# Supplementary Material

## Anti-virulence *C*-mannosides as antibiotic sparing, oral therapeutics for urinary tract infections

Laurel Mydock-McGrane[1,2,#], Zachary Cusumano[1,3,#], Zhenfu Han[2], Jerome S. Pinkner[3], Jana Binkley[3], Maria Kostakioti[3], Thomas Hannan[1,3], Roger Klein[3], Jan Crowley[5], Vasilios Kalas[3], Nigam P. Rath[4], Scott J. Hultgren[3,5], and James W. Janetka[2,5,*]

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1. **Prophylactic model of acute UTI with mannosides and prodrugs of mannoside 23 dosed at 25 mg/kg PO, 30 minutes prior to infection.**

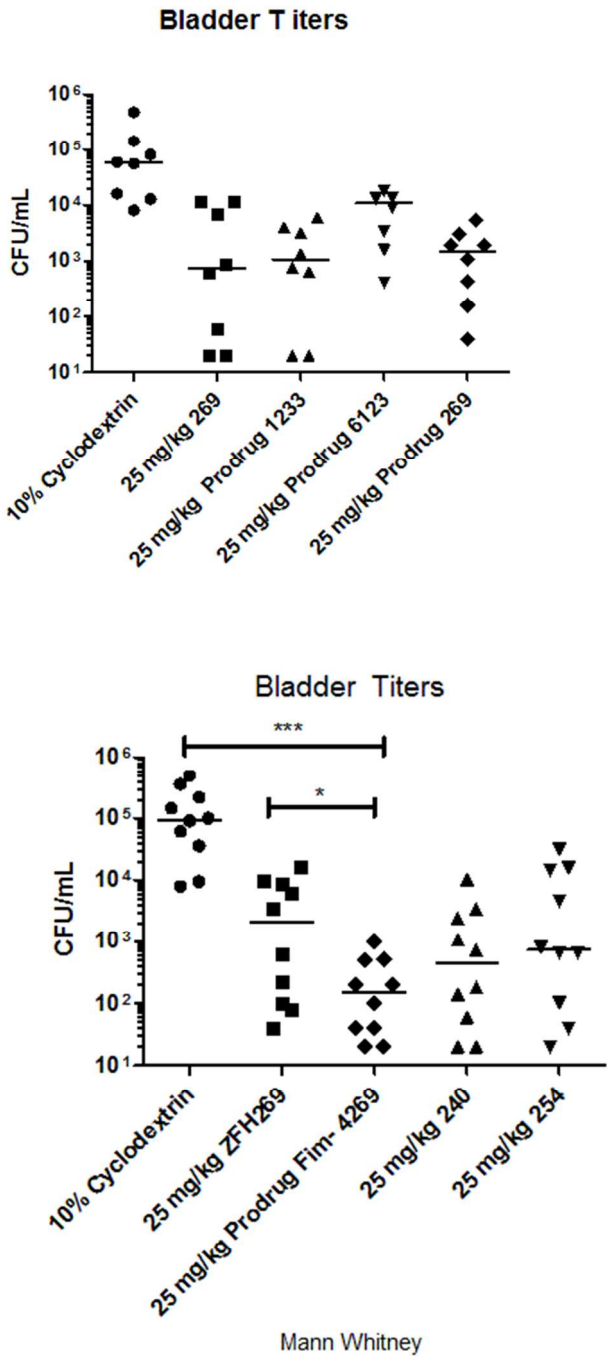
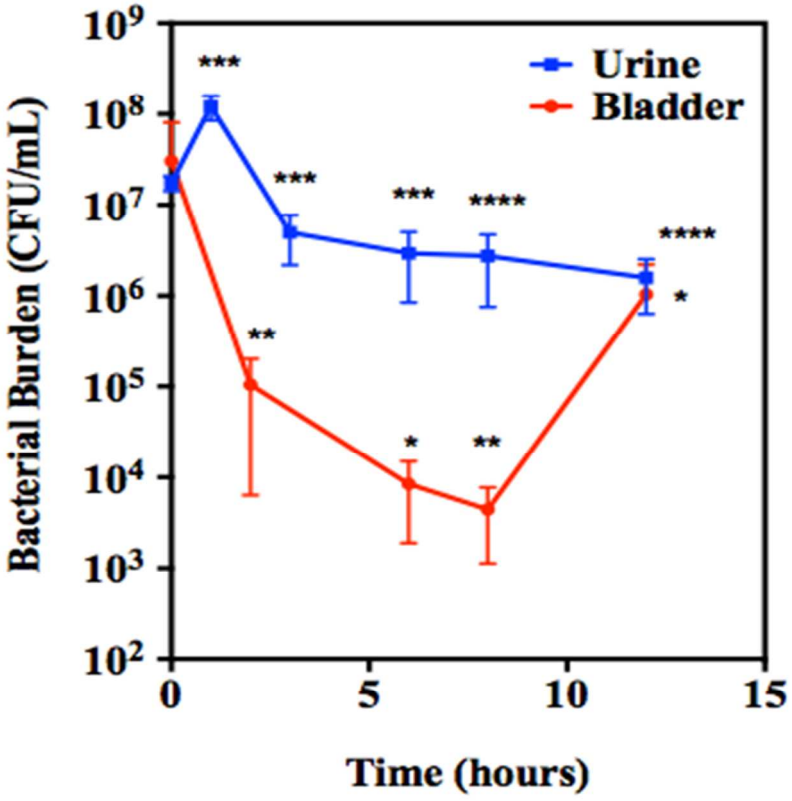



**Figure S1.** Bladder titers of **23** (269), **23a** (Prodrug 269), **23b** (Prodrug 1233), and **23c** (Prodrug 6123) following a 25 mg/kg PO dose in mice. Also shown on right is **23**, **23a**, **21R** (240), and **25** (254).

**2. Pharmacodynamics of 24 after oral dosing in chronic UTI model.**



## 3. Rat pharmacokinetics of 21R, 23, and 25

The pharmacokinetic profile of FIM-4269 (**23**), FIM-5240 (**21R**), FIM-5254 (**25**), was evaluated after a single intravenous (IV) injection or oral (PO) gavage dose to male Sprague Dawley (SD) rats. The study was performed by Seventh Wave Laboratories LLC
743 Spirit 40 Park Drive, Suite 209, Chesterfield, MO 63005.

Eighteen male rats were divided into six groups (3 rats per group). On Day 1 of the study, rats were administered **23**, **21R**, and **25** as a single IV dose at 3 mg/kg via surgically implanted jugular catheters at a dose volume of 1 mL/kg (Groups 1-3) or a single PO dose of 10 mg/kg at a dose volume of 2 mL/kg (Groups 4-6). The dose volume was calculated using individual rat body weight taken prior to dosing on Day 1. There were no abnormal clinical signs observed in any rats during the study. Blood was collected from all rats, via the tail vein, into $K_2$EDTA tubes at the protocol stated time points; 5, 15, 30 minutes, 1, 2, 4, 8 and 24 hour post dose for Groups 1-3 (IV Groups) and at 15 and 30 minutes, and 1, 2, 4, 8 and 24 hr post dose for Groups 4-6 (PO Groups). Plasma was separated via refrigerated centrifugation and Bioanalysis and PK analysis was performed to evaluate the systemic exposure to **23**, **21R**, and **25**. Using an internal standard, plasma and urine samples were analyzed for test article levels by LC–MS/MS with (+) ESI with Ion Spray detection using a Phenomenex Kinetex C18 (2.1 x 50 mm, 2.6 µm) reversed phase column with Mobile Phase, Aqueous: water containing 0.1% formic acid, Organic: acetonitrile containing 0.1% formic acid. The following MS/MS fragmentations [precursor mass/charge ratio (m/z)/product m/z] were found: compound **21R**, 418.20/238.20 amu; compound **23**, 404.20/242.20 amu; compound **25**, 414.20/252.20 amu.

Evidence of plasma exposure to FIM 4269, FIM-5240, FIM-5254, was observed in all FIM 4269, FIM-5240, FIM-5254-treated rats following a single IV and PO administration at 3 and 10 mg/kg, respectively. Exposure (as assessed by Cmax and AUCall) was the highest following IV administration for FIM-5240, followed by FIM-4269, and FIM-5254 with mean values of 3410 2440, 1590, 1360, and 337 ng/mL (Cmax) and 1440, 767, 521, 410, and 126 h*ng/mL (AUCall), respectively. FM-5240 had a moderate clearance in rat and the lowest of the three compounds at 34.9 mL/min/kg with the other compounds having markedly higher clearance values of 98.4 and 408 mL/min/kg for FIM-4269 and FIM-5254, respectively. All compounds had a short terminal half-life with values ranging from 0.28 to 0.49 hours following IV administration at 3 mg/kg.

Absorption (as assessed by Tmax) following PO administration at10 mg/kg was most rapid for FIM-5254, followed by FIM-5240, and FIM-4269 with mean Tmax values of 0.333, 0.667, and 1.42 hours, respectively. Exposure (as assessed by Cmax and AUCall) was the highest following PO administration for FIM-5240, followed by FIM-5254, and FIM-4269 with mean values of 18.6, 6.96, and 5.00 ng/mL (Cmax) and 93.7, 29.6, and 23.2 h*ng/mL (AUCall), respectively. The percent of the recovered dose in the urine was greatest for FIM-4269 followed by FIM5240, and FIM-5254 with mean values of 0.24, 0.19, and 0.13%, respectively. The low recovery of all five compounds in the urine over 24 hours suggests low renal clearance of unchanged parent compound following PO administration. Bioavailability for all compounds was low with the highest bioavailability observed for FIM-5254, followed by FIM-5240, and FIM-4269 with values of 7.05, 1.95, and 1.34, respectively.

**Table S1. Individual Animal and Group Mean Pharmacokinetic Summary Data; IV Administration**

| Group | Route | Dose (mg/kg) | Compound | Animal ID | $C_0$ (ng/mL) | $C_{max}$ (ng/mL) | $T_{max}$ (h) | $AUC_{all}$ (h*ng/mL) | $AUC_{INFobs}$ (h*ng/mL) | $Cl_{obs}$ (mL/min/kg) | $Vss_{obs}$ (L/kg) | $HL_{\lambda z}$ (h) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | IV | 3 | 23 (FIM-4269) | 1M001 | 2040 | 1280 | 0.083 | 485 | 480 | 104 | 1.85 | 0.326 |
| | | | | 1M002 | 3760 | 1980 | 0.083 | 622 | 619 | 80.8 | 0.965 | 0.301 |
| | | | | 1M003 | 2920 | 1520 | 0.083 | 455 | 454 | 110 | 1.16 | 0.308 |
| | | | | N | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| | | | | Mean | 2910 | 1590 | 0.083 | 521 | 518 | 98.4 | 1.33 | 0.312 |
| | | | | SD | 860 | 356 | 0.00 | 89.1 | 88.9 | 15.6 | 0.467 | 0.0129 |
| | | | | CV% | 29.6 | 22.3 | 0.0 | 17.1 | 17.2 | 15.8 | 35.2 | 4.1 |
| 2 | IV | 3 | 21R (FIM-5240) | 2M001 | 6520 | 4100 | 0.083 | 1480 | 1480 | 33.8 | 0.583 | 0.511 |
| | | | | 2M002 | 3520 | 2830 | 0.083 | 1470 | 1460 | 34.2 | 0.881 | 0.471 |
| | | | | 2M003 | 4710 | 3310 | 0.083 | 1370 | 1370 | 36.6 | 0.759 | 0.482 |
| | | | | N | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| | | | | Mean | 4920 | 3410 | 0.083 | 1440 | 1440 | 34.9 | 0.741 | 0.488 |
| | | | | SD | 1510 | 641 | 0.00 | 60.9 | 60.6 | 1.51 | 0.150 | 0.0209 |
| | | | | CV% | 30.7 | 18.8 | 0.0 | 4.2 | 4.2 | 4.3 | 20.2 | 4.3 |
| 3 | IV | 3 | 25 (FIM-5254) | 3M001 | 700 | 435 | 0.083 | 152 | 151 | 331 | 4.79 | 0.279 |
| | | | | 3M002 | 382 | 269 | 0.083 | 108 | 107 | 467 | 8.37 | 0.278 |
| | | | | 3M003 | 429 | 306 | 0.083 | 118 | 117 | 426 | 7.04 | 0.284 |
| | | | | N | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| | | | | Mean | 504 | 337 | 0.083 | 126 | 125 | 408 | 6.73 | 0.280 |
| | | | | SD | 172 | 87.1 | 0.00 | 23.0 | 23.0 | 69.7 | 1.81 | 0.0032 |
| | | | | CV% | 34.1 | 25.9 | 0.0 | 18.3 | 18.4 | 17.1 | 26.9 | 1.1 |

**Figure S2. 23 (FIM-4269) Plasma Concentration versus Time—Individual Animal Comparison; Group 1**

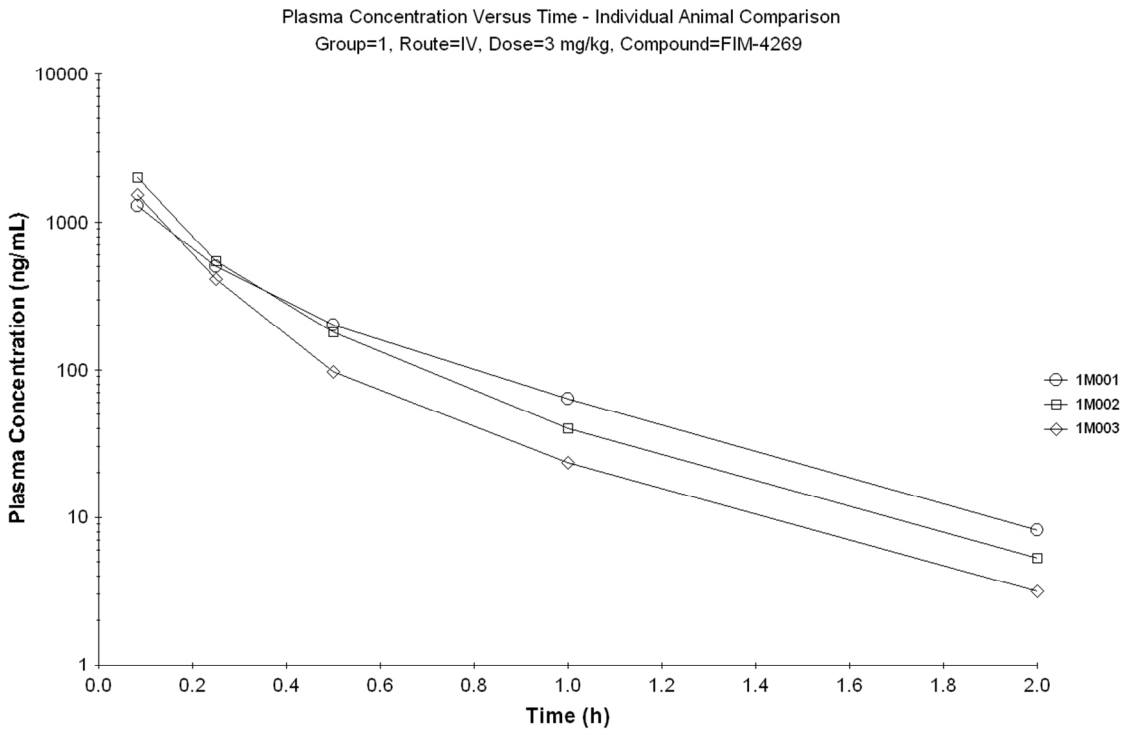



**Figure S3. 21R (FIM-5240) Plasma Concentration versus Time—Individual Animal Comparison; Group 2**

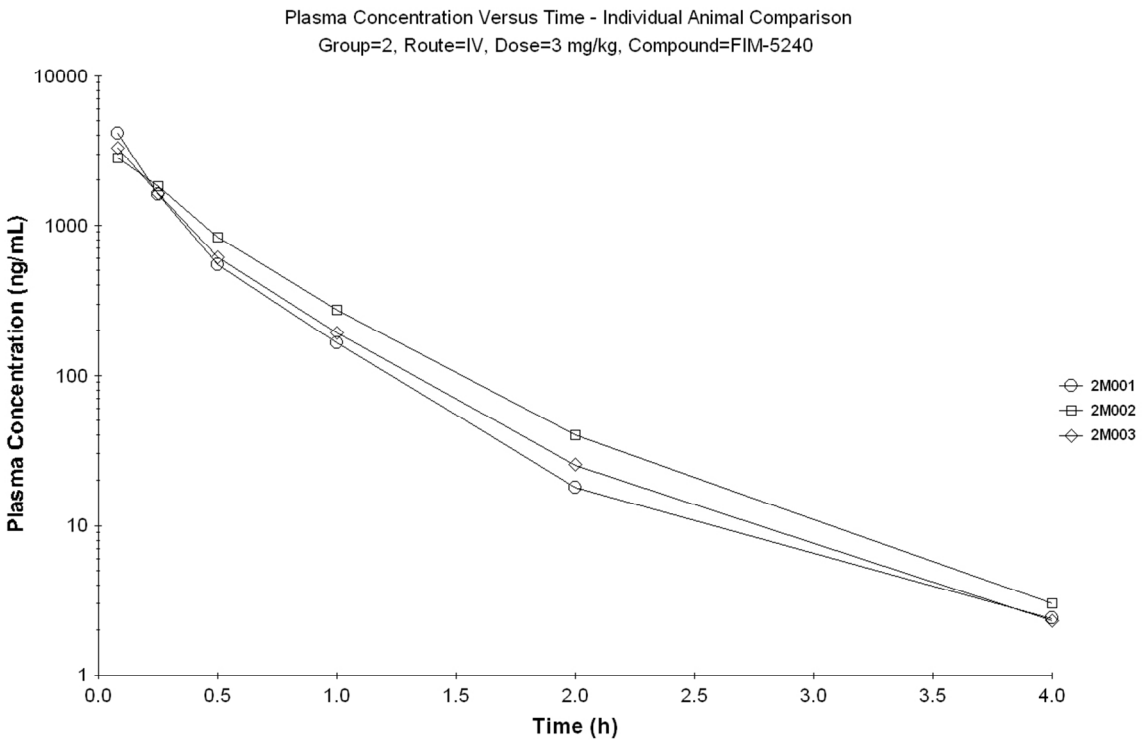



**Figure S4. 25 (FIM-5254) Plasma Concentration versus Time—Individual Animal Comparison; Group 3**

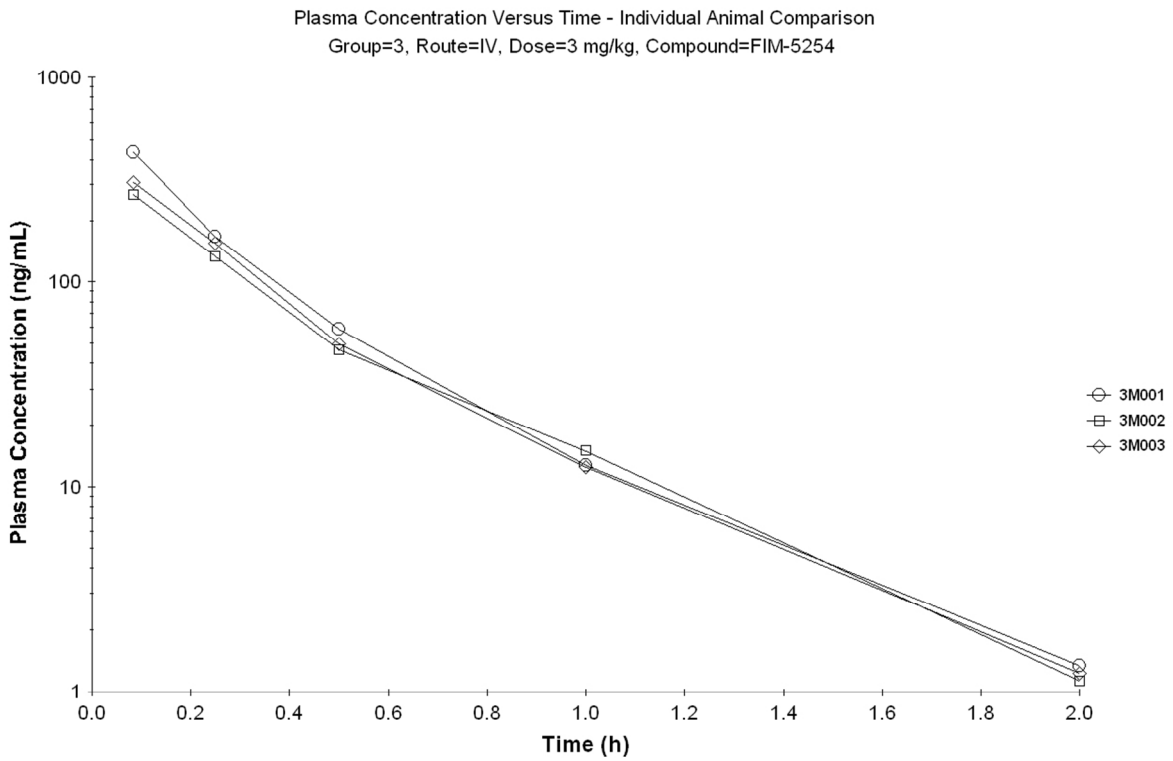



**Table S2. Individual Animal and Group Mean Pharmacokinetic Summary Data; PO Administration**

| Group | Route | Dose (mg/kg) | Compound | Animal ID | $C_{max}$ (ng/mL) | $T_{max}$ (h) | $AUC_{all}$ (h*ng/mL) | $AUC_{INFobs}$ (h*ng/mL) | $HL_{\lambda z}$ (h) | F (fraction) |
|---|---|---|---|---|---|---|---|---|---|---|
| 6 | PO | 10 | 23 (FIM-4269) | 6M001 | 4.41 | 2.00 | 34.6 | NC | NC | NA |
| | | | | 6M002 | 6.53 | 2.00 | 22.1 | NC | NC | NA |
| | | | | 6M003 | 4.05 | 0.250 | 12.8 | 16.0 | 2.85 | NA |
| | | | | N | 3 | 3 | 3 | 1 | 1 | 1 |
| | | | | Mean | 5.00 | 1.42 | 23.2 | 16.0 | 2.85 | 0.0134 |
| | | | | SD | 1.34 | 1.01 | 10.9 | NA | NA | NA |
| | | | | CV% | 26.8 | 71.3 | 47.2 | NA | NA | NA |
| 7 | PO | 10 | 21R (FIM-5240) | 7M001 | 23.8 | 0.500 | 83.6 | 75.0 | 3.56 | NA |
| | | | | 7M002 | 15.5 | 1.00 | 117 | 103 | 3.22 | NA |
| | | | | 7M003 | 16.4 | 0.500 | 80.9 | 74.0 | 3.81 | NA |
| | | | | N | 3 | 3 | 3 | 3 | 3 | 1 |
| | | | | Mean | 18.6 | 0.667 | 93.7 | 83.8 | 3.53 | 0.0195 |
| | | | | SD | 4.55 | 0.289 | 19.8 | 16.2 | 0.298 | NA |
| | | | | CV% | 24.5 | 43.3 | 21.2 | 19.3 | 8.4 | NA |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 8 | PO | 10 | 25 (FIM-5254) | 8M001 | 6.41 | 0.250 | 23.3 | NC | NC | NA |
| | | | | 8M002 | 8.53 | 0.500 | 18.3 | NC | NC | NA |
| | | | | 8M003 | 5.93 | 0.250 | 47.3 | NC | NC | NA |
| | | | | N | 3 | 3 | 3 | 0 | 0 | 1 |
| | | | | Mean | 6.96 | 0.333 | 29.6 | NA | NA | 0.0705 |
| | | | | SD | 1.38 | 0.144 | 15.5 | NA | NA | NA |
| | | | | CV% | 19.9 | 43.3 | 52.3 | NA | NA | NA |

**Figure S5. 23 (FIM-4269) Plasma Concentration versus Time—Individual Animal Comparison; Group 6**

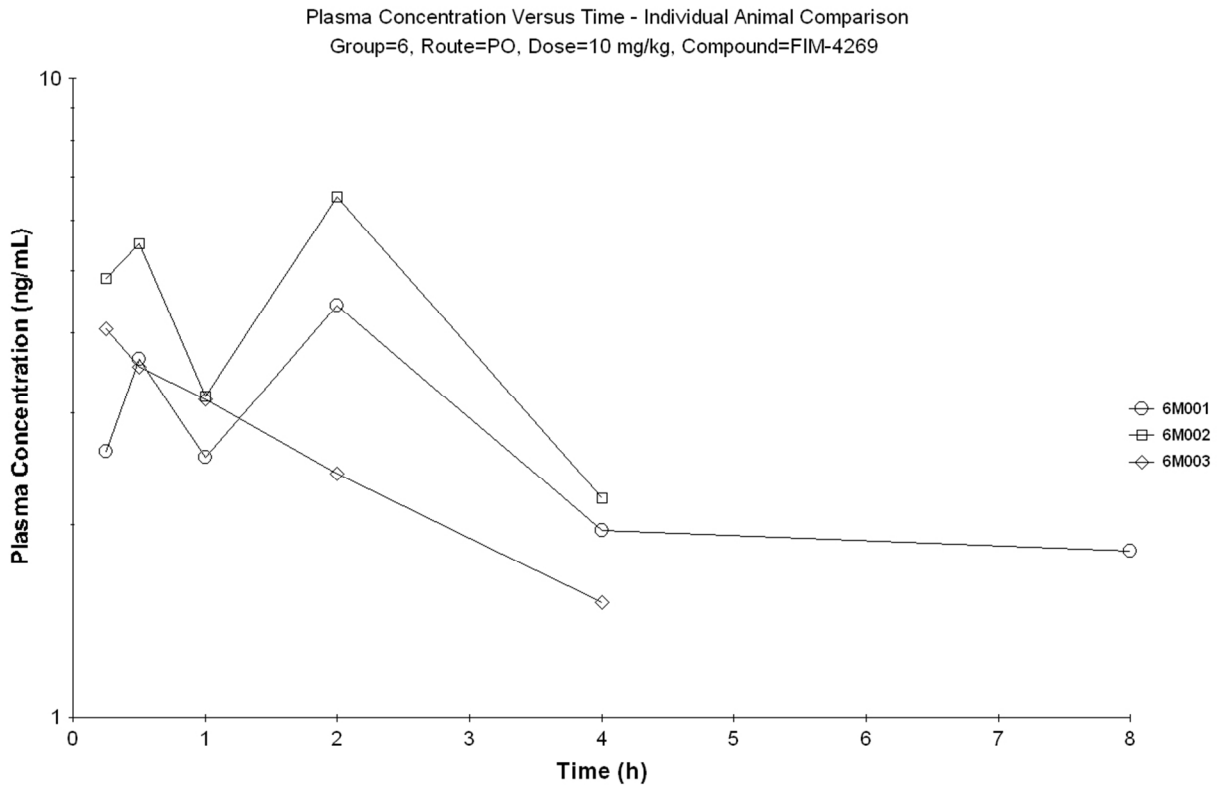



**Figure S6. 21R (FIM-5240) Plasma Concentration versus Time—Individual Animal Comparison; Group 7**

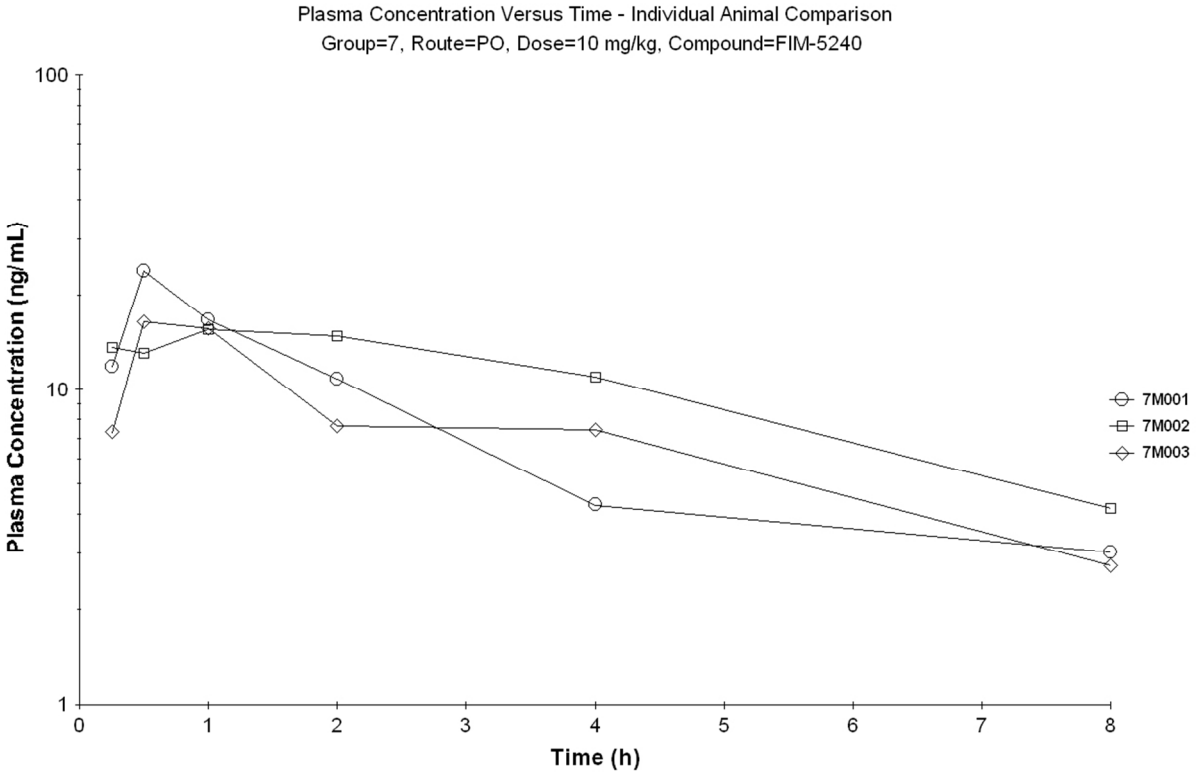
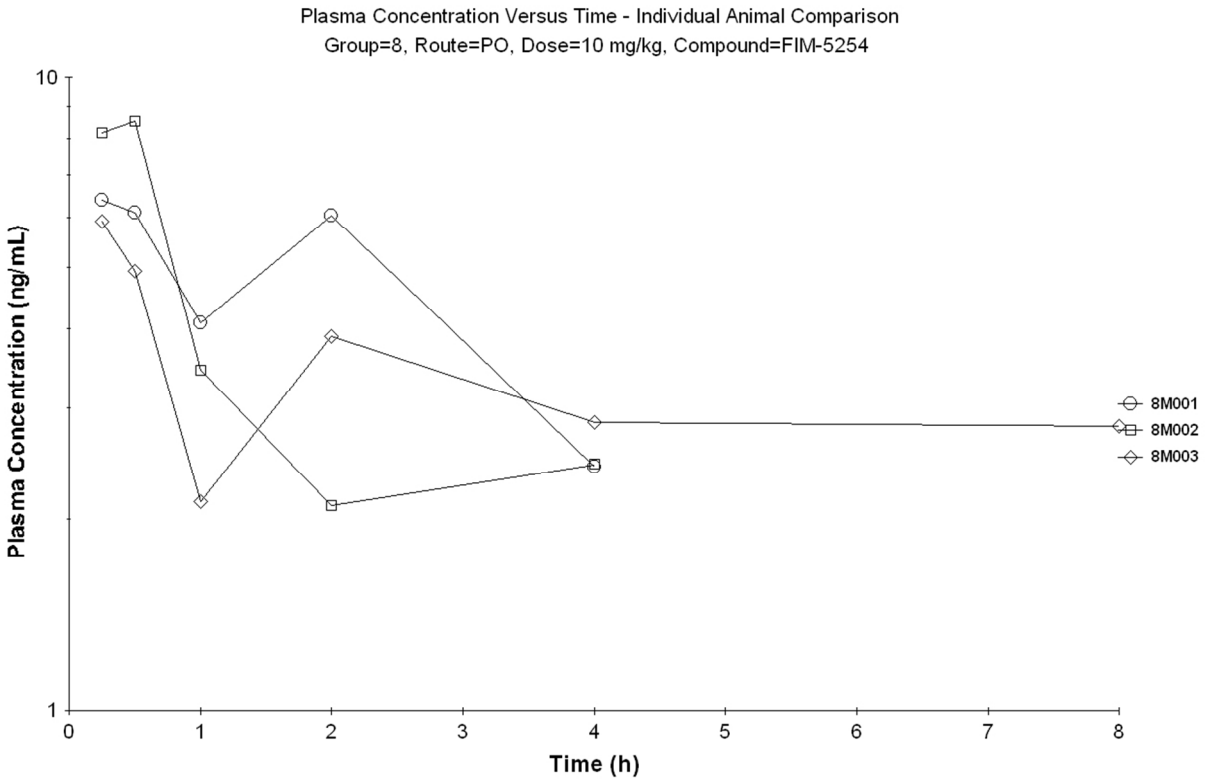

Plasma Concentration Versus Time - Individual Animal Comparison
Group=7, Route=PO, Dose=10 mg/kg, Compound=FIM-5240
Plasma Concentration (ng/mL)
100
10
1
0
1
2
3
4
5
6
7
8
Time (h)
7M001
7M002
7M003

**Figure S7. 25 (FIM-5254) Plasma Concentration versus Time—Individual Animal Comparison; Group 8**




All raw data, the Protocol, Protocol Amendments, supporting documents and any reports will be temporarily retained at Seventh Wave Laboratories LLC, 743 Spirit 40 Park Drive, Chesterfield, MO 63005 until transferred to the Sponsor or Sponsor's designee.

## 4. X-ray crystallization Studies of 23

**A. Protein Crystallization.** FimH lectin domain ($FimH_L$; residues 1-158) from E. coli strain J96 was expressed and purified as described previously and dialyzed into 10 mM MES pH 5.8, 50 mM NaCl. To generate crystals of $FimH_L$ bound to **23**, $FimH_L$ was pre-incubated with **23** at final concentrations of 1.27 mM (approx. 21 mg/mL) and 1.7 µM, respectively. 100 nL x 100 nL drops were set up using a TTP Labtech mosquito® crystal robot using 70 µL mother liquor (0.2 M AmSO4, 20% PEG 3350.) Cuboidal crystals appeared 72 hours of incubation at 18 °C. After approximately two weeks, crystals were transferred to mother liquor supplemented with 20% v/v glycerol as cryoprotectant and flash frozen in liquid nitrogen prior to storage and data collection.

**B. X-ray data collection and structure determination.** Diffraction data were collected at 100 ° K. X-rays were generated by a Rigaku MicroMax 007 generator coupled to a Rayonix Marmux X-ray source (Evanston, IL.) Images were collected on a Mar345 image plate dector. Data were indexed and integrated in iMosflm and scaled by Scala. The space group was then determined by POINTLESS. PHases were obtained by molecular replacement using PHASER and a .pdb of a previously solved $FimH_L$ structure (PDB ID 3MCY) stripped of all waters, additives, and ligands. The Fo-Fc map unambiguously indicated electron density corresponding to Mannoside **23** in two distinct binding conformations within the ligand binding pocket. Chemical restraints on **23** were generated by eLBOW and used in subsequent rounds of refinement with phenix.refine from the PHENIX package.

**Table S3. Data collection and refinement statistics**

| **Data Collection** | |
|---|---|
| Space Group | P 21 21 21 |
| Cell dimensions | |
| *a, b, c* (Å) | 48.97, 55.66, 60.89 |
| α, β, γ (°) | 90, 90, 90 |
| Resolution | 24.48 - 1.76 |
| Completeness (%) | 99.7 (24.48-1.76) |
| $R_{merge}$ (%) | 0.05 |
| $\langle I/\sigma(I) \rangle$ | 4.23 (at 1.76) |
| Total/Unique Reflections | 248304/17107 |

| **Refinement** | |
|---|---|
| $R_{work}/R_{free}$ | 0.1352 / 0.1718 |
| No. atoms (non-H) | |
| Protein | 1209 |
| Ligand | 58 |
| Water | 237 |
| Wilson B-Factor ($Å^2$) | 15.2 |
| R.M.S. Deviations | |
| Bond Lengths (Å) | 0.09 |
| Bond Angles (°) | 1.410 |

## 5. X-Ray Experimental, and Projection View of Compound 27.

Crystals of appropriate dimension were obtained from $Et_2O$/Hexanes. A crystal with approximate dimensions 0.52 x 0.46 x 0.37 $mm^3$ was mounted on a MiteGen cryoloop in random orientation. Preliminary examination and data collection were performed using a Bruker X8 Kappa Apex II Charge Coupled Device (CCD) Detector system single crystal X-Ray diffractometer equipped with an Oxford Cryostream LT device. All data were collected using graphite monochromated Mo Kα radiation (λ= 0.71073 Å) from a fine focus sealed tube X-Ray source. Preliminary unit cell constants were determined with a set of 36 narrow frame scans. Typical data sets consist of combinations of ϖ and φ scan frames with scan width of 0.5° and counting time of 15 seconds/frame at a crystal to detector distance of 4.0 cm. The collected frames were integrated using an orientation matrix determined from the narrow frame scans. Apex II and SAINT software packages[i] were used for data collection and data integration. Analysis of the integrated data did not show any decay. Final cell constants were determined by global refinement of reflections harvested from the complete data set. Collected data were corrected for systematic errors using SADABS[ii] based on the Laue symmetry using equivalent reflections.

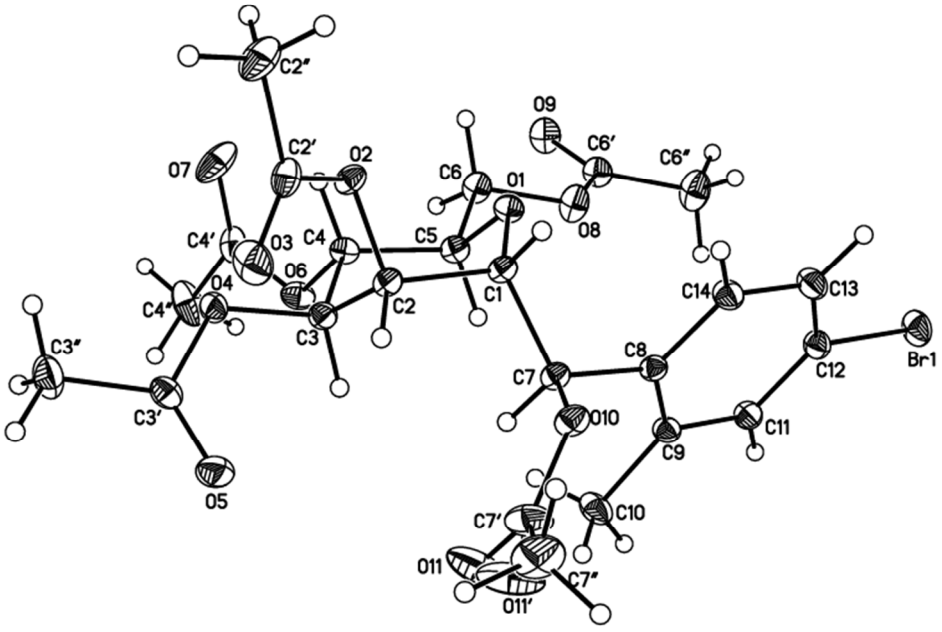



**Figure S8.** Compound **27** Projection view with 50% probability ellipsoids.

Structure solution and refinement were carried out using the SHELXTL- PLUS software package[iii]. The structure was solved by direct methods and refined successfully in the space group, P1. Full matrix least-squares refinements was carried out by minimizing $\Sigma w(F_o^2-F_c^2)^2$. The non-hydrogen atoms were refined anisotropically to convergence. All hydrogen atoms were treated using appropriate riding model (AFIX m3). Absolute structure was determined with the Flack x parameter refining to -0.011(1) for 6453 selected quotients using Parson's method[iv]

Complete listings of positional and isotropic displacement parameters for hydrogen atoms, anisotropic displacement parameters for the non-hydrogen atoms are listed below (**Tables S5-S9**). Table of calculated and observed structure factors are available in electronic format.

**Table S4. Crystal data and structure refinement for jj25315 (27).**

| | | |
|---|---|---|
| Identification code | j25315/lt/x8/Janetka-Mydock/Fimbrion | |
| Empirical formula | $C_{24}\ H_{29}\ Br\ O_{11}$ | |
| Formula weight | 573.38 | |
| Temperature | 100(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Triclinic | |
| Space group | P1 | |
| Unit cell dimensions | a = 8.1051(4) Å | α= 82.568(3)°. |
| | b = 8.9473(5) Å | β= 79.666(3)°. |
| | c = 9.4597(5) Å | γ = 88.134(2)°. |
| Volume | 669.18(6) $Å^3$ | |
| Z | 1 | |
| Density (calculated) | 1.423 $Mg/m^3$ | |
| Absorption coefficient | 1.592 $mm^{-1}$ | |
| F(000) | 296 | |
| Crystal size | 0.519 x 0.463 x 0.365 $mm^3$ | |
| Theta range for data collection | 2.206 to 39.500°. | |
| Index ranges | $-14\leq h\leq 14$, $-16\leq k\leq 16$, $-16\leq l\leq 16$ | |
| Reflections collected | 59865 | |
| Independent reflections | 15286 [R(int) = 0.022] | |
| Completeness to theta = 25.242° | 100.0 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.6514 and 0.6044 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 15286 / 4 / 340 | |
| Goodness-of-fit on $F^2$ | 1.054 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0240, wR2 = 0.0564 | |
| R indices (all data) | R1 = 0.0269, wR2 = 0.0573 | |
| Absolute structure parameter | -0.0108(10) | |
| Largest diff. peak and hole | 0.868 and -0.255 $e.Å^{-3}$ | |

**Table S5. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for jj25315.** U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Br(1) | 360(1) | 3108(1) | 413(1) | 22(1) |
| O(1) | 3160(1) | 4929(1) | 5885(1) | 14(1) |
| O(2) | 4149(1) | 7059(1) | 7644(1) | 15(1) |
| O(3) | 5720(1) | 9102(1) | 7614(1) | 25(1) |
| O(4) | 7354(1) | 5870(1) | 7477(1) | 16(1) |
| O(5) | 9062(1) | 7335(1) | 5744(1) | 24(1) |
| O(6) | 6921(1) | 2963(1) | 6793(1) | 18(1) |
| O(7) | 6165(2) | 2055(1) | 9141(1) | 30(1) |
| O(8) | 2666(1) | 2051(1) | 5175(1) | 21(1) |
| O(9) | 2256(1) | -335(1) | 6196(1) | 21(1) |
| O(10) | 4688(1) | 8165(1) | 3290(1) | 17(1) |
| O(11) | 7494(7) | 7919(8) | 2500(30) | 39(2) |
| O(11') | 7280(30) | 7997(17) | 2090(30) | 42(3) |
| C(1) | 3807(1) | 6395(1) | 5352(1) | 13(1) |
| C(2) | 5070(1) | 6835(1) | 6238(1) | 13(1) |
| C(2") | 3403(2) | 8516(2) | 9562(1) | 26(1) |
| C(2') | 4562(1) | 8291(1) | 8189(1) | 17(1) |
| C(3) | 6388(1) | 5593(1) | 6408(1) | 14(1) |
| C(3") | 9632(2) | 6964(2) | 8176(2) | 26(1) |
| C(3') | 8700(1) | 6780(1) | 6990(1) | 17(1) |
| C(4) | 5589(1) | 4065(1) | 6898(1) | 13(1) |
| C(4") | 8677(2) | 1126(2) | 7758(2) | 28(1) |
| C(4') | 7117(1) | 2066(1) | 8017(1) | 18(1) |
| C(5) | 4413(1) | 3776(1) | 5872(1) | 14(1) |
| C(6) | 3531(1) | 2292(1) | 6329(1) | 17(1) |
| C(6") | 1184(2) | 512(2) | 4014(2) | 27(1) |
| C(6') | 2071(1) | 653(1) | 5246(1) | 17(1) |
| C(7) | 4577(1) | 6547(1) | 3729(1) | 13(1) |
| C(7") | 6188(2) | 10410(2) | 2473(2) | 32(1) |
| C(7') | 6216(2) | 8722(1) | 2670(1) | 25(1) |
| C(8) | 3560(1) | 5793(1) | 2845(1) | 13(1) |

| | | | | |
|---|---|---|---|---|
| C(9) | 4344(1) | 4819(1) | 1877(1) | 14(1) |
| C(10) | 6212(1) | 4568(2) | 1575(1) | 21(1) |
| C(11) | 3356(1) | 4040(1) | 1154(1) | 15(1) |
| C(12) | 1636(1) | 4259(1) | 1374(1) | 16(1) |
| C(13) | 845(1) | 5263(1) | 2286(1) | 18(1) |
| C(14) | 1825(1) | 6013(1) | 3026(1) | 16(1) |

**Table S6. Bond lengths [Å] and angles [°] for jj25315.**

| | |
|---|---|
| Br(1)-C(12) | 1.8992(10) |
| O(1)-C(5) | 1.4233(12) |
| O(1)-C(1) | 1.4243(11) |
| O(2)-C(2') | 1.3507(13) |
| O(2)-C(2) | 1.4386(12) |
| O(3)-C(2') | 1.2094(15) |
| O(4)-C(3') | 1.3582(13) |
| O(4)-C(3) | 1.4344(12) |
| O(5)-C(3') | 1.2068(14) |
| O(6)-C(4') | 1.3515(14) |
| O(6)-C(4) | 1.4369(12) |
| O(7)-C(4') | 1.1954(16) |
| O(8)-C(6') | 1.3436(12) |
| O(8)-C(6) | 1.4411(13) |
| O(9)-C(6') | 1.2027(13) |
| O(10)-C(7') | 1.3532(15) |
| O(10)-C(7) | 1.4541(12) |
| O(11)-C(7') | 1.239(7) |
| O(11')-C(7') | 1.160(15) |
| C(1)-C(2) | 1.5249(13) |
| C(1)-C(7) | 1.5412(13) |
| C(1)-H(1) | 1.0000 |
| C(2)-C(3) | 1.5300(14) |
| C(2)-H(2) | 1.0000 |
| C(2")-C(2') | 1.4922(17) |
| C(2")-H(2"A) | 0.9800 |
| C(2")-H(2"B) | 0.9800 |
| C(2")-H(2"C) | 0.9800 |
| C(3)-C(4) | 1.5126(13) |
| C(3)-H(3) | 1.0000 |
| C(3")-C(3') | 1.4888(16) |
| C(3")-H(3"A) | 0.9800 |
| C(3")-H(3"B) | 0.9800 |
| C(3")-H(3"C) | 0.9800 |

| | |
|---|---|
| C(4)-C(5) | 1.5263(14) |
| C(4)-H(4) | 1.0000 |
| C(4")-C(4') | 1.4969(16) |
| C(4")-H(4"1) | 0.9800 |
| C(4")-H(4"2) | 0.9800 |
| C(4")-H(4"3) | 0.9800 |
| C(5)-C(6) | 1.5044(13) |
| C(5)-H(5) | 1.0000 |
| C(6)-H(6A) | 0.9900 |
| C(6)-H(6B) | 0.9900 |
| C(6")-C(6') | 1.4951(17) |
| C(6")-H(6"A) | 0.9800 |
| C(6")-H(6"B) | 0.9800 |
| C(6")-H(6"C) | 0.9800 |
| C(7)-C(8) | 1.5047(13) |
| C(7)-H(7) | 1.0000 |
| C(7")-C(7') | 1.4979(19) |
| C(7")-H(7"A) | 0.9800 |
| C(7")-H(7"B) | 0.9800 |
| C(7")-H(7"C) | 0.9800 |
| C(8)-C(14) | 1.3965(14) |
| C(8)-C(9) | 1.4010(14) |
| C(9)-C(11) | 1.3968(14) |
| C(9)-C(10) | 1.5049(15) |
| C(10)-H(10A) | 0.9800 |
| C(10)-H(10B) | 0.9800 |
| C(10)-H(10C) | 0.9800 |
| C(11)-C(12) | 1.3829(14) |
| C(11)-H(11) | 0.9500 |
| C(12)-C(13) | 1.3879(15) |
| C(13)-C(14) | 1.3877(15) |
| C(13)-H(13) | 0.9500 |
| C(14)-H(14) | 0.9500 |

| | |
|---|---|
| C(5)-O(1)-C(1) | 113.75(7) |
| C(2')-O(2)-C(2) | 115.78(8) |

| | |
|---|---|
| C(3')-O(4)-C(3) | 115.71(8) |
| C(4')-O(6)-C(4) | 117.58(8) |
| C(6')-O(8)-C(6) | 115.27(8) |
| C(7')-O(10)-C(7) | 117.15(9) |
| O(1)-C(1)-C(2) | 111.17(7) |
| O(1)-C(1)-C(7) | 112.46(7) |
| C(2)-C(1)-C(7) | 110.78(8) |
| O(1)-C(1)-H(1) | 107.4 |
| C(2)-C(1)-H(1) | 107.4 |
| C(7)-C(1)-H(1) | 107.4 |
| O(2)-C(2)-C(1) | 107.17(8) |
| O(2)-C(2)-C(3) | 109.27(7) |
| C(1)-C(2)-C(3) | 111.23(8) |
| O(2)-C(2)-H(2) | 109.7 |
| C(1)-C(2)-H(2) | 109.7 |
| C(3)-C(2)-H(2) | 109.7 |
| C(2')-C(2")-H(2"A) | 109.5 |
| C(2')-C(2")-H(2"B) | 109.5 |
| H(2"A)-C(2")-H(2"B) | 109.5 |
| C(2')-C(2")-H(2"C) | 109.5 |
| H(2"A)-C(2")-H(2"C) | 109.5 |
| H(2"B)-C(2")-H(2"C) | 109.5 |
| O(3)-C(2')-O(2) | 123.43(10) |
| O(3)-C(2')-C(2") | 125.18(11) |
| O(2)-C(2')-C(2") | 111.38(10) |
| O(4)-C(3)-C(4) | 107.00(8) |
| O(4)-C(3)-C(2) | 110.83(8) |
| C(4)-C(3)-C(2) | 111.64(8) |
| O(4)-C(3)-H(3) | 109.1 |
| C(4)-C(3)-H(3) | 109.1 |
| C(2)-C(3)-H(3) | 109.1 |
| C(3')-C(3")-H(3"A) | 109.5 |
| C(3')-C(3")-H(3"B) | 109.5 |
| H(3"A)-C(3")-H(3"B) | 109.5 |
| C(3')-C(3")-H(3"C) | 109.5 |
| H(3"A)-C(3")-H(3"C) | 109.5 |

| | |
|---|---|
| H(3"B)-C(3")-H(3"C) | 109.5 |
| O(5)-C(3')-O(4) | 122.66(10) |
| O(5)-C(3')-C(3") | 126.09(10) |
| O(4)-C(3')-C(3") | 111.24(9) |
| O(6)-C(4)-C(3) | 107.02(8) |
| O(6)-C(4)-C(5) | 108.63(8) |
| C(3)-C(4)-C(5) | 109.20(8) |
| O(6)-C(4)-H(4) | 110.6 |
| C(3)-C(4)-H(4) | 110.6 |
| C(5)-C(4)-H(4) | 110.6 |
| C(4')-C(4")-H(4"1) | 109.5 |
| C(4')-C(4")-H(4"2) | 109.5 |
| H(4"1)-C(4")-H(4"2) | 109.5 |
| C(4')-C(4")-H(4"3) | 109.5 |
| H(4"1)-C(4")-H(4"3) | 109.5 |
| H(4"2)-C(4")-H(4"3) | 109.5 |
| O(7)-C(4')-O(6) | 123.62(10) |
| O(7)-C(4')-C(4") | 125.84(11) |
| O(6)-C(4')-C(4") | 110.54(11) |
| O(1)-C(5)-C(6) | 107.48(8) |
| O(1)-C(5)-C(4) | 109.11(8) |
| C(6)-C(5)-C(4) | 111.88(8) |
| O(1)-C(5)-H(5) | 109.4 |
| C(6)-C(5)-H(5) | 109.4 |
| C(4)-C(5)-H(5) | 109.4 |
| O(8)-C(6)-C(5) | 106.42(8) |
| O(8)-C(6)-H(6A) | 110.4 |
| C(5)-C(6)-H(6A) | 110.4 |
| O(8)-C(6)-H(6B) | 110.4 |
| C(5)-C(6)-H(6B) | 110.4 |
| H(6A)-C(6)-H(6B) | 108.6 |
| C(6')-C(6")-H(6"A) | 109.5 |
| C(6')-C(6")-H(6"B) | 109.5 |
| H(6"A)-C(6")-H(6"B) | 109.5 |
| C(6')-C(6")-H(6"C) | 109.5 |
| H(6"A)-C(6")-H(6"C) | 109.5 |

| | |
|---|---|
| H(6"B)-C(6")-H(6"C) | 109.5 |
| O(9)-C(6')-O(8) | 122.70(10) |
| O(9)-C(6')-C(6") | 125.81(10) |
| O(8)-C(6')-C(6") | 111.49(9) |
| O(10)-C(7)-C(8) | 111.95(8) |
| O(10)-C(7)-C(1) | 104.30(7) |
| C(8)-C(7)-C(1) | 113.19(8) |
| O(10)-C(7)-H(7) | 109.1 |
| C(8)-C(7)-H(7) | 109.1 |
| C(1)-C(7)-H(7) | 109.1 |
| C(7')-C(7")-H(7"A) | 109.5 |
| C(7')-C(7")-H(7"B) | 109.5 |
| H(7"A)-C(7")-H(7"B) | 109.5 |
| C(7')-C(7")-H(7"C) | 109.5 |
| H(7"A)-C(7")-H(7"C) | 109.5 |
| H(7"B)-C(7")-H(7"C) | 109.5 |
| O(11')-C(7')-O(10) | 122.9(5) |
| O(11)-C(7')-O(10) | 122.7(3) |
| O(11')-C(7')-C(7") | 124.0(5) |
| O(11)-C(7')-C(7") | 125.4(3) |
| O(10)-C(7')-C(7") | 111.08(12) |
| C(14)-C(8)-C(9) | 119.72(9) |
| C(14)-C(8)-C(7) | 120.25(8) |
| C(9)-C(8)-C(7) | 119.98(8) |
| C(11)-C(9)-C(8) | 118.93(9) |
| C(11)-C(9)-C(10) | 118.04(9) |
| C(8)-C(9)-C(10) | 123.03(9) |
| C(9)-C(10)-H(10A) | 109.5 |
| C(9)-C(10)-H(10B) | 109.5 |
| H(10A)-C(10)-H(10B) | 109.5 |
| C(9)-C(10)-H(10C) | 109.5 |
| H(10A)-C(10)-H(10C) | 109.5 |
| H(10B)-C(10)-H(10C) | 109.5 |
| C(12)-C(11)-C(9) | 120.06(9) |
| C(12)-C(11)-H(11) | 120.0 |
| C(9)-C(11)-H(11) | 120.0 |

| | |
|---|---|
| C(11)-C(12)-C(13) | 121.79(9) |
| C(11)-C(12)-Br(1) | 117.87(8) |
| C(13)-C(12)-Br(1) | 120.33(8) |
| C(14)-C(13)-C(12) | 118.03(9) |
| C(14)-C(13)-H(13) | 121.0 |
| C(12)-C(13)-H(13) | 121.0 |
| C(13)-C(14)-C(8) | 121.39(9) |
| C(13)-C(14)-H(14) | 119.3 |
| C(8)-C(14)-H(14) | 119.3 |

Symmetry transformations used to generate equivalent atoms:

**Table S7. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for jj25315.** The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Br(1) | 20(1) | 25(1) | 24(1) | -9(1) | -8(1) | -3(1) |
| O(1) | 15(1) | 10(1) | 15(1) | 0(1) | -2(1) | -3(1) |
| O(2) | 19(1) | 13(1) | 11(1) | -3(1) | -2(1) | -2(1) |
| O(3) | 26(1) | 20(1) | 31(1) | -10(1) | -7(1) | -6(1) |
| O(4) | 16(1) | 17(1) | 16(1) | 0(1) | -4(1) | -5(1) |
| O(5) | 23(1) | 27(1) | 20(1) | -1(1) | 0(1) | -11(1) |
| O(6) | 16(1) | 16(1) | 20(1) | -3(1) | 1(1) | 3(1) |
| O(7) | 48(1) | 26(1) | 15(1) | -5(1) | -6(1) | 19(1) |
| O(8) | 30(1) | 12(1) | 22(1) | 1(1) | -11(1) | -8(1) |
| O(9) | 26(1) | 12(1) | 27(1) | 1(1) | -9(1) | -4(1) |
| O(10) | 21(1) | 13(1) | 15(1) | 2(1) | -2(1) | -5(1) |
| O(11) | 21(1) | 27(1) | 63(5) | 0(2) | 7(2) | -7(1) |
| O(11') | 37(4) | 30(2) | 50(5) | -5(3) | 21(3) | -13(3) |
| C(1) | 16(1) | 10(1) | 11(1) | -1(1) | -2(1) | -3(1) |
| C(2) | 16(1) | 12(1) | 12(1) | -1(1) | -2(1) | -3(1) |
| C(2") | 36(1) | 27(1) | 17(1) | -10(1) | -5(1) | 4(1) |
| C(2') | 22(1) | 16(1) | 17(1) | -6(1) | -8(1) | 1(1) |
| C(3) | 15(1) | 14(1) | 14(1) | -1(1) | -2(1) | -3(1) |
| C(3") | 26(1) | 26(1) | 29(1) | 3(1) | -14(1) | -9(1) |
| C(3') | 15(1) | 16(1) | 21(1) | -2(1) | -3(1) | -4(1) |
| C(4) | 14(1) | 11(1) | 15(1) | -1(1) | -1(1) | 0(1) |
| C(4") | 21(1) | 22(1) | 48(1) | -14(1) | -14(1) | 9(1) |
| C(4') | 21(1) | 15(1) | 22(1) | -8(1) | -10(1) | 5(1) |
| C(5) | 16(1) | 11(1) | 15(1) | -2(1) | -2(1) | -2(1) |
| C(6) | 22(1) | 12(1) | 18(1) | 1(1) | -6(1) | -6(1) |
| C(6") | 37(1) | 21(1) | 28(1) | -1(1) | -15(1) | -9(1) |
| C(6') | 19(1) | 12(1) | 21(1) | -2(1) | -4(1) | -4(1) |
| C(7) | 16(1) | 11(1) | 12(1) | -1(1) | -2(1) | -2(1) |
| C(7") | 43(1) | 20(1) | 31(1) | 4(1) | -2(1) | -14(1) |
| C(7') | 28(1) | 19(1) | 25(1) | -1(1) | 4(1) | -10(1) |
| C(8) | 14(1) | 13(1) | 11(1) | -3(1) | -1(1) | 0(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(9) | 13(1) | 16(1) | 13(1) | -4(1) | -1(1) | 0(1) |
| C(10) | 13(1) | 28(1) | 24(1) | -13(1) | -1(1) | 1(1) |
| C(11) | 15(1) | 17(1) | 15(1) | -6(1) | -2(1) | 0(1) |
| C(12) | 15(1) | 18(1) | 15(1) | -4(1) | -4(1) | -1(1) |
| C(13) | 14(1) | 23(1) | 19(1) | -8(1) | -3(1) | 1(1) |
| C(14) | 14(1) | 19(1) | 16(1) | -7(1) | -2(1) | 1(1) |

**Table S8. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for jj25315.**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1) | 2845 | 7124 | 5471 | 15 |
| H(2) | 5633 | 7793 | 5758 | 16 |
| H(2"A) | 2372 | 9016 | 9334 | 39 |
| H(2"B) | 3948 | 9145 | 10115 | 39 |
| H(2"C) | 3129 | 7536 | 10137 | 39 |
| H(3) | 7155 | 5562 | 5458 | 17 |
| H(3"A) | 8832 | 7119 | 9055 | 39 |
| H(3"B) | 10369 | 7838 | 7887 | 39 |
| H(3"C) | 10306 | 6056 | 8368 | 39 |
| H(4) | 4966 | 4013 | 7916 | 16 |
| H(4"1) | 8795 | 477 | 8652 | 43 |
| H(4"2) | 9653 | 1785 | 7457 | 43 |
| H(4"3) | 8606 | 500 | 6995 | 43 |
| H(5) | 5067 | 3788 | 4869 | 17 |
| H(6A) | 4351 | 1469 | 6480 | 21 |
| H(6B) | 2724 | 2326 | 7244 | 21 |
| H(6"A) | 492 | 1414 | 3847 | 41 |
| H(6"B) | 468 | -379 | 4250 | 41 |
| H(6"C) | 2011 | 409 | 3139 | 41 |
| H(7) | 5734 | 6101 | 3611 | 16 |
| H(7"A) | 5174 | 10772 | 3067 | 48 |
| H(7"B) | 7181 | 10782 | 2771 | 48 |
| H(7"C) | 6191 | 10782 | 1451 | 48 |
| H(10A) | 6777 | 5542 | 1318 | 32 |
| H(10B) | 6504 | 3964 | 771 | 32 |
| H(10C) | 6572 | 4033 | 2441 | 32 |
| H(11) | 3865 | 3358 | 510 | 19 |
| H(13) | -332 | 5431 | 2401 | 22 |
| H(14) | 1304 | 6691 | 3669 | 20 |

**Table S9. Torsion angles [°] for jj25315.**

| | |
|---|---|
| C(5)-O(1)-C(1)-C(2) | -58.95(10) |
| C(5)-O(1)-C(1)-C(7) | 65.93(10) |
| C(2')-O(2)-C(2)-C(1) | -136.07(9) |
| C(2')-O(2)-C(2)-C(3) | 103.29(9) |
| O(1)-C(1)-C(2)-O(2) | -69.46(9) |
| C(7)-C(1)-C(2)-O(2) | 164.73(7) |
| O(1)-C(1)-C(2)-C(3) | 49.92(10) |
| C(7)-C(1)-C(2)-C(3) | -75.90(9) |
| C(2)-O(2)-C(2')-O(3) | -6.07(15) |
| C(2)-O(2)-C(2')-C(2") | 173.29(9) |
| C(3')-O(4)-C(3)-C(4) | 152.01(9) |
| C(3')-O(4)-C(3)-C(2) | -86.06(10) |
| O(2)-C(2)-C(3)-O(4) | -50.01(10) |
| C(1)-C(2)-C(3)-O(4) | -168.12(7) |
| O(2)-C(2)-C(3)-C(4) | 69.17(10) |
| C(1)-C(2)-C(3)-C(4) | -48.95(10) |
| C(3)-O(4)-C(3')-O(5) | 0.53(16) |
| C(3)-O(4)-C(3')-C(3") | -178.75(10) |
| C(4')-O(6)-C(4)-C(3) | 120.84(9) |
| C(4')-O(6)-C(4)-C(5) | -121.38(9) |
| O(4)-C(3)-C(4)-O(6) | -67.85(10) |
| C(2)-C(3)-C(4)-O(6) | 170.74(8) |
| O(4)-C(3)-C(4)-C(5) | 174.75(8) |
| C(2)-C(3)-C(4)-C(5) | 53.33(10) |
| C(4)-O(6)-C(4')-O(7) | 6.56(17) |
| C(4)-O(6)-C(4')-C(4") | -172.87(9) |
| C(1)-O(1)-C(5)-C(6) | -174.86(8) |
| C(1)-O(1)-C(5)-C(4) | 63.64(10) |
| O(6)-C(4)-C(5)-O(1) | -175.67(7) |
| C(3)-C(4)-C(5)-O(1) | -59.28(10) |
| O(6)-C(4)-C(5)-C(6) | 65.53(10) |
| C(3)-C(4)-C(5)-C(6) | -178.08(8) |
| C(6')-O(8)-C(6)-C(5) | 167.55(9) |
| O(1)-C(5)-C(6)-O(8) | 68.60(10) |

| | |
|---|---|
| C(4)-C(5)-C(6)-O(8) | -171.65(8) |
| C(6)-O(8)-C(6')-O(9) | -0.66(17) |
| C(6)-O(8)-C(6')-C(6'') | 179.55(11) |
| C(7')-O(10)-C(7)-C(8) | -113.23(11) |
| C(7')-O(10)-C(7)-C(1) | 124.04(10) |
| O(1)-C(1)-C(7)-O(10) | 163.23(8) |
| C(2)-C(1)-C(7)-O(10) | -71.68(9) |
| O(1)-C(1)-C(7)-C(8) | 41.31(11) |
| C(2)-C(1)-C(7)-C(8) | 166.40(8) |
| C(7)-O(10)-C(7')-O(11') | 24(2) |
| C(7)-O(10)-C(7')-O(11) | -1.8(14) |
| C(7)-O(10)-C(7')-C(7'') | -171.87(10) |
| O(10)-C(7)-C(8)-C(14) | -70.87(12) |
| C(1)-C(7)-C(8)-C(14) | 46.67(12) |
| O(10)-C(7)-C(8)-C(9) | 111.62(10) |
| C(1)-C(7)-C(8)-C(9) | -130.85(9) |
| C(14)-C(8)-C(9)-C(11) | -2.56(14) |
| C(7)-C(8)-C(9)-C(11) | 174.97(9) |
| C(14)-C(8)-C(9)-C(10) | 177.51(10) |
| C(7)-C(8)-C(9)-C(10) | -4.96(15) |
| C(8)-C(9)-C(11)-C(12) | 1.40(15) |
| C(10)-C(9)-C(11)-C(12) | -178.67(10) |
| C(9)-C(11)-C(12)-C(13) | 1.10(16) |
| C(9)-C(11)-C(12)-Br(1) | -177.71(8) |
| C(11)-C(12)-C(13)-C(14) | -2.36(16) |
| Br(1)-C(12)-C(13)-C(14) | 176.42(8) |
| C(12)-C(13)-C(14)-C(8) | 1.15(16) |
| C(9)-C(8)-C(14)-C(13) | 1.30(16) |
| C(7)-C(8)-C(14)-C(13) | -176.22(10) |

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Symmetry transformations used to generate equivalent atoms:

**Table S10. Hydrogen bonds for jj25315 [Å and °].**

| D-H...A | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|---|---|---|---|---|
| C(1)-H(1)...O(9)#1 | 1.00 | 2.47 | 3.2835(13) | 138.6 |
| C(3")-H(3"B)...O(9)#2 | 0.98 | 2.49 | 3.4074(15) | 156.6 |
| C(3")-H(3"C)...Br(1)#3 | 0.98 | 3.07 | 3.8929(13) | 142.9 |
| C(6)-H(6A)...O(3)#4 | 0.99 | 2.56 | 3.5102(15) | 160.8 |
| C(6")-H(6"B)...O(5)#5 | 0.98 | 2.51 | 3.4382(16) | 157.8 |
| C(7")-H(7"C)...O(7)#6 | 0.98 | 2.33 | 3.3045(18) | 170.7 |
| C(11)-H(11)...O(7)#7 | 0.95 | 2.43 | 3.3197(14) | 156.3 |

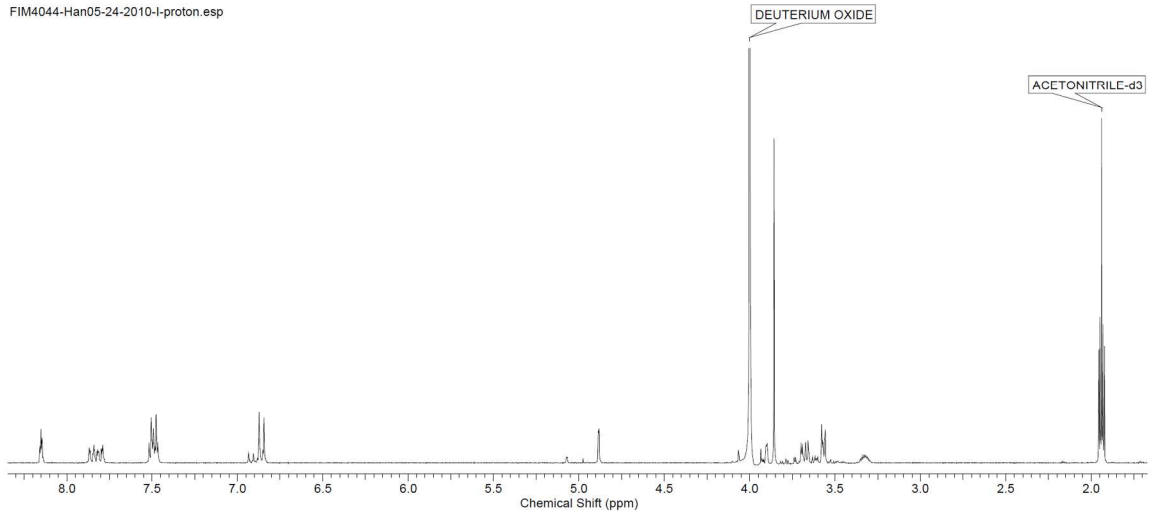
Symmetry transformations used to generate equivalent atoms:

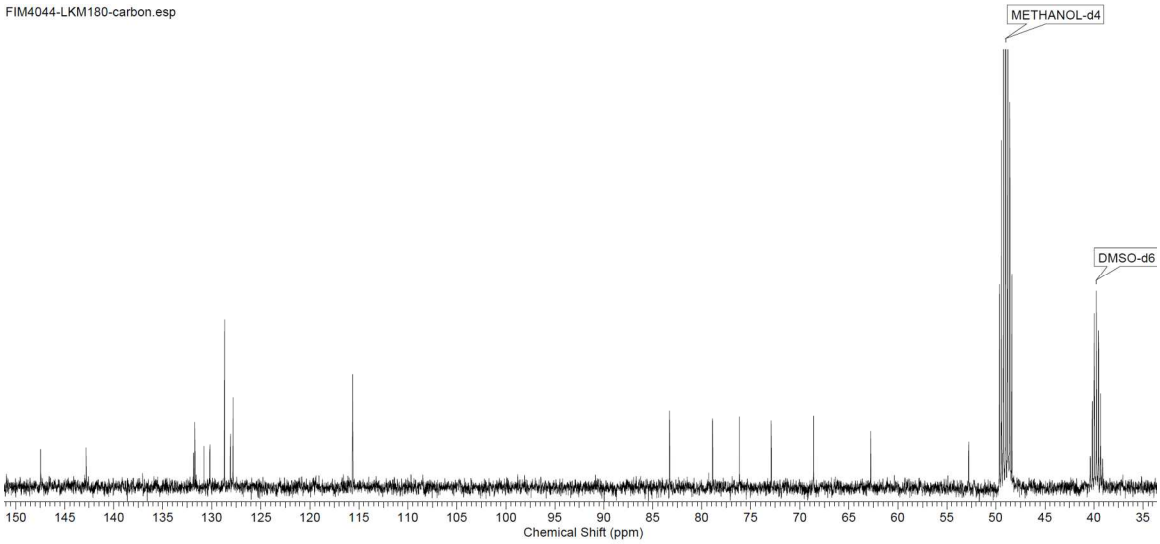
#1 x,y+1,z #2 x+1,y+1,z #3 x+1,y,z+1 #4 x,y-1,z

#5 x-1,y-1,z #6 x,y+1,z-1 #7 x,y,z-1

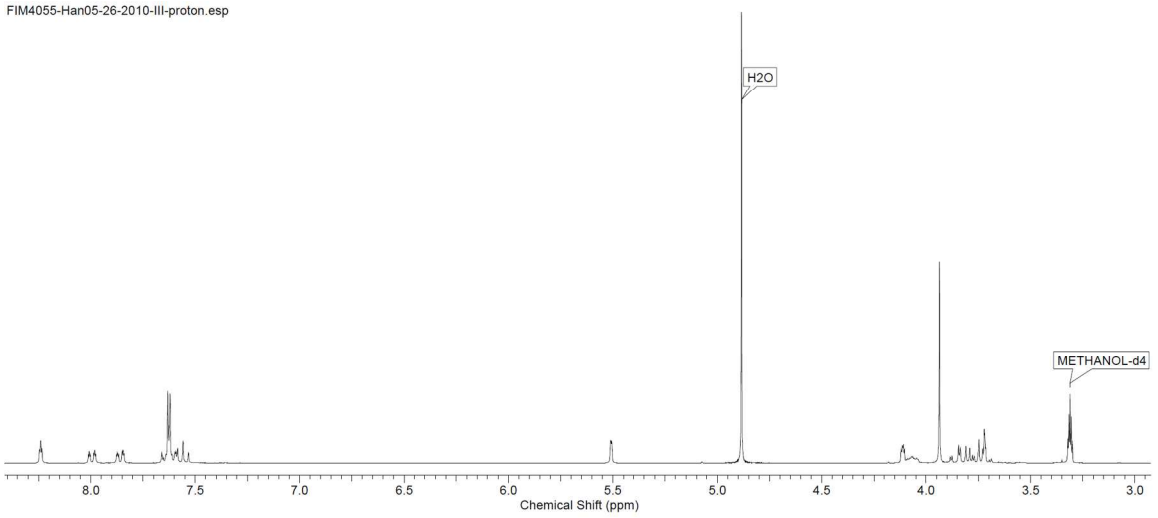
# 6. $^{1}$H and $^{13}$C NMR Spectra of New Compounds

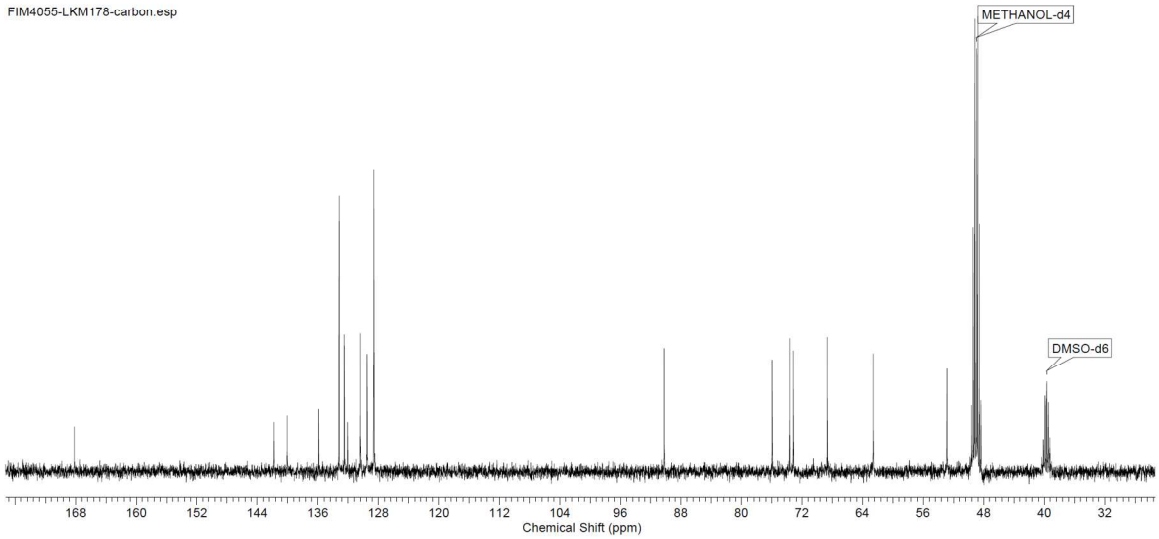
Compound 2



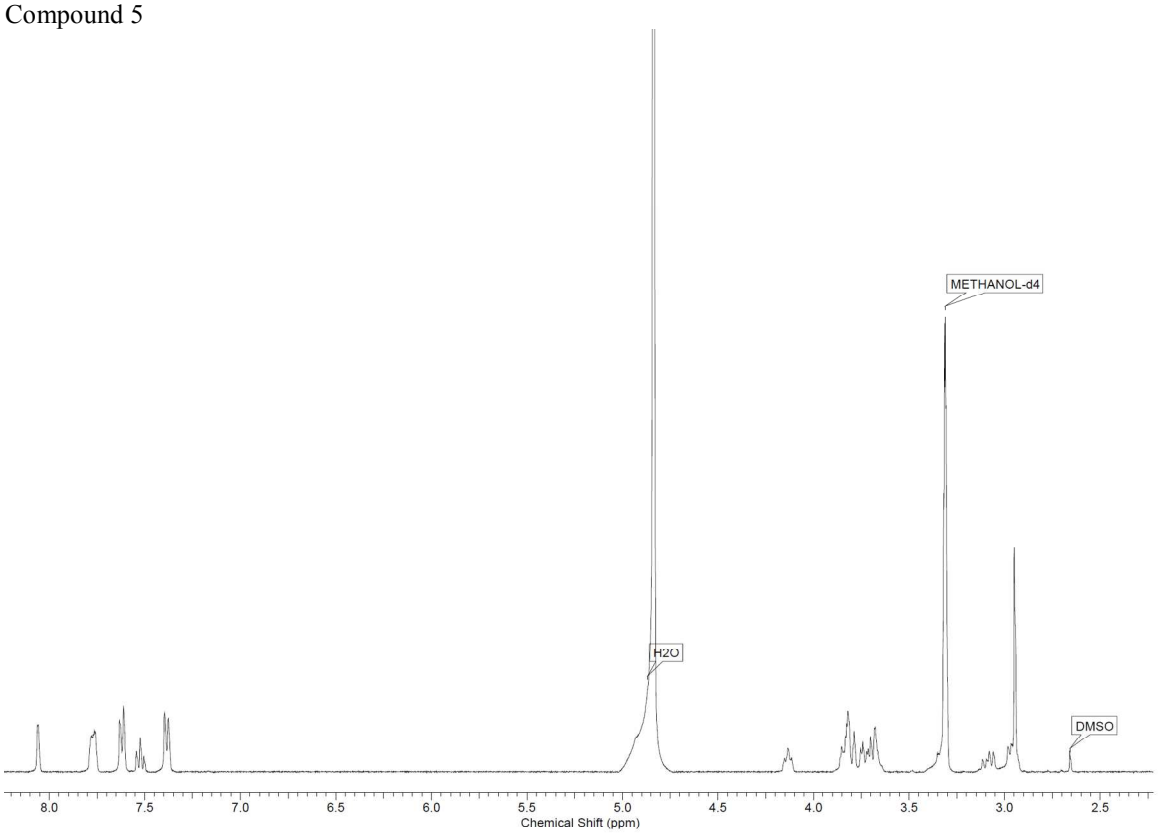



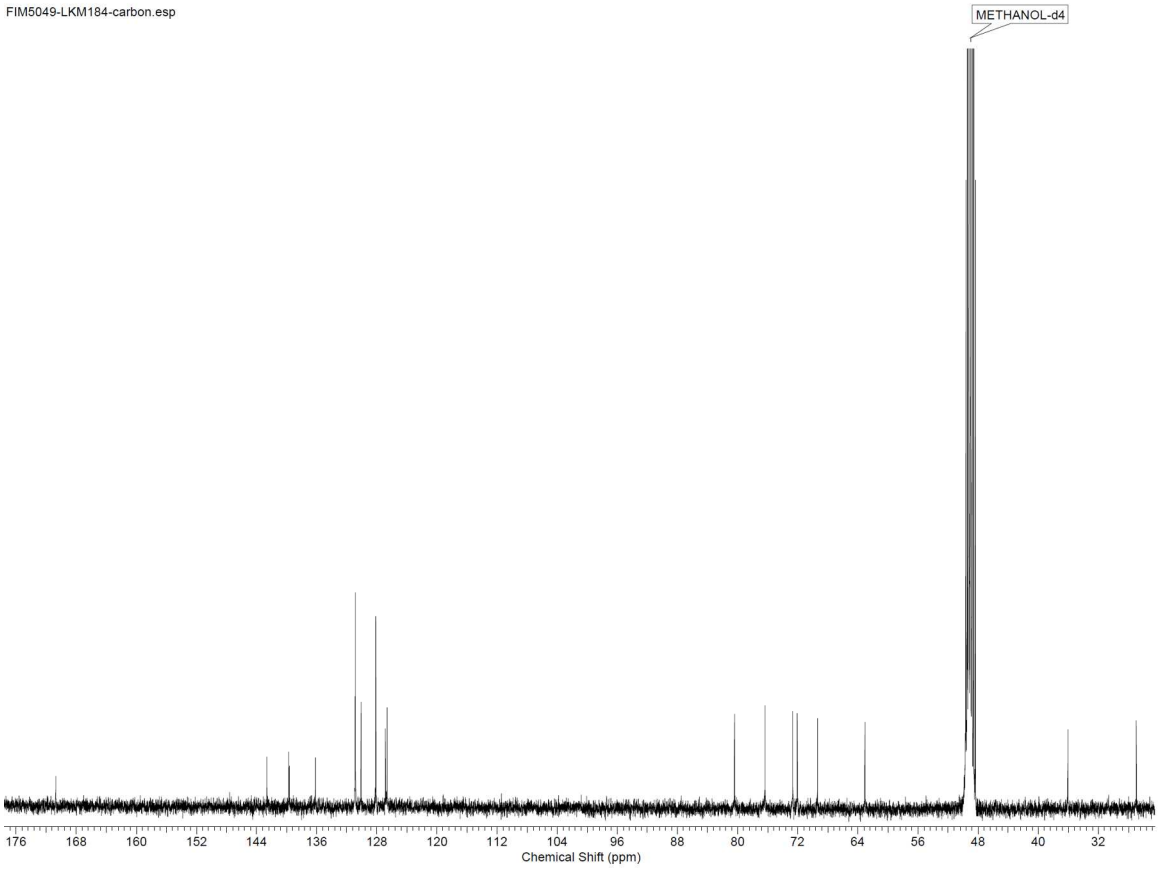

Compound 3


FIM4055-Han05-26-2010-III-proton.esp
H2O
METHANOL-d4
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
Chemical Shift (ppm)

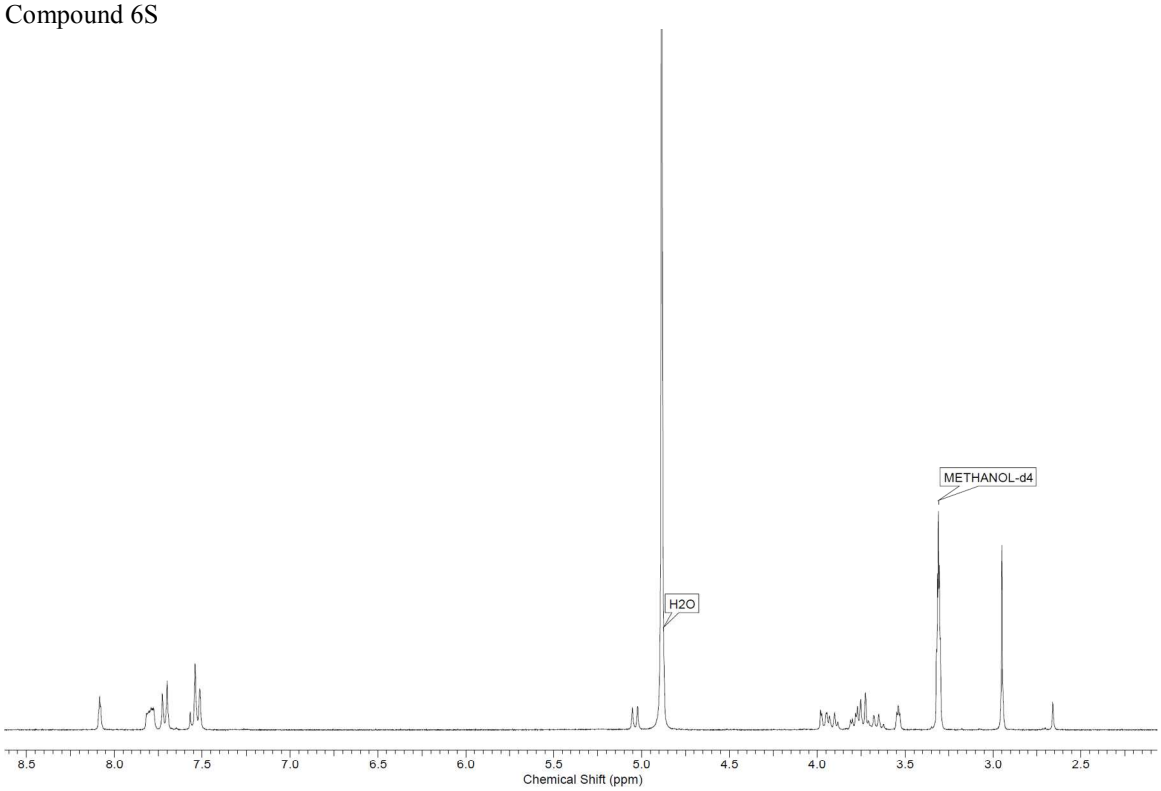

FIM4055-LKM178-carbon.esp
METHANOL-d4
DMSO-d6
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
Chemical Shift (ppm)

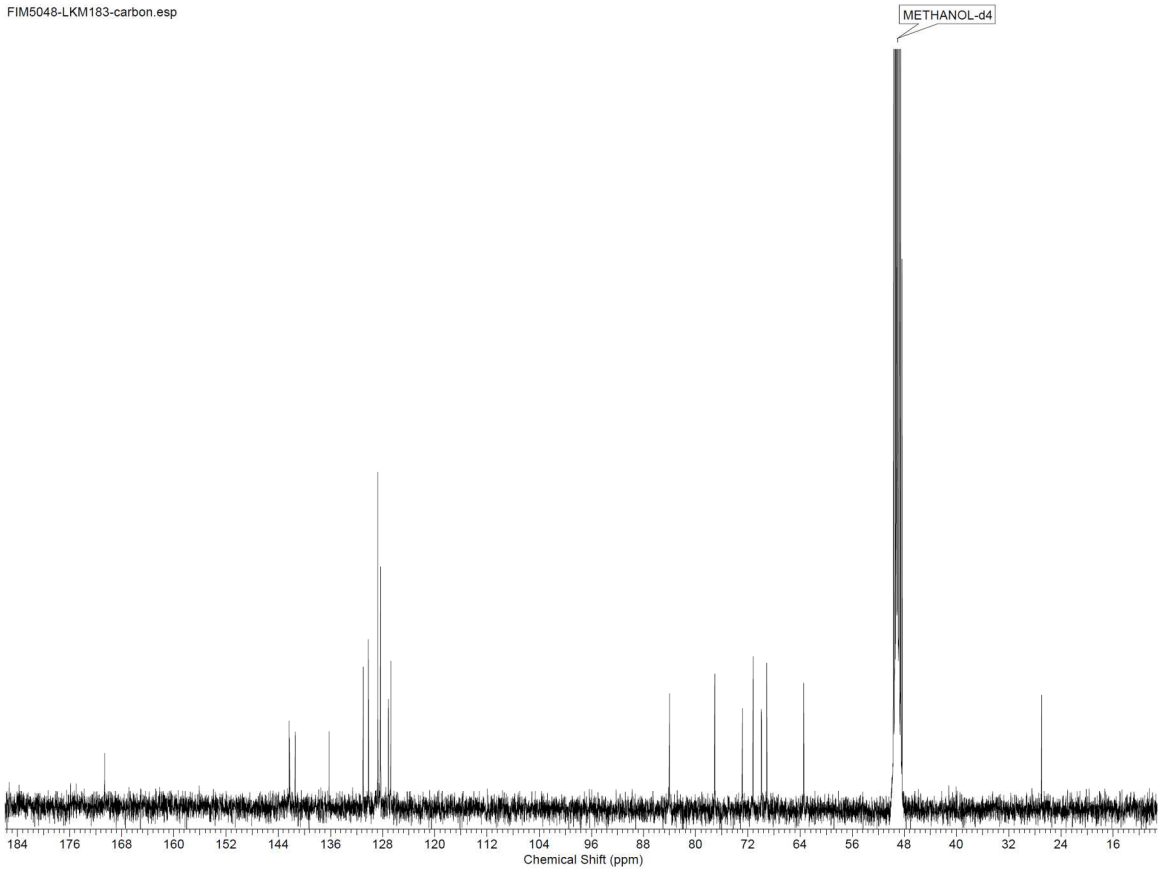
Compound 5


METHANOL-d4
H2O
DMSO
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
Chemical Shift (ppm)

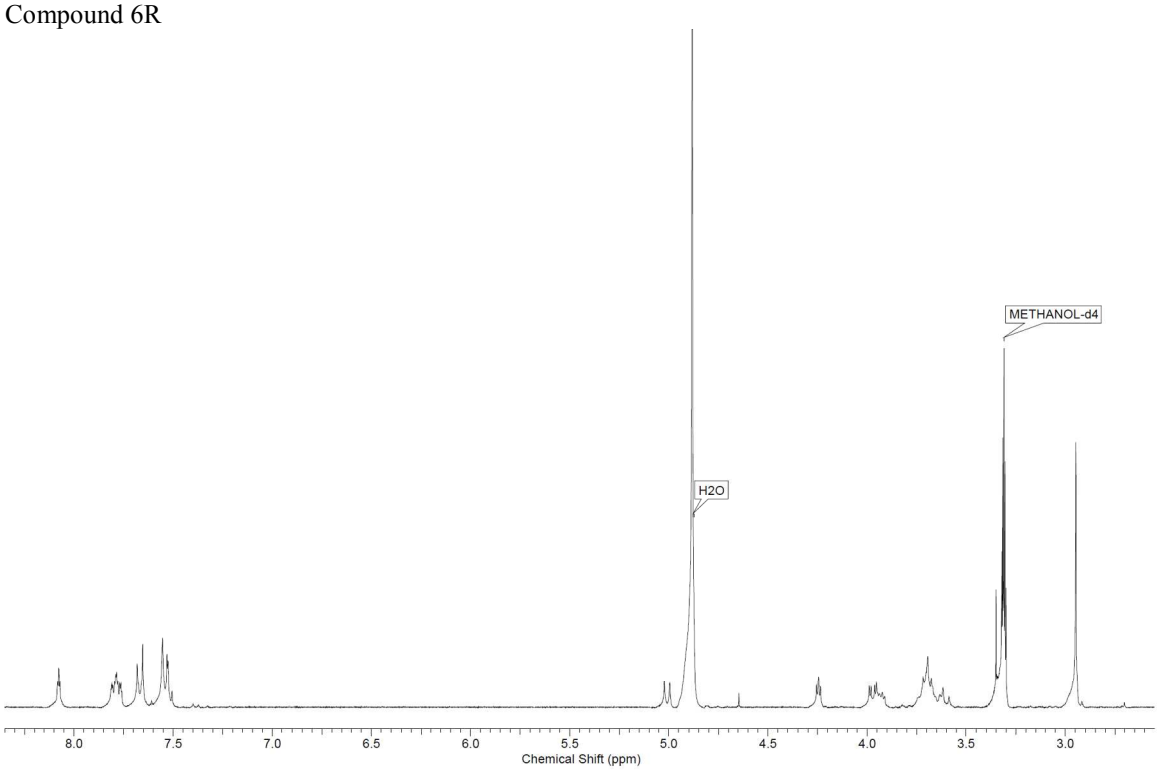

FIM5049-LKM184-carbon.esp
METHANOL-d4
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
Chemical Shift (ppm)

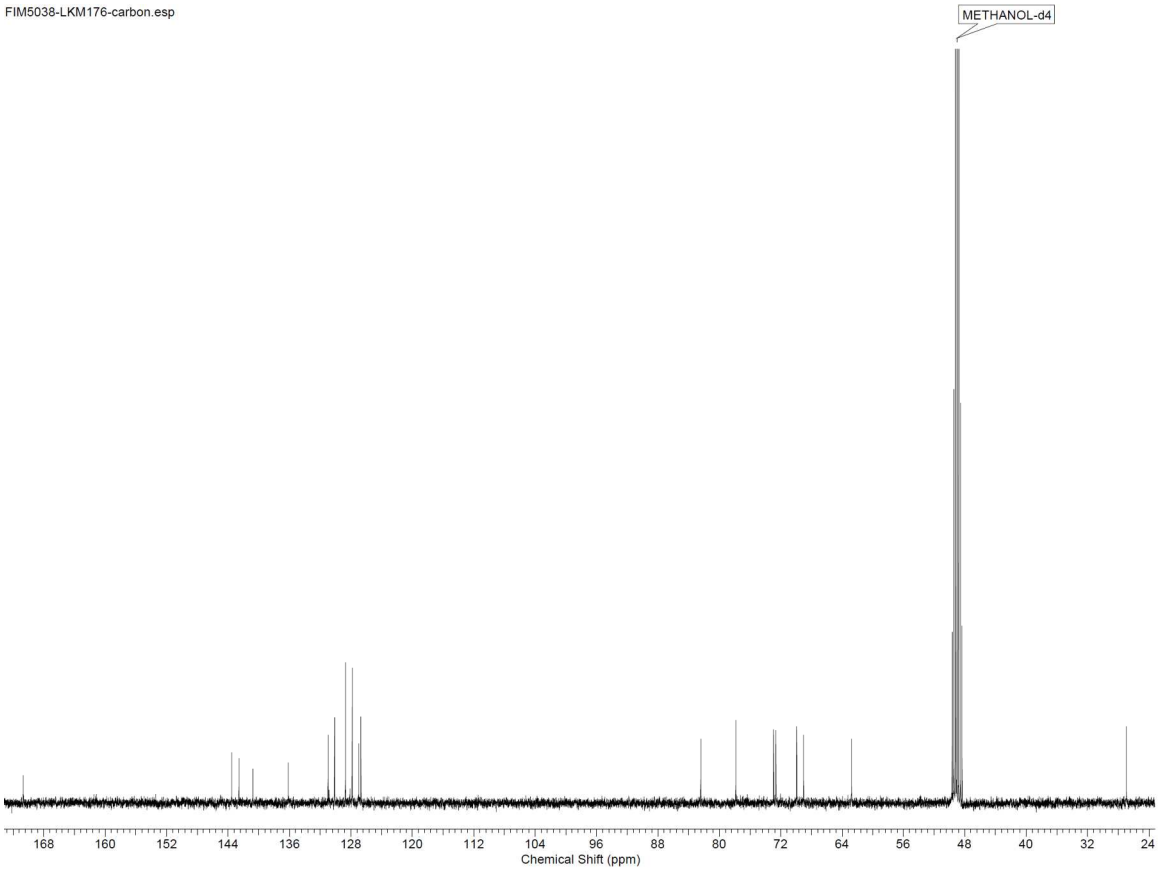
Compound 6S


METHANOL-d4
H2O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
Chemical Shift (ppm)

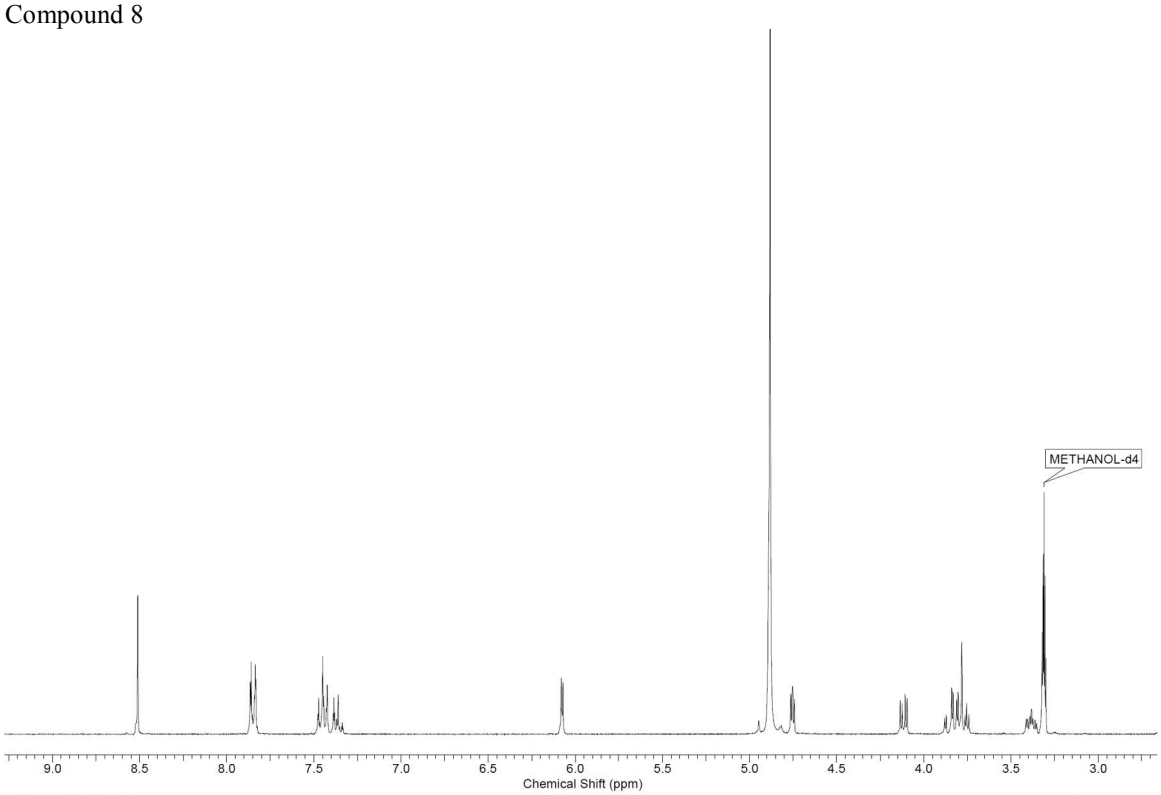

FIM5048-LKM183-carbon.esp
METHANOL-d4
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
Chemical Shift (ppm)

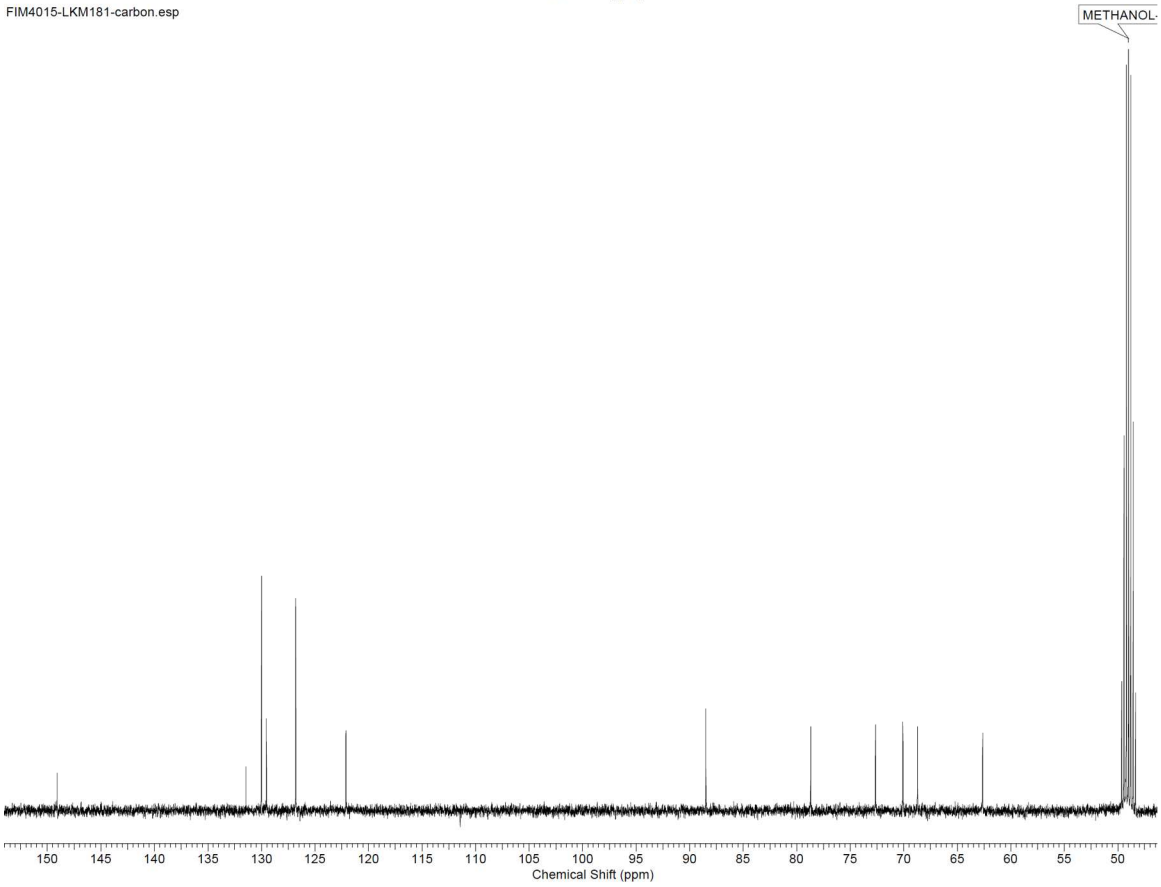
Compound 6R


METHANOL-d4
H2O
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
Chemical Shift (ppm)

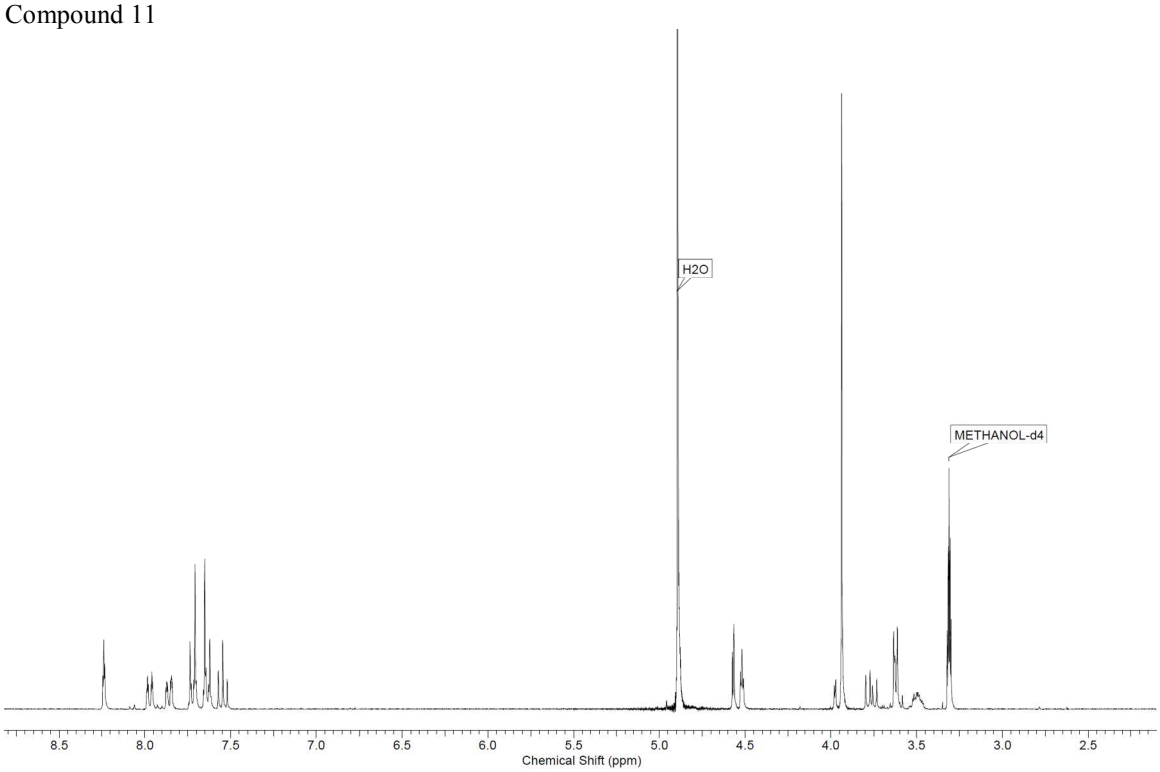

FIM5038-LKM176-carbon.esp
METHANOL-d4
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
Chemical Shift (ppm)

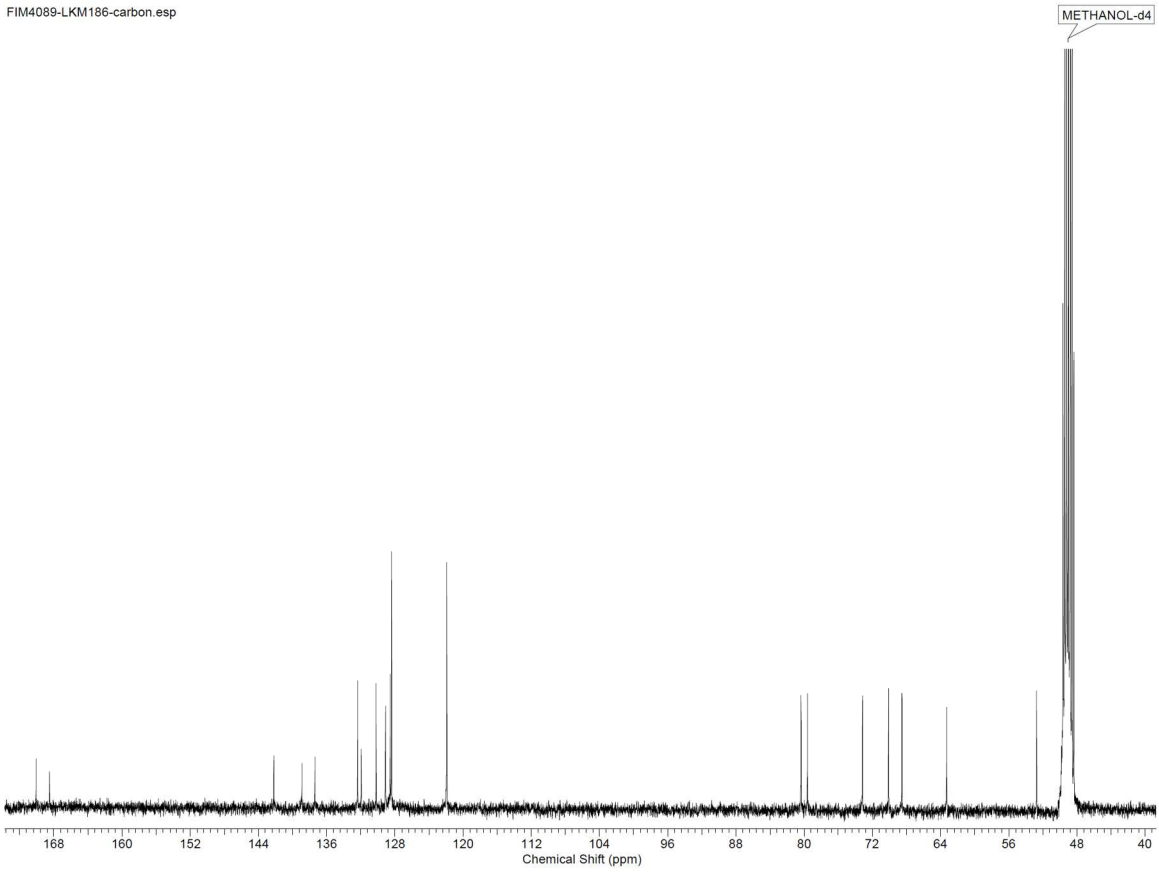
Compound 8


METHANOL-d4
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
Chemical Shift (ppm)

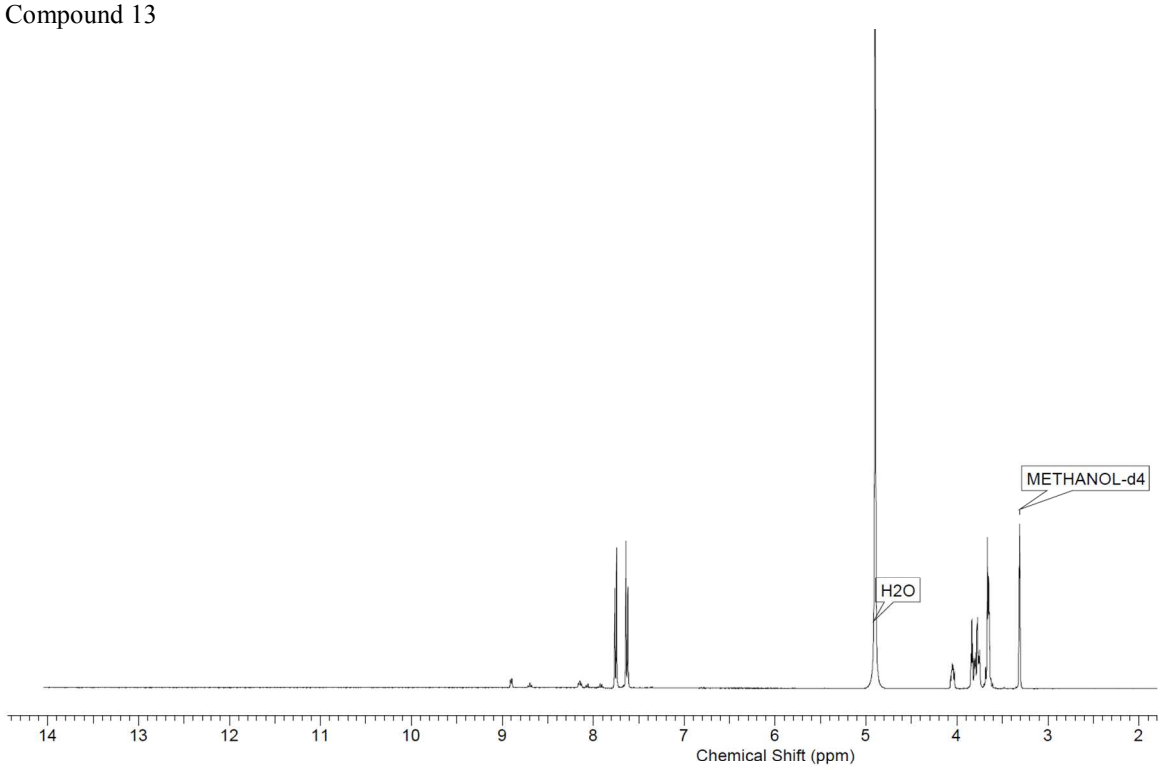

FIM4015-LKM181-carbon.esp
METHANOL-
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
Chemical Shift (ppm)

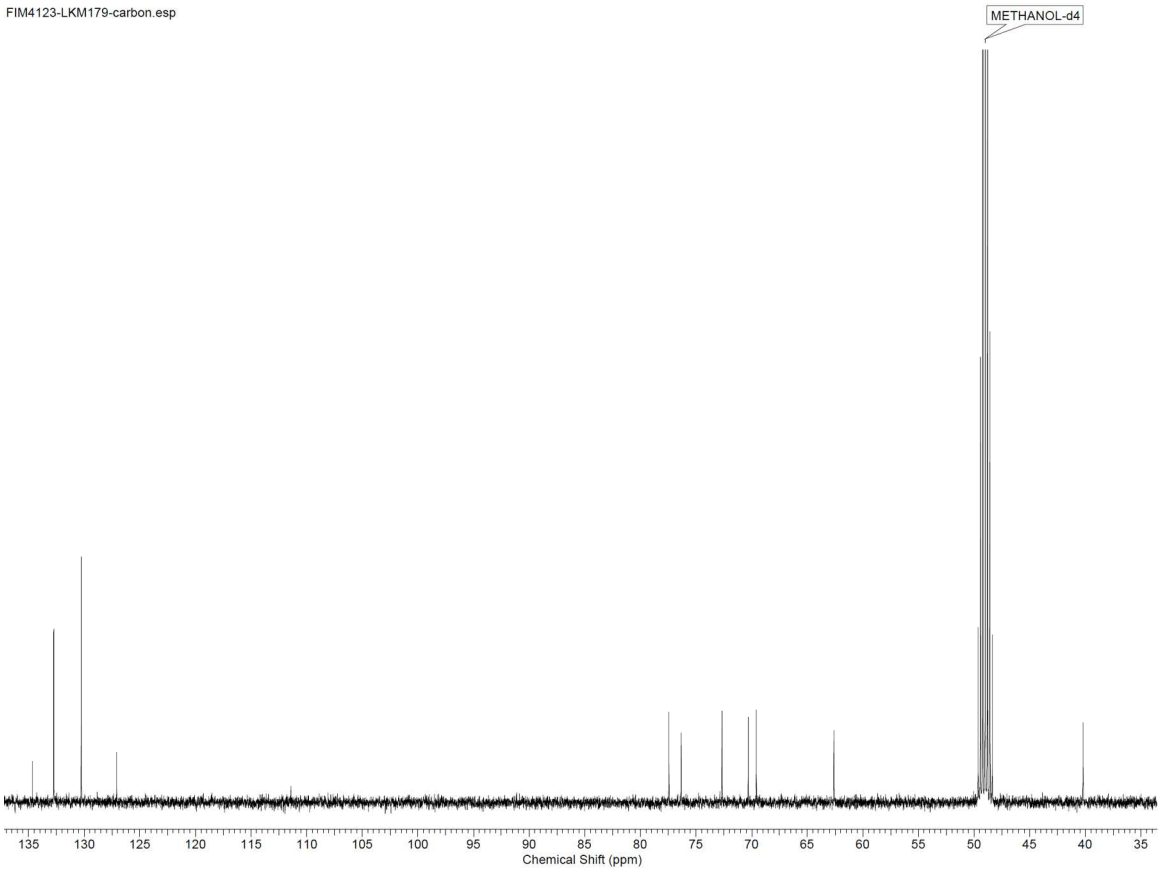
Compound 11


H2O
METHANOL-d4
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
Chemical Shift (ppm)

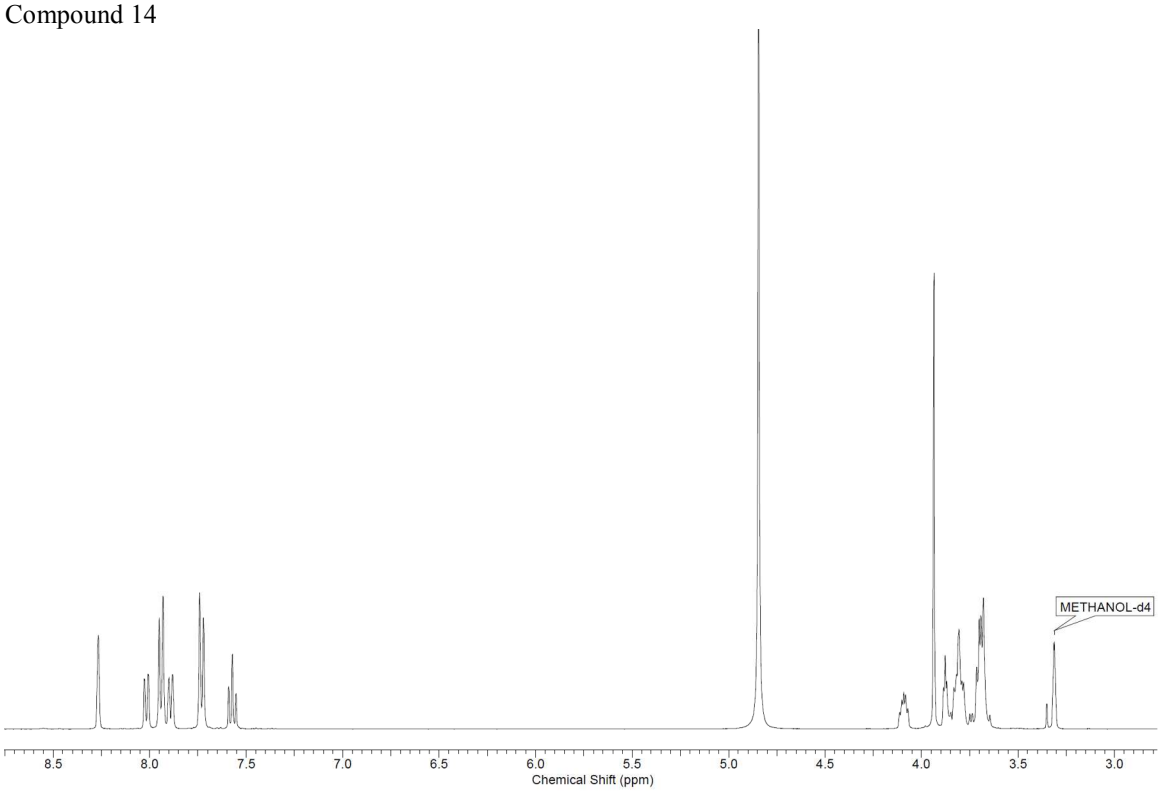

FIM4089-LKM186-carbon.esp
METHANOL-d4
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
Chemical Shift (ppm)

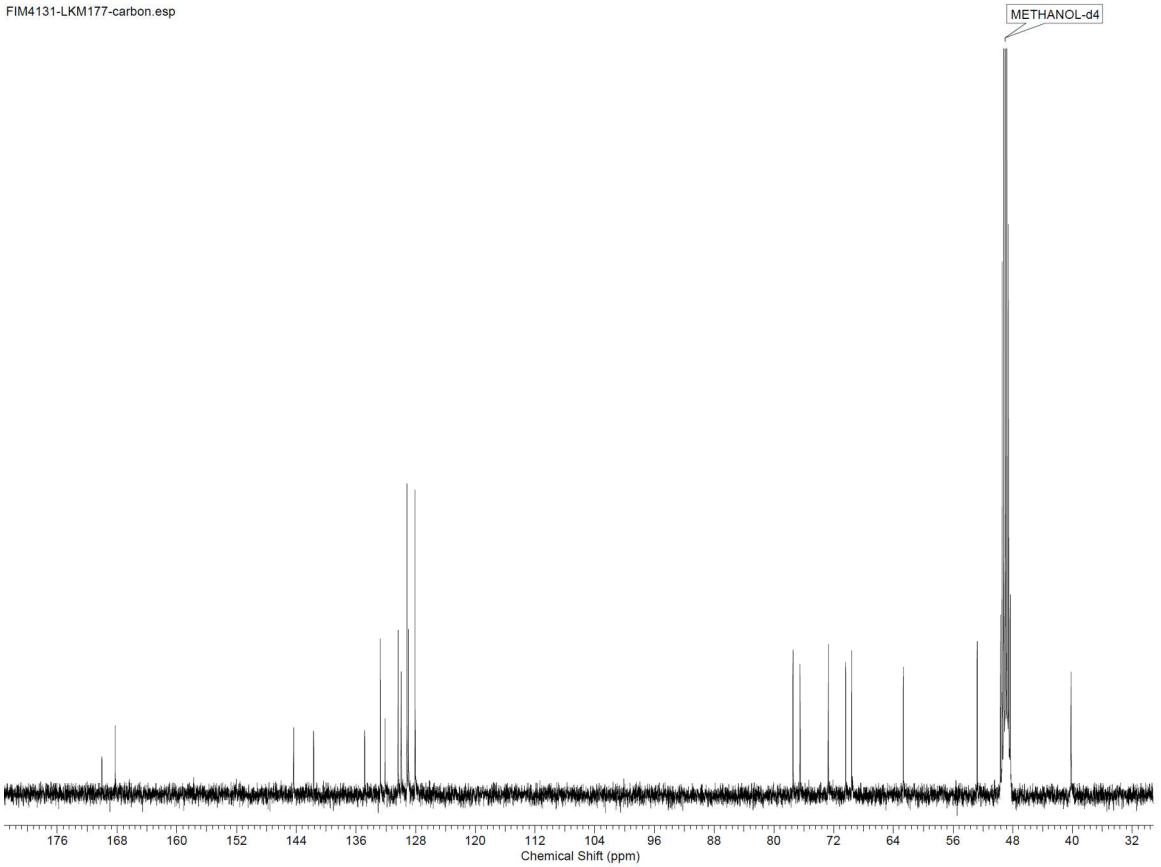
Compound 13


METHANOL-d4
H2O
14
13
12
11
10
9
8
7
6
5
4
3
2
Chemical Shift (ppm)

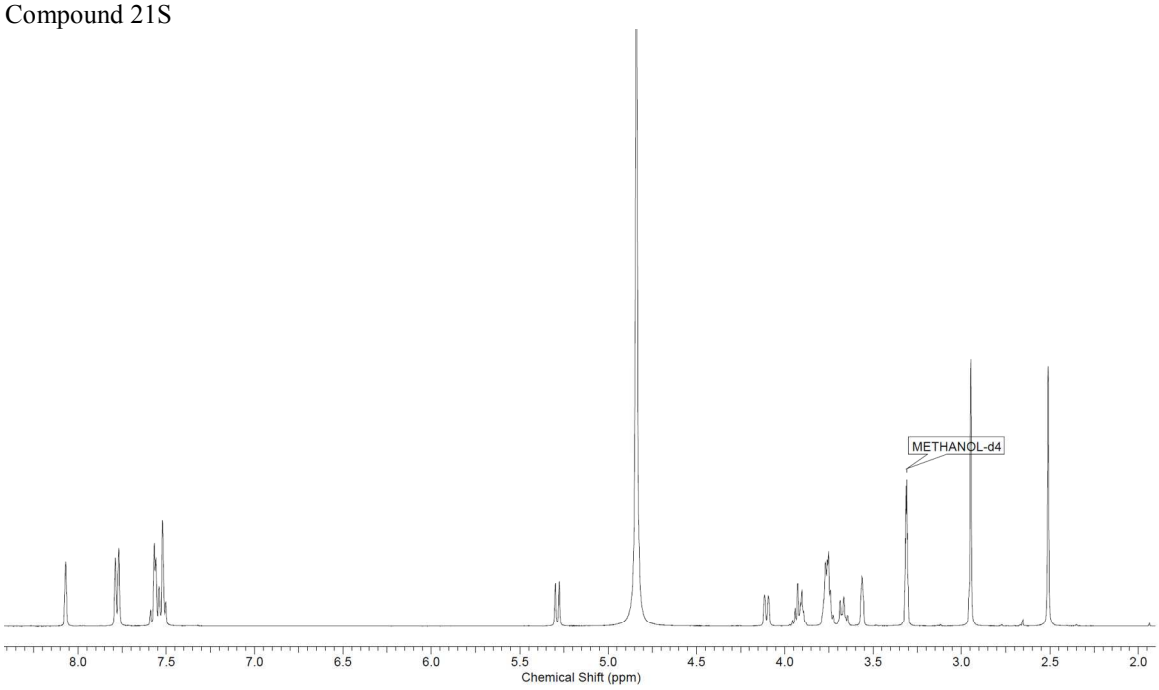

FIM4123-LKM179-carbon.esp
METHANOL-d4
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
Chemical Shift (ppm)

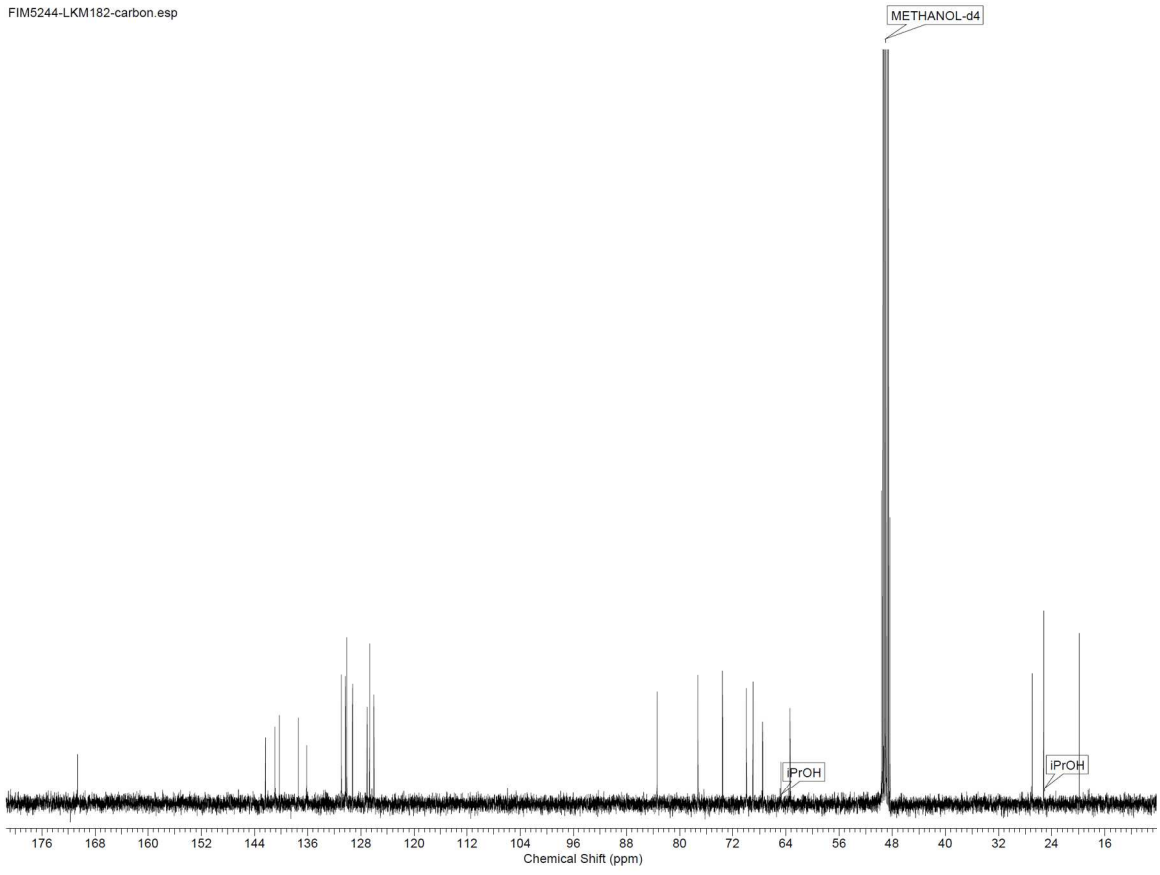
Compound 14


METHANOL-d4
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
Chemical Shift (ppm)

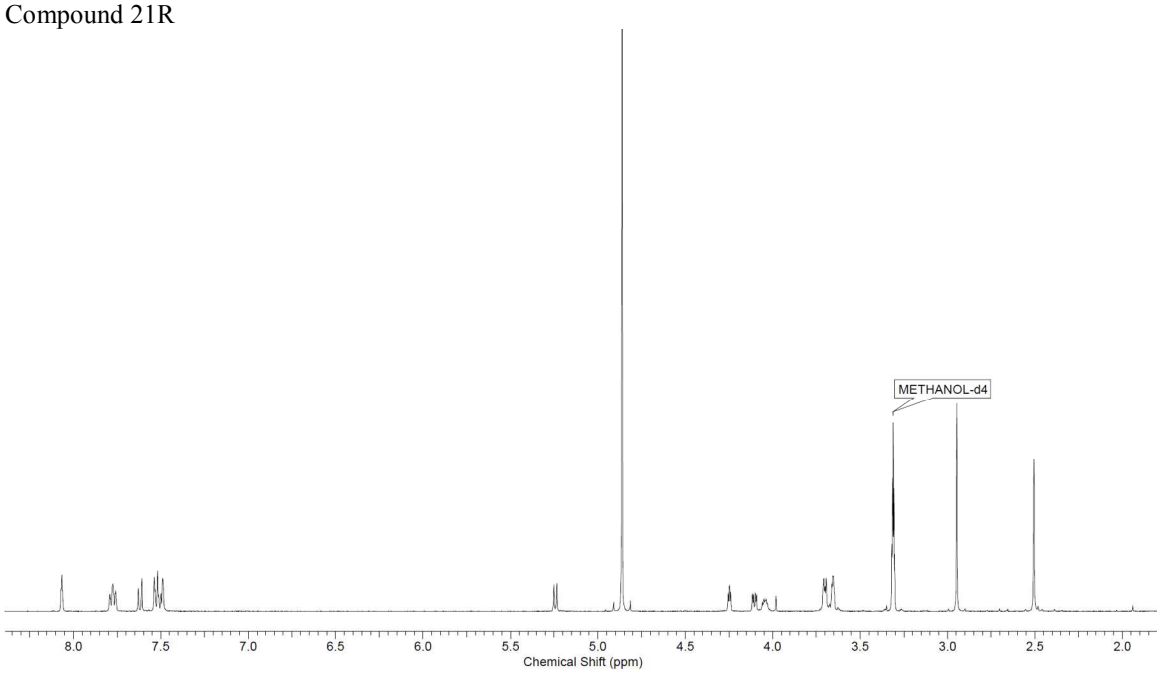

FIM4131-LKM177-carbon.esp
METHANOL-d4
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
Chemical Shift (ppm)

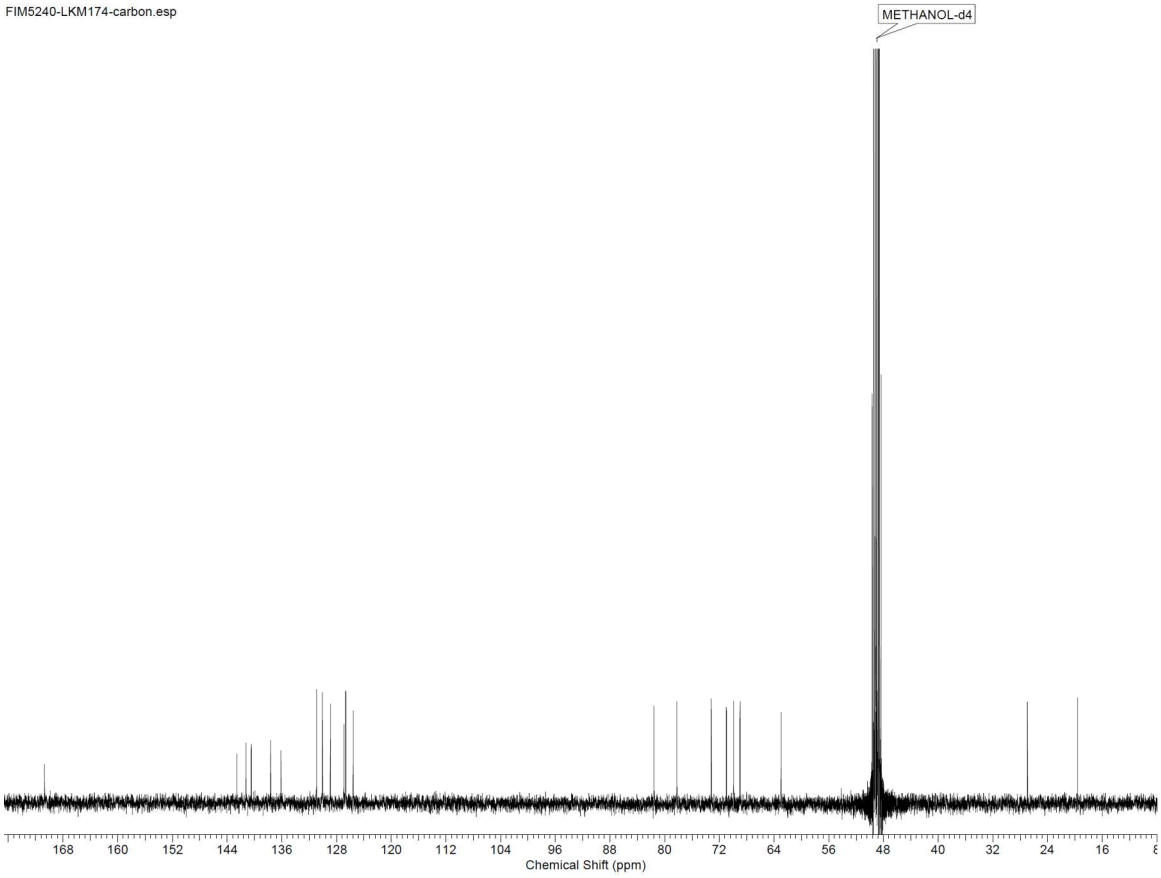
Compound 21S



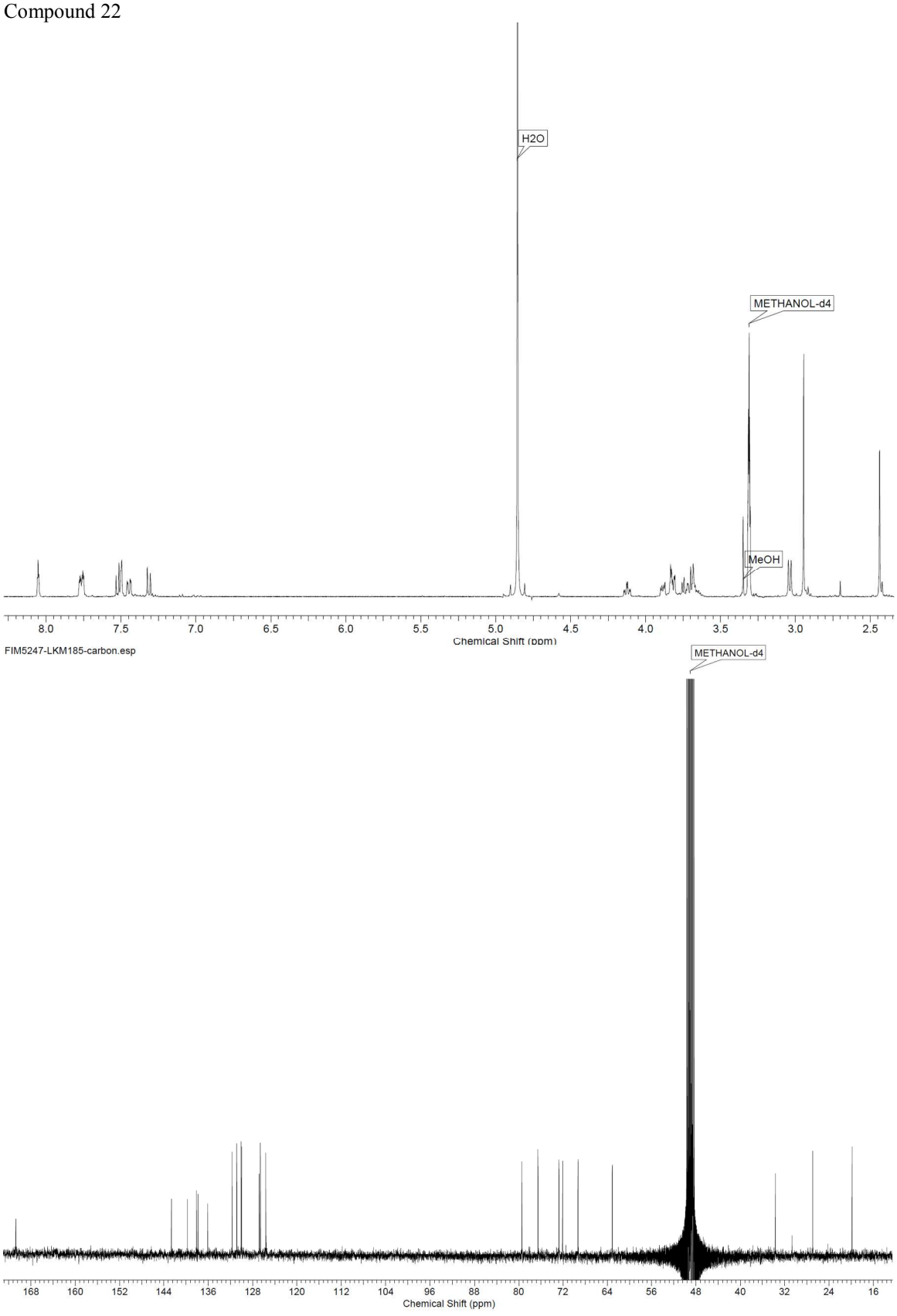



Compound 21R



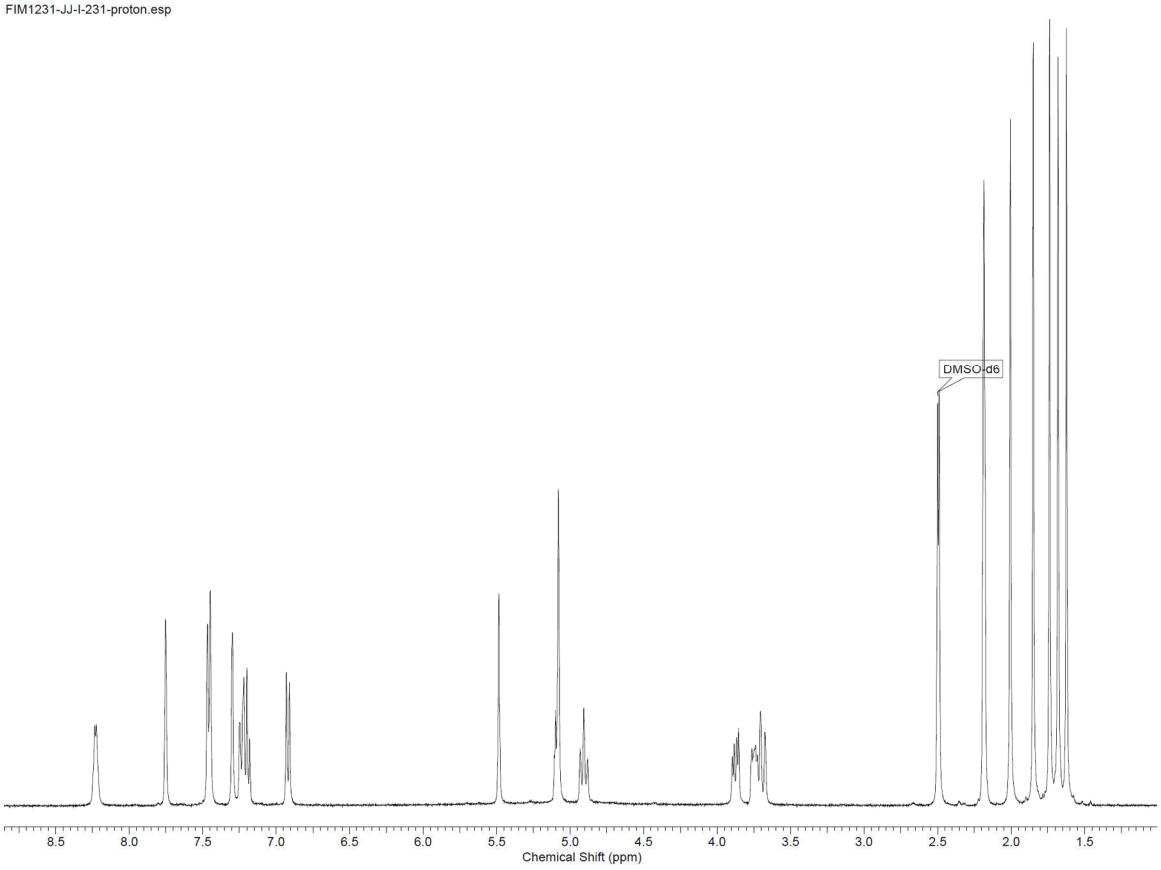



Compound 22

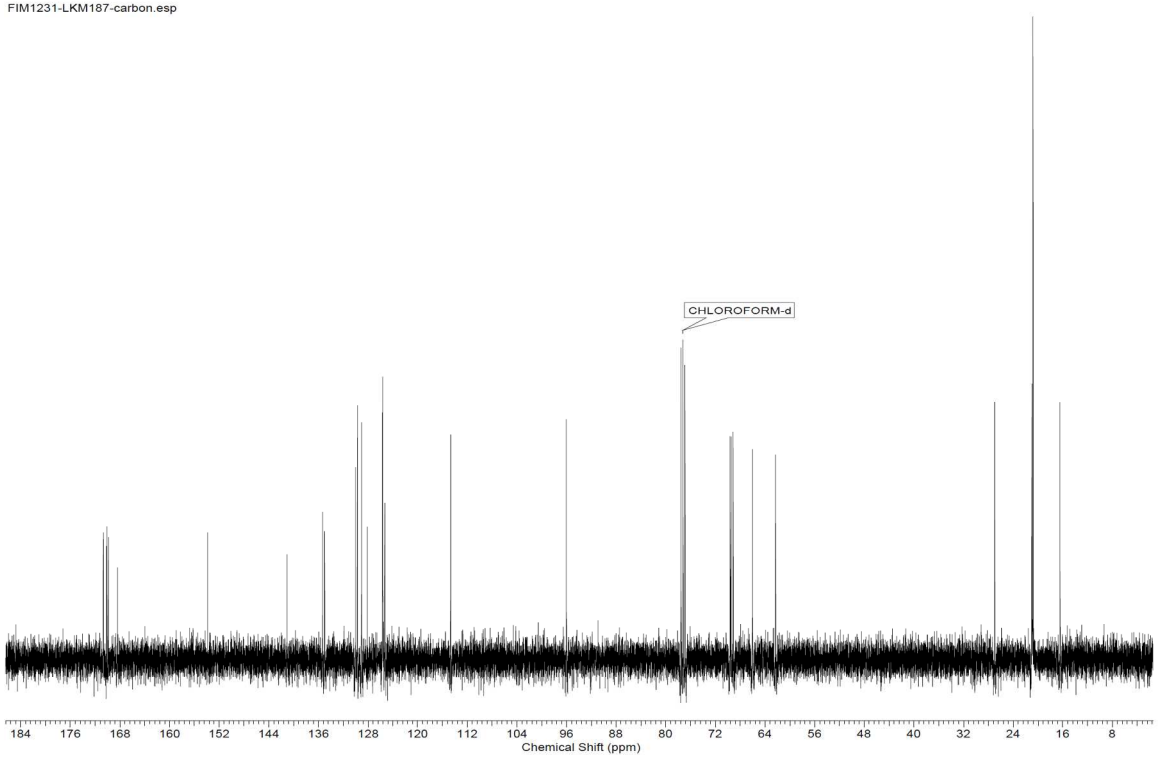

H2O
METHANOL-d4
MeOH
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
Chemical Shift (ppm)
FIM5247-LKM185-carbon.esp
METHANOL-d4
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
Chemical Shift (ppm)

Compound 23a

FIM1231-JJ-I-231-proton.esp

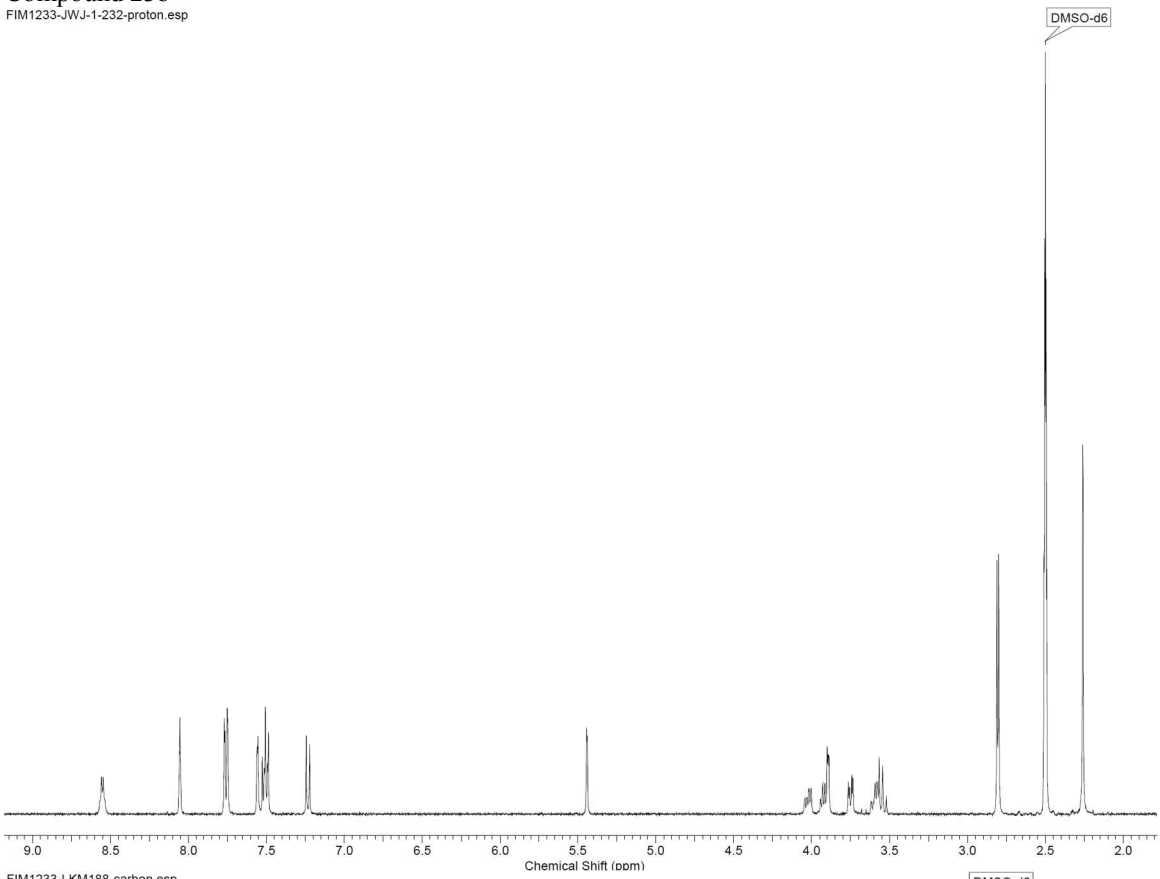



FIM1231-LKM187-carbon.esp

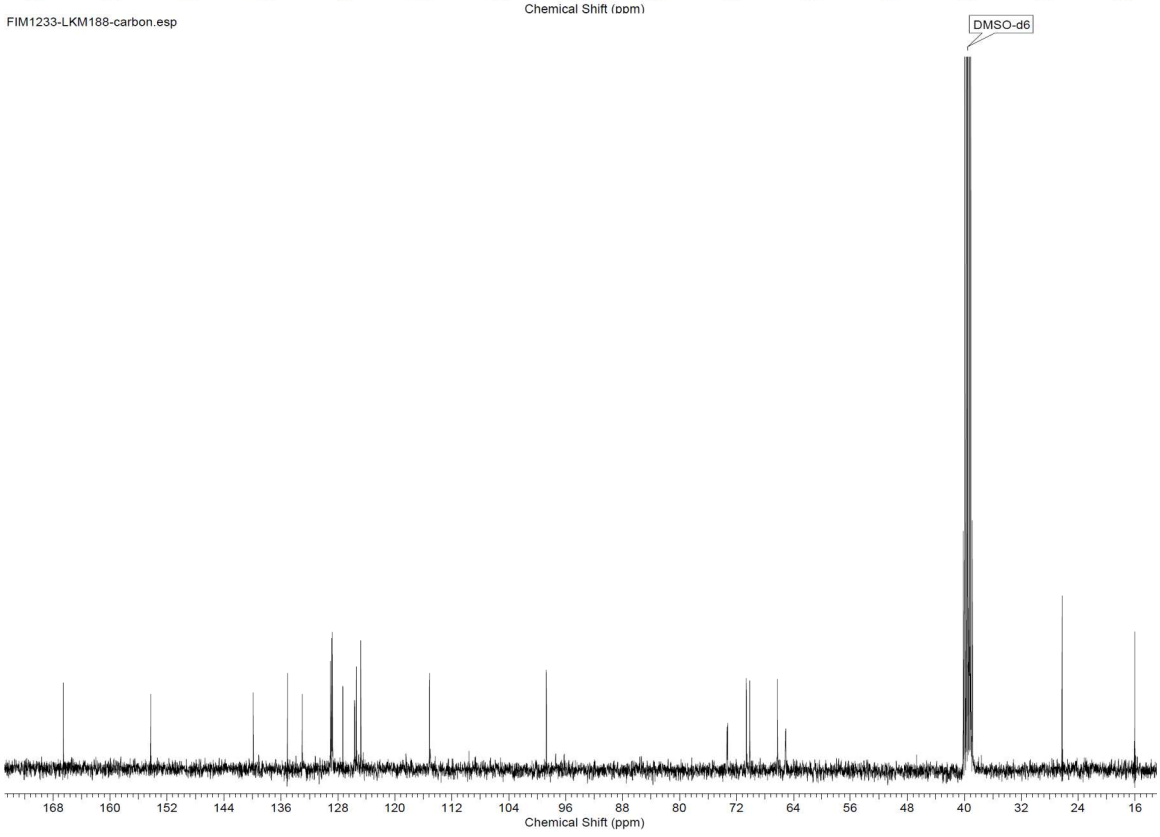


Compound 23b

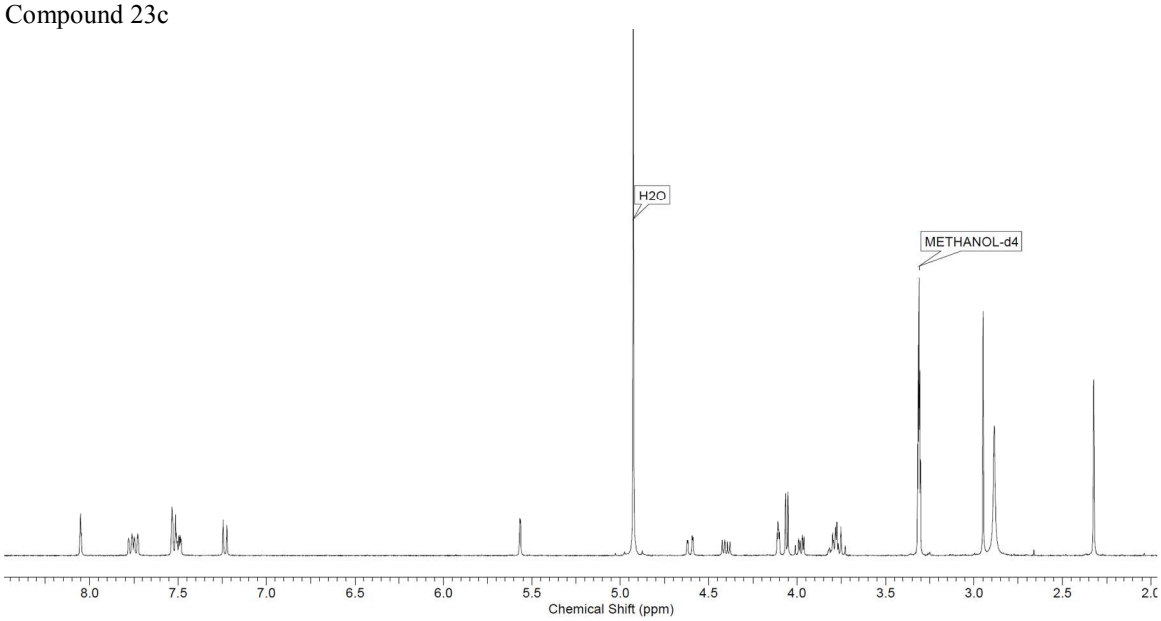
FIM1233-JWJ-1-232-proton.esp

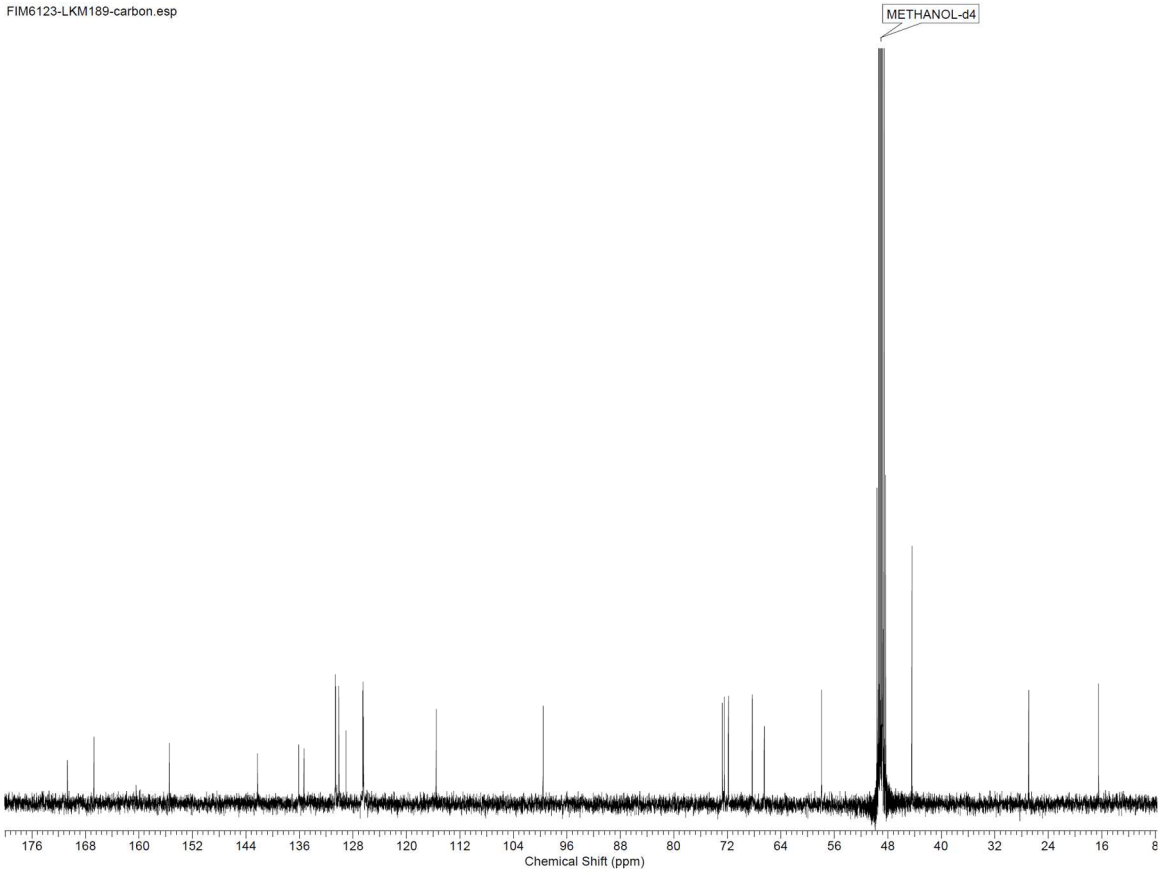



FIM1233-LKM188-carbon.esp

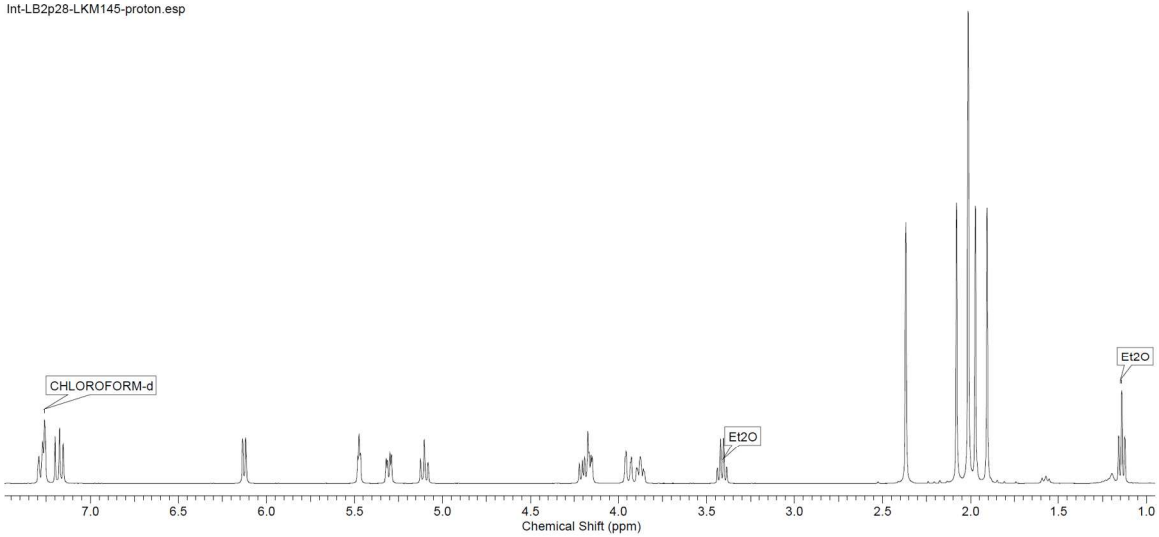


Compound 23c

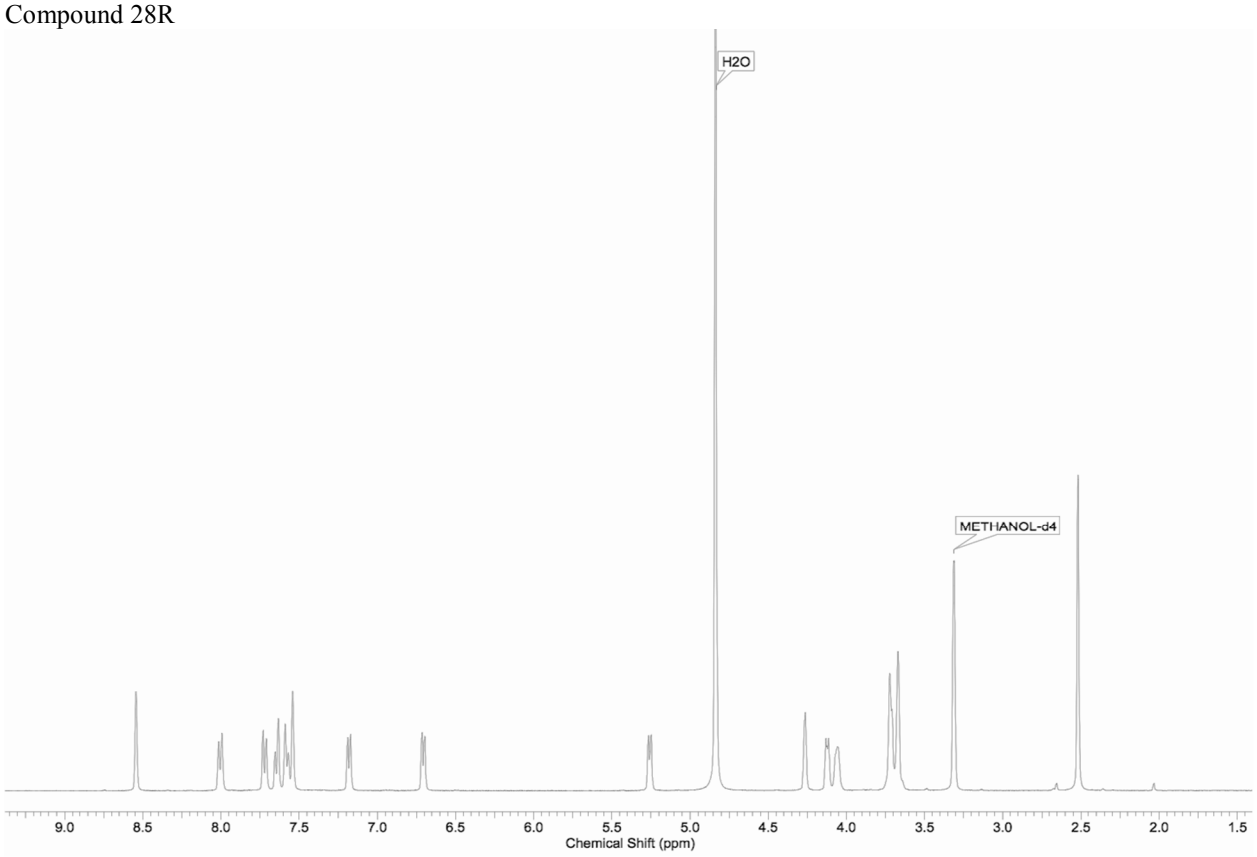


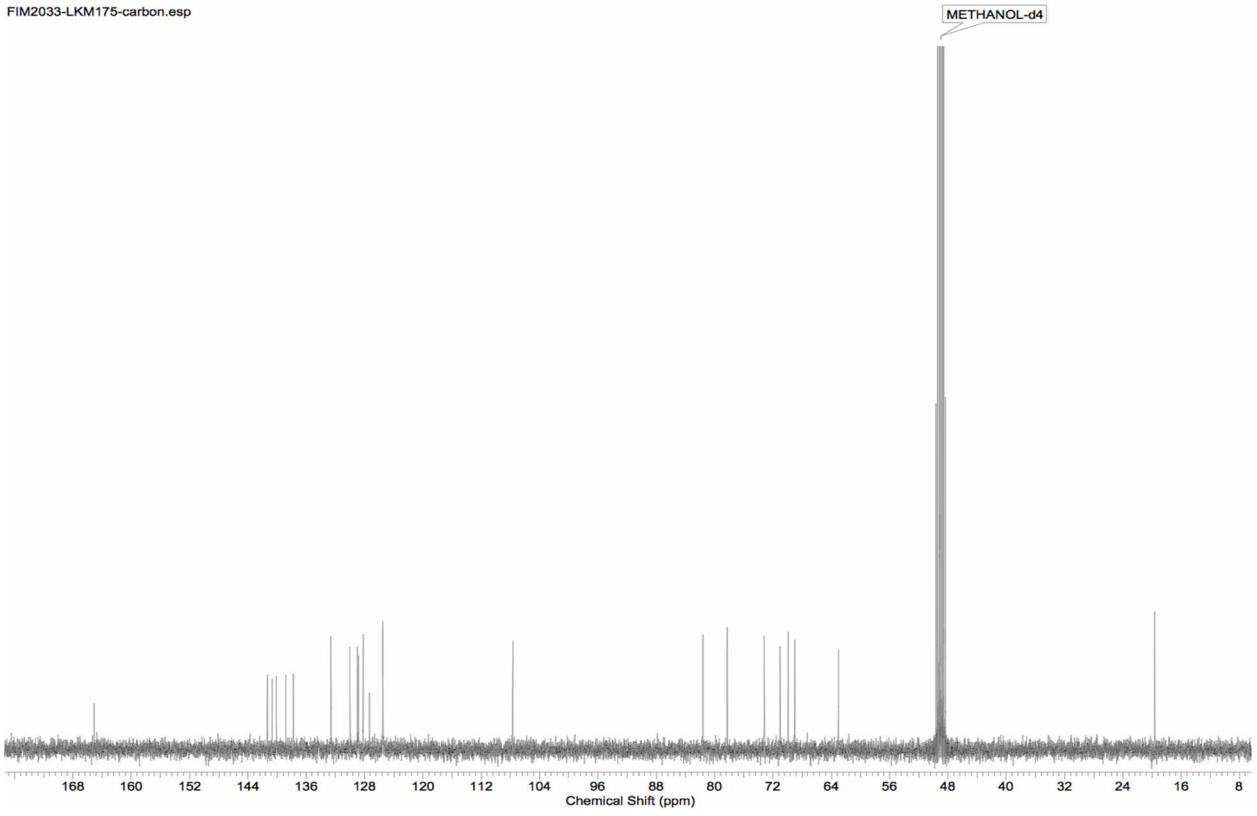



Compound 27

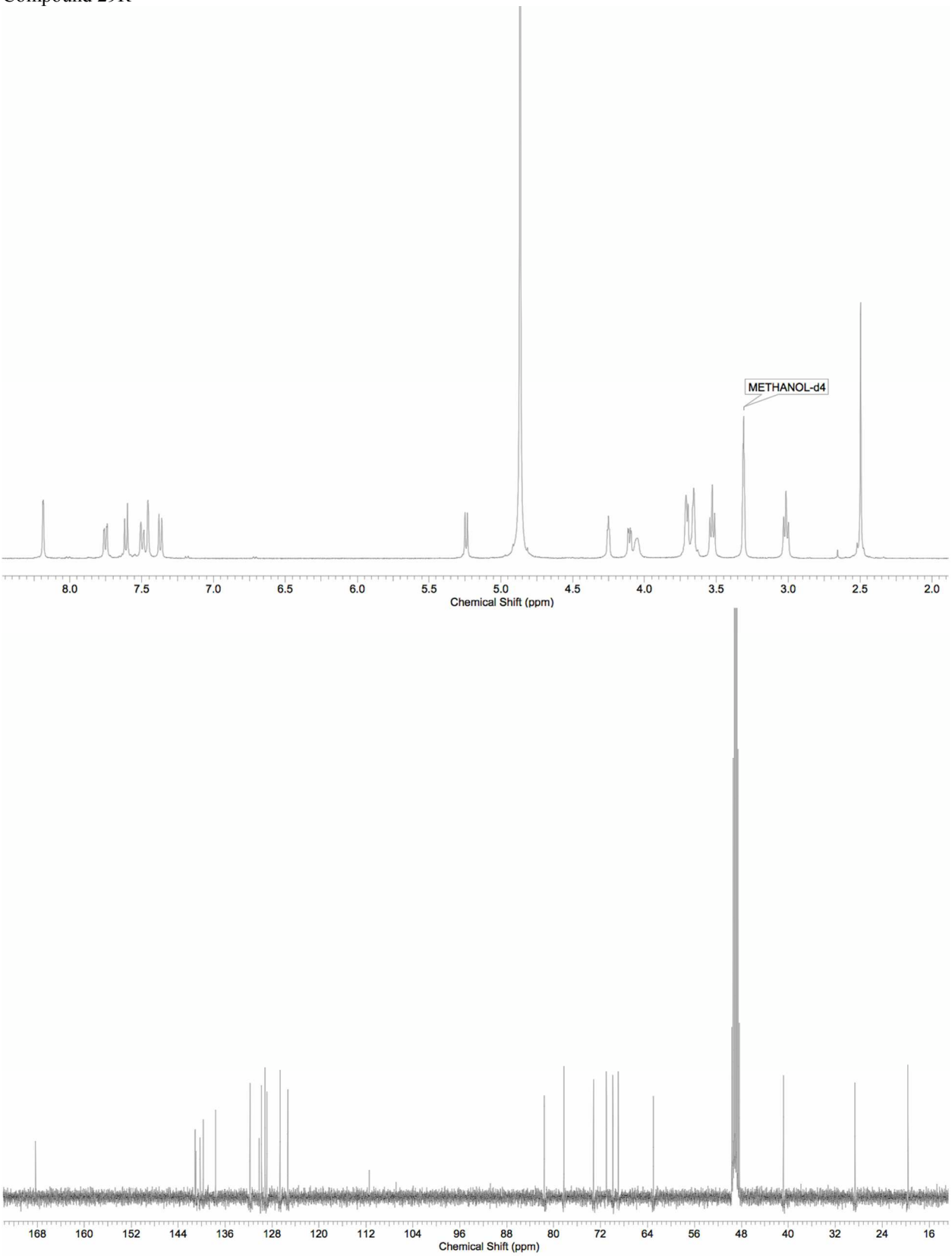

Int-LB2p28-LKM145-proton.esp
CHLOROFORM-d
Et2O
Et2O
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Chemical Shift (ppm)

Compound 28R

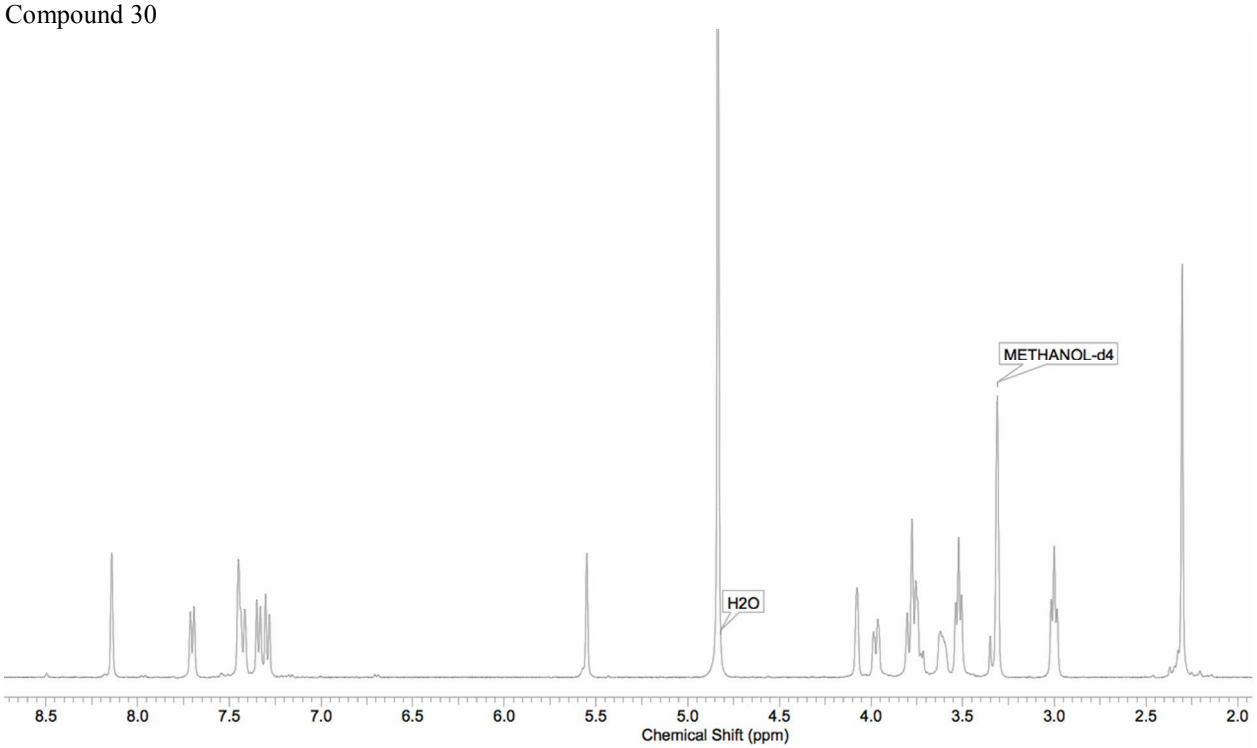


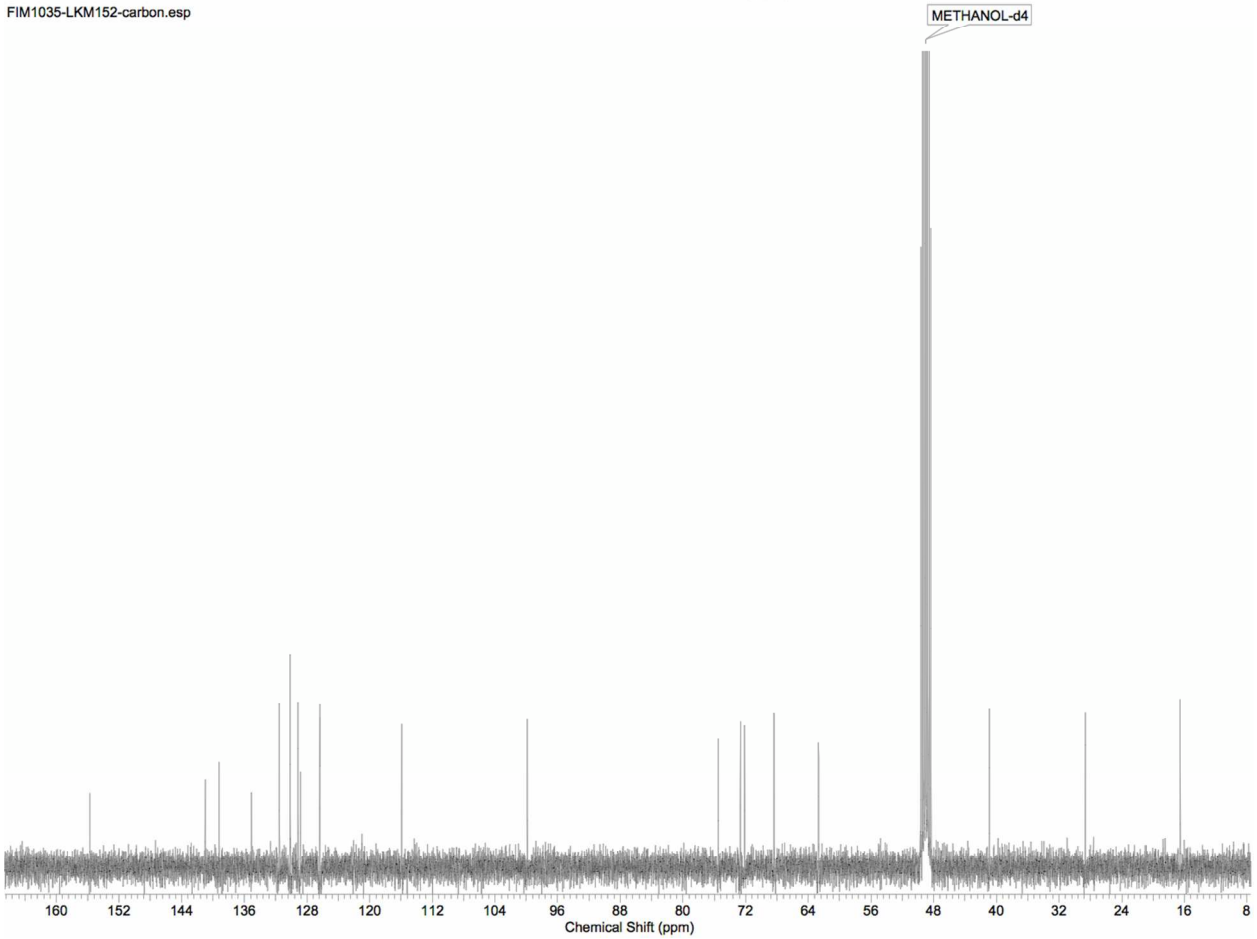



Compound 29R

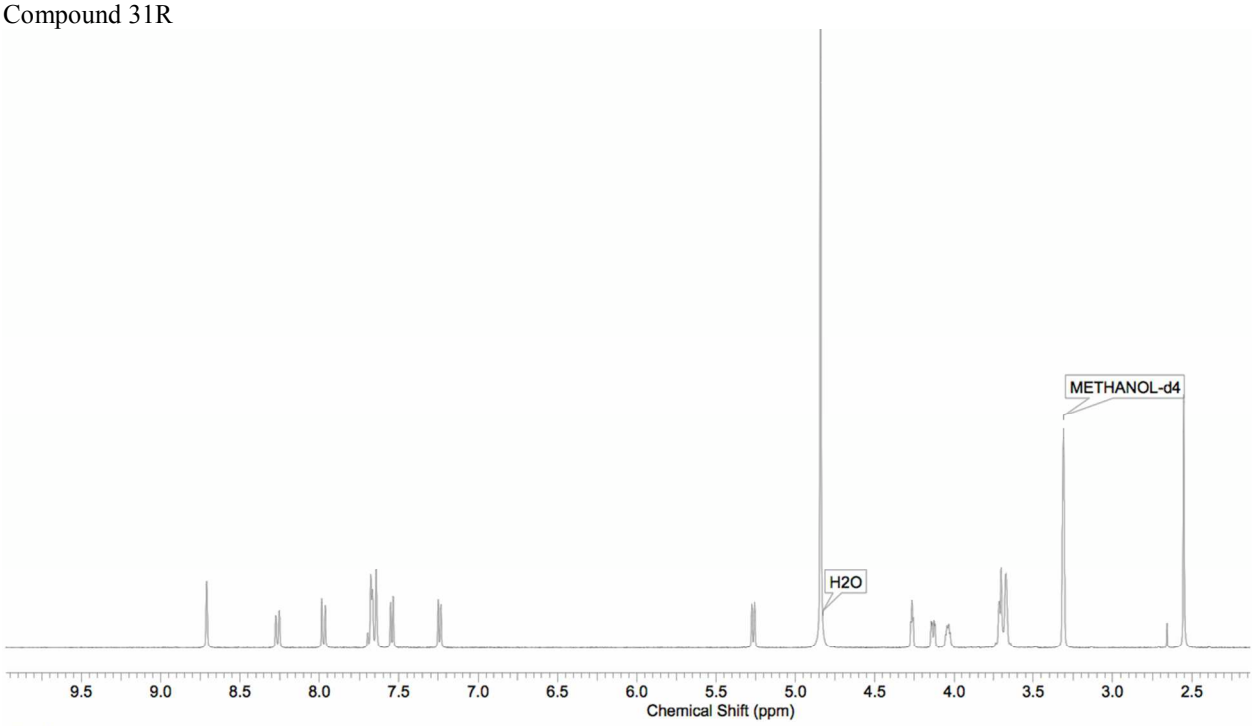


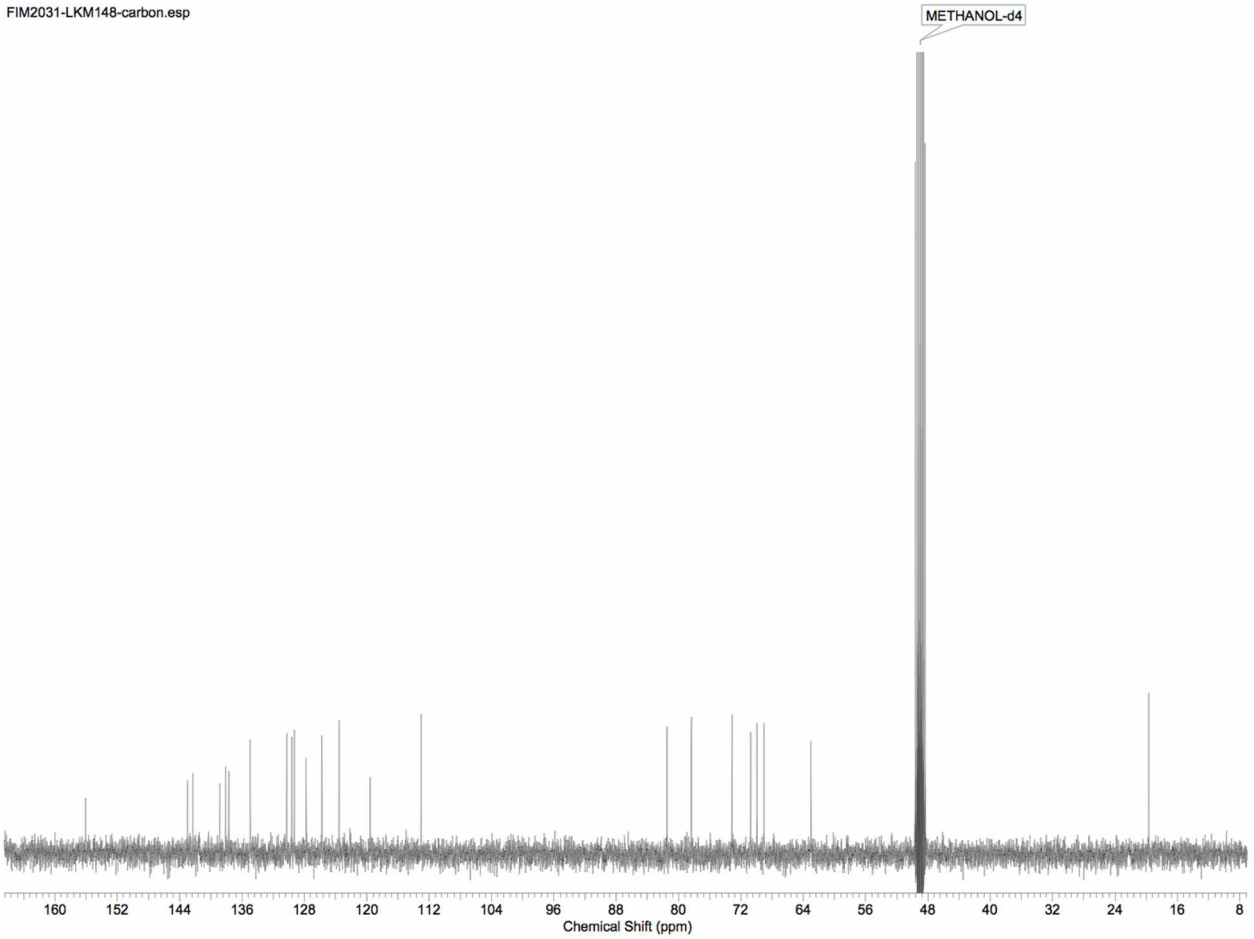
Compound 30


METHANOL-d4
H2O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
Chemical Shift (ppm)

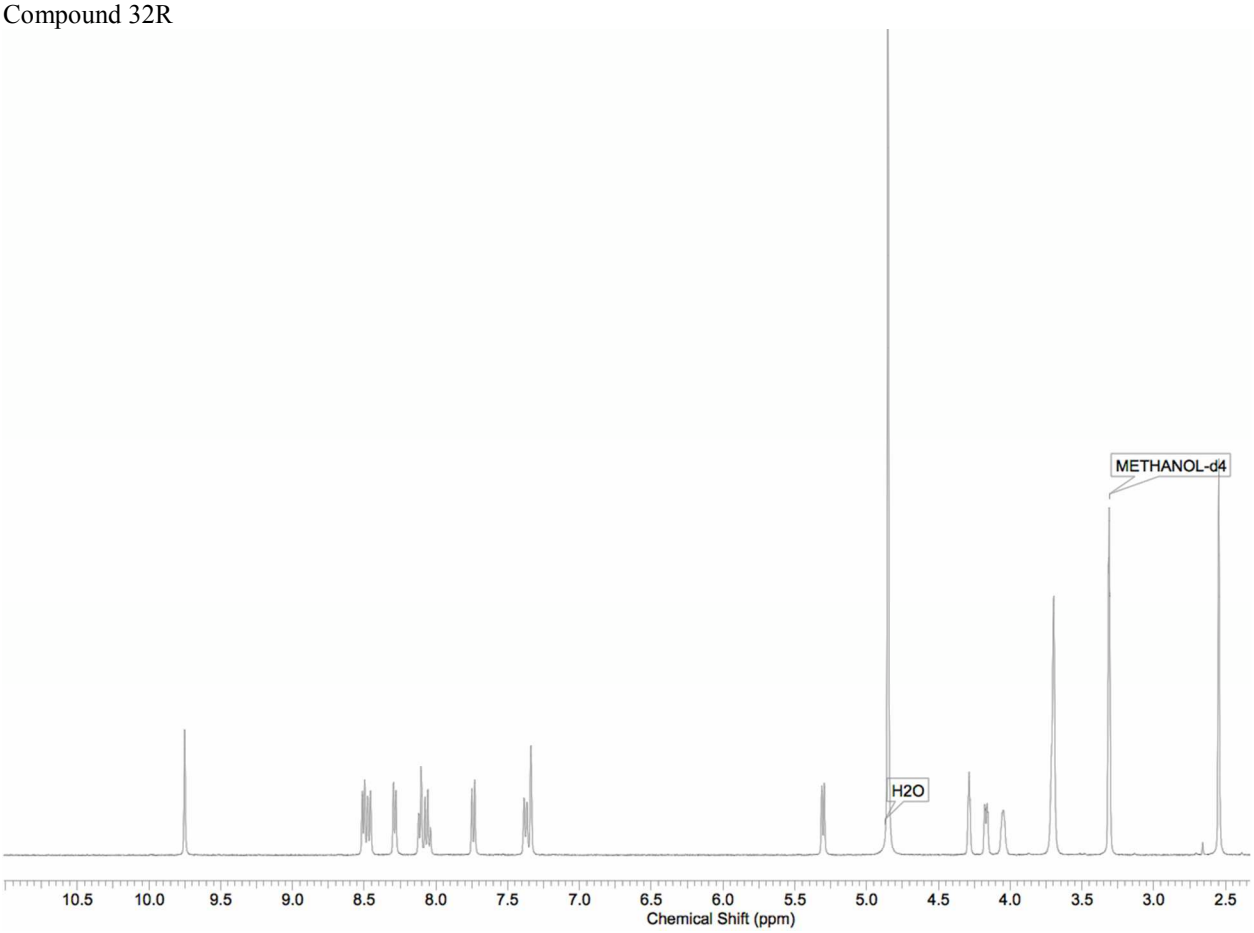

FIM1035-LKM152-carbon.esp
METHANOL-d4
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
Chemical Shift (ppm)

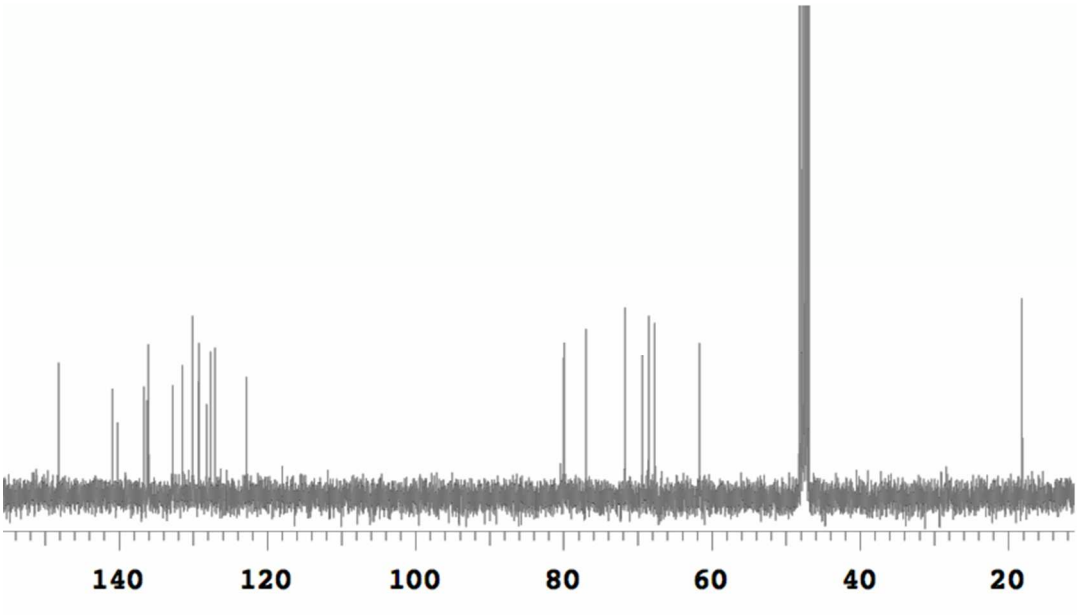
Compound 31R



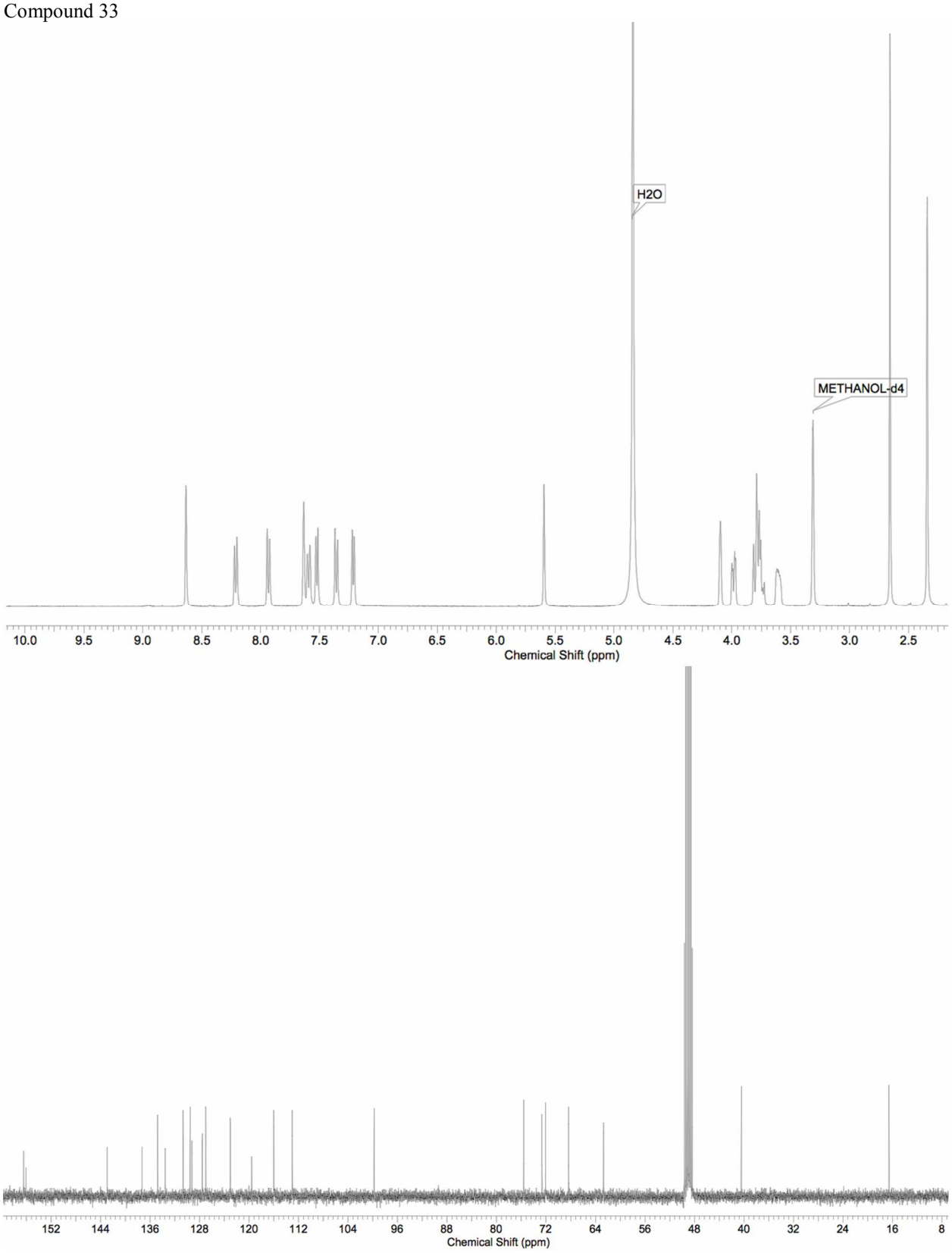



Compound 32R



Compound 33


H2O
METHANOL-d4
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5
Chemical Shift (ppm)
152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8
Chemical Shift (ppm)

**References.**


[i] Bruker Analytical X-Ray, Madison, WI, **2010**
[ii] Bruker Analytical X-Ray, Madison, WI, **2010**
[iii] Sheldrick, G.M. (**2008**). *Acta Cryst.* A64,112-122
[iv] Parsons, S. & Flack, H. (**2004**). Acta Cryst.,A, s61. 122