

Supporting Information

Synthetic Study of Pactamycin: Enantioselective Construction of the Pactamycin Core with Five Contiguous Stereocenters

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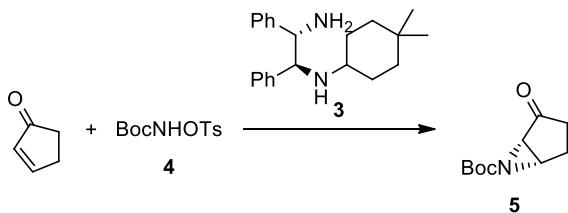
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1. General

Infrared (IR) spectra were recorded on a Fourier transform infrared spectrophotometer, equipped with ATR. Optical rotations were measured on a polarimeter with a sodium lump. NMR spectra were recorded with a 400 MHz spectrometer. Chemical shifts in CDCl_3 were reported downfield from TMS ($= 0$ ppm) for ^1H NMR. For ^{13}C NMR, chemical shifts were reported in the scale relative to the solvent signal [CHCl_3 (77.0 ppm)] as an internal reference. Positive-ion mass spectra were recorded by electrospray ionization (ESI-TOF). Column chromatography was performed with 63–230 mesh spherical neutral silica gel. Reactions were carried out in dry solvent. Other reagents were purified by the usual methods.

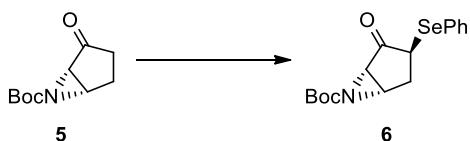
2. Experimental Procedures



tert-Butyl (1*S*,5*S*)-2-oxo-6-azabicyclo[3.1.0]hexane-6-carboxylate (5):

Compound **5** was prepared according to the reported method (Nemoto *et al.* *Tetrahedron* **2016**, doi:10.1016/j.tet.2016.02.067.). To a stirred mixture of **4** (1.25 g, 4.34 mmol), benzoic acid (530 mg, 4.34 mmol), sodium hydrogen carbonate (1.82 g, 21.7 mmol) and **3** (280 mg, 0.868 mmol) in chloroform (43.4 mL) at room temperature was added 2-cyclopenten-1-one (1.06 mL, 13.03 mmol). After being stirred for 14 h, the reaction was quenched by the addition of water. The resulting mixture was extracted with CH_2Cl_2 . The combined organic layers were dried over Na_2SO_4 , and concentrated *in vacuo*. The resulting residue was purified by silica gel column chromatography (*n*-hexane:EtOAc = 4:1) to give (1*S*,5*S*)-**5** as yellow oil (703 mg, 82% yield). The spectroscopic data was identical with the reported value. The enantiomeric excess was determined by the HPLC analysis (JASCO UV-970 (Detector) and PU-980 (Pump), DAICEL CHIRALPAK AD-H, *n*-hexane:2-propanol = 95:5, flow rate = 1 mL/min, λ = 214 nm, retention times; 12.3 min (1*R*,5*R*-isomer), 13.1 min. (1*S*,5*S*-isomer), 99% ee).

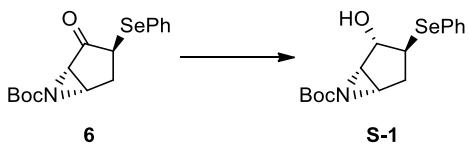
Note: Reaction scale 2.07 g of **4**: 72% yield, 98% ee. Reaction scale: 9.72 g of **4**: 66% yield, 96% ee. Reaction scale: \sim 2 g of **4** was suitable for preparing the highly enantio-enriched aziridine ketone adduct.



tert-Butyl (1*S*,3*S*,5*S*)-2-oxo-3-(phenylselanyl)-6-azabicyclo[3.1.0]hexane-6-carboxylate (6):

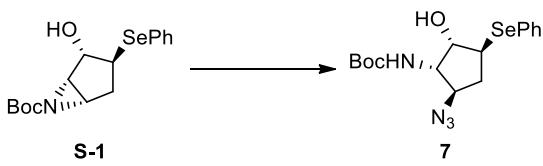
To a stirred solution of **5** (14.7 g, 74.5 mmol) in toluene (370 mL) was added *N,O*-bis(trimethylsilyl)acetamide (54.7 mL, 224 mmol) and DBU (2.20 mL, 14.9 mmol). The reaction mixture was stirred at room temperature for 15 h and cooled to -78 °C using a dry ice-acetone bath. A toluene solution of phenylselenenyl chloride (25.5 g, 149 mmol in 190 mL of toluene) was added dropwise to the reaction over 30 min, and then the reaction was stirred for 30 min. The reaction mixture was poured into saturated aq. NaHCO_3 solution and extracted with EtOAc. The organic extracts were washed with 10% aq. NH_4Cl solution and brine, dried

over sodium sulfate, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAc = 3/1) to give **6** (18.86 g, 71% yield) as yellow oil. IR (ATR) ν 2978, 1745, 1717, 1477, 1368, 1326, 1254, 1149, 843, 741, 691 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 1.41 (9H, s), 2.18 (1H, ddd, J = 3.2, 8.4, 14.8 Hz), 2.90 (1H, dd, J = 8.4, 14.8 Hz), 3.10 (1H, d, J = 3.2 Hz), 3.28 (1H, dd, J = 3.2, 3.2 Hz), 3.55 (1H, dd, J = 8.4, 8.4 Hz), 7.24–7.32 (3H, m), 7.54–7.58 (2H, m); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 27.7 (3C), 31.0, 41.1, 42.1, 42.8, 82.7, 127.3, 128.4, 129.2 (2C), 135.0 (2C), 158.1, 204.1; HRMS (ESI-TOF); calcd for $\text{C}_{16}\text{H}_{19}\text{NNaO}_3\text{Se}^+$ [$\text{M}+\text{Na}^+$]: 376.0422, found 376.0429; $[\alpha]_D^{24} +59.4$ (c 1.59, CHCl_3).



tert-Butyl

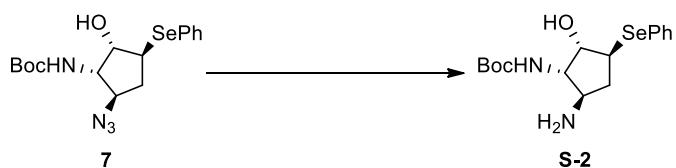
(1*S*,2*S*,3*S*,5*S*)-2-hydroxy-3-(phenylselanyl)-6-azabicyclo[3.1.0]hexane-6-carboxylate (S-1):
 Compound **6** (18.8 g, 53.5 mmol) in $\text{THF-H}_2\text{O}$ (267 mL-13 mL) was cooled to -78°C in a dry ice-acetone bath and NaBH_4 (2.03 g, 53.5 mmol) was then added to the mixture. After being stirred for 40 min, the reaction mixture was quenched with water and allowed to warm to room temperature. The whole was extracted with ethyl acetate, washed with brine, dried over sodium sulfate, and then concentrated *in vacuo*. The resulting crude product **S-1** (17.2 g) was used for the next step without further purification. IR (ATR) ν 3405, 2977, 1712, 1367, 1253, 1147, 1071, 1022, 837, 739, 691 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 1.43 (9H, s), 1.40–1.55 (1H, m), 1.89 (1H, ddd, J = 2.4, 10.4, 14.0 Hz), 2.65 (1H, dd, J = 7.6, 14.0 Hz), 2.84–2.90 (2H, m), 3.05–3.07 (1H, m), 4.16–4.21 (1H, m), 7.25–7.33 (3H, m), 7.58–7.61 (2H, m); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 27.8 (3C), 34.2, 40.5, 42.4, 44.2, 78.5, 81.9, 127.7, 128.1, 129.0 (2C), 135.4 (2C), 161.6; HRMS (ESI-TOF); calcd for $\text{C}_{16}\text{H}_{21}\text{NNaO}_3\text{Se}^+$ [$\text{M} + \text{Na}^+$]: 378.0579, found 378.0566; $[\alpha]_D^{25} +39.0$ (c 0.94, CHCl_3).



tert-Butyl [(1*S*,2*S*,3*S*,5*R*)-5-azido-2-hydroxy-3-(phenylselanyl)cyclopentyl]carbamate (7):

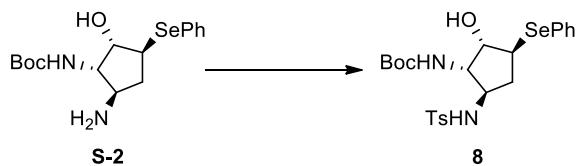
To a stirred solution of **S-1** (17.2 g, 48.5 mmol) in MeOH (320 mL) were added AcOH (14.0 mL), water (65.0 mL), and NaN_3 (15.8 g, 242.5 mmol). After being stirred at 40°C for 45 h, the reaction mixture was diluted with saturated aq. NaHCO_3 solution and extracted with EtOAc .

The combined organic layers were washed with brine, dried over sodium sulfate, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 3/1) to give **7** (16.96 g, 80 % yield in 2 steps) as pale yellow solid. Mp. 123–125 °C; IR (ATR) ν 3360, 2979, 2100, 1686, 1506, 1367, 1252, 1165, 1021, 741, 691 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 1.46 (9H, s), 1.56–1.62 (1H, m), 2.40 (1H, br-s), 2.65 (1H, ddd, *J* = 8.0, 8.4, 14.0 Hz), 3.44 (1H, ddd, *J* = 3.2, 8.0, 8.0 Hz), 3.77–3.90 (1H, m), 3.97–4.07 (1H, m), 4.15–4.19 (1H, m), 5.02 (1H, br-s), 7.27–7.33 (3H, m), 7.54–7.59 (2H, m); ¹³C-NMR (CDCl₃, 100 MHz) δ 28.3 (3C), 34.4, 42.9, 58.2, 63.9, 76.6, 80.2, 127.9, 128.8, 129.3 (2C), 134.2, 135.2, 155.7; HRMS (ESI-TOF); calcd for C₁₆H₂₂N₄NaO₃Se⁺ [M + Na⁺]: 421.0749, found 421.0749; $[\alpha]_D^{25}$ -63.4 (*c* 0.48, CHCl₃).



tert-Butyl (1*S*,2*S*,3*S*,5*R*)-5-amino-2-hydroxy-3-(phenylselanyl)cyclopentylcarbamate (S-2):

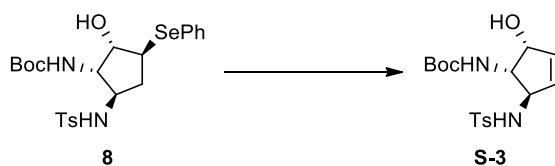
To a stirred solution of **7** (4.72 g, 11.88 mmol) and NH₄Cl (1.90 g, 35.64 mmol) in EtOH-H₂O (45 mL-15 mL) was added Zn powder (2.33 g, 35.64 mmol), and the reaction mixture was refluxed at 110 °C. After 2.5 h, the reaction mixture was diluted with EtOAc, and quenched with aq. NH₃ solution. The insoluble Zn powder was removed by filtration through celite. The filtrate was separated and the combined organic extracts were washed with brine, dried over sodium sulfate, and concentrated *in vacuo*. The resulting pale yellow solid **S-2** (4.41 g) was used for the next step without further purification. Mp. 143–146 °C; IR (ATR) ν 3363, 1680, 1533, 1327, 1249, 1170, 1058, 980, 737, 689 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 1.31 (1H, ddd, *J* = 8.0, 9.2, 14.0 Hz), 1.45 (9H, s), 1.38–2.27 (3H, broad peak), 2.54 (1H, ddd, *J* = 6.8, 8.4, 14.0 Hz), 3.14 (1H, dd, *J* = 8.4, 16.8 Hz), 3.38–3.44 (1H, m), 3.54–3.71 (1H, m), 4.11–4.15 (1H, m), 5.11 (1H, br-s), 7.25–7.32 (3H, m), 7.53–7.60 (2H, m); ¹³C-NMR (CDCl₃, 100 MHz) δ 28.4 (3C), 37.9, 44.0, 56.7, 60.8, 77.2, 79.8, 127.7, 129.2 (2C), 129.3, 134.1, 134.8, 156.4; HRMS (ESI-TOF); calcd for C₁₆H₂₄N₂NaO₃Se⁺ [M + Na⁺]: 395.0844, found 395.0860; $[\alpha]_D^{25}$ -88.6 (*c* 0.50, CHCl₃).



tert-Butyl

{(1*S*,2*S*,3*S*,5*R*)-2-hydroxy-5-[(4-methylphenyl)sulfonamide]-3-(phenylselanyl)cyclopentyl}carbamate (8):

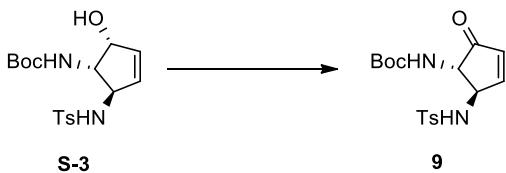
To a stirred solution of **S-2** (4.41 g) and *p*-toluenesulfonyl chloride (2.26 g, 11.88 mmol) in CH_2Cl_2 (118.8 mL) at 0 °C was added triethylamine (1.82 mL, 13.07 mmol), and the resulting solution was stirred at room temperature. After 2 h, the mixture was quenched with water. The combined organic layers were dried over sodium sulfate and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 3/1) to give **8** (5.80 g, 93% yield in 2 steps) as white solid. Mp. 151–153 °C; IR (ATR) ν 3434, 2977, 1685, 1508, 1366, 1304, 1159, 1093, 814, 741, 669 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 1.40 (9H, s), 1.53 (1H, ddd, J = 8.0, 8.0, 14.4 Hz), 2.41 (3H, s), 2.44 (1H, br-s), 2.59 (1H, ddd, J = 8.0, 8.0, 14.4 Hz), 3.30–3.45 (2H, m), 3.88–3.94 (1H, m), 4.09–4.11 (1H, m), 5.18 (1H, d, J = 6.0 Hz), 6.09 (1H, br-s), 7.23–7.33 (5H, m), 7.47–7.58 (2H, m), 7.70 (2H, d, J = 8.4 Hz); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 21.5, 28.3 (3C), 36.4, 43.4, 57.2, 58.4, 75.9, 80.3, 127.0 (2C), 128.0, 128.8, 129.3 (2C), 129.6 (2C), 134.3 (2C), 137.1, 143.2, 156.9; HRMS (ESI-TOF); calcd for $\text{C}_{23}\text{H}_{30}\text{N}_2\text{NaO}_5\text{SSe}^+$ [$\text{M} + \text{Na}^+$]: 549.0933, found 549.0934; $[\alpha]_D^{25} -114.9$ (*c* 0.50, CHCl_3).



tert-Butyl

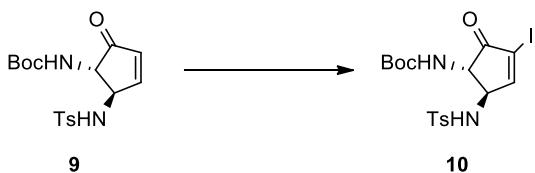
{(1*S*,2*R*,5*R*)-2-hydroxy-5-[(4-methylphenyl)sulfonamide]cyclopent-3-en-1-yl}carbamate (S-3):

To a stirred solution of **8** (2.62 g, 4.985 mmol) in THF (50 mL) at room temperature were added pyridine (0.80 mL, 9.971 mmol) and 30% hydrogen peroxide (1.54 mL, 14.95 mmol). After being stirred for 1 h, the reaction mixture was treated with saturated aq. NaHCO_3 solution and extracted with EtOAc. The organic extracts were washed with brine, dried over sodium sulfate, and concentrated *in vacuo*. The resulting pale yellow solid **S-3** (1.83 g) was used for the next step without further purification. Mp. 63–64 °C; IR (ATR) ν 3359, 1678, 1529, 1328, 1154, 1081, 919, 811, 734, 664 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 1.38 (9H, s), 2.40 (3H, s), 2.92 (1H, d, J = 6.0 Hz), 3.75 (1H, dd, J = 6.8, 13.2 Hz), 3.92–3.95 (1H, m), 4.50–4.54 (1H, m), 5.46 (1H, d, J = 7.2 Hz), 5.91–5.94 (1H, m), 6.05–6.07 (1H, m), 6.52 (1H, d, J = 2.0 Hz), 7.25 (2H, d, J = 8.0 Hz), 7.71 (2H, d, J = 8.0 Hz); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 21.4, 28.2 (3C), 58.3, 63.7, 72.2, 80.1, 127.1 (2C), 129.6 (2C), 132.6, 136.3, 137.1, 143.3, 157.0; HRMS (ESI-TOF); calcd for $\text{C}_{17}\text{H}_{24}\text{N}_2\text{NaO}_5\text{S}^+$ [$\text{M} + \text{Na}^+$]: 391.1298, found 391.1289; $[\alpha]_D^{19} -39.3$ (*c* 0.63, CHCl_3).



tert-Butyl {(1*S*,2*R*)-2-[(4-methylphenyl)sulfonamide]-5-oxocyclopent-3-en-1-yl}carbamate (9):

A solution of **S-3** (1.83 g) and IBX (4.187 g, 14.95 mmol) in DMSO (50 mL) was stirred at room temperature. After 3 h, the reaction was quenched by the addition of water and extracted with EtOAc. The organic extracts were washed with saturated aq. NaHCO₃ solution and brine, dried over sodium sulfate, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 1/1) to give **9** (1.789 g, 98% yield) as white solid. Mp. 117–120 °C; IR (ATR) ν 3251, 2979, 1728, 1509, 1367, 1329, 1160, 1092, 815, 668 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 1.40 (9H, s), 2.43 (3H, s), 3.83–3.87 (1H, m), 3.89 (1H, dd, *J* = 3.6, 3.6 Hz), 5.18 (1H, d, *J* = 3.6 Hz), 6.30 (1H, dd, *J* = 6.4, 1.6 Hz), 6.70 (1H, br-s), 7.30 (2H, d, *J* = 8.0 Hz), 7.79 (2H, d, *J* = 8.0 Hz), 7.81–7.83 (1H, m); ¹³C-NMR (CDCl₃, 100 MHz) δ 21.5, 28.1 (3C), 61.1, 62.1, 81.2, 127.5 (2C), 129.8 (2C), 132.3, 135.4, 143.9, 156.9, 160.1, 199.1; HRMS (ESI-TOF); calcd for C₁₇H₂₂N₂NaO₅S⁺ [M+Na⁺]: 389.1142, found 389.1124; [α]_D²⁵ +39.6 (*c* 0.50, CHCl₃).

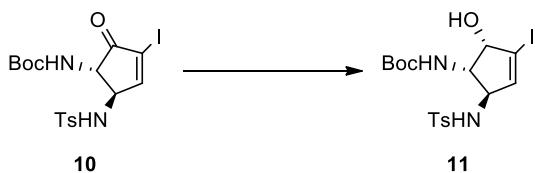


tert-Butyl

{(1*S*,5*R*)-3-iodo-5-[(4-methylphenyl)sulfonamide]-2-oxocyclopent-3-en-1-yl}carbamate (10):

To a stirred solution of **9** (2.09 g, 5.70 mmol) in CH₂Cl₂ (50 mL) was added iodine (4.34 g, 17.1 mmol) in pyridine (10 mL) at 0 °C. After being stirred for 15 min at room temperature, the reaction was quenched by the addition of 1N aq. HCl solution and extracted with CH₂Cl₂. The organic extracts were washed with 0.5 M aq. Na₂S₂O₃ solution, dried over sodium sulfate, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 4/1) to give **10** (2.27 g, 81% yield) as white solid. Mp. 143–145 °C; IR (ATR) ν 3260, 2978, 1697, 1497, 1367, 1333, 1254, 1153, 1087, 875, 814, 734, 661 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 1.39 (9H, s), 2.43 (3H, s), 3.92 (1H, dd, *J* = 3.2, 4.0 Hz), 4.00–4.04 (1H, m), 5.22 (1H, d, *J* = 4.8 Hz), 6.59 (1H, d, *J* = 3.2 Hz), 7.30 (2H, d, *J* = 8.0 Hz),

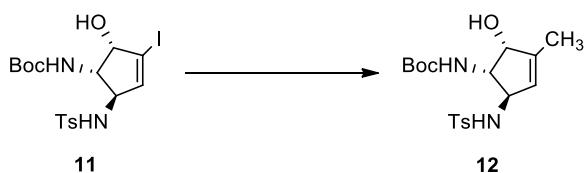
7.77 (2H, d, J = 8.0 Hz), 8.02 (1H, d, J = 2.0 Hz); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 21.6, 28.1 (3C), 59.7, 62.0, 81.4, 100.8, 127.4 (2C), 129.9 (2C), 135.5, 144.1, 156.2, 164.7, 195.1; HRMS (ESI-TOF); calcd for $\text{C}_{17}\text{H}_{21}\text{IN}_2\text{NaO}_5\text{S}^+$ [M+Na $^+$]: 515.0108, found 515.0112; $[\alpha]_D^{25} -48.3$ (c 1.00, CHCl_3).



tert-Butyl

{(1*S*,2*S*,5*R*)-2-hydroxy-3-iodo-5-[(4-methylphenyl)sulfonamide]cyclopent-3-en-1-yl}carbamate (11):

A solution of **10** (449 mg, 0.913 mol) and aluminum isopropoxide (3.73 g, 18.3 mmol) in isopropanol (30.4 mL) was stirred at 100 °C. After 2 h, the reaction was quenched by the addition of 1N aq. HCl solution and the resulting mixture was extracted with CH_2Cl_2 . The organic extracts were washed with water, dried over sodium sulfate, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 3/1) to give **11** (367 mg, 81% yield) as white solid. Mp. 144–145 °C; IR (ATR) ν 3864, 3422, 2977, 1684, 1514, 1326, 1156, 1087, 757, 654 cm^{-1} ; ^1H -NMR (CDCl_3 , 400 MHz) δ 1.40 (9H, s), 2.42 (3H, s), 2.53 (1H, d, J = 5.6 Hz), 3.83–3.88 (1H, m), 3.90–3.96 (1H, m), 4.50 (1H, dd, J = 5.2, 5.6 Hz), 5.37 (1H, d, J = 6.8 Hz), 6.36 (1H, d, J = 2.4 Hz), 6.55 (1H, br-s), 7.27 (2H, d, J = 8.4 Hz), 7.72 (2H, d, J = 8.4 Hz); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 21.5, 28.2 (3C), 57.7, 64.6, 79.3, 80.6, 98.9, 127.2 (2C), 129.7 (2C), 136.2, 143.5, 144.3, 156.8; HRMS (ESI-TOF); calcd for $\text{C}_{17}\text{H}_{23}\text{IN}_2\text{NaO}_5\text{S}^+$ [M+Na $^+$]: 517.0265, found 517.0280; $[\alpha]_D^{21} -118.3$ (c 0.50, CHCl_3).

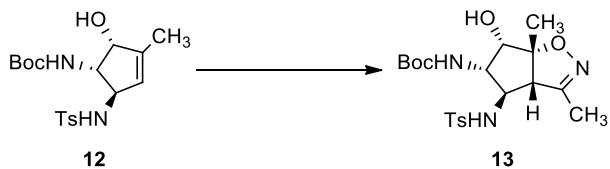


tert-Butyl

{(1*S*,2*R*,5*R*)-2-hydroxy-3-methyl-5-[(4-methylphenyl)sulfonamide]cyclopent-3-en-1-yl}carbamate (12):

To a stirred solution of **11** (589.2 mg, 1.19 mmol), $\text{Pd}(\text{dba})_2$ (68.5 mg, 0.119 mmol), and AsPh_3 (146 mg, 0.476 mmol) in freshly distilled HMPA (12 mL) at room temperature was added SnMe_4 (0.66 mL, 4.76 mmol). The resulting solution was heated to 60 °C and kept stirring for 1 h. The resulting mixture was cooled to room temperature and 1 M aq. KF solution was added to

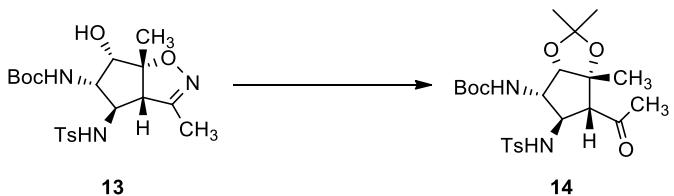
the mixture. The resulting mixture was stirred overnight. The mixture was filtered through celite and extracted with Et_2O . The organic extracts were washed with H_2O and brine, dried over sodium sulfate, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 3/1) to give **12** (361.7 mg, 79% yield) as white solid. Mp. 131–133 °C; IR (ATR) ν 3422, 2978, 1685, 1496, 1366, 1326, 1155, 1087, 1011, 814, 736, 661 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 1.40 (9H, s), 1.75 (3H, s), 2.41 (3H, s), 2.58 (1H, d, J = 6.4 Hz), 3.74–3.79 (1H, m), 3.86–3.90 (1H, m), 4.29 (1H, dd, J = 6.0, 6.4 Hz), 5.42 (1H, d, J = 6.4 Hz), 5.65 (1H, br-s), 6.37 (1H, d, J = 2.8 Hz), 7.25 (2H, d, J = 8.4 Hz), 7.72 (2H, d, J = 8.4 Hz); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 14.6, 21.5, 28.2 (3C), 58.9, 64.1, 75.4, 80.1, 127.2 (2C), 129.6 (2C), 129.8, 136.5, 142.3, 143.2, 156.9; HRMS (ESI-TOF); calcd for $\text{C}_{18}\text{H}_{26}\text{N}_2\text{NaO}_5\text{S}^+$ [$\text{M}+\text{Na}^+$]: 405.1455, found 405.1450; $[\alpha]_D^{25}$ –30.7 (*c* 0.50, CHCl_3).



tert-Butyl

{(3a*S*,4*R*,5*S*,6*S*,6a*R*)-6-hydroxy-3,6a-dimethyl-4-[(4-methylphenyl)sulfonamide]-3a,5,6,6a-tetrahydro-4*H*-cyclopenta[*d*]isoxazol-5-yl}carbamate (13):

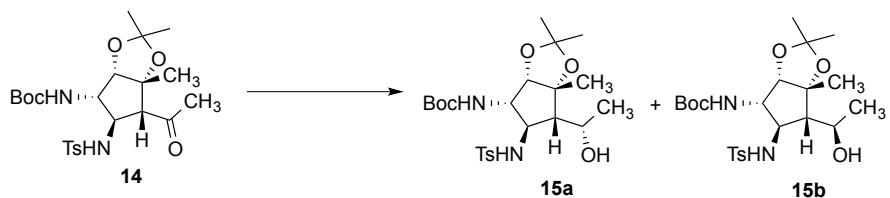
To a stirred solution of acetaldoxime (1.2 mL, 19.74 mmol) in CH_2Cl_2 (8.0 mL) at –78 °C was added *t*-butyl hypochlorite (2.1 mL, 18.51 mmol) slowly. After 5 min, the resulting solution was changed to blue. A solution of **12** (472 mg, 1.234 mmol) and triethylamine (2.6 mL, 18.51 mmol) in CH_2Cl_2 (4.0 mL) at 0 °C was added dropwise to the reaction over 30 min. The mixture was gradually allowed to warm to room temperature and kept stirring for 48 h. The reaction mixture was diluted with CH_2Cl_2 and washed with 10% aq. NH_4Cl solution. The organic layer was dried over sodium sulfate and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 3/1 to 1/1 to 1/2) to give **13** (361.6 mg, 66% yield) as white solid and **12** was recovered (79.0 mg, 17% yield). Mp. 190–193 °C; IR (ATR) ν 3303, 2977, 1696, 1523, 1451, 1366, 1323, 1235, 1158, 1092, 1051, 909, 860, 813, 668 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 1.34 (3H, s), 1.38 (9H, s), 2.20 (3H, s), 2.43 (3H, s), 3.30 (1H, d, J = 8.4 Hz), 3.40–3.58 (1H, broad peak), 3.70–3.79 (1H, m), 3.84–3.96 (2H, m), 5.07 (1H, d, J = 7.2 Hz), 6.27 (1H, br-s), 7.27 (2H, d, J = 8.4 Hz), 7.70 (2H, d, J = 8.4 Hz); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 14.7, 20.0, 21.5, 28.2 (3C), 55.4, 60.6, 61.1, 74.7, 80.6, 92.9, 126.9 (2C), 129.7 (2C), 136.6, 143.4, 156.5, 157.2; HRMS (ESI-TOF); calcd for $\text{C}_{20}\text{H}_{29}\text{N}_3\text{NaO}_6\text{S}^+$ [$\text{M}+\text{Na}^+$]: 462.1669, found 462.1669; $[\alpha]_D^{26}$ –114.9 (*c* 0.50, CHCl_3).



tert-butyl

[(3a*S*,4*S*,5*R*,6*R*,6a*R*)-6-acetyl-2,2,6a-trimethyl-5-(4-methylphenylsulfonamido)tetrahydro-3a*H*-cyclopenta[*d*][1,3]dioxol-4-yl]carbamate (14):

A suspension of **13** (228 mg, 0.518 mmol) and Raney Ni in MeOH (5.2 mL) was stirred at room temperature under a hydrogen atmosphere. After 7 h, the reaction was quenched by the addition of water. After removal of Raney Ni by decantation, the resulting mixture was extracted with CH₂Cl₂ and the organic extracts were dried over sodium sulfate and concentrated *in vacuo*. The resulting white solid (229 mg) was used for the next step without further purification. To a stirred solution of the product in 2,2-dimethoxypropane (5.2 mL) at room temperature was added *p*-toluenesulfonic anhydride (50.7 mg, 0.1554 mmol). After being stirred for 4 h, the reaction mixture was quenched with saturated aq. NaHCO₃ solution and extracted with Et₂O. The organic extracts were washed with brine, dried over sodium sulfate, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane:EtOAc = 4/1 to 1/1) to give **14** (147.5 mg, 59% yield in 2 steps) as white solid. Mp. 77–78 °C; IR (ATR) ν 3276, 2981, 2929, 1716, 1507, 1368, 1264, 1158, 733 cm⁻¹; ¹H-NMR (CDCl₃, 400 MHz) δ 1.25 (3H, s), 1.28 (3H, s), 1.44 (9H, s), 1.65 (3H, s), 2.12 (3H, s), 2.42 (3H, s), 2.94 (1H, d, *J* = 8.4 Hz), 3.97–4.09 (2H, m), 4.14 (1H, d, *J* = 4.4 Hz), 5.12 (1H, d, *J* = 8.4 Hz), 6.34 (1H, d, *J* = 3.6 Hz), 7.26 (2H, d, *J* = 8.4 Hz), 7.69 (2H, d, *J* = 8.4 Hz); ¹³C-NMR (CDCl₃, 100 MHz) δ 21.5, 25.5, 25.7, 27.7, 28.2 (3C), 31.2, 55.9, 59.0, 65.5, 80.5, 84.0, 85.8, 111.7, 127.4 (2C), 129.3 (2C), 137.0, 143.0, 156.8, 201.9; HRMS (ESI-TOF); calcd for C₂₃H₃₄N₂NaO₇S⁺ [M+Na⁺]: 505.1979, found 505.1955; $[\alpha]_D^{24}$ -46.2 (*c* 0.67, CHCl₃).

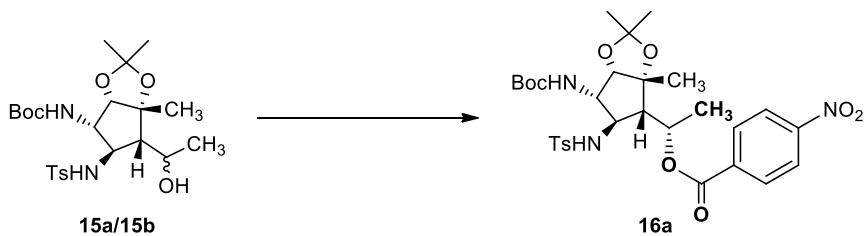


tert-Butyl

[(3a*S*,4*S*,5*R*,6*S*,6a*R*)-6-[(*S*)-1-hydroxyethyl]-2,2,6a-trimethyl-5-(4-methylphenylsulfonamido)tetrahydro-4*H*-cyclopenta[*d*][1,3]dioxol-4-yl]carbamate (15a):

To a stirred solution of **14** (83.1 mg, 0.172 mmol) in THF (3.44 mL) was added DIBAL-H (1.7

mL, 1.02 M in hexane, 1.72 mmol) at -78°C . After being stirred for 1.5 h, the reaction mixture was quenched with 2 M Rochelle salt solution, allowed to warm to room temperature, and kept stirring for 30 min. The resulting mixture was extracted with CH_2Cl_2 , dried over sodium sulfate, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAc = 2/1 to 1/1) to give **15a** and **15b** (75.9 mg, 91% yield, dr = 5:1) as white solid. Because of the difficult separation of **15a** and **15b**, the obtained mixture was utilized for the next reaction without further purification.



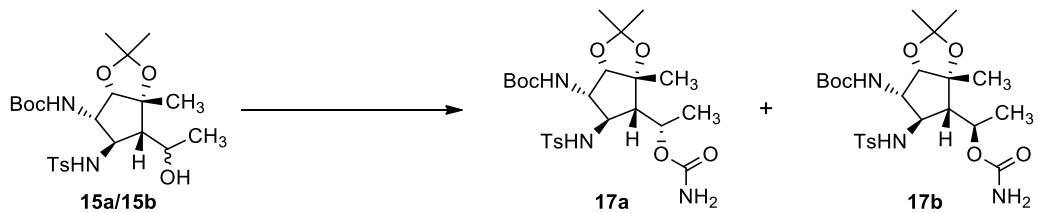
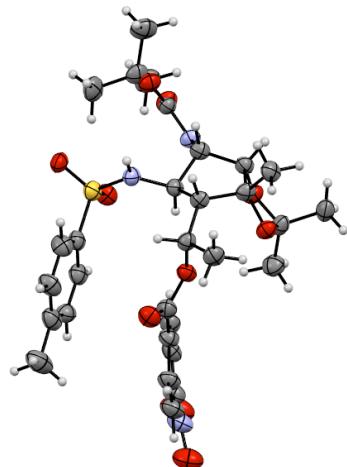
(S)-1-[(3a*R*,4*S*,5*R*,6*S*,6a*S*)-6-[(*tert*-butoxycarbonyl)amino]-2,2,3*a*-trimethyl-5-(4-methylphenylsulfonamido)tetrahydro-3*a*H-cyclopenta[*d*][1,3]dioxol-4-yl]ethyl 4-nitrobenzoate (16a):

To a stirred solution of the mixture of **15a** and **15b** (30.0 mg, 0.0619 mmol) in CH_2Cl_2 (1.2 mL) at 0°C was added DMAP (3.0 mg, 0.0247 mmol), *p*-nitrobenzoyl chloride (17.2 mg, 0.0928 mmol), and triethylamine (13.0 μL , 0.0928 mmol), and the reaction mixture was stirred at room temperature. After 24 h, the reaction was quenched with aq. NaHCO_3 , and the resulting mixture was extracted with EtOAc. The combined organic layers were washed with brine, and then dried over Na_2SO_4 . After concentration *in vacuo*, the obtained crude residue was purified by silica gel column chromatography (*n*-hexane/EtOAc = 5/1 to 3/1) to give pure **16a** (15.6 mg, 40% yield) as white solid; Mp. 52–53 $^{\circ}\text{C}$; IR (ATR) ν 3283, 2928, 1718, 1529, 1455, 1348, 1278, 1162, 1103, 721 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3 , 400MHz) δ 1.20–1.70 (12H, m), 1.40 (9H, s), 2.39 (3H, s) 3.80–3.90 (2H, m), 4.11 (1H, br-s), 4.90 (1H, d, J = 6.0 Hz), 5.29 (1H, d, J = 4.8 Hz), 5.40–5.48 (1H, m), 7.20–7.30 (2H, m), 7.74 (2H, d, J = 7.6 Hz), 8.12 (2H, d, J = 7.6 Hz), 8.21 (2H, d, J = 7.6 Hz), 8.25–8.35 (1H, m); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 18.0, 21.5, 25.9, 26.7, 27.2, 28.3 (3C), 54.8, 56.7, 57.3, 71.1, 79.9, 82.6, 84.1, 110.4, 123.3 (2C), 126.8 (2C), 129.6 (2C), 130.6 (2C), 136.2, 138.7, 143.1, 150.2, 156.0, 163.6; HRMS (ESI-TOF); calcd for $\text{C}_{30}\text{H}_{39}\text{N}_3\text{NaO}_{10}\text{S}^+ [\text{M}+\text{Na}^+]$: 656.2248, found: 656.2250; $[\alpha]_D^{22} +4.97$ (c 1.50, CHCl_3).

X-ray structure of **16a** (CCDC 1457178.)

Crystal Data: Colorless block crystal, $\text{C}_{30}\text{H}_{39}\text{N}_3\text{O}_{10}\text{S}$, M = 633.71, triclinic, a = 12.1373(3), b = 12.1458(3), c = 12.5058(3) \AA , V = 1593.07(6) \AA^3 , T = -180°C , space group P-1(#2), Z = 2, 17254 reflections measured, 5695 unique (Rint = 0.0671). R1 = 0.0634, wR2 = 0.1954.

ORTEP of **16a**



tert-Butyl

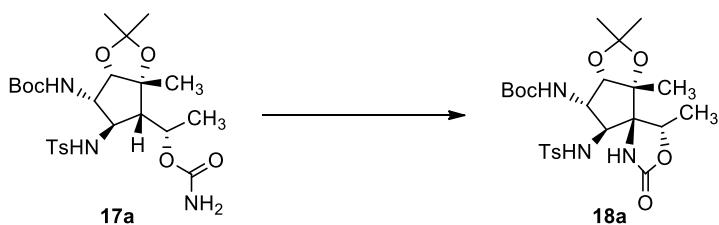
{(3a*S*,4*S*,5*R*,6*S*,6a*R*)-6-[(*S*)-1-(carbamoyloxy)ethyl]-2,2,6a-trimethyl-5-[(4-methylphenyl)sulfonamido]tetrahydro-4*H*-cyclopenta[*d*][1,3]dioxol-4-yl}carbamate (17a)

To a stirred solution of the mixture of **15a** and **15b** (162 mg, 0.334 mmol) in CH_2Cl_2 (3.3 mL) at 0 °C was added trichloroacetyl isocyanate (48 μL , 0.401 mmol), and the resulting mixture was allowed to warm to room temperature. After being stirred for 30 min, the reaction mixture was concentrated *in vacuo*. The residue was dissolved in MeOH (3.3 mL) and potassium carbonate (5.5 mg, 0.040 mmol) was added to the solution. After being stirred at room temperature for 3.5 h, the reaction mixture was poured into saturated aq. NH_4Cl solution and extracted with CH_2Cl_2 . The organic extracts were dried over sodium sulfate and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 1/1 to 1/2) to give **17a** (146 mg, 83% yield) and **17b** (31 mg, 17% yield) as white solid. Mp. 120–121 °C; IR (ATR) ν 3356, 2984, 1713, 1598, 1512, 1380, 1325, 1265, 1159, 739, 667 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3 , 400MHz) δ 0.79 (3H, d, J = 6.4 Hz), 1.31 (3H, s), 1.39 (3H, s), 1.42 (9H, s), 1.52 (3H, s), 2.08 (1H, dd, J = 11.6, 3.2 Hz), 2.38 (3H, s), 3.58–3.62 (1H, ddd, J = 8.0, 10.0, 11.2 Hz), 4.01 (1H, ddd, J = 4.8, 9.6, 10.0 Hz), 4.16 (1H, d, J = 4.4 Hz), 4.88–4.96 (2H, m), 5.06 (1H, d, J = 10.0 Hz), 5.90 (1H, d, J = 7.2 Hz), 7.25 (2H, d, J = 8.4 Hz), 7.75 (1H, d, J = 8.8 Hz), 7.85 (2H, d, J = 8.4 Hz);

¹³C-NMR (CDCl₃, 100 MHz) δ 16.8, 21.5, 25.9, 26.3, 26.6, 28.3 (3C), 53.1, 56.5, 56.9, 69.4, 79.3, 82.9, 84.0, 110.0, 127.0 (2C), 129.4 (2C), 138.8, 143.0, 155.8, 156.3; HRMS (ESI-TOF); calcd for C₂₄H₃₇N₃NaO₈S⁺ [M+Na⁺]: 550.2194, found 550.2202; [α]_D²⁰ +4.59 (c 1.07, CHCl₃).

NMR and MS data of compound **17b**:

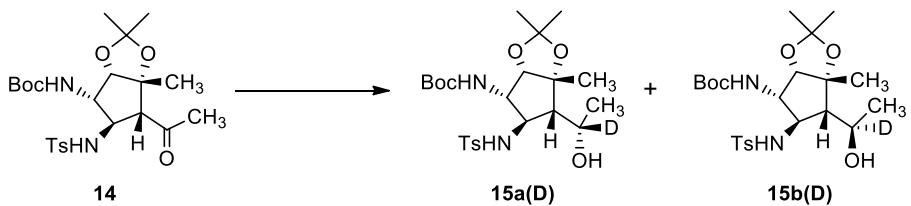
¹H-NMR (CDCl₃, 400MHz) δ 1.17 (3H, d, *J* = 6.4 Hz), 1.30–1.46 (9H, m), 1.40 (9H, s), 1.75–1.82 (1H, m), 2.40 (3H, s) 3.75–3.82 (2H, m), 4.08 (1H, d, *J* = 4.4 Hz), 4.88–4.99 (2H, m), 5.10 (1H, br-s), 5.96 (1H, d, *J* = 8.0 Hz), 7.25–7.32 (3H, m), 7.76 (2H, d, *J* = 8.4 Hz); ¹³C-NMR (CDCl₃) δ 18.5, 21.5, 25.9, 26.7, 27.9, 28.2 (3C), 53.2, 56.3, 58.5, 71.1, 79.9, 83.2, 84.5, 110.4, 126.6 (2C), 129.5 (2C), 139.2, 142.8, 156.1, 156.6; HRMS (ESI-TOF); calcd for C₂₄H₃₇N₃NaO₈S⁺ [M+Na⁺]: 550.2194, found 550.2199.



tert-butyl

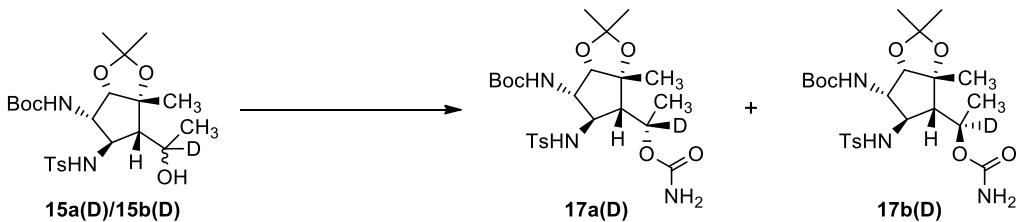
{(3a*R*,4*R*,5*S*,5'*S*,6*S*,6a*S*)-2,2,3a,5'-tetramethyl-5-(4-methylphenylsulfonamido)-2'-oxotetrahydrospiro[cyclopenta[d][1,3]dioxole-4,4'-oxazolidin]-6-yl}carbamate (**18a**)

To a stirred solution of **17a** (17.1 mg, 0.0324 mmol) in benzene (0.65 mL) at room temperature were added magnesium oxide (3.0 mg, 0.0745 mmol), PhI(OCO*t*Bu)₂ (18.4 mg, 0.0453 mmol) and Rh₂(OAc)₄ (2.1 mg, 4.86 μmol), and the mixture was heated to 40 °C. After being stirred for 6 h, the reaction mixture was concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 3/1 to 1/2) to give **18a** (11.9 mg, 70% yield) as white solid and **14** was recovered (4.8 mg, 30% yield). Mp. 160–161 °C; IR (ATR) ν 3289, 2927, 1752, 1513, 1456, 1385, 1333, 1262, 1162, 1095 cm⁻¹; ¹H-NMR (CDCl₃, 400MHz) δ 1.18 (3H, d, *J* = 6.8 Hz), 1.43 (9H, s), 1.36–1.45 (9H, m), 2.38 (3H, s), 3.95–3.99 (1H, m), 4.03–4.09 (1H, m), 4.24 (1H, d, *J* = 4.0 Hz), 4.60–4.66 (1H, m), 5.16 (1H, d, *J* = 9.2 Hz), 6.04 (1H, d, *J* = 8.4 Hz), 7.00 (1H, br-s), 7.25 (2H, d, *J* = 8.4 Hz), 7.74 (2H, d, *J* = 8.4 Hz); ¹³C-NMR (CDCl₃, 100 MHz) δ 15.2, 21.5, 21.5, 25.9, 26.8, 28.4 (3C), 54.9, 57.5, 69.1, 77.2, 80.3, 80.9, 87.5, 111.1, 126.8 (2C), 129.6 (2C), 138.9, 143.1, 156.2, 159.1; HRMS (ESI-TOF); calcd for C₂₄H₃₅N₃NaO₈S⁺ [M+Na⁺]: 548.2037, found 548.2015; [α]_D²⁰ −0.94 (c 0.54, CHCl₃).



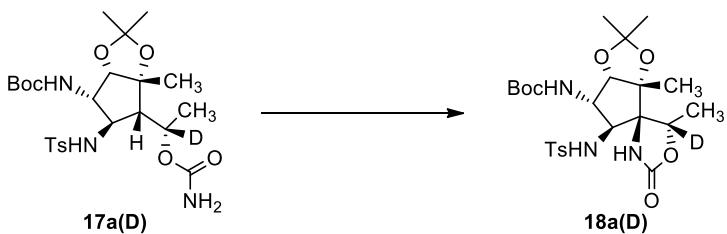
Compound 15a(D) and 15b(D)

To a stirred solution of **14** (34.1 mg, 0.070 mmol) in MeOH (1.4 mL) was added NaBD₄ (29 mg, 0.70 mmol) at 0 °C. After being stirred for 4 h at room temperature, the reaction mixture was quenched with H₂O. The resulting mixture was extracted with EtOAc, dried over sodium sulfate, and concentrated *in vacuo*. The obtained mixture of **15a(D)** and **15b(D)** were used for the next step without further purification.



Compound 17a(D):

To a stirred solution of the crude mixture of **15a(D)** and **15b(D)** (34 mg, 0.070 mmol) in CH₂Cl₂ (1.4 mL) at 0 °C was added trichloroacetyl isocyanate (10 µL, 0.084 mmol), and the resulting mixture was allowed to warm to room temperature. After being stirred for 30 min, the reaction mixture was concentrated *in vacuo*. The residue was dissolved in MeOH (1.4 mL) and potassium carbonate (1.1 mg, 8.4 µmol) was added to the reaction. The resulting mixture was stirred at room temperature. After 3.5 h, the reaction mixture was poured into saturated aq. NH₄Cl solution and extracted with CH₂Cl₂. The organic extracts were dried over sodium sulfate and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 1/1 to 1/2) to give **17a(D)** (17.6 mg, 48% yield) and **17b(D)** (17.5 mg, 47%) as white solid. Mp. 105–106 °C; IR (ATR) ν 3853, 3735, 2930, 1716, 1507, 1375, 1266, 1160, 743, 671 cm⁻¹; ¹H-NMR (CDCl₃, 400MHz) δ 0.92 (3H, s), 1.20–1.51 (18H, m), 1.92 (1H, d, *J* = 11.4 Hz), 2.39 (3H, s) 3.63–3.69 (1H, m), 3.90–3.95 (1H, m), 4.14 (1H, d, *J* = 4.2 Hz), 4.80–4.90 (1H, broad peak), 5.02 (1H, d, *J* = 9.6 Hz), 5.47 (1H, d, *J* = 8.4 Hz), 7.26–7.29 (2H, m), 7.73–7.80 (1H, m), 7.82 (2H, d, *J* = 7.8 Hz); ¹³C-NMR (CDCl₃) δ 17.0, 21.5, 26.0, 26.6, 26.7, 28.3 (3C), 53.8, 56.7, 57.0, 69.3 (t), 79.5, 82.8, 84.1, 110.2, 127.0 (2C), 129.5 (2C), 138.9, 143.1, 155.9, 156.1; HRMS (ESI-TOF); calcd for C₂₄H₃₆DN₃NaO₈S⁺ [M+Na⁺]: 551.2256, found 551.2249; [α]_D²¹ -5.61 (c 0.50, CHCl₃).

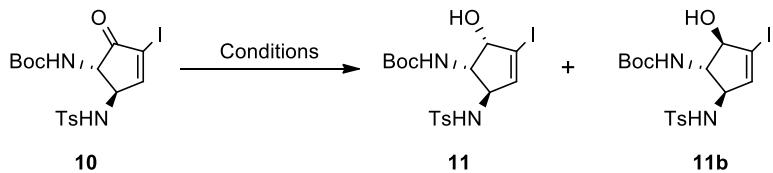


Compound 18a(D)

To a stirred solution of **17a(D)** (11.2 mg, 0.0211 mmol) in benzene (0.42 mL) at room temperature were added magnesium oxide (2.0 mg, 0.0487 mmol), $\text{PhI}(\text{OCO}t\text{Bu})_2$ (12 mg, 0.0296 mmol) and $\text{Rh}_2(\text{OAc})_4$ (1.4 mg, 3.177 μmol), and the mixture was heated to 40 $^{\circ}\text{C}$. After being stirred for 6 h, the reaction mixture was concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAt = 3/1 to 1/2) to give **18a(D)** (8.9 mg, 80% yield) as white solid and **14** was recovered (1.6 mg, 15% yield). Mp. 164–165 $^{\circ}\text{C}$; IR (ATR) ν 3275, 2931, 1751, 1509, 1456, 1368, 1333, 1161, 1094, 918 cm^{-1} ; $^1\text{H-NMR}$ (CDCl_3 , 400MHz) δ 1.17 (3H, s), 1.43 (9H, s), 1.35–1.46 (9H, m), 2.38 (3H, s), 3.97–4.10 (2H, m), 4.22 (1H, d, J = 4.0 Hz), 5.17 (1H, d, J = 9.2 Hz), 6.07 (1H, d, J = 9.2 Hz), 6.91 (1H, br-s), 7.23–7.28 (2H, m), 7.74 (2H, d, J = 8.4 Hz); $^{13}\text{C-NMR}$ (CDCl_3) δ 15.1, 21.5, 21.5, 25.9, 26.8, 28.3 (3C), 54.8, 57.4, 68.9, 77.0 (t), 80.4, 80.9, 87.4, 111.2, 126.7 (2C), 129.5 (2C), 138.9, 143.0, 156.2, 159.0; HRMS (ESI-TOF); calcd for $\text{C}_{24}\text{H}_{34}\text{DN}_3\text{NaO}_8\text{S}^+ [\text{M}+\text{Na}^+]$: 549.2100, found 549.2102; $[\alpha]_D^{21} +0.17$ (c 0.34, CHCl_3).

3. Optimization of the Reaction Conditions

(1) Diastereoselective reduction of the ketone in **10**



Entry	Conditions	dr ^a
1	NaBH_4 , $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}$, MeOH , 0 $^{\circ}\text{C}$	11:11b = 1:1
2	NaBH_4 , $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}$, <i>i</i> -PrOH, -78 to 0 $^{\circ}\text{C}$	11:11b = 1:1
3	DIBAH, CH_2Cl_2 , -78 $^{\circ}\text{C}$	11:11b = 1:1
4	DIBAH, toluene, -78 $^{\circ}\text{C}$	11:11b = 1:1
5	$\text{Al}(\text{O}-i\text{-Pr})_3$, <i>i</i> -PrOH, reflux, then 1N HCl	11:11b = 8:1

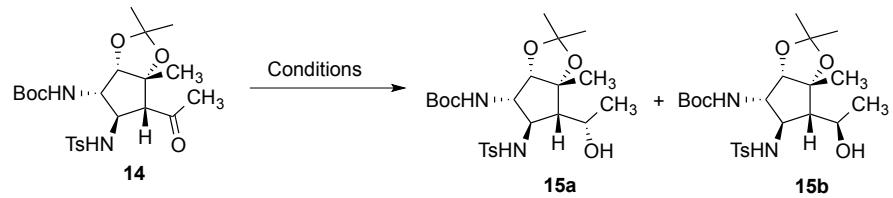
^aDiastereomeric ratio was determined by ^1H NMR analysis of the crude mixture.

(2) Regio- and stereoselective 1,3-dipolar cycloaddition of **12**

Entry	X	Base	Solvent	Time	Results
1 ^a	3	EtMgBr	CH ₂ Cl ₂	18 h	13 was not obtained.
2	3	Et ₃ Al	CH ₂ Cl ₂	18 h	13 was not obtained.
3	5	NEt ₃	CH ₂ Cl ₂	48 h	50% yield
4	5	i-Pr ₂ NEt	CH ₂ Cl ₂	48 h	38% yield
5	5	NEt ₃	(CH ₂ Cl) ₂	48 h	31% yield
6	5	NEt ₃	THF	48 h	25% yield
7	5	NEt ₃	benzene	48 h	<10% yield
8 ^b	15	NEt ₃	CH ₂ Cl ₂	48 h	66% yield (80% brsm)

^a Ref. Carreira et al. *Org. Lett.* **2007**, 9, 3857. ^b 16 equiv of acetaldoxime was used.

(3) Diastereoselective reduction of the methyl ketone in **14**



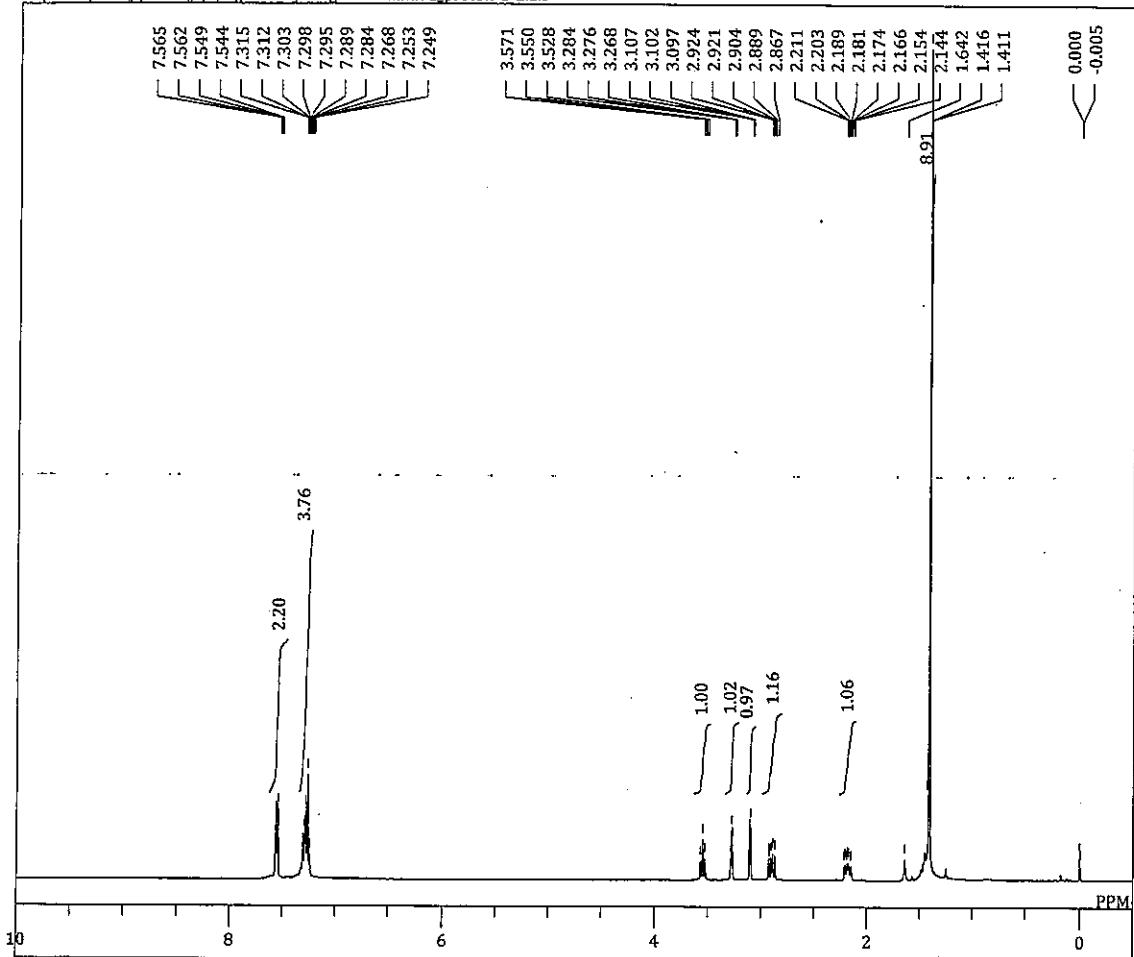
Entry	Conditions	Yield (15a + 15b)	dr ^a
1	DIBAH, CH ₂ Cl ₂ , -78 °C	80%	15a:15b = 1:2
2	DIBAH, THF, -78 °C	91%	15a:15b = 5:1
3	NaBH4, MeOH, rt	65% (14: 10%)	15a:15b = 1:1
4	L-selectride, THF, -78 °C	no reaction	—

^a Diastereomeric ratio was determined by ¹H NMR analysis of the isolated mixture.

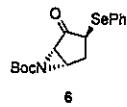
4. ¹H and ¹³C NMR Charts of New Compounds (S16–S33)

01

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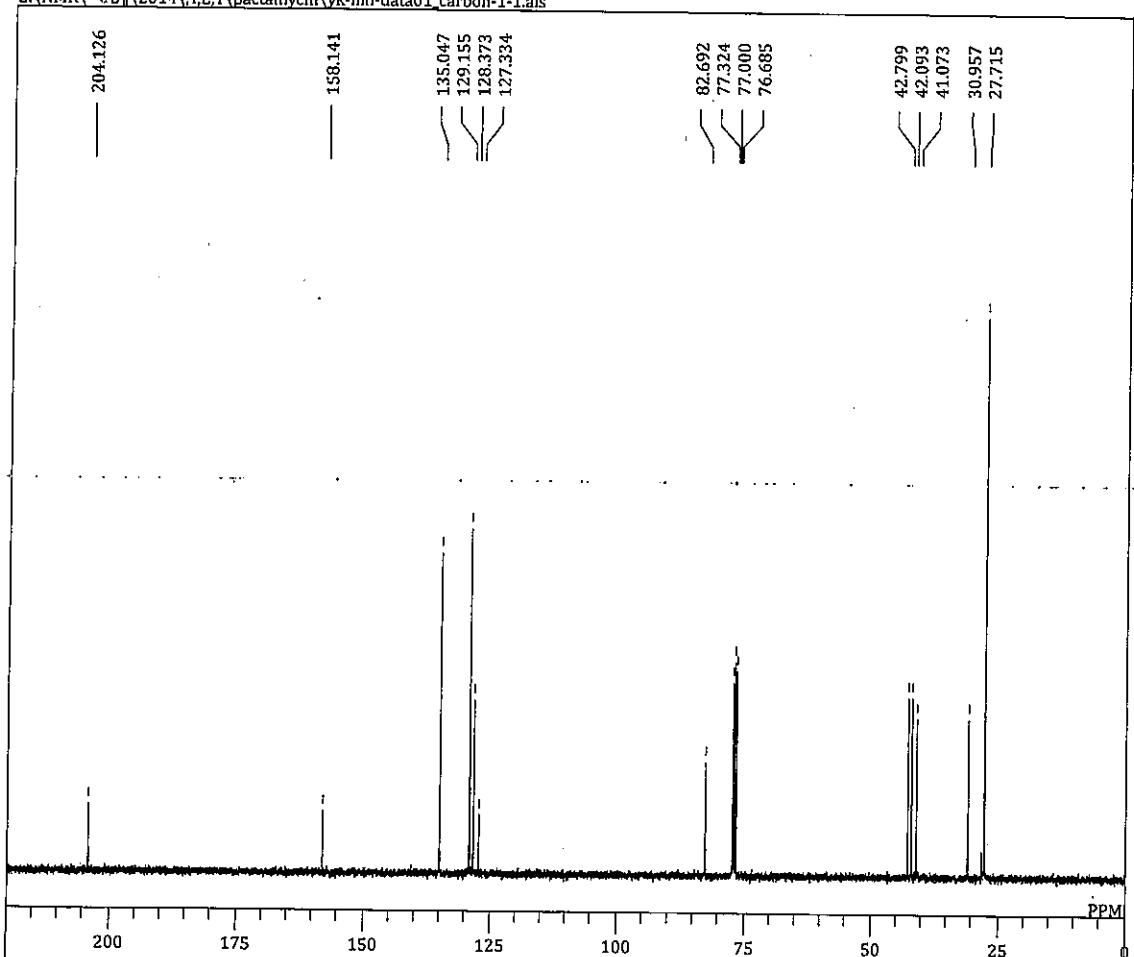


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 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 36

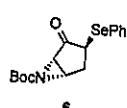


01

E:\NMR\12\ET\2014\YE\pactamycin\yk-mh-data01_carbon-1-1.als



DFILE yk-mh-data01_carbon-1-1.als
 COMNT 01
 DATIM 2013-09-13 16:54:16
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 73
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.02 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60



S16

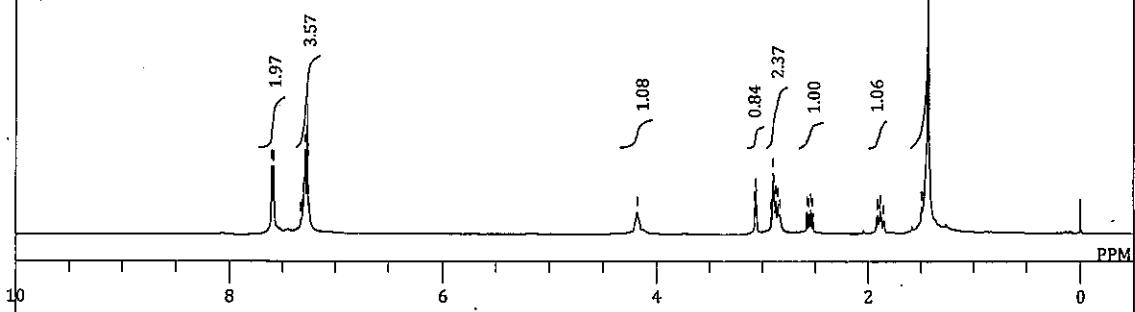
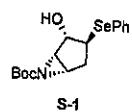
02

E:\NMR\^2\ET\2014\Y\B\pactamycin\yk-mh-data02_proton-1-1.als

7.603
7.587
7.583
7.329
7.326
7.309
7.293
7.287
7.268
7.253

4.177
3.059
3.070
3.067
3.055
3.052
2.914
2.895
2.868
2.849
2.831
2.575
2.557
2.540
2.521
1.915
1.910
1.890
1.881
1.855
1.851
1.497
1.487
1.483
1.451
0.000
-0.003

DFILE yk-mh-data02_proton-1-1.als
COMNT 02
DATIM 2013-09-13 22:13:23
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 kHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 12
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.01 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32



02

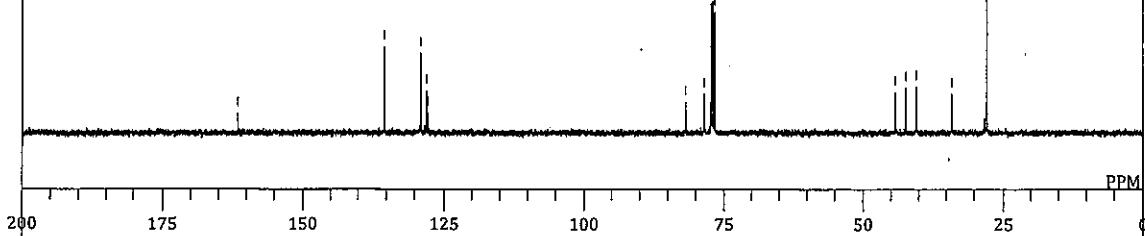
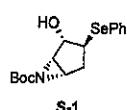
E:\NMR\^2\ET\2014\Y\B\pactamycin\yk-mh-data02_carbon-1-1.als

161.5524
135.4448
129.0351
128.0658
127.734

81.901
78.516
77.315
77.000
76.676

44.220
42.351
40.492
34.160
27.848

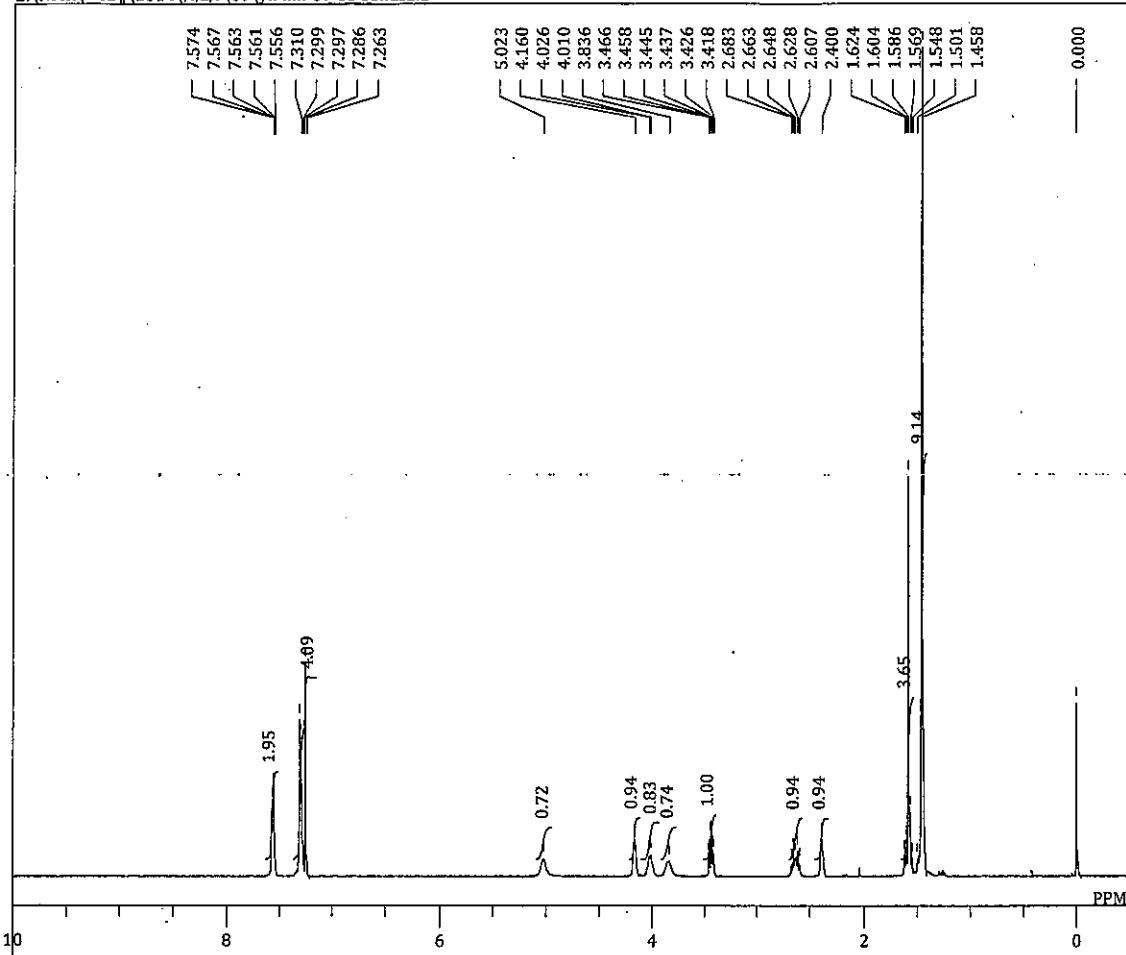
DFILE yk-mh-data02_carbon-1-1.als
COMNT 02
DATIM 2013-09-13 22:16:51
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 kHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 75
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.02 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 50



S17

yk-mh-09-55-solids2

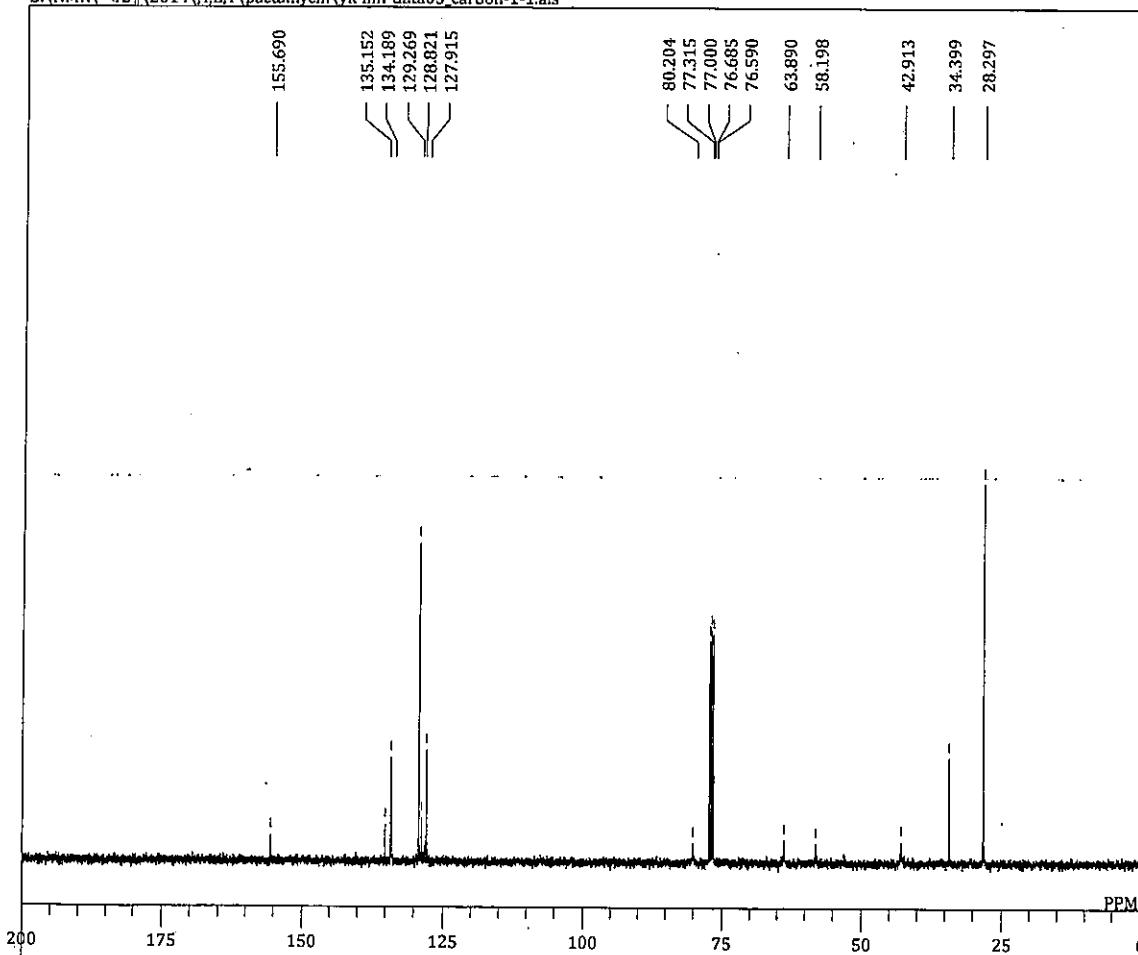
E:\NMR\12\ET\2014\Y\09\yk-mh-09-55-solids2.1



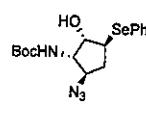
DFILE yk-mh-09-55-solids2.1
 COMNT yk-mh-09-55-solids2
 DATIM 2014-06-17 22:02:06
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 399.78 MHz
 OBSET 4.19 kHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 5998.80 Hz
 SCANS 8
 ACQTM 2.7312 sec
 PD 2.0000 sec
 PW1 5.75 usec
 IRNUC 1H
 CTEMP 21.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 17

data03

E:\NMR\12\ET\2014\Y\09\pactamycin\yk-mh-data03_carbon-1-1.als

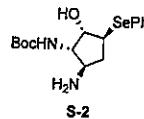
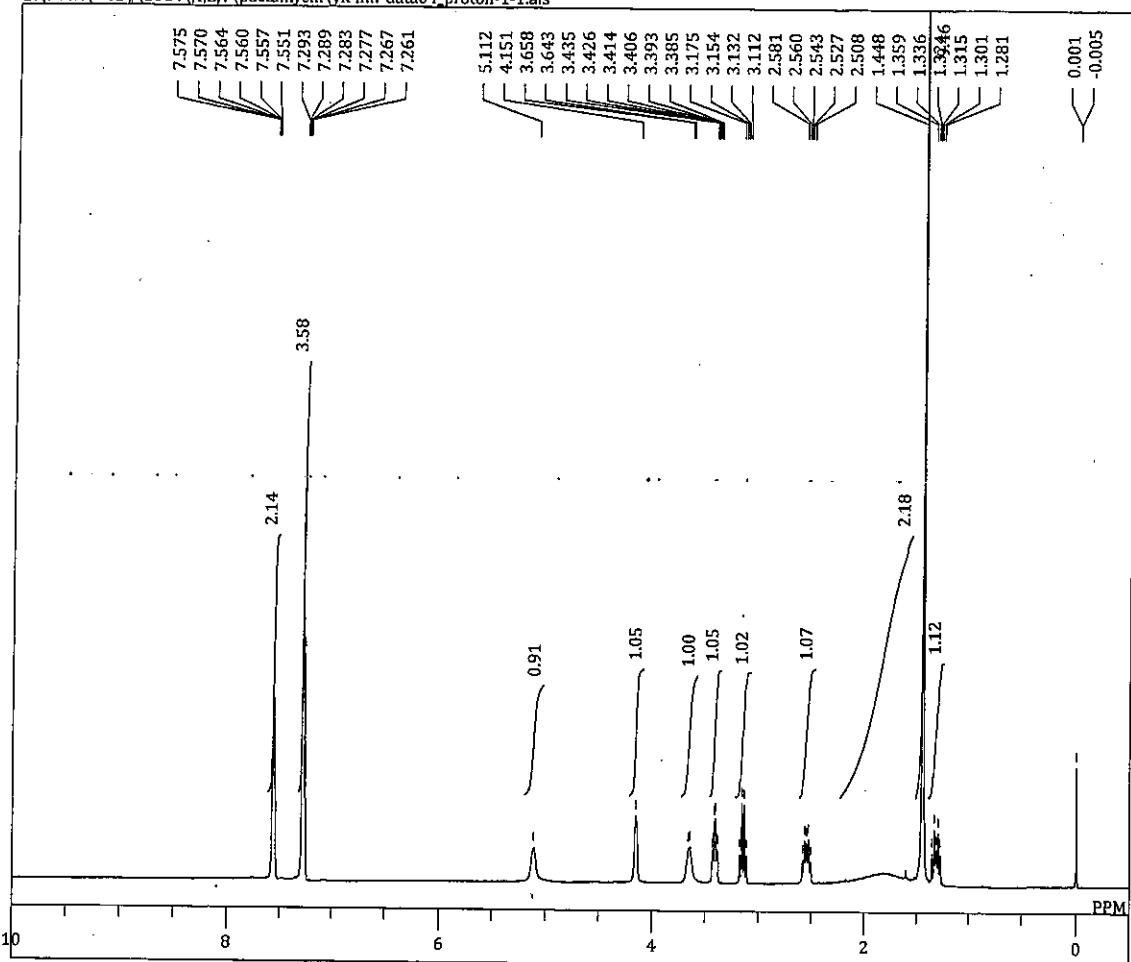


DFILE yk-mh-data03_carbon-1-1.als
 COMNT data03
 DATIM 2013-09-20 16:40:31
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 62
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.02 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60



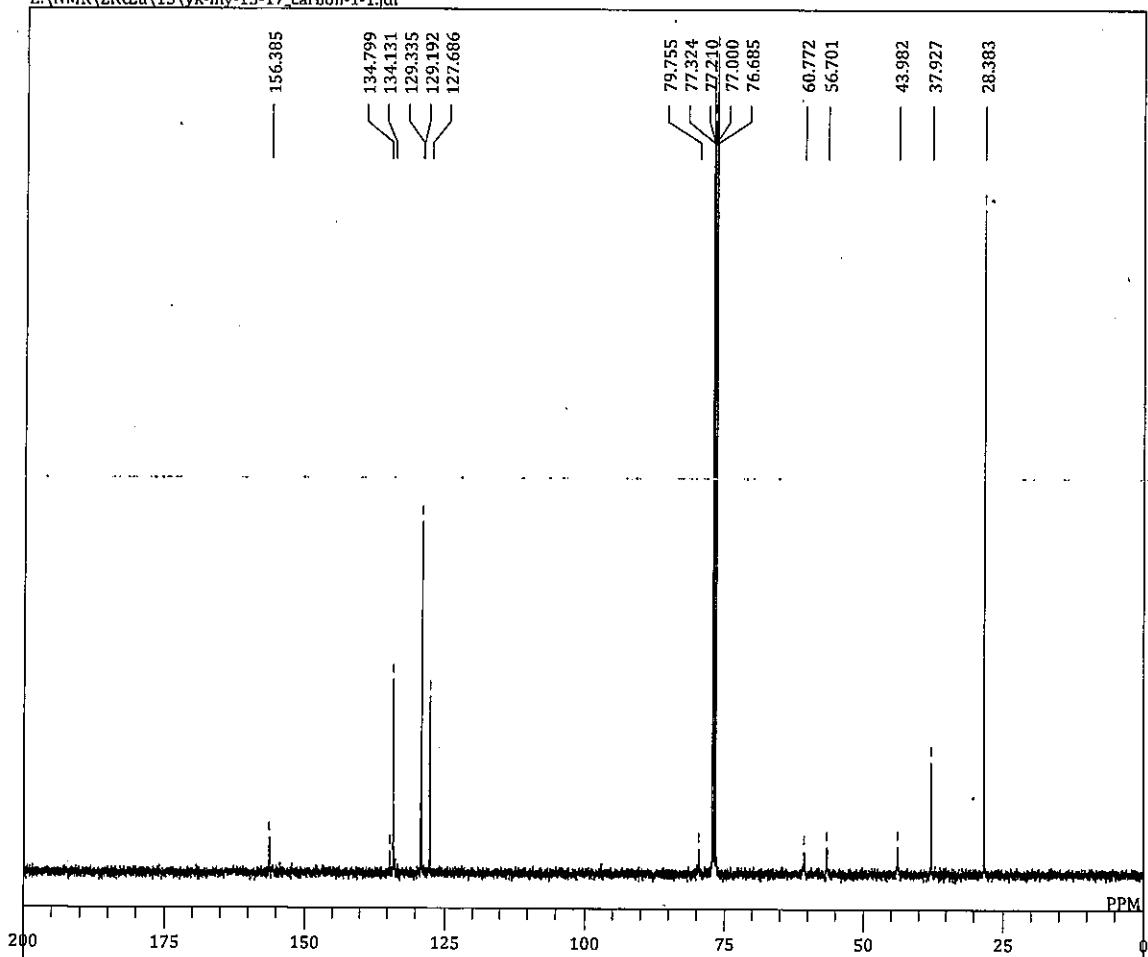
data04

E:\NMR\12\2014\Y.E.Y\pactamycin\yk-mh-data04_proton-1-1.als

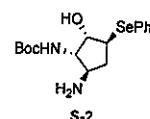


yk-my-13-17_yk-my-13-17_proton

E:\NMR\12\2014\yk-my-13-17_carbon-1-1.jdf

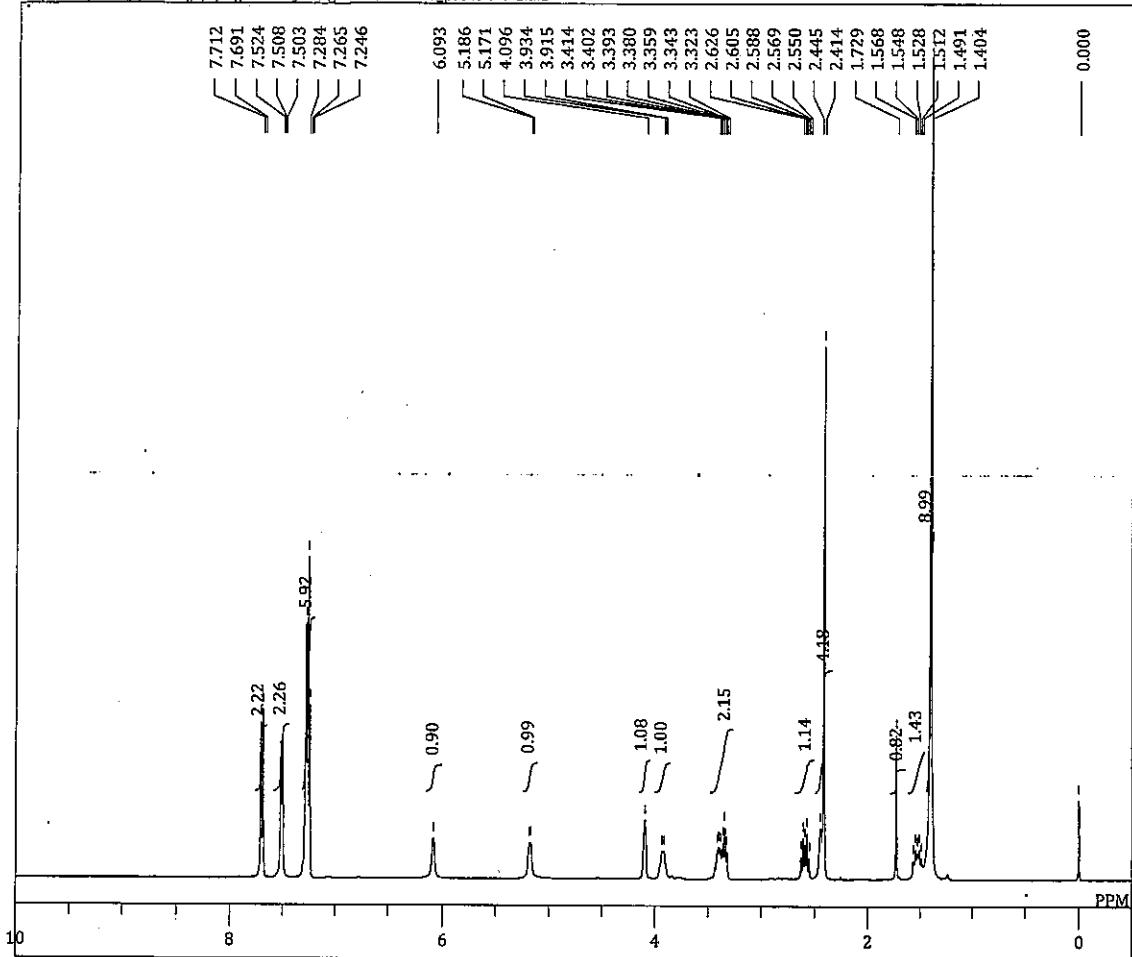


DFILE yk-my-13-17_carbon-1-1.jdf
 COMNT yk-my-13-17_yk-my-13-17_prot
 DATIM 2016-01-06 17:41:02
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 398
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 2.77 usec
 IRNUC 1H
 CTEMP 23.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60



data05

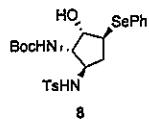
E:\NMR\12\AE\2014\Y\20140529\pactamycin\yk-mh-data05_proton-2-1.als



```

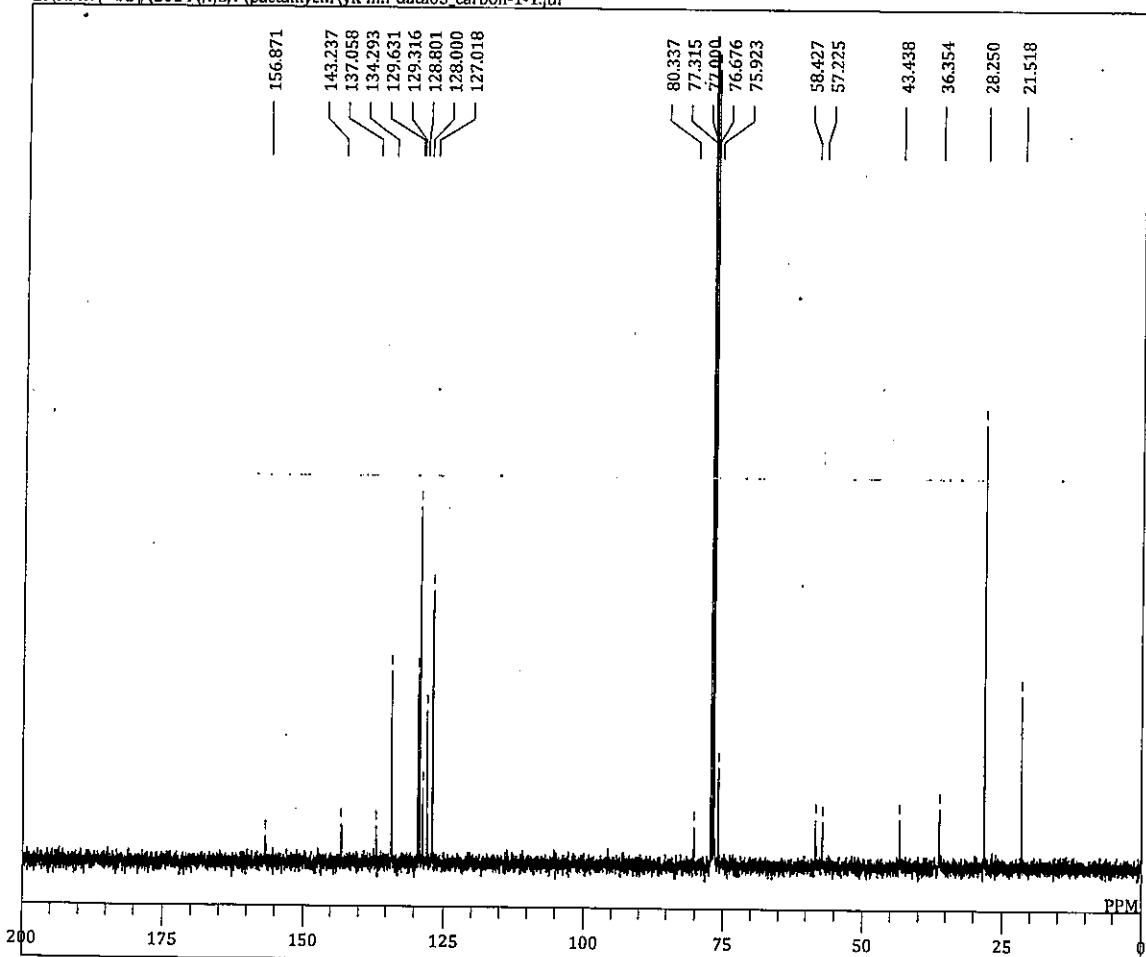
DFILE  yk-mh-data05_proton-2-1.als
COMNT  data05
DATIM  2013-09-21 21:45:30
OBNUC  1H
EXMOD  proton.jxp
OBFRQ  399.78 MHz
OBSET  4.19 kHz
OBFIN  7.29 Hz
POINT  13107
FREQU  6002.40 Hz
SCANS  16
ACQTM  2.1837 sec
PD      5.0000 sec
PW1    5.01 usec
IRNUC  1H
CTEMP  20.3 c
SLVNT  CDCL3
EXREF  0.00 ppm
BF     0.12 Hz
RGAIN  36

```



data05

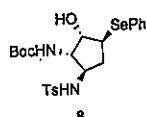
E:\NMR\12\ET\2014\Y\ET\pactamycin\vk-mh-data05_carbon-1-1.pdf



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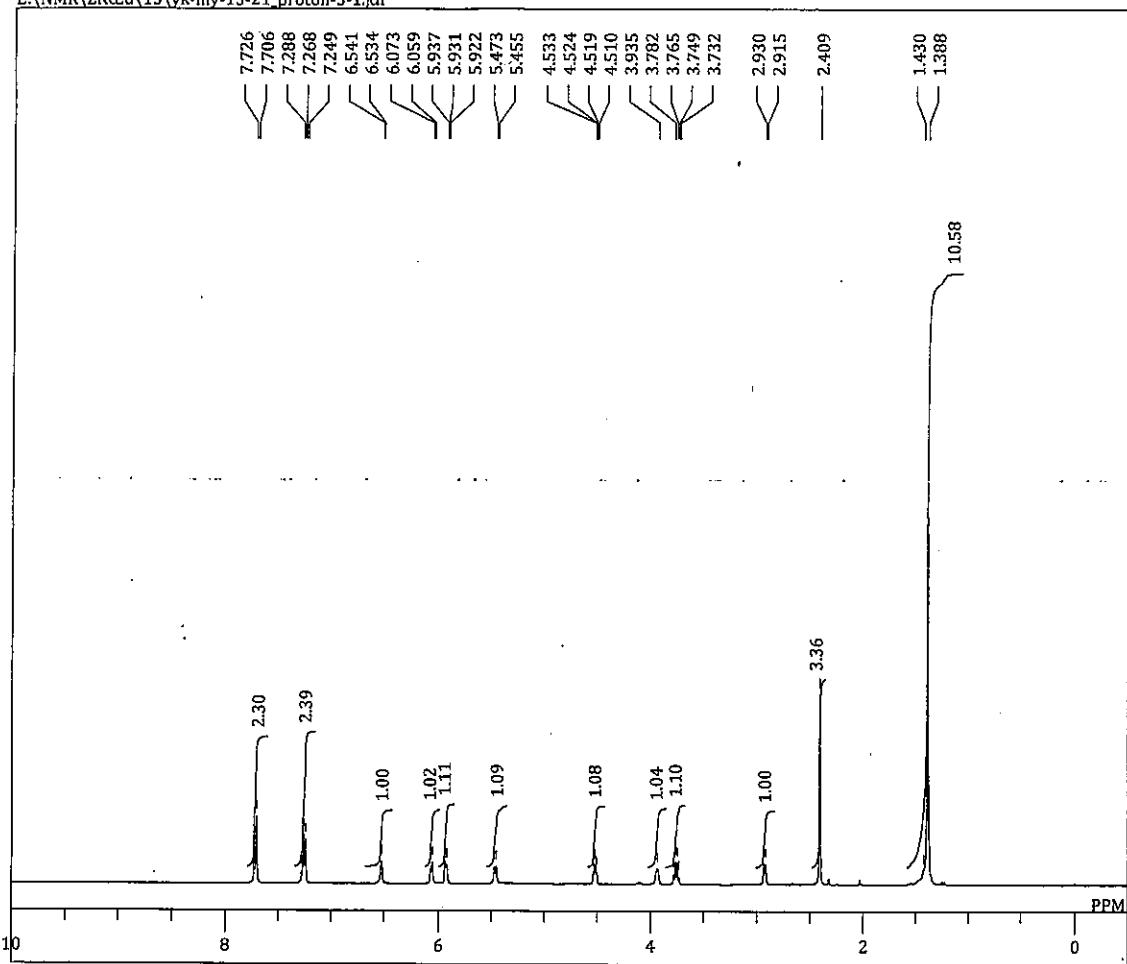
FILE      yk-mh-data05_carbon-1-1.jdf
COMNT    data05
DATIM    2013-09-21 21:50:02
OBNUC    13C
EXMOD   carbon.jxp
OBFRQ   100.53 MHz
OBSET    5.35 KHz
OBFIN   5.86 Hz
POINT    32767
FREQU   31407.04 Hz
SCANS    169
ACQTM    1.0433 sec
PD       2.0000 sec
PW1      3.02 usec
IRNUC   1H
CTEMP   20.6 c
SLVNT   CDCL3
EXREF   77.00 ppm
BF       1.20 Hz
PGAIN   60

```



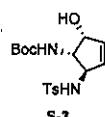
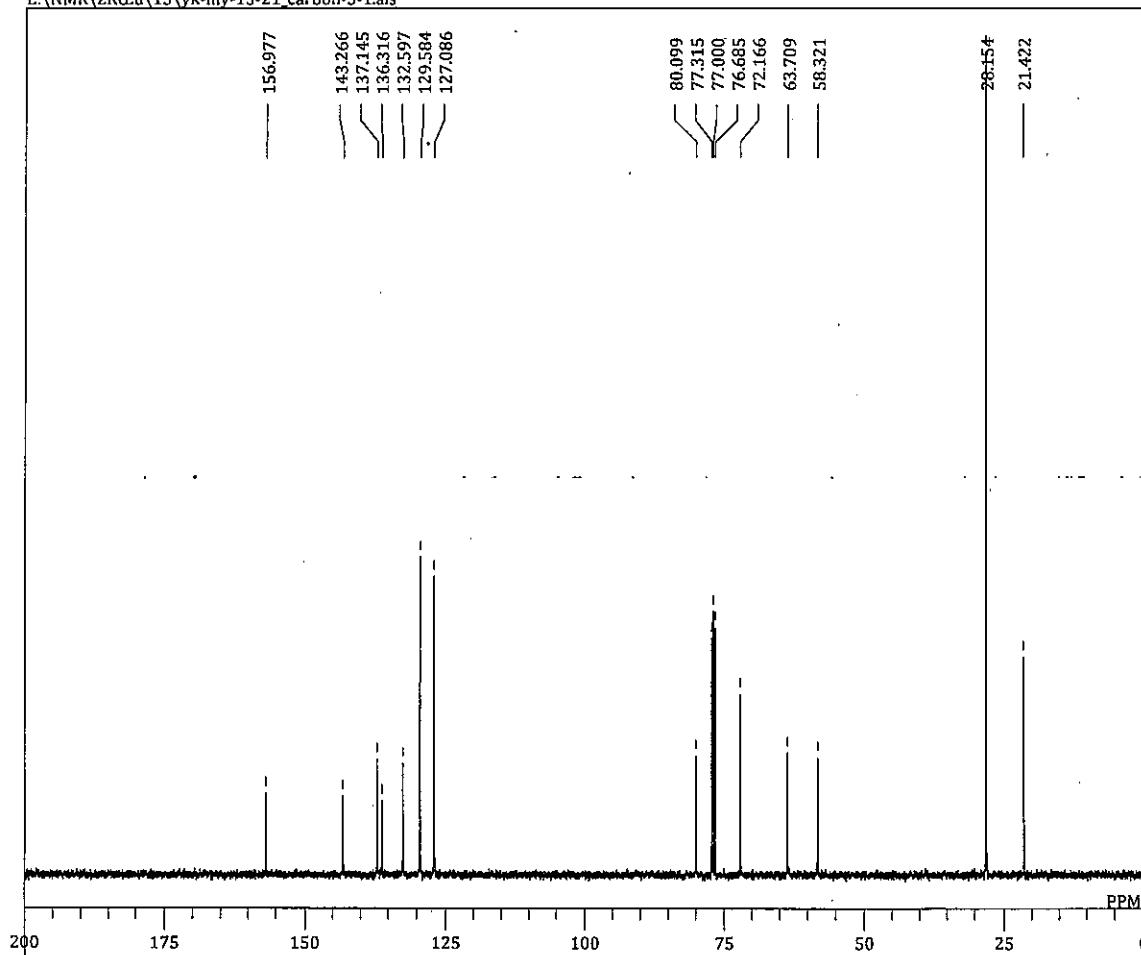
yk-my-13-21

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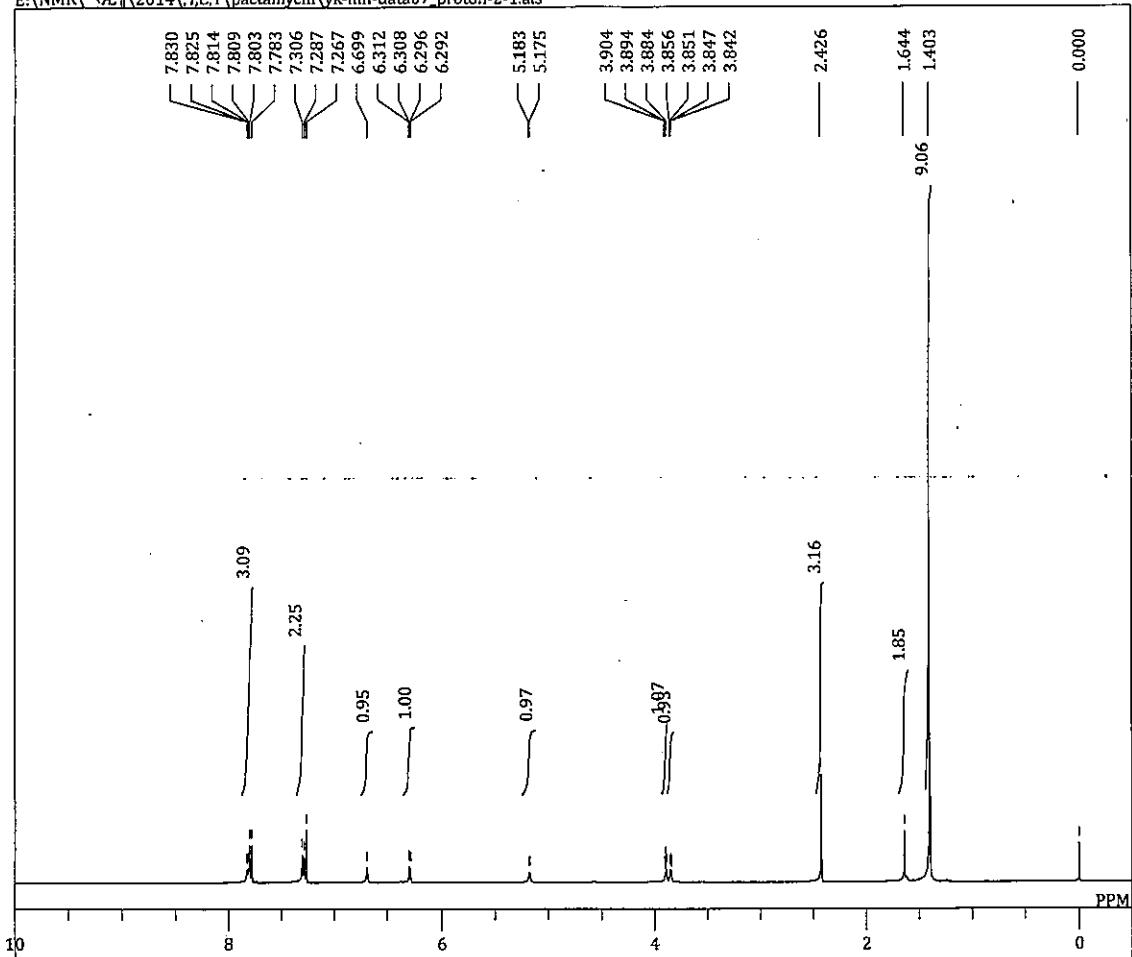
yk-my-13-21_yk-my-13-21_proton

E:\NMR\ŽRCG\13\yk-my-13-21_carbon-3-1.als

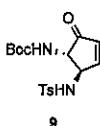


data07

E:\NMR\12\ET\2014\Y\pactamycin\yk-mh-data07_proton-2-1.als

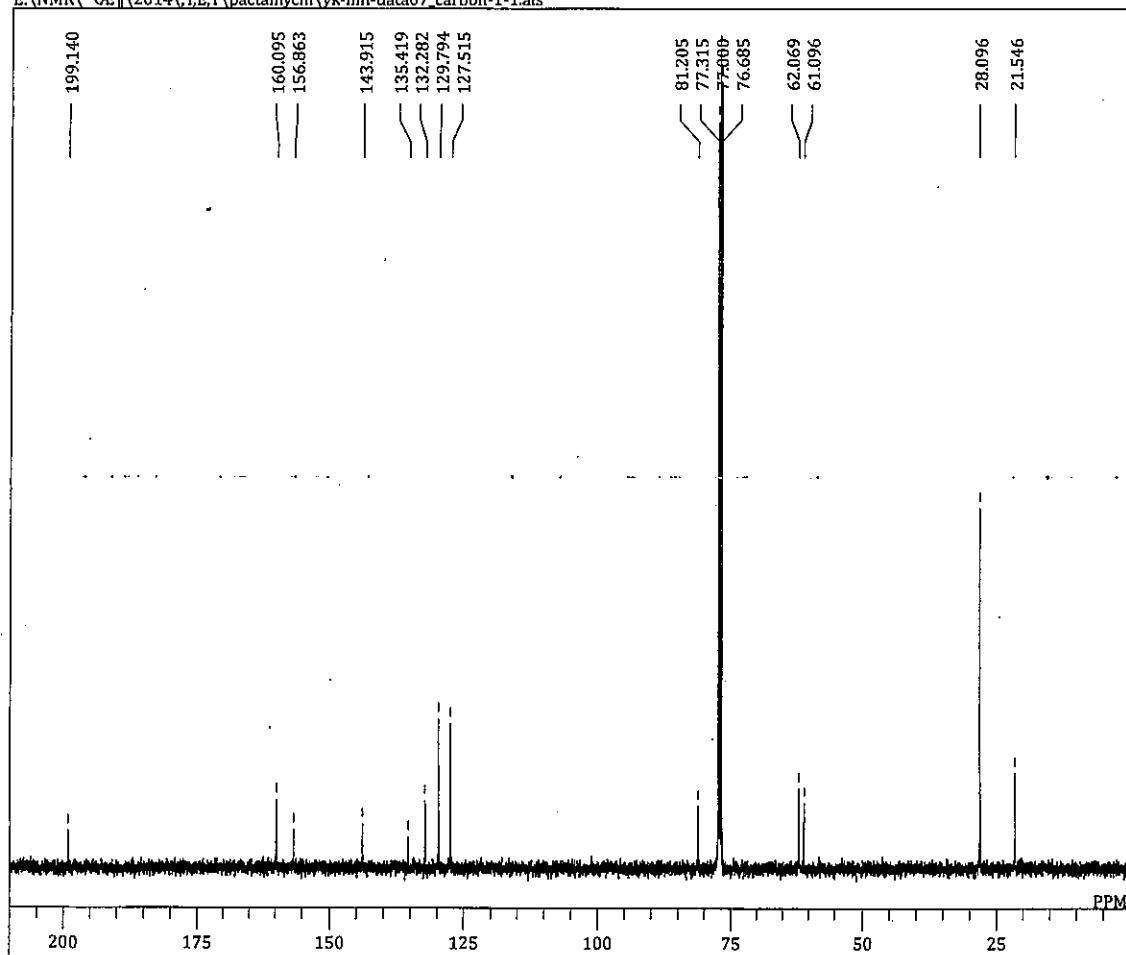


DFILE yk-mh-data07_proton-2-1.als
 COMNT data07
 DATIM 2013-10-01 18:31:58
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 399.78 MHz
 OBSET 4.19 kHz
 OBFIN 7.29 Hz
 POINT 13107
 FREQU 6002.40 Hz
 SCANS 16
 ACQTM 2.1837 sec
 PD 5.0000 sec
 PW1 5.01 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 42

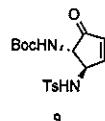


data07

E:\NMR\12\ET\2014\Y\pactamycin\yk-mh-data07_carbon-1-1.als



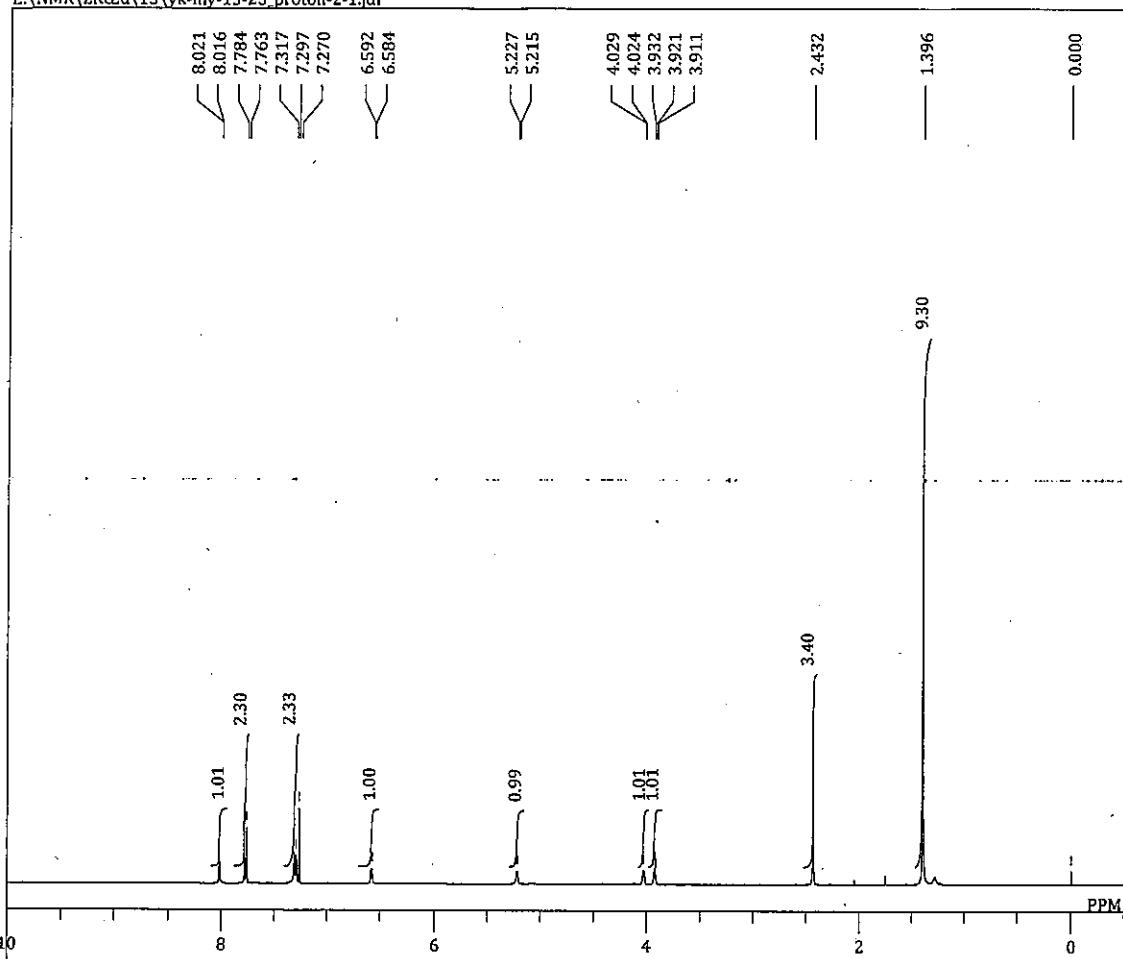
DFILE yk-mh-data07_carbon-1-1.als
 COMNT data07
 DATIM 2013-10-01 18:36:00
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 412
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.02 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60



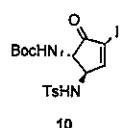
S22

yk-my-13-23

E:\NMR\ZRCE0\13\yk-my-13-23_proton-2-1.jdf

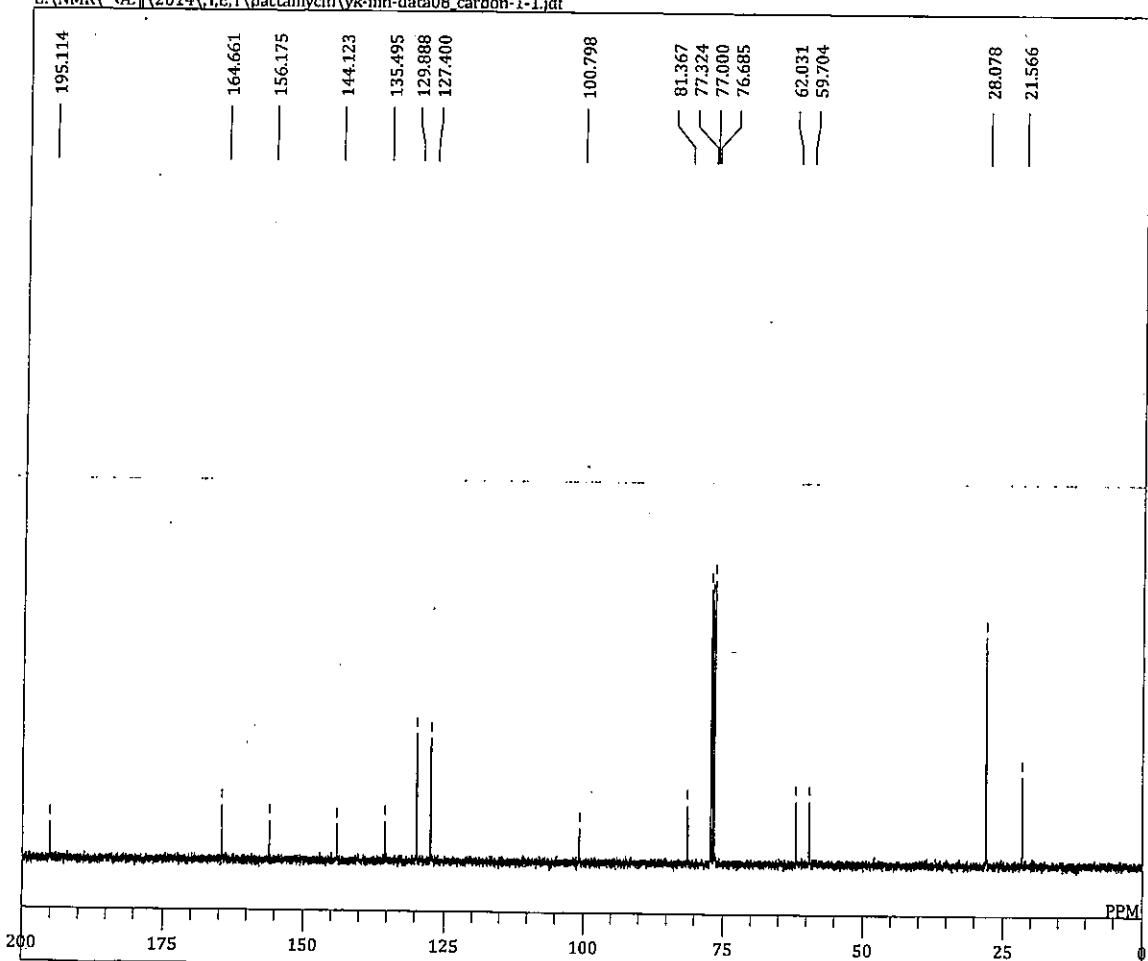


YK-MY-13-23_proton-2-1.jdf
 yk-my-13-23
 2016-01-14 15:10:45
 1H
 proton.jxp
 399.78 MHz
 4.19 kHz
 7.29 Hz
 16384
 7503.00 Hz
 8
 2.1837 sec
 5.0000 sec
 5.01 usec
 1H
 21.7 c
 CDCL3
 0.00 ppm
 0.12 Hz
 36

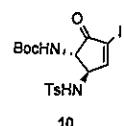


data08

E:\NMR\ZRCE0\2014\YK\pactamycin\yk-mh-data08_carbon-1-1.jdf



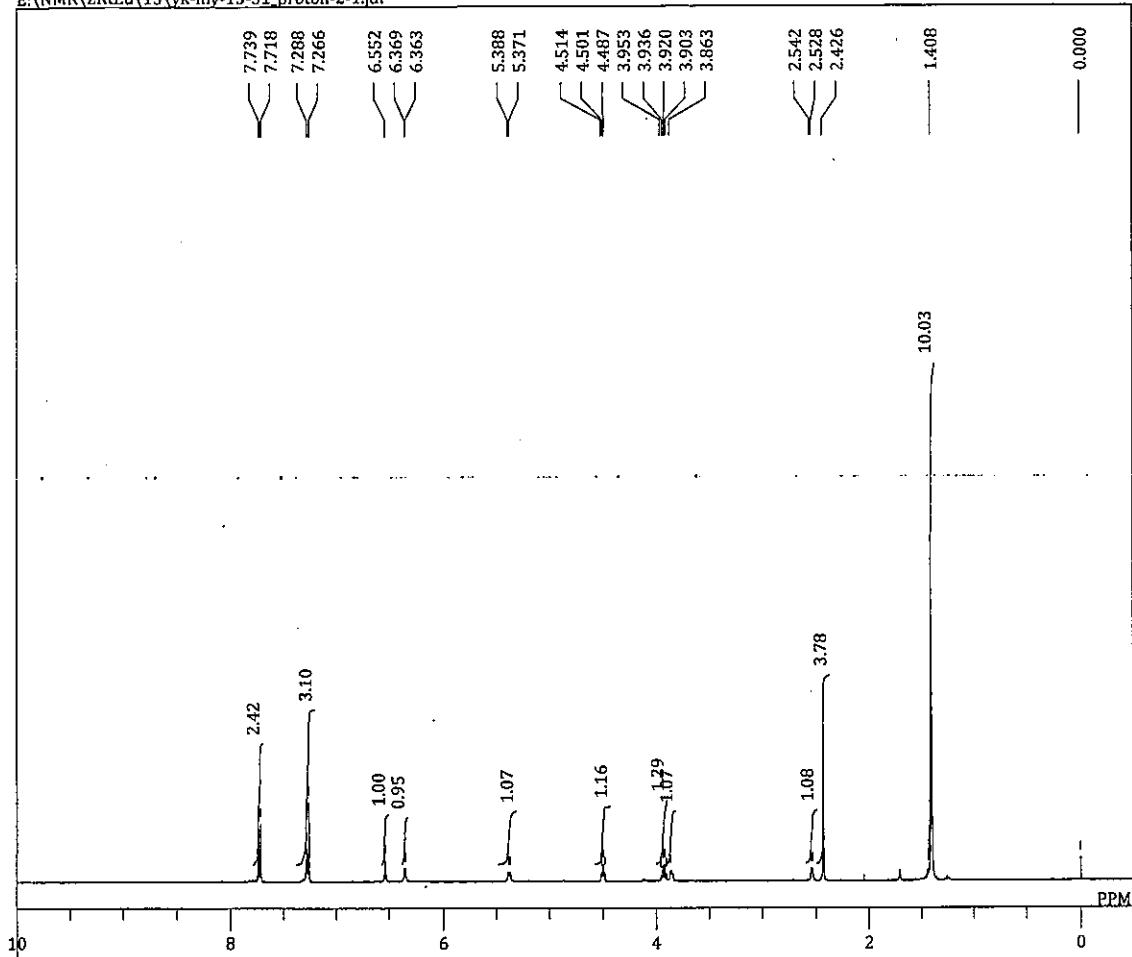
YK-MH-DATA08_CARBON-1-1.jdf
 data08
 2013-10-05 17:00:55
 13C
 carbon.jxp
 100.53 MHz
 5.35 kHz
 5.86 Hz
 32767
 31407.04 Hz
 71
 1.0433 sec
 2.0000 sec
 3.02 usec
 1H
 20.0 c
 CDCL3
 77.00 ppm
 1.20 Hz
 60



S 23

yk-my-13-31-

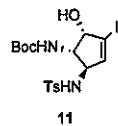
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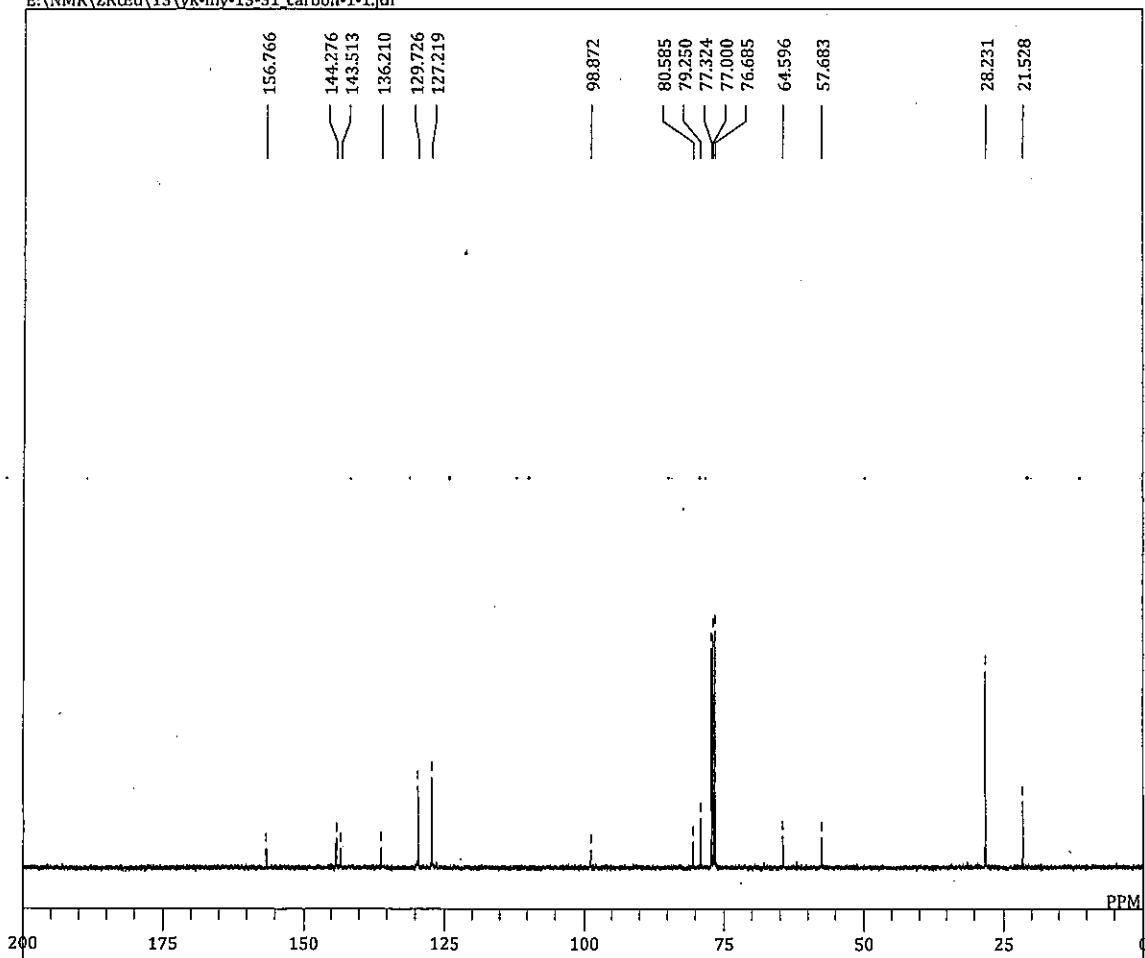
yk-my-13-31_proton-2-1.jdf
yk-my-13-31_
2016-01-20 13:17:36
1H
proton.jxp
399.78 MHz
4.19 KHz
7.29 Hz
16384
7503.00 Hz
8
2.1837 sec
5.0000 sec
5.01 usec
1H
21.2 c
CDCL3
0.00 ppm
0.12 Hz
40

```



yk-my-13-31-

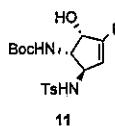
E:\NMR\ŽROEG\13\yk-my-13-31_carbon-1-1.jdf



```

DFILE  yk-my-13-31_carbon-1-1.jdf
COMNT  yk-my-13-31_
DATIM  2016-01-19 21:08:37
OBNUC  13C
EXMOD carbon.jxp
OBFRQ  100.53 MHz
OBSET  5.35 KHz
OBFIN  5.86 Hz
POINT  32767
FREQU  31407.04 Hz
SCANS  180
ACQTM  1.0433 sec
PD      2.0000 sec
PW1    2.77 usec
IRNUC  1H
CTEMP  21.4 c
SLVNT  CDCL3
EXREF  77.00 ppm
BF      1.20 Hz
RGAIN  60

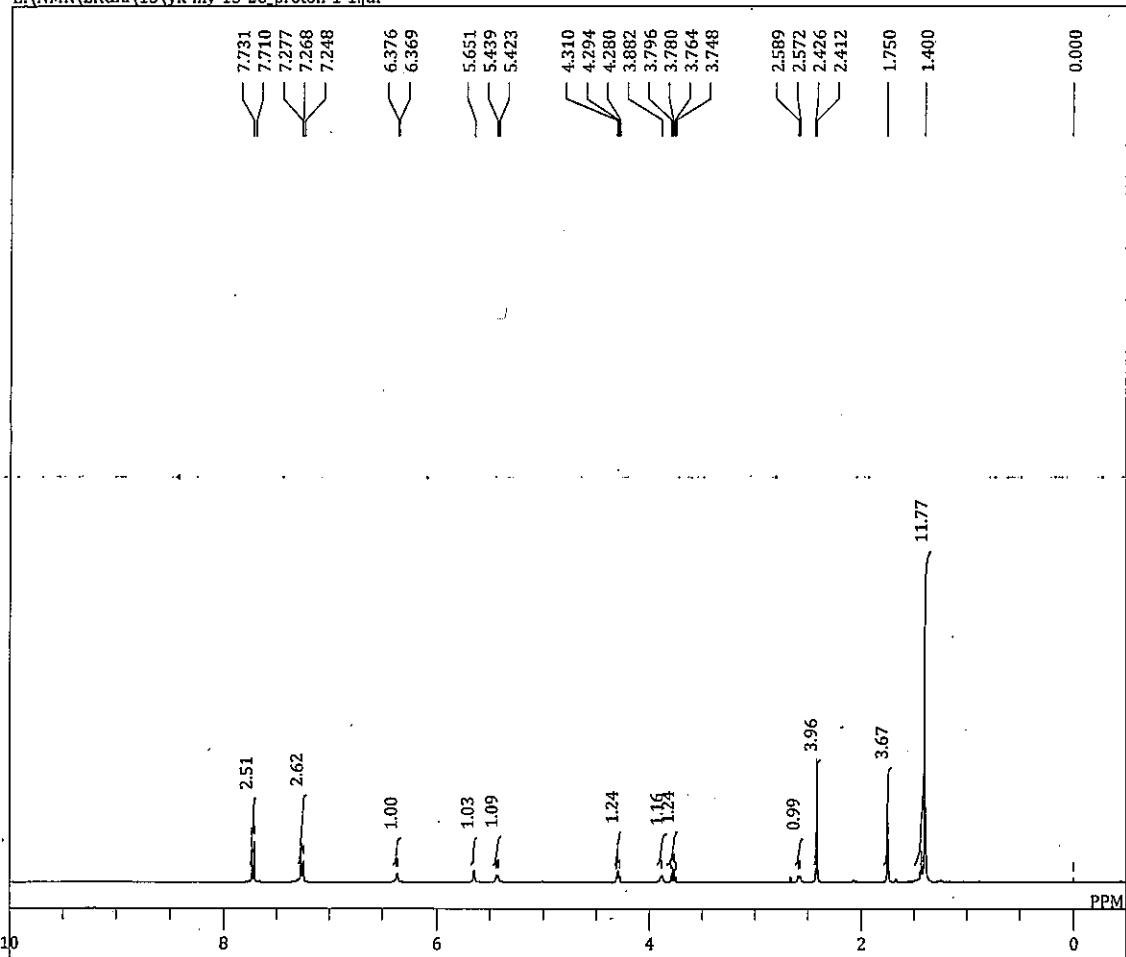
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524

yk-my-13-26

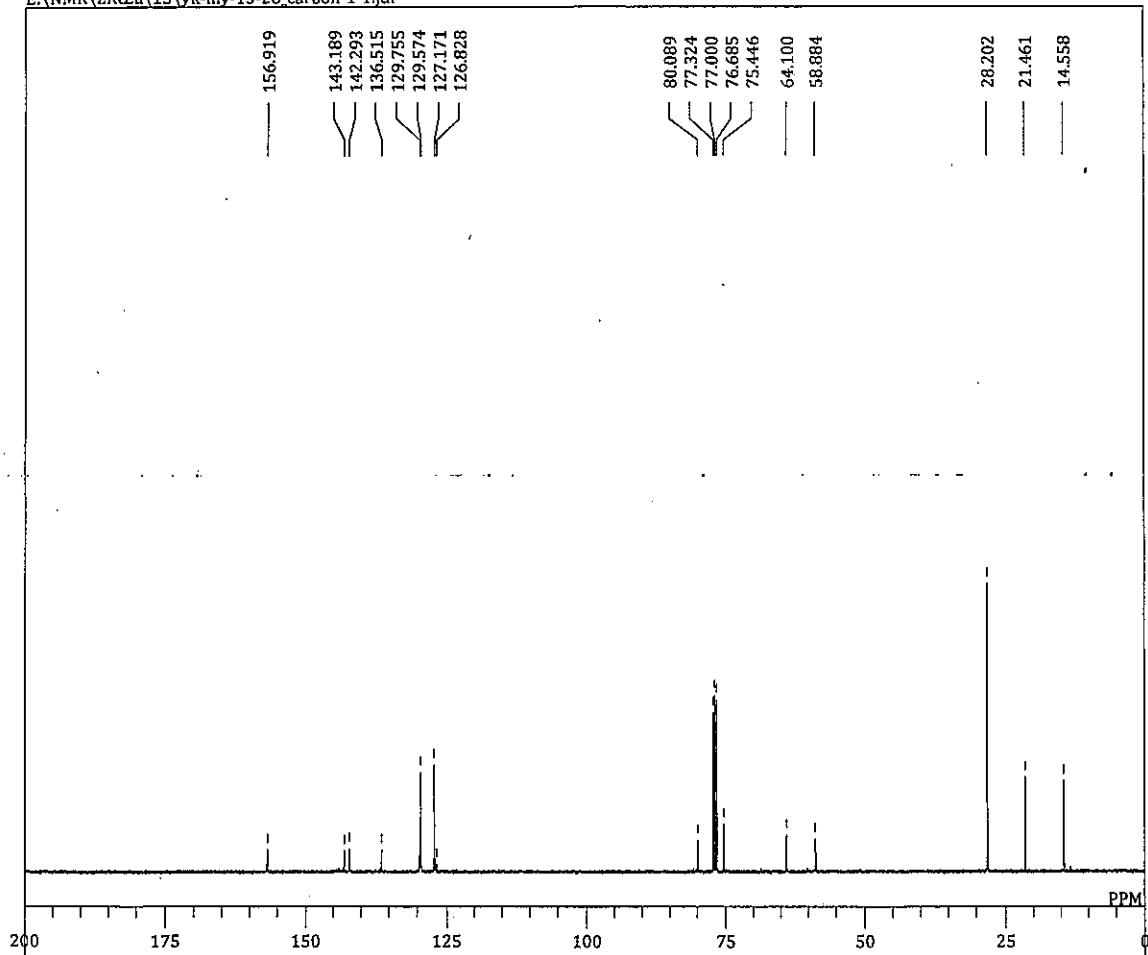
E:\NMR\ŽRCE\13\yk-my-13-26_proton-1-1.jdf



DFILE yk-my-13-26_proton-1-1.jdf
 COMNT yk-my-13-26
 DATIM 2016-01-16 14:10:07
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 399.78 MHz
 OBSET 4.19 kHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 8
 ACQTM 2.1837 sec
 PD 5.0000 sec
 PW1 5.01 usec
 IRNUC 1H
 CTEMP 21.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 28

yk-my-13-26_yk-my-13-26_proton

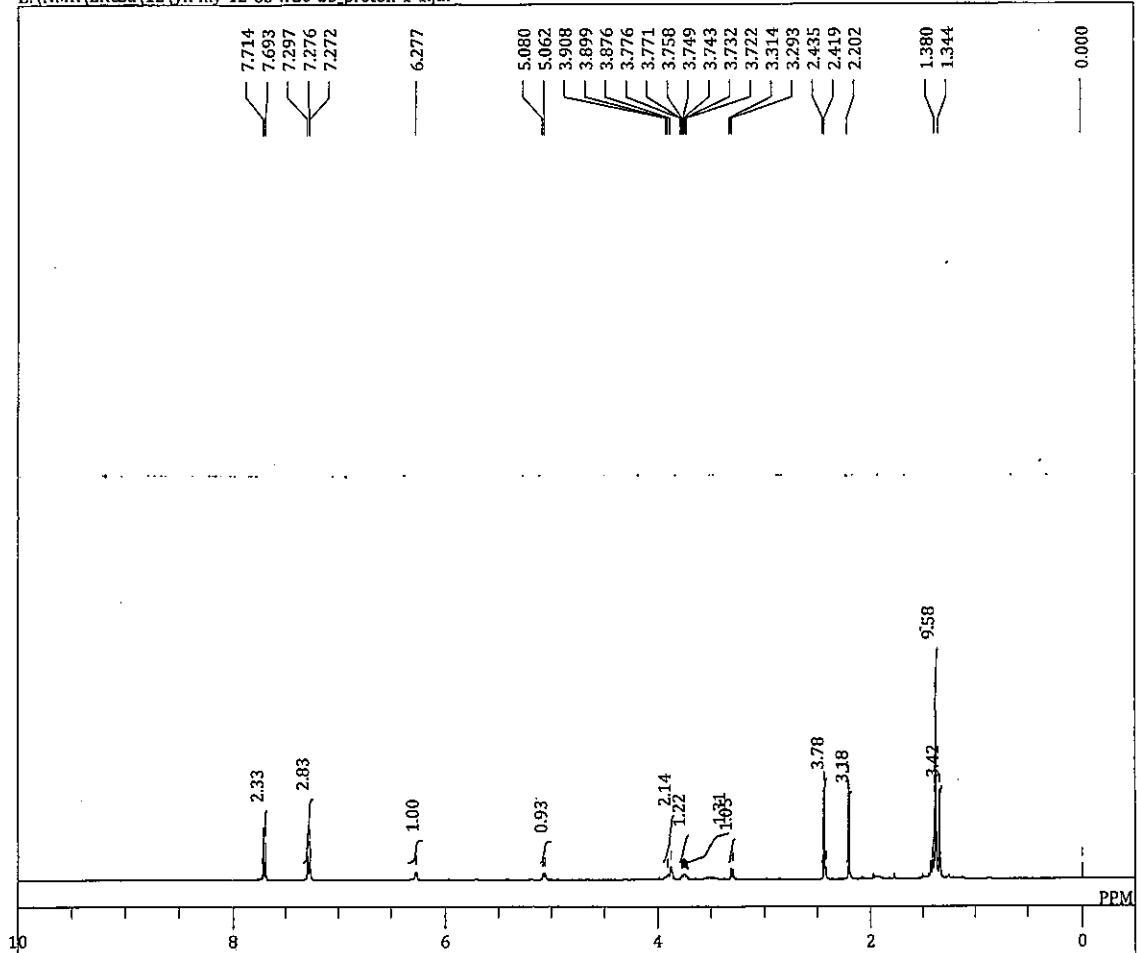
E:\NMR\ŽRCE\13\yk-my-13-26_carbon-1-1.jdf



DFILE yk-my-13-26_carbon-1-1.jdf
 COMNT yk-my-13-26_yk-my-13-26_proton
 DATIM 2016-01-16 14:11:34
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 281
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 2.77 usec
 IRNUC 1H
 CTEMP 22.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60

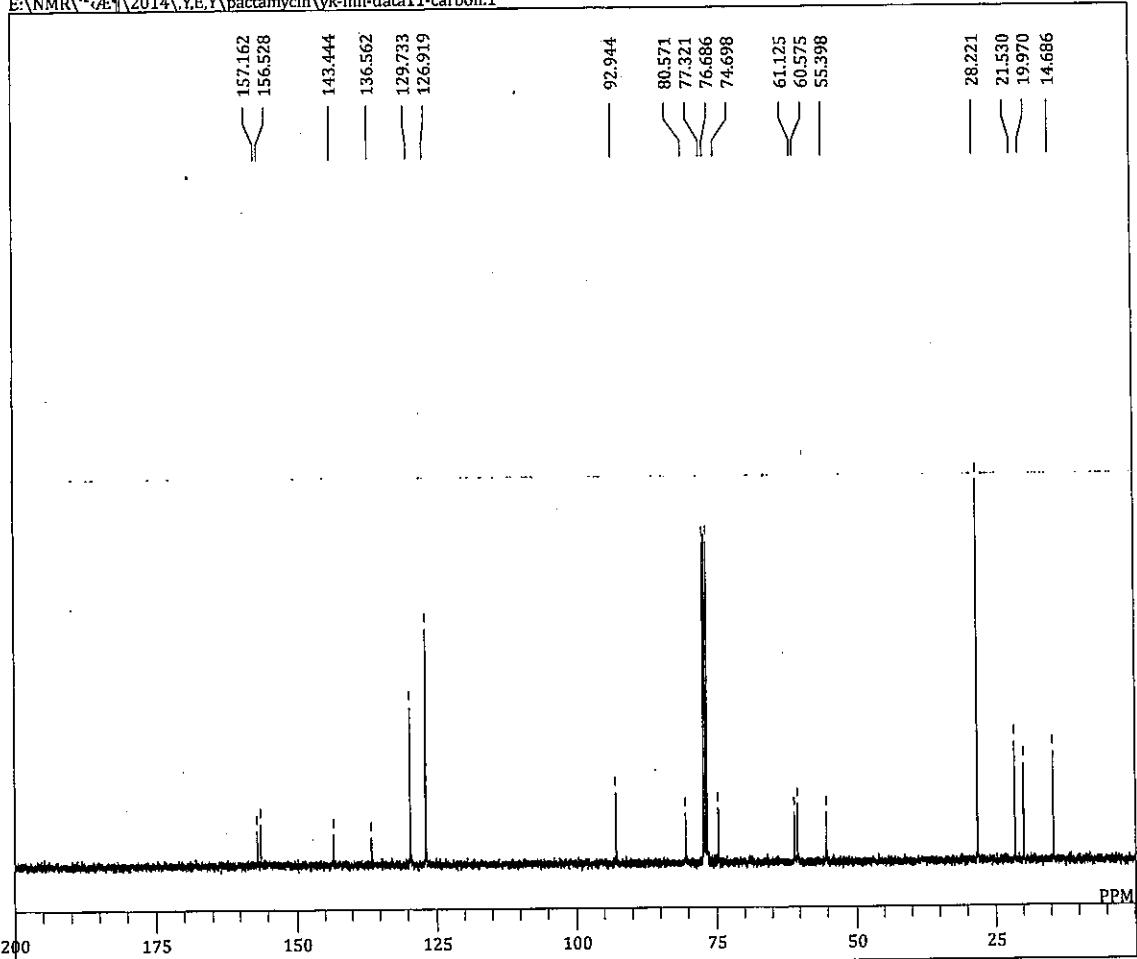
yk-my-12-63-fr20-23

E:\NMR\ZRC\112\yk-my-12-63-fr20-23_proton-1-1.jdf



yk-mh-data11-carbon

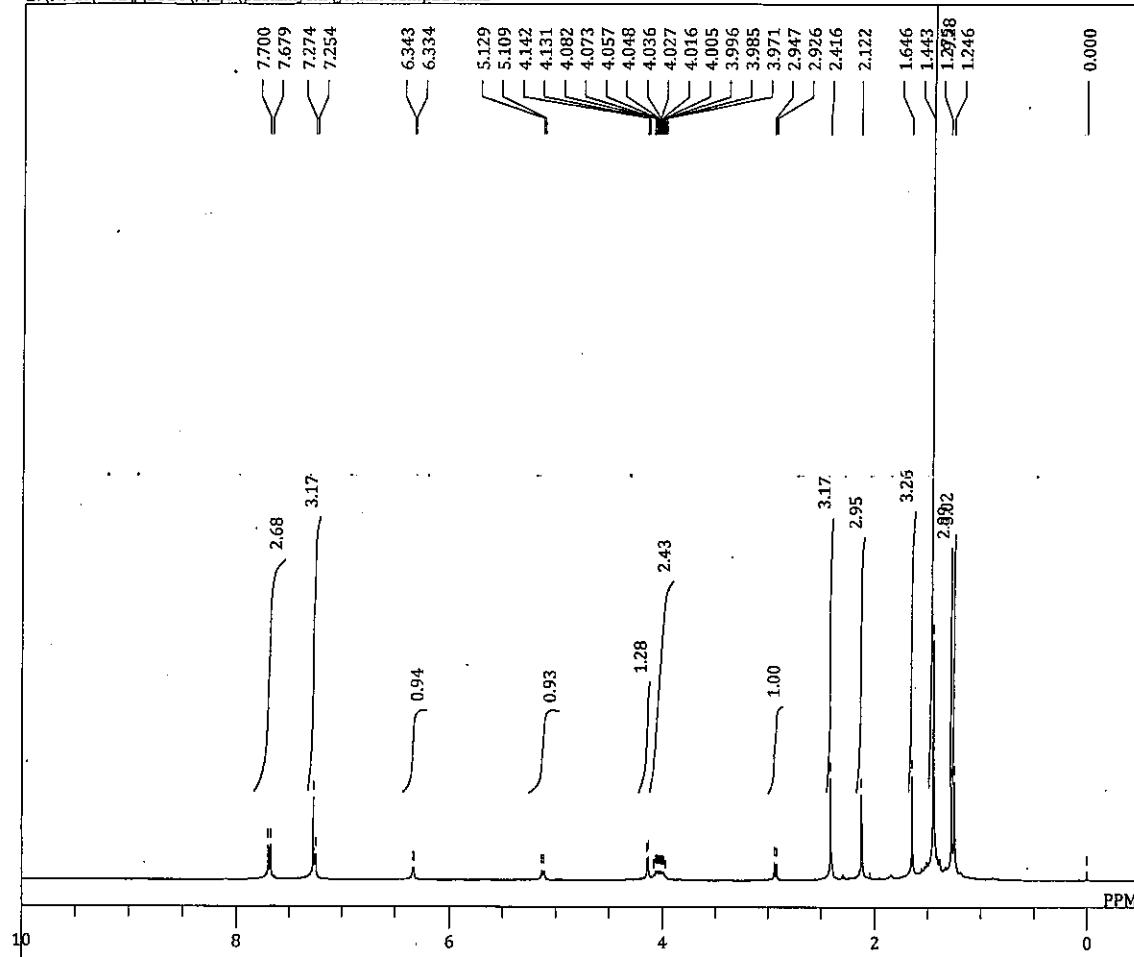
E:\NMR\13C\2014\Y.E.Y\pactamycin\yk-mh-data11-carbon.1



DFILE yk-my-12-63-fr20-23_proton-1-1
 COMNT yk-my-12-63-fr20-23_2015-11-20 11:26:41
 DATIM 2015-11-20 11:26:41
 IRNUC 1H
 EXMOD proton.jxp
 OBFRQ 399.78 MHz
 OBSET 4.19 kHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 8
 ACQTM 2.1837 sec
 PD 5.0000 sec
 PW1 5.01 usec
 IRNUC 1H
 CTEMP 19.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 34

yk-mh-comp21-2

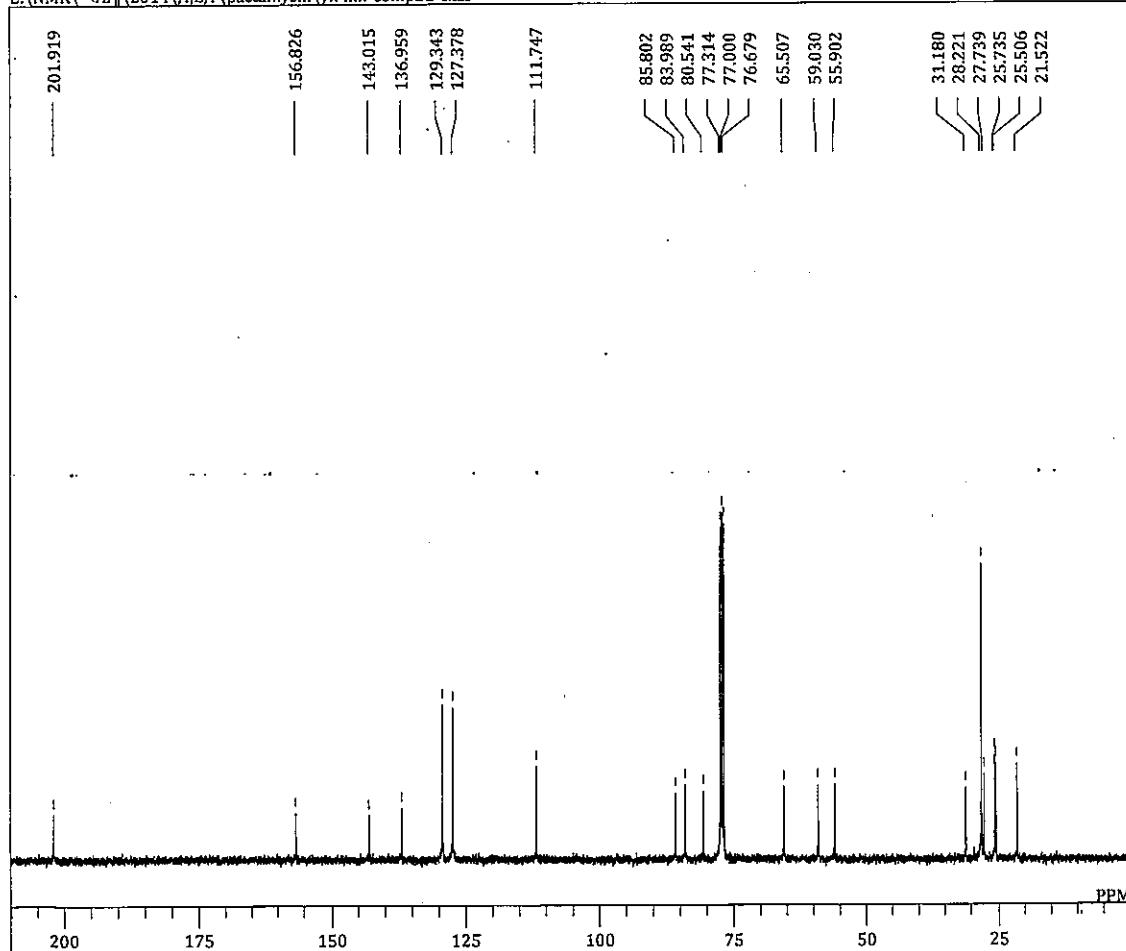
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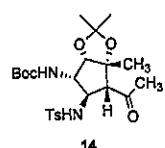
DFILE yk-mh-comp21-2.als
 COMNT yk-mh-comp21-2
 DATIM 2014-12-04 19:19:35
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 399.78 MHz
 OBSET 4.19 kHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 5998.80 Hz
 SCANS 8
 ACQTM 2.7312 sec
 PD 2.0000 sec
 PW1 5.75 usec
 IRNUC
 CTEMP 25.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 11

yk-mh-comp21-c

E:\NMR\^2\ET\2014\Y\B\Y\pactamycin\yk-mh-comp21-c.als



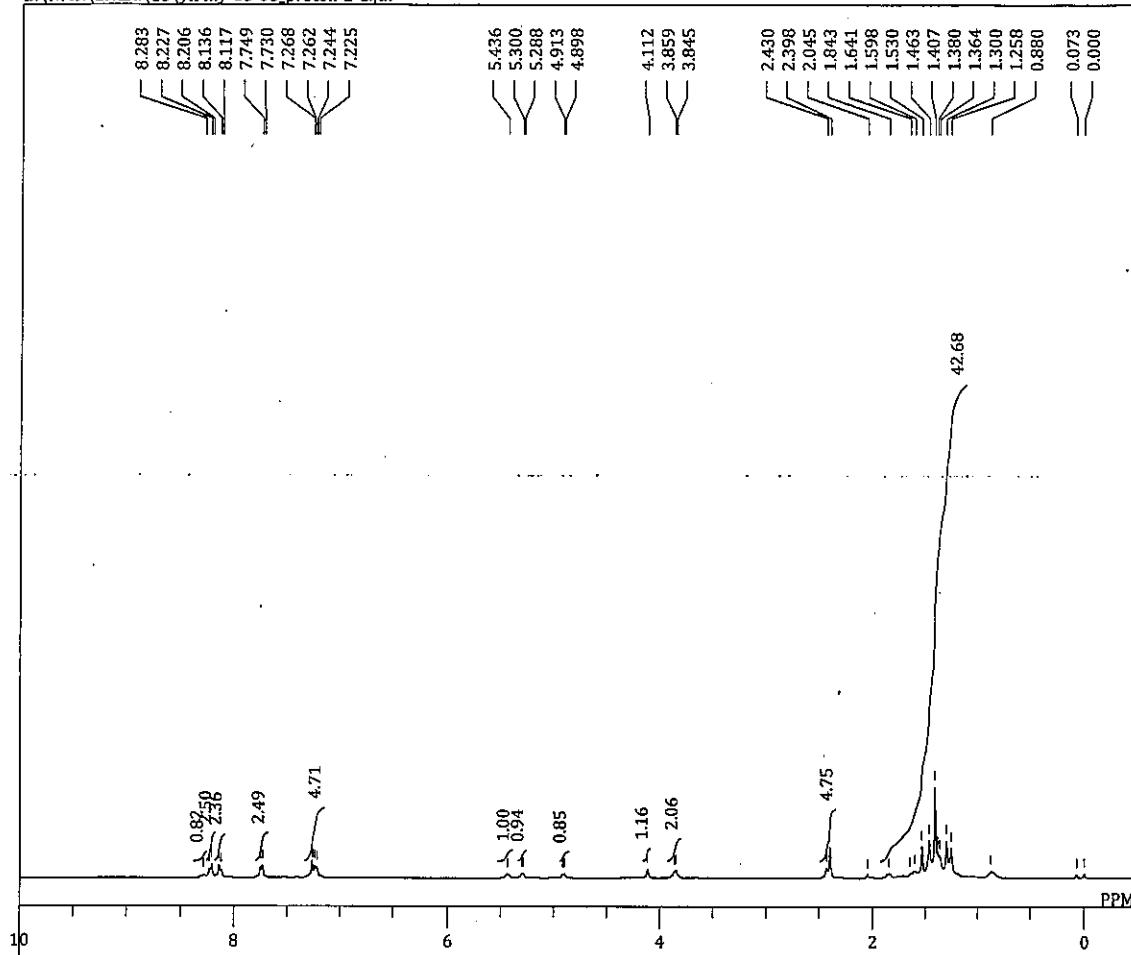
DFILE yk-mh-comp21-c.als
 COMNT yk-mh-comp21-c
 DATIM 2014-12-04 20:39:11
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 32768
 FREQU 25188.92 Hz
 SCANS 1000
 ACQTM 1.3009 sec
 PD 1.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 24



S 27

yk-my-13-68

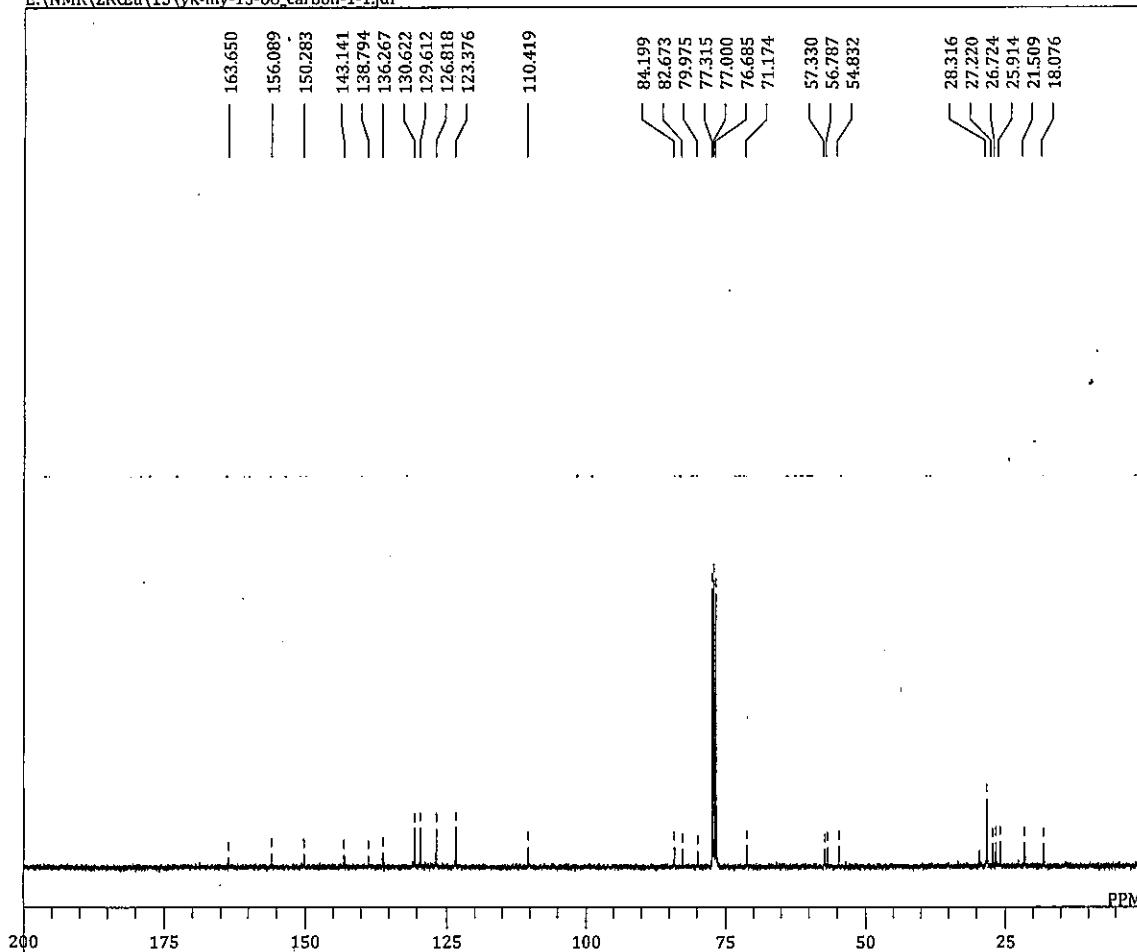
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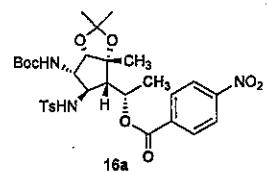
DFILE yk-my-13-68_proton-2-1.jdf
 COMNT yk-my-13-68
 DATIM 2016-03-10 13:38:02
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 399.78 MHz
 OBSET 4.19 kHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 8
 ACQTM 2.1837 sec
 PD 5.0000 sec
 PW1 5.01 usec
 IRNUC 1H
 CTEMP 23.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 34

single pulse decoupled gated NOE

E:\NMR\ZRE01\13\yk-my-13-68_carbon-1-1.jdf

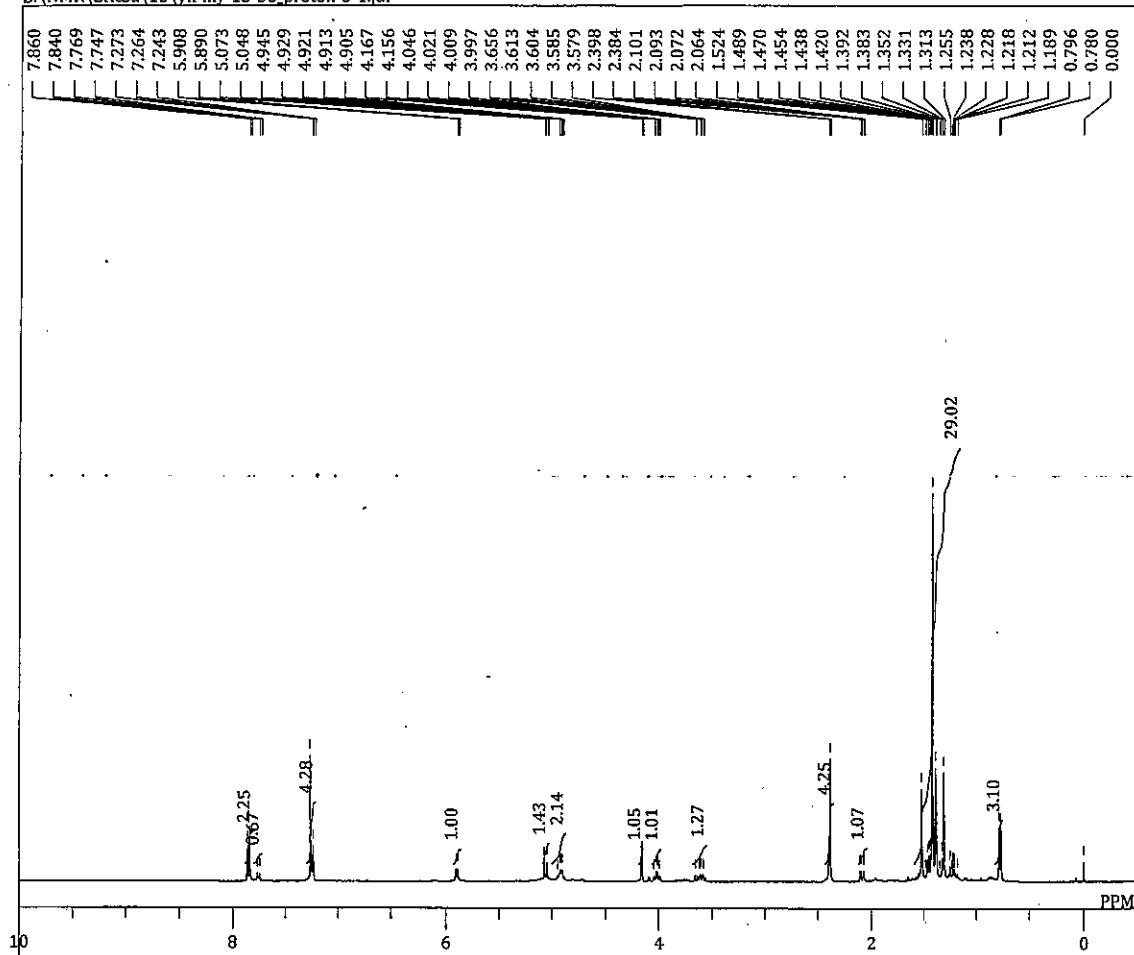


DFILE yk-my-13-68_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2016-03-10 13:39:28
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 331
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 2.77 usec
 IRNUC 1H
 CTEMP 23.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60

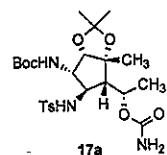


yk-my-13-36

E:\NMR\ZRC0\13\yk-my-13-36_proton-3-1.jdf

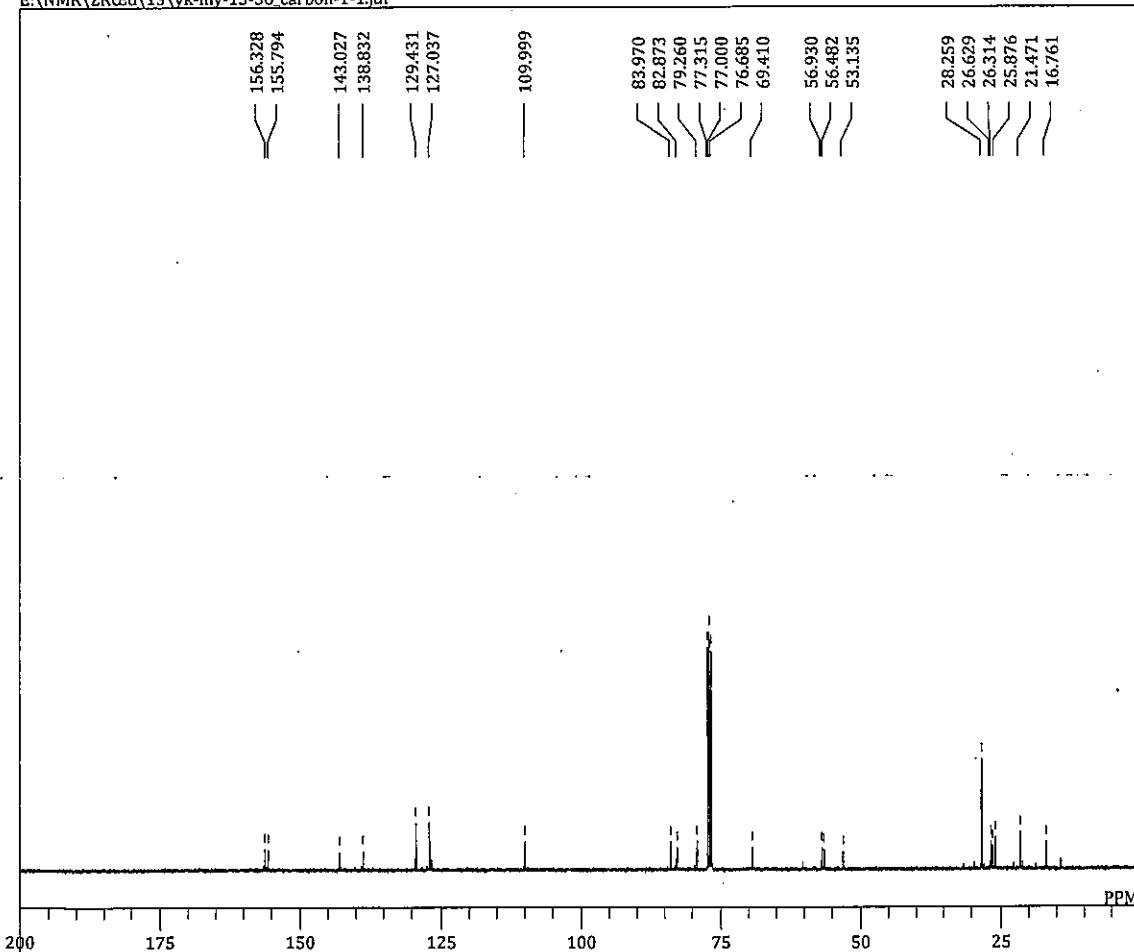


DFILE yk-my-13-36_proton-3-1.jdf
 COMNT yk-my-13-36
 DATIM 2016-01-27 23:23:57
 1H
 EXMOD proton.jxp
 OBFRQ 399.78 MHz
 OBSET 4.19 kHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 8
 ACQTM 2.1837 sec
 PD 5.0000 sec
 PW1 5.01 usec
 1H
 IRNUC 20.2 c
 CTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.12 Hz
 RGAIN 30

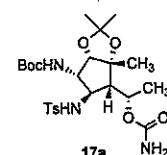


yk-my-13-36_yk-my-13-36_proton

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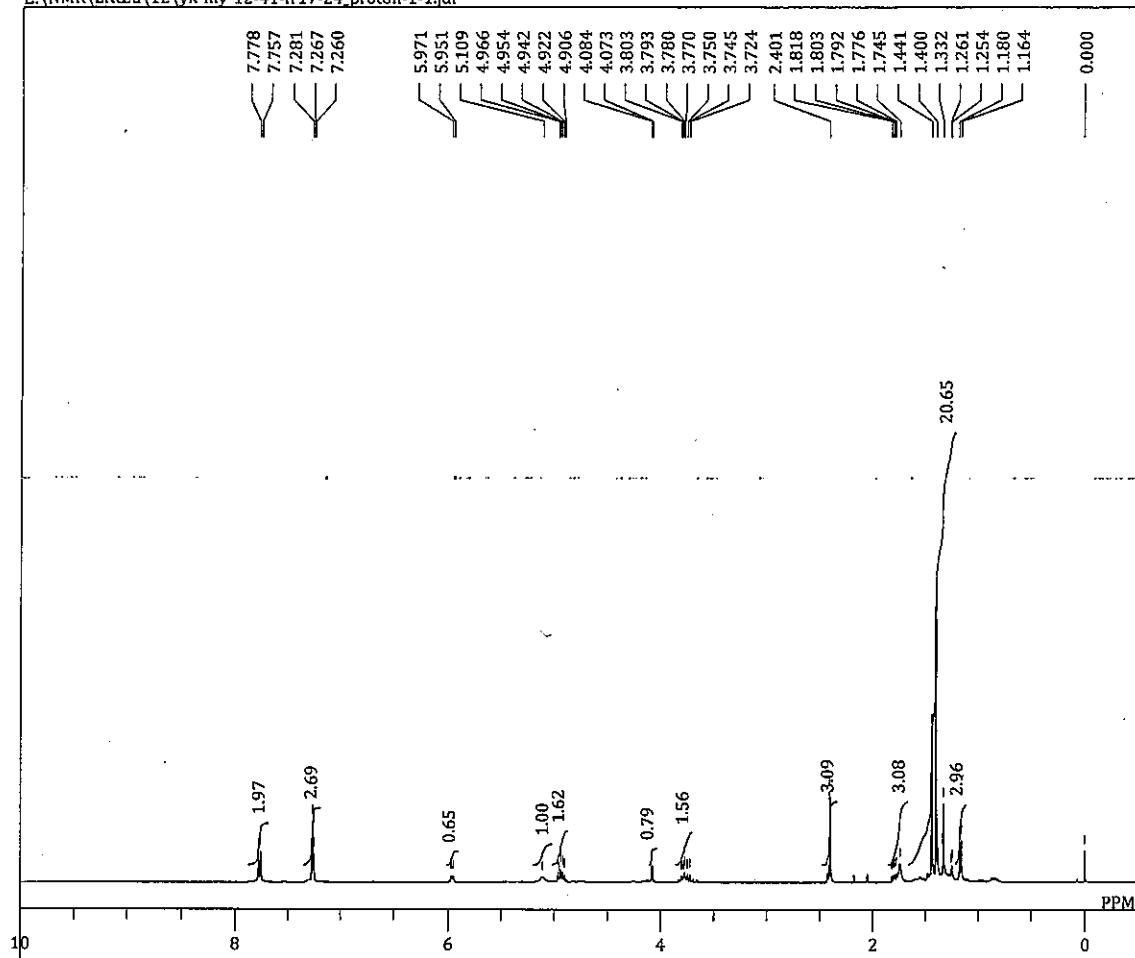
DFILE yk-my-13-36_carbon-1-1.jdf
 COMNT yk-my-13-36_yk-my-13-36_proto
 DATIM 2016-01-26 12:30:38
 13C
 EXMOD carbon.jxp
 OBFRQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 310
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 2.77 usec
 1H
 IRNUC 19.9 c
 CTEMP CDCL3
 SLVNT 77.00 ppm
 EXREF 1.20 Hz
 RGAIN 60



S 29

yk-my-12-41-fr17-24

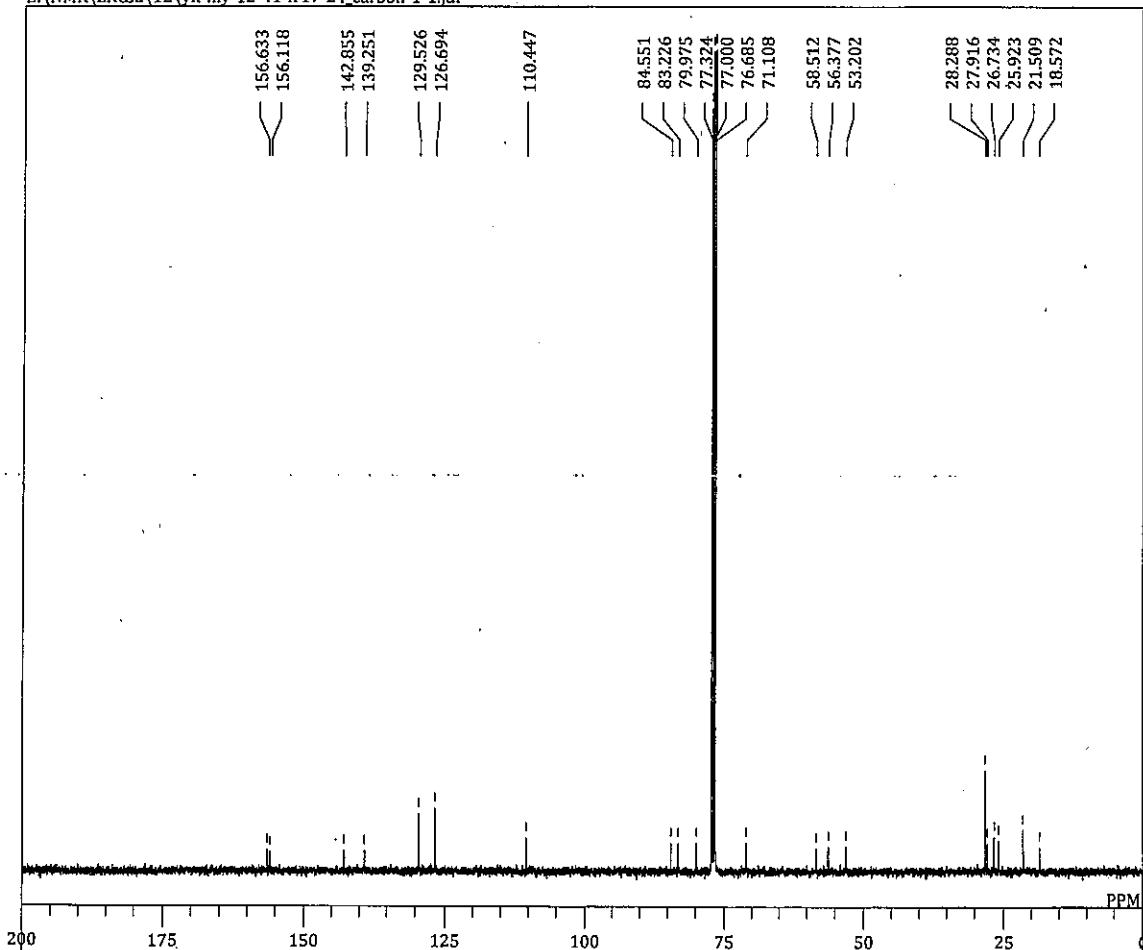
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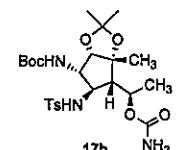
DFILE yk-my-12-41-fr17-24_proton-1-1
 COMNT yk-my-12-41-fr17-24
 DATIM 2015-10-30 13:33:54
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 399.78 MHz
 OBSET 4.19 kHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 8
 ACQTM 2.1837 sec
 PD 5.0000 sec
 PW1 5.01 usec
 IRNUC 1H
 CTEMP 20.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 42

yk-my-12-41-fr17-24

E:\NMR\ZRC\12\yk-my-12-41-fr17-24_carbon-1-1.jdf

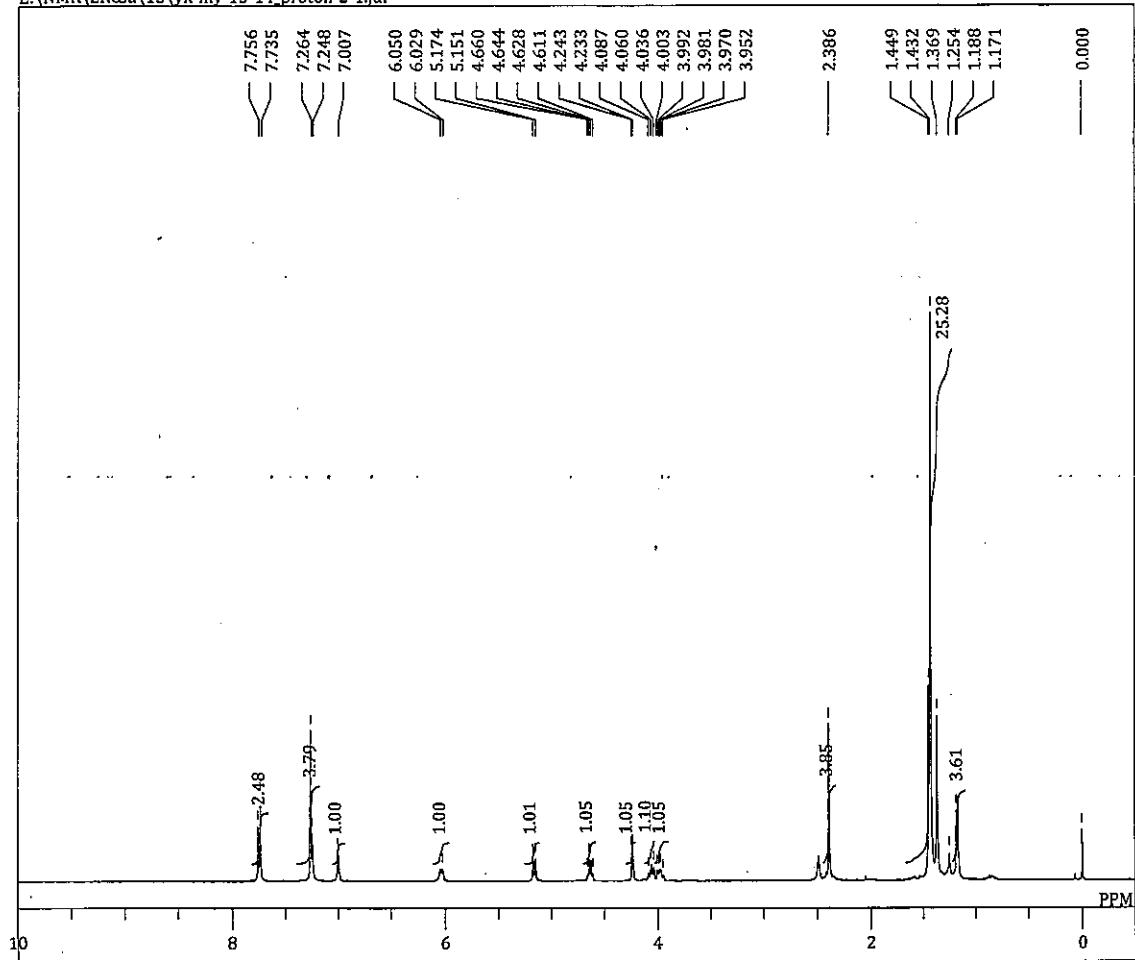


DFILE yk-my-12-41-fr17-24_carbon-1-1
 COMNT yk-my-12-41-fr17-24
 DATIM 2015-10-30 13:53:52
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 469
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 2.77 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60

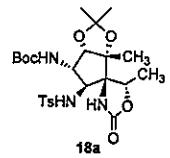


yk-my-13-14

E:\NMR\ŽRŒG\13\yk-my-13-14_proton-2-1.jdf

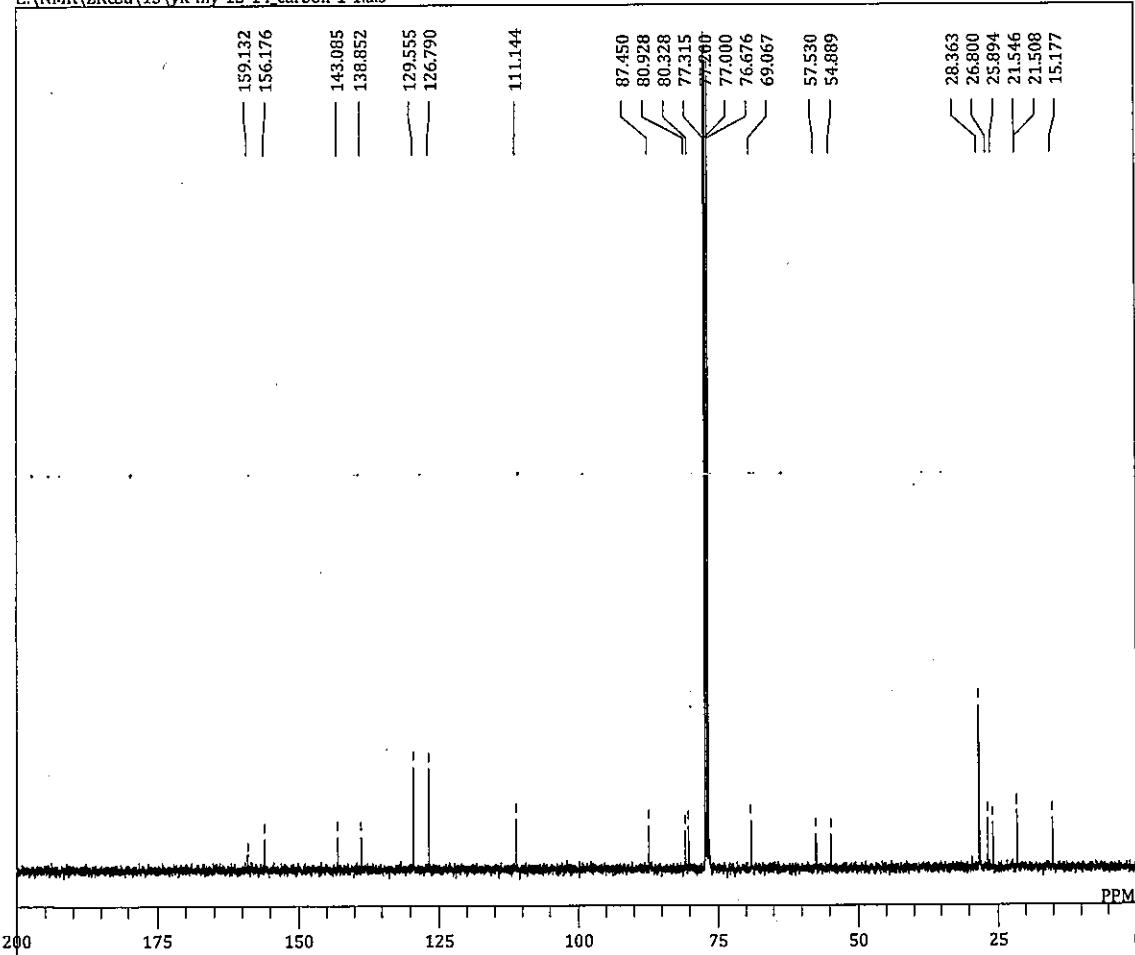


DFILE yk-my-13-14_proton-2-1.jdf
 COMNT yk-my-13-14_
 DATIM 2016-01-05 14:52:37
 1H
 EXMOD proton.jxp
 OBFRQ 399.78 MHz
 OBSET 4.19 kHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 8
 ACQTM 2.1837 sec
 PD 5.0000 sec
 PW1 5.01 usec
 1H
 IRNUC 23.3 c
 CTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.12 Hz
 RGAIN 40

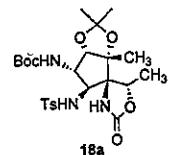


yk-my-13-14

E:\NMR\ŽRŒG\13\yk-my-13-14_carbon-1-1.als



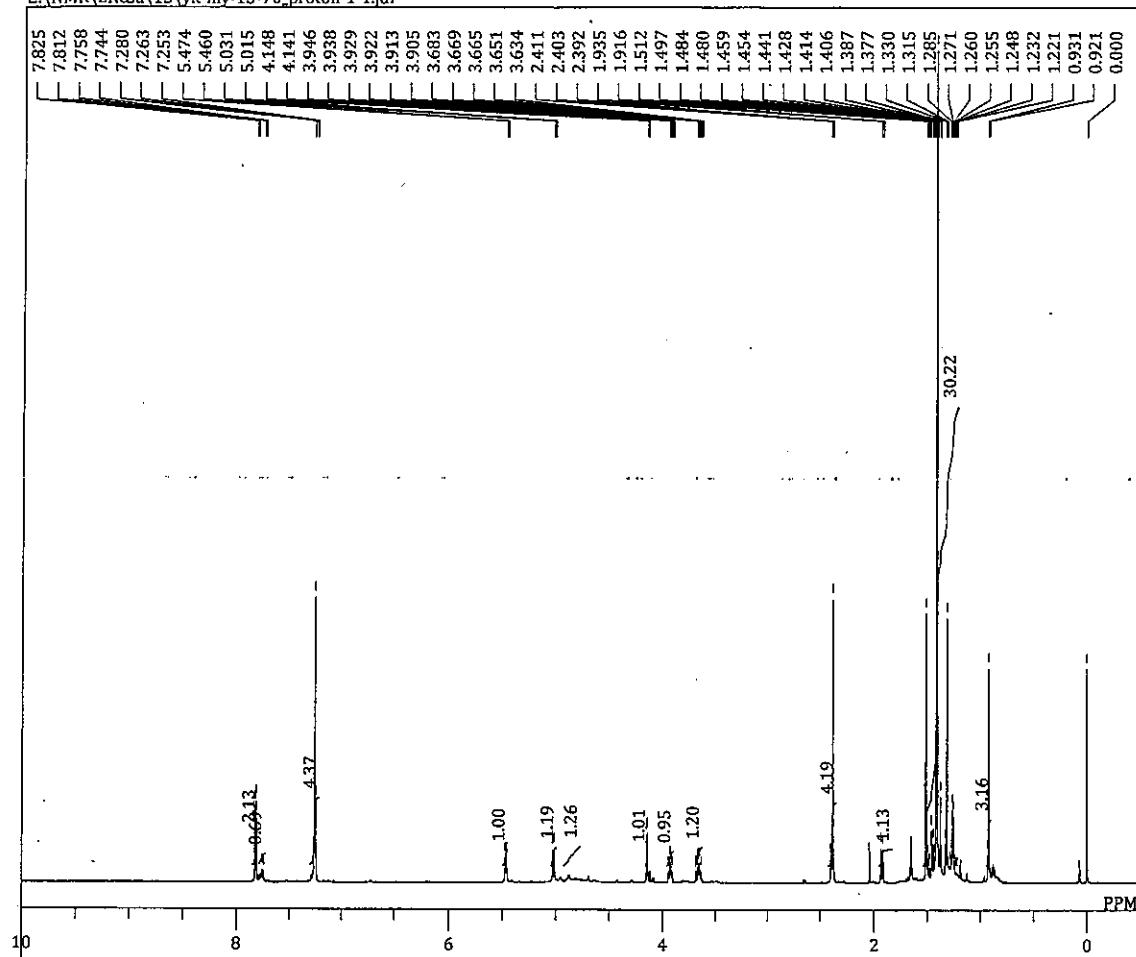
DFILE yk-my-13-14_carbon-1-1.als
 COMNT yk-my-13-14_
 DATIM 2016-01-05 16:07:45
 13C
 EXMOD carbon.jxp
 OBFRQ 100.53 MHz
 OBSET 5.35 kHz
 OBFIN 5.86 Hz
 POINT 26214
 FREQU 25125.63 Hz
 SCANS 463
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 2.77 usec
 1H
 IRNUC 24.0 c
 CTEMP CDCL3
 SLVNT 77.00 ppm
 EXREF 1.20 Hz
 RGAIN 60



S 3

yk-my-13-70

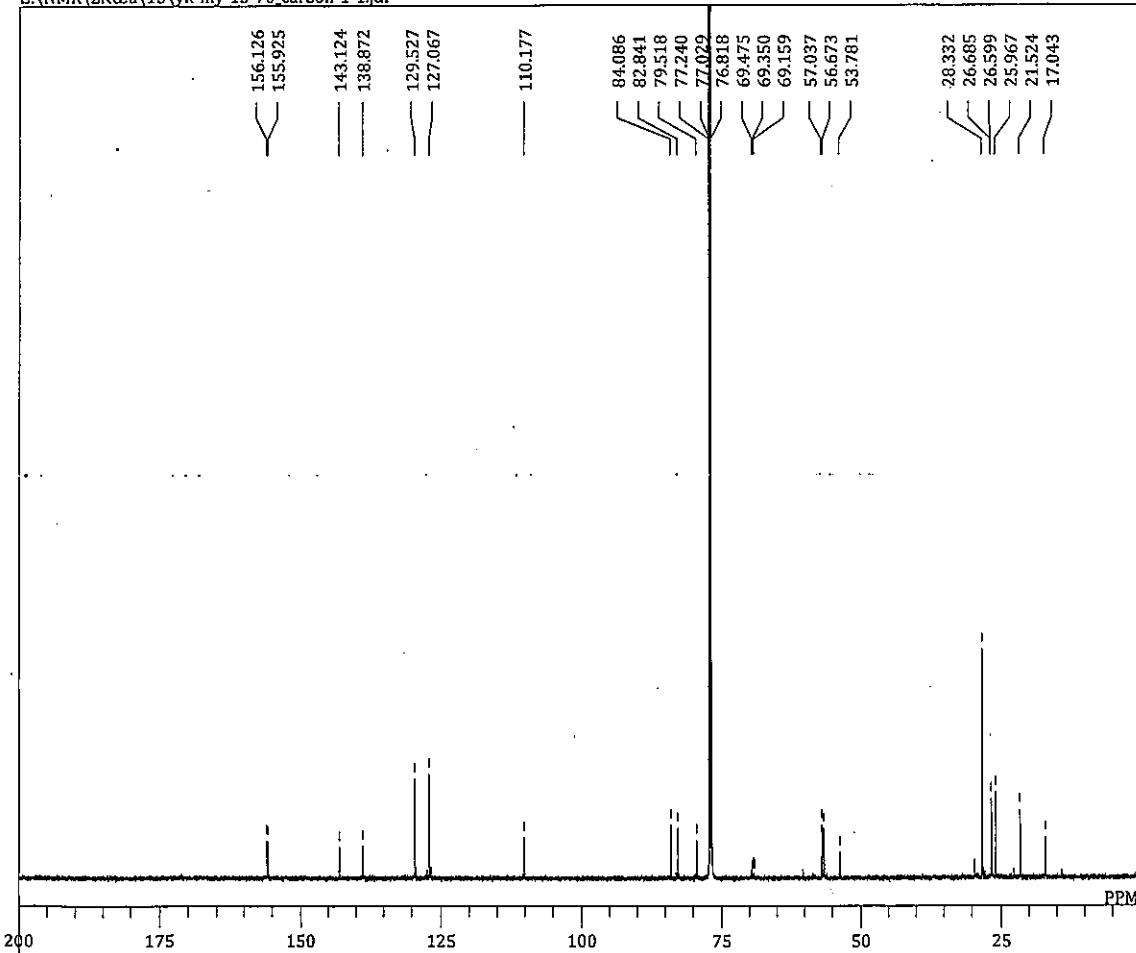
E:\NMR\ZRC01\13\yk-my-13-70_proton-1-1.jdf



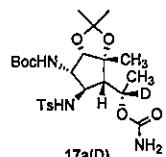
DFILE yk-my-13-70_proton-1-1.jdf
 COMNT yk-my-13-70
 DATIM 2016-03-03 19:29:32
 1H
 OBNUC proton.jxp
 EXMOD 600.17 MHz
 OBFRQ 5.30 kHz
 OBSET 5.47 Hz
 OBFIN 16384
 POINT 11261.26 Hz
 SCANS 8
 ACQTM 1.4549 sec
 PD 5.0000 sec
 PW1 5.97 usec
 1H
 IRNUC 22.9 c
 CTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF BF 0.12 Hz
 RGAIN 44

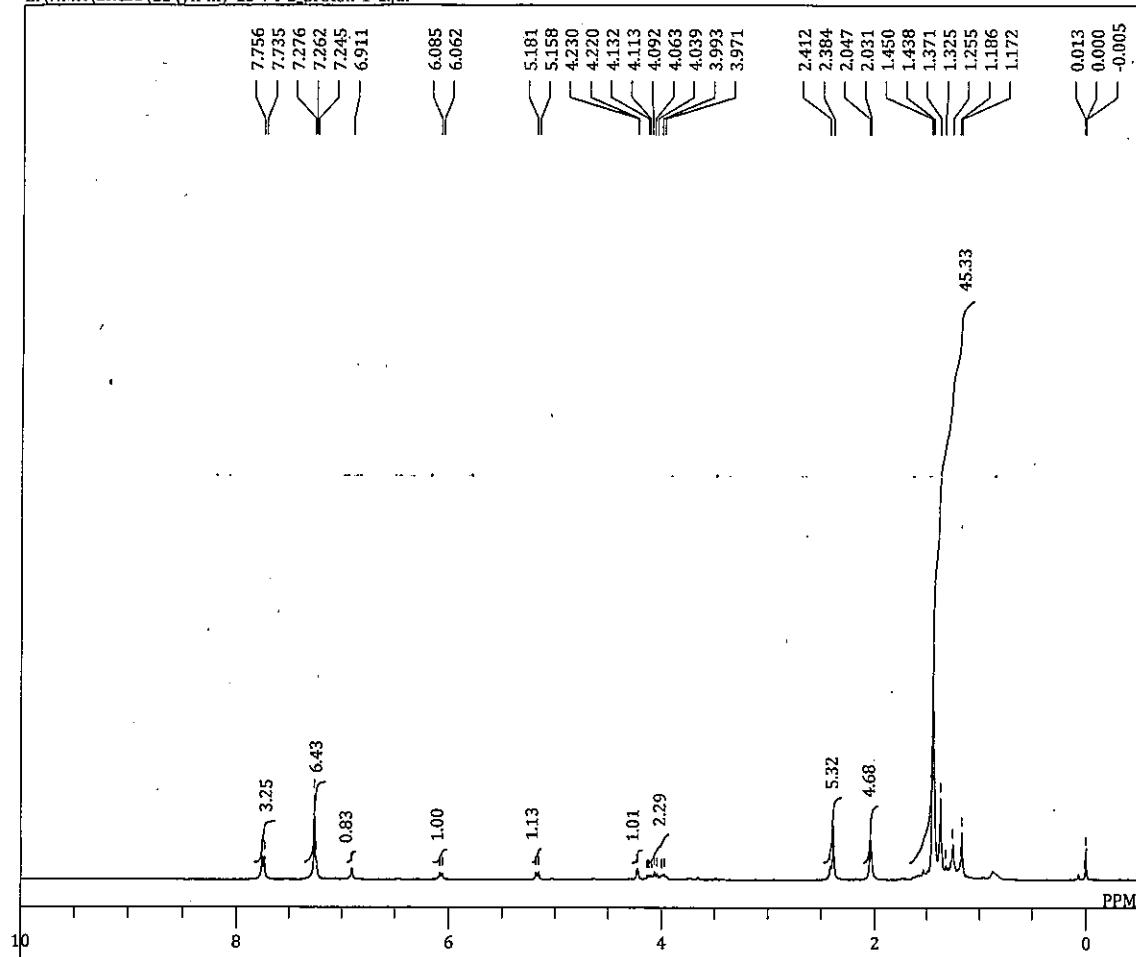
single pulse decoupled gated NOE

E:\NMR\ZRC01\13\yk-my-13-70_carbon-1-1.jdf

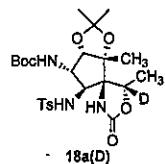


DFILE yk-my-13-70_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2016-03-03 19:31:54
 13C
 EXMOD carbon.jxp
 OBFRQ 150.92 MHz
 OBSET 8.52 kHz
 OBFIN 1.74 Hz
 POINT 32767
 FREQU 47348.49 Hz
 SCANS 5289
 ACQTM 0.6921 sec
 PD 2.0000 sec
 PW1 3.45 usec
 1H
 IRNUC 23.8 c
 CTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF BF 1.20 Hz
 RGAIN 54

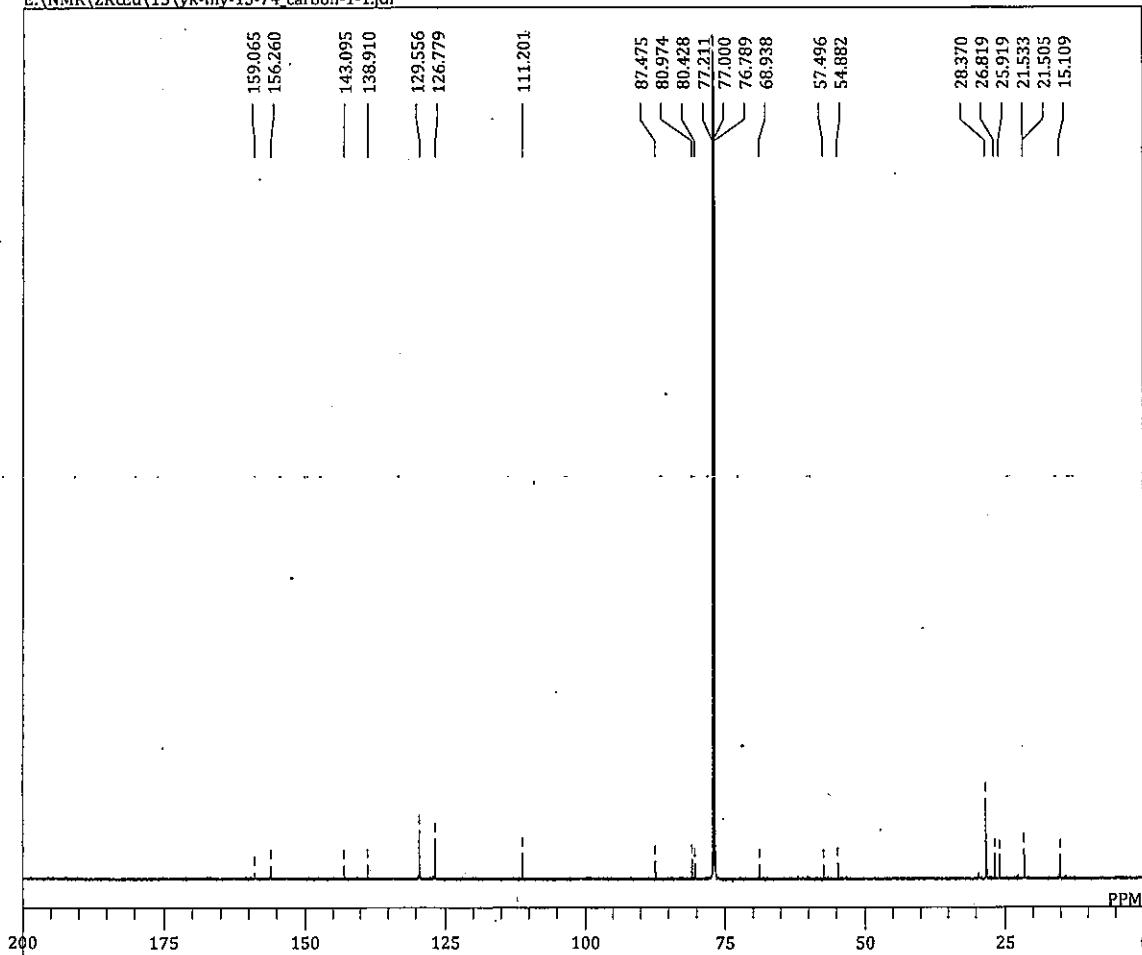




DFILE yk-my-13-74-2_proton-1-1.jdf
 COMNT yk-my-13-
 DATIM 2016-03-10 13:31:55
 1H
 proton.jxp
 OBRQ 399.78 MHz
 OBSET 4.19 KHz
 OBPIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 8
 ACQTM 2.1837 sec
 PD 5.0000 sec
 PW1 5.01 usec
 1H
 IRNUC 23.4 c
 CTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF BF 0.12 Hz
 RGAIN 44



single pulse decoupled gated NOE



DFILE yk-my-13-74_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2016-03-09 12:38:19
 13C
 carbon.jxp
 OBRQ 150.92 MHz
 OBSET 8.52 KHz
 OBPIN 1.74 Hz
 POINT 32767
 FREQU 47348.49 Hz
 SCANS 3905
 ACQTM 0.6921 sec
 PD 2.0000 sec
 PW1 3.45 usec
 1H
 IRNUC 26.0 c
 CTEMP CDCL3
 SLVNT 77.00 ppm
 EXREF 1.20 Hz
 RGAIN 54

