SUPPORTING INFORMATION

Cerium(III) Chloride-Mediated Reactions of Sulfonamide Dianions.

*David C. Johnson II and Theodore S. Widlanski**

Department of Chemistry, Indiana University, Bloomington IN 47405, USA

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N-Methyl methanesulfonamide (34). A 2.0M solution of methyl amine in methanol (60 ml) is cooled to 0 °C then methanesulfonyl chloride (2 ml, 25.74 mmol) is added dropwise. The reaction is stirred overnight at RT. All volatiles are removed under reduced pressure. Excess amine salt is triturated from DCM and filtered. The filtrate is purified by silica-gel chromatography (1:1 Hex: EtOAc) providing **34** (1.577 g, 14.45 mmol) as a solid; yield: 56%. (The product is soluble in water) ¹H NMR (400 MHz, CDCl₃): δ= 4.93 (br s, 1H), 2.88 (s, 3 H), 2.72 (d, *J* = 5.1 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ= 38.25, 29.32; IR (film) 3589, 3298, 3020 2936, 2821, 2360, 1473, 1406, 1313, 1155, 1071; HRMS (EI) *m/z* calcd for C₂H₇NO₂S (M⁺): 109.01975, found 109.01968.

N-Ethyl methanesulfonamide (36). Methanesulfonyl chloride (2 ml, 25.74 mmol) is dissolved in THF (60 ml) and cooled to 0 °C then ethyl amine (gas) is bubbled through for 10 minutes. The reaction is stirred overnight at RT under an atmosphere of ethylamine. The precipitate is filtered and washed with THF. All volatiles are removed by rotary evaporator. Purification by silica-gel chromatography (1:1 Hex: EtOAc) provides **35** (2.145 g, 17.41 mmol) as a solid; yield: 68%. ¹H NMR (400 MHz, CDCl₃): δ= 4.95(br s, 1H), 3.10 (m, 2 H), 2.90 (s, 3 H), 1.16 (t, *J* = 7.6 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ= 39.95, 38.28, 15.47; IR (film) 3586, 3285, 3023, 2983, 2938, 2882, 2359, 2340, 1439, 1413, 1384, 1316, 1153, 1064; HRMS (EI) *m/z* calcd for C₃H₉NO₂S (M⁺): 123.03540, found 123.03502.

General procedure for the synthesis of N-substituted methanesulfonamides

The amine (0.2713 mol) is dissolved in Et₂O (700 ml) and cooled to 0 °C then methanesulfonyl chloride (10 ml, 0.1292 mol) is added dropwise. The reaction is stirred overnight at RT. The precipitate is filtered and washed with Et₂O. The organic phase is washed with 2 M hydrochloric acid then NaCl. The ether is dried (Na₂SO₄) and removed by rotary evaporator. The syrup obtained is dissolved in THF (200 ml) and stirred for 3 hrs with a solution of 10% MeOH/ NaHCO₃ (100 ml). All volatiles are removed by rotary evaporator and the syrup obtained is portioned between DCM and water. The organic phase is separated, dried (Na₂SO₄) and evaporated. Purification by silica-gel chromatography uses the solvent systems indicated.

***N*-isoPropyl methanesulfonamide (38).** Eluent = 2:1 Hex: EtOAc provides **38** (7.09 g, 0.0517 mol) as a solid; yield: 40%. ¹H NMR (400 MHz, CDCl₃): δ= 4.60 (br s, 1H), 3.62 (sept, *J* = 6.6 Hz, 1 H), 2.95 (s, 3 H), 1.22 (d, *J* = 6.4 Hz); ¹³C NMR (100 MHz, CDCl₃): δ= 46.34, 41.82, 24.18; IR (film) 3585, 3286, 2976, 2937, 2878, 1467, 1436, 1412, 1389, 1371, 1318, 1136, 1013; HRMS (EI) *m/z* calcd for C₄H₁₁NO₂S (M⁺): 137.05105, found 137.05157.

***N*-Hexyl methanesulfonamide (40).** Eluent = 2:1 Hex: EtOAc provides **40** (22.700 g, 0.1266 mol) as a solid; yield: 98%. ¹H NMR (400 MHz, CDCl₃): δ= 4.59 (br s, 1 H), 3.10 (q, *J* = 7.1 Hz, 2 H), 2.94 (s, 3H), 1.55 (quint, *J* = 7.3 Hz, 2 H), 1.37- 1.24 (m, 6 H), 0.88 (t, *J* = 6.6 Hz); ¹³C NMR (100 MHz, CDCl₃): δ= 43.49, 40.27, 31.45, 30.17, 26.37, 22.64, 14.11; IR (film) 3251, 3023, 2955, 2926, 2860, 2253, 1468, 1412, 1303, 1135, 1076; HRMS (EI) *m/z* calcd for C₇H₁₈NO₂S (M + H⁺): 180.10582, found 180.10616.

N-Phenyl methanesulfonamide (42). Eluent = 2:1 Hex: EtOAc provides **42** (22.122 g, 0.1292 mol) as a solid; yield: quantitative. ^1H NMR (400 MHz, CDCl_3): δ = 7.37- 7.17 (m, 5 H), 7.03 (br s, 1H), 3.02 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ = 136.93, 129.84 (2), 125.57, 120.96 (2), 39.40; IR (film) 3587, 3262, 3085, 3050, 3023, 2934, 1600, 1495, 1406, 1326, 1222, 1152; HRMS (EI) m/z calcd for $\text{C}_7\text{H}_9\text{NO}_2\text{S}$ (M^+): 171.03540, found 171.03620.

N-Benzhydryl methanesulfonamide (44). Trituration from DCM provides **44** (29.376 g, 0.1124 mol) as a solid; yield: 87%. ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ = 8.34 (br s), 7.40- 7.24 (m, 10 H), 5.62 (s, 1H), 2.62 (s, 3 H); ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$): δ = 142.21, 128.45, 127.12, 60.36, 41.26; IR (diamond window) 3295, 3204, 3060, 3014, 1493, 1450, 1430, 1318, 1295, 1148, 1046; HRMS (CH_4) m/z calcd for $\text{C}_{13}\text{H}_{12}\text{N}$ ($\text{M}^+ - \text{CH}_3\text{SO}_2$): 182.09697, found 182.09650.

N-Benzyl methanesulfonamide (49)¹ Eluent = 2:1 Hex: EtOAc provides **49** (22.260 g, 0.1202 mol) as a solid; yield: 93%. ^1H NMR (400 MHz, CDCl_3): δ = 7.28 (m, 5H), 5.39 (t, J = 6.0 Hz, 1 H), 4.19 (d, J = 6.4 Hz, 2 H), 2.72 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ = 136.91, 128.74, 127.89, 46.94, 40.65; IR (film) 3235, 3039, 3021, 2933, 2360, 2257, 1456, 1438, 1412, 1337, 1299, 1137, 1062; HRMS (CH_4) m/z calcd for $\text{C}_8\text{H}_{11}\text{NO}_2\text{S}$ (M^+): 185.05105, found 185.05109.

***N*-Benzyl-deuteriomethanesulfonamide (50).** **49** (100 mg, 0.5398 mmol) is dissolved in THF 1.1ml) and cooled to -75°C . A 2.23 M solution of BuLi in hexanes (0.48 ml, 1.079 mmol) is added dropwise. The reaction is warmed to -30°C over a period of 15 minutes and stirred at this temperature for 45 minutes. Perdueteroacetic acid (0.1 ml, 1.746 mmol) is added, the reaction is warmed to RT and the THF removed by rotary evaporator. The syrup obtained is dissolved in EtOAc and extracted with NaHCO_3 . The EtOAc phase is dried (Na_2SO_4) and evaporated to dryness yielding **50** (99 mg, 0.5315 mmol); yield: 98% with 92% deuterium incorporation. ^1H NMR (400 MHz, CDCl_3): δ = 7.37 (m, 5H), 5.22 (m, 1 H), 4.31 (d, J = 6.2 Hz, 2 H), 2.83 (t, J = 1.7 Hz, 2 H); ^2H NMR (61 MHz, CHCl_3): δ = 2.83 (t, J = 2.0 Hz); ^{13}C NMR (100 MHz, CDCl_3): δ = 136.90, 128.94, 128.10, 128.02, 47.47, 40.78 (t, J = 21.4 Hz); IR (film) 3235, 3088, 3034, 3016, 2963, 2857, 1456, 1436, 1308, 1239, 1154, 1061; HRMS (CH_4) m/z calcd for $\text{C}_8\text{H}_{10}\text{DNO}_2\text{S}$ (M^+): 186.05722, found 186.05772.

Table 5: Deuterium incorporation into *N*-benzyl- α , *N*-dilithio methanesulfonamide.

Entry	$\text{CH}_3\text{SO}_2\text{NHBn}$ (eq)	BuLi (eq)	Temp ($^{\circ}\text{C}$)	$\alpha\text{-CH}_2\text{D}$	$\alpha\text{-CH}_3$	% Yield
1 ^a	1	2.1	-78 to 0	92	8	95
2	1	2.0	-78 to -30	92	8	98

^a Published protocol for generation of sulfonamide dianion.²

1,2,5,6-Di-*O*-isopropylidene-3-oxo- α -D-glucofuranose (16).³ Diacetone-D-glucose (**51**) (2.5 g, 9.605 mmol) was dissolved in DCM (60 ml) and cooled to 0 °C. Dess-Martin-Periodinane⁴ (6.125 g, 14.44 mmol) was added then the ice-bath was removed. The reaction was stirred overnight at RT then quenched with NaHCO₃ (100 ml) containing Na₂S₃O₆ (2 g). The reaction was diluted with DCM and extracted three times with NaHCO₃ (100 ml) containing Na₂S₃O₆ (2 g). The organic phase was separated, dried (Na₂SO₄) and evaporated by rotary evaporator. The gum obtained was filtered through a 1" cake of silica-gel (Et₂O) providing **16** in equilibrium with its hydrate. The keto-form was obtained by drying in a desiccator under high-vacuum over P₂O₅ for several days providing **16** as a wax (2.143 g, 8.298 mmol); yield: 86%. ¹H NMR (400 MHz, CDCl₃): δ = 6.12 (d, *J* = 4.5 Hz, 1H), 4.39-4.33 (m, 3 H), 4.03- 4.01 (m, 2 H), 1.47 (s, 3 H), 1.45 (s, 3 H), 1.31 (s, 6 H); ¹³C NMR (100 MHz, CDCl₃): δ = 209.05, 114.44, 110.53, 103.27, 79.10, 77.40, 76.52, 64.43, 27.71, 27.32, 26.12, 25.44; IR (film) 2989, 2939, 2360, 2341, 1773, 1375, 1263, 1216, 1156, 1081, 1023; HRMS (CH₄) *m/z* calcd for C₁₁H₁₅O₆ (M⁺ - CH₃): 243.08687, found 243.08740.

5'-(*O*-*tert*-Butyldimethylsilyl)-3'-ketothymidine (19).⁵ The method of Bender and Moffett⁶ was employed for the preparation of **19** using **52**⁵ (500 mg, 1.403 mmol) to provide **19** (477 mg, 1.346 mmol); yield: 96%. The product was obtained as a 99: 1 ratio of ketone **19** to β -elimination product **53**.

^1H NMR (400 MHz, CDCl_3): δ = 9.41 (br s, 1 H), 7.64 (d, J = 1.3 Hz, 1 H), 6.49 (dd, J = 8.3, 6.4 Hz, 1 H), 4.12 (br s, 1H), 3.98 (d, J = 1.7 Hz, 2 H), 2.97 (A of ABX, J_{AB} = 18.4 Hz, J_{AX} = 6.4 Hz, 1 H), 2.40 (B of ABX, J_{BA} = 18.4 Hz, J_{BX} = 8.3 Hz, 1 H), 1.94 (d, J = 1.2 Hz, 3 H), 0.88 (s, 9 H), 0.09 (s, 3 H), 0.06 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ = 209.62, 163.89, 150.64, 134.79, 112.12, 83.02, 81.25, 62.95, 42.61, 26.02, 18.52, 12.72, -5.39; IR (film) 3190, 3063, 2955, 2929, 2858, 1770, 1699, 1683, 1471, 1277, 1256, 1172, 1123, 1077, 1051; HRMS (CH_4) m/z calcd for $\text{C}_{12}\text{H}_{17}\text{N}_2\text{O}_6\text{Si}$ (M^+ - C_4H_9): 297.09067, found 297.09096.

3-(*O*-*tert*-Butyldimethylsilyl)-estrone (21).⁷ TBS-Cl (251 mg, 1.664 mmol) was added to a stirring solution of estrone (**54**) (300 mg, 1.110 mmol) and DMAP (10 mg, 0.08185 mmol) in pyridine (6 ml). The mixture was stirred until complete by TLC at which time the pyridine was removed rotary evaporation. The resulting gum was purified by silica-gel chromatography (3:1 Hex: EtOAc) to give **21** (326 mg, 0.8453 mmol); yield: 76%. ^1H NMR (400 MHz, CDCl_3): δ = 7.12 (d, J = 8.4 Hz, 1 H), 6.63 (dd, J = 8.8, 2.2 Hz, 1H), 6.57 (d, J = 2.0 Hz, 1 H), 2.85 (m, 2 H), 2.50 (dd, J = 18.8, 8.7 Hz, 1 H), 2.38 (m, 1 H), 2.24 (m, 1 H), 2.19- 1.37 (m, 11 H), 0.98 (s, 9 H), 0.91 (s, 3 H), 0.19 (s, 6 H); ^{13}C NMR (100 MHz, CDCl_3): δ = 221.19, 153.64, 137.77, 132.60, 126.31, 120.17, 117.48, 50.60, 48.18, 44.18, 38.47, 36.05, 31.75, 29.65, 26.73, 26.01, 25.87, 21.75, 18.34, 14.04, -4.22; IR (film) 3046, 3034, 2947, 2927, 2877, 2857, 1733, 1605, 1493, 1471, 1284, 1258, 1248, 1157, 1084, 1052; HRMS (CH_4) m/z calcd for $\text{C}_{24}\text{H}_{36}\text{O}_2\text{Si}$ (M^+): 384.24846, found 384.24945.

5'-Deoxy-2', 3'-isopropylidene-5', 5'-(*N, N'*-diphenylethylenediamino)-uridine (56**).** The method of Tronchet and Valero⁸ was employed using **55**⁹ (700 mg, 2.463 mmol) to provide **56** (697 mg, 1.463 mmol); yield: 59%. ¹H NMR (400 MHz, CDCl₃): δ = 7.28 (m, 5 H), 6.83 (m 6 H), 5.97 (d, J = 2.5 Hz, 1 H), 5.81 (d, J = 2.3 Hz, 1 H), 5.48 (d, J = 8.0 Hz, 1 H), 4.89 (t, J = 6.0 Hz, 1 H), 4.59 (dd, J = 6.6, 2.5 Hz, 1 H), 4.49 (dd, J = 5.3, 2.2 Hz, 1 H), 3.90- 3.64 (m, 4 H), 1.43 (s, 3 H), 1.31 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ = 163.12, 150.15, 146.82, 146.40, 140.73, 129.59, 129.47, 118.69, 118.59, 115.23, 114.02, 113.74, 102.95, 89.44, 86.03, 84.03, 79.45, 73.00, 48.20, 46.83, 27.33, 25.78; IR (film) 3352, 3082, 3066, 2989, 2938, 1704, 1599, 1580, 1449, 1282, 1238, 1213, 1110, 1085; HRMS (FAB) m/z calcd for C₂₆H₂₈N₄O₅Na (M + Na⁺): 499.19577, found 499.19807.

2', 3'-Isopropylidene-uridine-5'-aldehyde (28**).**¹⁰ The method of Ranganathan, Jones and Moffatt¹¹ was employed with the following modifications: 1) THF was removed completely after filtration by rotary evaporation then the aqueous phase was washed with EtOAc; 2) The aqueous phase was evaporated to dryness under vacuum at 50 °C overnight to provide the hydrate of **28**. The free aldehyde was liberated as reported.¹¹ From **56** (476 mg, 0.9989 mmol) to **28** (127 mg, 0.4502 mmol); yield: 45%. ¹H NMR (400 MHz, CDCl₃): δ = 9.81 (br s, 1 H), 9.41 (s, 1 H), 7.28 (d, J = 8.0 Hz, 1 H), 5.78 (d, J = 8.1 Hz, 1 H), 5.51 (s, 1 H), 5.20 (br d, J = 6.2 Hz, 1 H), 5.10 (br d, J = 6.2 Hz, 1 H), 4.54 (br s, 1 H), 1.52 (s, 3 H), 1.35 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ = 199.52, 163.86, 150.75, 144.42, 113.75,

102.95, 100.21, 94.22, 85.00, 83.88, 26.52, 24.84; IR (film) 3405, 3221, 3105, 3070, 2990, 2938, 2829, 2256, 1694, 1461, 1383, 1273, 1213, 1159, 1090, 1058; HRMS (CH₄) *m/z* calcd for C₁₂H₁₅N₂O₆ (MH⁺): 283.09301, found 283.09170.

7-Hydroxy-heptanenitrile (58).¹² Powdered KCN (995 mg, 15.29 mmol) was dried under vacuum at 100 °C overnight. After cooling to RT, dry CH₃CN (3 ml), 6-bromohexanol (1 ml, 7.643 mmol) and 18-crown-6 (202 mg, 0.7463 mmol) were introduced. The suspension was stirred for two days at 80 °C. After cooling to RT, CH₃CN was removed by rotary evaporation and the resulting slurry was partitioned between DCM and Na₂CO₃. The DCM was dried (Na₂SO₄) and evaporated. Flash-column chromatography (2:1 Et₂O: Hex) provided **58** as an oil (780 mg, 6.211 mmol); yield: 80%. ¹H NMR (400 MHz, CDCl₃): δ= 3.61 (t, *J* = 6.9 Hz, 2 H), 2.33 (t, *J* = 6.9 Hz, 2 H), 1.81 (br s, 1 H), 1.65 (p, *J* = 6.9 Hz, 2 H), 1.55 (p, *J* = 6.9 Hz, 2 H), 1.46 (m, 2 H), 1.39 (m, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ= 119.93, 62.61, 32.38, 28.53, 25.39, 25.09, 17.17; IR (neat) 3403, 2936, 2862, 2247, 1462, 1426, 1056; HRMS (CH₄) *m/z* calcd for C₇H₁₄NO C₇H₁₄ON (MH⁺): 128.10754, found 128.10756.

7-Oxo-heptanenitrile (32).¹³ A solution of oxalyl chloride (0.129 ml, 1.479 mmol) in DCM (3.8 ml) was cooled to −50 °C and distilled DMSO (0.209 ml, 2.958 mmol) was added dropwise. After stirring five min, **58** (in 1 ml DCM) was added dropwise. Stirring was continued for 15 min, at which time TEA (0.94 ml, 6.722 mmol) was added. The reaction was warmed to RT and quenched with H₂O. The DCM phase was dried (Na₂SO₄) and evaporated. Silica-gel chromatography (2:1 Et₂O: Hex) provided **32** as an oil (80 mg, 0.639 mmol); yield: 48%. ¹H NMR (400 MHz, CDCl₃): δ= 9.73 (br s, 1 H), 2.44 (dt, *J* = 6.9, 6.9, 0.91 Hz, 2 H), 2.31 (t, *J* = 7.3 Hz, 2 H), 1.63 (h, *J* = 7.3 Hz, 2 H), 1.55 (p, *J* = 6.9 Hz, 2

H), 1.46 (m, 2 H), 1.45 (m, 2 H); ^{13}C NMR (100 MHz, CDCl_3): δ = 202.04, 119.62, 43.47, 28.10, 25.16, 21.13, 16.99; IR (neat) 2940, 2868, 2729, 2246, 1722, 1462, 1427, 1392; HRMS (CH_4) m/z calcd for $\text{C}_7\text{H}_{10}\text{NO}$ ($\text{M}^+ - \text{H}$): 124.07624, found 124.07641.

Table 6: Qualitative 2-D NOESY interactions for nucleosides^a

R	Nucleoside	Interaction					
		1'-4'	2'-OH	2'-H ₆	OH-5'	OH-H ₆	3''-4'
Bn	18	m	m	w	-	m	m
Me	35	s	s	s	w	s	s
Et	37	s	s	s	-	s	m
<i>i</i> Pr	39	m	s	m	w	s	s
Hexyl	41	m	s	s	m	s	s
Phenyl	43	s	m	m	-	s	m
Benzyhydrol	45	m	m	m	-	m	m
Bn	20	m	-	-	-	m	-

^aIntensity ratings: w = weak; m = medium; s = strong

Figure 1: Diagram for nomenclature of 2-D NOESY interactions

R' = OTBS; R'' = H: (**18, 35, 37, 39, 41, 43, 45**)

R' = H; R'' = CH₃: (**20**)

Determination of detrimental water in a preparation of dehydrated cerium(III) chloride.

Cerium(III) chloride heptahydrate (223 mg, 0.5985 mmol) was dehydrated according to the method of Dimitrov.¹⁴ The dehydrated CeCl₃ (146 mg, 0.5924) was stirred overnight in THF (2.37 ml) under an atmosphere of N₂. Separately, *N*-benzyl methanesulfonamide (107 mg, 0.5780 mmol) in THF (0.50 ml) is cooled to –75 °C and titrated BuLi (0.49 ml, 1.156 mmol) is added dropwise under an atmosphere of N₂. The temperature of the cold bath is raised gradually to –30 °C over 15 min and the solution stirred for an additional 45 min at –30 °C. After cooling the dianion solution to –75 °C, it is introduced via cannula into the cerium solution, also cooled to –75 °C. The temperature of the cold bath is raised gradually to –30 °C over 15 min and the solution stirred for an additional 45 min at –30 °C. The heterogeneous yellow/ light orange cerium-dianion solution is recooled to –30 °C. The reaction is quenched with a 1: 1 mixture of CD₃CO₂D: D₂O (2 ml) and warmed to RT, at which time water is added (10 ml/ mmol). THF is removed by rotary evaporator and the resulting slurry is partitioned between ethyl acetate and water. The ethyl acetate phase is extracted with water then NaHCO₃. The aqueous phases, kept separate, are individually extracted with ethyl acetate then all organic phases are combined, dried (Na₂SO₄) and concentrated by rotary evaporation of the volatiles. No further purification was performed.

The control was performed using the conditions above with the omission of cerium.

Table 7: Deuterium incorporation into *N*-benzyl methanesulfonamide in the presence of CeCl₃.

	Control				Cerium			
	Manual		Gaussian Line Shape		Manual		Gaussian Line Shape	
	CH ₃	CH ₂ D	CH ₃	CH ₂ D	CH ₃	CH ₂ D	CH ₃	CH ₂ D
Area	0.42805	1.4516	220.22	872.32	0.49429	1.5728	121.29	410.98
Area/ H	0.14268	0.7258	73.407	436.16	0.16476	0.78625	40.43	205.49
%	16	84	14	86	17	83	16	84
Average	CH ₃		CH ₂ D		CH ₃		CH ₂ D	
	15		85		17		83	

Determination of mole fraction of detrimental water in CeCl_3

Reaction

NMR Data

Avg % composition	83	17
control adjustment	+ 15	- 15
corrected % composition	98	2

Molar Ratio

starting material = 0.000578 mol		
product = 0.000566 mol	0.000555 mol	0.000011 mol

$$\begin{aligned} \text{mol } \alpha\text{CH}_3 &= \text{mol H}_2\text{O} \\ 0.000011 &= \text{mol H}_2\text{O} \quad \therefore \quad 0.00020 = \text{grams H}_2\text{O} \end{aligned}$$

Mass H_2O

$$\frac{\text{mass H}_2\text{O}}{\text{measured mass of CeCl}_3 \text{ reagent}} = \frac{0.00020 \text{ g}}{0.146 \text{ g}} = 0.1\%$$

$$n_{\text{H}_2\text{O}} = \frac{100 \text{ g (0.1 \%)}}{18.01 \text{ g/mol}}$$

$$n_{\text{CeCl}_3} = \frac{100 \text{ g (99.9 \%)}}{246.45 \text{ g/mol}}$$

$$n_{\text{H}_2\text{O}} = 0.006$$

$$n_{\text{CeCl}_3} = 0.4$$

Mole Fraction H_2O

$$n_{\text{total}} = 0.406$$

$$\text{mole fraction H}_2\text{O} = \frac{n_{\text{H}_2\text{O}}}{n_{\text{total}}} = 0.01$$

Figure 2: MS spectrum for *N*-benzyl-2-hydroxy-4, 8-dimethyl-nona-3,7-diene-1-methylenesulfonamide (15).

Figure 3: Proton NMR spectrum for 1,2,5,6-di-*O*-isopropylidene-3-oxo- α -D-glucofuranose (16).

Figure 4: Carbon NMR spectrum for 1,2,5,6-di-*O*-isopropylidene-3-oxo- α -D-glucofuranose (16).

Figure 5: IR spectrum for 1,2,5,6-di-*O*-isopropylidene-3-oxo- α -D-glucofuranose (16).

Figure 6: MS spectrum for 1,2,5,6-di-*O*-isopropylidene-3-oxo- α -D-glucofuranose (16).

DJ0046 Scan 11 RT=1:02 100%=276895 mv 16 Aug 101 14:41
HRP +CI 212C 3000 RES / DCI CH4

Figure 7: Proton NMR spectrum for 3- β -(*N*-benzyl methanesulfonamide)-1,2,5,6-di-*O*-isopropylidene- α -D-allofuranose (17).

Figure 8: HMQC spectrum for 3- β -(*N*-benzyl methanesulfonamide)-1,2,5,6-di-*O*-isopropylidene- α -D-allofuranose (17).

Figure 9: Detailed HMQC spectrum for 3- β -(*N*-benzyl methanesulfonamide)-1,2,5,6-di-*O*-sopropylidene- α -D-allofuranose (17).

Figure 10: Carbon NMR spectrum for 3- β -(*N*-benzyl methanesulfonamide)-1,2,5,6-di-*O*-isopropylidene- α -D-allofuranose (17).

Figure 11: IR spectrum for 3- β -(*N*-benzyl methylenesulfonamide)-1,2,5,6-di-*O*-isopropylidene- α -D-allofuranose (17).

Figure 12: MS spectrum for 3- β -(*N*-benzyl methanesulfonamide)-1,2,5,6-di-*O*-isopropylidene- α -D-allofuranose (17).

DJ0044 Scan 34 RT=2:12 100%=138922 mv 27 Sep 102 9:24
HRP +EI 4F 3000 RES / DEI

Figure 13: Proton NMR spectrum for 3'- α -(*N*-benzyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (18).

Figure 14: HMQC spectrum for 3'- α -(*N*-benzyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (18).

Figure 15: Detailed HMQC spectrum for 3'- α -(*N*-benzyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (18).

Figure 16: Carbon NMR spectrum for 3'- α -(*N*-benzyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (18).

Figure 17: IR spectrum for 3'- α -(*N*-benzyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (18).

Figure 18: MS spectrum for 3'- α -(*N*-benzyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (18).

Figure 19: Proton NMR spectrum for 5'-(*O*-*tert*-butyldimethylsilyl)-3'-ketothymidine (19).

Figure 20: Carbon NMR spectrum for 5'-(*O*-*tert*-butyldimethylsilyl)-3'-ketothymidine (19).

IR Spectrum (Wavenumbers (cm⁻¹) vs. % Transmittance) showing characteristic peaks for compound (19).

Chemical structure of (19): Cc1c[nH]c(=O)n1C2OC(C2)C3OC(C3)C4OC(C4)C5OC(C5)C6OC(C6)C7OC(C7)C8OC(C8)C9OC(C9)C10OC(C10)C11OC(C11)C12OC(C12)C13OC(C13)C14OC(C14)C15OC(C15)C16OC(C16)C17OC(C17)C18OC(C18)C19OC(C19)C20OC(C20)C21OC(C21)C22OC(C22)C23OC(C23)C24OC(C24)C25OC(C25)C26OC(C26)C27OC(C27)C28OC(C28)C29OC(C29)C30OC(C30)C31OC(C31)C32OC(C32)C33OC(C33)C34OC(C34)C35OC(C35)C36OC(C36)C37OC(C37)C38OC(C38)C39OC(C39)C40OC(C40)C41OC(C41)C42OC(C42)C43OC(C43)C44OC(C44)C45OC(C45)C46OC(C46)C47OC(C47)C48OC(C48)C49OC(C49)C50OC(C50)C51OC(C51)C52OC(C52)C53OC(C53)C54OC(C54)C55OC(C55)C56OC(C56)C57OC(C57)C58OC(C58)C59OC(C59)C60OC(C60)C61OC(C61)C62OC(C62)C63OC(C63)C64OC(C64)C65OC(C65)C66OC(C66)C67OC(C67)C68OC(C68)C69OC(C69)C70OC(C70)C71OC(C71)C72OC(C72)C73OC(C73)C74OC(C74)C75OC(C75)C76OC(C76)C77OC(C77)C78OC(C78)C79OC(C79)C80OC(C80)C81OC(C81)C82OC(C82)C83OC(C83)C84OC(C84)C85OC(C85)C86OC(C86)C87OC(C87)C88OC(C88)C89OC(C89)C90OC(C90)C91OC(C91)C92OC(C92)C93OC(C93)C94OC(C94)C95OC(C95)C96OC(C96)C97OC(C97)C98OC(C98)C99OC(C99)C100OC(C100)C101OC(C101)C102OC(C102)C103OC(C103)C104OC(C104)C105OC(C105)C106OC(C106)C107OC(C107)C108OC(C108)C109OC(C109)C110OC(C110)C111OC(C111)C112OC(C112)C113OC(C113)C114OC(C114)C115OC(C115)C116OC(C116)C117OC(C117)C118OC(C118)C119OC(C119)C120OC(C120)C121OC(C121)C122OC(C122)C123OC(C123)C124OC(C124)C125OC(C125)C126OC(C126)C127OC(C127)C128OC(C128)C129OC(C129)C130OC(C130)C131OC(C131)C132OC(C132)C133OC(C133)C134OC(C134)C135OC(C135)C136OC(C136)C137OC(C137)C138OC(C138)C139OC(C139)C140OC(C140)C141OC(C141)C142OC(C142)C143OC(C143)C144OC(C144)C145OC(C145)C146OC(C146)C147OC(C147)C148OC(C148)C149OC(C149)C150OC(C150)C151OC(C151)C152OC(C152)C153OC(C153)C154OC(C154)C155OC(C155)C156OC(C156)C157OC(C157)C158OC(C158)C159OC(C159)C160OC(C160)C161OC(C161)C162OC(C162)C163OC(C163)C164OC(C164)C165OC(C165)C166OC(C166)C167OC(C167)C168OC(C168)C169OC(C169)C170OC(C170)C171OC(C171)C172OC(C172)C173OC(C173)C174OC(C174)C175OC(C175)C176OC(C176)C177OC(C177)C178OC(C178)C179OC(C179)C180OC(C180)C181OC(C181)C182OC(C182)C183OC(C183)C184OC(C184)C185OC(C185)C186OC(C186)C187OC(C187)C188OC(C188)C189OC(C189)C190OC(C190)C191OC(C191)C192OC(C192)C193OC(C193)C194OC(C194)C195OC(C195)C196OC(C196)C197OC(C197)C198OC(C198)C199OC(C199)C200OC(C200)C201OC(C201)C202OC(C202)C203OC(C203)C204OC(C204)C205OC(C205)C206OC(C206)C207OC(C207)C208OC(C208)C209OC(C209)C210OC(C210)C211OC(C211)C212OC(C212)C213OC(C213)C214OC(C214)C215OC(C215)C216OC(C216)C217OC(C217)C218OC(C218)C219OC(C219)C220OC(C220)C221OC(C221)C222OC(C222)C223OC(C223)C224OC(C224)C225OC(C225)C226OC(C226)C227OC(C227)C228OC(C228)C229OC(C229)C230OC(C230)C231OC(C231)C232OC(C232)C233OC(C233)C234OC(C234)C235OC(C235)C236OC(C236)C237OC(C237)C238OC(C238)C239OC(C239)C240OC(C240)C241OC(C241)C242OC(C242)C243OC(C243)C244OC(C244)C245OC(C245)C246OC(C246)C247OC(C247)C248OC(C248)C249OC(C249)C250OC(C250)C251OC(C251)C252OC(C252)C253OC(C253)C254OC(C254)C255OC(C255)C256OC(C256)C257OC(C257)C258OC(C258)C259OC(C259)C260OC(C260)C261OC(C261)C262OC(C262)C263OC(C263)C264OC(C264)C265OC(C265)C266OC(C266)C267OC(C267)C268OC(C268)C269OC(C269)C270OC(C270)C271OC(C271)C272OC(C272)C273OC(C273)C274OC(C274)C275OC(C275)C276OC(C276)C277OC(C277)C278OC(C278)C279OC(C279)C280OC(C280)C281OC(C281)C282OC(C282)C283OC(C283)C284OC(C284)C285OC(C285)C286OC(C286)C287OC(C287)C288OC(C288)C289OC(C289)C290OC(C290)C291OC(C291)C292OC(C292)C293OC(C293)C294OC(C294)C295OC(C295)C296OC(C296)C297OC(C297)C298OC(C298)C299OC(C299)C300OC(C300)C301OC(C301)C302OC(C302)C303OC(C303)C304OC(C304)C305OC(C305)C306OC(C306)C307OC(C307)C308OC(C308)C309OC(C309)C310OC(C310)C311OC(C311)C312OC(C312)C313OC(C313)C314OC(C314)C315OC(C315)C316OC(C316)C317OC(C317)C318OC(C318)C319OC(C319)C320OC(C320)C321OC(C321)C322OC(C322)C323OC(C323)C324OC(C324)C325OC(C325)C326OC(C326)C327OC(C327)C328OC(C328)C329OC(C329)C330OC(C330)C331OC(C331)C332OC(C332)C333OC(C333)C334OC(C334)C335OC(C335)C336OC(C336)C337OC(C337)C338OC(C338)C339OC(C339)C340OC(C340)C341OC(C341)C342OC(C342)C343OC(C343)C344OC(C344)C345OC(C345)C346OC(C346)C347OC(C347)C348OC(C348)C349OC(C349)C350OC(C350)C351OC(C351)C352OC(C352)C353OC(C353)C354OC(C354)C355OC(C355)C356OC(C356)C357OC(C357)C358OC(C358)C359OC(C359)C360OC(C360)C361OC(C361)C362OC(C362)C363OC(C363)C364OC(C364)C365OC(C365)C366OC(C366)C367OC(C367)C368OC(C368)C369OC(C369)C370OC(C370)C371OC(C371)C372OC(C372)C373OC(C373)C374OC(C374)C375OC(C375)C376OC(C376)C377OC(C377)C378OC(C378)C379OC(C379)C380OC(C380)C381OC(C381)C382OC(C382)C383OC(C383)C384OC(C384)C385OC(C385)C386OC(C386)C387OC(C387)C388OC(C388)C389OC(C389)C390OC(C390)C391OC(C391)C392OC(C392)C393OC(C393)C394OC(C394)C395OC(C395)C396OC(C396)C397OC(C397)C398OC(C398)C399OC(C399)C400OC(C400)C401OC(C401)C402OC(C402)C403OC(C403)C404OC(C404)C405OC(C405)C406OC(C406)C407OC(C407)C408OC(C408)C409OC(C409)C410OC(C410)C411OC(C411)C412OC(C412)C413OC(C413)C414OC(C414)C415OC(C415)C416OC(C416)C417OC(C417)C418OC(C418)C419OC(C419)C420OC(C420)C421OC(C421)C422OC(C422)C423OC(C423)C424OC(C424)C425OC(C425)C426OC(C4

DJ0041 Scan 16 RT=1:00 100%=199400 mv 27 Sep 102 8:55
 HRP +EI 32F 3000 RES / DEI

Figure 22: MS spectrum for 5'-(*O*-*tert*-butyldimethylsilyl)-3'-ketothymidine (19).

Figure 23: Proton NMR spectrum for 3'- α -(*N*-benzyl methanesulfonamide)-5'-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy-thymidine (20).

Figure 24: HMQC spectrum for 3'- α -(*N*-benzyl methanesulfonamide)-5'-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy-thymidine (20).

Figure 25: Detailed HMQC spectrum for 3'- α -(*N*-benzyl methanesulfonamide)-5'-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy-thymidine (20).

Figure 26: Carbon NMR spectrum for 3'- α -(*N*-benzyl methanesulfonamide)-5'-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy-thymidine (20).

Chemical structure of compound 20 is shown in the center of the plot:

Cc1c[nH]c2c1O[C@H](C[C@@H](O)[C@H]2OSi(C)(C)C)N3C(=O)C(=O)N3

Compound 20 is a substituted pyrimidine derivative. It features a 4-methyl-2,4-dihydropyrimidin-5(1H)-one core. The sugar moiety is a 2,3,4-tri-O-isopropylidene-β-D-ribofuranose derivative, specifically a 2,3,4-tri-O-isopropylidene-β-D-ribofuranose derivative. The sugar is linked to the pyrimidine ring via a glycosidic bond at the C5 position. The pyrimidine ring is substituted with a methyl group at the C4 position and a carbonyl group at the C2 position. The sugar moiety is a 2,3,4-tri-O-isopropylidene-β-D-ribofuranose derivative.

Key IR peaks (Wavenumbers, cm⁻¹):

- 3408.43
- 3290.29
- 3069.84
- 3038.70
- 2954.32
- 2929.23
- 2884.32
- 2856.82
- 1698.84
- 1682.99
- 1471.50
- 1431.82
- 1330.83
- 1274.45
- 1154.48
- 1097.63
- 419.94
- 402.99

Figure 28: MS spectrum for 3'- α -(*N*-benzyl methanesulfonamide)-5'-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy-thymidine (20).

Figure 29: Proton NMR spectrum for 3-(*O*-*tert*-butyldimethylsilyl)-estrone (21).

CC1(C)CCC2(C3CC4C(C1)C(=O)CC[C@H]4C[C@@H]3CC[C@H]2C5=CC=C(C=C5)OSi(C)(C)C(C)C)C
(21)
 $C_{24}H_{36}O_2Si$
 Exact Mass: 384.24846
 Mol. Wt.: 384.62694

Figure 31: IR spectrum for 3-(*O*-*tert*-butyldimethylsilyl)-estrone (21).

DJ0047 Scan 62 RT=4:13 100%=54233 mv 26 Jul 102 13:57
HRP +CI 46E 3000 RES / DCI CH4

Figure 32: MS spectrum for 3-(*O*-*tert*-butyldimethylsilyl)-estrone (21).

Figure 33: Proton NMR spectrum for 17 α -(*N*-benzyl methanesulfonamide)-3-(*O*-*tert*-butyldimethylsilyl)- β -estradiol (22).

Figure 34: Carbon NMR spectrum for 17 α -(*N*-benzyl methanesulfonamide)-3-(*O*-*tert*-butyldimethylsilyl)- β -estradiol (22).

Figure 35: IR spectrum for 17 α -(*N*-benzyl methanesulfonamide)-3-(*O*-*tert*-butyldimethylsilyl)- β -estradiol (22).

Figure 36: MS spectrum for 17 α -(*N*-benzyl methylenesulfonamide)-3-(-*O*-*tert*-butyldimethylsilyl)- β -estradiol (22).

DJ0042 Scan 61 RT=3:59 100%=16685 mv 27 Sep 102 9:06
HRP +EI 12F 3000 RES / DEI

Figure 37: Proton NMR spectrum for 5 α -androstan-17 α -(*N*-benzyl methanesulfonamide)-3 β -17 β -diol (24).

Figure 38: Carbon NMR spectrum for 5 α -androstan-17 α -(*N*-benzyl methanesulfonamide)-3 β -17 β -diol (24).

Figure 39: IR spectrum for 5 α -androstan-17 α -(*N*-benzyl methanesulfonamide)-3 β - 17 β -diol (24).

Figure 40: MS spectrum for 5 α -androstan-17 α -(*N*-benzyl methanesulfonamide)-3 β - 17 β -diol (24).

Figure 41: Proton NMR spectrum for 5 α -androstan-17 α -(*N*-benzyl methanesulfonamide)-17 β -ol-3 β acetate (25).

Figure 42: Carbon NMR spectrum for 5 α -androstan-17 α -(*N*-benzyl methylenesulfonamide)-17 β -ol-3 β acetate (25).

Figure 43: IR spectrum for 5 α -androstan-17 α -(*N*-benzyl methanesulfonamide)-17 β -ol-3 β acetate (25).

Figure 44: MS spectrum for 5 α -androstan-17 α -(*N*-benzyl methanesulfonamide)-17 β -ol-3 β acetate (25).

Figure 45: Proton NMR spectrum for 3- β -(*N*-benzyl methanesulfonamide)- α -hydroxy-cholesterol (27).

Figure 46: Carbon NMR spectrum for 3-β-(*N*-benzyl methanesulfonamide)-α-hydroxy-cholesterol (27).

Figure 47: IR spectrum for 3- β -(*N*-benzyl methanesulfonamide)- α -hydroxy-cholesterol (27).

Figure 48: MS spectrum for 3-β-(*N*-benzyl methylenesulfonamide)-α-hydroxy-cholesterol (27).

DJ0065 Scan 53 RT=3:27 100%=18555 mv 4 Oct 102 14:35
HRP +CI 10F3X 3000 RES / DEI

Figure 49: Proton NMR spectrum for 2', 3'-isopropylidene-uridine-5'-aldehyde (28).

Figure 50: Carbon NMR spectrum for 2', 3'-isopropylidene-uridine-5'-aldehyde (28).

$C_{12}H_{14}N_2O_6$
Exact Mass: 282.08519
Mol. Wt.: 282.24944

Figure 51: IR spectrum for 2', 3'-isopropylidene-uridine-5'-aldehyde (28).

Figure 52: MS spectrum for 2', 3'-isopropylidene-uridine-5'-aldehyde (28).

DJ0067 Scan 20 RT=1:16 100%=152729 mv 7 Oct 102 13:21
HRP +CI 46F 3000 RES / DCI CH4

$C_{12}H_{15}N_2O_6^+$
Exact Mass: 283.09301
Mol. Wt.: 283.25738

Figure 53: MS spectrum for 5'-(*N*-benzyl methanesulfonamide)-2', 3'-isopropylidene-uridine (29).

50F PDT 1000 RES/FAB NBA/NA 14 Oct 2 11:05 am Peak Match +EI
 DJ0076 Gr 1 U1 490.0 scans 28-34 100%= 2247mV

Figure 54: MS spectrum for (1S)-(3-benzylsulfamoyl-2-hydroxy-1-methyl-propyl)-carbamic acid 9H-fluoren-9-ylmethyl ester (31).

Figure 55: Proton NMR spectrum for 7-oxo-heptanenitrile (32).

Figure 56: Carbon NMR spectrum for 7-oxo-heptanenitrile (32).

Figure 57: IR spectrum for 7-oxo-heptanenitrile (32).

Figure 58: MS spectrum for 7-oxo-heptanenitrile (32).

DJ0072 Scan 25 RT=1:36 100%=24751 mv 7 Aug 102 13:32
HRP +CI 72E 3000 RES / DCI CH4

Figure 59: Proton NMR spectrum for *N*-benzyl-7-cyano-2-hydroxy-heptanesulfonamide (33).

Figure 60: Carbon NMR spectrum for *N*-benzyl-7-cyano-2-hydroxy-heptanesulfonamide (33).

Figure 61: IR spectrum for *N*-benzyl-7-cyano-2-hydroxy-heptanesulfonamide (33).

Figure 62: MS spectrum for *N*-benzyl-7-cyano-2-hydroxy-heptanesulfonamide (33).

Figure 63: Proton NMR spectrum for *N*-Methyl methanesulfonamide (34).

Figure 64: Carbon NMR spectrum for *N*-Methyl methanesulfonamide (34).

Figure 65: IR spectrum for *N*-Methyl methanesulfonamide (34).

Figure 66: MS spectrum for *N*-Methyl methanesulfonamide (34).

File : C:\HPCHEM\1\DATA\DJ5.D
Operator : A.R.
Acquired : 24 Mar 103 9:38 am using AcqMethod TWA
Instrument : 5989 - In
Sample Name: 206F
Misc Info : DB-1,T.INJ.250,T.COL.50-250,15/MIN
Vial Number: 1

Figure 67: Proton NMR spectrum for 3'- α -(*N*-Methyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (35).

Figure 68: Detailed HMQC spectrum for 3'- α -(*N*-Methyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (35).

Figure 69: HMQC spectrum for 3'- α -(*N*-Methyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (35).

Figure 70: Carbon NMR spectrum for 3'- α -(*N*-Methyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (35).

Figure 71: IR spectrum for 3'- α -(*N*-Methyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (35).

Figure 72: MS spectrum for 3'- α -(*N*-Methyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (35).

Figure 73: Proton NMR spectrum for *N*-Ethyl methanesulfonamide (36).

Figure 74: Carbon NMR spectrum for *N*-Ethyl methanesulfonamide (36).

Figure 75: IR spectrum for *N*-Ethyl methanesulfonamide (36).

Figure 76: MS spectrum for *N*-Ethyl methanesulfonamide (36).

File : C:\HPCHEM\1\DATA\DJ15.D
Operator : A.R.
Acquired : 25 Mar 103 8:17 am using AcqMethod TWA
Instrument : 5989 - In
Sample Name: 204F
Misc Info : DB-1,T.INJ.250,T.COL.60-250,15/MIN
Vial Number: 1

Figure 77: Proton NMR spectrum for 3'- α -(*N*-Ethyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (37).

Figure 78: HMQC NMR spectrum for 3'- α -(*N*-Ethyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (37).

Figure 79: Detailed HMQC NMR spectrum for 3'- α -(*N*-Ethyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (37).

Figure 80: Carbon NMR spectrum for 3'- α -(*N*-Ethyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (37).

Figure 81: IR spectrum for 3'- α -(*N*-Ethyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (37).

Figure 82: MS spectrum for 3'- α -(*N*-Ethyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (37).

Figure 83: Proton NMR spectrum for *N*-isoPropyl methanesulfonamide (38).

Figure 84: Carbon NMR spectrum for *N-isopropyl* methanesulfonamide (38).

Figure 85: IR spectrum for *N*-*iso*Propyl methanesulfonamide (38).

Figure 86: MS spectrum for *N*-isoPropyl methanesulfonamide (38).

File : C:\HPCHEM\1\DATA\DJ12.D
Operator : A.R.
Acquired : 24 Mar 103 1:06 pm using AcqMethod TWA
Instrument : 5989 - In
Sample Name: 198F-IPR
Misc Info : DB-1,T.INJ.250,T.COL.50-250,15/MIN
Vial Number: 1

Figure 87: Proton NMR spectrum for 3'- α -(*N*-isoPropyl methylenesulfonamide)-2', 5'-bis-(*O*-tert-butyltrimethylsilyl)-3'- β -hydroxy- uridine (39).

Figure 88: HMQC NMR spectrum for 3'- α -(*N*-isoPropyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (39).

Figure 89: Carbon NMR spectrum for 3'- α -(*N*-isoPropyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (39).

Figure 90: IR spectrum for 3'- α -(*N*-isoPropyl methylenesulfonamide)-2', 5'-bis-(*O*-tert-butyltrimethylsilyl)-3'- β -hydroxy- uridine (39).

Figure 91: MS spectrum for 3'- α -(*N*-*iso*Propyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (39).

Figure 92: Proton NMR spectrum for *N*-Hexyl methanesulfonamide (40).

Figure 93: Carbon NMR spectrum for *N*-Hexyl methanesulfonamide (40).

Figure 94: IR spectrum for *N*-Hexyl methanesulfonamide (40).

Figure 95: MS spectrum for *N*-Hexyl methanesulfonamide (40).

Figure 96: Proton NMR spectrum for 3'- α -(*N*-Hexyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (41).

Figure 97: HMQC spectrum for 3'- α -(*N*-Hexyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (41).

Figure 98: Detailed HMQC spectrum for 3'- α -(*N*-Hexyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (41).

Figure 99: Carbon NMR spectrum 3'- α -(*N*-Hexyl methanesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (41).

Figure 100: IR spectrum for 3'- α -(*N*-Hexyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (41).

Figure 101: MS spectrum for 3'- α -(*N*-Hexyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (41).

Figure 102: Proton NMR spectrum for *N*-Phenyl methanesulfonamide (42).

Figure 103: Carbon NMR spectrum for *N*-Phenyl methanesulfonamide (42).

Figure 104: IR spectrum for *N*-Phenyl methanesulfonamide (42).

Figure 105: MS spectrum for *N*-Phenyl methanesulfonamide (42).

File : C:\HPCHEM\1\DATA\DJ1.D
Operator : A.R.
Acquired : 21 Mar 103 10:38 am using AcqMethod TWA
Instrument : 5989 - In
Sample Name: 214F
Misc Info : DB-1,T.INJ.250,T.COL.60-250,15/MIN
Vial Number: 1

Figure 106: Proton NMR spectrum for 3'- α -(*N*-Phenyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (43).

Figure 107: HMQC spectrum for 3'- α -(*N*-Phenyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (43).

Figure 108: Detailed HMQC spectrum for 3'- α -(*N*-Phenyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (43).

Figure 109: Carbon NMR spectrum for 3'- α -(*N*-Phenyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (43).

Figure 110: IR spectrum for 3'- α -(*N*-Phenyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (43).

Figure 111: MS spectrum for 3'- α -(*N*-Phenyl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (43).

Figure 112: Proton NMR spectrum for *N*-Benzhydryl methanesulfonamide (44).

Figure 113: Carbon NMR spectrum for *N*-Benzhydryl methanesulfonamide (44).

Figure 114: IR spectrum for *N*-Benzhydryl methanesulfonamide (44).

DJ0111 Scan 19 RT=1:16 100%=71243 mv 3 Jul 101 11:06
HRP +CI 112C 3000 RES / DCI CH4

Figure 116: Proton NMR spectrum for 3'- α -(*N*-Benzhydryl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (45).

Figure 117: HMQC spectrum for 3'- α -(*N*-Benzhydryl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (45).

Figure 118: Detailed HMQC spectrum for 3'- α -(*N*-Benzhydryl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (45).

Figure 119: Carbon NMR spectrum 3'- α -(*N*-Benzhydryl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (45).

Figure 120: IR spectrum for 3'- α -(*N*-Benzhydryl methylenesulfonamide)-2', 5'-bis-(*O*-*tert*-butyldimethylsilyl)-3'- β -hydroxy- uridine (45).

Figure 121: MS spectrum for 3'- α -(*N*-Benzhydryl methylenesulfonamide)-2', 5'-bis-(*O*-tert-butyltrimethylsilyl)-3'- β -hydroxy- uridine (45).

Figure 122: Proton NMR spectrum for 3-β-methylenesulfonamide-1,2: 5, 6-di-*O*-isopropylidene-α-D-allofuranose (46).

Figure 123: Carbon NMR spectrum for 3- β -methylenesulfonamide-1,2: 5, 6-di-*O*-isopropylidene- α -D-allofuranose (46).

Figure 124 : IR spectrum for 3- β -methylenesulfonamide-1,2: 5, 6-di-*O*-isopropylidene- α -D-allofuranose (46).

Figure 125: MS spectrum for 3- β -methylenesulfonamide-1,2: 5, 6-di-*O*-isopropylidene- α -D-allofuranose (46).

Figure 126: Proton NMR spectrum for 2-(*N*-benzyl methanesulfonamide)-2-hydroxy-1-phenylpropane (48).

Figure 127: Carbon NMR spectrum for 2-(*N*-benzyl methanesulfonamide)-2-hydroxy-1-phenylpropane (48).

Figure 128: IR spectrum for 2-(*N*-benzyl methanesulfonamide)-2-hydroxy-1-phenylpropane (48).

Figure 129: MS spectrum for 2-(*N*-benzyl methanesulfonamide)-2-hydroxy-1-phenylpropane (48).

Figure 130: Proton NMR spectrum for *N*-benzyl methanesulfonamide (49).

Figure 131: Carbon NMR spectrum for *N*-benzyl methanesulfonamide (49).

Figure 132: IR spectrum for *N*-benzyl methanesulfonamide (49).

Figure 133: MS spectrum for *N*-benzyl methanesulfonamide (49).

DJ0076 Scan 12 RT=1:08 100%=570706 mv 17 Apr 101 11:45
HRP +CI 138B 3000 RES / DCI CH4

$C_8H_{11}NO_2S$
Exact Mass: 185.05105
Mol. Wt.: 185.24448

Figure 134: Proton NMR spectrum for *N*-benzyl-deuteromethanesulfonamide (50).

Figure 135: Proton-decoupled deuterium NMR spectrum for *N*-benzyl-deuteromethanesulfonamide (50).

Figure 136: Carbon NMR spectrum for *N*-benzyl-deuteriomethanesulfonamide (50).

Figure 137: IR spectrum for *N*-benzyl-deuteriomethanesulfonamide (50).

Figure 138: MS spectrum for *N*-benzyl-deuteriomethanesulfonamide (50).

DJ0093 Scan 14 RT=0:52 100%=149844 mv 16 Oct 102 14:23
 HRP +CI 56F 3000 RES / DCI CH4

Figure 139: Proton NMR spectrum for 5'-deoxy-2', 3'-isopropylidene-5', 5'-(*N*, *N*'-diphenylethylenediamino)-uridine (56).

Figure 140: Carbon NMR spectrum for 5'-deoxy-2', 3'-isopropylidene-5', 5'-(*N*, *N'*-diphenylethylenediamino)-uridine (56).

Figure 141: IR spectrum for 5'-deoxy-2', 3'-isopropylidene-5', 5'-(*N*, *N*-diphenylethylenediamino)-uridine (56).

Figure 142: MS spectrum for 5'-deoxy-2', 3'-isopropylidene-5', 5'-(*N,N*-diphenylethylenediamino)-uridine (56).

Figure 143: Proton NMR spectrum for 7-hydroxy-heptanenitrile (58).

Figure 144: Carbon NMR spectrum for 7-hydroxy-heptanenitrile (58).

Figure 145: IR spectrum for 7-hydroxy-heptanenitrile (58).

DJ0073 Scan 37 RT=2:24 100%=63353 mv 7 Aug 102 13:40
HRP +CI 48E 3000 RES / DCI CH4

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