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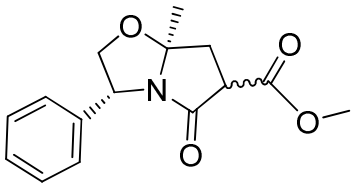
I. General

All reactions were carried out in oven-dried glassware under atmosphere of argon or nitrogen with magnetic stirring, unless otherwise noted. Bicyclic lactam **I-IV** were prepared from established literature procedures.[1,2] Lithium hexamethyldisilazane and aryl flurorides were purchased from Aldrich. Dichloromethane was distilled over CaO before use.
All NMR spectra were recorded in DMSO-D_6 or $CDCl_3$, unless otherwise noted.

II. Synthesis of monoacylated bicyclic lactam

General procedure for the synthesis of monoacylated bicyclic lactams (1-4). A solution of **lactam** in THF was added to a cooled (-78^{o}C) solution of LiHMDS in THF. After stirring the solution at -78^{o}C for 30 minutes methyl chloroformate was added and stirred for 30 minutes at -78^{o}C (The reaction progress was monitored by quenching saturated NH_4Cl and extracting with EtOAC, spotting the reaction mass over an analytical silica gel TLC plate and visualizing respective spots using 254 nm UV light and $KMnO_4$ charring shows yellow colour, 30 % EtOAC/Hexane, (SM and product was same Rf if double run the TLC its slight polar than the SM). The reaction mixture was quenched by addition saturated NH_4Cl and extracting with EtOAC.The combined organic layer was dried over Na_2SO_4 and concentrated on reduced pressure to afford the desired **monoester**, the crude was proceeds next step without further purification.



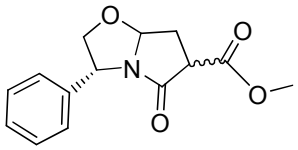
Compound 1. The general procedure was followed. Bicyclic lactam **I** (2g, 9.2mmol), LHMDS, 1M in THF (9.2mL, 9.2mmol), methyl chloroformate (1.1mL, 13.8mmol) and THF (20mL). Flash column chromatography furnished the product (1.2g, 73%) as off white solid.

^{1}H NMR (400 MHz, $CDCl_3$) 7.36-7.32 (m, 2H), 7.29-7.21 (m, 3H), 5.19-5.17 (m, 1H), 4.69-4.64 (m, 1H), 4.19-4.16 (m, 0.5H), 4.11-4.07 (m, 0.5H), 3.97-3.95 (m, 0.5H), 3.83 (s, 1.5H), 3.81 (s, 1.5H), 3.83-3.72 (m, 0.5H), 2.75-2.65 (m, 1H), 2.56-2.47 (m, 1H), 1.60 (s, 1.5H), 1.53 (s, 1.5H).

^{13}C NMR (100 MHz, $CDCl_3$) 172.7, 169.1, 139.2, 129.1, 127.8, 125.9, 98.2, 73..2, 57.7, 52.7, 51.1, 36.7, 24.5.

FTIR (thin film) 2978, 2784, 1713, 1435 cm^{-1}

LC-MS calc. for $C_{15}H_{17}NO_4$ (M + H) 276.12, found 276.34



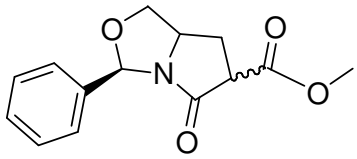
Compound 2. The general procedure was followed. Bicyclic lactam **II** (10 g, 49.26 mmol), LHMDS, 1M in THF (123.15mL, 123.15 mmol), methyl chloroformate (5.72mL, 73.15mmol) and THF (75 mL). Flash column chromatography furnished the product (12g, crude) as white solid.

^{1}H NMR (400 MHz, $CDCl_3$) 7.39-7.35 (m, 2H), 7.31-7.28 (m, 3H), 5.42-5.36 (m, 1H), 4.98-4.93 (m, 1H), 4.69-4.62 (m, 1H), 4.10-4.06 (t, J = 12Hz, J = 12Hz, 0.5H), 3.80-3.77 (m, 0.5H), 3.76-4.68 (m, 3H), 2.77-2.68 (m, 1H), 2.33-2.27 (m, 1H), obscured peak under 3.76-4.68 (m, 1H)

^{13}C NMR (100 MHz, $CDCl_3$) 172.6, 169.1, 139.2, 129.1, 127.8, 125.9, 98.2, 73.3, 57.7, 52.7, 51.1, 36.8, 24.6.

FTIR (thin film) 2965, 2864, 1728, 1448 cm^{-1}

LC-MS calc. for $C_{15}H_{15}NO_4$ (M + H) 262.1, found 262.00



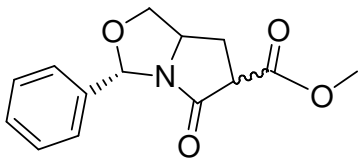
Compound 3. The general procedure was followed. Bicyclic lactam **III** (1g, 4.93mmol), LHMDS, 1M solution in THF (12.3mL, 12.3mmol), methyl chloroformate (0.6mL, 7.32mmol) and THF (20mL). Flash column chromatography furnished the product (1g, 77%) as white solid.

^{1}H NMR (400 MHz, $CDCl_3$) 7.46-7.31 (m, 5H), 6.32(s, 1H), 4.33-4.25(m, 1.5H), 4.17-4.09(m, 0.5H), 3.92-3.91(m, 0.5H), 3.81-3.79(d, J=9.2Hz, 3H), 3.72-3.66(m, 1 H), 3.54-3.51(m, 0.5H), 2.81-2.75(m, 0.5 H), 2.64-2.54(m, 0.5H), 2.46-2.39(m, 0.5H), 2.19-2.12(m, 0.5H)

^{13}C NMR (100 MHz, $CDCl_3$) 172.1, 171.9, 169.7, 169.4, 138.3, 137.9, 128.7, 128.8, 128.5, 126.2, 125.9, 87.3, 87.1, 71.8, 71.6, 57.9, 56.9, 53.0, 52.8, 52.0, 51.3, 27.7, 27.5.

FTIR (thin film) 2953, 1715, 1166 cm^{-1}

LC-MS calc. for $C_{14}H_{15}NO_4$ (M + H) 262.2 found 262.12

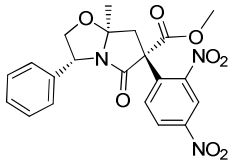


Compound 4. The general procedure was followed. Bicyclic lactam **IV** (1g, 4.93mmol), LHMDS, 1M solution in THF (12.3mL, 12.3mmol), methyl chloroformate (0.6mL, 7.32mmol) and THF (20mL). Flash column chromatography furnished the product (1.1g, 79%) as white solid.

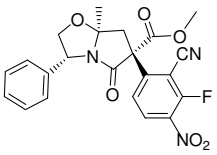
^{1}H NMR (400 MHz, $CDCl_3$) 7.45-7.31 (m, 5H), 6.33(s, 1H), 4.33-4.25(m, 1.5H), 4.16-4.09(m, 0.5H), 3.92-3.9(m, 0.5H), 3.81(d, J=9.2Hz, 3H), 3.7-3.66(m, 1 H), 3.54-3.51(m, 0.5H), 2.81-2.75(m, 0.5 H), 2.62-2.54(m, 0.5H), 2.46-2.39(m, 0.5H), 2.19-2.12(m, 0.5H)
^{13}C NMR (100 MHz, $CDCl_3$) 172.0, 171.9, 169.6, 169.4, 138.2, 137.9, 128.7, 128.6, 128.5, 126.0, 125.9, 87.3, 87.1, 71.8, 71.6, 57.9, 56.9, 53.0, 52.8, 52.0, 51.3, 27.7, 27.5.
FTIR (thin film) 2953, 1715, 1166 cm^{-1}
LC-MS calc. for $C_{14}H_{15}NO_4$ (M + H) 262.2 found 262.16

III. Arylation of monoacylated bicyclic lactam

General procedure for the arylation of monoacylated bicyclic lactams. To a suspension of sodium hydride (50% in mineral oil) in dry tetrahydrofuran at 0℃ was added dropwise a solution of monoacylated bicyclic lactam in THF. The reaction mixture was stirred at 0℃ for 1h. Then 2,4-dinitro fluorobenzene was charged dropwise for 5 min. The resulting pale yellow solution was stirred at 0℃ for 2 to 3 h. Reaction progress was monitored by TLC (eluent: 30% EA in n-hexane). After completion of the reaction, the reaction mixture was neutralised (pH:6-6.5) with 1N HCl solution and water added water, ethyl acetate and stirred for 5 min, the organic layer was separated and the aq layer was extraced with ethyl acetete. The combined organic layers were washed with water and brine, dried over anh. sodium sulphate and evaporated under vacuum to generate the crude product. It was purified by column chromatograpgy (silica:100-200 mesh, eluent: 12-15% EtOAc in peteher) to afford white solid compound.



Compound 8. The general procedure was followed. Bicyclic lactam **1** (3g, 10.9mmol), aryl fluoride **5** (7.6g, 10.9mmol) NaH in 50% dispersion in oil (0.6g, 12.1mmol) and THF (20mL). Flash column chromatography furnished the product (3.5g, 72%) as white solid.
^{1}H NMR (400 MHz, DMSO-D_6) 8.96-8.95 (d, J = 4Hz, 1H), 8.79-8.77 (m, 1H), 7.78-7.76 (d, J = 8Hz, 1H), 7.46-7.44 (m, 2H), 7.38-7.36 (m, 3H), 5.29-5.25 (m, 1H), 4.84-4.80 (m, 1H), 4.05-4.01 (m, 1H), 3.62 (s, 3H), 3.51-3.47 (d, J = 16Hz, 1H), 2.75-2.72 (m, 1H), 1.19-1.17 (d, J = 8Hz, 3H).
^{13}C NMR (100 MHz, DMSO-D_6) 173.1, 168.8, 147.8, 147.5, 140.2, 139.4, 131.7, 129.8, 129.3, 128.1, 126.0, 122.4, 97.9, 73.6, 66.9, 58.8, 53.9, 47.3, 25.1.
FTIR (thin film) 3029, 2941, 1732, 1523 cm^{-1}
HRMS calc. for $C_{21}H_{19}N_3NaO_8$ 464.1172, found 464.1172
Specific Optical Rotation $[\alpha]^{25}_{589}$: +82.200° ($CHCl_3$, c=1)



Compound 9. The general procedure was followed. Bicyclic lactam 1 (3g, 10.9mmol), aryl fluoride 6 (7.4g, 10.9mmol), NaH (0.6g, 12.1 mmol) and THF (20mL). Flash column chromatography furnished the product (4.0g, 75%) as white solid.

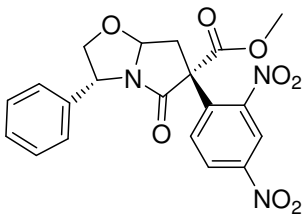
^{1}H NMR (400 MHz, $CDCl_3$) 8.38-8.34 (t, J = 8Hz, J = 8Hz, 1H), 7.52-7.50 (d, J = 8Hz, 1H), 7.43-7.39 (m, 2H), 7.36-7.33 (m, 1H), 7.26-7.25 (m, 1H), 5.36-5.32 (t, J = Hz, J = Hz, 1H), 4.76-4.72 (t, J = Hz, J = Hz, 1H), 4.22-4.18 (m, 1H), 3.87 (s, 3H), 3.80-3.77 (d, J = Hz, 1H), 2.78-2.74 (m, 1H), 1.22 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) 171.9, 167.7, 158.9, 156.1, 150.9, 138.3, 130.3, 129.1, 128.1, 125.5, 125.3, 124.4, 110.8, 97.7, 73.5, 66.5, 58.8, 54.4, 47.4, 25.4.

^{19}F NMR (MHz, $CDCl_3$) (-)108.08 - (-)108.11 (d, J = 12Hz, 1F)

FTIR (thin film) 3039, 2901, 1712, 1503 cm^{-1}

HRMS calc. for $C_{22}H_{18}FN_3NaO_6$ 462.1180, found 462.1180. Specific Optical Rotation $[\alpha]^{25}_{589}$: +112.200° ($CHCl_3$, c=1)



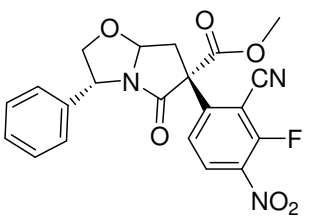
Compound 10. The general procedure was followed. Bicyclic lactam **2** (8 g, 30.65 mmol), aryl fluoride **5** (5.7 g, 30.65 mmol), NaH (3 g, 122.4mmol) and tetrahydrofuran (80 mL). The crude product was recrystallized in EtOH to afford off white solid compound **(**7.2g, 55 %)

^{1}H NMR (400 MHz, DMSO-D_6) 9.00-9.04 (d, J = 2.33 Hz, 1 H), 8.48-8.46 (d, J = 8.4Hz,J=2.8 Hz, 1H), 7.75-7.73 (d, J = 8.64 Hz,1H), 7.44-7.40 (m, J = 7.57Hz, 2H), 7.38-7.36 (m, J = 7.27Hz, 1H), 7.31-7.28 (d, J = 1.8Hz, 1H), 7.28-7.32 (m, 1H), 5.30-5.26 (m, J = 6.53Hz, 2H), 4.74-7.70 (dd, J = 8.8Hz, J = 7.44Hz,1H), 4.01-3.96 (dd,J = 9.06Hz, J = 7.3Hz, 1H), 3.75 (s, 3H), 3.66-3.61 (dd, J = 15.4Hz, J=3.18 Hz, 1H), 2.74-2.68 (dd, J = 15.4Hz, J = 6.33Hz, 0.33H),

^{13}C NMR (100 MHz, DMSO-D_6): 173.1, 168.0, 148.0, 147.4, 141.0, 138.3, 131.1, 128.2, 125.9, 121.6, 75.1, 65.1, 59.0, 53.7, 39.6.

FTIR (thin film) 3397, 2953, 2238, 1746, 1704, 1545, 1355, 1229 cm^{-1}

HRMS calc. for $C_{20}H_{17}N_3NaO_8$ 450.1016, found 450.1012 ; $[\alpha]^{23}$ D=+55.80°($CHCl_3$,c=1)



Compound 11. The general procedure was followed. Bicyclic lactam **2** (0.4 g, 1.53 mmol), aryl fluoride **6** (0.28 g, 1.53 mmol), NaH (0.14 g, 6.12 mmol) and tetrahydrofuran (8 mL). The crude product was recrystallized from EtOH to afford off white solid compound. **(**0.350 g, 58 %).

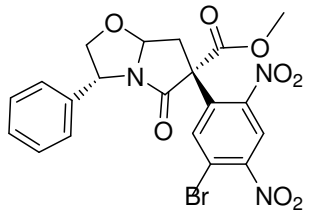
^{1}H NMR (400 MHz,$CDCl_3$) 8.30-8.26 (dd, J = 8.82Hz, J = 7.95Hz, 1H), 7.47-7.42(d, J = 9.16,J = 1.83Hz, 1H), 7.42-7.26(m, J = 7.44, 3H), 7.27(d, 1H), 7.25(t, 1H), 5.29-5.23(m, J = 6.47, 2H), 4.75-4.70 (dd, J = 9.04Hz ,J = 7.45Hz ,1H), 4.0-3.96 (dd, J = 9.15, J =7.2Hz,1H), 3.87(s, 3H), 3.61-3.57(m, 1H), 2.94-2.89 (dd, J = 14.92,J = 6Hz,1H)

^{13}C NMR (100MHz,$CDCl_3$): 171.7, 167.7, 159.2, 155.5, 150.8, 138.1, 136.8, 130.3, 130.3, 129.1, 128.3, 125.9, 124.1, 124.0, 110.9, 105.4, 90.0, 75.3, 65.3, 58.8, 54.4, 39.3.

FTIR (thin film): 3429, 2924, 2238, 1740, 1709, 1540, 1363, 1240, 1091 cm^{-1}

HRMS calc. for $C_{21}H_{16}FN_3NaO_8$ 448.1023, found 448.1023.

Specific Optical Rotation $[\alpha]^{25}_{589}$: +103.600° ($CHCl_3$, c=1)



Compound 12. The general procedure was followed. Bicyclic lactam **2** (0.4 g, 1.53 mmol), aryl fluoride **7** (0.4 g, 1.53 mmol), NaH (0.14 g, 6.12 mmol) and tetrahydrofuran (8 mL). The crude product was recrystallized from EtOH to afford off white solid compound **(**0.57 g, 75 %)

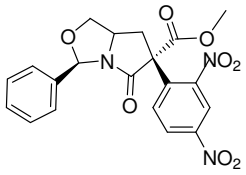
^{1}H NMR (400 MHz, $CDCl_3$) : 8.71(s, 1H), 7.94 (s, 1H), 7.45-7.28(m, 5H), 5.31-5.27(m, 2H), 4.38(dd, J=8.99, J=7.46Hz, 1H), 3.99-3.95(dd, J= 8.95, J= 6.88Hz, 1H), 3.76(s, 1H), 3.63-3.58 (m, 1H), 2.73-2.68(m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): 172.3, 167.7, 148.6, 146.4, 139.5, 138.1, 136.7, 129.2, 128.3, 125.8, 123.7, 121.4, 89.9, 65.3, 58.8, 53.8, 39.6.

FTIR (thin film): 3416, 1742,1708, 1584, 1531,1338, 704 cm^{-1}

HRMS calc. for $C_{20}H_{16}BrN_3NaO_8$ 528.0121, found 528.0118

Specific Optical Rotation $[\alpha]^{25}_{589}$: +2.200° ($CHCl_3$, c=1)



Compound 13. The general procedure was followed. Bicyclic lactam **3** (0.5g, 1.92mmol), aryl fluoride **5** (0.36g, 1.92 mmol), NaH (0.1g, 2.1 mmol) and tetrahydrofuran (15mL). Flash column chromatography furnished the product (0.53g, 65%) as white solid.

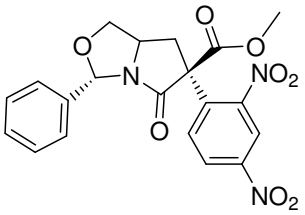
^{1}H NMR (400 MHz, $CDCl_3$) 8.99(s, 1H), 8.45-8.44(d, J = 8Hz,1H), 7.75-7.74(d, J=8.8Hz, 1H), 7.58-7.51(m, 5H), 6.43(s,1H), 4.41-4.39(m, 1H), 4.09-4.05(m, 1H), 3.96-3.92(t, J=8.4Hz, J=7.6Hz, 1H), 3.78(s, 3H), 3.46-3.42(dd, J=5.2, J = 10.4Hz, 1H), 2.58-2.52(m, 1H).

^{13}C NMR (400 MHz, $CDCl_3$) 171.9, 168.4, 148.0, 147.4, 140.7, 137.8, 131.3, 129.2, 128.7, 128.1, 125.9, 121.6, 87.94, 71.7, 66.6, 56.1, 53.8, 36.3

FTIR (KBr) 2938, 1737, 1533, 1349 cm^{-1}

HRMS calc. for $C_{20}H_{17}N_3NaO_8$ 450.1016 found 450.1016.

Specific Optical Rotation $[\alpha]^{25}_{589}$: +13.500° ($CHCl_3$, c=1)



Compound 14. The general procedure was followed. Bicyclic lactam **4** (5g, 19.16mmol), aryl fluoride **5** (3.56g, 19.16 mmol), NaH (1.01g, 21mmol) and tetrahydrofuran (75mL). Flash column chromatography furnished the product (6.5g, 80%) as white solid.

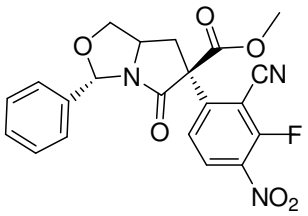
^{1}H NMR (400 MHz, $CDCl_3$) 8.98(s, 1H), 8.45-8.41(d, J = 8Hz,1H), 7.78-7.73(d, J=8.8Hz, 1H), 7.52-7.38(m, 5H), 6.41(s, 1H), 4.41-4.39(m, 1H), 4.09-4.04(m, 1H), 3.92-3.88 (t, J=8.4Hz, J=7.6Hz, 1H), 3.76(s, 3H), 3.46-3.42(dd, J=5.2, 10.4Hz, 1H), 2.58-2.52(m, 1H).

^{13}C NMR (400 MHz, $CDCl_3$) 171.9, 168.4, 148.0, 147.4, 140.7, 137.8, 131.3, 129.2, 128.7, 128.1, 125.9, 121.6, 87.94, 71.7, 66.6, 56.1, 53.8, 36.3

FTIR (KBr) 2938, 1737, 1533, 1349 cm^{-1}

HRMS calculated for $C_{20}H_{17}N_3NaO_8$ 450.1016 found 450.1008.

Specific Optical Rotation $[\alpha]^{25}_{589}$: -37.000° ($CHCl_3$, c=1)



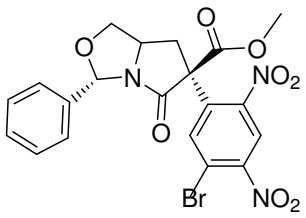
Compound 15. The general procedure was followed. Bicyclic lactam **4** (0.5g, 1.91mmol), aryl fluoride **6** (0.351g, 1.91mmol), NaH (101mg, 2.1mmol) and tetrahydrofuran 7.5 ml. Flash column chromatography furnished the product (0.56g, 69%) as light yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$) 8.27(t, J=7.6, 8.4Hz, 1H), 7.47(d, J=6.4, 2H), 7.43(d, J=6, 1H), 7.41-7.26(m, 3H), 6.43(s, 1H), 4.37(t, J=6.8, 7.6Hz, 1H), 4.08-4.03(m, 1H), 3.9(t, J=7.2,7.2Hz, 1H), 3.8(s, 3H), 3.44(dd, J=5.6, 14.4, 1H), 2.76-2.71(m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) 170.6, 168.0, 158.7, 156.0, 150.6, 137.5, 136.7, 130.3, 129.2, 128.7, 125.9, 124.2, 110.9, 105.2, 87.9, 71.3, 66.5, 56.3, 54.4, 36.4.

FTIR (thin film) 2963, 2234, 1743, 1531,1349 cm^{-1}

LC-MS calc. for $C_{20}H_{16}FN_3O_8$ (M + H) 426.3, found 426.11.

HPLC purity: 99.35% using using Chiralpak AD-H column (hexane/EtOH 40:60); flow rate 0.80 mL/min; $\tau_{product}$ = 23.33 min.
Specific Optical Rotation $[\alpha]^{25}_{589}$: -11.280° ($CHCl_3$, c=1)



Compound 16. The general procedure was followed. Bicyclic lactam **4** (0.5g, 1.91mmol), aryl fluoride **7** (0.506g, 1.91mmol), NaH (101mg, 2.1mmol) and tetrahydrofuran 7.5 ml .Flash column chromatography furnished the product (0.57g, 59%) as light yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$) 8.67(s, 1H), 7.92(s, 1H), 7.38-7.52(m, 5H), 6.44(s, 1H), 4.38(t, J=6.4, J=7.6Hz, 1H), 4.11-4.07(m, 1H), 3.91(t, J=8.4, J=8.0Hz, 1H), 3.77(s, 3H), 3.43(m, 1H), 2.57-2.51(m, 1H)

^{13}C NMR (100 MHz, $CDCl_3$) 171.1, 168.0, 148.5, 146.4, 139.1, 137.6, 136.9, 129.1, 128.7, 125.8, 123.6, 121.3, 87.6, 71.5, 66.2, 59.9, 53.8, 36.3.

FTIR (thin film) 2993, 1737, 1527, 700 cm^{-1}

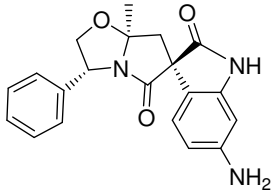
LC-MS calc. for $C_{20}H_{16}BrN_3O_8$ (M- H) 505.2, found 442.1.

HPLC purity: 99.32% using using Eclipse XDB-C8 column (0.01M Ammonium Acetate/ACN 0:30, 4/70, 15/95, 15.1/30); flow rate 0.80 mL/min; $\tau_{product}$ = 7.67 min.

Specific Optical Rotation $[\alpha]^{25}_{589}$: -8.650° ($CHCl_3$, c=1)

IV. Formation of spiropyrrolidoneoxindole scaffold

General procedure for the synthesis of spiropyrrolidoneoxindole scaffold. A flask was charged with the substrate, it was then evacuated with argon three times. The appropriate volume of tetrahydrofuran was added, followed by ammonium formate and Pd-C (10% w/w). The solution was stirred at reflux for 12h. Once TLC indicates complete consumption of the starting material, the reaction was exposed to air and was filtered through a bed of celite. The filtrate was diluted with ethyl acetate and was washed with water. The organic layer was dried over anhydrous magnesium sulfate and was evaporated under vaccum to obtain the crude product, which was purified by flash column chromatography (2-3 % methanol in DCM), to generate the desired compound as off white solid.



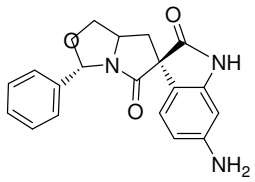
Compound 17. The general procedure was followed. Bicyclic lactam **8** (200mg, 0.45mmol), ammonium formate (200mg, 2.27mmol), 10% (w/w) Pd-C (50mg) and THF (5mL). Flash column chromatography furnished the product (100mg, 63%) as white solid.

^{1}H NMR (400 MHz, DMSO-D_6) 10.43 (s, 1H), 7.41-7.37 (m, 2H), 7.31-7.23 (m, 3H), 7.13-7.11 (d, J = 8Hz, 1H), 6.23-6.21 (m, 1H), 6.17-6.16 (d, J = 4Hz, 1H), 5.29 (m, 2H), 5.10-5.07 (t, J = 8Hz, J = 8Hz, 1H), 4.82-4.77 (t, J = 8Hz, J = 8Hz, 1H), 4.02-3.96 (m, 1H), 3.30 (s, 3H), 2.78-2.75 (d, J = 12Hz, 1H), 2.40-2.37 (d, J = 12Hz, 1H), 1.65 (m, 3H).

^{13}C NMR (100 MHz, DMSO-D_6) 176.9, 176.4, 150.4, 143.7, 140.5, 129.1, 127.8, 125.8, 123.9, 117.4, 107.5, 98.5, 96.6, 73.3, 61.4, 60.1, 43.9, 25.1.

FTIR (thin film) 3045, 2981, 1680, 1493 cm^{-1}

HRMS calc. for $C_{20}H_{19}N_3NaO_3$ 372.1426, found 372.1422



Compound 18. The general procedure was followed. Bicyclic lactam **14** (1g, 2.34 mmol), ammonium formate (913mg, 11.7mmol), 10% (w/w) Pd-C (200mg) and tetrahydrofuran (30mL). Flash column chromatography furnished the product (490mg, 62%) as white solid.

^{1}H NMR (400 MHz, $CDCl_3$) 10.4(s, 1H), 7.44-7.36(m, 5H), 6.98(d, J=8.4,1H), 6.16-6.14(d, J=9.2Hz, 2H), 6.11(s, 1H), 5.26(s, 2H), 4.54-4.47(m, 1H), 4.31(t, J=7.8Hz,J=6.4Hz, 1H), 3.67(t, J=8.0, J=8.4Hz, 1H), 2.54-2.44(m, 2H).

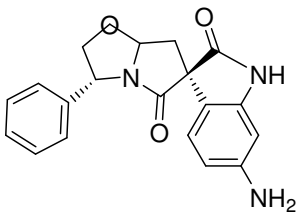
^{13}C NMR (100 MHz, DMSO-D_6) 176.4, 174.4, 149.8, 143.0, 138.7, 128.6, 128.4, 126.1, 123.2, 116.8, 106.9, 95.9, 86.7, 71.5, 61.3, 56.7, 33.4.

FTIR (KBr) 3454, 2937,1729, 1395, 1166 cm^{-1}

LC-MS calc. for $C_{19}H_{17}N_3O_3$ (M + H) 336.3, found 334.3 as M-H

HPLC purity: 98.18% using using Eclipse XDB-C8 column (0.01M Ammonium Acetate/ACN 0:30, 4/70, 15/95, 15.1/30); flow rate 0.80 mL/min; $\tau_{product}$ = 4.62 min.

Specific Optical Rotation $[\alpha]^{25}_{589}$: +199.600° ($CHCl_3$, c=1)



Compound 19. The general procedure was followed. **10** (6 g, 0.45mmol), ammonium formate (200 mg, 2.27 mmol), $Pd(OH)_2$ (600 mg, 10% w/w) and THF (120 mL). Flash column chromatography furnished the product **19** (4.6 g, 63 %) as off white solid.

^{1}H NMR (400 MHz, DMSO-D_6) 8.77 (s, 1H), 7.35-7.33 (m, 2H), 7.29-7.25 (m, 3H), 6.93-6.91 (d, J = 7.58 Hz, 1H), 6.25-6.22 (m, 1H), 6.14 (s, 1H), 5.61-5.86 (dd, J = 6.16 Hz, J = 3.85 Hz,1H), 5.21-5.17 (m, 1H), 4.73-4.68 (dd, J =

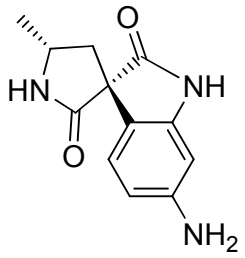
8.88 Hz, J = 7.74 Hz, 1H), 3.99-3.95 (dd, J = 8.8 Hz, J = 7.1 Hz, 1H), 2.86-2.81 (dd, J = 14.04 Hz, J = 4.29 Hz, 1H), 2.64-2.59 (d, J = 13.87 Hz, 6.27 Hz, 1H).

^{13}C NMR (100 MHz, DMSO-D_6): 174.2, 169.5, 150.2, 143.17, 139.5, 128.7, 127.6, 126.0, 123.2, 112.1, 107.3, 93.9, 90.5, 75.8, 60.9, 59.2, 57.2, 54.9, 25.1.

FTIR (thin film) : 3416, 3106, 1742, 1584, 1531,1338 cm^{-1}

LC-MS calc. for $C_{20}H_{19}N_3O_3$ (M + H) 336.36, found 336.36.

Specific Optical Rotation $[\alpha]^{25}_{589}$: +55.800° ($CHCl_3$, c=1)



Compound 20. Compound **17** (300mg, 0.91 mmol) was dissolved in dichloromethane (7mL) under nitrogen atmosphere and was cooled to -78°C. Titanium chloride (1mL, 0.91mmol) was added to the solution and the resulting mixture was stirred for 10-15minutes. Triethylsilane (0.1mL, 0.3 mmol) was then added to the reaction mixture, which was then stirred for another 30minutes. The mixture was then allowed to warm to rt over night. Once TLC confirms total consumption of starting material the reaction was quenched with ammonium chloride solution (10mL), extracted with dichloromethane (3 X 10), dried over anhydrous magnesium sulfate, filtered and evaporated under reduced pressure to yield the crude product which was directly taken to the next step.

The crude alcohol was dissolved in dichloromethane and was treated with PPh_3 (240mg, 0.9mmol), followed by CCl_4 (146mg, 0.95mmol) and the resulting mixture was refluxed until TLC confirms total consumption of starting alcohol. The reaction mixture was then cooled to rt, quenched with water and extracted with ethyl acetate (10 X 2). The organic layer was dried over magnesium sulfate, filtered and evaporated under reduced pressure to the yield the crude compound. It was taken to the next step without any purification.

The chloride was dissolved in dioxane (5mL) and treated with DBU (275mg, 0.9mmol). The resulting mixture was refluxed for about 4h, after which TLC confirmed the complete consumption of starting material. The reaction was cooled to rt, quenched with water and the mixture extracted with dichloromethane (10mL). The organic layer was dried with anh. magnesium sulfate, filtered and evaporated under reduced pressure to yield the intermediate enamide, which was immediately taken to the final step, due to the sensitive nature of the compound.

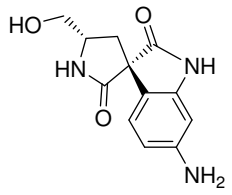
The enamide was dissolved in 1:1 mixture of methanol and 6M HCl (20mL). It was allowed to stir for 4h at about 50°C and then was allowed to cool to rt. It was then concentrated under reduced pressure and was extracted with dichloromethane (10mL x 3). The combined organic layers were then washed with saturated sodium bicarbonate solution, dried and concentrated under reduced pressure, to yield the crude compound, which was then purified by flash column chromatography (2→10% DCM-MeOH) to generate the desired compound (76mg, 30%).

^{1}H NMR (400 MHz, DMSO-D_6) 10.50 (s, 1H), 6.75-6.76 (d, J = 8Hz, 1H), 6.23-6.17 (m, 2H), 4.18-4.16 (m, 1H), 2.32-2.27 (m, 1H), 2.03-1.99 (m, 1H), 1.05-1.03 (d, J = 7.96Hz, 3H).

^{13}C NMR (100 MHz, DMSO-D_6) 177.9, 172.4, 150.0, 140.0, 128.7, 118.9, 107.5, 107.2, 58.9, 57.9, 49.1, 38.2, 22.5

FTIR (thin film): 3151, 2954, 1745, 1680, 1599 cm^{-1}

HRMS calc. for $C_{20}H_{19}N_3NaO_3$ 256.1008, found 256.1002



Compound 21. To a solution of compound **18** (200 mg, 0.6mmol) in dry dichloromethane(5ml) was added 1 ml of trifluoro acetic acid at room temparature. The reaction mass was stirred for 3 h and checked TLC (10 methanol in DCM). After the reaction was completed the reaction mass was concentrated under vaccum and the residue was triturated with diethyl ether to get pure compound **21** (103mg, 70%) as off white solid. ^{1}H NMR (400 MHz, DMSO-D6) 10.33(s, 1H), 8.18(s, 1H), 6.96(d, J=7.6, 1H), 6.33-6.29(m, 2H), 3.81-3.79(m, 1H), 3.5(d, J=5.6, 2H), 2.23(d, J=6.8, 2H), 1.99(s, 1H). ^{13}C NMR (100 MHz, DMSO-D_6) 177.4, 172.8, 143.4, 123.3, 122.3, 109.6, 98.5, 64.8, 59.7, 57.5, 53.2, 33.8, 14.0. FTIR (KBr) 3224, 2920, 1719 cm^{-1}. HRMS calc. for $C_{12}H_{13}N_3NaO_3$ 270.0957, found 270.0824. Specific Optical Rotation $[\alpha]^{25}_{589}$: +15.280° ($CHCl_3$, c=1)

1. a) L. B. Burgess, A. I. Meyers, *J. Am. Chem. Soc*. **1991**, *113*, 9858-9859; b) M. Penhoat, S. Leleu, G. Dupass, C. Papamicaël, F. Marsais, V. Levacher, *Tetlett*. **2005**, *46*, 8385-8389.
2. M. D. Groaning, A. I. Meyers, *Tetlett*. **1999**, *40*, 4639-4642.
3. M. J. Beard, J. H. Bailey, D. T. Cherry, M. G. Moloney; S. B. Shim, *Tetrahedron* ,**1996**, *52(10)*, 3719 – 3740.
4. Y. Hamada, O. Hara, A. Kawai, K. Yasushi, T. Shioiri, *Tetrahedron*, **1991** , *47(40)*, 8635 - 8652

Spectra:
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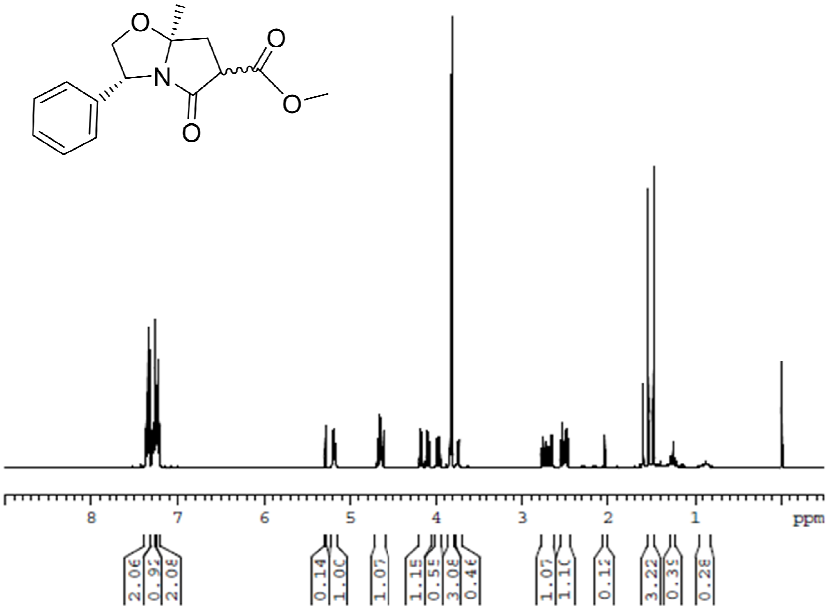

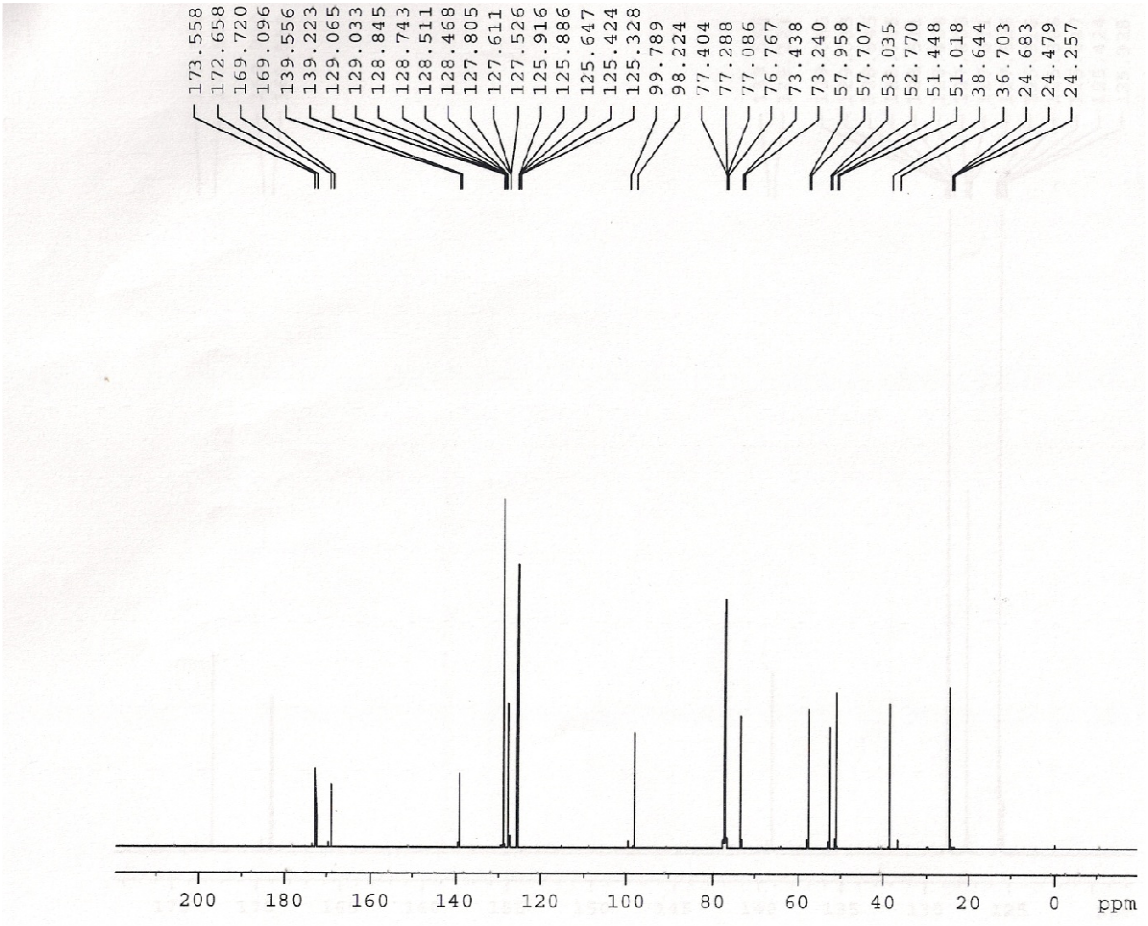
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139.223
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129.033
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127.805
127.611
127.526
125.916
125.886
125.647
125.424
125.328
99.789
98.224
77.404
77.288
77.086
76.767
73.438
73.240
57.958
57.707
53.035
52.770
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51.018
38.644
36.703
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24.257
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160
140
120
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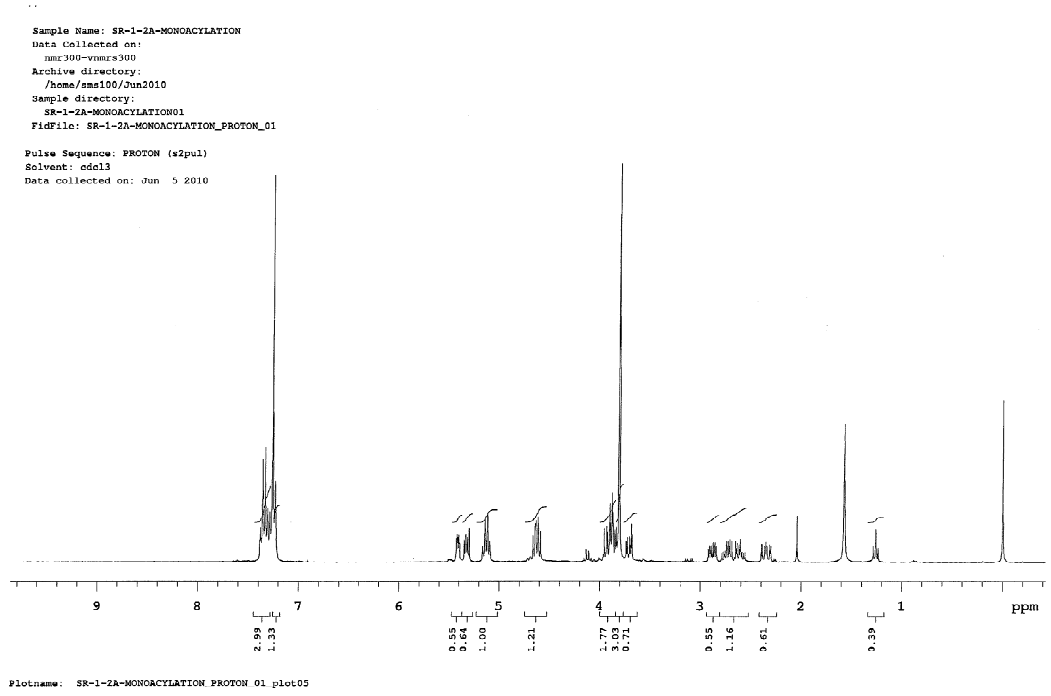
80
60
40
20
0
ppm

Compound **2**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Sample directory:
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Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 5 2010
9
8
7
6
5
4
3
2
1
ppm
2.99
1.33
0.55
0.64
1.00
1.21
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3.03
0.71
0.55
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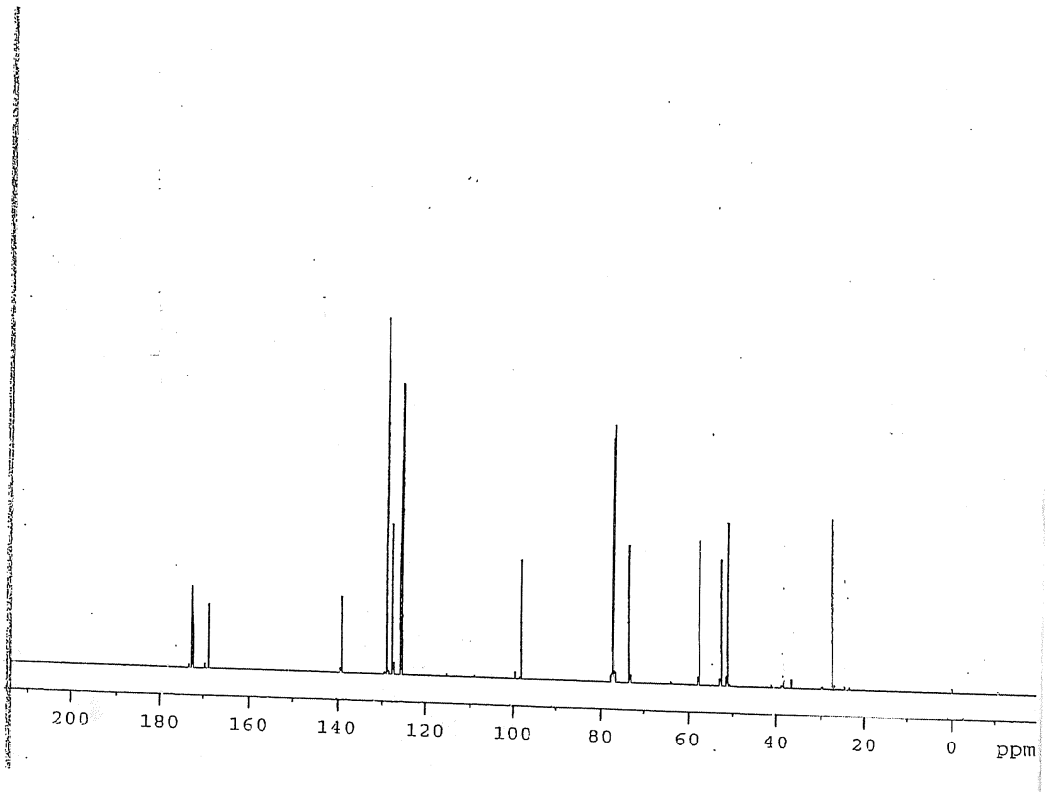
0.61
0.39
Plotname: SR-1-2A-MONOACYLATION_PROTON_01_plot05


200
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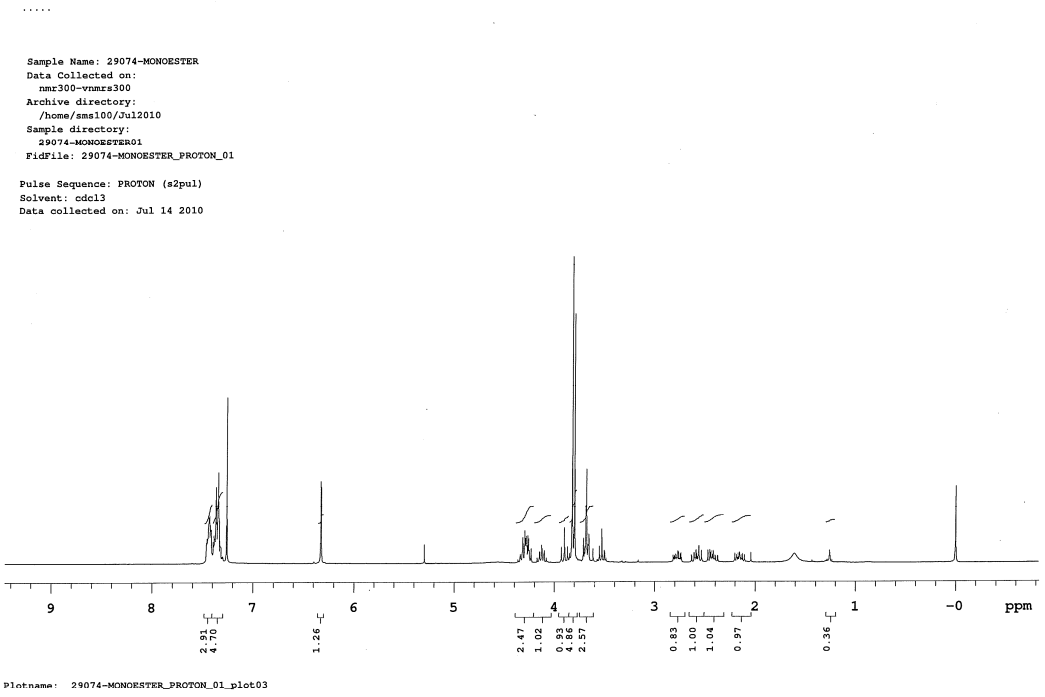
160
140
120
100
80
60
40
20
0
ppm

Compound 3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Archive directory:
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Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 14 2010
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8
7
6
5
4
3
2
1
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ppm
2.91
4.70
1.26
2.47
1.02
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0.36
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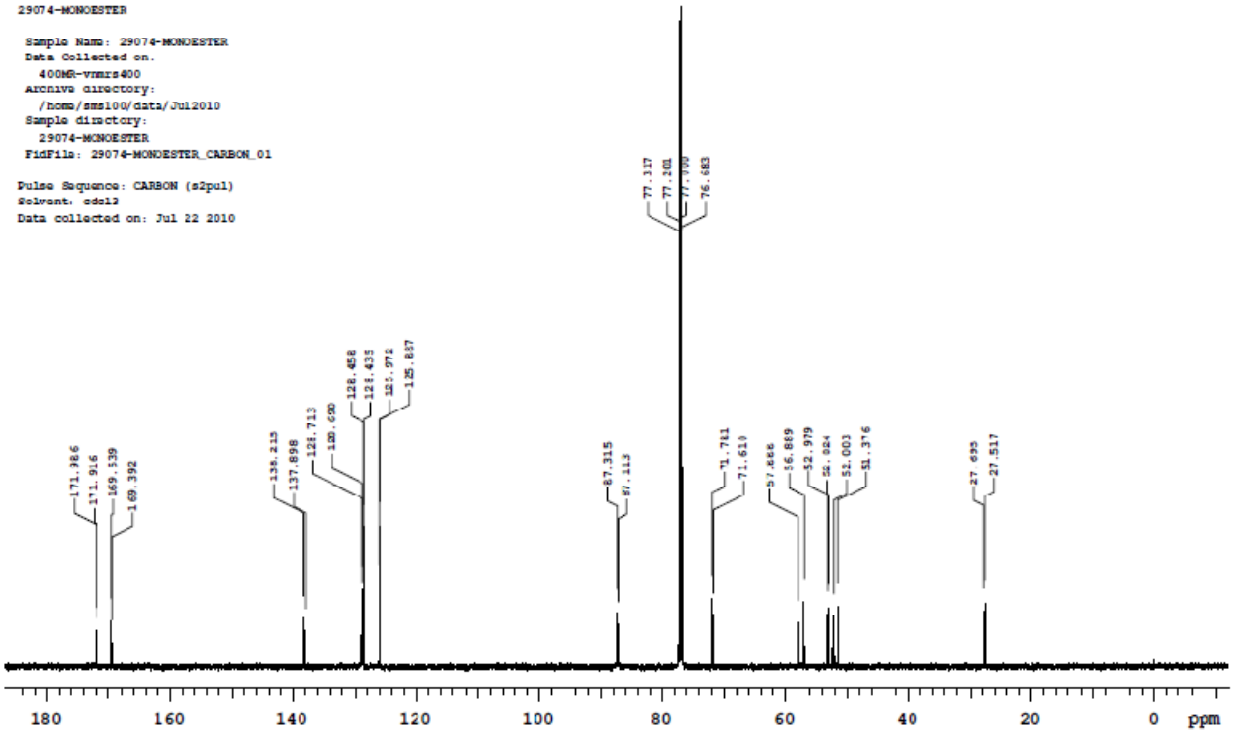
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Data Collected on.
400MR-vnmrs400
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Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 22 2010
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169.392
138.215
137.898
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128.458
128.433
125.857
87.315
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52.979
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51.376
27.693
27.517
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77.000
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160
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120
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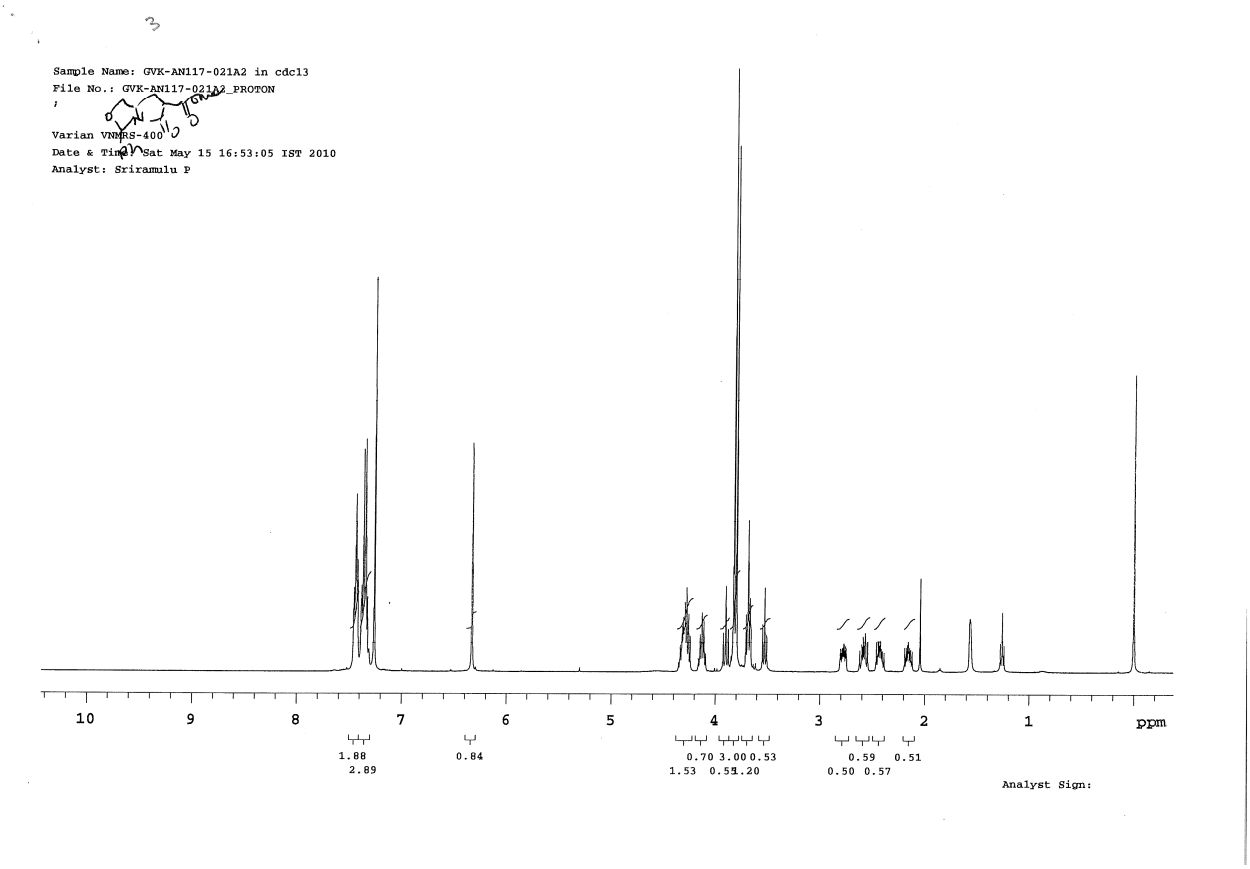
80
60
40
20
0
ppm

Compound **4**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Varian VNMRS-400
Date & Time Sat May 15 16:53:05 IST 2010
Analyst: Sriramulu P
10
9
8
7
6
5
4
3
2
1
ppm
1.88
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0.53
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0.50
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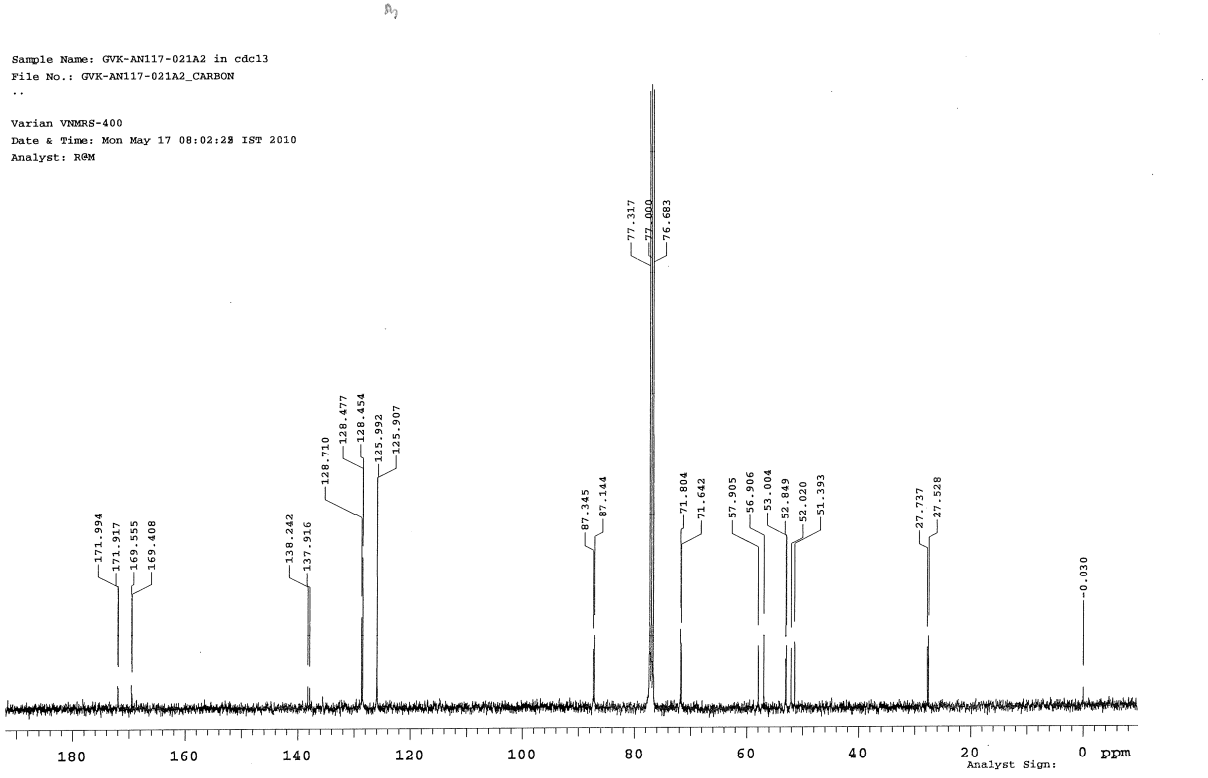
0.57
0.51
Analyst Sign:


Sample Name: GVK-AN117-021A2 in cdcl3
File No.: GVK-AN117-021A2_CARBON
..
Varian VNMRS-400
Date & Time: Mon May 17 08:02:28 IST 2010
Analyst: R@M
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171.917
169.555
169.408
138.242
137.916
128.710
128.477
128.454
125.992
125.907
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77.000
76.683
71.804
71.642
57.905
56.906
53.004
52.849
52.020
51.393
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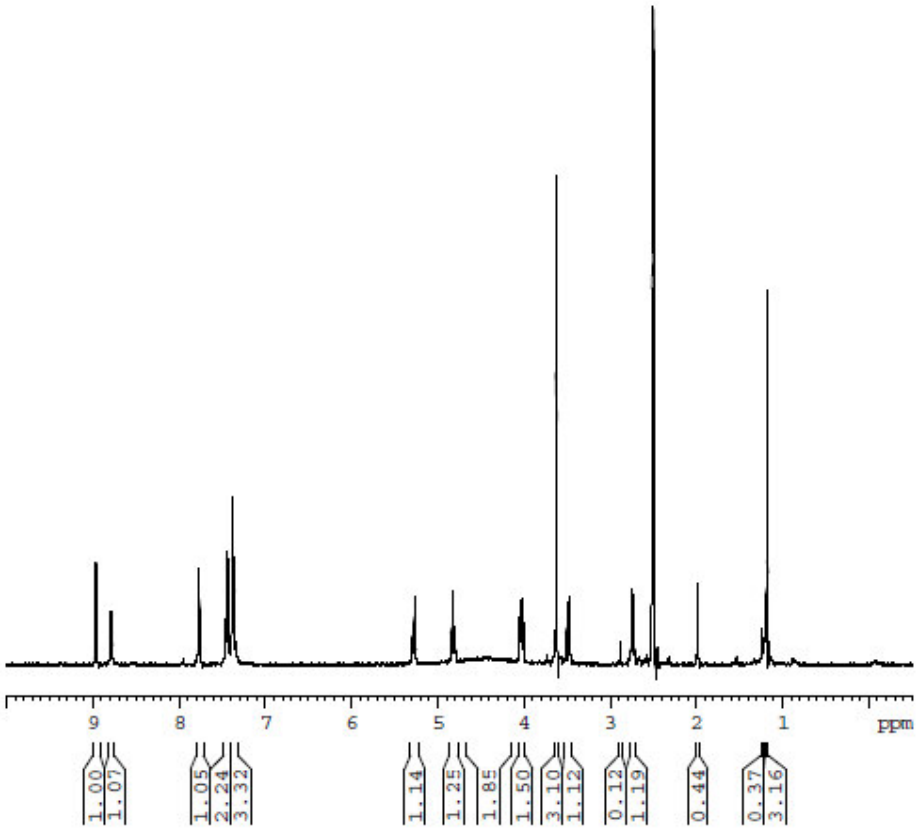
-0.030
180
160
140
120
100
80
60
40
20
0 ppm
Analyst Sign:

Compound **8**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7
6
5
4
3
2
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1.07
1.05
2.24
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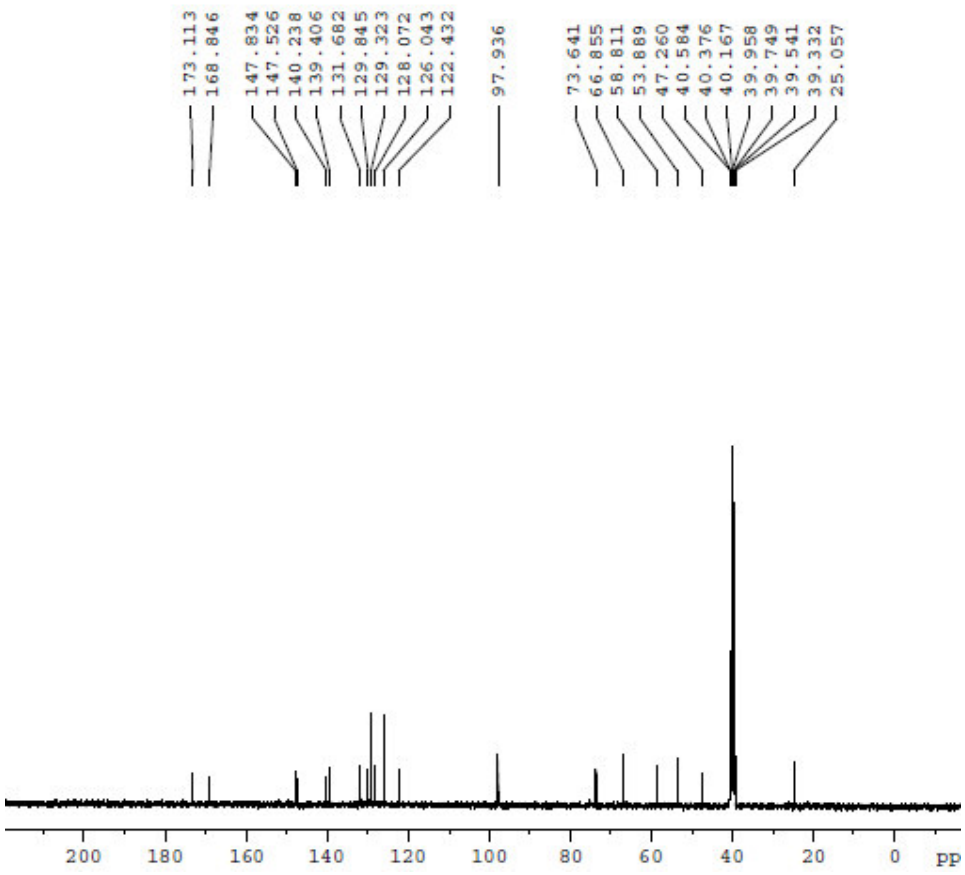
0.12
1.19
0.44
0.37
3.16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73.641
66.855
58.811
53.889
47.260
40.584
40.376
40.167
39.958
39.749
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80
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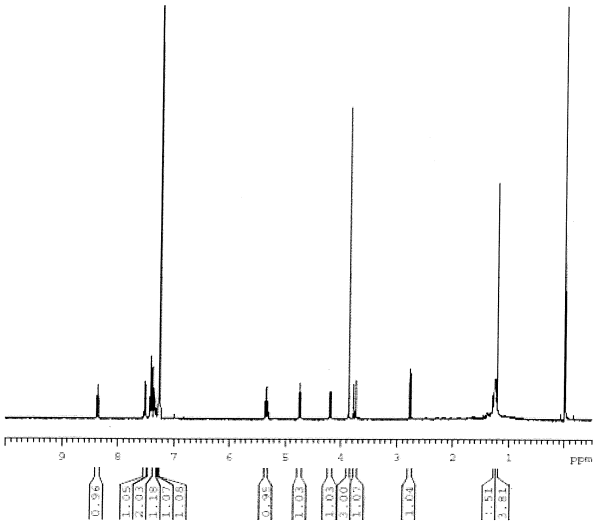
20
0
pp

Compound **9**


9
9
8
7
6
5
4
3
2
1
ppm
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1.05
2.03
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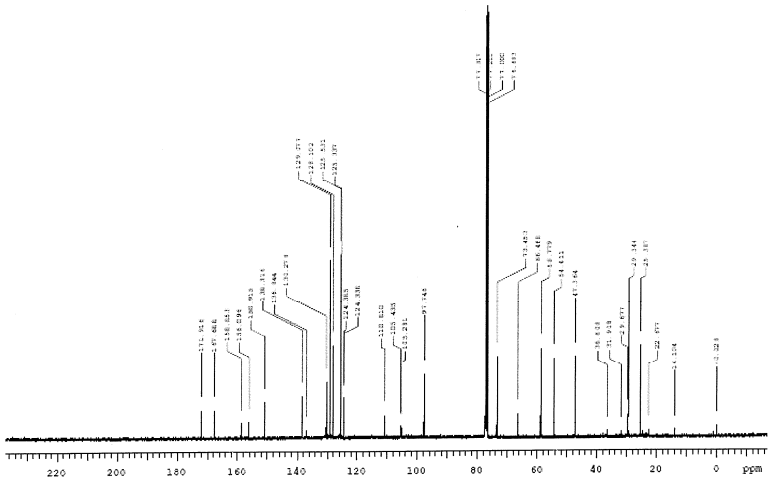
1.01
1.06
0.94
1.03
1.03
2.00
1.07
1.04
1.51
2.84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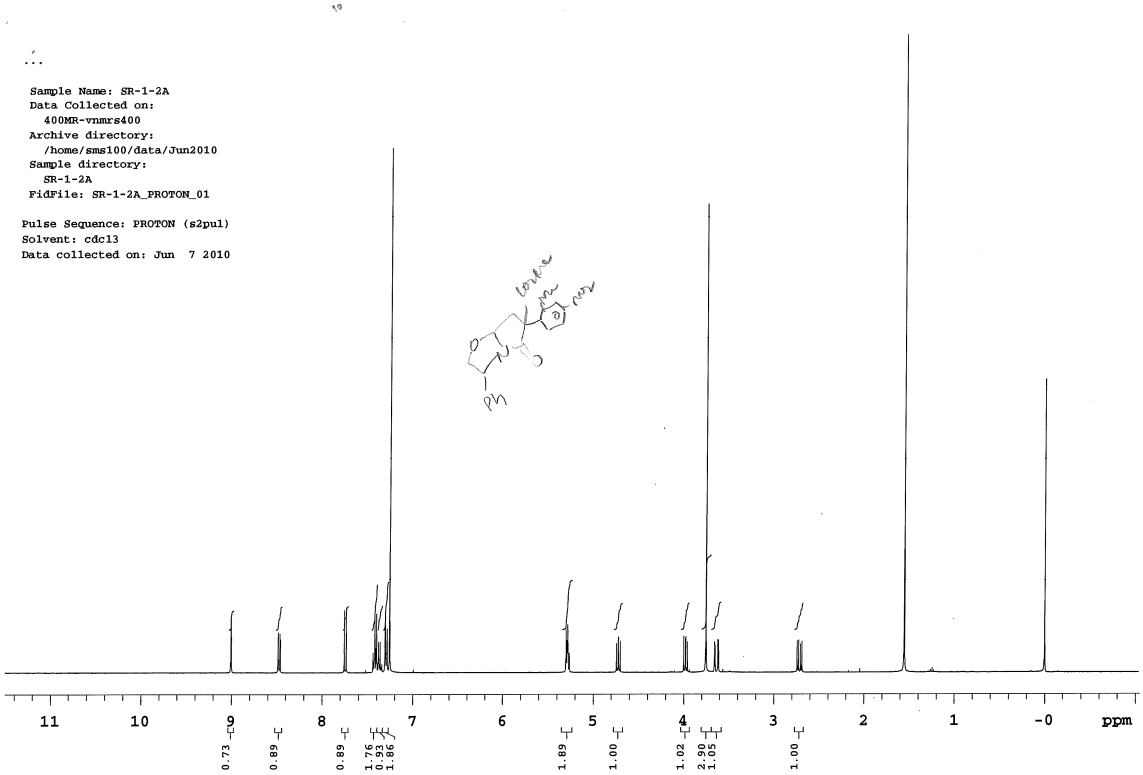
80
60
40
20
0
ppm

Compound **10**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Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 7 2010
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
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1.76
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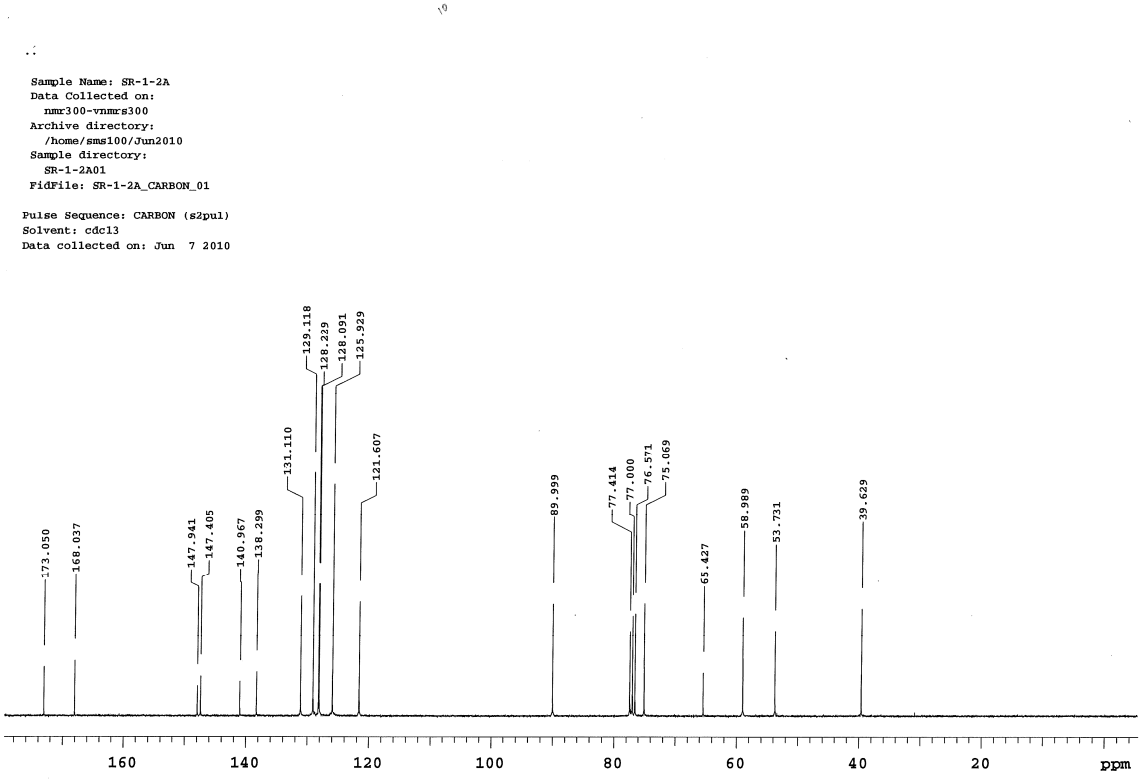
1.86
1.89
1.00
1.02
2.90
1.05
1.00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Archive directory:
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Sample directory:
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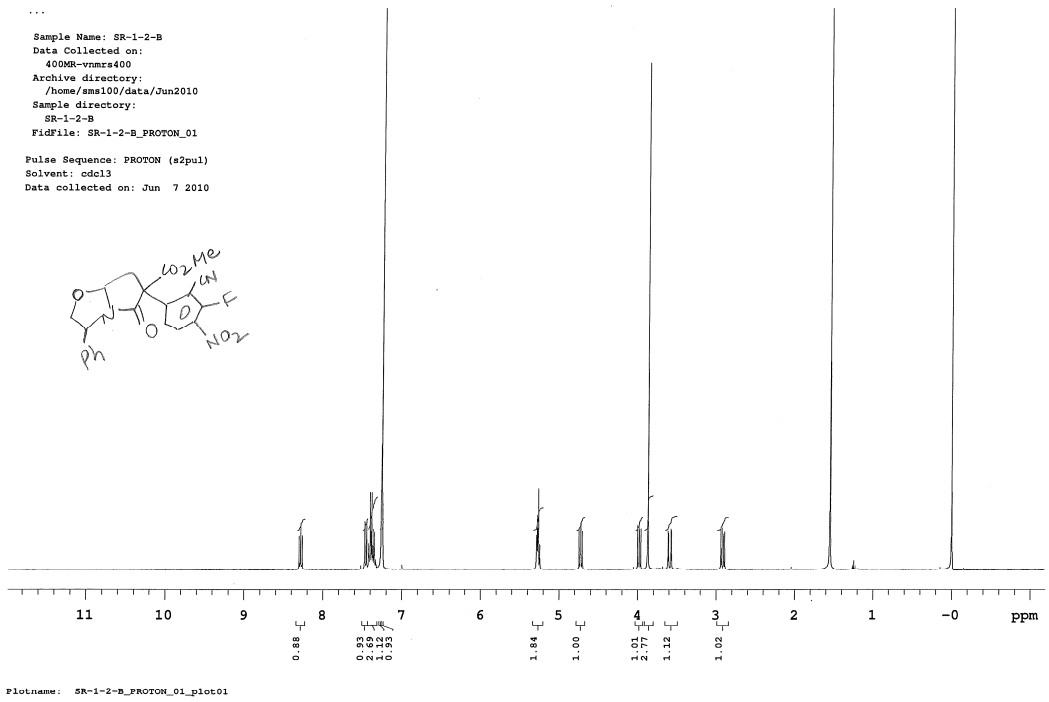
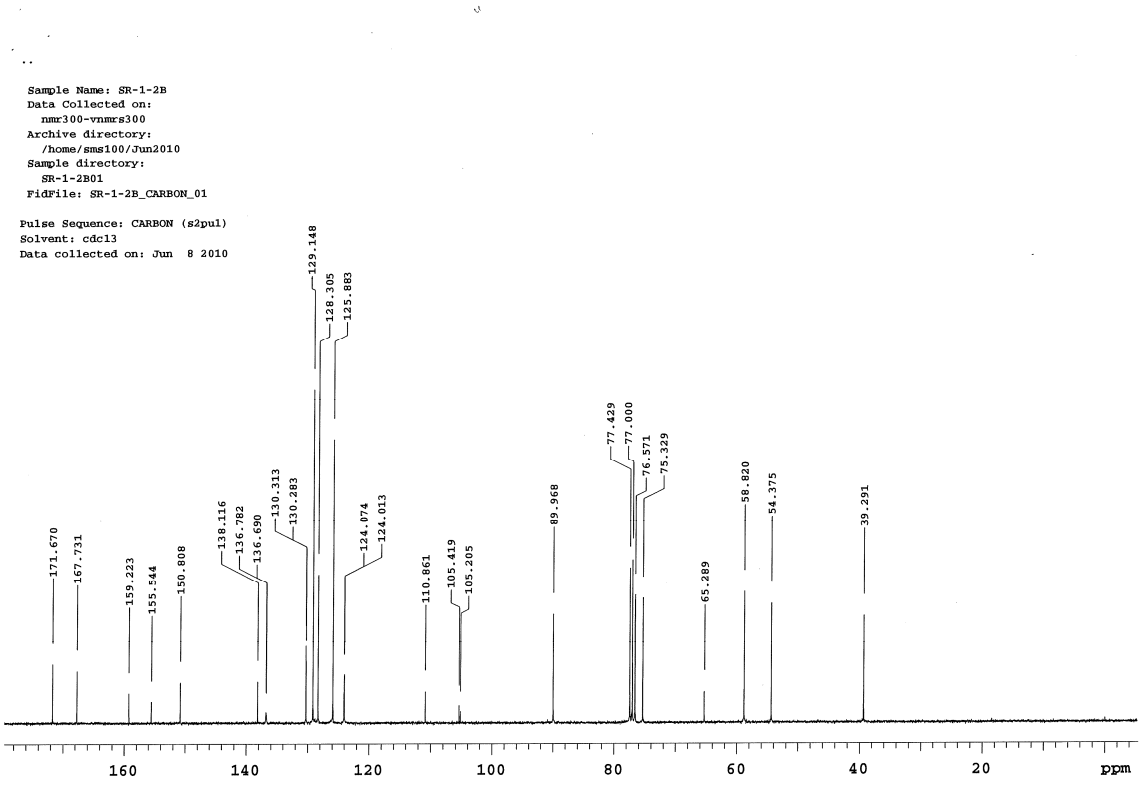
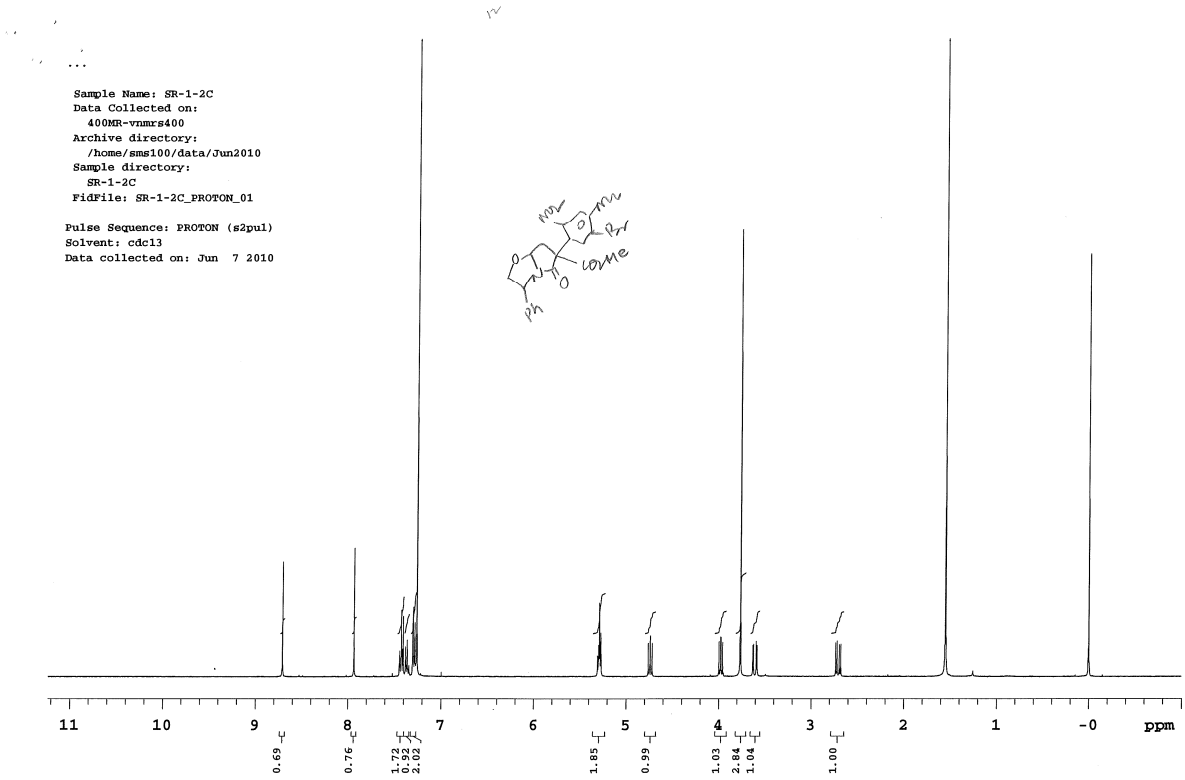
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 7 2010
173.050
168.037
147.941
147.405
140.967
138.299
131.110
129.118
128.229
128.091
125.929
121.607
89.999
77.414
77.000
76.571
75.069
65.427
58.989
53.731
39.629
160
140
120
100
80
60
40
20
ppm

Compound **11**


Sample Name: SR-1-2-B
Data Collected on:
400MR-vnmrs400
Archive directory:
/home/sms100/data/Jun2010
Sample directory:
SR-1-2-B
FidFile: SR-1-2-B_PROTON_01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 7 2010
CO2Me
CN
F
NO2
Ph
O
O
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.88
0.93
2.85
1.12
0.93
1.84
1.00
1.01
2.77
1.12
1.02
Plotname: SR-1-2-B_PROTON_01_plot01

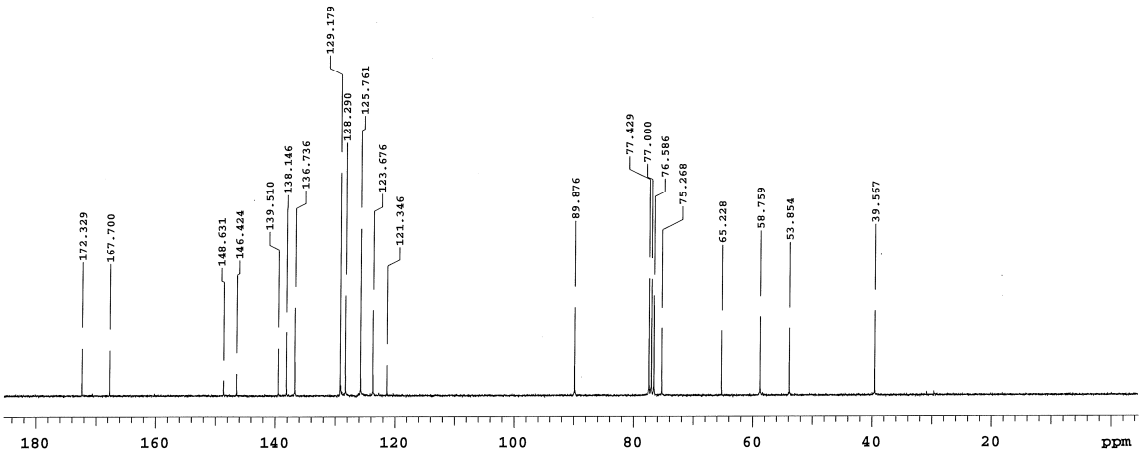

Sample Name: SR-1-2B
Data Collected on:
nmr300-vnmrs300
Archive directory:
/home/sms100/Jun2010
Sample directory:
SR-1-2B01
FidFile: SR-1-2B_CARBON_01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 8 2010
171.670
167.731
159.223
155.544
150.808
138.116
136.782
136.690
130.313
130.283
129.148
128.305
125.883
124.074
124.013
110.861
105.419
105.205
89.968
77.429
77.000
76.571
75.329
65.289
58.820
54.375
39.291
160
140
120
100
80
60
40
20
ppm

Compound **12**

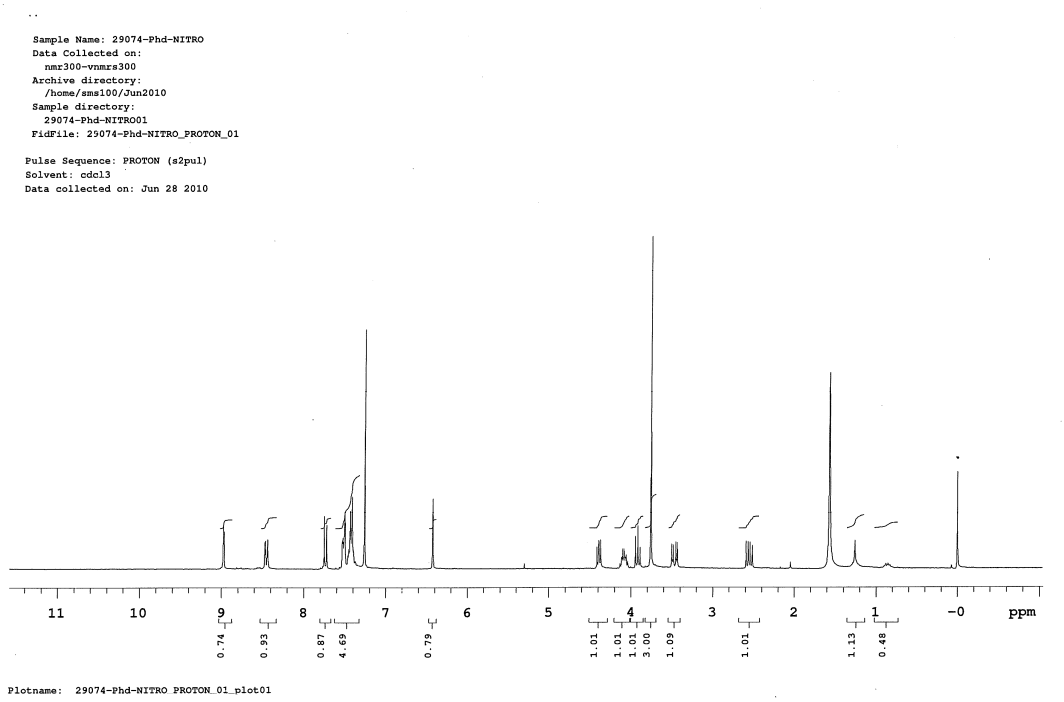

Sample Name: SR-1-2C
Data Collected on:
400MR-vnmrs400
Archive directory:
/home/sms100/data/Jun2010
Sample directory:
SR-1-2C
FidFile: SR-1-2C_PROTON_01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 7 2010
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.69
0.76
1.72
0.92
2.02
1.85
0.99
1.03
2.84
1.04
1.00

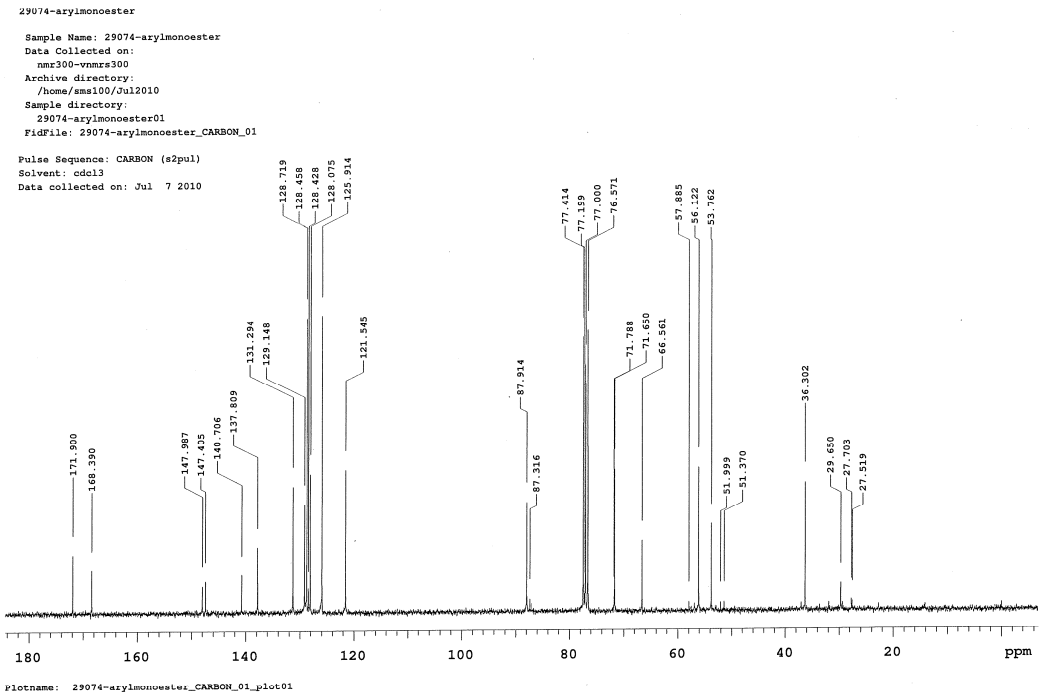
Sample Name: SR-1-2C
Data Collected on:
nmr300-vnmrs300
Archive directory:
/home/sms100/Jun2010
Sample directory:
SR-1-2C02
FidFile: SR-1-2C_CARBON_01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 8 2010

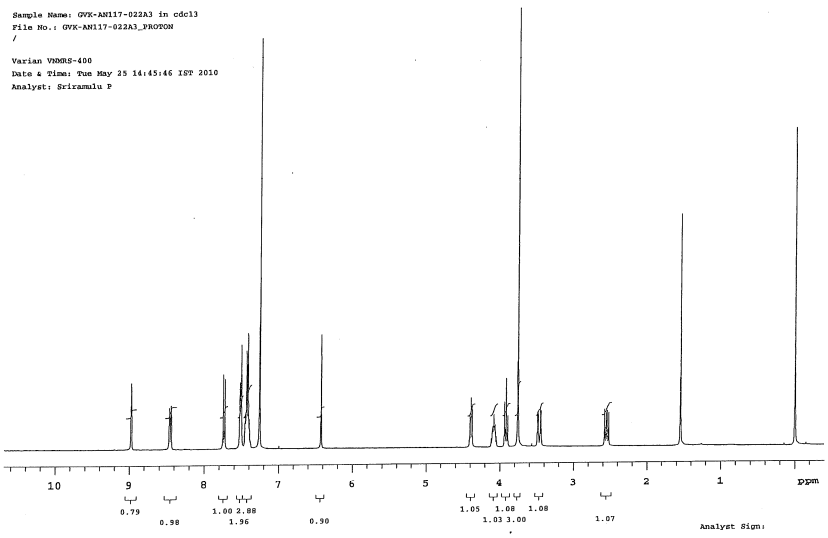


Compound **13**


Sample Name: 29074-Phd-NITRO
Data Collected on:
nmr300-vnmrs300
Archive directory:
/home/sms100/Jun2010
Sample directory:
29074-Phd-NITRO01
FidFile: 29074-Phd-NITRO_PROTON_01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 28 2010
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.74
0.93
0.87
4.69
0.79
1.01
1.01
1.01
3.00
1.09
1.01
1.13
0.48
Plotname: 29074-Phd-NITRO_PROTON_01_plot01

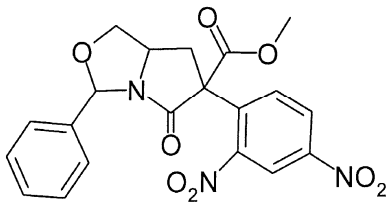

29074-arylmonoester
Sample Name: 29074-arylmonoester
Data Collected on:
nmr300-vnmrs300
Archive directory:
/home/sms100/Jul2010
Sample directory:
29074-arylmonoester01
FidFile: 29074-arylmonoester_CARBON_01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 7 2010
171.900
168.390
147.987
147.435
140.706
137.809
131.294
129.348
128.719
128.458
128.428
128.075
125.914
121.545
87.914
87.316
77.414
77.159
77.000
76.571
71.788
71.650
66.561
57.885
56.122
53.762
51.999
51.370
36.302
29.650
27.703
27.519
180
160
140
120
100
80
60
40
20
ppm
Plotname: 29074-arylmonoester_CARBON_01_plot01

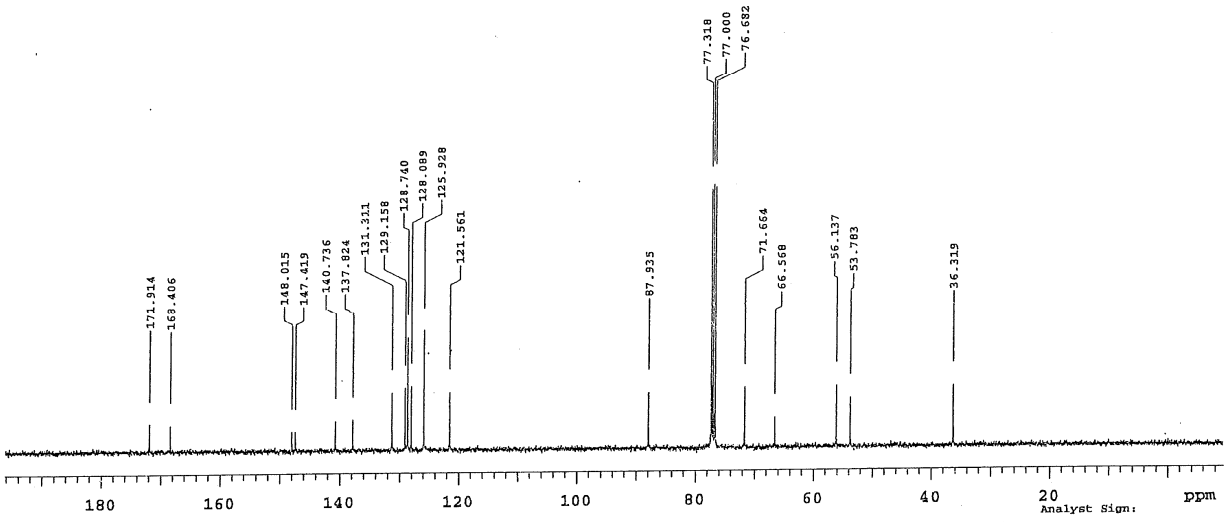
Compound **14**


Sample Name: GVK-AN117-022A3 in cdcl3
File No.: GVK-AN117-022A3_PROPON
/
Varian VNMRS-400
Date & Time: Tue May 25 14:45:46 IST 2010
Analyst: Sriramulu P
10
9
8
7
6
5
4
3
2
1
ppm
0.79
0.98
1.00 2.88
1.96
0.90
1.05
1.03 3.00
1.08
1.08
1.07
Analyst Sign:

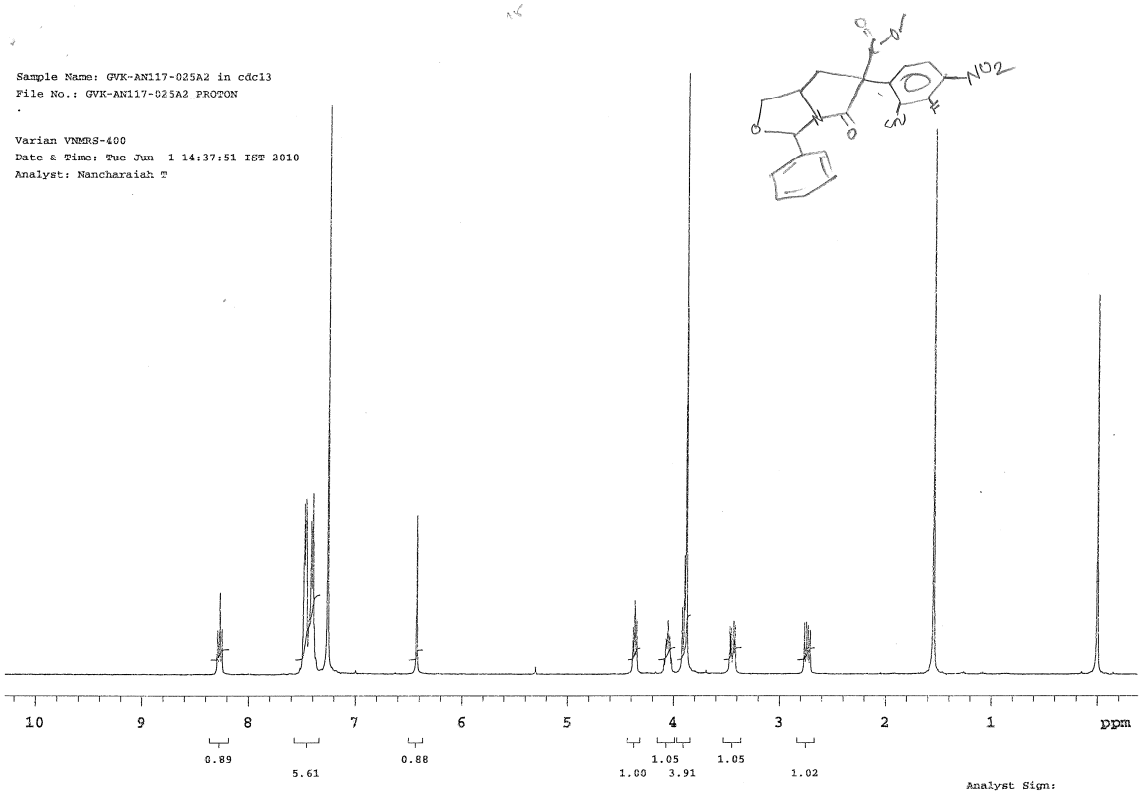
Sample: DJ110552-022A3 in cdcl3
File: DJ110552-022A3_CARBON

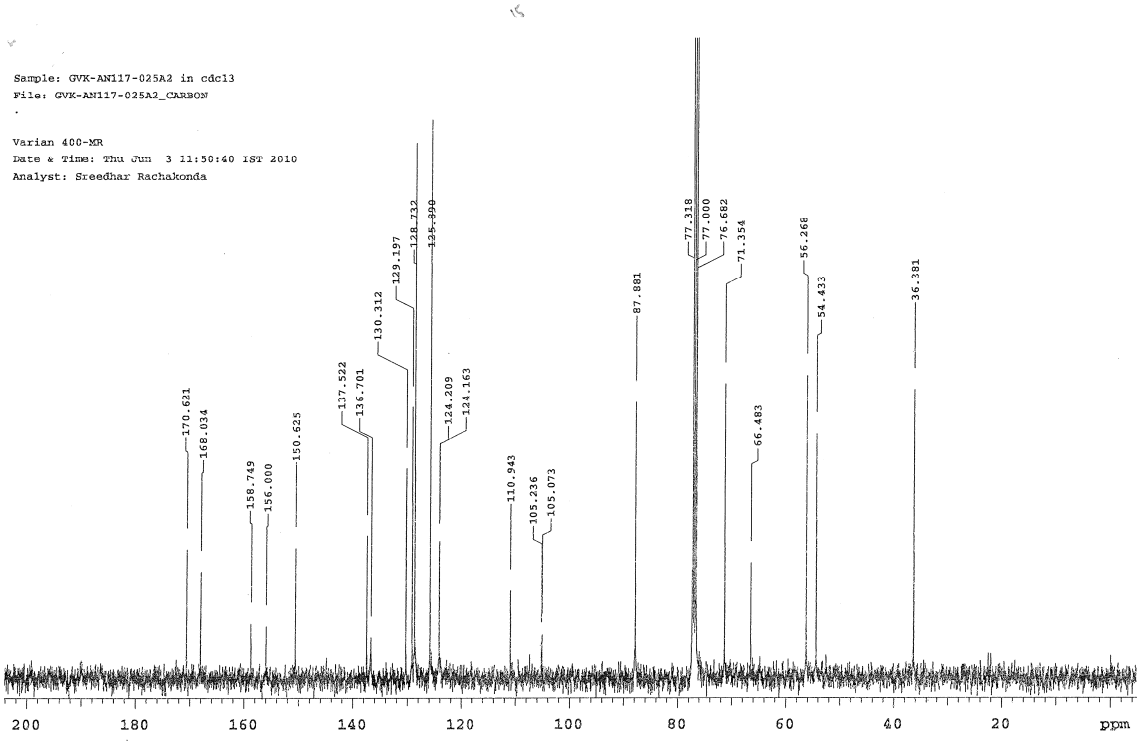
Varian 400-MR
Date & Time: Thu May 27 10:27:10 IST 2010
Analyst: Rama Krishna





Compound **15**


Sample Name: GVK-AN117-025A2 in cdcl3
File No.: GVK-AN117-025A2 PROTON
Varian VNMRS-400
Date & Time: Tue Jun 1 14:37:51 IST 2010
Analyst: Nancharaiah T
NO2
10
9
8
7
6
5
4
3
2
1
ppm
0.89
5.61
0.88
1.00
3.91
1.05
1.05
1.02
Analyst Sign:


Sample: GVK-AN117-025A2 in cdcl3
File: GVK-AN117-025A2_CARBON
Varian 400-MR
Date & Time: Thu Jun 3 11:50:40 IST 2010
Analyst: Sreedhar Rachakonda
170.621
168.034
158.749
156.000
150.625
137.522
136.701
130.312
129.197
128.732
125.390
124.209
124.163
110.943
105.236
105.073
87.881
77.318
77.000
76.682
71.354
66.483
56.268
54.433
36.381
200
180
160
140
120
100
80
60
40
20
ppm
Analyst Sign:

GVK BIO

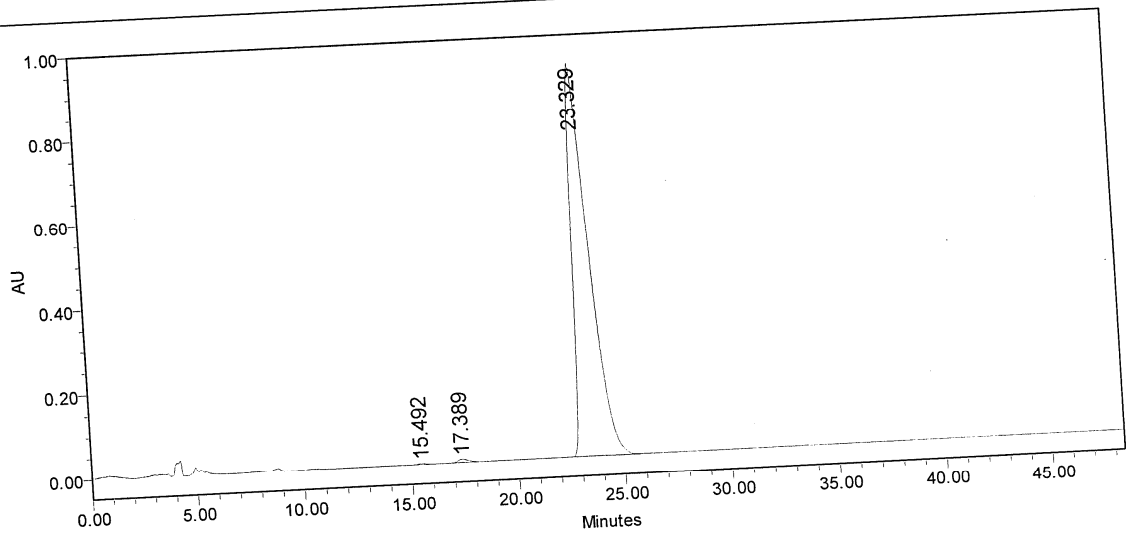
Project Name: NRM1_2010\NRM1_GSK_JUNE_10\NRM1_PDA4

GVK BIOSCIENCES (P) LTD

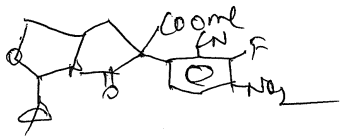
MCL Analytical-Nacharam

Sample Name:	GVK-AN117-025A2	Acquired By:	nrm12_mcl
Vial:	50	Date Acquired:	6/4/2010 8:22:26 AM IST
Injection Volume:	15.00 ul	Proc. Chnl. Descr.:	PDA MaxPlot (190.0 nm to 800.0 nm)
Run Time:	60.0 Minutes		

Column:CHIRALPAK AD-H (4.6X250mm)5µ
Mobile Phase: A: Hexane B:EtOH
A:B (40:60)
Flow Rate:0.8 ml/min
TEMP:ABIENT

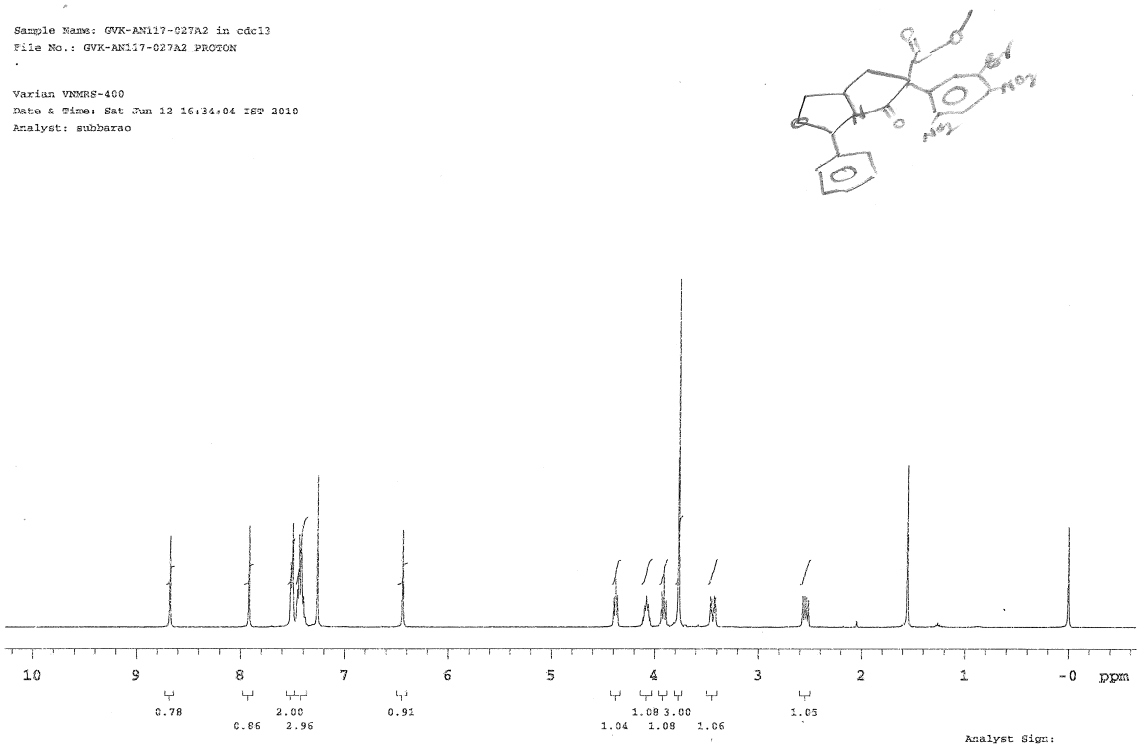


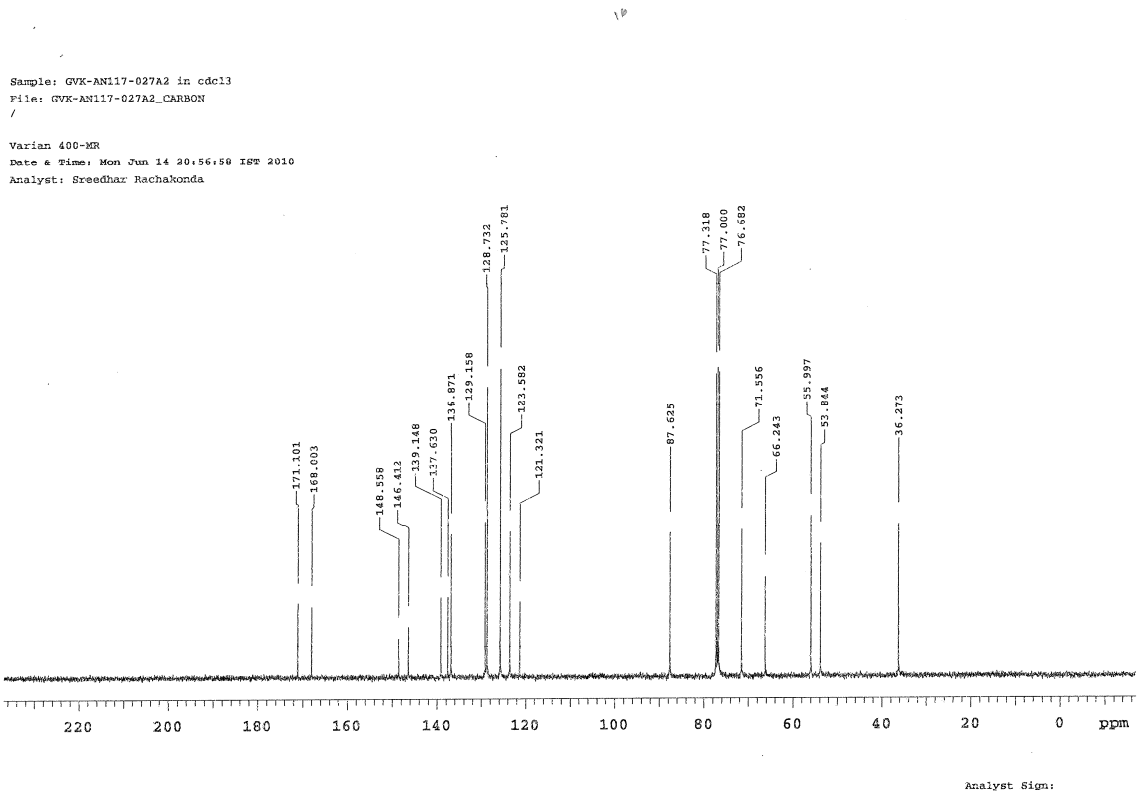
	RT	Area	% Area
1	15.49	58420	0.12
2	17.39	264906	0.53
3	23.33	49628093	99.35



Report Method Name: CHIRAL_ADH Printed 9:29:23 AM Asia/Calcutta 6/4/2010 Page: 1 of 1

Compound **16**


Sample Name: GVK-AN117-027A2 in cdcl3
File No.: GVK-AN117-027A2 PROTON
Varian VNMRS-400
Date & Time: Sat Jun 12 16:34:04 IST 2010
Analyst: subbarao
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.78
0.86
2.00
2.96
0.91
1.04
1.08
3.00
1.08
1.06
1.05
Analyst Sign:


Sample: GVK-AN117-027A2 in cdcl3
File: GVK-AN117-027A2_CARBON
/
Varian 400-MR
Date & Time: Mon Jun 14 20:56:50 IST 2010
Analyst: Sreedhar Rachakonda
171.101
168.003
148.558
146.412
139.148
137.630
136.871
129.158
128.732
125.781
123.582
121.321
87.625
77.318
77.000
76.682
71.556
66.243
55.997
53.844
36.273
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Analyst Sign:

GVK BIO

Eclipse XDBC8_DUP

Project Name: NRM1_2010\NRM1_JUNE_2010\NRM12_PDA05

GVK BIOSCIENCES (P) LTD

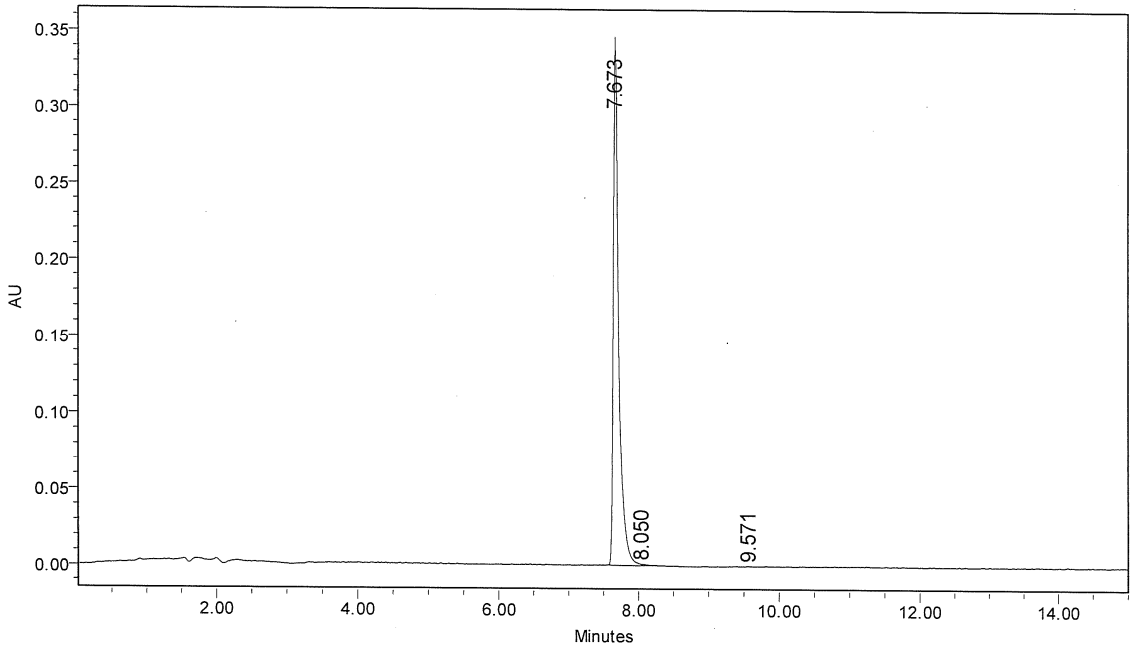
MCL Analytical-Nacharam

Sample Name:	GVK-AN117-027A2	Acquired By:	nrm12_mcl
Vial:	7	Date Acquired:	6/14/2010 1:03:28 PM IST
Injection Volume:	2.00 ul	Proc. Chnl. Descr.:	PDA MaxPlot (190.0 nm to 800.0 nm)
Run Time:	15.0 Minutes		

Chromatographic Conditions:

Column:Eclipse XDB-C8(4.6X150mm)5µ
Mobile Phase:A:0.01M Ammonium Acetate B:ACN
T/%B:0/30,4/70,6/95,15/95,15.1/30.
Flow Rate:0.8 ml/min
Temp:Ambient
Diluent:ACN

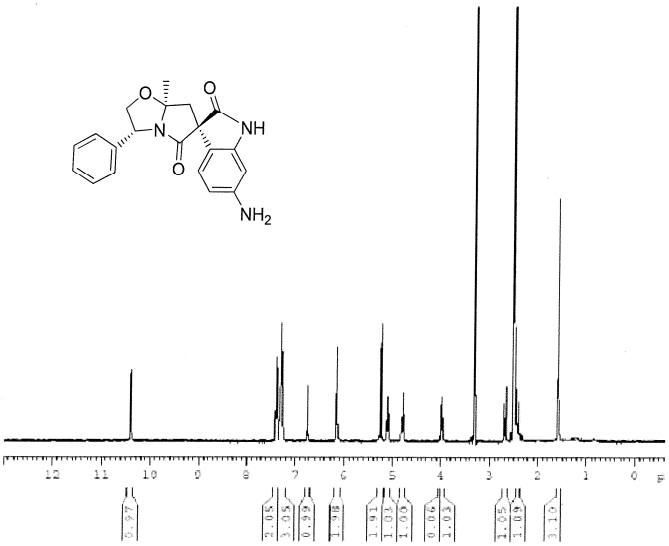
Purity Chromatogram

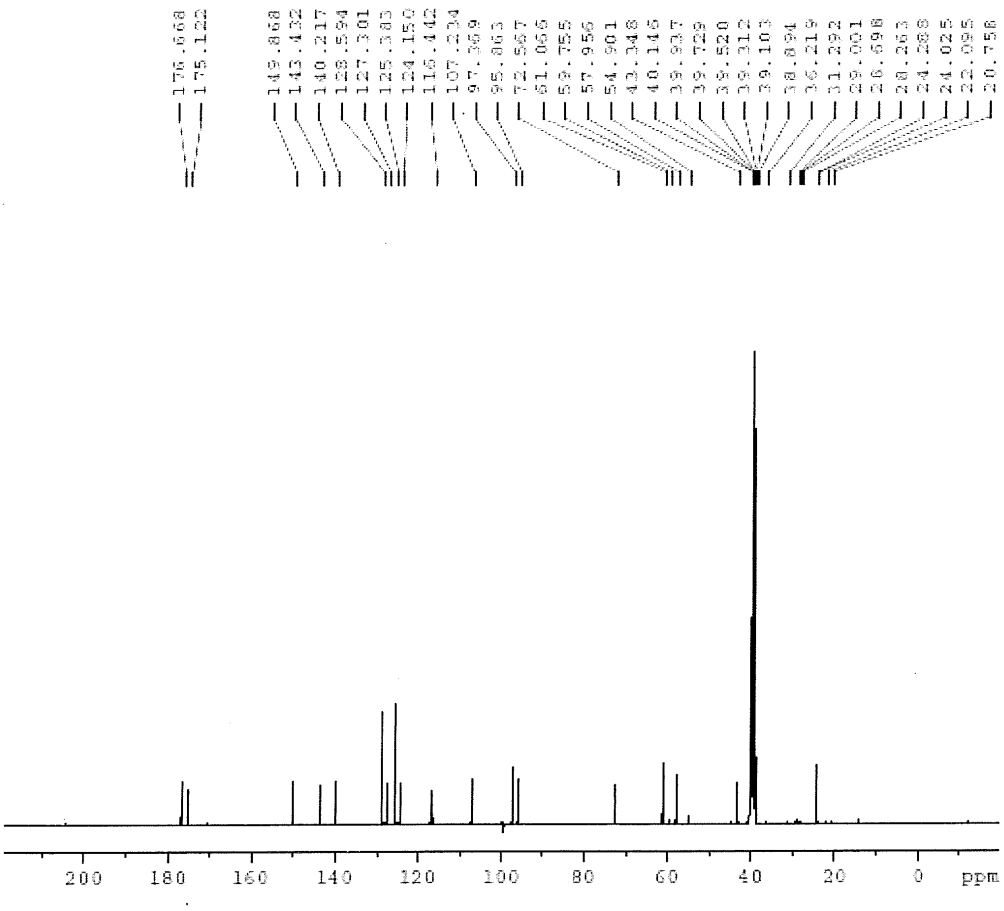


	RT	Area	% Area
1	7.67	1849174	99.32
2	8.05	8871	0.48
3	9.57	3733	0.20

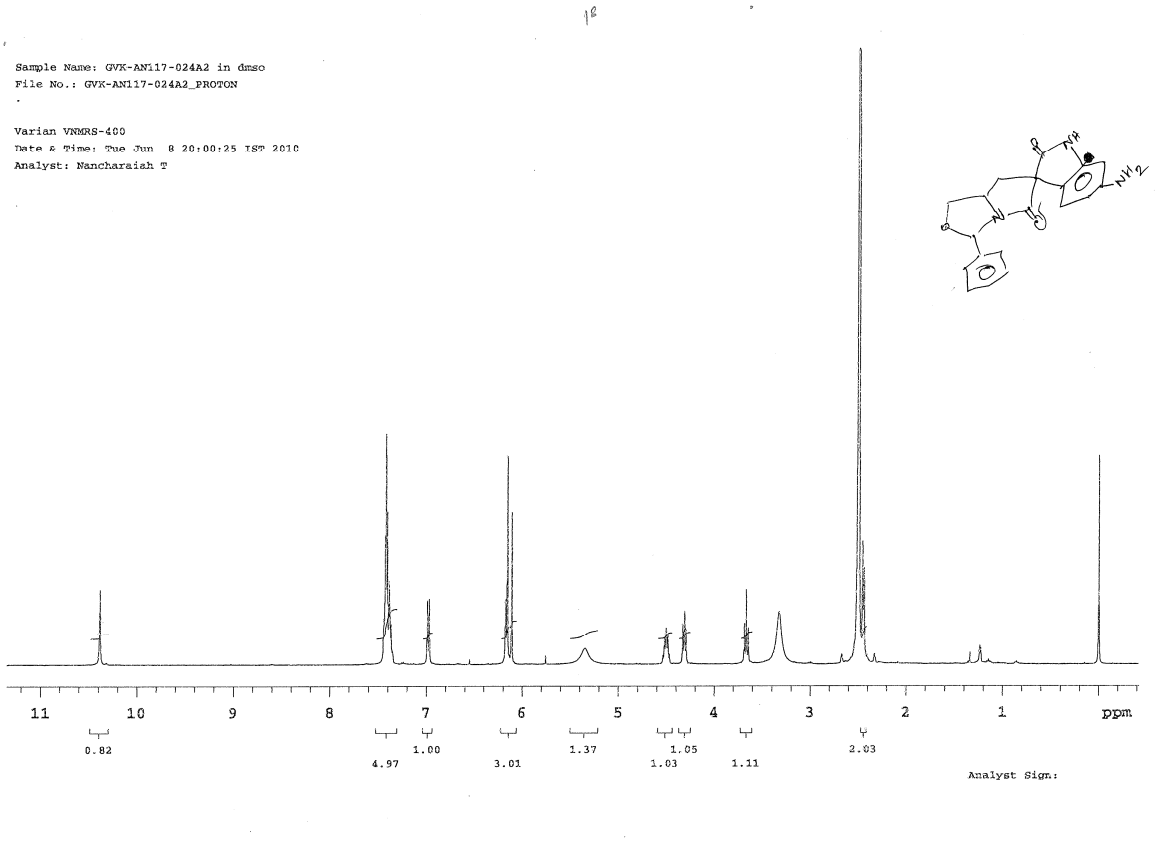
Report Method Name: Eclipse XDBC8_ Printed 1:21:16 PM Asia/Calcutta 6/14/2010 Page: 1 of 1

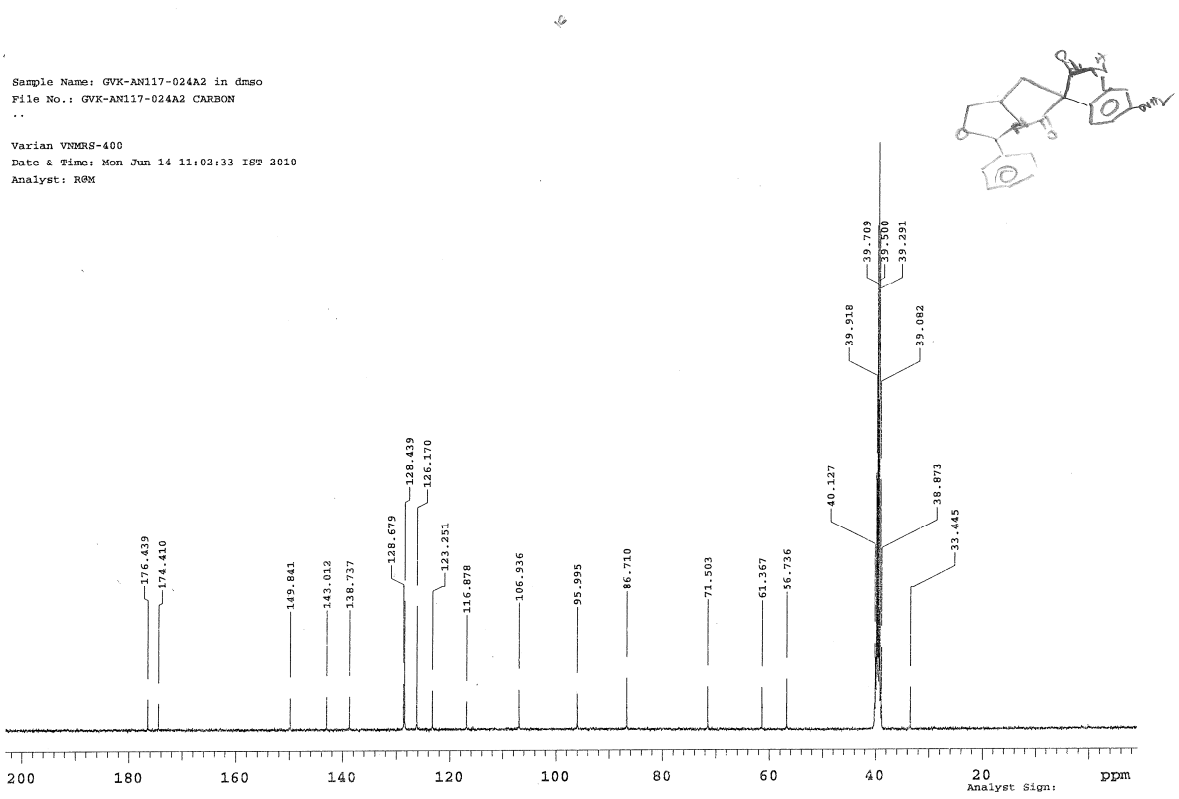
Compound **17**


O
O
NH
N
O
NH2
12
11
10
9
8
7
6
5
4
3
2
1
0
0.97
2.05
3.05
0.99
1.98
1.91
1.02
1.06
0.06
1.03
1.05
1.06
3.10


176.668
175.122
149.868
143.432
140.217
128.594
127.301
125.385
124.150
116.442
107.234
97.369
95.863
72.567
61.066
59.755
57.956
54.901
43.348
40.145
39.937
39.729
39.520
39.312
39.103
38.894
36.219
31.292
29.001
26.698
20.263
24.288
24.025
22.095
20.758
200
180
160
140
120
100
80
60
40
20
0
ppm

Compound **18**


Sample Name: GVK-AN117-024A2 in dmso
File No.: GVK-AN117-024A2_PROTON
Varian VNMRS-400
Analyst: Nancharaiah T
11
10
9
8
7
6
5
4
3
2
1
ppm
0.82
4.97
1.00
3.01
1.37
1.03
1.05
1.11
2.03
Analyst Sign:


Sample Name: GVK-AN117-024A2 in dmso
File No.: GVK-AN117-024A2 CARBON
..
Varian VNMRS-400
Date & Time: Mon Jun 14 11:02:33 IST 2010
Analyst: R&M
176.439
174.410
149.841
143.012
138.737
128.679
128.439
126.170
123.251
116.878
106.936
95.995
86.710
71.503
61.367
56.736
40.127
39.709
39.500
39.291
39.082
38.873
33.445
200
180
160
140
120
100
80
60
40
20
ppm
Analyst Sign:

GVK BIO

Eclipse XDBC8_DUP

Project Name: NRM1_2010\NRM1_JUNE_2010\NRM12_PDA05

GVK BIOSCIENCES (P) LTD

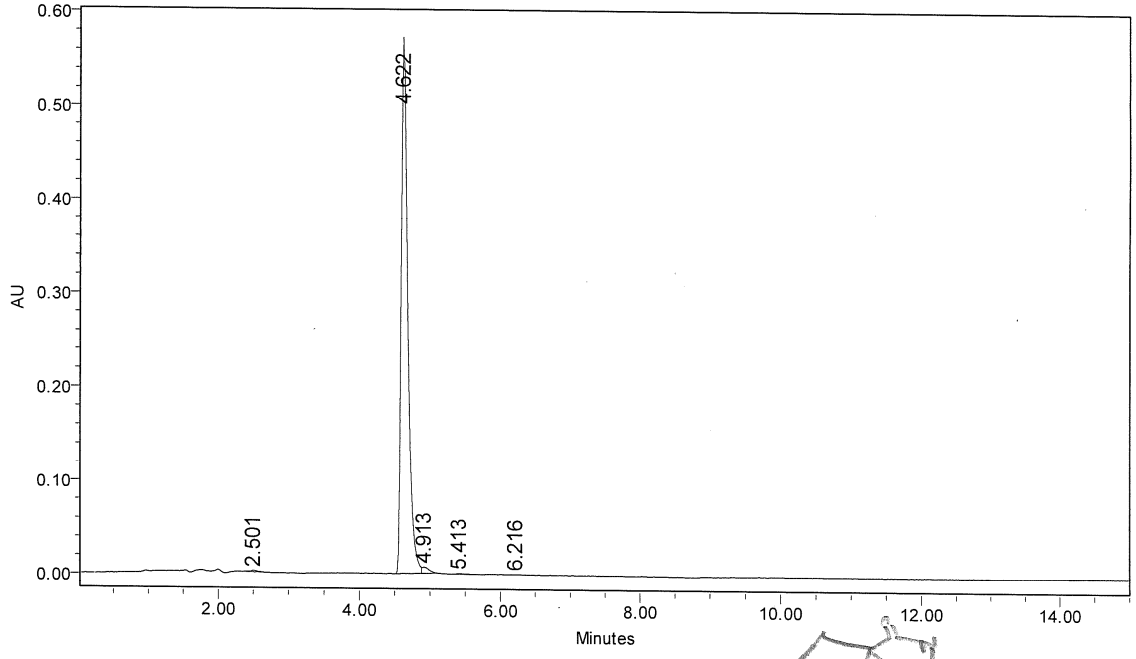
MCL Analytical-Nacharam

Sample Name:	GVK-AN117-024A2	Acquired By:	nrm12_mcl
Vial:	6	Date Acquired:	6/14/2010 12:43:34 PM IST
Injection Volume:	2.00 ul	Proc. Chnl. Descr.:	PDA MaxPlot (190.0 nm to 800.0 nm)
Run Time:	15.0 Minutes		

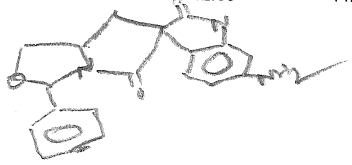
Chromatographic Conditions:

Column:Eclipse XDB-C8(4.6X150mm)5µ
Mobile Phase:A:0.01M Ammonium Acetate B:ACN
T/%B:0/30,4/70,6/95,15/95,15.1/30.
Flow Rate:0.8 ml/min
Temp:Ambient
Diluent:ACN

Purity Chromatogram

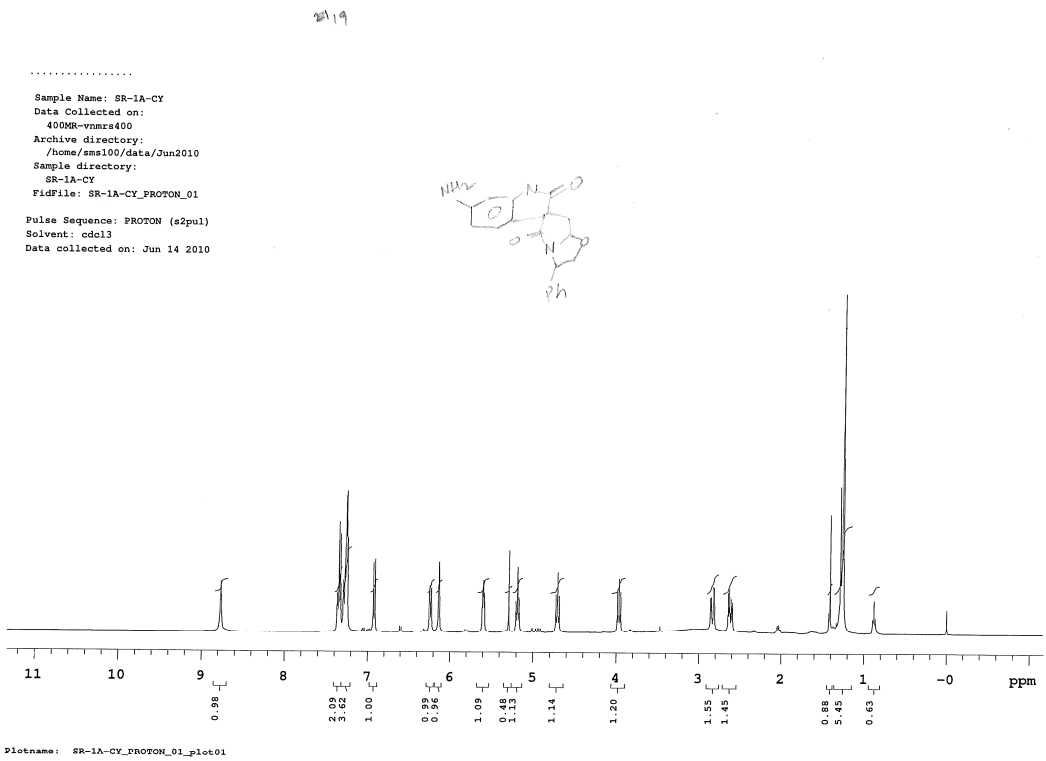


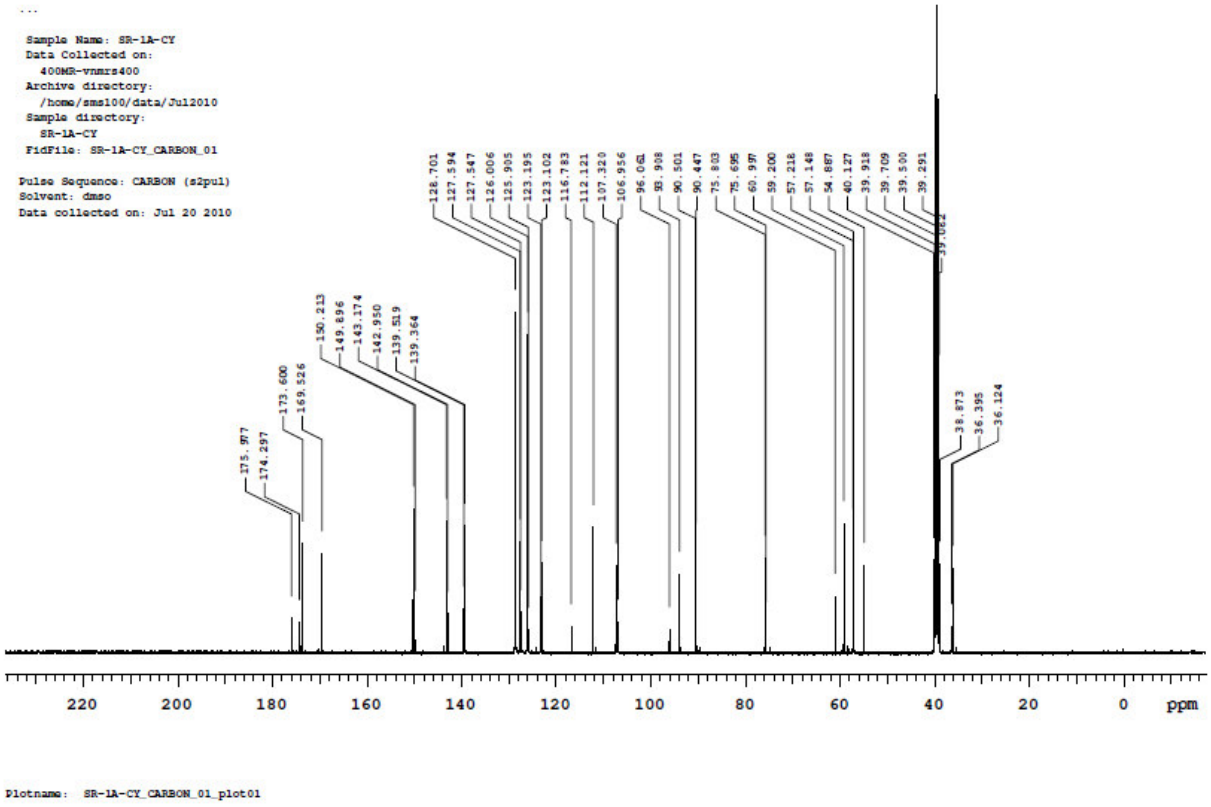
	RT	Area	% Area
1	2.50	12610	0.33
2	4.62	3723200	98.18
3	4.91	46511	1.23
4	5.41	6628	0.17
5	6.22	3454	0.09



Report Method Name: Eclipse XDBC8_ Printed 1:03:44 PM Asia/Calcutta 6/14/2010 Page: 1 of 1

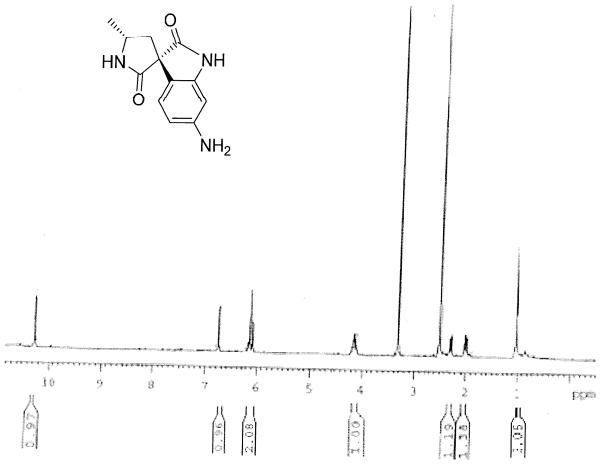
Compound **19**

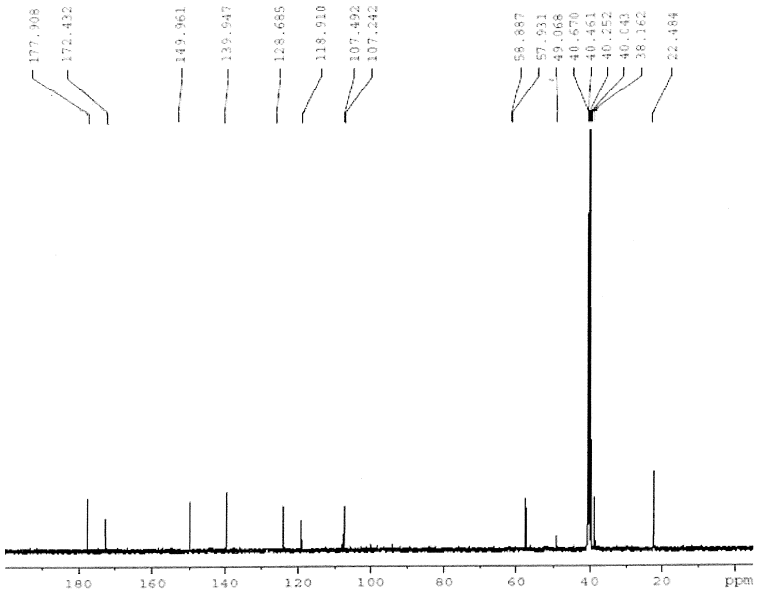

Sample Name: SR-1A-CY
Data Collected on:
400MR-vnmrs400
Archive directory:
/home/sms100/data/Jun2010
Sample directory:
SR-1A-CY
FidFile: SR-1A-CY_PROTON_01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 14 2010
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.98
2.09
3.02
1.00
0.99
0.96
1.09
0.48
1.13
1.14
1.20
1.55
1.45
0.88
5.45
0.63
Plotname: SR-1A-CY_PROTON_01_plot01



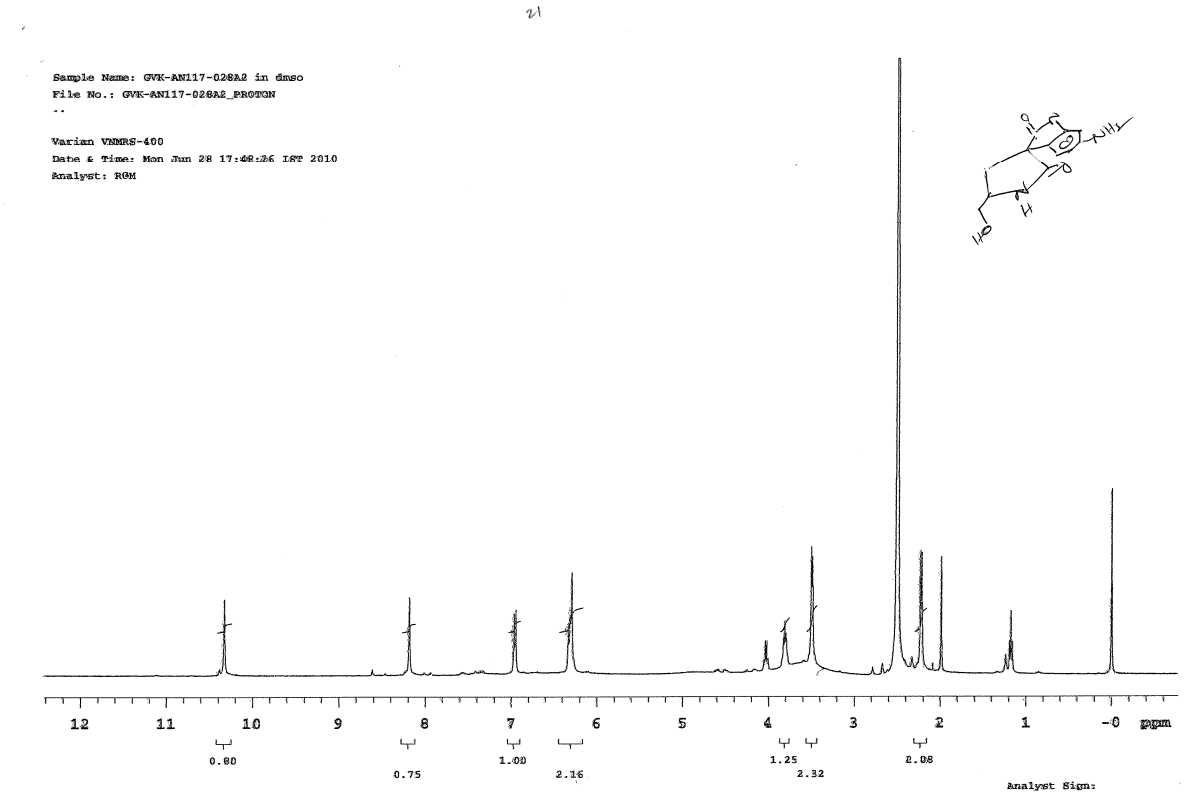
Plotname: SR-1A-CY_CARBON_01_plot01

Compound **20**




177.908
172.432
149.961
139.947
128.685
118.910
107.492
107.242
58.887
57.931
49.068
40.670
40.461
40.252
40.043
38.162
22.484
180
160
140
120
100
80
60
40
20
ppm

Compound **21**


21
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File No.: GVK-AN117-028A2_PROTON
Varian VNMRS-400
Date & Time: Mon Jun 28 17:49:26 IST 2010
Analyst: RGM
12
11
10
9
8
7
6
5
4
3
2
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-0
ppm
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0.75
1.00
2.16
1.25
2.32
2.08
Analyst Sign:

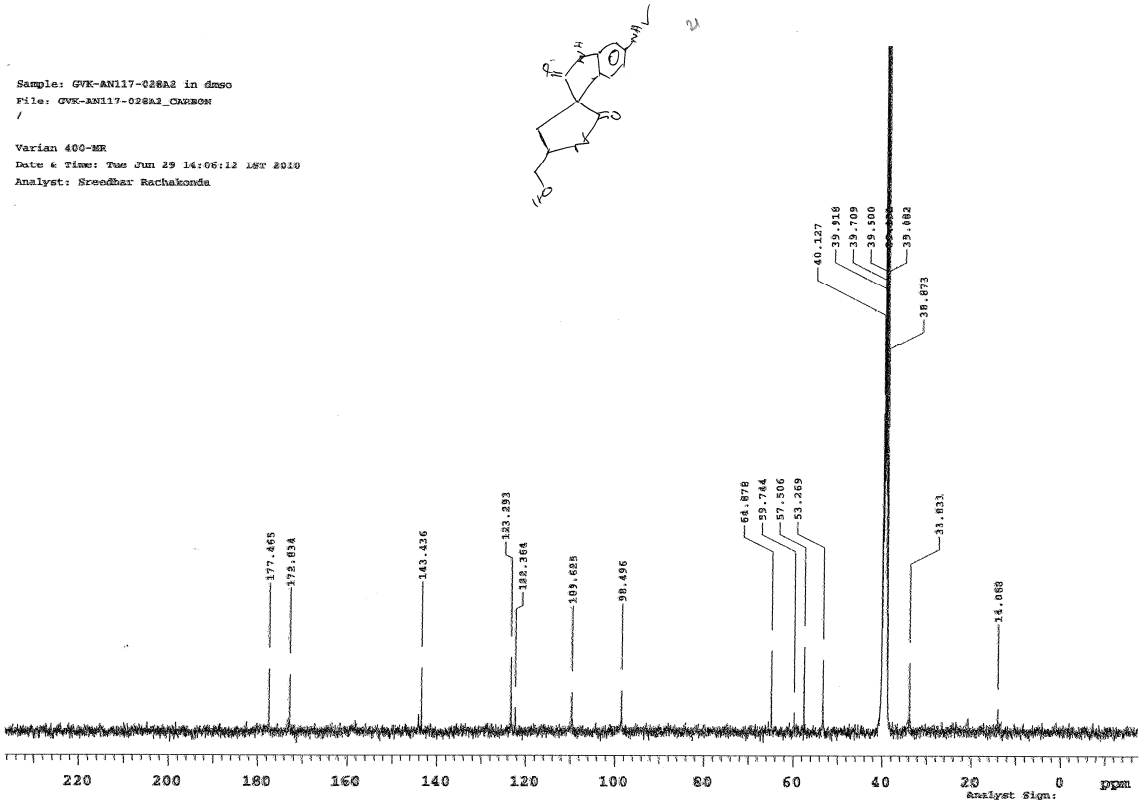
Sample: GVK-AN117-026A2 in dmso
File: GVK-AN117-026A2_CARBON
/
Varian 400-MR
Date & Time: Tue Jun 29 14:06:12 IST 2010
Analyst: Sreedhar Rachakonda
21
177.465
172.834
143.436
133.293
132.364
109.628
98.496
64.878
59.744
57.506
53.269
40.127
39.916
39.709
39.500
39.082
38.873
33.831
14.068
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Analyst Sign:

Table 1. Crystal data and structure refinement for **10**.

Identification code	egvk23	
Empirical formula	C_{20} H_{17} N_3 O_8	
Formula weight	427.37	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 9.7745(6) Å	a= 90°.
	b = 9.7915(4) Å	b= 90°.
	c = 20.5118(9) Å	g = 90°.
Volume	1963.12(17) $Å^3$	
Z	4	
Density (calculated)	1.446 Mg/m^3	
Absorption coefficient	0.114 mm^{-1}	
F(000)	888	
Crystal size	0.34 x 0.30 x 0.24 mm^3	
Theta range for data collection	2.88 to 26.37°.	
Index ranges	-12<=h<=11, -12<=k<=11, -20<=l<=25	
Reflections collected	7779	
Independent reflections	4020 [R(int) = 0.0317]	
Completeness to theta = 26.37°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9732 and 0.9623	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4020 / 0 / 281	
Goodness-of-fit on F^2	0.833	
Final R indices [I>2sigma(I)]	R1 = 0.0406, wR2 = 0.0662	
R indices (all data)	R1 = 0.0805, wR2 = 0.0733	
Absolute structure parameter	-0.4(11)	
Largest diff. peak and hole	0.152 and -0.165 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for egvk23. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	8616(3)	4824(2)	705(1)	42(1)
C(2)	8680(3)	5650(2)	1791(1)	45(1)
C(3)	9546(4)	6530(3)	1316(1)	71(1)
C(4)	8821(3)	3175(2)	1531(1)	36(1)
C(5)	9127(2)	2388(2)	882(1)	32(1)
C(6)	9032(3)	3528(2)	358(1)	39(1)
C(7)	10607(3)	1886(2)	972(1)	41(1)
C(8)	11931(3)	-31(3)	1279(2)	67(1)
C(9)	8148(3)	1212(2)	768(1)	32(1)
C(10)	8167(3)	375(2)	211(1)	33(1)
C(11)	7289(3)	-709(2)	118(1)	35(1)
C(12)	6310(3)	-945(2)	579(1)	35(1)
C(13)	6186(3)	-131(2)	1121(1)	44(1)
C(14)	7112(3)	917(2)	1212(1)	42(1)
C(15)	7431(3)	6380(2)	2039(1)	40(1)
C(16)	6126(3)	6023(2)	1859(1)	46(1)
C(17)	5003(3)	6718(2)	2106(1)	60(1)
C(18)	5185(4)	7774(3)	2536(1)	68(1)
C(19)	6486(4)	8143(3)	2718(1)	74(1)
C(20)	7611(4)	7460(2)	2474(1)	61(1)
N(1)	8389(2)	4457(2)	1378(1)	35(1)
N(2)	9149(2)	595(2)	-330(1)	38(1)
N(3)	5350(3)	-2068(2)	477(1)	44(1)
O(1)	9717(2)	5778(2)	744(1)	70(1)
O(2)	8969(2)	2729(2)	2076(1)	49(1)
O(3)	11601(2)	2558(2)	859(1)	54(1)
O(4)	10609(2)	617(2)	1215(1)	50(1)
O(5)	10237(2)	1155(2)	-217(1)	57(1)
O(6)	8811(2)	180(2)	-866(1)	53(1)
O(7)	5731(2)	-3042(2)	150(1)	58(1)
O(8)	4214(2)	-1972(2)	723(1)	63(1)

Table 3. Bond lengths [Å] and angles [°] for egvk23.

C(1)-O(1)	1.427(3)
C(1)-N(1)	1.443(3)
C(1)-C(6)	1.511(3)
C(1)-H(1)	0.9800
C(2)-N(1)	1.471(2)
C(2)-C(15)	1.503(4)
C(2)-C(3)	1.552(3)
C(2)-H(2)	0.9800
C(3)-O(1)	1.395(3)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
C(4)-O(2)	1.209(2)
C(4)-N(1)	1.361(3)
C(4)-C(5)	1.565(3)
C(5)-C(9)	1.516(3)
C(5)-C(7)	1.539(3)
C(5)-C(6)	1.553(3)
C(6)-H(6A)	0.9700
C(6)-H(6B)	0.9700
C(7)-O(3)	1.196(3)
C(7)-O(4)	1.339(3)
C(8)-O(4)	1.446(3)
C(8)-H(8A)	0.9600
C(8)-H(8B)	0.9600
C(8)-H(8C)	0.9600
C(9)-C(14)	1.392(3)
C(9)-C(10)	1.407(3)
C(10)-C(11)	1.377(3)
C(10)-N(2)	1.482(3)
C(11)-C(12)	1.365(3)
C(11)-H(11)	0.9300
C(12)-C(13)	1.373(3)
C(12)-N(3)	1.461(3)
C(13)-C(14)	1.380(3)
C(13)-H(13)	0.9300
C(14)-H(14)	0.9300
C(15)-C(16)	1.373(4)

C(15)-C(20)	1.395(3)
C(16)-C(17)	1.387(4)
C(16)-H(16)	0.9300
C(17)-C(18)	1.370(3)
C(17)-H(17)	0.9300
C(18)-C(19)	1.375(5)
C(18)-H(18)	0.9300
C(19)-C(20)	1.380(4)
C(19)-H(19)	0.9300
C(20)-H(20)	0.9300
N(2)-O(6)	1.219(2)
N(2)-O(5)	1.219(3)
N(3)-O(8)	1.223(3)
N(3)-O(7)	1.224(2)

O(1)-C(1)-N(1)	103.01(18)
O(1)-C(1)-C(6)	112.0(2)
N(1)-C(1)-C(6)	106.47(16)
O(1)-C(1)-H(1)	111.7
N(1)-C(1)-H(1)	111.7
C(6)-C(1)-H(1)	111.7
N(1)-C(2)-C(15)	114.5(2)
N(1)-C(2)-C(3)	100.63(17)
C(15)-C(2)-C(3)	113.07(19)
N(1)-C(2)-H(2)	109.4
C(15)-C(2)-H(2)	109.4
C(3)-C(2)-H(2)	109.4
O(1)-C(3)-C(2)	107.49(18)
O(1)-C(3)-H(3A)	110.2
C(2)-C(3)-H(3A)	110.2
O(1)-C(3)-H(3B)	110.2
C(2)-C(3)-H(3B)	110.2
H(3A)-C(3)-H(3B)	108.5
O(2)-C(4)-N(1)	125.64(19)
O(2)-C(4)-C(5)	125.80(19)
N(1)-C(4)-C(5)	108.55(18)
C(9)-C(5)-C(7)	111.67(18)
C(9)-C(5)-C(6)	113.59(18)
C(7)-C(5)-C(6)	111.6(2)

C(9)-C(5)-C(4)	112.64(18)
C(7)-C(5)-C(4)	103.66(19)
C(6)-C(5)-C(4)	102.91(15)
C(1)-C(6)-C(5)	107.02(16)
C(1)-C(6)-H(6A)	110.3
C(5)-C(6)-H(6A)	110.3
C(1)-C(6)-H(6B)	110.3
C(5)-C(6)-H(6B)	110.3
H(6A)-C(6)-H(6B)	108.6
O(3)-C(7)-O(4)	125.6(2)
O(3)-C(7)-C(5)	124.4(2)
O(4)-C(7)-C(5)	110.0(2)
O(4)-C(8)-H(8A)	109.5
O(4)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
O(4)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(14)-C(9)-C(10)	114.8(2)
C(14)-C(9)-C(5)	121.08(19)
C(10)-C(9)-C(5)	124.1(2)
C(11)-C(10)-C(9)	123.6(2)
C(11)-C(10)-N(2)	114.34(19)
C(9)-C(10)-N(2)	122.1(2)
C(12)-C(11)-C(10)	118.1(2)
C(12)-C(11)-H(11)	120.9
C(10)-C(11)-H(11)	120.9
C(11)-C(12)-C(13)	121.6(2)
C(11)-C(12)-N(3)	118.6(2)
C(13)-C(12)-N(3)	119.8(2)
C(12)-C(13)-C(14)	118.8(2)
C(12)-C(13)-H(13)	120.6
C(14)-C(13)-H(13)	120.6
C(13)-C(14)-C(9)	122.9(2)
C(13)-C(14)-H(14)	118.5
C(9)-C(14)-H(14)	118.5
C(16)-C(15)-C(20)	118.8(3)
C(16)-C(15)-C(2)	122.9(2)
C(20)-C(15)-C(2)	118.3(3)

C(15)-C(16)-C(17)	120.8(2)
C(15)-C(16)-H(16)	119.6
C(17)-C(16)-H(16)	119.6
C(18)-C(17)-C(16)	120.2(3)
C(18)-C(17)-H(17)	119.9
C(16)-C(17)-H(17)	119.9
C(17)-C(18)-C(19)	119.5(3)
C(17)-C(18)-H(18)	120.2
C(19)-C(18)-H(18)	120.2
C(18)-C(19)-C(20)	120.7(3)
C(18)-C(19)-H(19)	119.6
C(20)-C(19)-H(19)	119.6
C(19)-C(20)-C(15)	119.9(3)
C(19)-C(20)-H(20)	120.0
C(15)-C(20)-H(20)	120.0
C(4)-N(1)-C(1)	113.66(17)
C(4)-N(1)-C(2)	122.72(17)
C(1)-N(1)-C(2)	108.88(16)
O(6)-N(2)-O(5)	123.9(2)
O(6)-N(2)-C(10)	116.8(2)
O(5)-N(2)-C(10)	119.3(2)
O(8)-N(3)-O(7)	124.2(2)
O(8)-N(3)-C(12)	117.8(2)
O(7)-N(3)-C(12)	118.0(2)
C(3)-O(1)-C(1)	107.6(2)
C(7)-O(4)-C(8)	116.3(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for egvk23. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	44(2)	35(1)	46(1)	1(1)	1(1)	2(1)
C(2)	48(2)	37(1)	49(1)	-11(1)	-7(1)	-5(2)
C(3)	72(3)	55(2)	87(2)	-18(2)	21(2)	-24(2)
C(4)	34(2)	35(1)	39(1)	-4(1)	-1(1)	-4(1)
C(5)	31(2)	27(1)	37(1)	-1(1)	6(1)	1(1)
C(6)	44(2)	37(1)	36(1)	3(1)	4(1)	-2(1)
C(7)	36(2)	42(1)	45(1)	-6(1)	3(1)	0(1)
C(8)	46(2)	57(2)	98(2)	-4(2)	-6(2)	20(2)
C(9)	32(2)	29(1)	34(1)	1(1)	3(1)	4(1)
C(10)	33(2)	31(1)	34(1)	3(1)	4(1)	7(1)
C(11)	40(2)	27(1)	39(1)	-1(1)	-2(1)	2(1)
C(12)	31(2)	26(1)	48(1)	2(1)	-6(1)	-2(1)
C(13)	39(2)	51(1)	43(1)	3(1)	8(1)	-10(2)
C(14)	42(2)	45(1)	39(1)	-9(1)	5(1)	-6(1)
C(15)	55(2)	31(1)	33(1)	-2(1)	-2(1)	3(1)
C(16)	54(2)	35(1)	50(1)	-7(1)	8(2)	-1(2)
C(17)	57(2)	51(2)	72(2)	-4(2)	18(2)	-3(2)
C(18)	84(3)	58(2)	63(2)	-2(2)	24(2)	18(2)
C(19)	116(4)	52(2)	54(2)	-22(2)	-5(2)	28(2)
C(20)	77(3)	51(2)	55(2)	-20(1)	-20(2)	10(2)
N(1)	40(1)	29(1)	36(1)	-6(1)	1(1)	3(1)
N(2)	45(2)	28(1)	42(1)	-1(1)	10(1)	6(1)
N(3)	40(2)	36(1)	58(1)	7(1)	-9(1)	-4(1)
O(1)	93(2)	46(1)	69(1)	-11(1)	26(1)	-29(1)
O(2)	65(1)	44(1)	37(1)	2(1)	-4(1)	1(1)
O(3)	36(1)	56(1)	69(1)	-3(1)	4(1)	-10(1)
O(4)	38(1)	39(1)	72(1)	7(1)	1(1)	7(1)
O(5)	51(1)	56(1)	64(1)	-16(1)	25(1)	-14(1)
O(6)	64(1)	60(1)	34(1)	-4(1)	5(1)	12(1)
O(7)	61(2)	31(1)	80(1)	-9(1)	-2(1)	2(1)
O(8)	38(1)	56(1)	94(1)	-7(1)	6(1)	-12(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for egvk23.

	x	y	z	U(eq)
H(1)	7799	5233	509	50
H(2)	9249	5369	2161	53
H(3A)	9082	7383	1223	86
H(3B)	10429	6736	1508	86
H(6A)	9909	3649	145	47
H(6B)	8357	3288	30	47
H(8A)	12199	-411	867	100
H(8B)	11877	-746	1598	100
H(8C)	12595	633	1415	100
H(11)	7363	-1265	-248	42
H(13)	5491	-283	1422	53
H(14)	7040	1449	1586	50
H(16)	5993	5307	1568	55
H(17)	4125	6466	1980	72
H(18)	4432	8237	2703	82
H(19)	6611	8861	3009	89
H(20)	8487	7720	2600	73

Table 1. Crystal data and structure refinement for **14**.

Identification code	egvk22	
Empirical formula	C20 H17 N3 O8	
Formula weight	427.37	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21 1	
Unit cell dimensions	a = 6.8966(12) Å	α= 90°.
	b = 11.252(2) Å	β= 103.09(2)°.
	c = 12.799(4) Å	γ = 90°.
Volume	967.4(4) Å^3	
Z	2	
Density (calculated)	1.467 Mg/m^3	
Absorption coefficient	0.116 mm^{-1}	
F(000)	444	
Crystal size	0.42 x 0.32 x 0.14 mm^3	
Theta range for data collection	3.03 to 26.37°.	
Index ranges	-8<=h<=8, -13<=k<=14, -15<=l<=15	
Reflections collected	4448	
Independent reflections	3445 [R(int) = 0.0239]	
Completeness to theta = 26.37°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9840 and 0.9530	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3445 / 1 / 281	
Goodness-of-fit on F^2	0.856	
Final R indices [I>2sigma(I)]	R1 = 0.0446, wR2 = 0.0737	
R indices (all data)	R1 = 0.0968, wR2 = 0.0854	
Absolute structure parameter	0.3(13)	
Largest diff. peak and hole	0.134 and -0.142 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for egvk22. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	1729(5)	10335(3)	4806(3)	48(1)
C(2)	-443(4)	9277(3)	3351(3)	44(1)
C(3)	-1106(5)	10552(3)	3448(3)	60(1)
C(4)	3034(5)	9035(3)	3548(3)	42(1)
C(5)	2075(4)	8425(3)	2469(3)	41(1)
C(6)	-122(4)	8855(3)	2269(3)	52(1)
C(7)	3306(5)	8918(3)	1725(3)	51(1)
C(8)	4162(6)	10688(4)	942(3)	82(1)
C(9)	2216(4)	7088(3)	2554(3)	38(1)
C(10)	1897(5)	6302(3)	1679(3)	47(1)
C(11)	2021(4)	5097(3)	1798(3)	56(1)
C(12)	2464(5)	4630(3)	2817(4)	53(1)
C(13)	2794(5)	5325(3)	3703(3)	54(1)
C(14)	2666(4)	6552(3)	3560(3)	47(1)
C(15)	2144(4)	9995(3)	5961(3)	44(1)
C(16)	2057(5)	8835(3)	6307(3)	52(1)
C(17)	2463(5)	8569(3)	7390(3)	64(1)
C(18)	2922(5)	9446(4)	8149(3)	64(1)
C(19)	3002(5)	10608(4)	7811(3)	60(1)
C(20)	2628(4)	10880(3)	6732(3)	51(1)
N(1)	1562(3)	9353(2)	4040(2)	41(1)
N(2)	1392(5)	6739(3)	567(3)	65(1)
N(3)	2567(5)	3323(3)	2943(4)	79(1)
O(1)	-211(3)	10874(2)	4530(2)	63(1)
O(2)	4800(3)	9205(2)	3890(2)	60(1)
O(3)	4603(4)	8379(2)	1438(2)	72(1)
O(4)	2877(3)	10045(2)	1493(2)	62(1)
O(5)	1911(4)	6145(3)	-120(2)	98(1)
O(6)	457(4)	7670(3)	374(2)	82(1)
O(7)	2237(5)	2725(3)	2142(3)	120(1)
O(8)	2987(5)	2941(3)	3851(4)	114(1)

Table 3. Bond lengths [Å] and angles [°] for egvk22.

C(1)-O(1)	1.439(4)
C(1)-N(1)	1.465(4)
C(1)-C(15)	1.490(4)
C(1)-H(1)	0.9800
C(2)-N(1)	1.465(3)
C(2)-C(3)	1.520(4)
C(2)-C(6)	1.527(4)
C(2)-H(2)	0.9800
C(3)-O(1)	1.429(4)
C(3)-H(3B)	0.9700
C(3)-H(3A)	0.9700
C(4)-O(2)	1.212(3)
C(4)-N(1)	1.358(4)
C(4)-C(5)	1.550(4)
C(5)-C(9)	1.510(4)
C(5)-C(7)	1.517(4)
C(5)-C(6)	1.556(4)
C(6)-H(6B)	0.9700
C(6)-H(6A)	0.9700
C(7)-O(3)	1.205(4)
C(7)-O(4)	1.321(4)
C(8)-O(4)	1.446(4)
C(8)-H(8A)	0.9600
C(8)-H(8C)	0.9600
C(8)-H(8B)	0.9600
C(9)-C(14)	1.391(4)
C(9)-C(10)	1.404(4)
C(10)-C(11)	1.364(5)
C(10)-N(2)	1.471(5)
C(11)-C(12)	1.375(5)
C(11)-H(11)	0.9300
C(12)-C(13)	1.354(5)
C(12)-N(3)	1.479(5)
C(13)-C(14)	1.393(4)
C(13)-H(13)	0.9300
C(14)-H(14)	0.9300
C(15)-C(16)	1.384(4)

C(15)-C(20)	1.389(5)
C(16)-C(17)	1.383(5)
C(16)-H(16)	0.9300
C(17)-C(18)	1.371(5)
C(17)-H(17)	0.9300
C(18)-C(19)	1.383(5)
C(18)-H(18)	0.9300
C(19)-C(20)	1.380(5)
C(19)-H(19)	0.9300
C(20)-H(20)	0.9300
N(2)-O(5)	1.221(4)
N(2)-O(6)	1.226(4)
N(3)-O(7)	1.204(4)
N(3)-O(8)	1.212(4)

O(1)-C(1)-N(1)	103.1(3)
O(1)-C(1)-C(15)	108.1(3)
N(1)-C(1)-C(15)	116.1(3)
O(1)-C(1)-H(1)	109.8
N(1)-C(1)-H(1)	109.8
C(15)-C(1)-H(1)	109.8
N(1)-C(2)-C(3)	99.0(2)
N(1)-C(2)-C(6)	104.6(2)
C(3)-C(2)-C(6)	118.4(3)
N(1)-C(2)-H(2)	111.3
C(3)-C(2)-H(2)	111.3
C(6)-C(2)-H(2)	111.3
O(1)-C(3)-C(2)	104.3(3)
O(1)-C(3)-H(3B)	110.9
C(2)-C(3)-H(3B)	110.9
O(1)-C(3)-H(3A)	110.9
C(2)-C(3)-H(3A)	110.9
H(3B)-C(3)-H(3A)	108.9
O(2)-C(4)-N(1)	125.9(3)
O(2)-C(4)-C(5)	125.6(3)
N(1)-C(4)-C(5)	108.5(3)
C(9)-C(5)-C(7)	111.9(3)
C(9)-C(5)-C(4)	111.7(3)
C(7)-C(5)-C(4)	102.2(3)

C(9)-C(5)-C(6)	111.5(3)
C(7)-C(5)-C(6)	116.5(3)
C(4)-C(5)-C(6)	102.3(3)
C(2)-C(6)-C(5)	106.4(2)
C(2)-C(6)-H(6B)	110.4
C(5)-C(6)-H(6B)	110.4
C(2)-C(6)-H(6A)	110.4
C(5)-C(6)-H(6A)	110.4
H(6B)-C(6)-H(6A)	108.6
O(3)-C(7)-O(4)	124.0(3)
O(3)-C(7)-C(5)	124.6(3)
O(4)-C(7)-C(5)	111.3(3)
O(4)-C(8)-H(8A)	109.5
O(4)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
O(4)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
H(8C)-C(8)-H(8B)	109.5
C(14)-C(9)-C(10)	115.3(3)
C(14)-C(9)-C(5)	119.7(3)
C(10)-C(9)-C(5)	125.0(3)
C(11)-C(10)-C(9)	122.8(3)
C(11)-C(10)-N(2)	115.8(3)
C(9)-C(10)-N(2)	121.4(3)
C(10)-C(11)-C(12)	118.7(4)
C(10)-C(11)-H(11)	120.6
C(12)-C(11)-H(11)	120.6
C(13)-C(12)-C(11)	122.2(3)
C(13)-C(12)-N(3)	119.2(4)
C(11)-C(12)-N(3)	118.6(4)
C(12)-C(13)-C(14)	117.9(4)
C(12)-C(13)-H(13)	121.0
C(14)-C(13)-H(13)	121.0
C(9)-C(14)-C(13)	123.1(4)
C(9)-C(14)-H(14)	118.5
C(13)-C(14)-H(14)	118.5
C(16)-C(15)-C(20)	118.0(3)
C(16)-C(15)-C(1)	123.2(3)
C(20)-C(15)-C(1)	118.9(3)

C(17)-C(16)-C(15)	120.8(3)
C(17)-C(16)-H(16)	119.6
C(15)-C(16)-H(16)	119.6
C(18)-C(17)-C(16)	121.1(4)
C(18)-C(17)-H(17)	119.5
C(16)-C(17)-H(17)	119.5
C(17)-C(18)-C(19)	118.6(4)
C(17)-C(18)-H(18)	120.7
C(19)-C(18)-H(18)	120.7
C(20)-C(19)-C(18)	120.8(4)
C(20)-C(19)-H(19)	119.6
C(18)-C(19)-H(19)	119.6
C(19)-C(20)-C(15)	120.8(4)
C(19)-C(20)-H(20)	119.6
C(15)-C(20)-H(20)	119.6
C(4)-N(1)-C(2)	114.1(3)
C(4)-N(1)-C(1)	123.8(3)
C(2)-N(1)-C(1)	111.6(2)
O(5)-N(2)-O(6)	123.3(4)
O(5)-N(2)-C(10)	118.0(4)
O(6)-N(2)-C(10)	118.6(3)
O(7)-N(3)-O(8)	125.2(4)
O(7)-N(3)-C(12)	117.9(5)
O(8)-N(3)-C(12)	116.9(4)
C(3)-O(1)-C(1)	107.7(2)
C(7)-O(4)-C(8)	117.3(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for egvk22. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	46(2)	32(2)	74(3)	-8(2)	27(2)	-2(2)
C(2)	35(2)	36(2)	65(2)	0(2)	18(2)	1(2)
C(3)	55(2)	43(2)	87(3)	12(2)	25(2)	17(2)
C(4)	42(2)	41(2)	46(2)	0(2)	15(2)	1(2)
C(5)	36(2)	39(2)	51(2)	5(2)	15(2)	4(2)
C(6)	42(2)	48(2)	63(3)	2(2)	8(2)	4(2)
C(7)	57(2)	48(2)	49(2)	2(2)	15(2)	-2(2)
C(8)	119(3)	68(3)	65(3)	10(2)	33(3)	-28(3)
C(9)	31(2)	38(2)	45(2)	1(2)	11(2)	2(2)
C(10)	41(2)	50(2)	50(3)	-2(2)	13(2)	4(2)
C(11)	40(2)	49(2)	82(3)	-13(2)	16(2)	-1(2)
C(12)	44(2)	32(2)	85(3)	4(2)	21(2)	-1(2)
C(13)	46(2)	45(2)	75(3)	16(2)	23(2)	1(2)
C(14)	44(2)	43(2)	56(3)	-1(2)	16(2)	2(2)
C(15)	41(2)	38(2)	57(3)	-7(2)	20(2)	-3(2)
C(16)	60(2)	37(2)	60(3)	-8(2)	17(2)	-7(2)
C(17)	75(3)	47(2)	68(3)	1(2)	13(2)	-7(2)
C(18)	69(2)	60(3)	61(3)	-6(2)	14(2)	-10(2)
C(19)	53(2)	58(3)	73(3)	-18(2)	20(2)	-5(2)
C(20)	46(2)	36(2)	76(3)	-12(2)	24(2)	-2(2)
N(1)	38(1)	36(2)	51(2)	-3(1)	13(1)	3(1)
N(2)	61(2)	74(3)	58(3)	-13(2)	11(2)	1(2)
N(3)	62(2)	50(3)	125(4)	-1(3)	23(3)	-6(2)
O(1)	74(2)	48(2)	71(2)	-1(2)	24(1)	22(1)
O(2)	35(1)	75(2)	70(2)	-15(2)	15(1)	-1(1)
O(3)	80(2)	60(2)	93(2)	9(2)	58(2)	7(1)
O(4)	78(2)	45(2)	67(2)	11(1)	26(1)	-2(1)
O(5)	126(3)	107(3)	68(2)	-21(2)	35(2)	18(2)
O(6)	99(2)	78(2)	61(2)	-2(2)	3(1)	29(2)
O(7)	152(3)	51(2)	154(4)	-26(2)	26(2)	-4(2)
O(8)	147(3)	55(2)	139(4)	32(2)	29(3)	1(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for egvk22.

	x	y	z	U(eq)
H(1)	2734	10902	4687	58
H(2)	-1274	8717	3642	53
H(3B)	-643	11064	2945	72
H(3A)	-2546	10603	3314	72
H(6B)	-354	9501	1753	62
H(6A)	-1027	8210	1993	62
H(8A)	4743	10143	523	123
H(8C)	5197	11074	1457	123
H(8B)	3397	11273	478	123
H(11)	1810	4602	1201	68
H(13)	3097	4993	4387	65
H(14)	2892	7035	4165	56
H(16)	1721	8228	5806	62
H(17)	2424	7782	7606	76
H(18)	3175	9263	8876	76
H(19)	3310	11214	8316	72
H(20)	2701	11666	6520	61