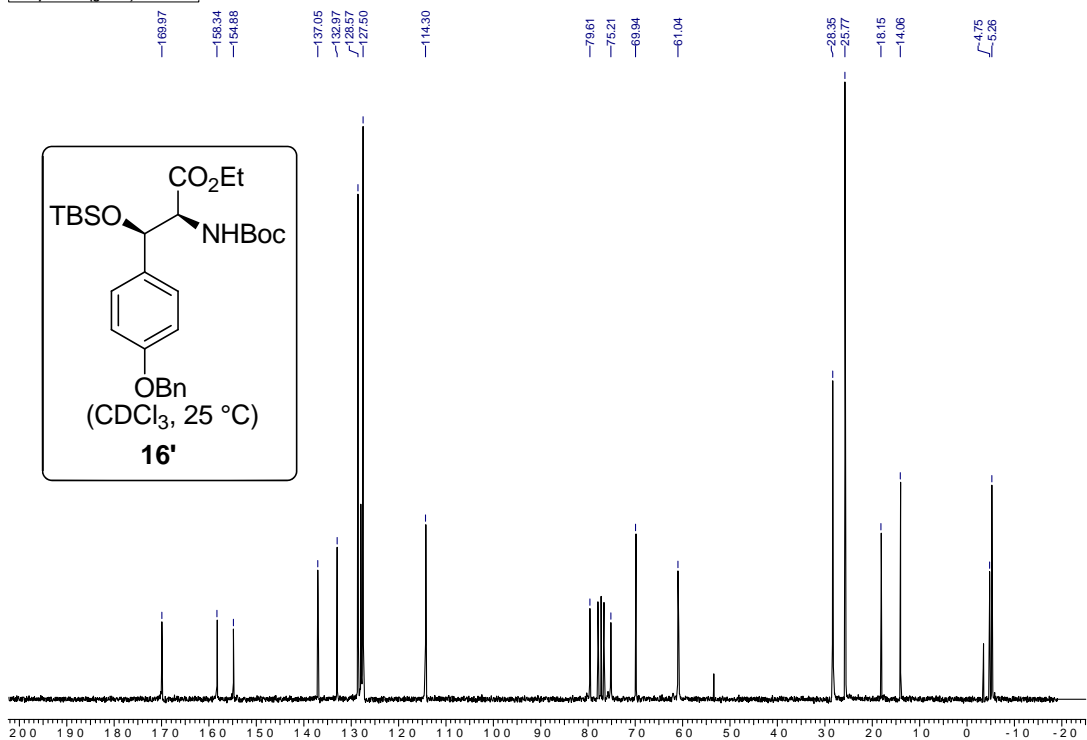
22 Dec 2010

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| Temperature (grad C) | 0.000 | | | Points Count | 32768 |
| | | | | Sweep Width (Hz) | 4139.07 |



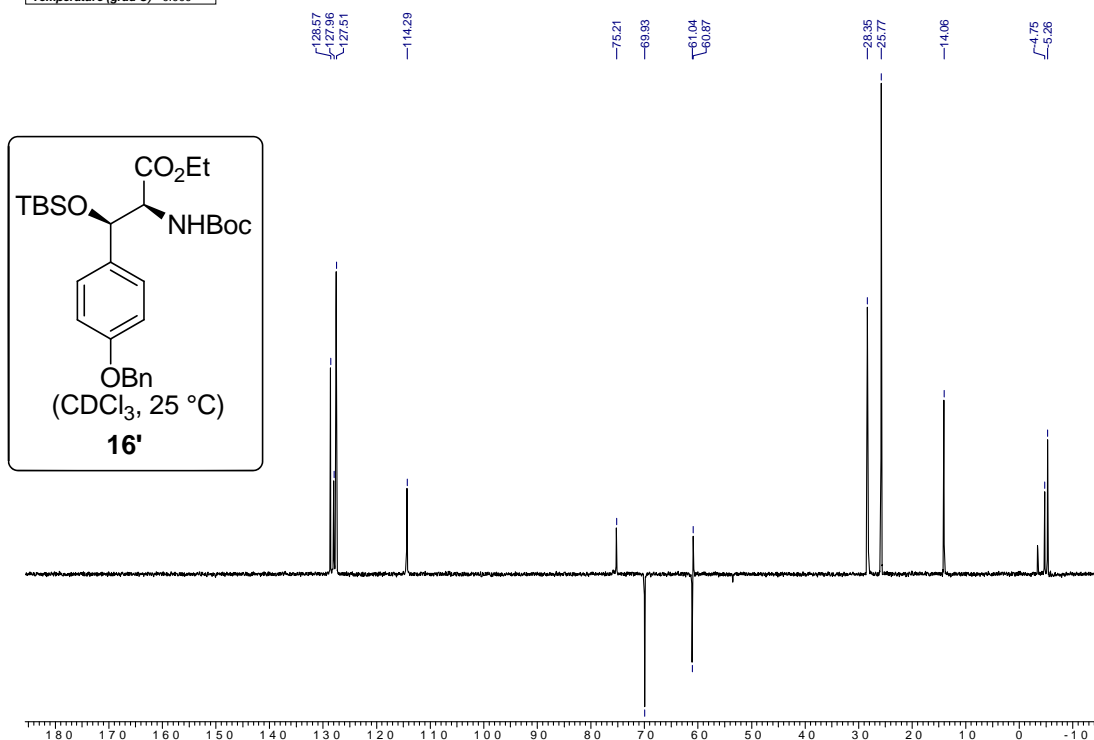
22 Dec 2010

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| Frequency (MHz) | 50.32 | Nucleus | ¹³ C | Original Points Count | 32768 |
| Temperature (grad C) | 0.000 | | | Points Count | 32768 |
| | | | | Sweep Width (Hz) | 11990.41 |



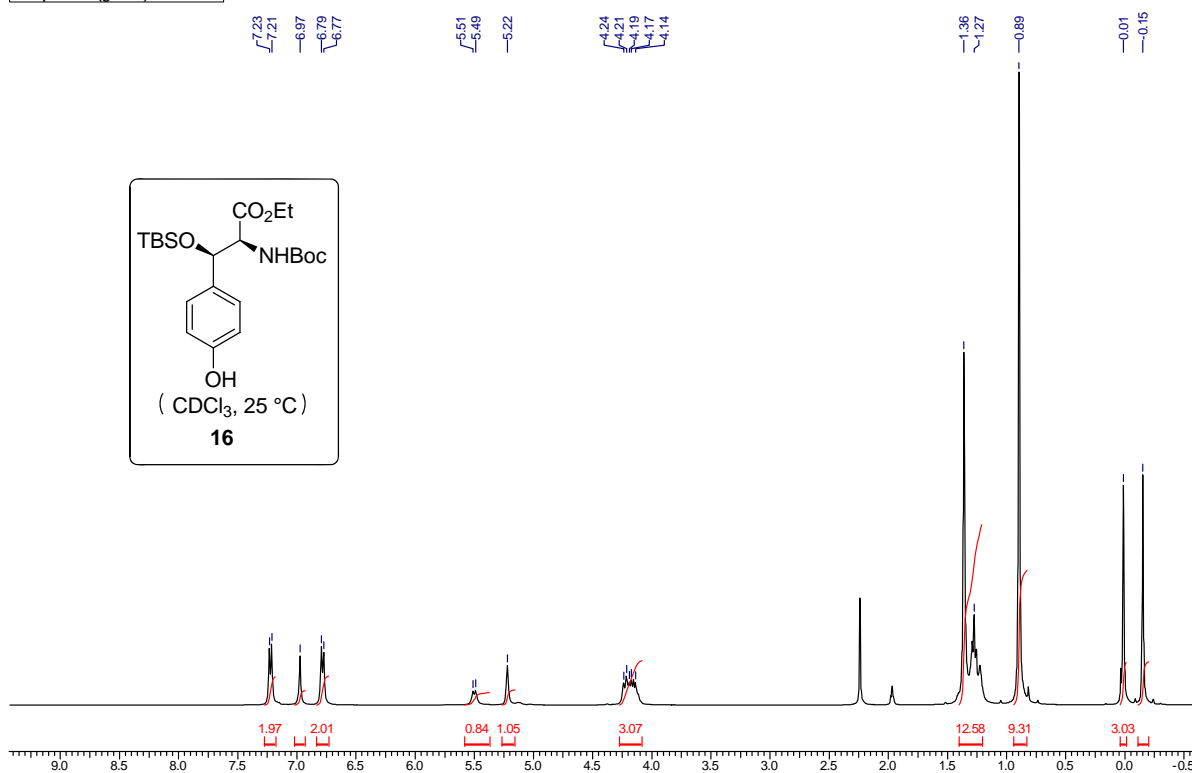
22 Dec 2010

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| Temperature (grad C) | 0.000 | | | | | | | | |



28 May 2011
 Alok/10997 (303K)

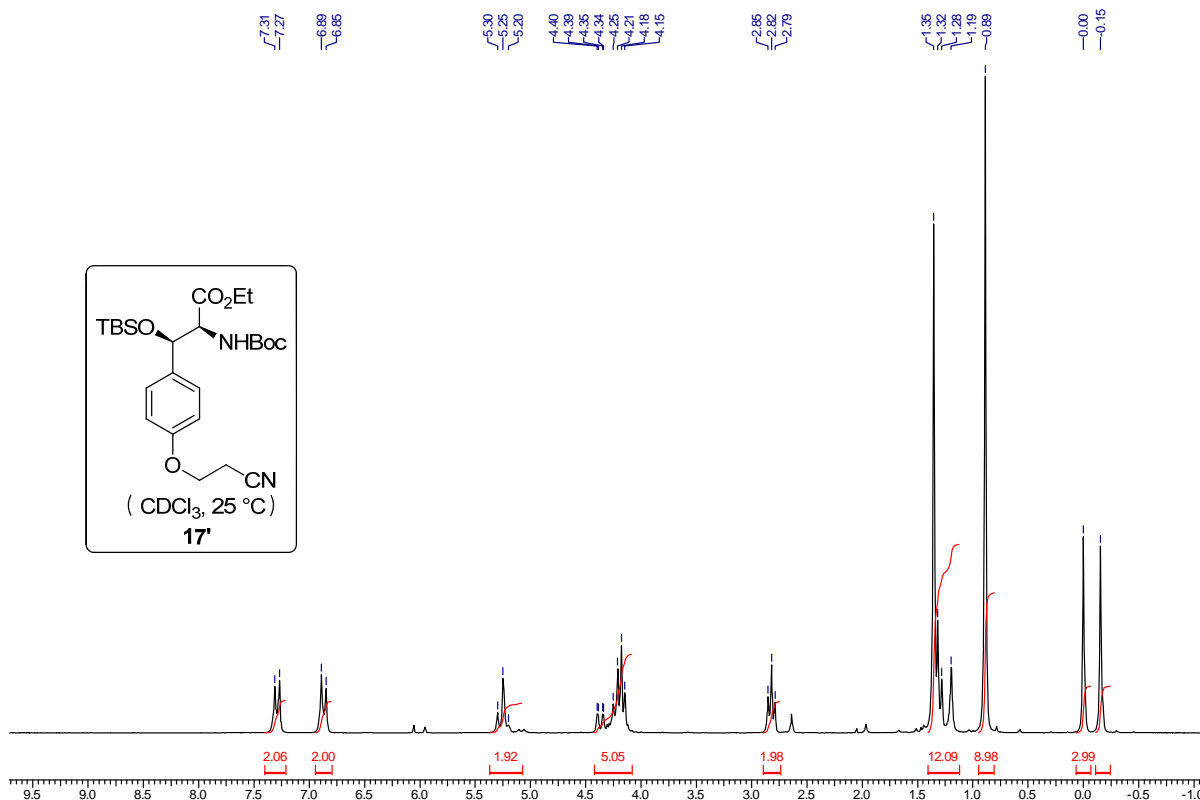
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| Temperature (grad C) | 0.000 | | | | | | | | |



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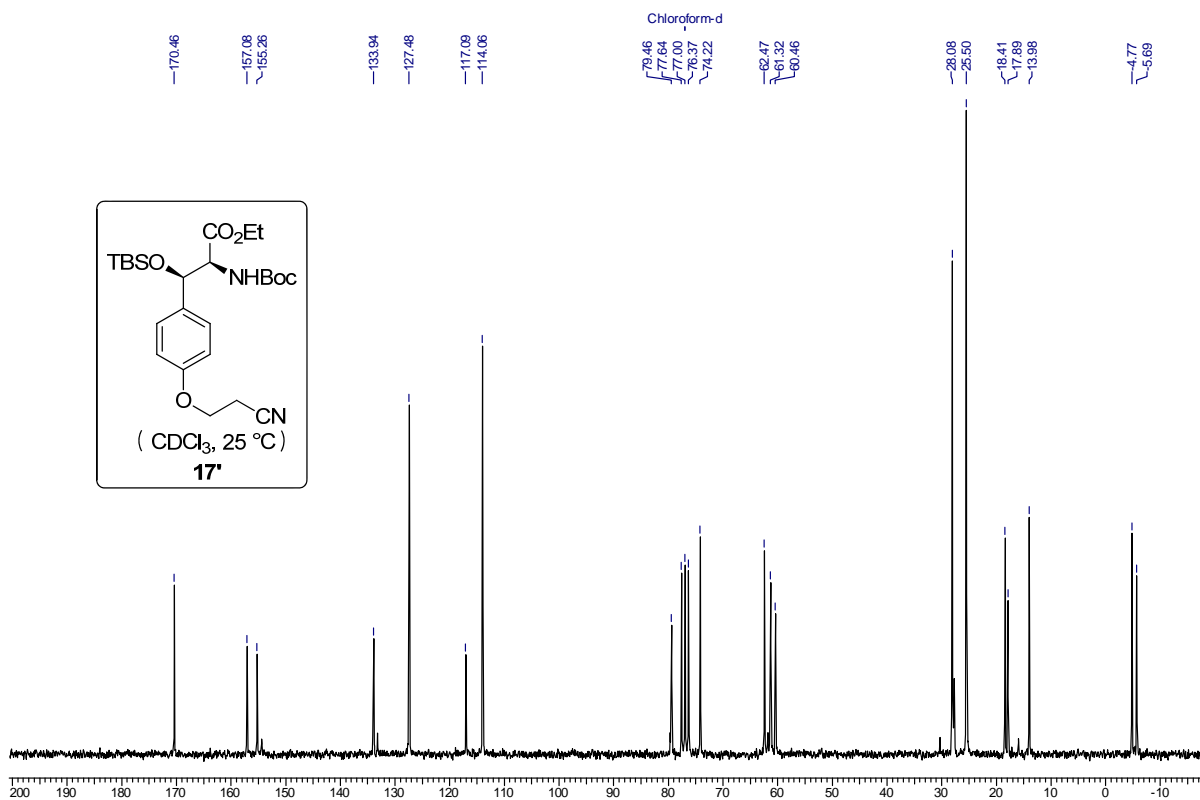
2 May 2011
 Alok/10997

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| Frequency (MHz) | 200.13 | Nucleus | 1H | Original Points Count | 32768 |
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| | | | | Sweep Width (Hz) | 4139.07 |



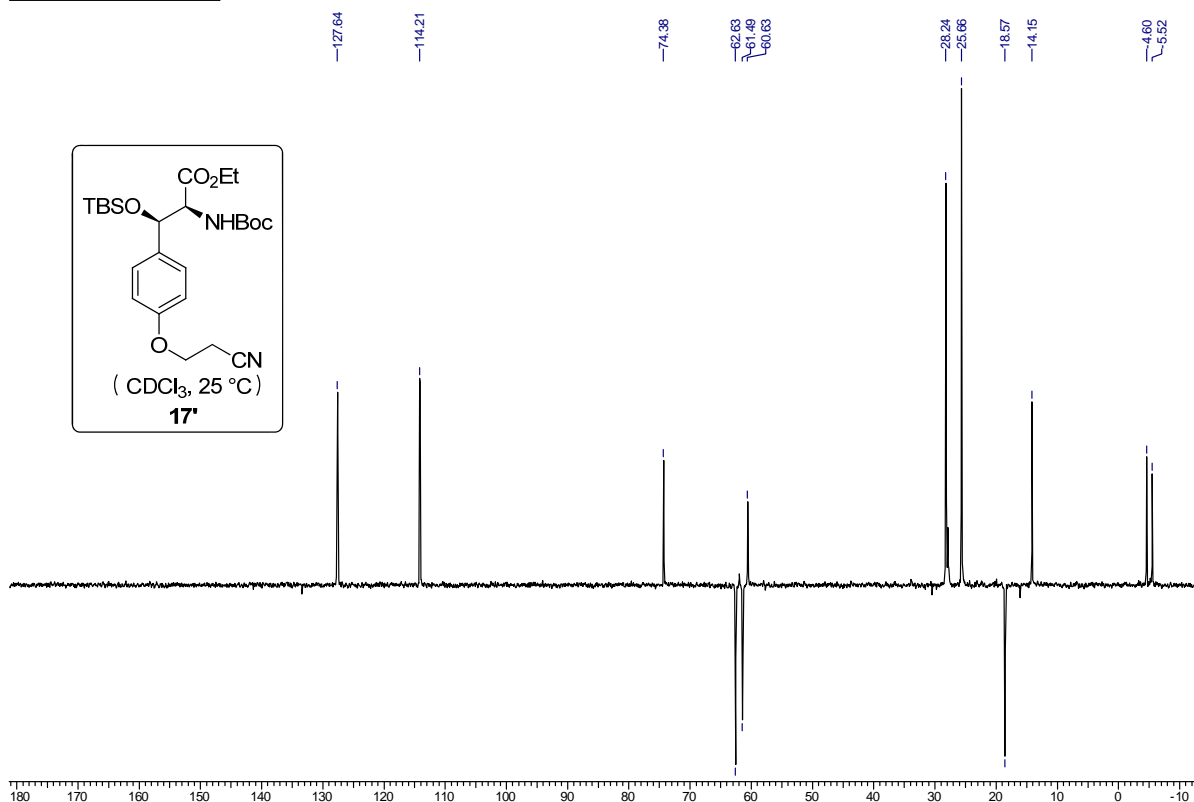
9 May 2011
 Alok/10997

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| Frequency (MHz) | 50.32 | Nucleus | 13C | Original Points Count | 32768 |
| Temperature (grad C) | 0.000 | | | Points Count | 32768 |
| | | | | Sweep Width (Hz) | 11990.41 |



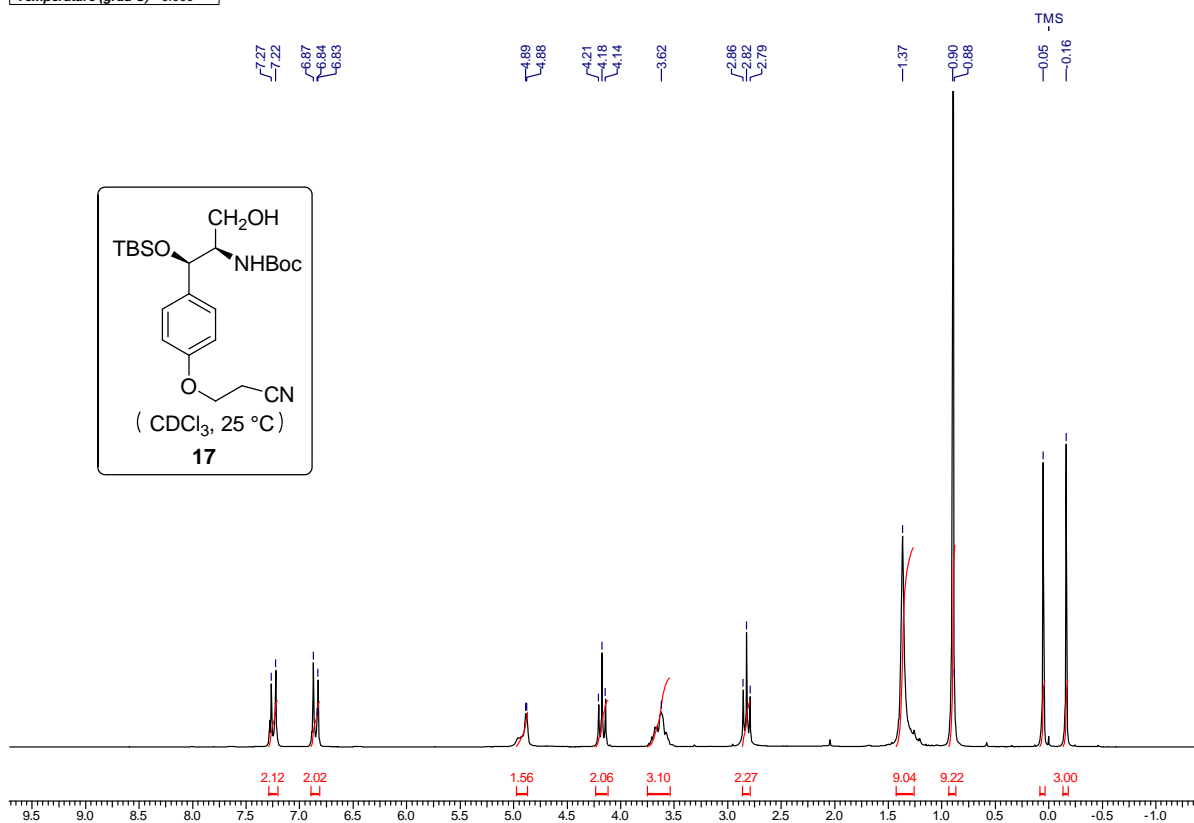
9 May 2011
 Alok/10997

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| Frequency (MHz) | 50.32 | Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 11990.41 |
| Temperature (grad C) | 0.000 | | | | | | | | |



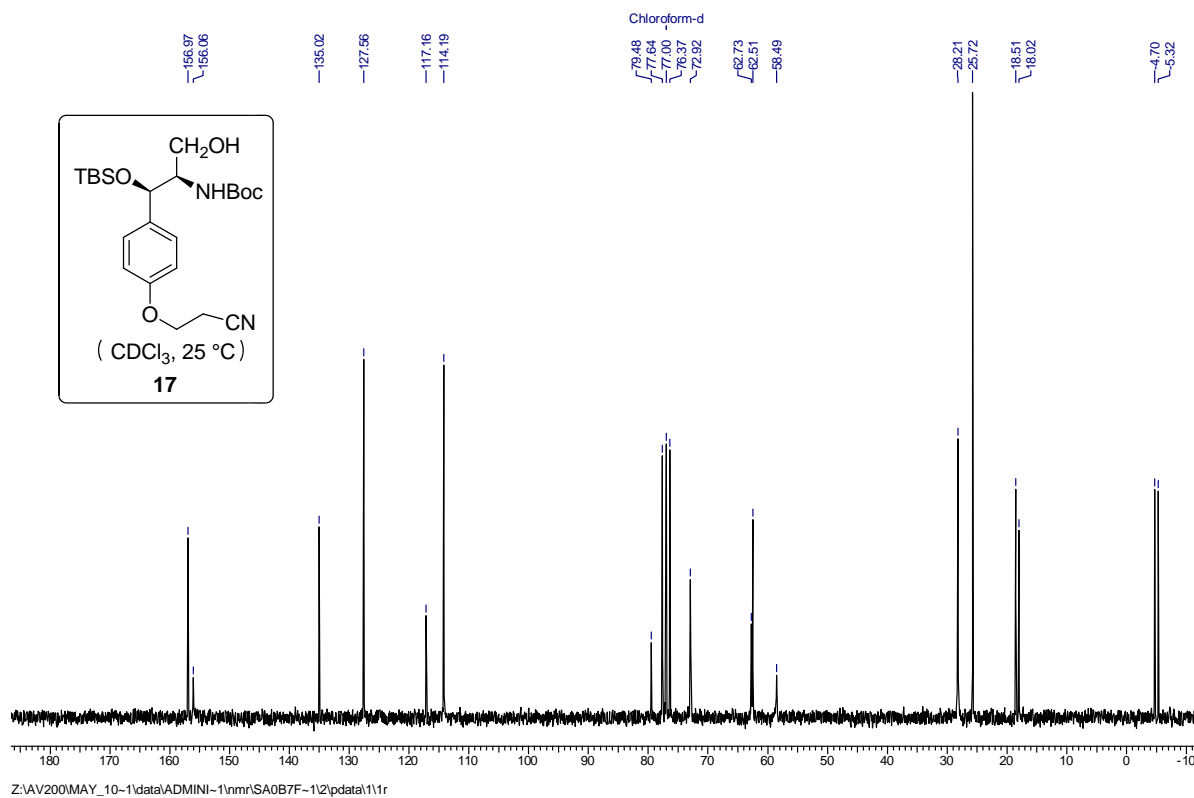
20 Jan 2011

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| Frequency (MHz) | 200.13 | Nucleus | 1H | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 4139.07 |
| Temperature (grad C) | 0.000 | | | | | | | | |



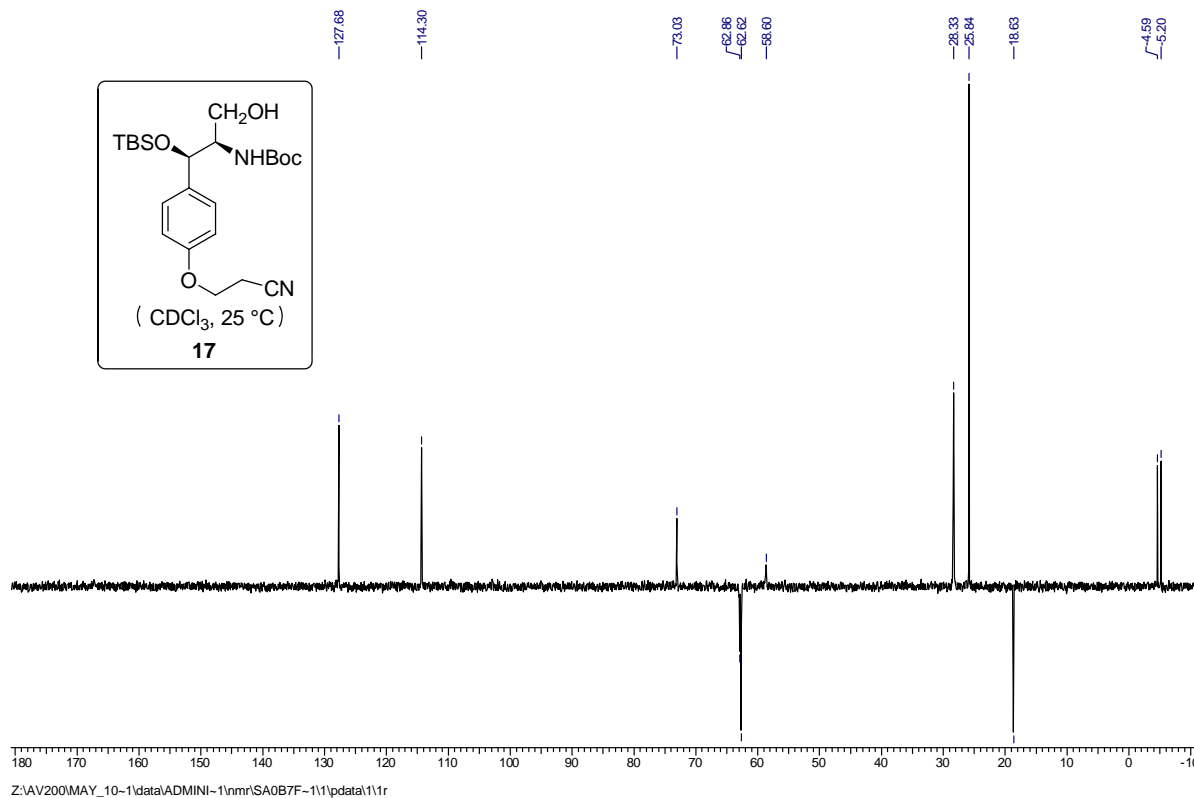
30 May 2011
 Alok/10997

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|------------------------|--------|---------|------------|-----------------------|---------------------|------------------|----------|
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| Frequency (MHz) | 50.32 | Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 |
| Temperature (grad C) | 0.000 | | | | | Sweep Width (Hz) | 11990.41 |



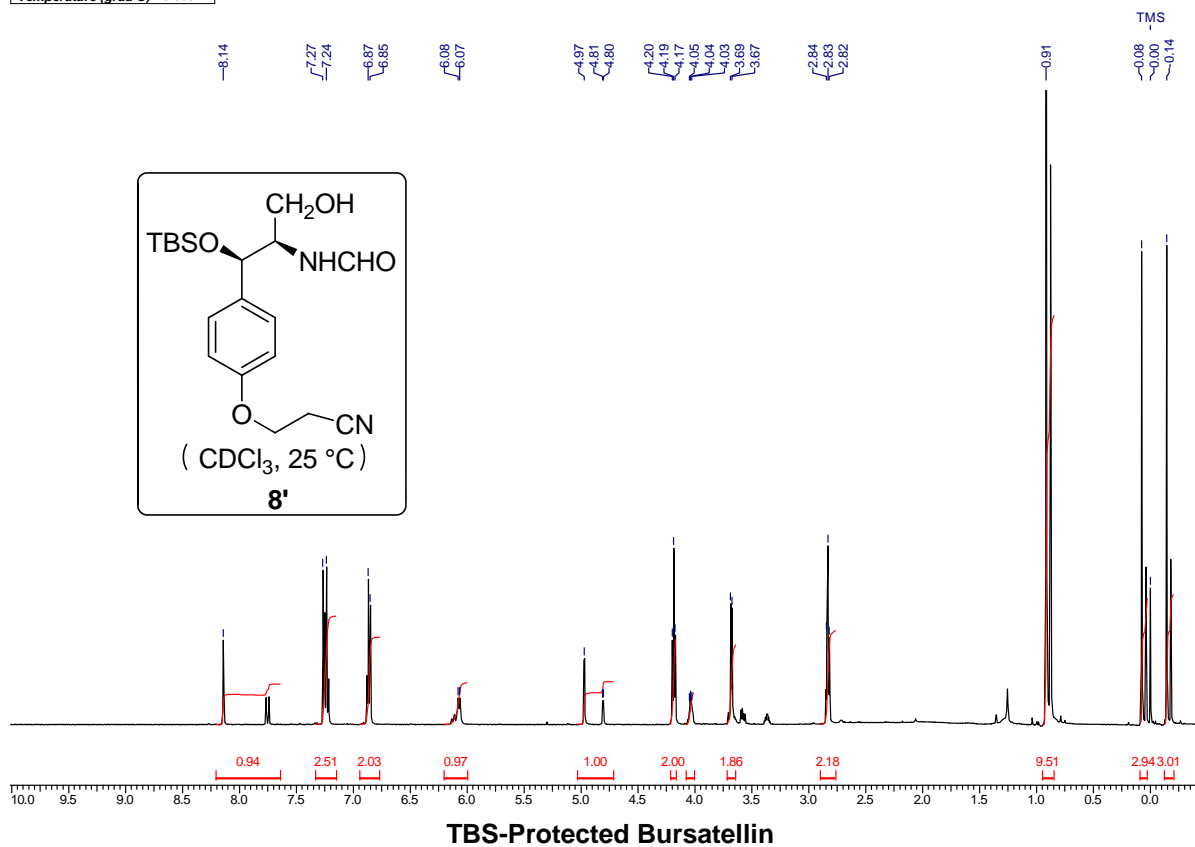
30 May 2011
 Alok/10997

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|------------------------|--------|---------|------------|-----------------------|---------------------|------------------|----------|
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| Frequency (MHz) | 50.32 | Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 |
| Temperature (grad C) | 0.000 | | | | | Sweep Width (Hz) | 11990.41 |



20 May 2011
 1H

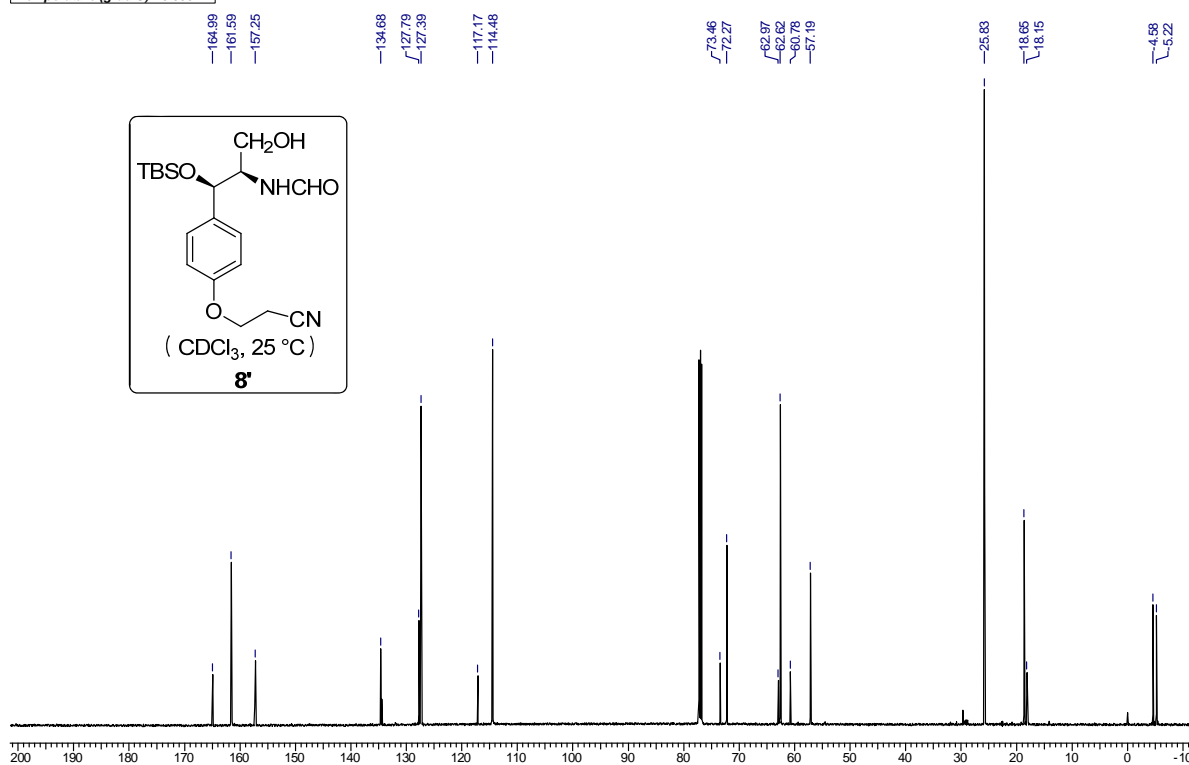
| | | | | | | | |
|------------------------|--------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 3.2768 | Comment | 1H | Date | 25/05/2010 22:14:54 | Frequency (MHz) | 500.13 |
| Nucleus | 1H | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 10000.00 |
| Temperature (grad C) | 0.000 | | | | | | |



TBS-Protected Bursatellin

20 May 2011
 13C

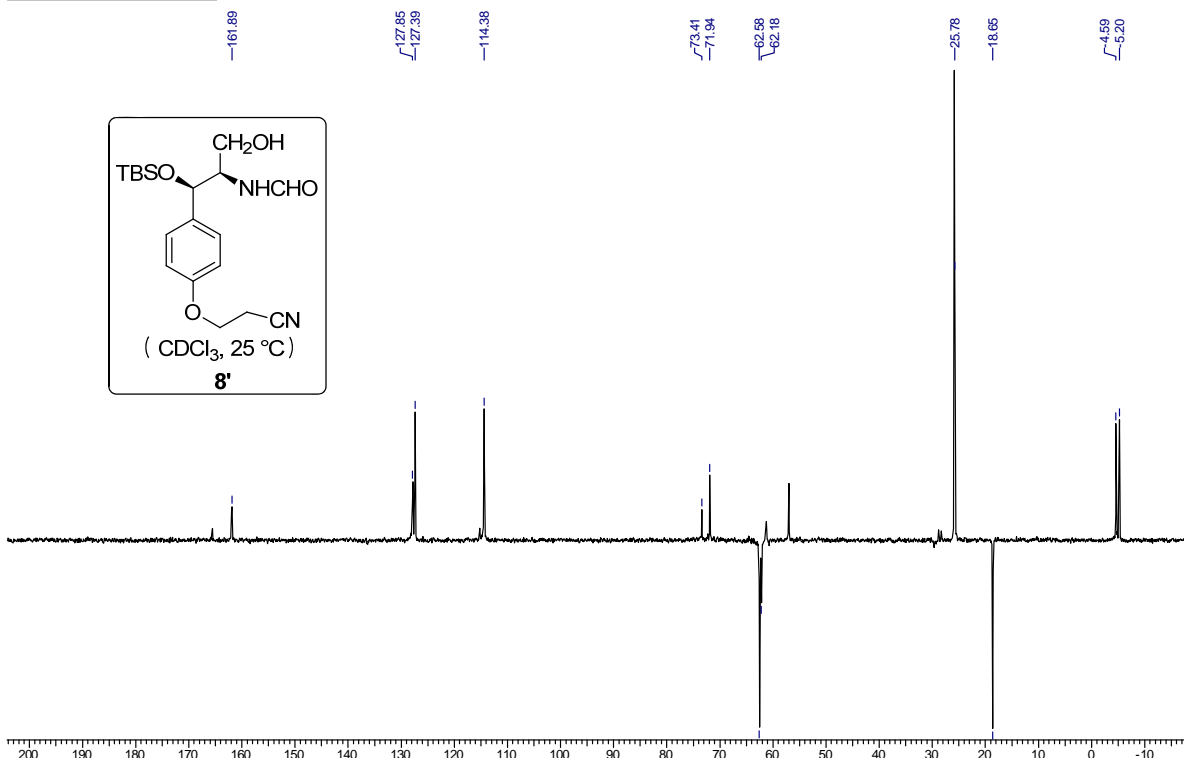
| | | | | | | | |
|------------------------|--------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.1010 | Comment | 13C | Date | 26/05/2010 13:13:32 | Frequency (MHz) | 125.76 |
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 29761.90 |
| Temperature (grad C) | 0.000 | | | | | | |



TBS-Protected Bursatellin

20 May 2011
 Alok/10997

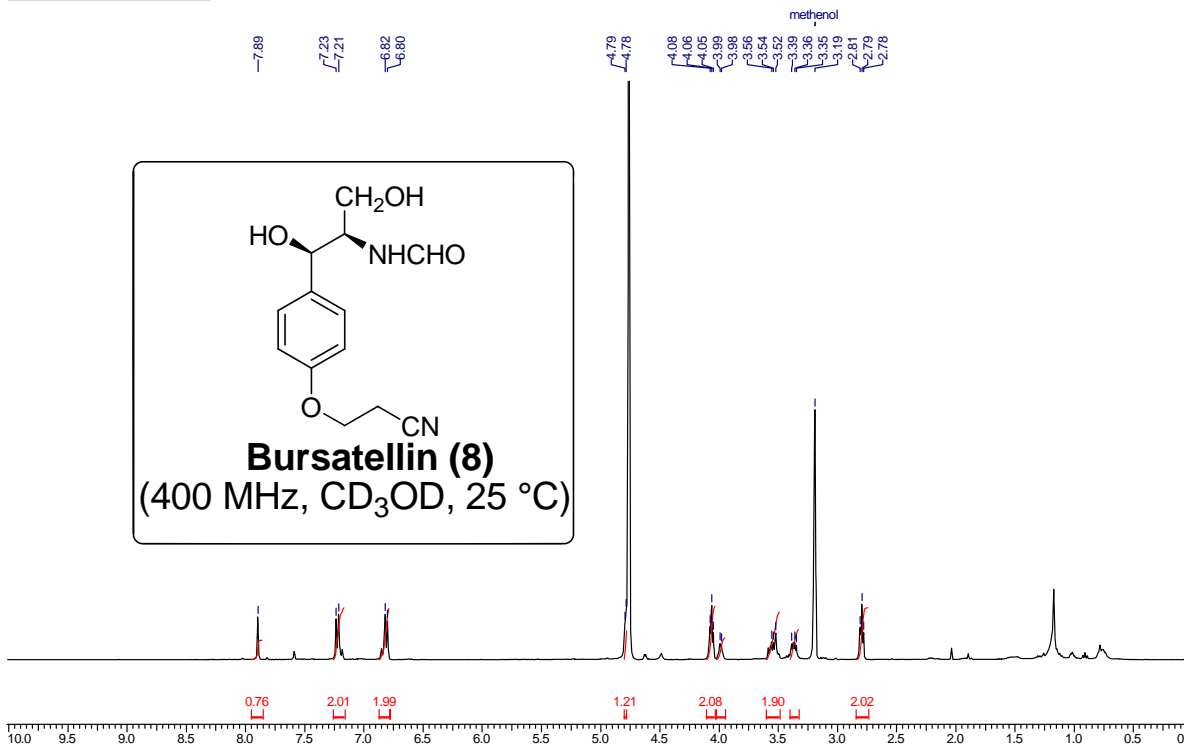
| | | | | | | | |
|------------------------|--------|---------|-----------------|-----------------------|---------------------|--------------|-------|
| Acquisition Time (sec) | 2.7329 | Comment | Alok/10997 | Date | 06/11/2010 16:10:58 | | |
| Frequency (MHz) | 50.32 | Nucleus | ¹³ C | Original Points Count | 32768 | Points Count | 32768 |
| Temperature (grad C) | 0.000 | | | Sweep Width (Hz) | 11990.41 | | |



(-)-Bursatellin

19 May 2011
 Thu2av400#009
 Alok

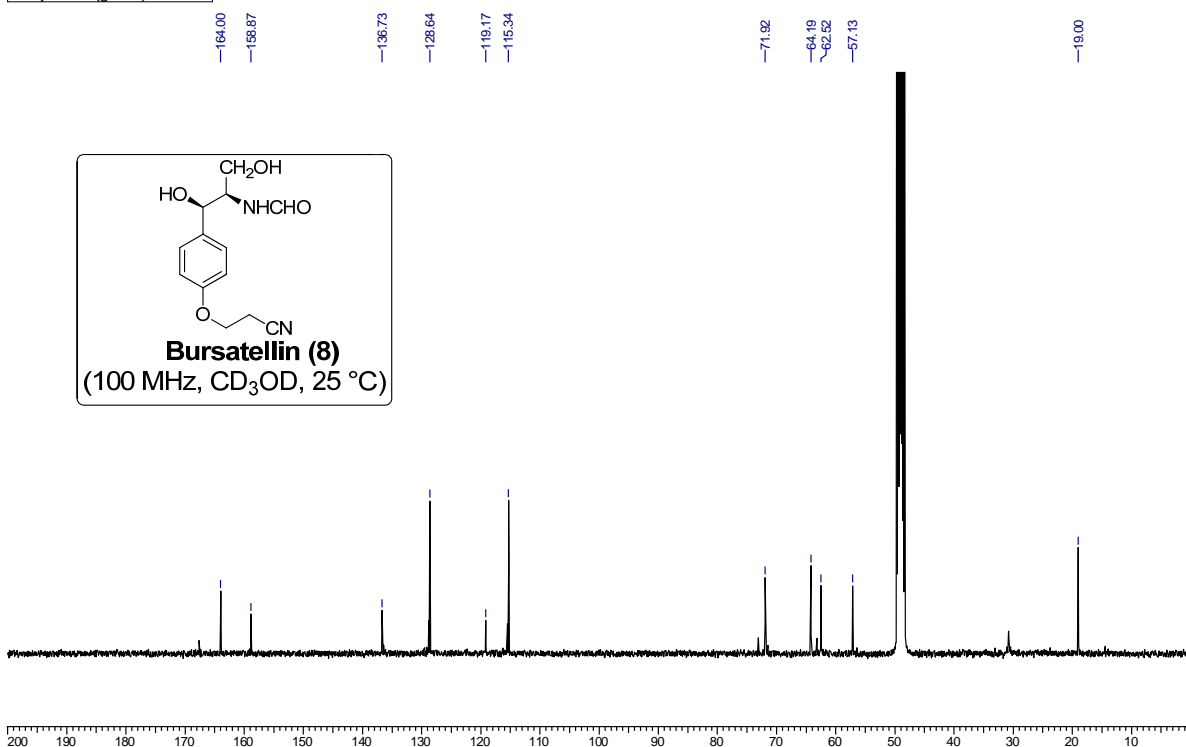
| | | | | | | | | |
|------------------------|----------------|-----------------------|-------|--------------|---------------------|------------------|-----------------|--------|
| Acquisition Time (sec) | 3.9846 | Comment | Alok | Date | 07/02/2011 09:30:44 | | Frequency (MHz) | 400.13 |
| Nucleus | ¹ H | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 8223.68 | |
| Temperature (grad C) | 0.000 | | | | | | | |



(-)-Bursatellin

19 May 2011
 Thu2av400#009
 Alok

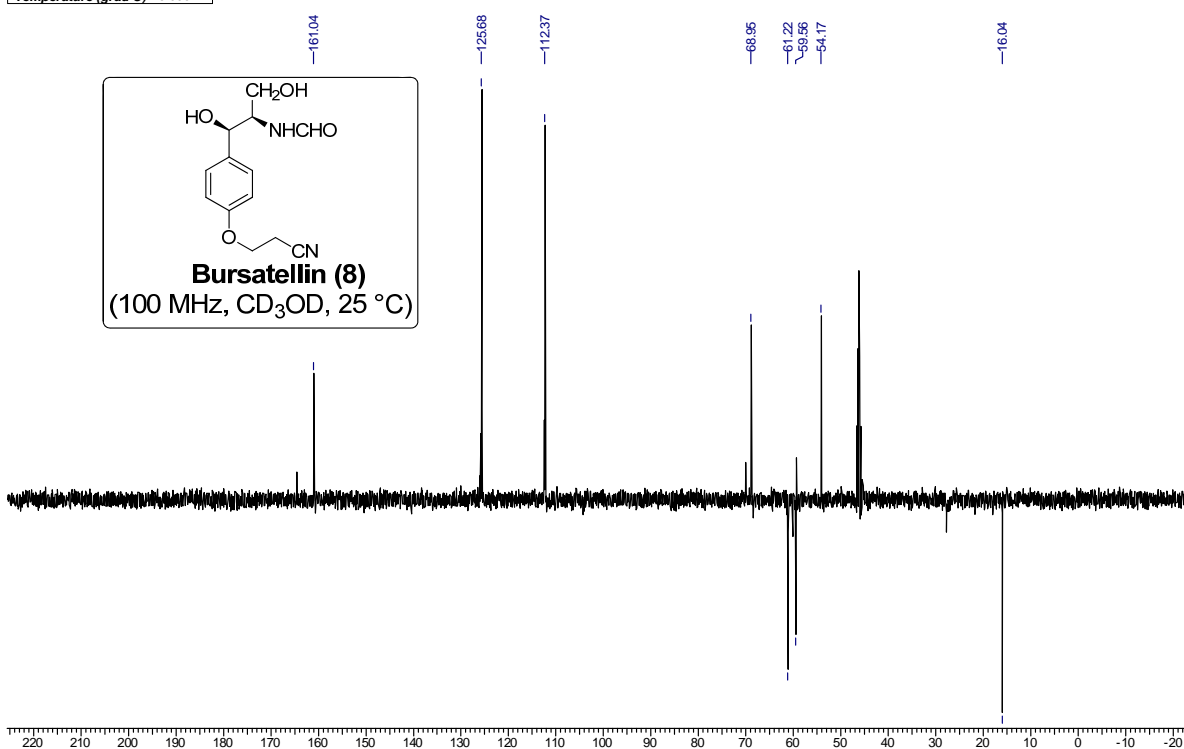
| | | | | | | | |
|------------------------|--------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.2976 | Comment | Alok | Date | 07/02/2011 09:31:46 | Frequency (MHz) | 100.61 |
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 25252.53 |
| Temperature (grad C) | 0.000 | | | | | | |



(-)-Bursatellin

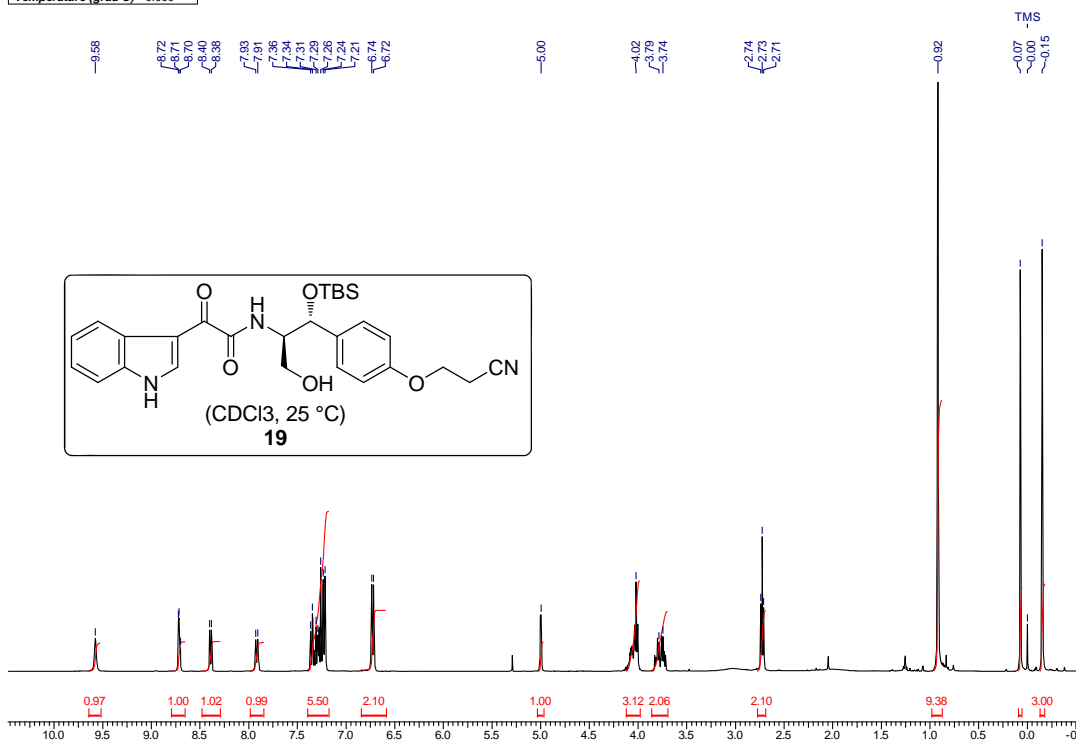
19 May 2011
 Thu2av400#009
 Alok

| | | | | | | | |
|------------------------|--------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.2976 | Comment | Alok | Date | 07/02/2011 09:30:54 | Frequency (MHz) | 100.61 |
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 25252.53 |
| Temperature (grad C) | 0.000 | | | | | | |



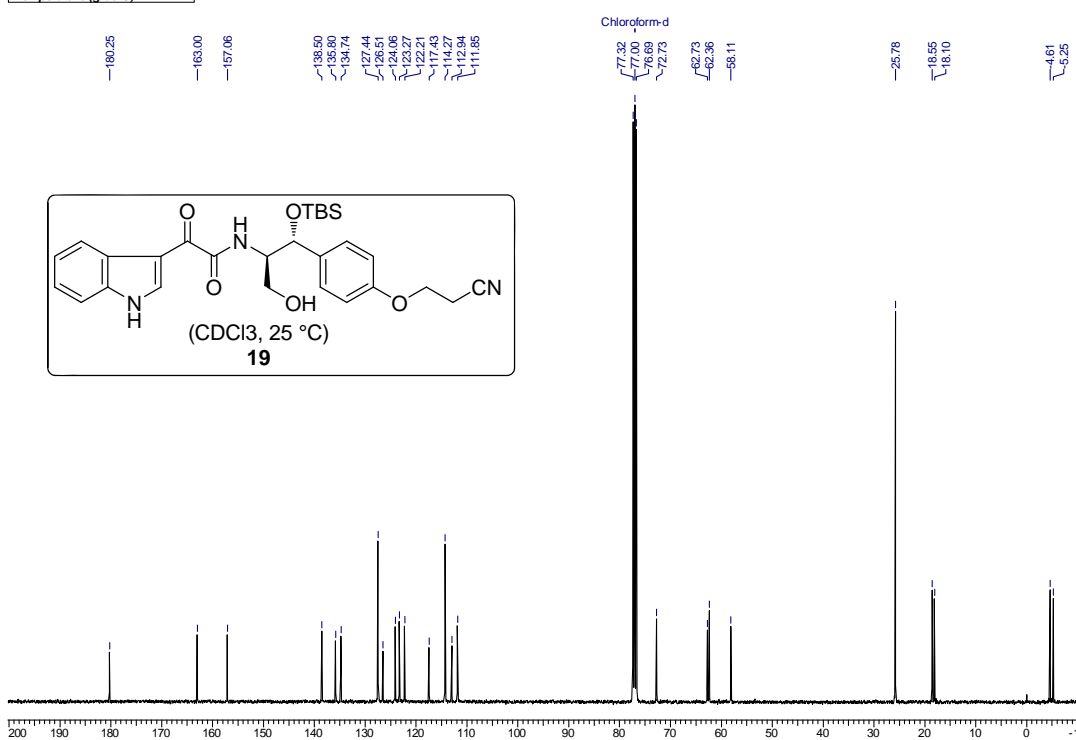
3 Jan 2011

| | | | | | | | | | |
|------------------------|--------|---------|----------------|-----------------------|---------------------|--------------|-------|------------------|---------|
| Acquisition Time (sec) | 3.9846 | Comment | Alck10997 | Date | 03/01/2011 16:27:34 | | | | |
| Frequency (MHz) | 400.13 | Nucleus | ¹ H | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 8223.68 |
| Temperature (grad C) | 0.000 | | | | | | | | |



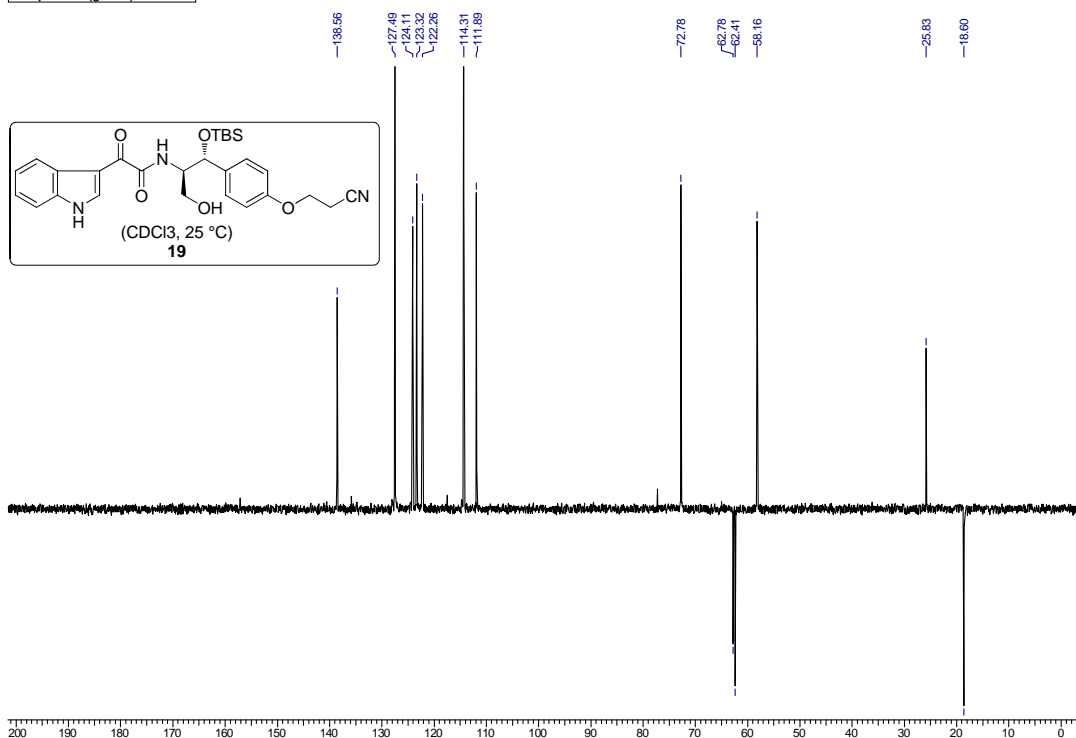
3 Jan 2011

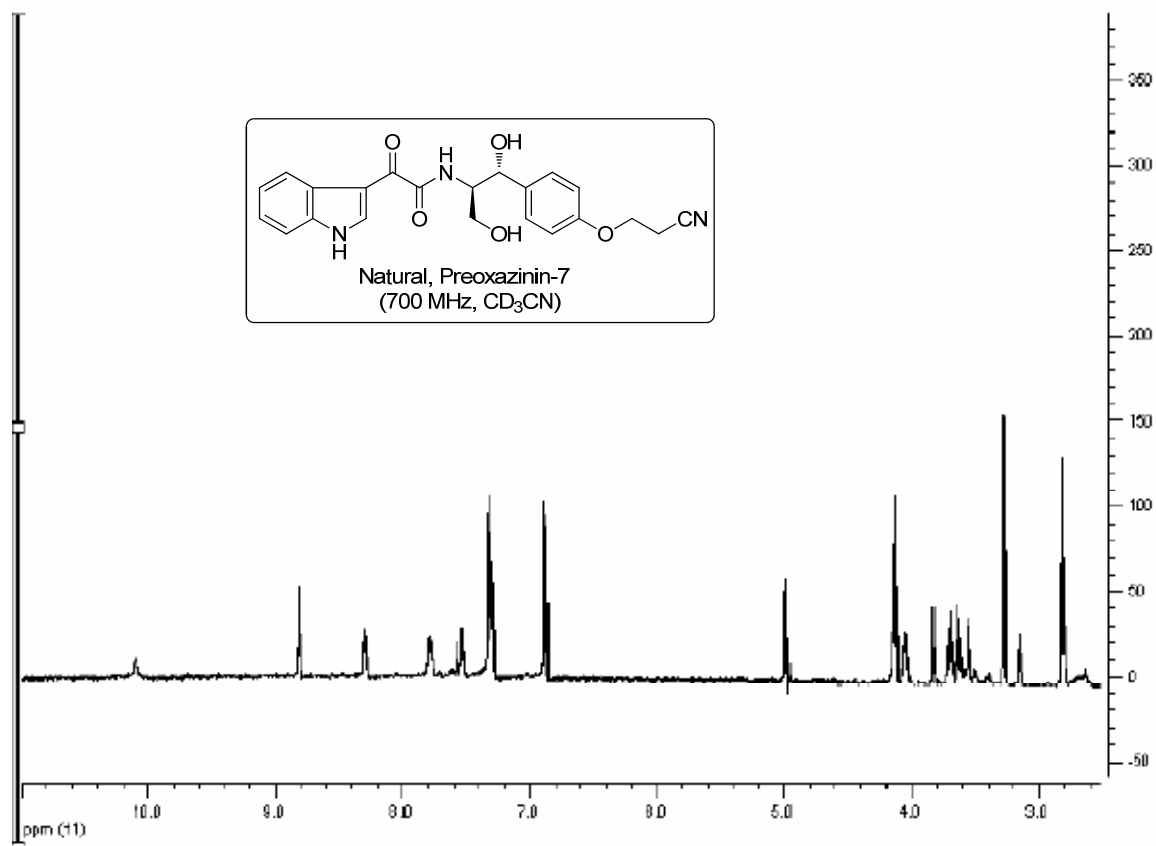
| | | | | | | | | |
|------------------------|-----------------|-----------------------|-----------------|--------------|---------------------|------------------|-----------------|--------|
| Acquisition Time (sec) | 1.3631 | Comment | ¹³ C | Date | 03/01/2011 16:27:50 | | Frequency (MHz) | 100.61 |
| Nucleus | ¹³ C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 24038.46 | |
| Temperature (grad C) | 0.000 | | | | | | | |



3 Jan 2011

| Acquisition Time (sec) | Comment | DEPT | Date | 03/01/2011 16:27:42 | Frequency (MHz) | 100.61 |
|------------------------|---------|-----------------------|--------------|---------------------|------------------|----------|
| Nucleus | 13C | Original Points Count | Points Count | 32768 | Sweep Width (Hz) | 24038.46 |
| Temperature (grad C) | 0.000 | | | | | |

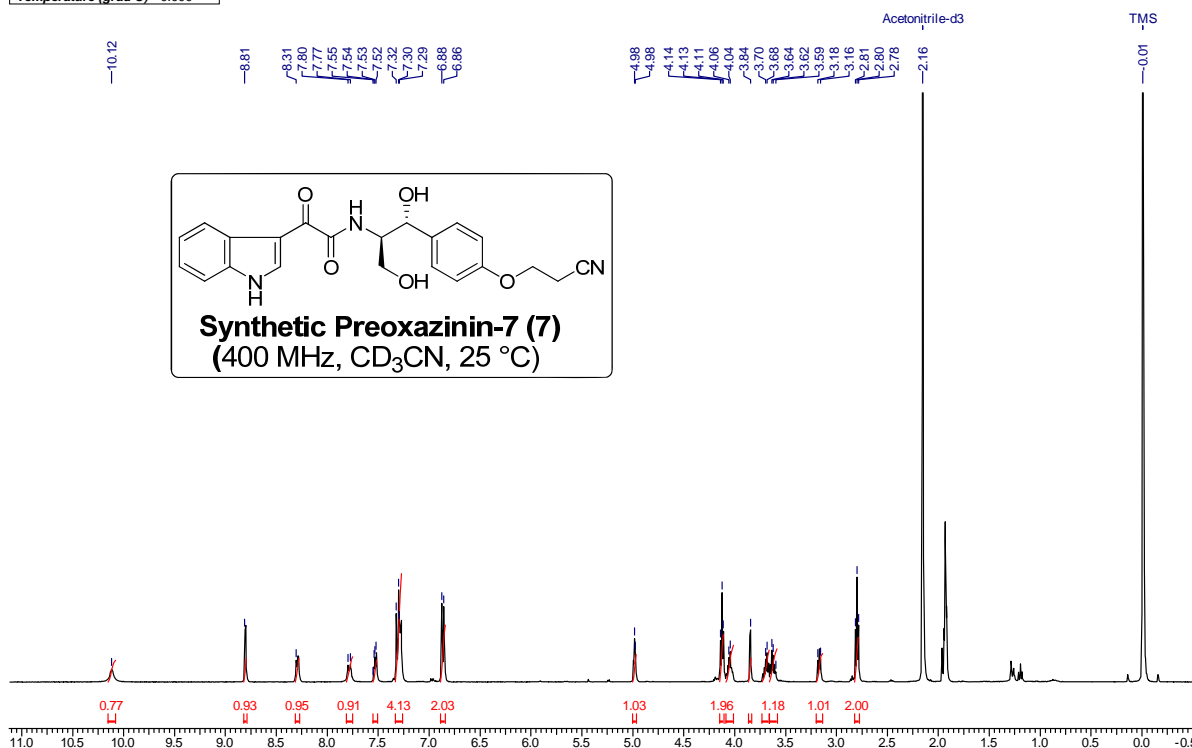




Preoxazin

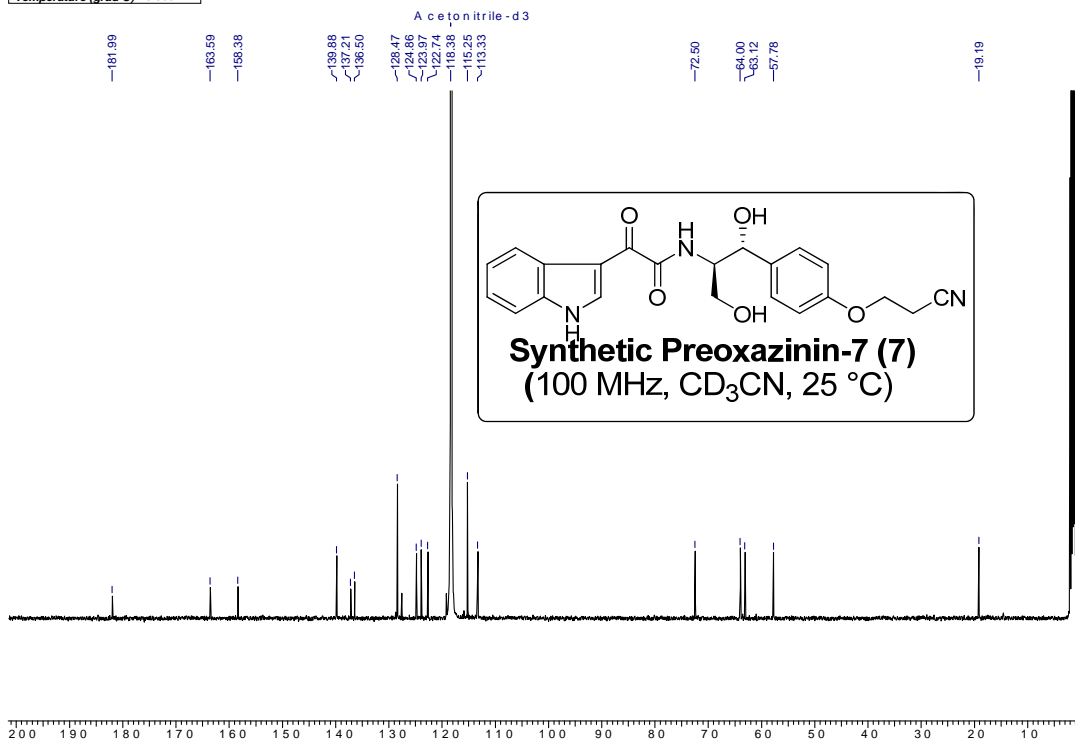
18 May 2011
 Thu2av400#009
 Alok R/10997

| | | | | | |
|------------------------|--------|---------|--------------|-----------------------|---------------------|
| Acquisition Time (sec) | 3.9846 | Comment | Alok R/10997 | Date | 17/05/2010 21:04:00 |
| Frequency (MHz) | 400.13 | Nucleus | 1H | Original Points Count | 32768 |
| Temperature (grad C) | 0.000 | | | Points Count | 32768 |
| | | | | Sweep Width (Hz) | 8223.68 |



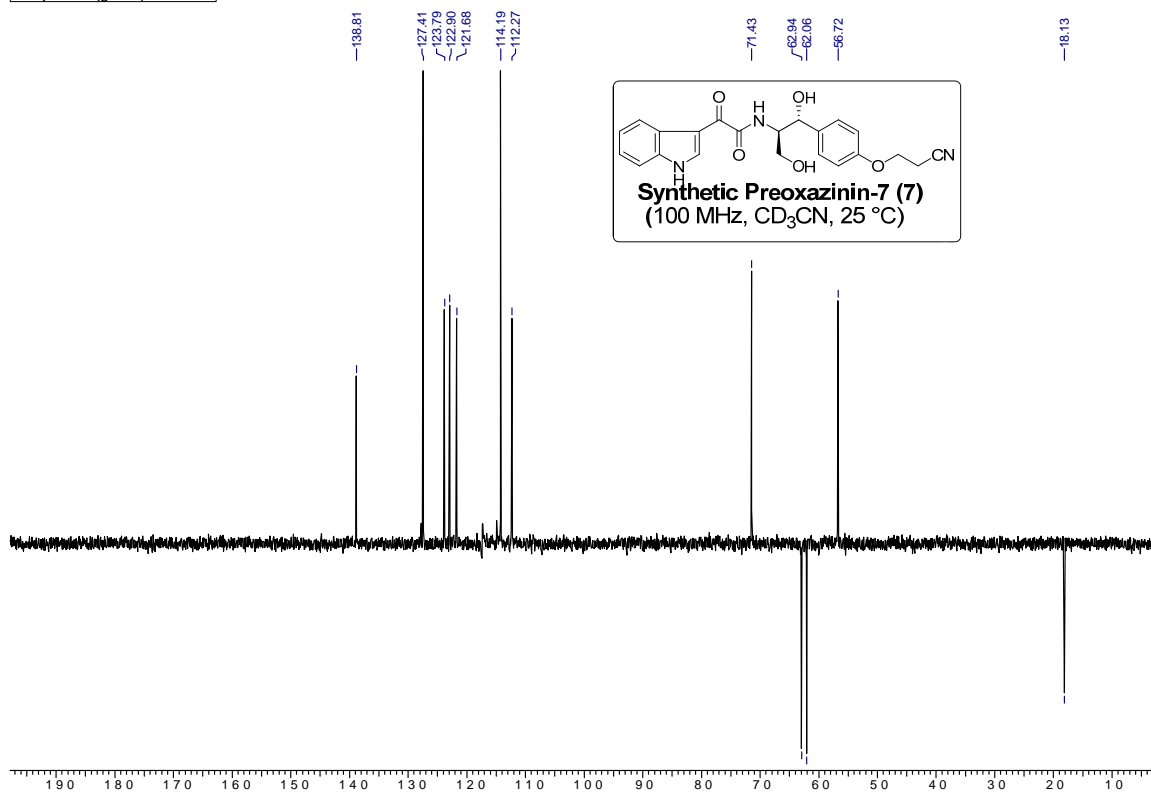
23 Dec 2010

| | | | | | |
|------------------------|--------|---------|-----------------|-----------------------|---------------------|
| Acquisition Time (sec) | 1.3631 | Comment | Alok R/10997 | Date | 18/05/2010 10:30:42 |
| Frequency (MHz) | 100.61 | Nucleus | ¹³ C | Original Points Count | 32768 |
| Temperature (grad C) | 0.000 | | | Points Count | 32768 |
| | | | | Sweep Width (Hz) | 24038.46 |



23 Dec 2010

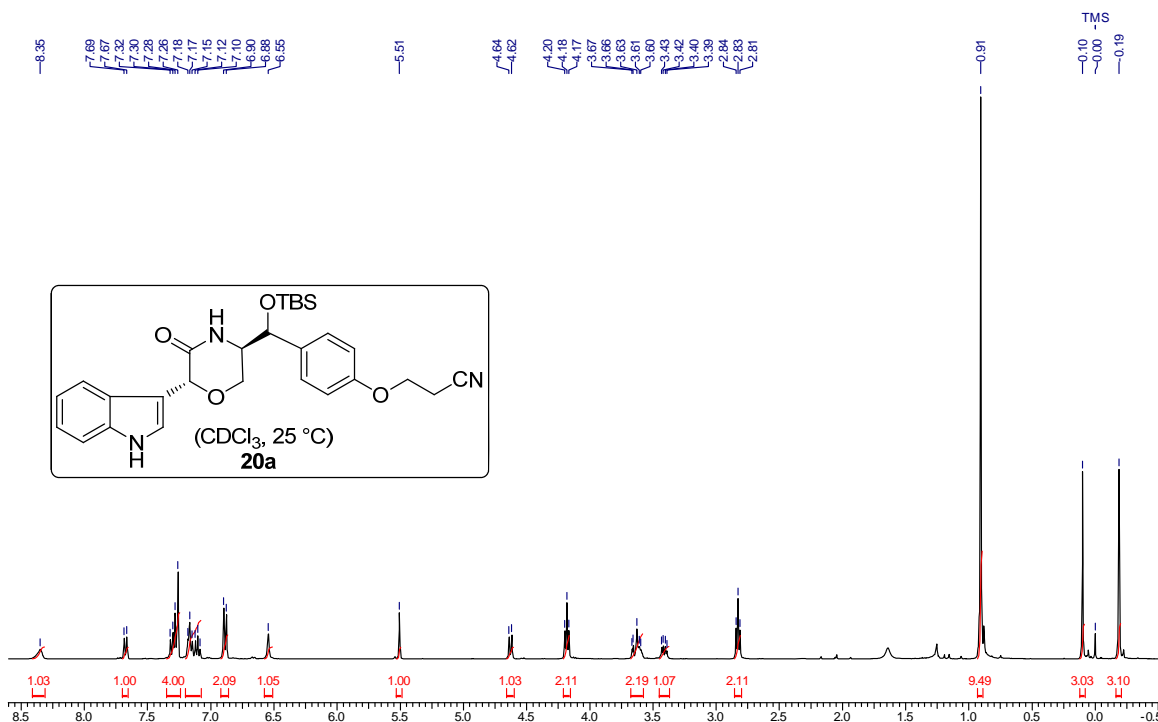
| | | | | | |
|------------------------|--------|---------|-----------------|-----------------------|---------------------|
| Acquisition Time (sec) | 1.3631 | Comment | Alok R/10997 | Date | 18/05/2010 18:39:38 |
| Frequency (MHz) | 100.61 | Nucleus | ¹³ C | Original Points Count | 32768 |
| Temperature (grad C) | 0.000 | | | Points Count | 32768 |
| | | | | Sweep Width (Hz) | 24038.46 |



TBS-protected oxazin-5

28 Apr 2011
 mon5av400#002
 VHP

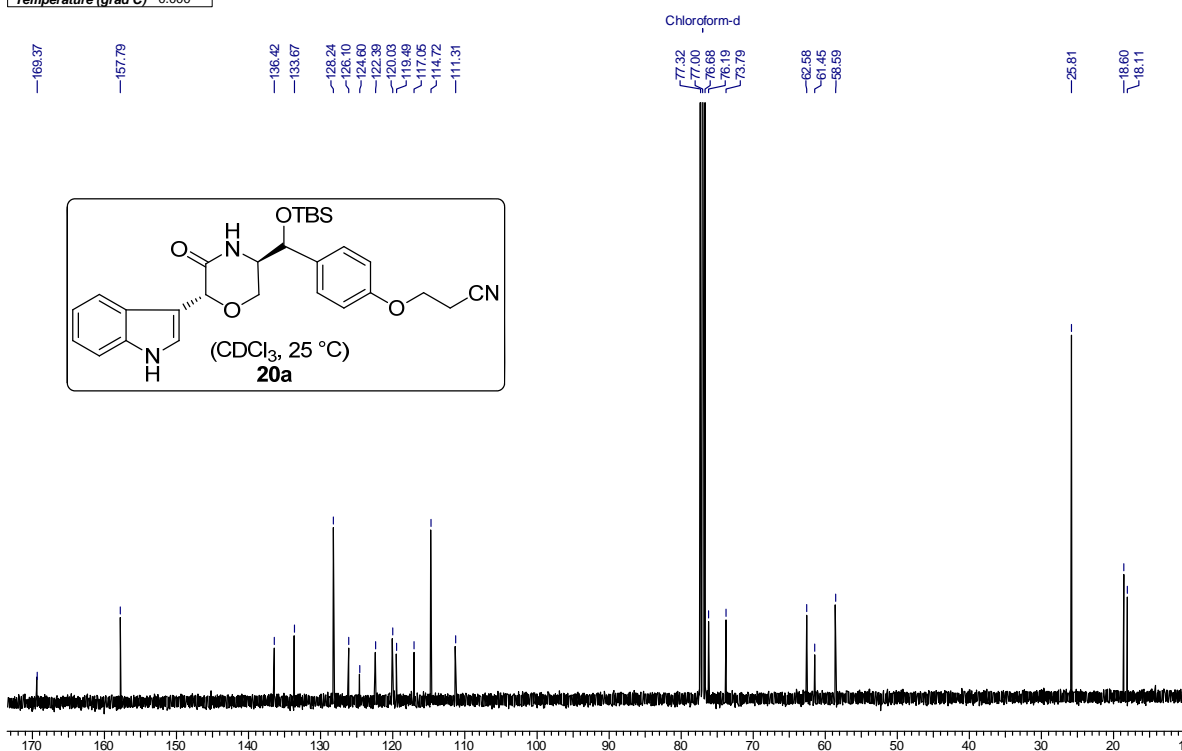
| | | | | | | | |
|------------------------|----------------|-----------------------|-------|--------------|---------------------|------------------|---------|
| Acquisition Time (sec) | 3.9846 | Comment | Alok | Date | 28/03/2011 15:45:38 | Frequency (MHz) | 400.13 |
| Nucleus | ¹ H | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 8223.68 |
| Temperature (grad C) | 0.000 | | | | | | |



TBS-protected oxazin-5

28 Apr 2011
 mon5av400#002
 13C

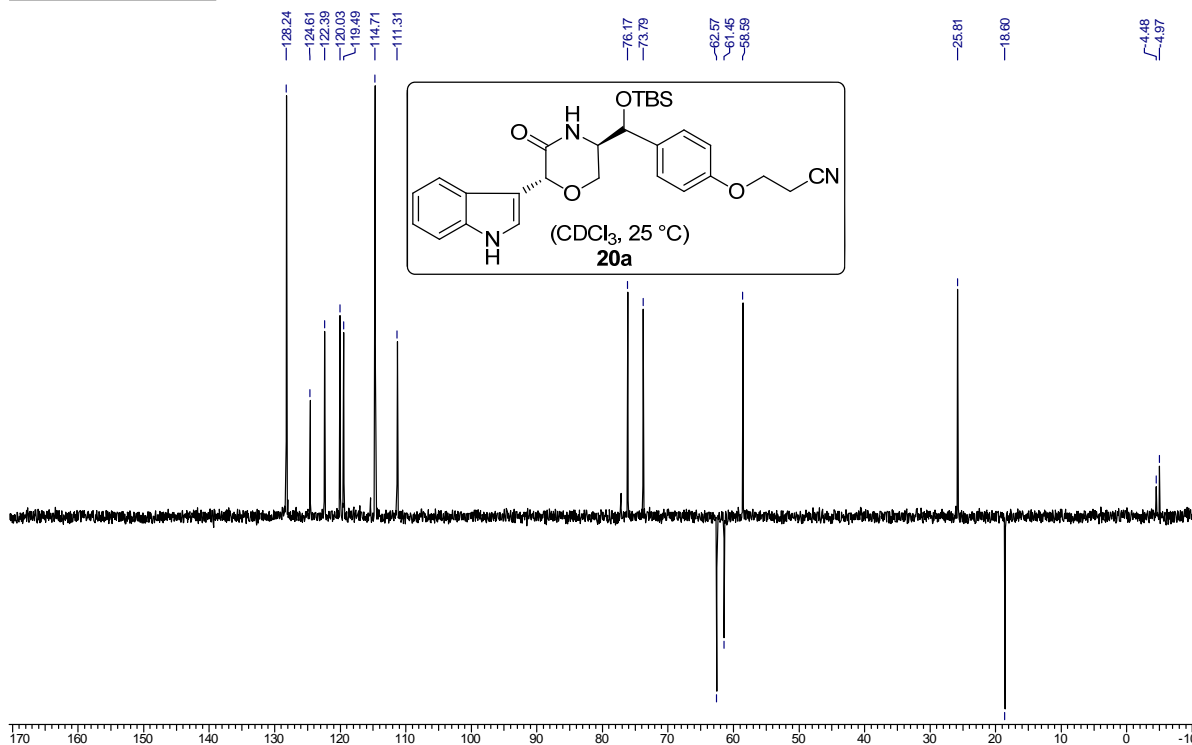
| | | | | | | | |
|------------------------|-----------------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.8350 | Comment | 13C | Date | 28/03/2011 15:44:12 | Frequency (MHz) | 100.61 |
| Nucleus | ¹³ C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 17857.14 |
| Temperature (grad C) | 0.000 | | | | | | |



TBS-protected oxazin-5

28 Apr 2011
 mon5av400#002
 DEPT

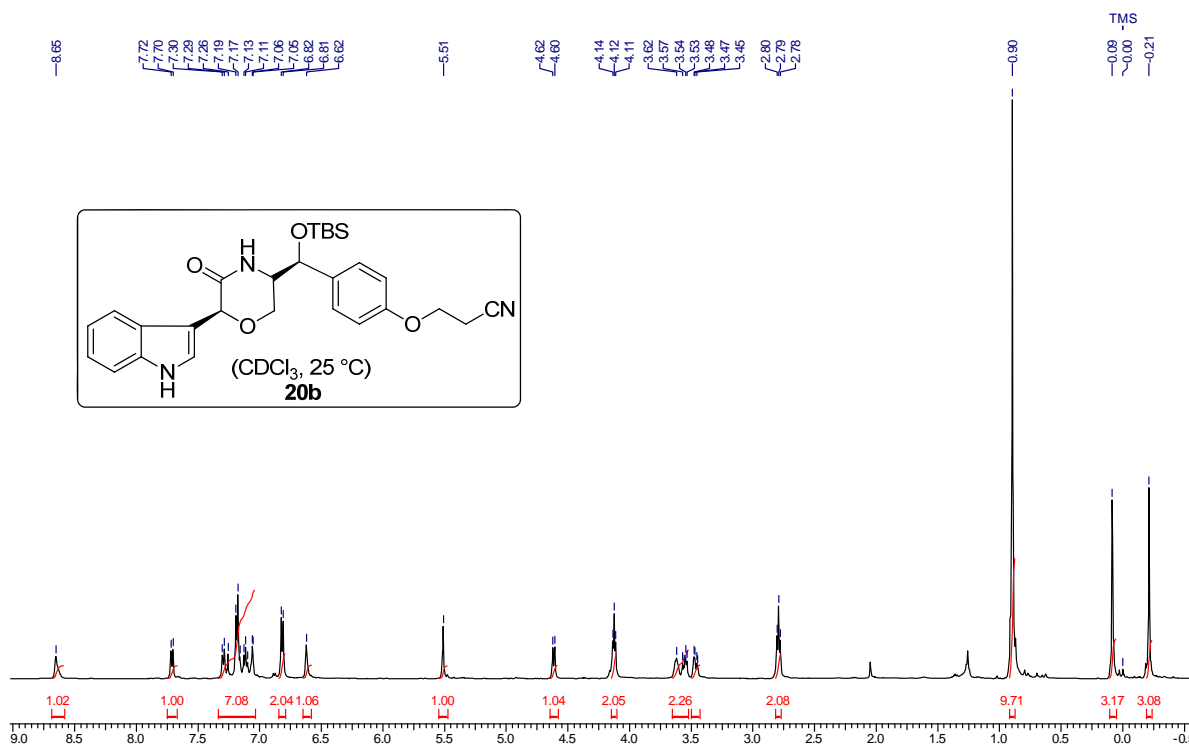
| | | | | | | | |
|------------------------|-----------------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.2976 | Comment | DEPT | Date | 28/03/2011 15:45:30 | Frequency (MHz) | 100.61 |
| Nucleus | ¹³ C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 25252.53 |
| Temperature (grad C) | 0.000 | | | | | | |



TBS-protected oxazin-6

28 Apr 2011
 wed5av500#009
 alok

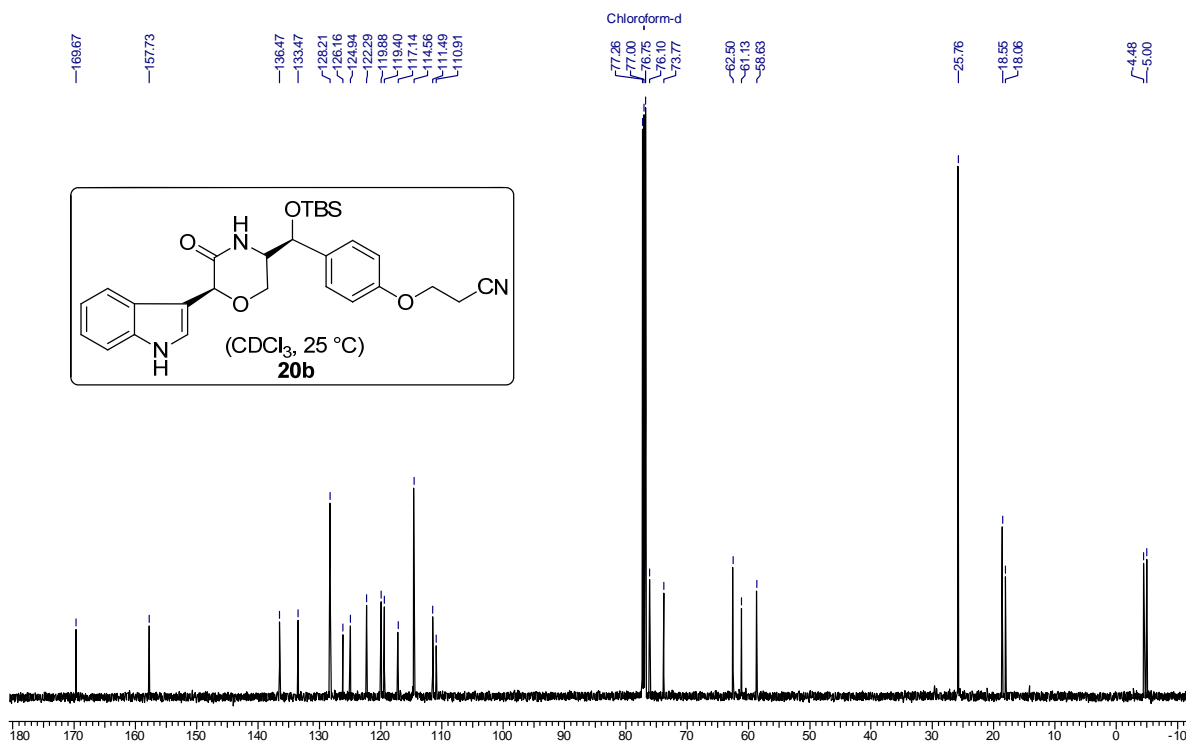
| | | | | | | | |
|------------------------|----------------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 3.2768 | Comment | alok | Date | 23/03/2011 17:33:42 | Frequency (MHz) | 500.13 |
| Nucleus | ¹ H | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 10000.00 |
| Temperature (grad C) | 0.000 | | | | | | |



TBS-protected oxazin-6

28 Apr 2011
 wed5av500#009
 13C

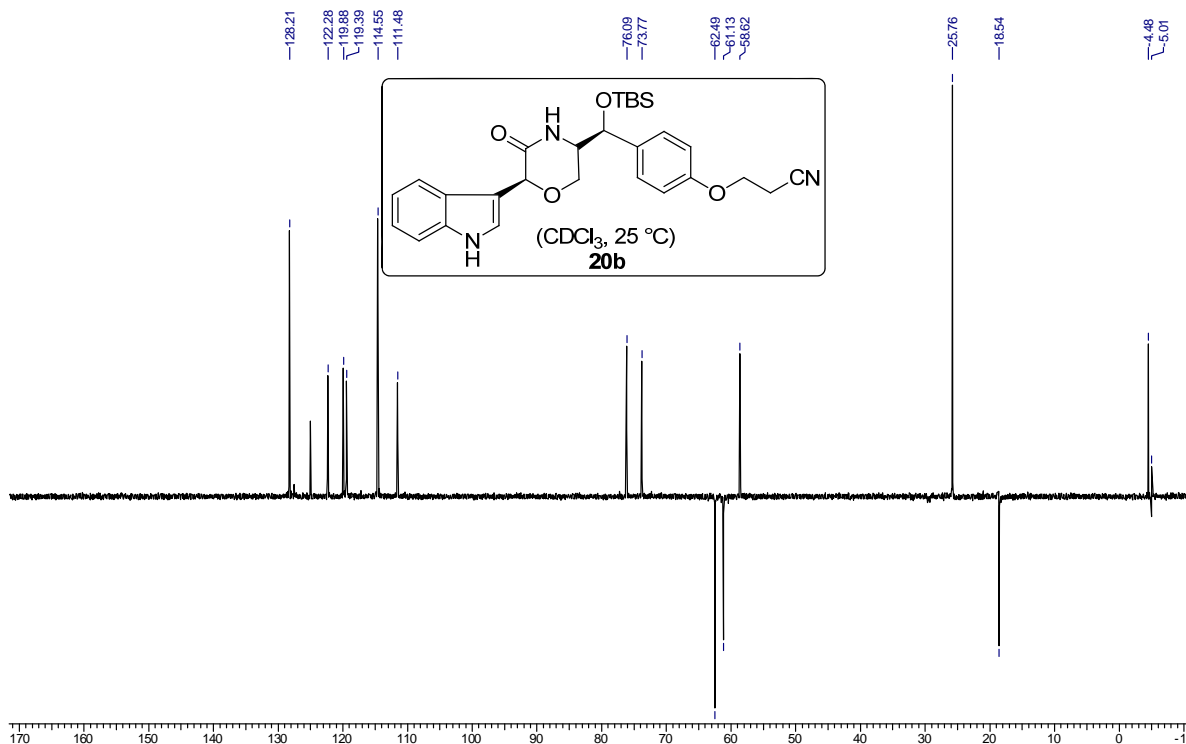
| | | | | | | | |
|------------------------|--------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.1010 | Comment | 13C | Date | 23/03/2011 18:14:02 | Frequency (MHz) | 125.76 |
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 29761.90 |
| Temperature (grad C) | 0.000 | | | | | | |

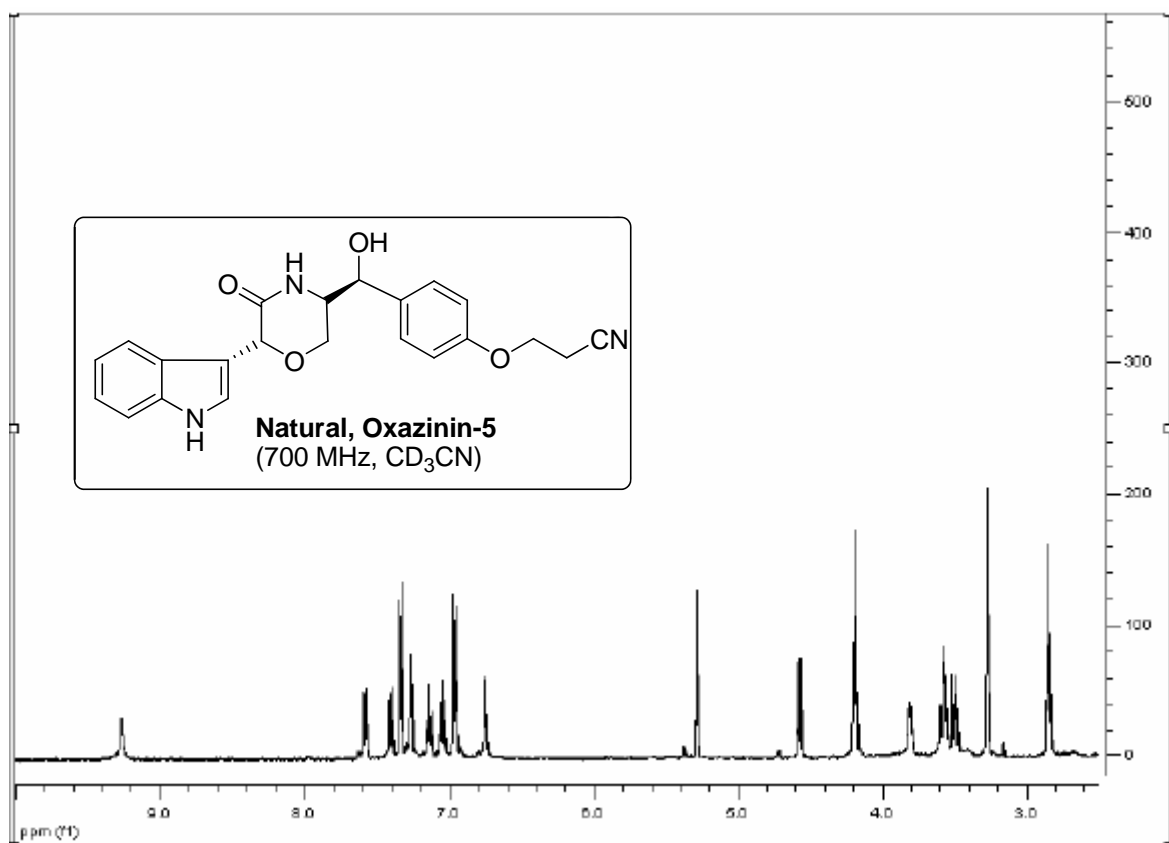


TBS-protected oxazin-6

28 Apr 2011
 wed5av500#009
 DEPT135

| | | | | | | | |
|------------------------|--------|-----------------------|---------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.1010 | Comment | DEPT135 | Date | 23/03/2011 17:55:36 | Frequency (MHz) | 125.76 |
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 29761.90 |
| Temperature (grad C) | 0.000 | | | | | | |

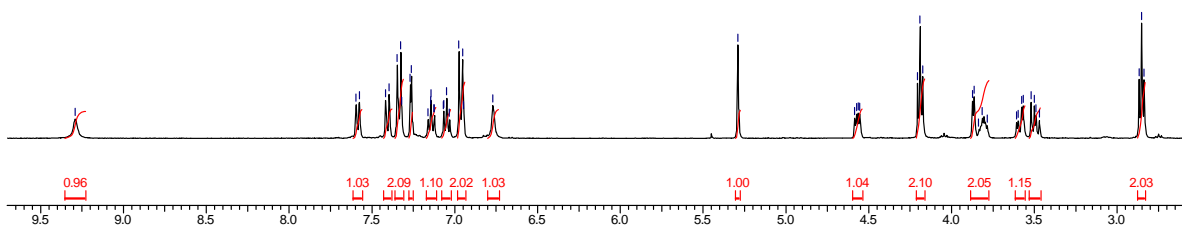
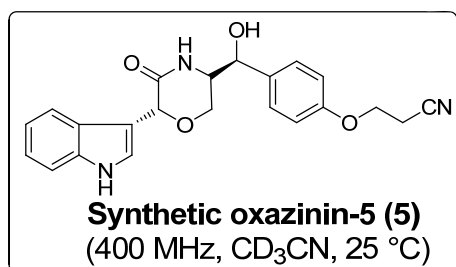




oxazin-5

28 Apr 2011
 wed3av400#012
 VHP

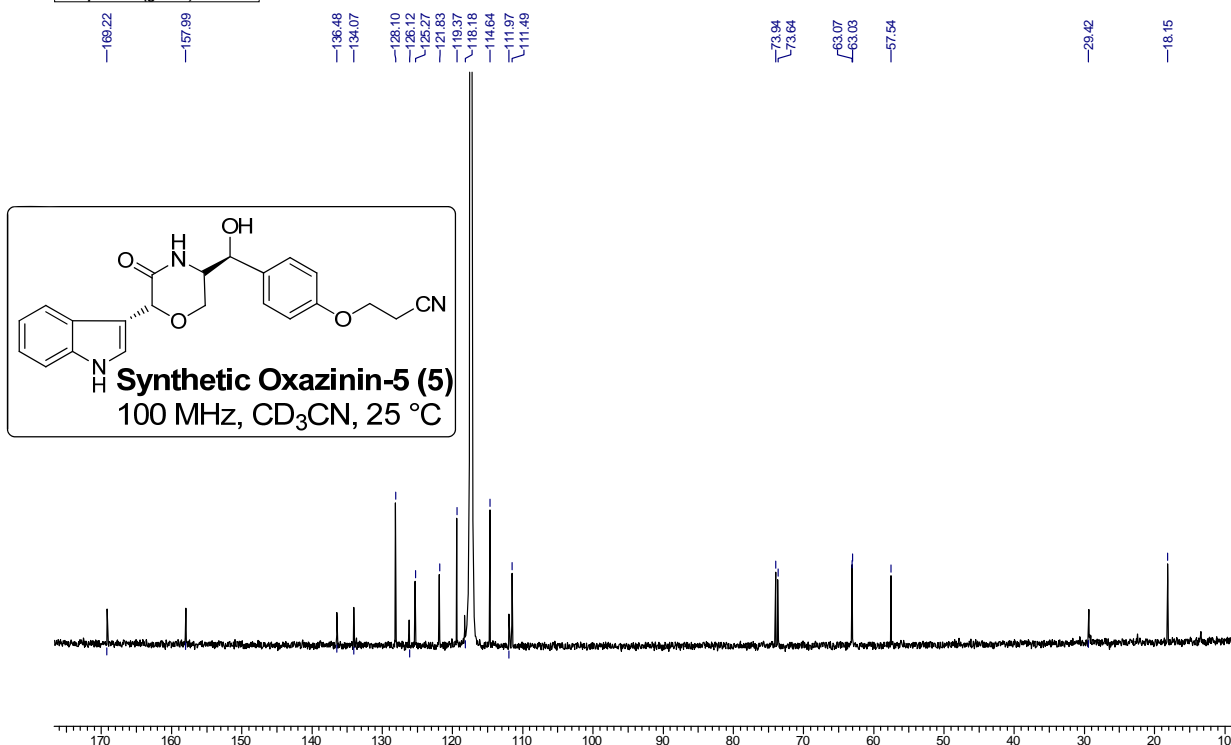
| | | | | | |
|------------------------|--------|---------|-------------|-----------------------|---------------------|
| Acquisition Time (sec) | 4.0894 | Comment | Alok Ranjan | Date | 13/04/2011 22:44:38 |
| Frequency (MHz) | 400.13 | Nucleus | 1H | Original Points Count | 32768 |
| Temperature (grad C) | 0.000 | | | Points Count | 32768 |
| | | | | Sweep Width (Hz) | 8012.82 |



oxazinin-5

16 May 2011
 wed3av400#012
 13C

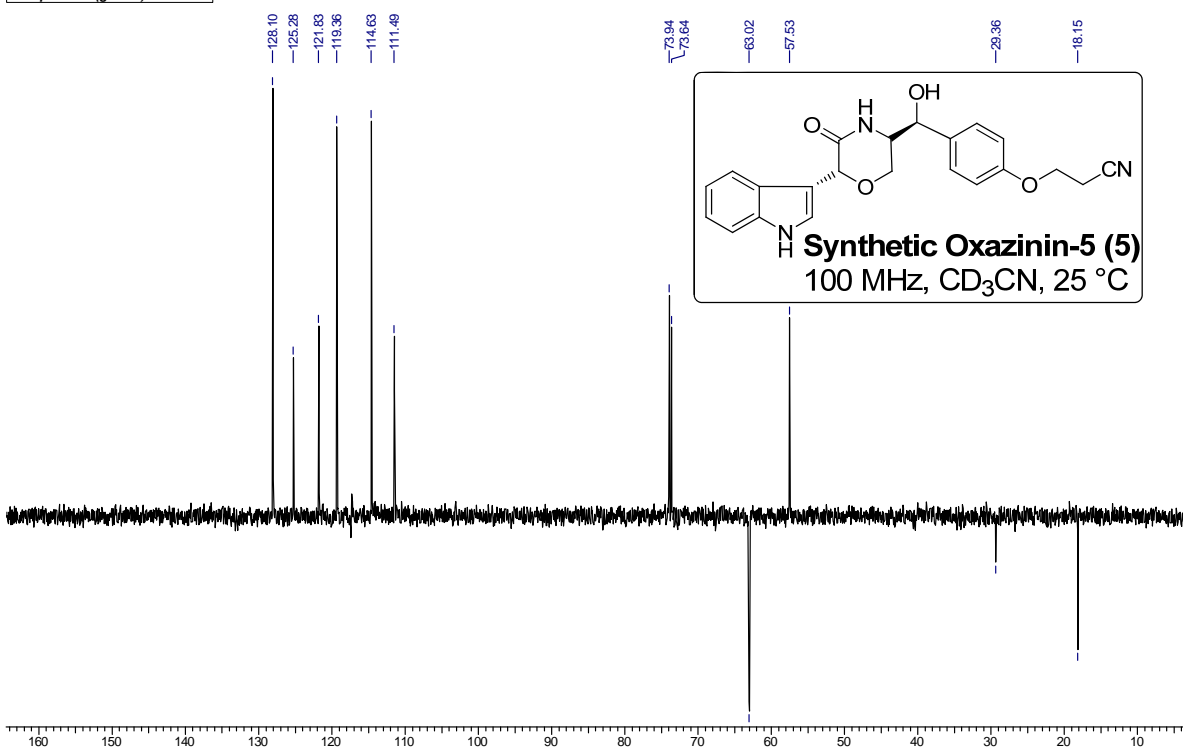
| | | | | | | | |
|------------------------|--------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.2976 | Comment | 13C | Date | 14/04/2011 10:21:50 | Frequency (MHz) | 100.61 |
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 25252.53 |
| Temperature (grad C) | 0.000 | | | | | | |



oxazinin-5

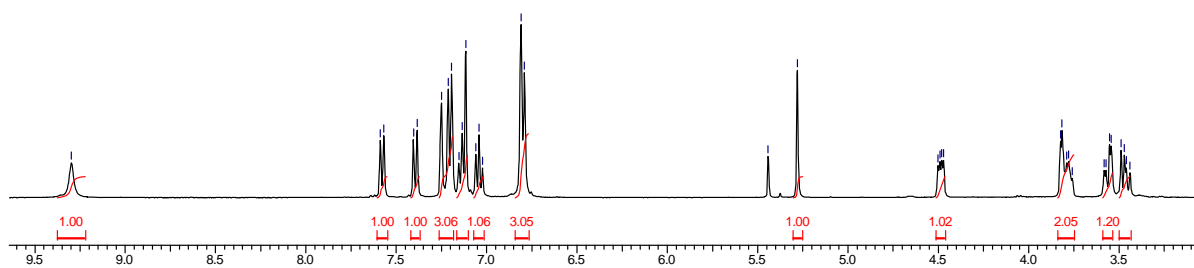
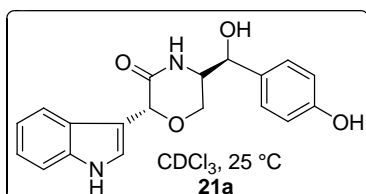
16 May 2011
 wed3av400#012
 DEPT

| | | | | | | | |
|------------------------|--------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.2976 | Comment | DEPT | Date | 14/04/2011 10:22:24 | Frequency (MHz) | 100.61 |
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 25252.53 |
| Temperature (grad C) | 0.000 | | | | | | |



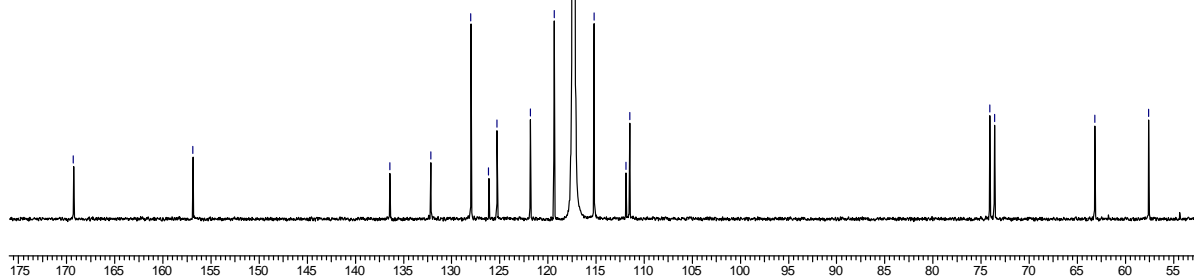
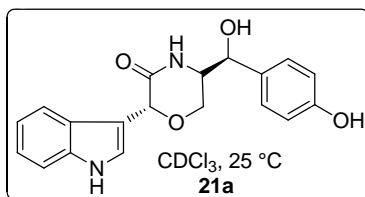
17 May 2011
 Mon1av400#005
 Vijendra

| Acquisition Time (sec) | Comment | Vijendra | Date | 03/05/2011 10:05:10 | Frequency (MHz) | 400.13 | | | |
|------------------------|---------|-----------------------|-------|---------------------|-----------------|------------------|---------|----------------------|-------|
| Nucleus | 1H | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 8012.82 | Temperature (grad C) | 0.000 |



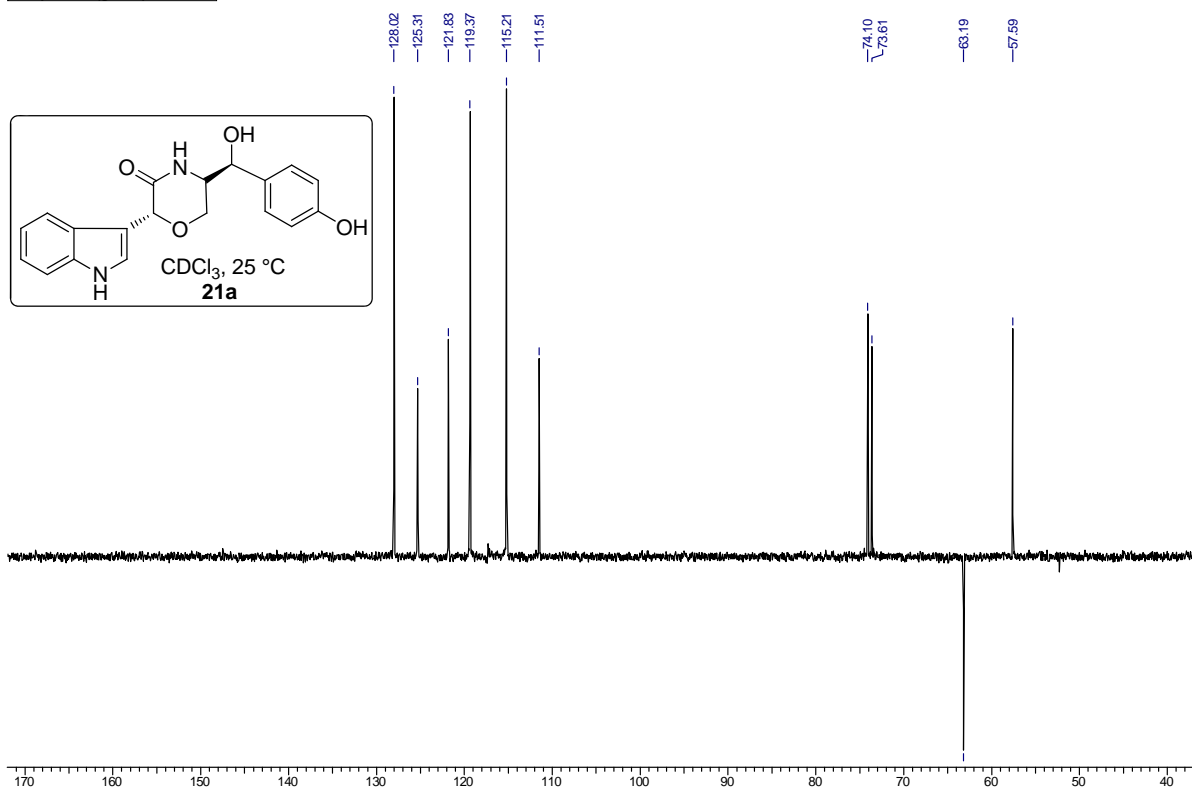
17 May 2011
 Mon1av400#005
 Vijendra

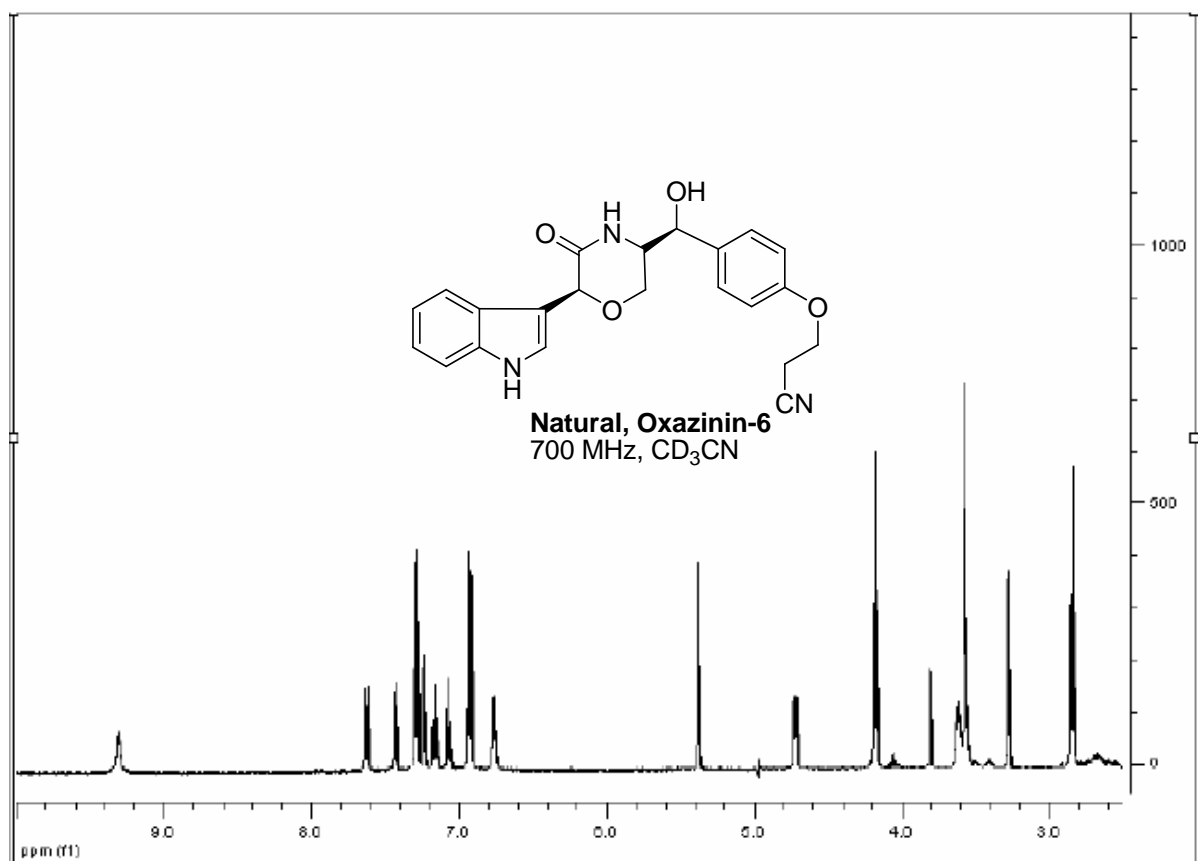
| Acquisition Time (sec) | Comment | Vijendra | Date | 03/05/2011 10:06:22 | Frequency (MHz) | 100.61 | | | |
|------------------------|---------|-----------------------|-------|---------------------|-----------------|------------------|----------|----------------------|-------|
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 24038.46 | Temperature (grad C) | 0.000 |



17 May 2011
Mon1av400#005
DEPT

| | | | | | | | |
|------------------------|-----------------|-----------------------|----------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.3631 | Comment | Vijendra | Date | 03/05/2011 10:06:00 | Frequency (MHz) | 100.61 |
| Nucleus | ¹³ C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 24038.46 |
| Temperature (grad C) | 0.000 | | | | | | |



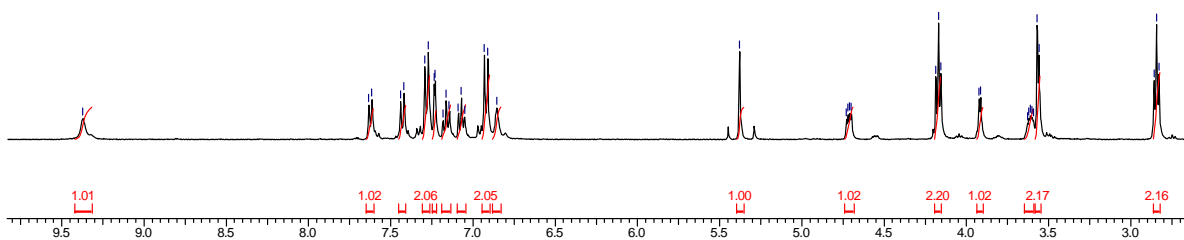
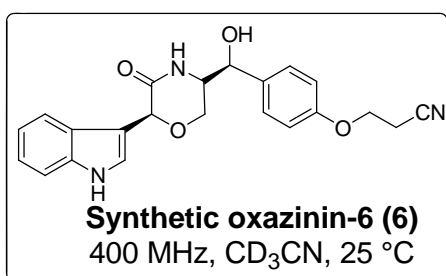


oxazin-6

17 May 2011
 sat5av400#009
 1H

| | | | | | | | |
|------------------------|--------|-----------------------|----------|--------------|---------------------|----------------------|---------|
| Acquisition Time (sec) | 4.0894 | Comment | Vijendra | Date | 30/04/2011 17:36:36 | Frequency (MHz) | 400.13 |
| Nucleus | 1H | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 8012.82 |
| | | | | | | Temperature (grad C) | 0.000 |

9.37
 7.63
 7.61
 7.44
 7.42
 7.29
 7.27
 7.24
 7.23
 7.15
 7.09
 7.07
 7.05
 6.93
 6.91
 6.86
 5.38
 4.73
 4.72
 4.71
 4.70
 4.19
 4.17
 4.16
 3.92
 3.91
 3.83
 3.81
 3.69
 3.69
 3.57
 3.56
 2.86
 2.84
 2.83

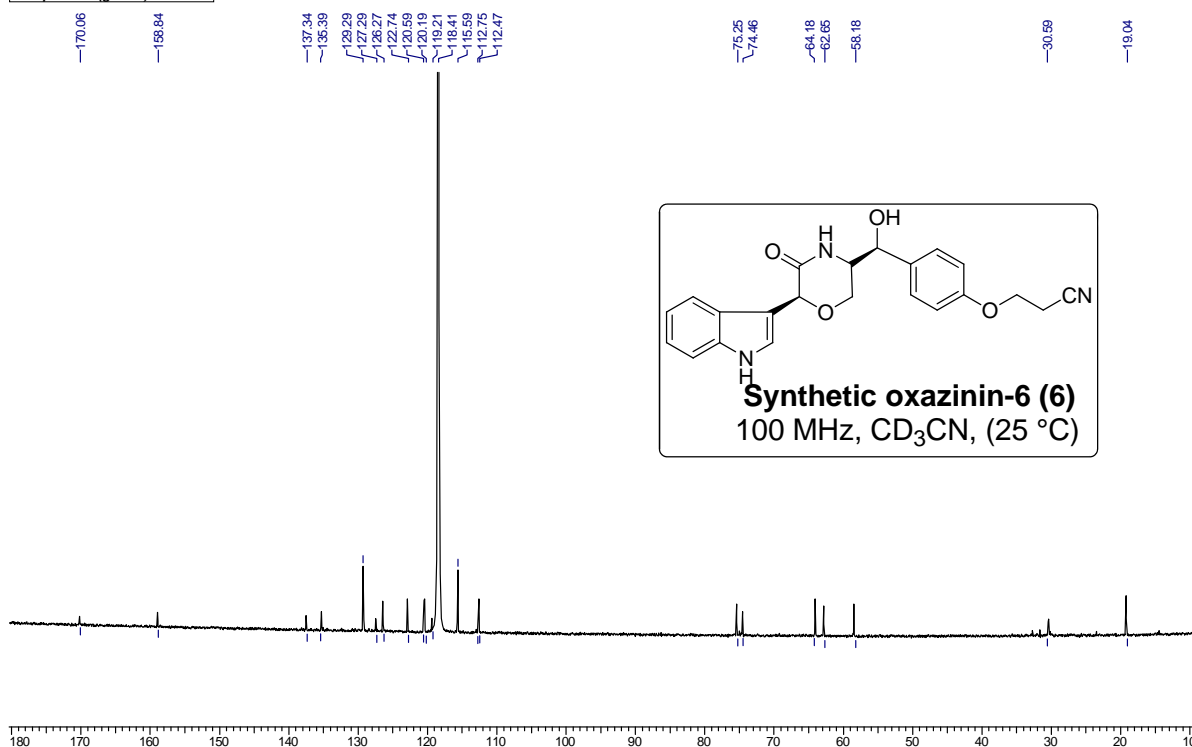


1.01
 1.02
 2.06
 2.05
 1.00
 1.02
 2.20
 1.02
 2.17
 2.16

oxazinin-6

17 May 2011
 sat5av400#009
 13c

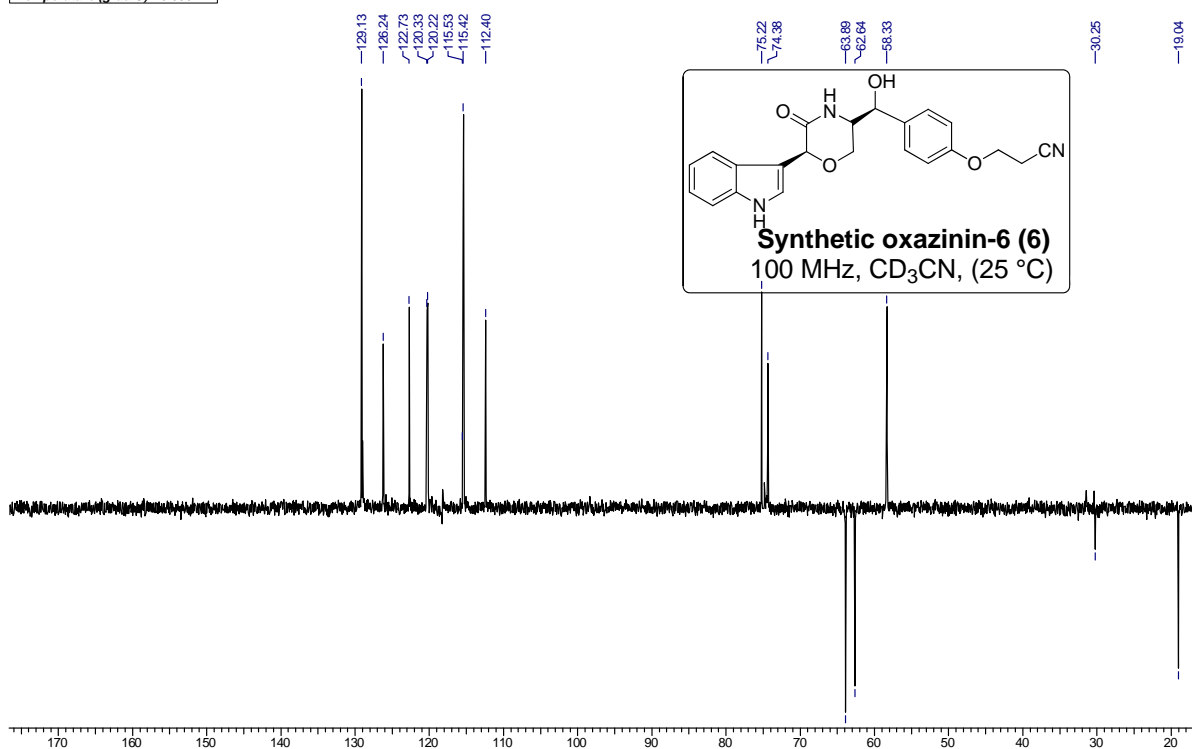
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|------------------------|--------|-----------------------|-------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.3631 | Comment | 13c | Date | 01/05/2011 09:36:18 | Frequency (MHz) | 100.61 |
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 24038.46 |
| Temperature (grad C) | 0.000 | | | | | | |



oxazinin-6

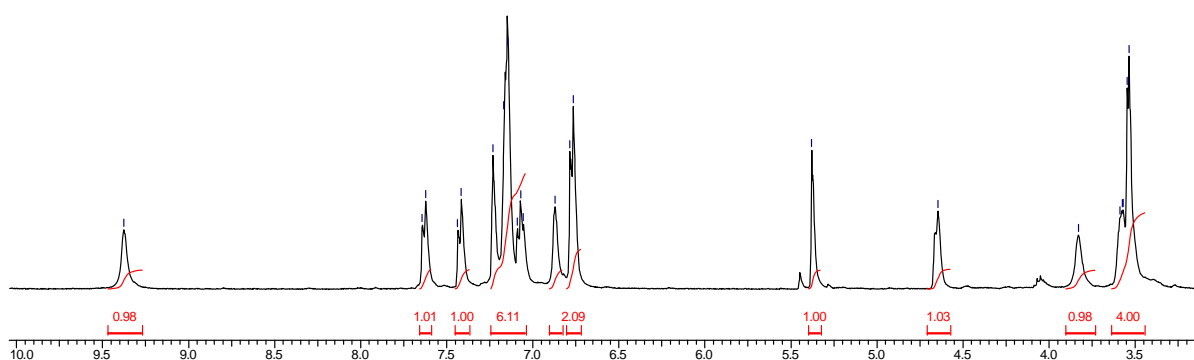
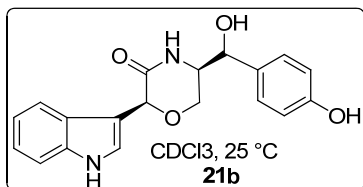
17 May 2011
 sat5av400#009
 Vijendra

| | | | | | | | |
|------------------------|--------|-----------------------|----------|--------------|---------------------|------------------|----------|
| Acquisition Time (sec) | 1.3631 | Comment | Vijendra | Date | 01/05/2011 09:36:58 | Frequency (MHz) | 100.61 |
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 24038.46 |
| Temperature (grad C) | 0.000 | | | | | | |



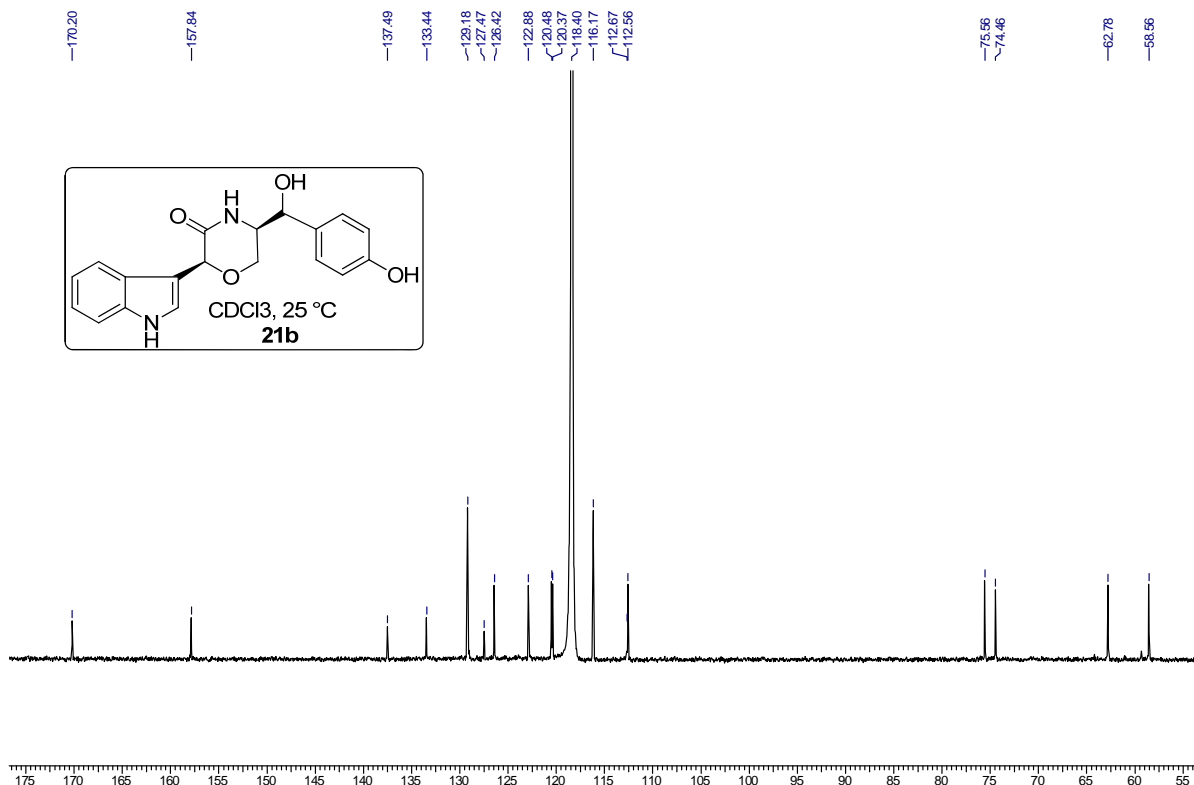
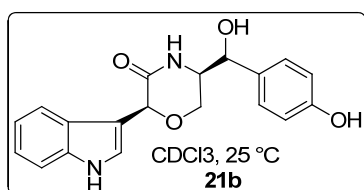
17 May 2011
 Thu2av400#009
 1H side product

| Acquisition Time (sec) | Comment | Vijendra | Date | 13/05/2011 08:57:00 | Frequency (MHz) | 400.13 | | | |
|------------------------|---------|-----------------------|-------|---------------------|-----------------|------------------|---------|----------------------|-------|
| Nucleus | 1H | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 8012.82 | Temperature (grad C) | 0.000 |

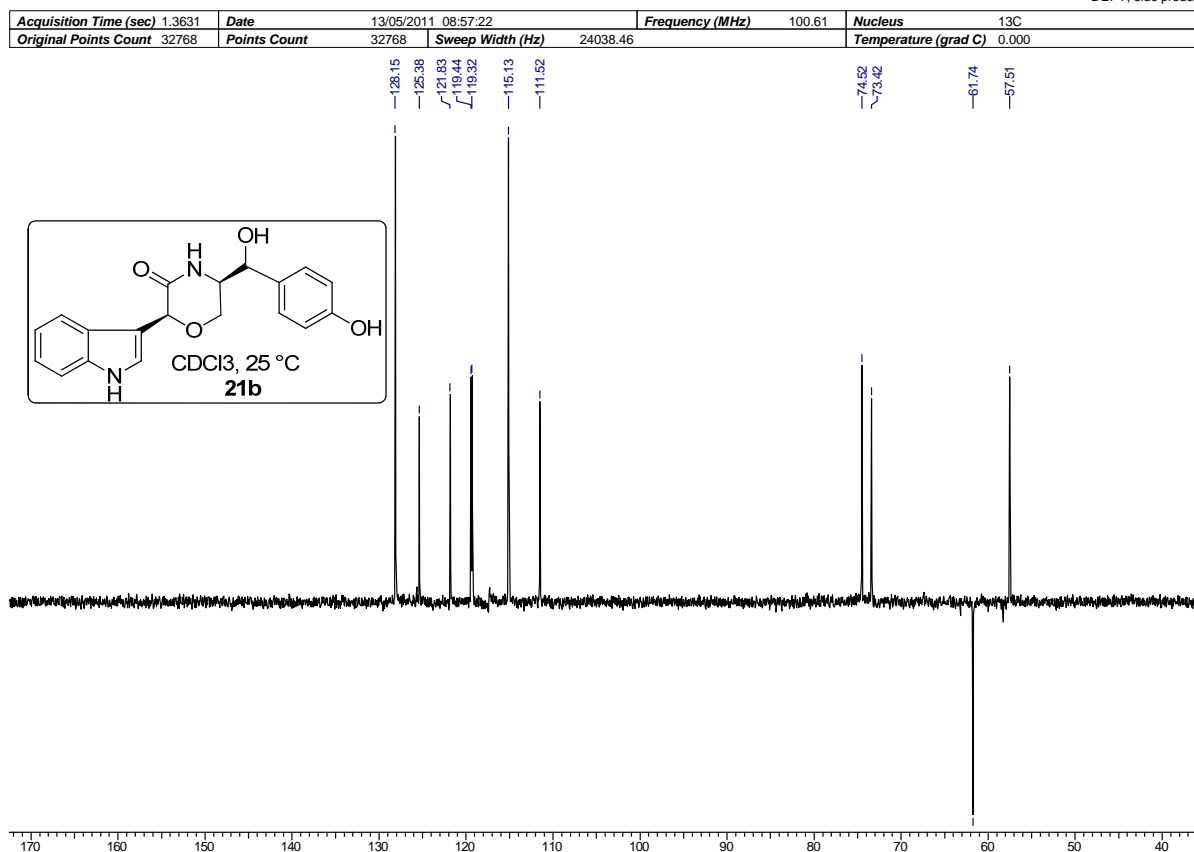


17 May 2011
 Thu2av400#009
 13C side product

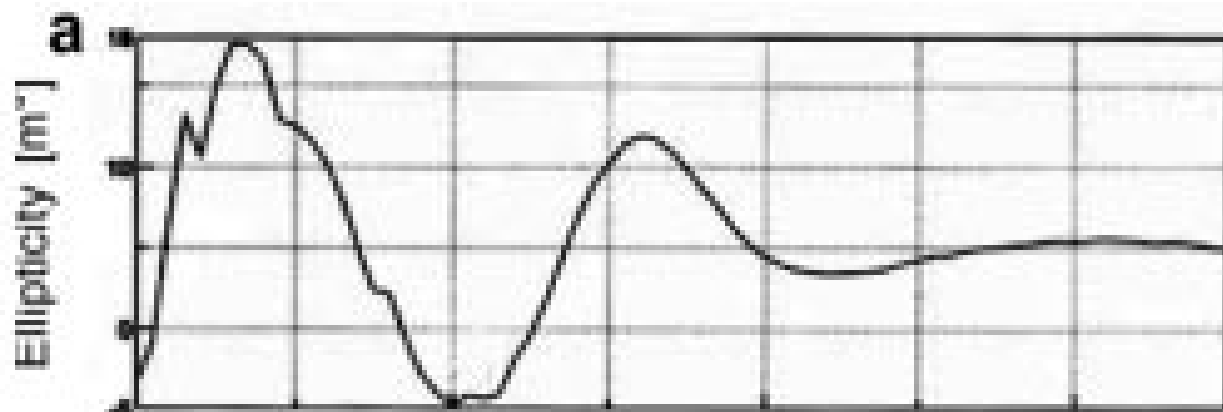
| Acquisition Time (sec) | Comment | 13C | Date | 13/05/2011 08:59:50 | Frequency (MHz) | 100.61 | | | |
|------------------------|---------|-----------------------|-------|---------------------|-----------------|------------------|----------|----------------------|-------|
| Nucleus | 13C | Original Points Count | 32768 | Points Count | 32768 | Sweep Width (Hz) | 24038.46 | Temperature (grad C) | 0.000 |



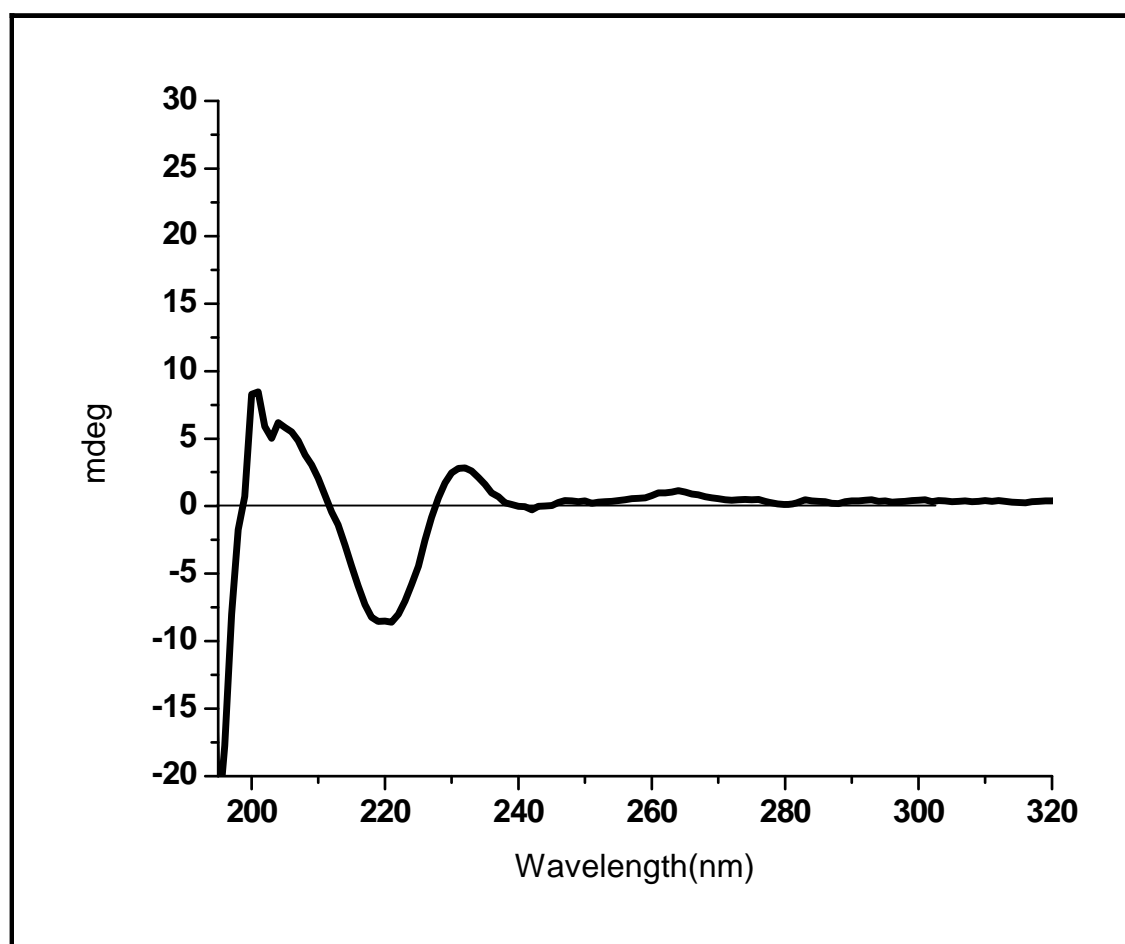
17 May 2011
Thu2av400#009
DEPT, side product



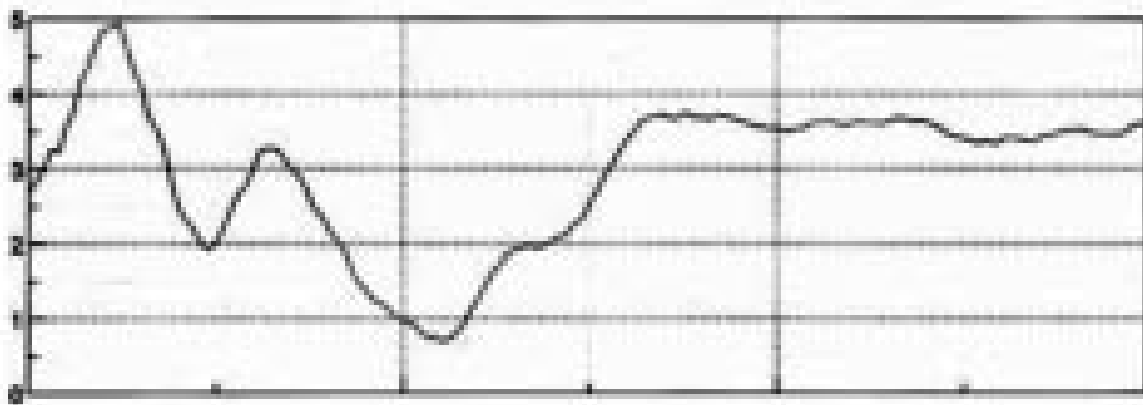
Reported CD Spectra of Natural Oxazinin-5



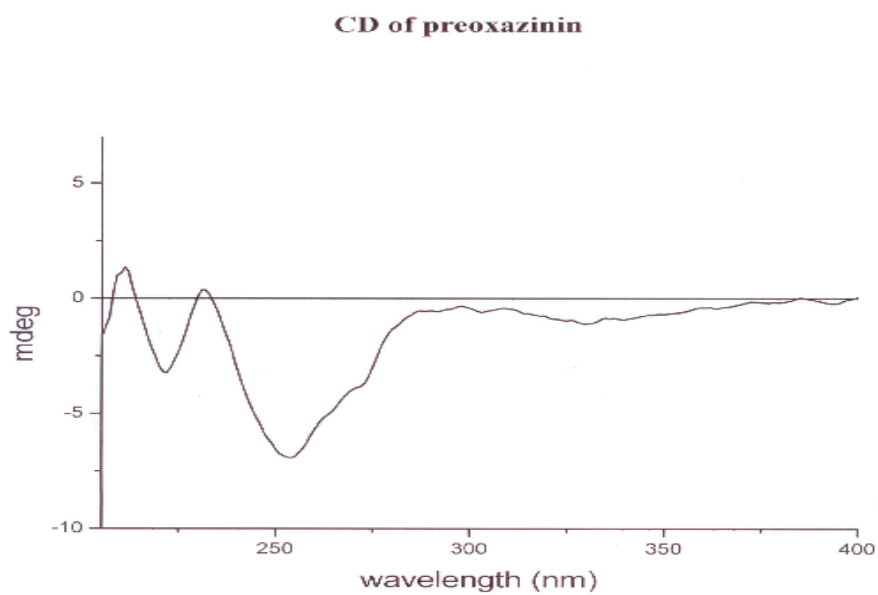
CD Spectra of Synthetic Oxazinin-5



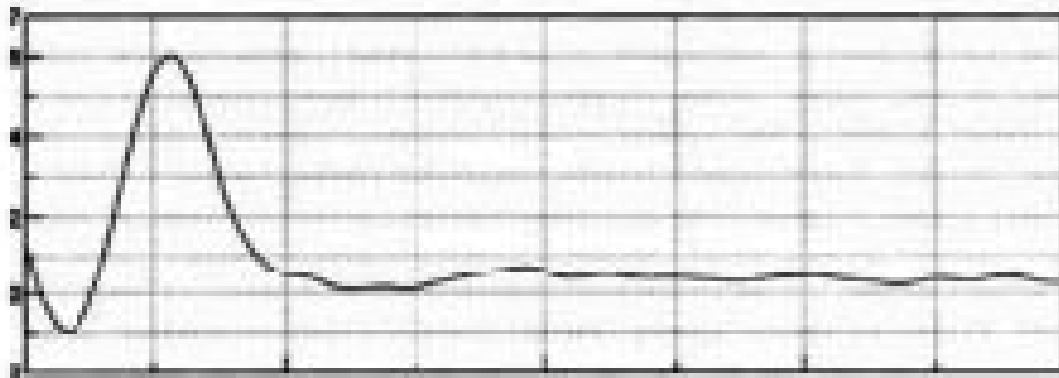
Reported CD spectra of Natural Preoxazinin-7:



CD spectra of Synthetic Preoxazinin -7:



Reported CD spectra of Natural Oxazinin-6:



CD Spectra of Synthetic oxazinin-6:

