

Supporting Information

Experimental Section

All the reagents and solvents were purchased commercially and used without purification unless otherwise noted. ^1H and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded in CDCl_3 on a JEOL Alpha 500 or JEOL Lambda 500 spectrometers. Chemical shifts are reported in part per million (ppm) with tetramethylsilane (TMS) as an internal standard. Coupling constants (J) are given in hertz (Hz) and the abbreviations s, d, t, q, br and m refer to singlet, doublet, triplet, quartet, broad and multiplet respectively. All the assignments were made based on ^1H - ^1H COSY, HMQC and HMBC methods. Mass spectra (MS) were obtained with a Micromass Platform LCTM or a Micromass Q-ToFTM 2. Infrared spectra (IR) were recorded on a PerkinElmer Paragon 1000 spectrometer as KBr pellets and are reported as reciprocal centimeters (cm^{-1}). Elemental analyses were performed by a PerkinElmer 2400 CHN analyzer. Melting Points were measured using a Mettler FP61 melting point instrument and are uncorrected.

5-*O*-Desosaminyl-6-*O*-methylerythronolide A (1). A mixture of clarithromycin (20 g, 26.74 mmol) and 2M aqueous hydrochloric acid (200 mL) was stirred at room temperature for 3h. The reaction mixture was washed with CHCl_3 , and then was adjusted to pH10 with 2M NaOH solution, and was extracted with ethyl acetate. The organic layer was washed with brine, dried over MgSO_4 and concentrated in vacuo. The residue was crystallized from ethyl acetate to afford 8.31g (53%) of **1** as a colorless powder: mp 237 – 239 °C; IR (KBr) 1728, 1693 cm^{-1} ; HRMS (ES) m/z 590.3894 [(M+H)⁺; calcd for $\text{C}_{30}\text{H}_{56}\text{NO}_{10}$: 590.3904]. Anal. Calcd for $\text{C}_{30}\text{H}_{55}\text{NO}_{10}$: C, 61.10; H, 9.40; N, 2.37. Found: C, 60.83; H, 9.30; N, 2.36.

2, 3-Di-*O*-Acetyl-5-*O*-desosaminyl-6-*O*-methylerythronolide A (2). To a solution of **1** (10.0 g, 17.0 mmol) in THF (150 mL) was added 4'-dimethylaminopyridine (1.04 g, 8.51 mmol), pyridine (11.0 mL, 0.14 mol) and acetyl chloride (4.82 mL, 67.8 mmol). The reaction mixture was stirred at room temperature for 4days. After evaporation of the solvent, the residue was diluted with ethyl acetate and washed with brine. The organic layer was dried over MgSO_4 and concentrated under reduced pressure. The residue was purified by column chromatography eluting with MeOH/ CHCl_3 (2/ 98) to afford 7.82g (68%) of **2** as slight yellow amorphous. Crystals for X-ray analysis was obtained by recrystallization from

Et₂O/ *n*-hexane: mp 211–213 °C. IR (KBr) 1749, 1742, 1694 cm⁻¹
; HRMS (ES) *m/z* 674.4118 [(M+H)⁺; calcd for C₃₄H₆₀NO₁₂:674.4116]. Anal. Calcd for C₃₄H₅₉NO₁₂: C, 60.60; H, 8.83; N, 2.08. Found: C, 60.55; H, 8.95; N, 2.08.

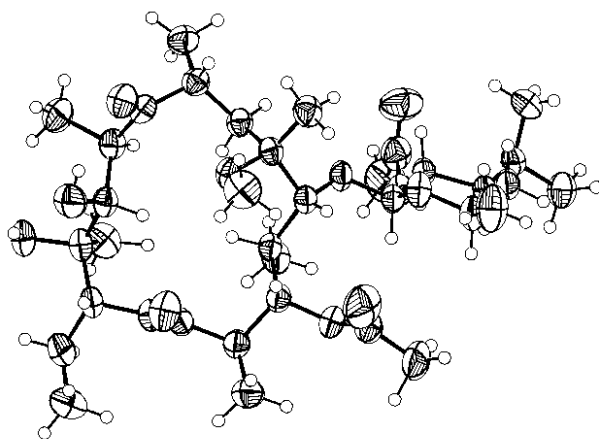


Chart 1. ORTEP drawing of the X-ray crystal structure of compound **2**

3-*O*-Acetyl-5-*O*-desosaminyl-6-*O*-methylethyronolide A (3a). A solution of **2** (3.47 g, 5.15 mmol) in MeOH (30 mL) was stirred at room temperature for 2 days. After evaporation of the solvent, the residue was purified by column chromatography eluting with MeOH/CHCl₃ (4/ 96) to afford 3.20g (98%) of **3a** as colorless amorphous: IR (KBr) 1739, 1688 cm⁻¹; HRMS (ES) *m/z* 632.4019[(M+H)⁺; calcd for C₃₂H₅₈NO₁₁: 632.4010]. Anal. Calcd for C₃₂H₅₇NO₁₁: C, 60.83; H, 9.09; N, 2.22. Found: C, 60.52; H, 9.08; N, 2.25.

2'-*O*-Acetyl-5-*O*-desosaminyl-6-*O*-methylethyronolide A (4)¹⁰. To a solution of **1** (11.78 g, 0.02 mol) in acetone (100 mL) was added acetic anhydride (2.27 mL, 24 mmol) at 0 °C. After stirring at room temperature for 6 h, the reaction mixture was evaporated in vacuo. The residue was partitioned between CH₂Cl₂ and the organic layer was saturated Na₂CO₃

solution, washed with brine, and dried over MgSO₄. After evaporation of the solvent, the residue was purified by crystallization from Et₂O/ *n*-hexane to give 12.17g (96%) of **4** as a colorless powder: LRMS (FAB) *m/z* 632(M+H)⁺.

3-O-Cyanoacetyl-5-O-desosaminyl-6-O-methylerythronolide A (3b). To a solution of **4** (816 mg, 1.29 mmol) in CH₂Cl₂ (20 mL) was added cyanoacetic acid (0.68 g, 7.84 mmol), 1-[3-(dimethylamino)propyl]-3-ethylcarbodiimide hydrochloride (EDC-HCl) (1.48 g, 7.78 mmol), and 4'-dimethylaminopyridine (DMAP) (0.16 g, 1.31 mmol) at 0°C. After stirring at room temperature for 2days, the reaction mixture was partitioned between ethyl acetate and water, and the pH of the aqueous layer was adjusted to 10 with 2M NaOH solution. The organic layer was washed with saturated NH₄Cl solution, and then dried over MgSO₄. After evaporation of the solvent under reduced pressure, the residue was dissolved in MeOH (20 mL) and the solution was stirred at room temperature for 2days. The reaction mixture was evaporated in vacuo, and the residue was purified by column chromatography eluting with MeOH/ CHCl₃/ 25% NH₄OH (15.5/ 1/ 0.1) to afford 0.62 g (73%) of **3b** as colorless amorphous, along with 91 mg (12%) of recovered starting material **1**. **3b**: IR (KBr) 1753, 1693 cm⁻¹; HRMS (ES) *m/z* 657.3958 [(M+H)⁺; calcd for C₃₃H₅₇N₂O₁₁: 657.3962]. Anal. Calcd for C₃₃H₅₆N₂O₁₁·H₂O: C, 58.73; H, 8.66; N, 4.15. Found: C, 58.97; H, 8.56; N, 4.12.

3-O-(N-tert-Butoxycarbonyl)glycyl-5-O-desosaminyl-6-O-methylerythronolide A (3d). 2'-Acetate of the title compound was prepared from compound **4** (1.0 g, 1.58 mmol) and *N*-(tert-butoxycarbonyl)glycine (553 mg, 3.16 mmol) by the same procedures as described for **3b**. Purification by column chromatography eluting with acetone/ *n*-hexane/ Et₃N (6/ 10/ 0.2) to afford 1.2 g (96%) of 2'-Ac **3d**. This product (0.8 g, 1.0mmol) was dissolved in MeOH (8 mL) and refluxed for 2h. After evaporation of the solvent, the residue was purified by column chromatography eluting with CHCl₃/ MeOH/ 25% NH₄OH (30/ 1/ 0.1) to afford 0.74 g (98%) of **3d** as colorless amorphous: IR (KBr) 1757, 1741, 1715 cm⁻¹; HRMS (ES) *m/z* 747.4635 [(M+H)⁺; calcd for C₃₇H₆₇N₂O₁₃: 747.4643]. Anal. Calcd for C₃₇H₆₆N₂O₁₃·¹/₂H₂O: C, 58.79; H, 8.93; N, 3.71. Found: C, 58.77; H, 8.92; N, 3.65.

3-O-(N-Benzyloxycarbonyl)glycyl-5-O-desosaminyl-6-O-methylerythronolide A (3e). The title compound was prepared from compound **4** (2.0 g, 3.17 mmol) and *N*-(tert-benzyloxycarbonyl)glycine (1.99 g, 9.52 mmol) by the same procedures as

described for **3b**. Purification by column chromatography gave 2.40 g (97%) of **3d** as colorless amorphous: IR (KBr) 1761, 1748, 1742, 1738, 1732, 1716, 1698 cm^{-1} ; HRMS (ES) m/z 781.4493 [(M+H)⁺; calcd for $\text{C}_{40}\text{H}_{65}\text{N}_2\text{O}_{13}$: 781.4487]. Anal. Calcd for $\text{C}_{40}\text{H}_{64}\text{N}_2\text{O}_{13}$: C, 61.52; H, 8.26; N, 3.59. Found: C, 61.32; H, 8.07; N, 3.54.

5-O-Desosaminyl-3-O-glycyl-6-O-methylerythronolide A (3c). A mixture of **3e** (2.0 g, 2.56 mmol), 10% palladium carbon (0.4 g), and ammonium formate (1.6 g, 25.4 mmol) in MeOH (20 mL) was stirred at room temperature for 1.5h. The reaction mixture was passed through a celite pad, and the filtrate was concentrated in vacuo. The residue was purified by crystallization from ethyl acetate/ *n*-hexane to afford 0.95 g (50%) of **3c** as colorless powder: mp 187–190 °C; IR (KBr) 1745, 1738, 1725, 1698 cm^{-1} ; HRMS (ES) m/z 647.4122 [(M+H)⁺; calcd for $\text{C}_{32}\text{H}_{59}\text{N}_2\text{O}_{11}$: 647.4119]. Anal. Calcd for $\text{C}_{32}\text{H}_{58}\text{N}_2\text{O}_{11}$: C, 59.42; H, 9.04; N, 4.33. Found: C, 59.41; H, 9.11; N, 4.24.

(E)-5-O-Desosaminyl-6-O-methylerythronolide A 9-O-(Phenylmethyl)oxime (5). A mixture of clarithromycin 9-oxime¹¹ and 2M aqueous hydrochloric acid (70 mL) was stirred at room temperature for 15h. The reaction mixture was washed with CHCl_3 , and then was adjusted to pH10 with 2M NaOH solution. The solution was extracted with ethyl acetate and washed with brine. The organic layer was dried over MgSO_4 and evaporated in vacuo. The residue was crystallized from 2-propanol to afford 2.55g (45%) of (*E*)-5-*O*-desosaminyl-6-*O*-methylerythronolide A 9-oxime. To a solution of the oxime (1.53 g, 2.52 mmol) in THF (30 mL) was added benzyl chloride (0.87 g, 6.87 mmol), powdered potassium hydroxide (0.34 g, 6.06 mmol), and tetra-*n*-butylammonium iodide (0.19 g, 0.51 mmol) at 0 °C. After stirring at room temperature for 22h, the reaction was quenched by addition of 50% dimethylamine solution and the reaction mixture was partitioned between ethyl acetate and water. The organic layer was washed with brine, dried over MgSO_4 and evaporated in vacuo. The residue was purified by column chromatography eluting with CHCl_3 / MeOH / 25% NH_4OH (21/ 1/ 0.1) to give 1.09 g (62%) of **5** as colorless amorphous, along with 0.53 g (27%) of 2'-*O*-phenylmethylated derivative of **5** as colorless amorphous: IR (KBr) 1735, 1630 cm^{-1} ; HRMS (ES) m/z 695.4485 [(M+H)⁺; calcd for $\text{C}_{37}\text{H}_{63}\text{N}_2\text{O}_{10}$: 695.4482]. Anal. Calcd for $\text{C}_{37}\text{H}_{62}\text{N}_2\text{O}_{10}$: C, 63.95; H, 8.99; N, 4.03. Found: C, 64.04; H, 9.06; N, 3.99.

(E)-5-O-Desosaminyl-3-O-tetrahydropyranyl-6-O-methylerythronolide A 9-O-(phenylmethyl)oxime (7). To a solution of **5** (1.09 g, 1.57 mmol) in acetone (20 mL) was added acetic anhydride (0.24 g, 2.35 mmol) at room temperature. After stirring at room temperature for 19h, the reaction mixture was partitioned between ethyl acetate and saturated NaHCO₃ solution. The organic layer was washed with brine, dried over MgSO₄ and evaporated in vacuo to afford 1.1 g of crude 2'-acetate **6**, which was used without further purification. To a solution of **6** in CH₂Cl₂ (20 mL) was added 3,4-dihydro-2H-pyran (0.40 g, 4.76 mmol), *p*-toluenesulfonic acid monohydrate (0.45 g, 2.37 mmol), and molecular sieves 4A (2.0 g) at room temperature. After stirring at room temperature for 9h, the reaction mixture was filtered and the filtrate was partitioned between CHCl₃ and saturated NaHCO₃ solution. The organic layer was dried over MgSO₄ and evaporated in vacuo. The residue was dissolved in MeOH (20 mL) and the solution was stirred at room temperature for 20h. The reaction mixture was evaporated in vacuo, and the residue was purified by column chromatography eluting with MeOH/ CHCl₃/ 25% NH₄OH (15.5/ 1/ 0.1) to afford and 0.68 g (56%) of **7** as colorless amorphous, along with 179 mg (16%) of recovered starting material **5**.

7: IR (KBr) 1734, 1630 cm⁻¹. HRMS (ES) *m/z* 779.5060 [(M+H)⁺; calcd for C₄₂H₇₁N₂O₁₁: 779.5058]. Anal. Calcd for C₄₂H₇₀N₂O₁₁: C, 64.76; H, 9.06; N, 3.60. Found: C, 64.68; H, 9.18; N, 3.54.

3-O-(2-Tetrahydropyranyl)-5-O-desosaminyl-6-O-methylerythronolide A (3f). A mixture of **7** (0.3 g, 0.39 mmol) and 10% palladium carbon (100 mg), ammonium formate (40 mg, 0.63 mmol), and 99% formic acid (0.12 mL, 3.13 mmol) in MeOH (3 mL) was stirred at 50 °C for 0.5h. After removal of the insoluble by filtration, evaporation of the solvent under reduced pressure afforded 244 mg of crude 3-O-(2-tetrahydropyranyl)-5-O-desosaminyl-6-O-methylerythronolide A 9-oxime. To a solution of the oxime (220 mg, 0.32 mmol) in EtOH (1 mL) and water (1.5 mL) was added sodium bisulfite (432 mg, 4.15 mmol), 99% formic acid (0.02 mL, 0.52 mmol). After refluxing for 30minutes, the mixture was adjusted to pH10 with 2M sodium hydroxide solution and extracted with ethyl acetate. The organic layer was dried over MgSO₄ and evaporated in vacuo. The residue was purified by column chromatography eluting with CHCl₃ / MeOH / 25% NH₄OH (21/ 1/ 0.1) to give 130 mg (51%) of **3f** as colorless amorphous: IR (KBr) 1735, 1694 cm⁻¹; HRMS (ES) *m/z* 674.4474 [(M+H)⁺; calcd for

C₃₅H₆₄NO₁₁: 674.4479]. Anal. Calcd for C₃₅H₆₃NO₁₁: C, 62.38; H, 9.42; N, 2.08. Found: C, 62.31; H, 9.43; N, 1.96.

3-O-(2-Nitro)phenyl-5-O-desosaminyl-6-O-methylerythronolide A (3g). To a solution of 1.18g (2.0 mmol) of **1** in THF (15 mL) was added 1-fluoro-2-nitrobenzene (2.1 mL, 20.0 mmol) and 60% sodium hydride (0.28 g, 17.0 mmol) at 0 °C, and the reaction mixture was allowed to warm to room temperature. After stirring for 24h, the reaction mixture was partitioned between ethyl acetate and brine. The organic layer was dried over MgSO₄ and evaporated in vacuo. The residue was purified by column chromatography eluting with CHCl₃ / MeOH / 25% NH₄OH (95/ 5/ 0.5) to give 560mg (39%) of **3g** as slight yellow amorphous: IR (KBr) 1732, 1694 cm⁻¹; HRMS (ES) *m/z* 711.4058 [(M+H)⁺; calcd for C₃₆H₅₉N₂O₁₂: 711.4068]. Anal. Calcd for C₃₆H₅₈N₂O₁₂·H₂O: C, 59.32; H, 8.30; N, 3.84. Found: C, 59.02; H, 7.91; N, 3.70.

3-O-Phenylacetyl-5-O-desosaminyl-6-O-methylerythronolide A (3h). To a solution of **4** (1.26 g, 2.0 mmol) in pyridine (6.0 mL) was added 4-dimethylaminopyridine (122 mg, 1.0 mmol) and phenylacetyl chloride (0.66 mL, 5.0mmol) at 0 °C. After stirring at room temperature for 22 h, the reaction mixture was partitioned between ethyl acetate and brine. The organic layer was dried over MgSO₄ and evaporated in vacuo. The residue was purified by column chromatography eluting with acetone/ *n*-hexane/ Et₃N (4/ 10/ 0.05) to give 730 mg (49%) of 2'-Ac-**3h**. This acetate was dissolved in methanol (10 mL) and the solution was refluxed for 6h, and then evaporated in vacuo. The residue was diluted with ethyl acetate and washed with saturated NaHCO₃ solution, brine successively, dried over MgSO₄ and evaporated in vacuo. The residue was purified by column chromatography eluting with CHCl₃ / MeOH / 25% NH₄OH (20/ 1/ 0.1) to afford 490 mg (71%) of **3h** as slight yellow amorphous:IR (KBr) 1745, 1734, 1695cm⁻¹; HRMS (ES) *m/z* 708.4308 [(M+H)⁺; calcd for C₃₈H₆₂NO₁₁: 708.4323]. Anal. Calcd for C₃₈H₆₁NO₁₁: C, 64.47; H, 8.69; N, 1.98. Found: C, 64.48; H, 8.76; N, 1.88.

3-O-Benzylsulfonyl-5-O-desosaminyl-6-O-methylerythronolide A (3i). To a solution of **4** (1.26 g, 2.0 mmol) in CH₂Cl₂ (10 mL) and pyridine (2 mL) was added benzylsulfonyl chloride (953 mg, 5.0 mmol) at 0 °C. After stirring at room temperature for 23h, the reaction mixture was partitioned between ethyl acetate and saturated NaHCO₃ solution. The

organic layer was washed with saturated NH₄Cl solution and brine successively, dried over MgSO₄ and evaporated in vacuo. The residue was purified by column chromatography eluting with acetone/ *n*-hexane/ Et₃N (4/ 10/ 0.05) to give 1.37g (87%) of 2'-Ac-**3i** as colorless amorphous. The acetate (0.48 g, 0.61 mmol) was dissolved in MeOH (10 mL) and the solution was stirred at room temperature for 20.5h. After evaporation of the solvent, the residue was purified by column chromatography eluting with CHCl₃ / MeOH / 25% NH₄OH (21/ 1/ 0.1) to afford 210mg (46%) of **3i** as colorless amorphous: IR (KBr) 1738, 1693 cm⁻¹; HRMS (ES) *m/z* 744.3981 [(M+H)⁺; calcd for C₅₀H₅₄N₃OS: 744.3988]. Anal. Calcd for C₃₇H₆₁NO₁₂S·³/₂H₂O: C, 57.64; H, 8.37; N, 1.82. Found: C, 57.69; H, 8.04; N, 1.79.

3-O-(4-Nitrophenyl)acetyl-5-O-desosaminyl-6-O-methylerythronolide A (3j). 2'-Acetate of the title compound was prepared from compound **4** (5.0 g, 7.91 mmol) and 4-nitrophenylacetic acid (4.30 g, 23.7 mmol) by the same procedures as described for **3b**. Purification by column chromatography eluting with acetone/ *n*-hexane/ Et₃N (6/ 10/ 0.2) afforded 5.03 g (80%) of 2'-*O*-acetyl-**3j** as light brown amorphous. A solution of this product in MeOH (50 mL) was stirred at room temperature for 15h, and evaporated in vacuo. The residue was crystallized from isopropylether to afford 3.86 g (81%) of **3j** as colorless powder: mp 157–159 °C; IR (KBr) 1742, 1732, 1694 cm⁻¹; HRMS (ES) *m/z* 753.4189 [(M+H)⁺; calcd for C₃₈H₆₁N₂O₁₃: 753.4174]. Anal. Calcd for C₃₈H₆₀N₂O₁₃·¹/₂H₂O: C, 59.90; H, 8.07; N, 3.68. Found: C, 59.79; H, 7.97; N, 3.70.

3-O-(4-Nitro)benzoyl-5-O-desosaminyl-6-O-methylerythronolide A (3k). 2'-Acetate of the title compound was prepared from compound **4** (5.0 g, 7.91 mmol) and 4-nitrobenzoic acid (3.97 g, 23.7 mmol) by the same procedures as described for **3b**. Purification of the crude product by column chromatography eluting with acetone/ *n*-hexane/ Et₃N (6/ 10/ 0.2) afforded to 6.03 g (98%) of 2'-Ac-**3k** as slight yellow amorphous. This product in MeOH (50 mL) was stirred at room temperature for 15h, and evaporated in vacuo. The residue was purified by column chromatography eluting with acetone/ *n*-hexane/ Et₃N (6/ 10/ 0.2) to afford 5.25 g (92%) of **3i** as slight yellow amorphous: IR (KBr) 1740, 1694, 1610 cm⁻¹; HRMS (ES) *m/z* 739.4006 [(M+H)⁺; calcd for C₃₇H₅₉N₂O₁₃: 739.4017]. Anal. Calcd for C₃₇H₅₈N₂O₁₃: C, 60.15; H, 7.91; N, 3.79. Found: C, 60.05; H, 8.03; N, 3.63.

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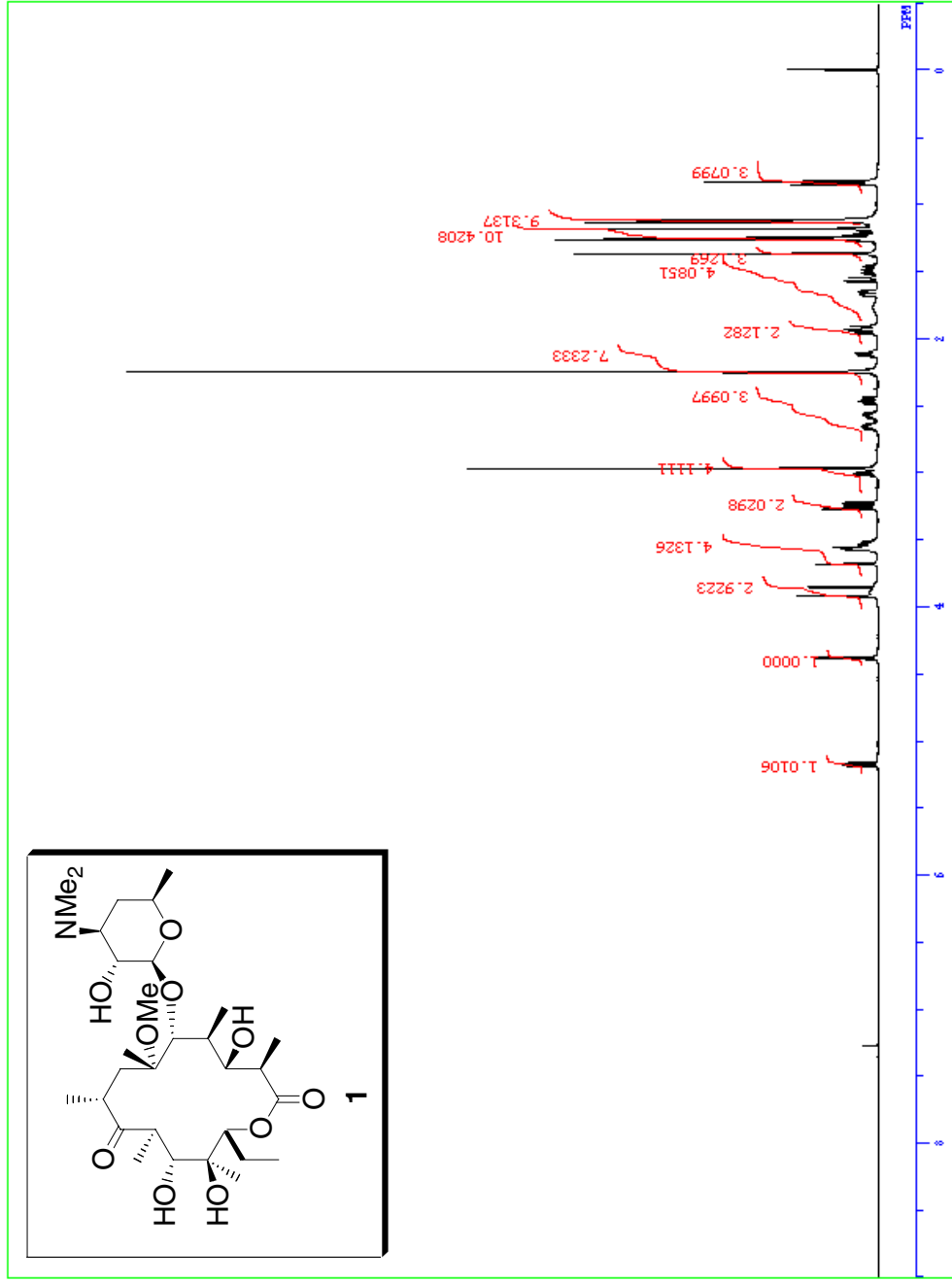


Table 1. Proton and carbon assignments of compound **1**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.66	m	-	C ₁	175.0
2-Me	1.26	m	-	C ₂	44.6
C ₃ -H	3.50 - 3.58	m	-	C ₂ -Me	15.2
C ₄ -H	2.12	dq	7.3	C ₃	79.0
4-Me	1.13	m	-	C ₄	35.9
C ₅ -H	3.69	d	1.8	C ₄ -Me	8.2
6-Me	1.37	s	-	C ₅	88.5
6-OMe	2.97	s	-	C ₆	78.0
C ₇ -H	1.56	dd	4.6, 1.8	C ₆ -Me	18.8
	1.90 - 1.96	m	-	C ₆ -OMe	49.5
C ₈ -H	2.58	m	-	C ₇	38.7
8-Me	1.13	m	-	C ₈	45.5
C ₁₀ -H	3.01	m	-	C ₈ -Me	17.7
10-Me	1.13	m	-	C ₉	220.7
C ₁₁ -H	3.86	d	1.2	C ₁₀	37.5
12-Me	1.18	s	-	C ₁₀ -Me	12.6
C ₁₃ -H	5.18	dd	11.0, 2.4	C ₁₁	69.8
C ₁₄ -H	1.49	m	-	C ₁₂	74.2
	1.90 - 1.96	m	-	C ₁₂ -Me	16.2
C ₁₅ -H	0.84	t	7.6	C ₁₃	76.6
C ₁ '-H	4.38	d	7.3	C ₁₄	21.4
C ₂ '-H	3.24	dd	10.4, 7.3	C ₁₅	10.4
C ₃ '-H	2.47	ddd	12.2, 10.4, 4.3	C ₁ '	106.8
C ₃ '-NMe ₂	2.25	s	-	C ₂ '	70.7
C ₄ '-H	1.20 - 1.28	m	-	C ₃ '	65.7
	1.67	dq	12.2, 1.8, 1.8, 1.8	C ₃ '-NMe ₂	40.2
C ₅ '	3.50 - 3.58	m	-	C ₄ '	28.0
C ₅ '-Me	1.26	m	-	C ₅ '	70.3
OH	3.27	s	-	C ₅ '-Me	21.2
	3.50 - 3.58	m	-		
	3.89	br	-		
	3.92	s	-		

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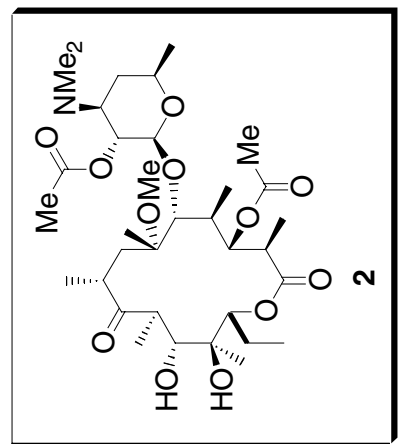
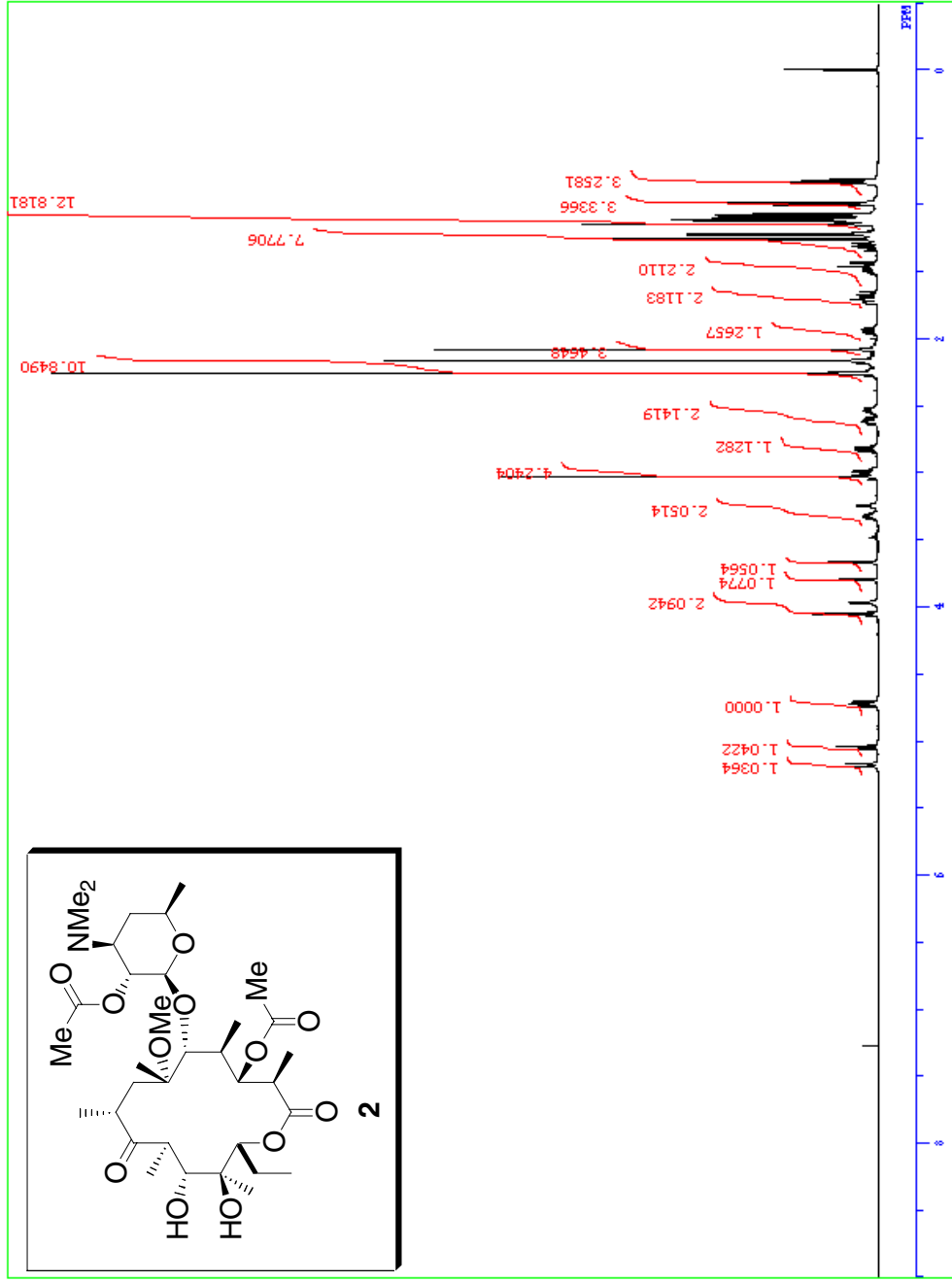


Table 2. Proton and carbon assignments of compound **2**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.83	m	-	C ₁	173.6
2-Me	1.08	d	6.7	C ₂	42.7
C ₃ -H	5.05	d	11.0	C ₂ -Me	15.2
C ₄ -H	2.21 - 2.14	m	-	C ₃	77.8
4-Me	0.99	d	7.3	C ₄	35.8
C ₅ -H	3.67	d	3.1	C ₄ -Me	8.7
6-Me	1.26	s	-	C ₅	79.8
6-OMe	3.03	s	-	C ₆	77.9
C ₇ -H	1.66 - 1.74	m	-	C ₆ -Me	19.3
	1.45	dd	14.7, 1.2	C ₆ -OMe	50.1
C ₈ -H	2.54	m	-	C ₇	38.3
8-Me	1.11	d	7.3	C ₈	45.3
C ₁₀ -H	2.99	m	-	C ₈ -Me	17.9
10-Me	1.12	d	6.7	C ₉	220.7
C ₁₁ -H	3.80	d	1.2	C ₁₀	37.3
12-Me	1.15	s	-	C ₁₀ -Me	12.5
C ₁₃ -H	5.18	dd	11.0, 2.4	C ₁₁	69.5
C ₁₄ -H	1.94	m	-	C ₁₂	74.2
	1.46 - 1.52	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.83	t	7.3	C ₁₃	77.0
C ₁ '-H	4.06	d	7.3	C ₁₄	21.2
C ₂ '-H	4.73	dd	10.4, 7.3	C ₁₅	10.4
C ₃ '-H	2.62	m	-	C ₁ '	100.6
C ₃ '-NMe ₂	2.26	m	-	C ₂ '	71.3
C ₄ '-H	1.66 - 1.74	m	-	C ₃ '	63.5
	1.27 - 1.34	m	-	C ₃ '-NMe ₂	40.6
C ₅ '	3.33	m	-	C ₄ '	30.7
C ₅ '-Me	1.23	d	6.1	C ₅ '	69.3
OH	3.25	br	-	C ₅ '-Me	20.9
	3.97	br	-	C ₃ '-COCH ₃	21.2
C ₂ '-COCH ₃	2.08	s	-	C ₂ '-COCH ₃	21.4
C ₃ -COCH ₃	2.17	s	-	C ₂ '-COCH ₃	169.8
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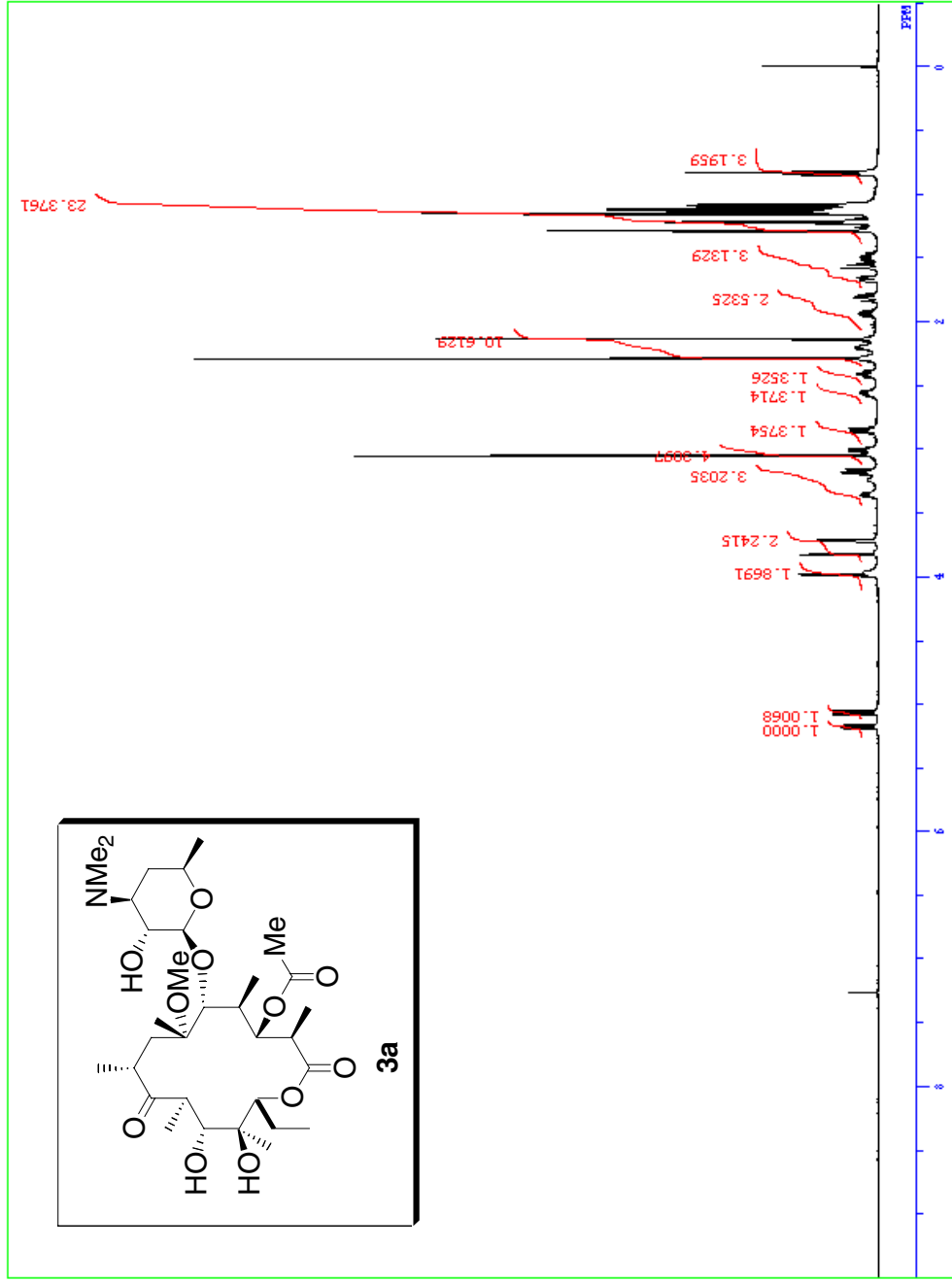


Table 3. Proton and carbon assignments of compound **3a**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.86	m	-	C ₁	173.7
2-Me	1.08	d	6.7	C ₂	42.8
C ₃ -H	5.07	d	11.0	C ₂ -Me	15.2
C ₄ -H	2.21	m	-	C ₃	77.9
4-Me	1.15	d	7.9	C ₄	36.0
C ₅ -H	3.72	d	3.1	C ₄ -Me	8.9
6-Me	1.29	s	-	C ₅	80.9
6-OMe	3.05	s	-	C ₆	77.9
C ₇ -H	1.57	dd	14.7, 11.6	C ₆ -Me	19.3
	1.81	dd	14.7, 1.2	C ₆ -OMe	50.2
C ₈ -H	2.56	m	-	C ₇	38.7
8-Me	1.11	d	7.3	C ₈	45.4
C ₁₀ -H	3.01	m	-	C ₈ -Me	18.0
10-Me	1.13	d	7.3	C ₉	220.7
C ₁₁ -H	3.83	d	1.2	C ₁₀	37.3
12-Me	1.16	s	-	C ₁₀ -Me	12.5
C ₁₃ -H	5.18	dd	11.0, 1.8	C ₁₁	69.5
C ₁₄ -H	1.50	m	-	C ₁₂	74.2
	1.94	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.84	t	7.3	C ₁₃	77.0
C ₁ '-H	3.99	d	7.3	C ₁₄	21.2
C ₂ '-H	3.18	dd	7.3	C ₁₅	10.4
C ₃ '-H	2.41	m	-	C ₁ '	103.1
C ₃ '-NMe ₂	2.29	s	-	C ₂ '	70.4
C ₄ '-H	1.16 – 1.23	m	-	C ₃ '	66.1
	1.67	dq	12.2, 1.8, 1.8, 1.8	C ₃ '-NMe ₂	40.3
C ₅ '	3.36	m	-	C ₄ '	28.5
C ₅ '-Me	1.22	d	6.1	C ₅ '	69.7
OH	3.24	br	-	C ₅ '-Me	21.1
	3.97	br	-	C ₃ -COCH	21.3
C ₃ -COCH ₃	2.14	s	-	C ₃ -COCH	170.5

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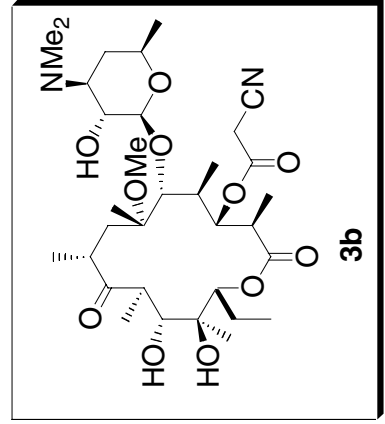
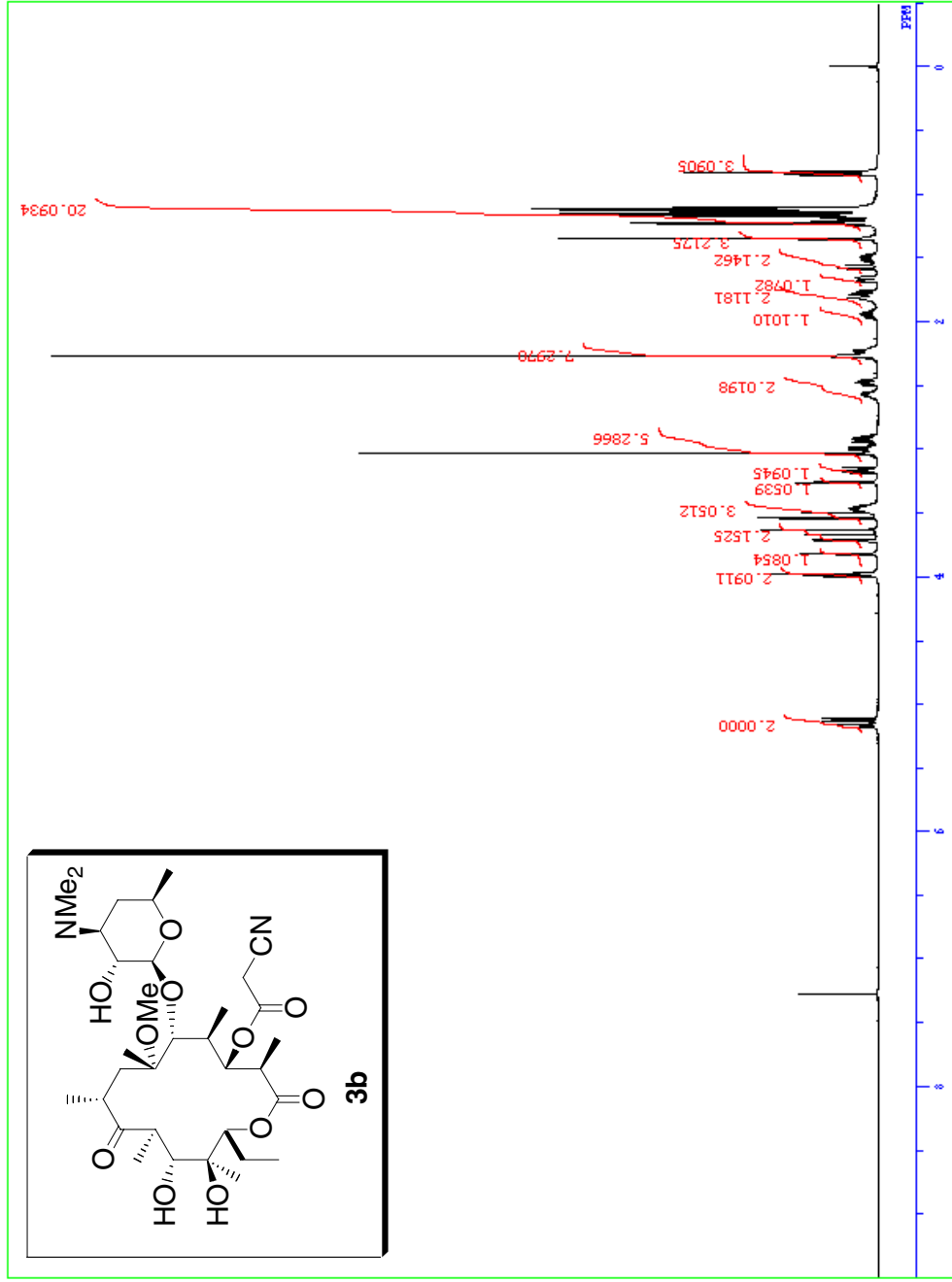


Table 4. Proton and carbon assignments of compound **3b**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.93	m	-	C ₁	173.1
2-Me	1.11	d	7.3	C ₂	42.7
C ₃ -H	5.13	d	11.0	C ₂ -Me	15.2
C ₄ -H	2.23	m	-	C ₃	81.7
4-Me	1.17	d	7.3	C ₄	36.4
C ₅ -H	3.72	d	3.1	C ₄ -Me	8.8
6-Me	1.35	s	-	C ₅	84.3
6-OMe	3.04	s	-	C ₆	78.0
C ₇ -H	1.79	dd	14.7, 11.6	C ₆ -Me	19.4
	1.57	dd	14.7, 1.8	C ₆ -OMe	49.9
C ₈ -H	2.57	m	-	C ₇	38.7
8-Me	1.12	d	7.3	C ₈	45.2
C ₁₀ -H	3.00	m	-	C ₈ -Me	17.8
10-Me	1.13	d	7.3	C ₉	220.6
C ₁₁ -H	3.82	d	1.2	C ₁₀	37.3
12-Me	1.16	s	-	C ₁₀ -Me	12.4
C ₁₃ -H	5.18	dd	11.0, 2.4	C ₁₁	69.5
C ₁₄ -H	1.95	m	-	C ₁₂	74.1
	1.50	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.84	t	7.3	C ₁₃	77.4
C ₁ '-H	3.99	d	7.3	C ₁₄	21.1
C ₂ '-H	3.17	dd	10.4, 7.3	C ₁₅	10.4
C ₃ '-H	2.48	m	-	C ₁ '	105.1
C ₃ '-NMe ₂	2.27	s	-	C ₂ '	70.4
C ₄ '-H	1.20 – 1.25	m	-	C ₃ '	65.9
	1.67	dq	12.8, 1.8, 1.8, 1.8	C ₃ '-NMe ₂	40.3
C ₅ '	3.48	m	-	C ₄ '	28.1
C ₅ '-Me	1.23	d	6.1	C ₅ '	69.9
OH	3.26	s	-	C ₅ '-Me	21.1
	3.98	s	-	C ₃ -COCH ₂ CN	25.0
C ₃ -COCH ₂ CN	3.52	d	18.9	C ₃ -COCH ₂ CN	113.1
	3.65	d	18.9	C ₃ -COCH ₂ CN	163.4

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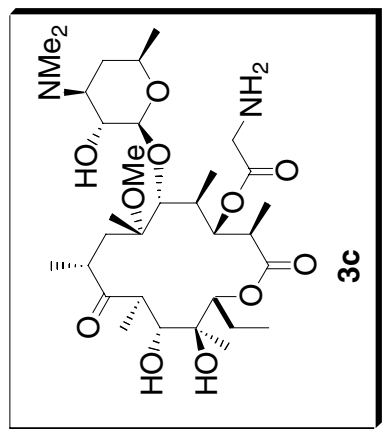
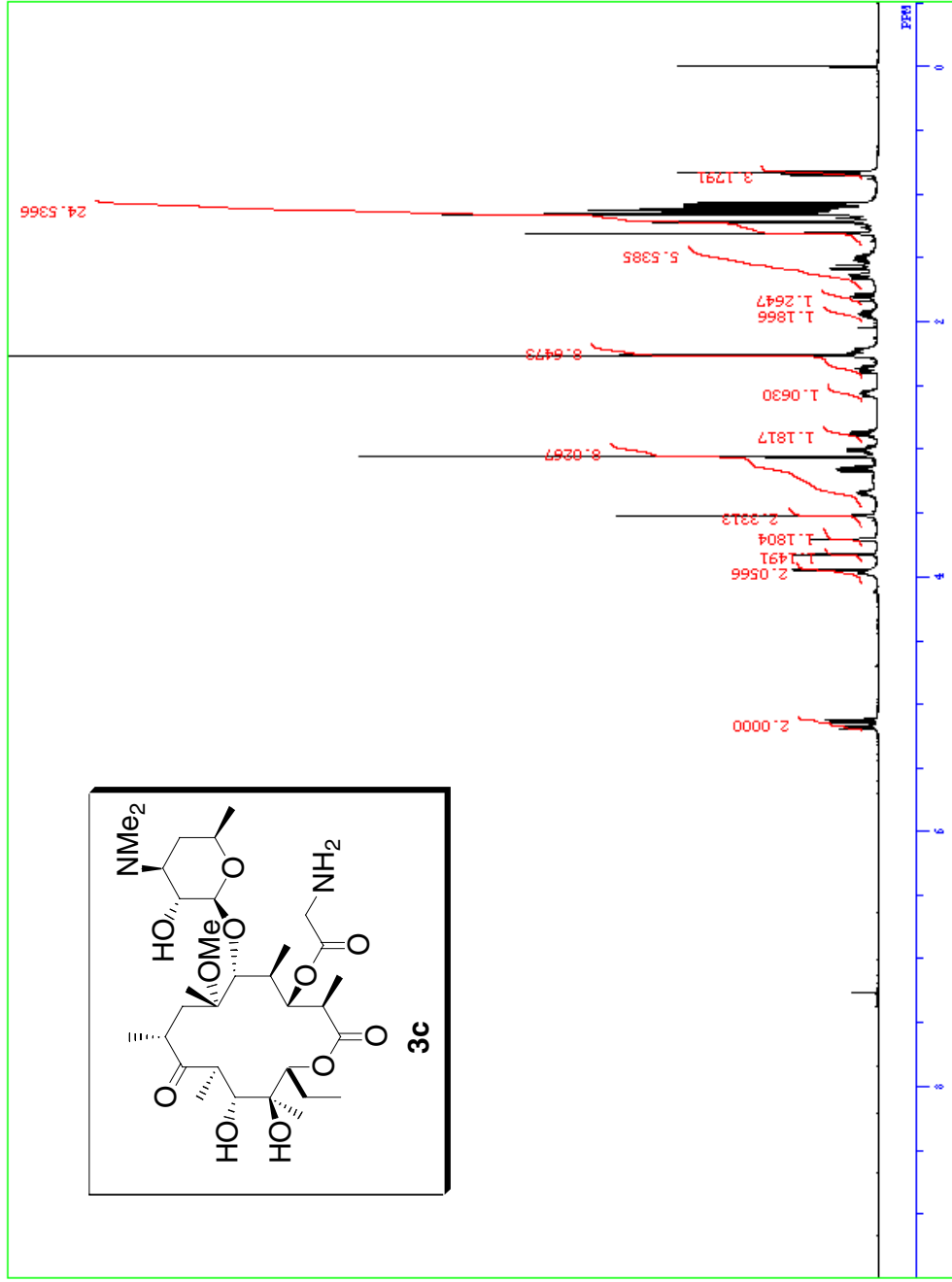


Table 5. Proton and carbon assignments of compound **3c**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.88	m	-	C ₁	173.6
2-Me	1.08	d	6.7	C ₂	42.8
C ₃ -H	5.13	d	11.0	C ₂ -Me	15.2
C ₄ -H	2.20 – 2.27	m	-	C ₃	78.7
4-Me	1.15	d	6.1	C ₄	36.2
C ₅ -H	3.71	d	3.4	C ₄ -Me	8.9
6-Me	1.31	s	-	C ₅	81.8
6-OMe	3.06	s	-	C ₆	78.0
C ₇ -H	1.81	dd	14.6, 11.9	C ₆ -Me	19.3
	1.57	dd	14.6, 1.5	C ₆ -OMe	50.1
C ₈ -H	2.57	m	-	C ₇	38.7
8-Me	1.12	d	7.9	C ₈	45.4
C ₁₀ -H	3.01	m	-	C ₈ -Me	17.9
10-Me	1.13	d	7.0	C ₉	220.6
C ₁₁ -H	3.83	d	1.5	C ₁₀	37.3
12-Me	1.16	s	-	C ₁₀ -Me	12.5
C ₁₃ -H	5.19	dd	11.3, 2.1	C ₁₁	69.5
C ₁₄ -H	1.95	m	-	C ₁₂	74.2
	1.50	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.84	t	7.3	C ₁₃	77.1
C ₁ '-H	3.95	d	7.3	C ₁₄	21.2
C ₂ '-H	3.16	dd	10.4, 7.3	C ₁₅	10.4
C ₃ '-H	2.37	m	-	C ₁ '	103.7
C ₃ '-NMe ₂	2.27	s	-	C ₂ '	70.4
C ₄ '-H	1.16 – 1.19	m	-	C ₃ '	66.1
	1.65	dq	12.8, 1.8, 1.8, 1.8	C ₃ '-NMe ₂	40.3
C ₅ '	3.35	m	-	C ₄ '	28.4
C ₅ '-Me	1.22	d	6.1	C ₅ '	69.8
C ₃ -COCH ₂ NH ₂	3.53	s	-	C ₅ '-Me	21.2
OH	3.98	br	-	C ₃ -COCH ₂ NH	44.3
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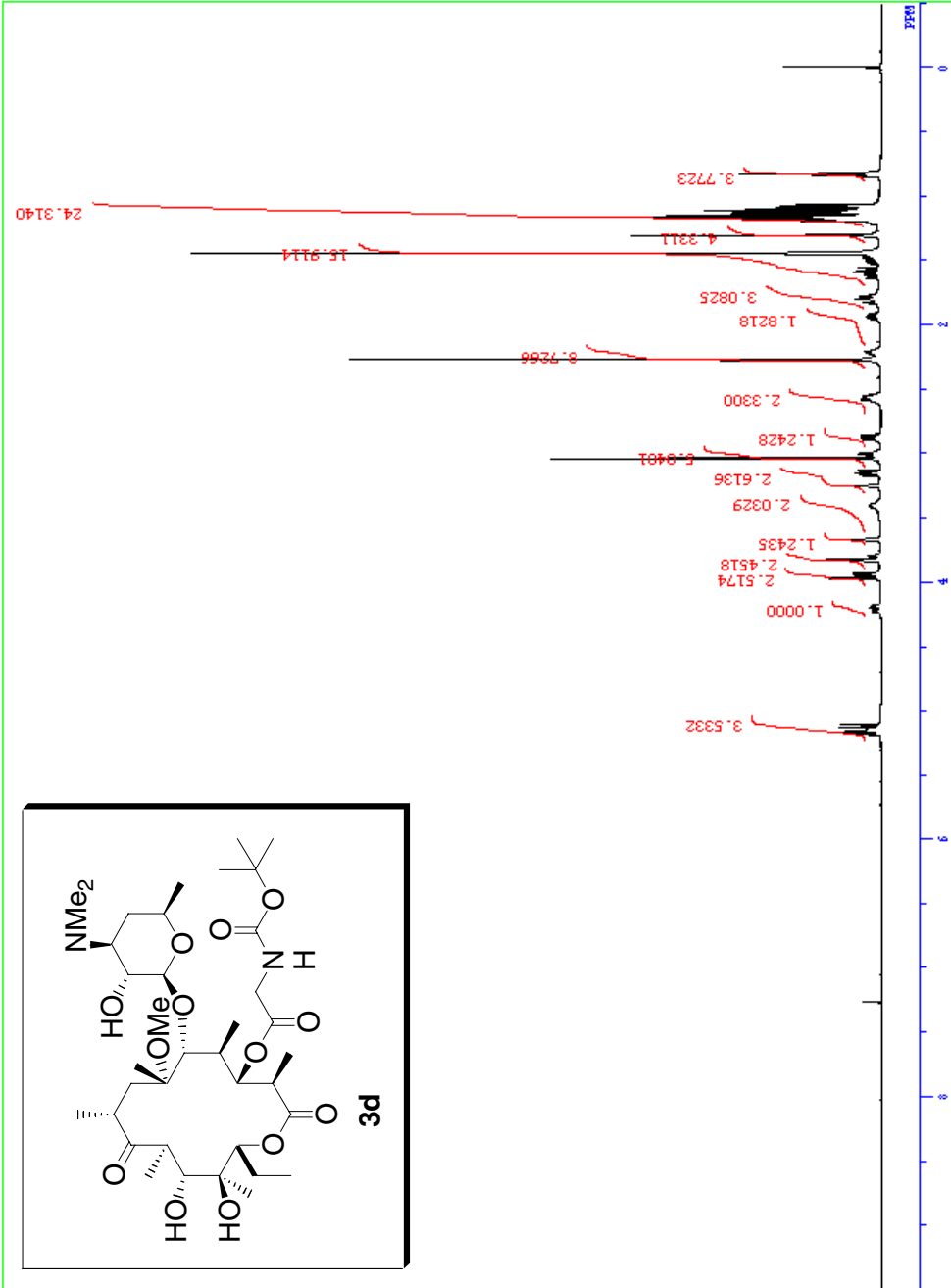
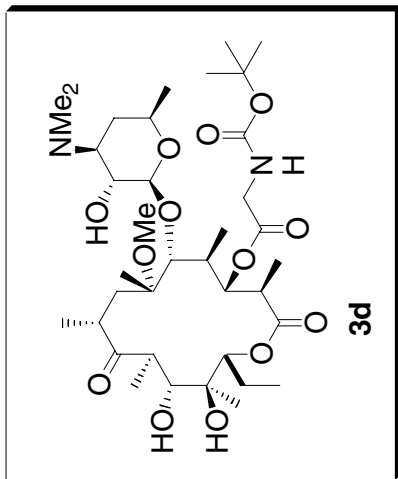


Table 6. Proton and carbon assignments of compound **3d**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	Peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.88	m	-	C ₁	173.5
2-Me	1.08	d	7.3	C ₂	42.9
C ₃ -H	5.13	d	11.6	C ₂ -Me	15.2
C ₄ -H	2.22	m	-	C ₃	79.5
4-Me	1.17	d	5.5	C ₄	36.2
C ₅ -H	3.68	d	3.1	C ₄ -Me	8.9
6-Me	1.31	s	-	C ₅	82.3
6-OMe	3.04	s	-	C ₆	78.0
C ₇ -H	1.58	d	14.6	C ₆ -Me	19.3
	1.80	dd	14.6, 12.2	C ₆ -OMe	50.0
C ₈ -H	2.53 – 2.62	m	-	C ₇	38.7
8-Me	1.11	d	8.5	C ₈	45.3
C ₁₀ -H	3.00	m	-	C ₈ -Me	17.9
10-Me	1.13	d	6.7	C ₉	220.7
C ₁₁ -H	3.83	s	-	C ₁₀	37.3
12-Me	1.16	s	-	C ₁₀ -Me	12.5
C ₁₃ -H	5.18	dd	11.6, 2.4	C ₁₁	69.5
C ₁₄ -H	1.45 – 1.53	m	-	C ₁₂	74.2
	1.94	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.83	t	7.3	C ₁₃	77.2
C ₁ '-H	3.94	d	7.3	C ₁₄	21.2
C ₂ '-H	3.16	dd	10.4, 7.3	C ₁₅	10.4
C ₃ '-H	2.53 – 2.62	m	-	C ₁ '	103.9
C ₃ '-NMe ₂	2.28	s	-	C ₂ '	70.5
C ₄ '-H	1.16 – 1.22	m	-	C ₃ '	65.4
	1.63	m	-	C ₃ '-NMe ₂	40.2
C ₅ '	3.41	m	-	C ₄ '	28.4
C ₅ '-Me	1.19	d	6.1	C ₅ '	69.5
OH	3.26	br s	-	C ₅ '-Me	21.1
	3.98	s	-	-CO ₂ C(CH ₃) ₃	28.3
C ₃ -COCH ₂ NH-	4.21	dd	18.3, 7.3	-CO ₂ C(CH ₃) ₃	79.9
	3.79 – 3.83	m	-	C ₃ -COCH ₂ NH-	42.6
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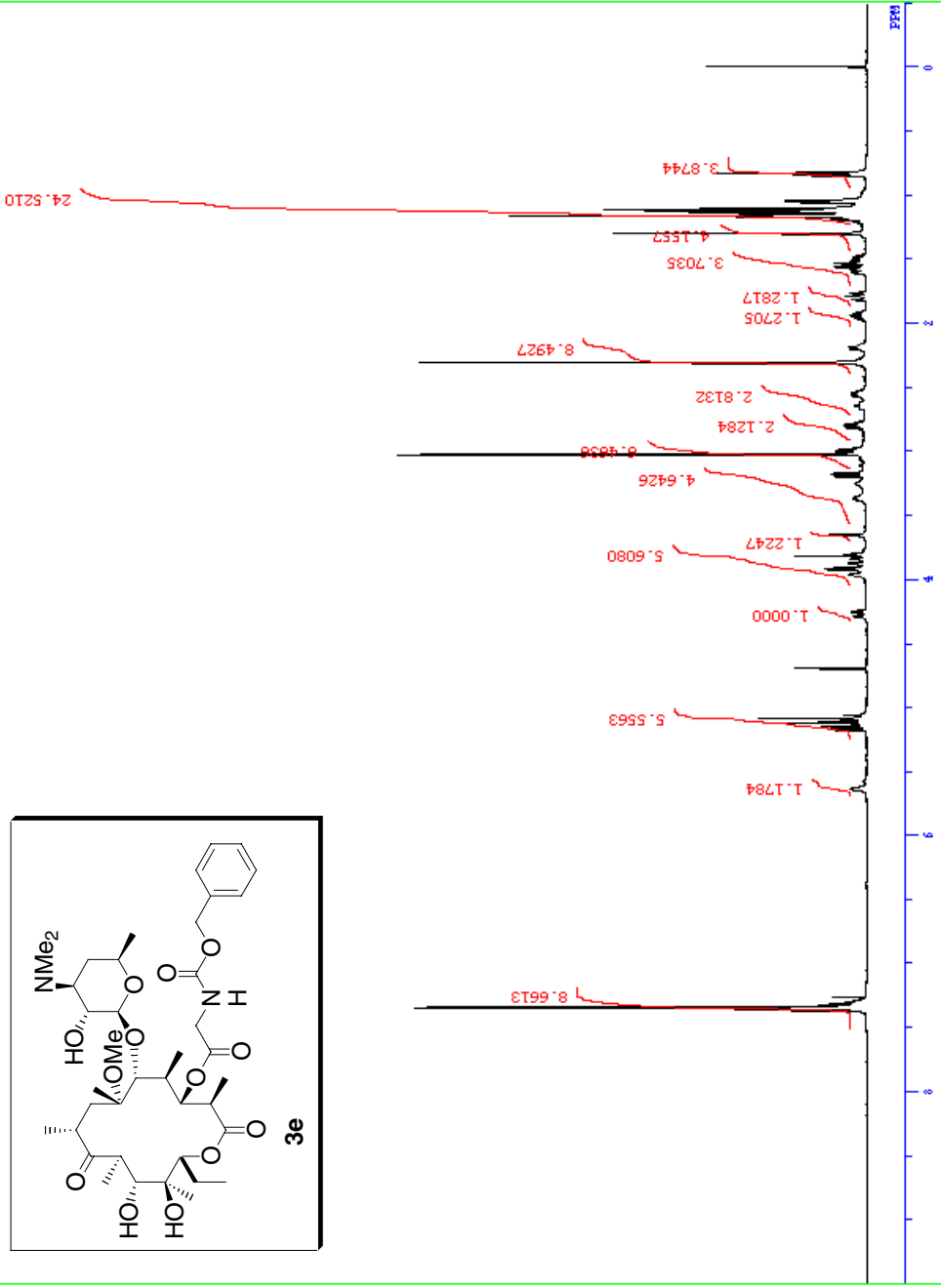


Table 7. Proton and carbon assignments of compound **3e**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.80	m	-	C ₁	173.5
2-Me	1.05	d	6.7	C ₂	42.9
C ₃ -H	5.10	d	10.7	C ₂ -Me	15.2
C ₄ -H	2.19	m	-	C ₃	79.8
4-Me	1.13	d	8.5	C ₄	36.2
C ₅ -H	3.66	d	2.7	C ₄ -Me	8.9
6-Me	1.30	s	-	C ₅	83.2
6-OMe	3.03	s	-	C ₆	78.0
C ₇ -H	1.55	dd	14.6, 1.2	C ₆ -Me	19.4
	1.79	dd	14.6, 11.9	C ₆ -OMe	50.0
C ₈ -H	2.56	m	-	C ₇	38.7
8-Me	1.11	d	7.3	C ₈	45.3
C ₁₀ -H	3.00	m	-	C ₈ -Me	17.9
10-Me	1.16	s	-	C ₉	220.7
C ₁₁ -H	3.82	d	1.5	C ₁₀	37.3
12-Me	0.84	t	7.3	C ₁₀ -Me	12.5
C ₁₃ -H	5.17	dd	11.3, 2.4	C ₁₁	69.5
C ₁₄ -H	1.51	m	-	C ₁₂	74.2
	1.95	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.84	t	7.3	C ₁₃	77.1
C ₁ '-H	3.92	d	7.0	C ₁₄	21.2
C ₂ '-H	3.18	dd	10.4, 7.3	C ₁₅	10.4
C ₃ '-H	2.64	m	-	C ₁ '	104.1
C ₃ '-NMe ₂	2.31	s	-	C ₂ '	70.3
C ₄ '-H	1.13 – 1.19	m	-	C ₃ '	65.7
	1.60	m	-	C ₃ '-NMe ₂	40.0
C ₅ '	3.37	m	-	C ₄ '	28.6
C ₅ '-Me	1.17	d	4.9	C ₅ '	69.2
OH	3.26	br	-	C ₅ '-Me	21.1
	3.97	br	-	C ₃ -COCH ₂ NH-	43.1
-CO ₂ CH ₂ Ph	5.08	d	12.2	C ₃ -COCH ₂ NH-	170.0
	5.14	d		-CO ₂ CH ₂ Ph	67.0
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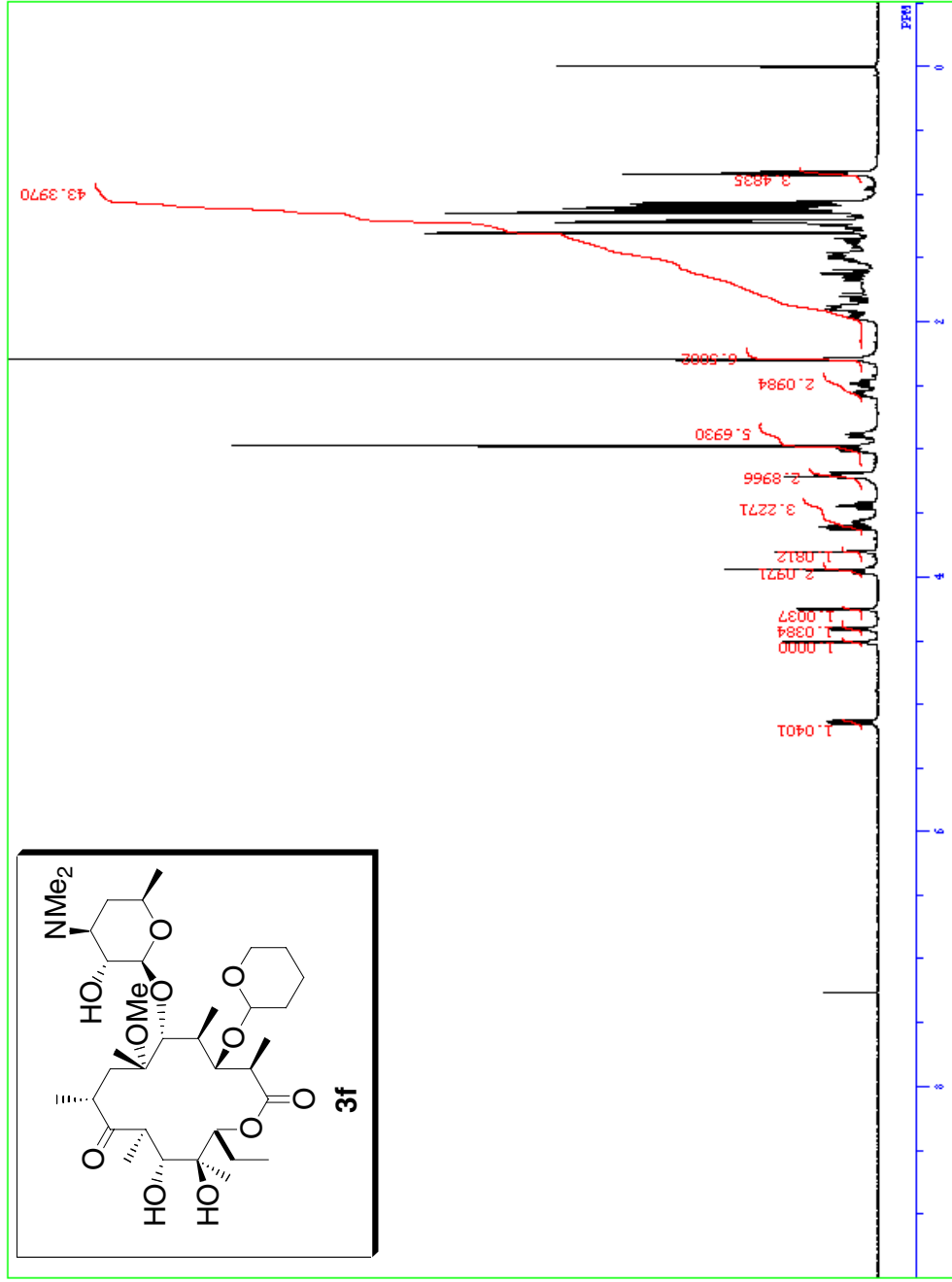


Table 8. Proton and carbon assignments of compound **3f**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.89	m	-	C ₁	175.3
2-Me	1.22	d	7.0	C ₂	44.4
C ₃ -H	3.62	d	11.3	C ₂ -Me	15.5
C ₄ -H	1.88 – 1.99	m	-	C ₃	80.9
4-Me	1.07	d	7.6	C ₄	36.7
C ₅ -H	4.23	d	4.0	C ₄ -Me	9.1
6-Me	1.31	s	-	C ₅	79.2
6-OMe	2.98	s	-	C ₆	78.0
C ₇ -H	1.61	dd	14.6, 1.5	C ₆ -Me	19.5
	1.81	dd	14.6, 12.2	C ₆ -OMe	50.0
C ₈ -H	2.57	m	-	C ₇	38.8
8-Me	1.10	d	7.3	C ₈	45.5
C ₁₀ -H	3.01	m	-	C ₈ -Me	18.0
10-Me	1.12	d	7.0	C ₉	221.1
C ₁₁ -H	3.81	d	1.2	C ₁₀	37.3
12-Me	1.15	s	-	C ₁₀ -Me	12.5
C ₁₃ -H	5.14	dd	11.3, 2.4	C ₁₁	69.4
C ₁₄ -H	1.35 – 1.56	m	-	C ₁₂	74.3
	1.88 – 1.99	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.84	t	7.3	C ₁₃	76.6
C ₁ '-H	4.52	d	7.6	C ₁₄	22.8
C ₂ '-H	3.20	dd	10.6, 7.6	C ₁₅	10.5
C ₃ '-H	2.49	ddd	12.2, 10.4, 4.3	C ₁ '	101.9
C ₃ '-NMe ₂	2.30	s	-	C ₂ '	70.8
C ₄ '-H	1.17-1.27	m	-	C ₃ '	66.0
	1.66	m	-	C ₃ '-NMe ₂	40.4
C ₅ '	3.58	m	-	C ₄ '	29.0
C ₅ '-Me	1.22	d	6.4	C ₅ '	68.7
THP(C ₁ -H)	4.41	dd	9.4, 1.2	C ₅ '-Me	21.2
THP(C ₂ -H)	1.88 – 1.99	m	-	THP(C ₁)	102.6
THP(C ₃ -H)	1.35 - 1.56	m	-	THP(C ₂)	31.4
	1.88 – 1.99	m	-	THP(C ₃)	21.2
THP(C ₄ -H)	1.35 – 1.56	m	-	THP(C ₄)	25.4
THP(C ₅ -H)	3.45	ddd	11.6, 11.6, 2.4	THP(C ₅)	66.3
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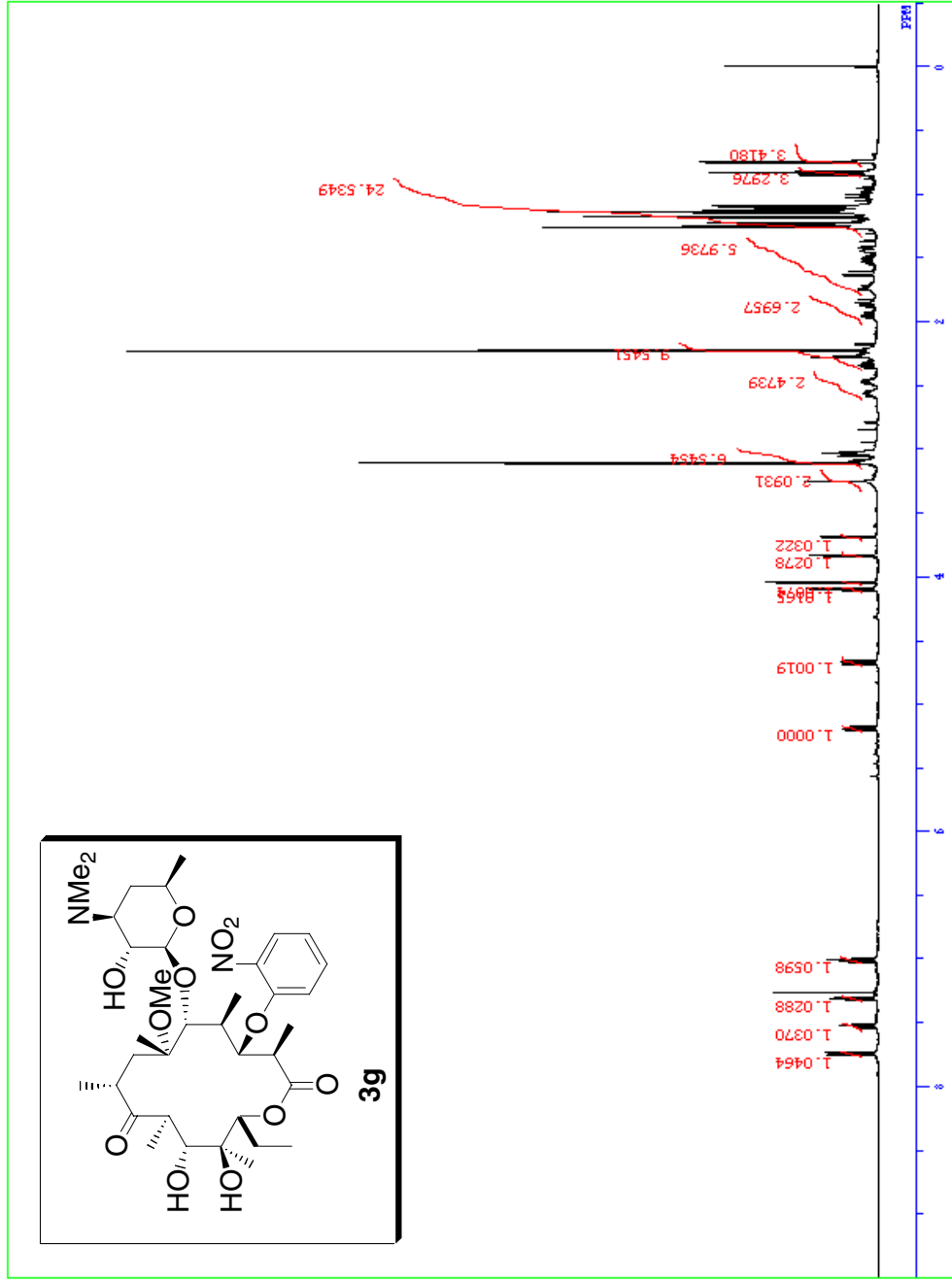


Table 9. Proton and carbon assignments of compound **3g**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	3.08 – 3.11	m	-	C ₁	174.4
2-Me	1.10	d	7.3	C ₂	44.0
C ₃ -H	4.68	d	10.4	C ₂ -Me	15.4
C ₄ -H	2.25 – 2.28	m	-	C ₃	82.5
4-Me	1.24	d	7.3	C ₄	37.0
C ₅ -H	3.69	d	3.7	C ₄ -Me	8.3
6-Me	1.26	s	-	C ₅	78.6
6-OMe	3.11	s	-	C ₆	78.2
C ₇ -H	1.62	dd	14.7, 1.2	C ₆ -Me	19.3
	1.85	dd	14.7, 12.2	C ₆ -OMe	50.2
C ₈ -H	2.57	m	-	C ₇	38.6
8-Me	1.13	d	7.3	C ₈	45.5
C ₁₀ -H	3.02 – 3.05	m	-	C ₈ -Me	17.8
10-Me	1.15	d	6.7	C ₉	220.9
C ₁₁ -H	3.84	d	1.8	C ₁₀	37.3
12-Me	1.18	s	-	C ₁₀ -Me	12.5
C ₁₃ -H	5.19	dd	11.0, 2.4	C ₁₁	69.6
C ₁₄ -H	1.52	m	-	C ₁₂	74.2
	1.96	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.84	t	7.3	C ₁₃	77.3
C ₁ '-H	4.10	d	7.3	C ₁₄	21.1
C ₂ '-H	3.02 – 3.05	m	-	C ₁₅	10.4
C ₃ '-H	2.34	m	-	C ₁ '	101.3
C ₃ '-NMe ₂	2.23	s	-	C ₂ '	70.4
C ₄ '-H	1.02	m	12.8, 1.8, 1.8,	C ₃ '	65.2
	1.43	dq	1.8	C ₃ '-NMe ₂	40.2
C ₅ '	2.47	m	-	C ₄ '	28.4
C ₅ '-Me	0.75	d	6.1	C ₅ '	69.0
OH	3.26	s	-	C ₅ '-Me	20.8
	4.05	s	-	-Ph(C ₁)	152.2
-Ph(C ₃ -H)	7.75	dd	7.9, 1.8	-Ph(C ₂)	140.2
-Ph(C ₄ -H)	7.01	t	7.9	-Ph(C ₃)	125.4
-Ph(C ₅ -H)	7.53	m	-	-Ph(C ₄)	120.1
-Ph(C ₆ -H)	7.31	d	8.6	-Ph(C ₅)	133.6
				-Ph(C ₃)	113.4

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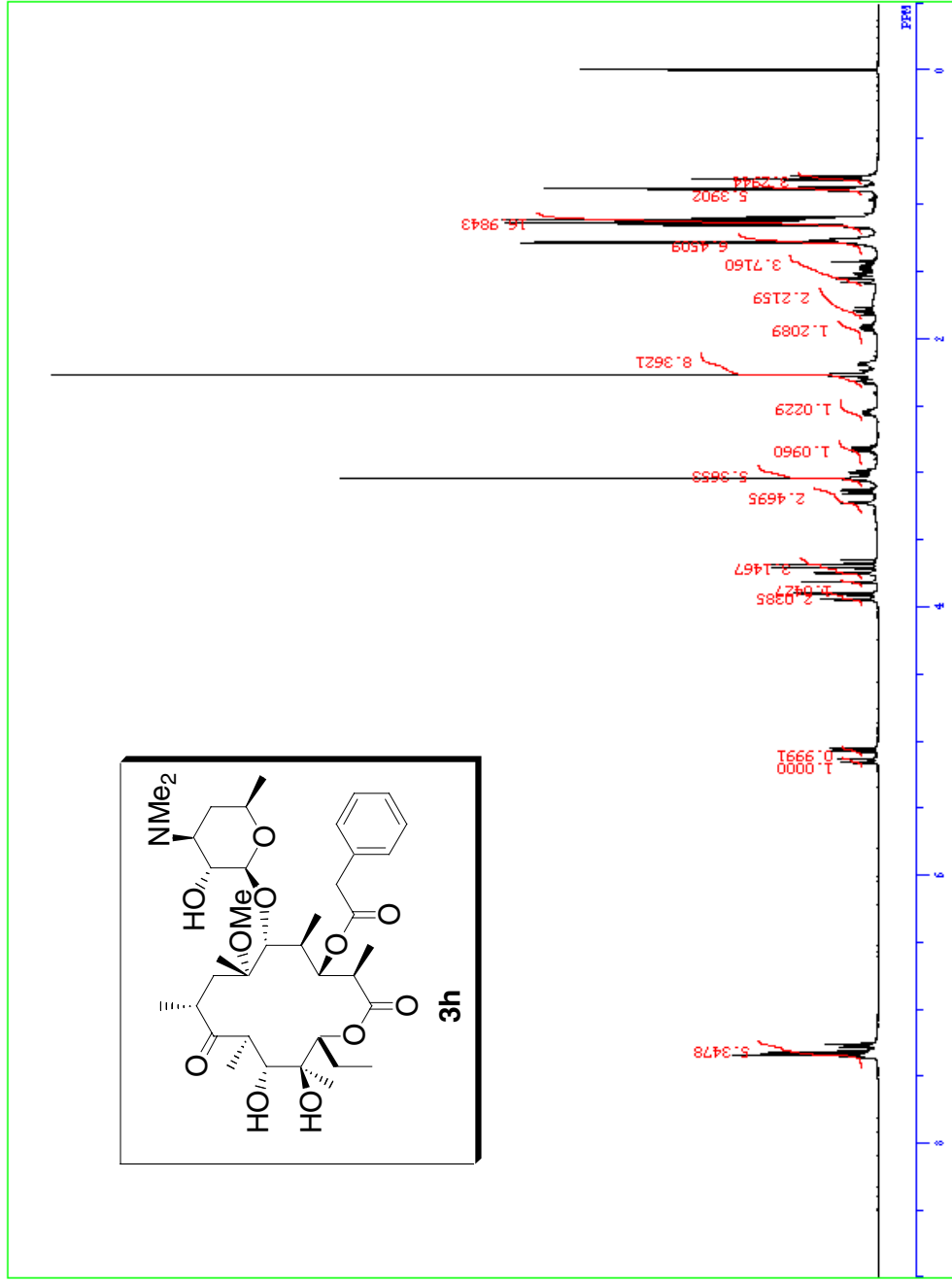


Table 10. Proton and carbon assignments of compound **3h**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.83	m	-	C ₁	173.6
2-Me	0.89	d	6.4	C ₂	43.0
C ₃ -H	5.07	d	11.3	C ₂ -Me	15.0
C ₄ -H	2.20	m	-	C ₃	78.5
4-Me	1.16	d	7.6	C ₄	36.2
C ₅ -H	3.75	d	3.7	C ₄ -Me	9.0
6-Me	1.29	s	-	C ₅	81.1
6-OMe	3.05	s	-	C ₆	78.0
C ₇ -H	1.80	dd	14.9, 11.6	C ₆ -Me	19.4
	1.57	dd	14.9, 1.5	C ₆ -OMe	50.1
C ₈ -H	2.56	m	-	C ₇	38.7
8-Me	1.11	d	7.3	C ₈	45.3
C ₁₀ -H	2.98 – 3.08	m	-	C ₈ -Me	18.0
10-Me	1.12	d	7.0	C ₉	220.6
C ₁₁ -H	3.82	d	1.5	C ₁₀	37.3
12-Me	1.15	s	-	C ₁₀ -Me	12.5
C ₁₃ -H	5.15	dd	11.3, 2.4	C ₁₁	69.4
C ₁₄ -H	1.41 – 1.55	m	-	C ₁₂	74.2
	1.92	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.81	t	7.3	C ₁₃	77.0
C ₁ '-H	3.91	d	7.3	C ₁₄	21.1
C ₂ '-H	3.14	dd	10.4, 7.3	C ₁₅	10.4
C ₃ '-H	2.32	m	-	C ₁ '	103.3
C ₃ '-NMe ₂	2.27	s	-	C ₂ '	70.5
C ₄ '-H	1.07 – 1.19	m	-	C ₃ '	65.8
	1.41 – 1.55	m	-	C ₃ '-NMe ₂	40.3
C ₅ '	2.98 – 3.08	m	-	C ₄ '	28.3
C ₅ '-Me	1.13	d	6.1	C ₅ '	69.4
OH	3.23	br s	-	C ₅ '-Me	21.0
	3.95	br s	-	C ₃ -COCH ₂ Ph	41.5
-COCH ₂ Ph	3.67	d	15.2	C ₃ -COCH ₂ Ph	171.1
	3.73	d	15.2	C ₃ -COCH ₂ Ph	127.3
-COCH ₂ Ph	7.26 – 7.36	m	-		128.6
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					133.6

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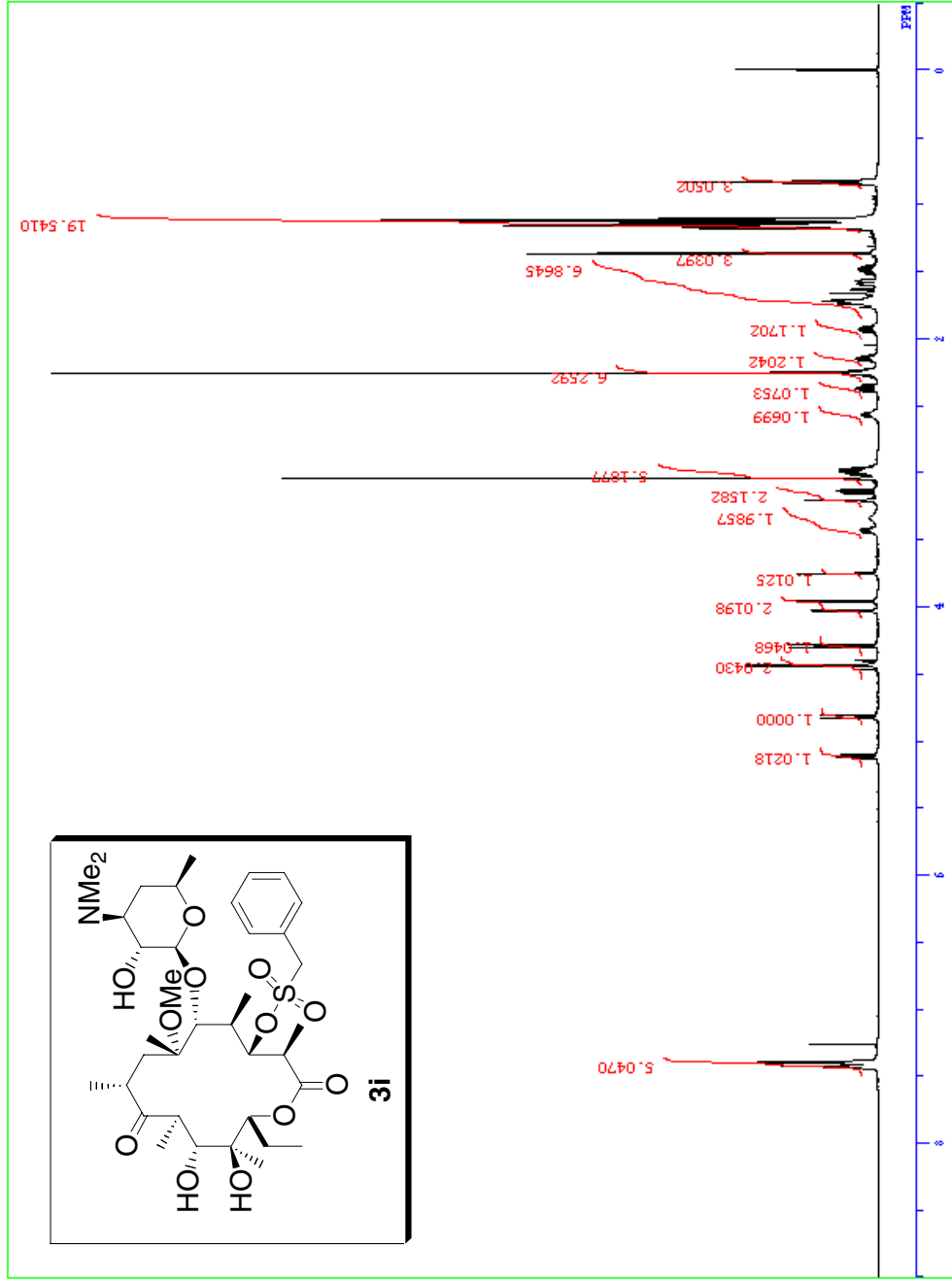


Table 11. Proton and carbon assignments of compound **3i**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.95 – 3.02	m	-	C ₁	173.3
2-Me	1.17	d	6.7	C ₂	43.6
C ₃ -H	4.82	d	11.0	C ₂ -Me	16.5
C ₄ -H	2.15	m	-	C ₃	85.9
4-Me	1.15	d	7.9	C ₄	37.9
C ₅ -H	4.03	d	4.9	C ₄ -Me	8.8
6-Me	1.37	s	-	C ₅	80.0
6-OMe	3.05	s	-	C ₆	78.3
C ₇ -H	1.65	dd	14.7, 1.8	C ₆ -Me	19.5
	1.72 – 1.77	m	-	C ₆ -OMe	50.5
C ₈ -H	2.57	m	-	C ₇	39.0
8-Me	1.12	d	7.3	C ₈	45.1
C ₁₀ -H	2.95 – 3.02	m	-	C ₈ -Me	18.0
10-Me	1.12	d	6.7	C ₉	220.7
C ₁₁ -H	3.76	d	1.2	C ₁₀	37.2
12-Me	1.12	s	-	C ₁₀ -Me	12.3
C ₁₃ -H	5.12	dd	11.6, 2.4	C ₁₁	69.2
C ₁₄ -H	1.49	m	-	C ₁₂	74.2
	1.93	m	-	C ₁₂ -Me	16.0
C ₁₅ -H	0.84	t	7.3	C ₁₃	77.4
C ₁ '-H	4.30	d	7.3	C ₁₄	21.0
C ₂ '-H	3.14	dd	9.8, 7.3	C ₁₅	10.4
C ₃ '-H	2.37	m	-	C ₁ '	102.7
C ₃ '-NMe ₂	2.26	s	-	C ₂ '	70.7
C ₄ '-H	1.14 – 1.20	m	12.8, 1.8, 1.8, 1.8	C ₃ '	65.8
	1.58	dq		C ₃ '-NMe ₂	40.2
C ₅ '	3.44	m	-	C ₄ '	28.3
C ₅ '-Me	1.16	d	5.5	C ₅ '	68.8
OH	3.21	s	-	C ₅ '-Me	21.1
	3.34	br	-	C ₃ -SO ₂ CH ₂ Ph	57.5
-SO ₂ CH ₂ Ph	4.46	d	14.0	C ₃ -SO ₂ CH ₂ Ph	128.0
	4.46	d			128.8
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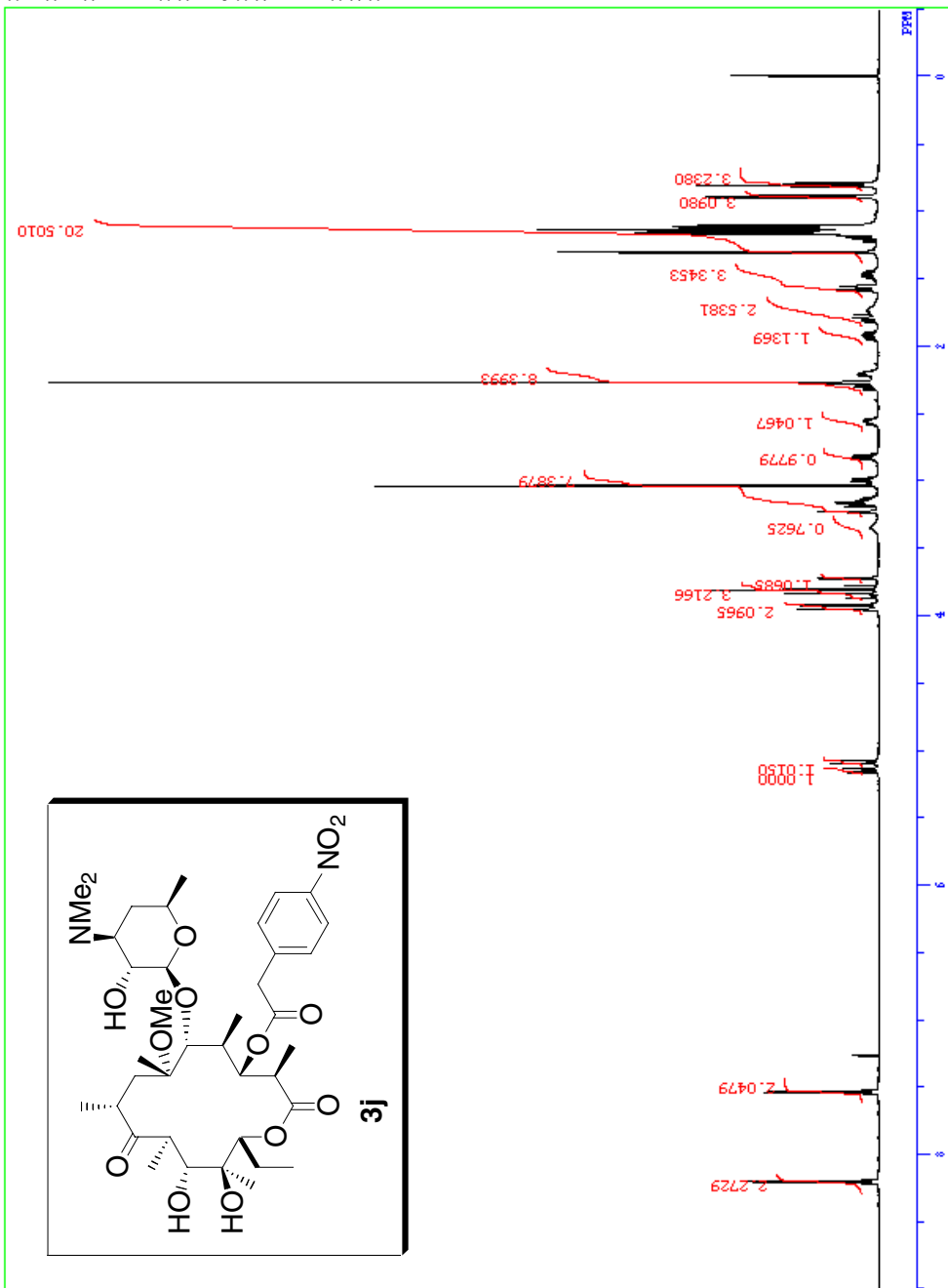


Table 12. Proton and carbon assignments of compound **3j**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.83	m	-	C ₁	173.4
2-Me	0.90	d	6.7	C ₂	42.9
C ₃ -H	5.09	d	11.0	C ₂ -Me	15.1
C ₄ -H	2.21	m	-	C ₃	79.4
4-Me	1.16	d	7.6	C ₄	36.3
C ₅ -H	3.72	d	3.7	C ₄ -Me	9.0
6-Me	1.31	s	-	C ₅	82.2
6-OMe	3.04	s	-	C ₆	78.0
C ₇ -H	1.79	dd	14.6, 11.9	C ₆ -Me	19.4
	1.56 – 1.59	m	-	C ₆ -OMe	50.1
C ₈ -H	2.56	m	-	C ₇	38.8
8-Me	1.12	d	7.0	C ₈	45.3
C ₁₀ -H	3.00	m	-	C ₈ -Me	17.9
10-Me	1.12	d	7.0	C ₉	220.6
C ₁₁ -H	3.81	s	-	C ₁₀	37.3
12-Me	1.15	s	-	C ₁₀ -Me	12.4
C ₁₃ -H	5.15	dd	11.3, 2.1	C ₁₁	69.5
C ₁₄ -H	1.47	m	-	C ₁₂	74.2
	1.93	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.81	t	7.3	C ₁₃	77.2
C ₁ '-H	3.92	d	7.3	C ₁₄	21.1
C ₂ '-H	3.17	dd	10.4, 7.3	C ₁₅	10.4
C ₃ '-H	2.30	m	-	C ₁ '	104.0
C ₃ '-NMe ₂	2.27	s	-	C ₂ '	70.4
C ₄ '-H	1.16 – 1.23	m	-	C ₃ '	66.2
	1.56 – 1.59	m	-	C ₃ '-NMe ₂	40.3
C ₅ '	3.15	m	-	C ₄ '	28.2
C ₅ '-Me	1.17	d	6.1	C ₅ '	69.7
OH	3.23 3.35 3.96	br s	-	C ₅ '-Me	21.1
		br	-	C ₃ -COCH ₂ Ph	41.1
		s	-	C ₃ -COCH ₂ Ph	169.9
-COCH ₂ Ph	3.96	d	15.5	C ₃ -COCH ₂ Ph	123.7
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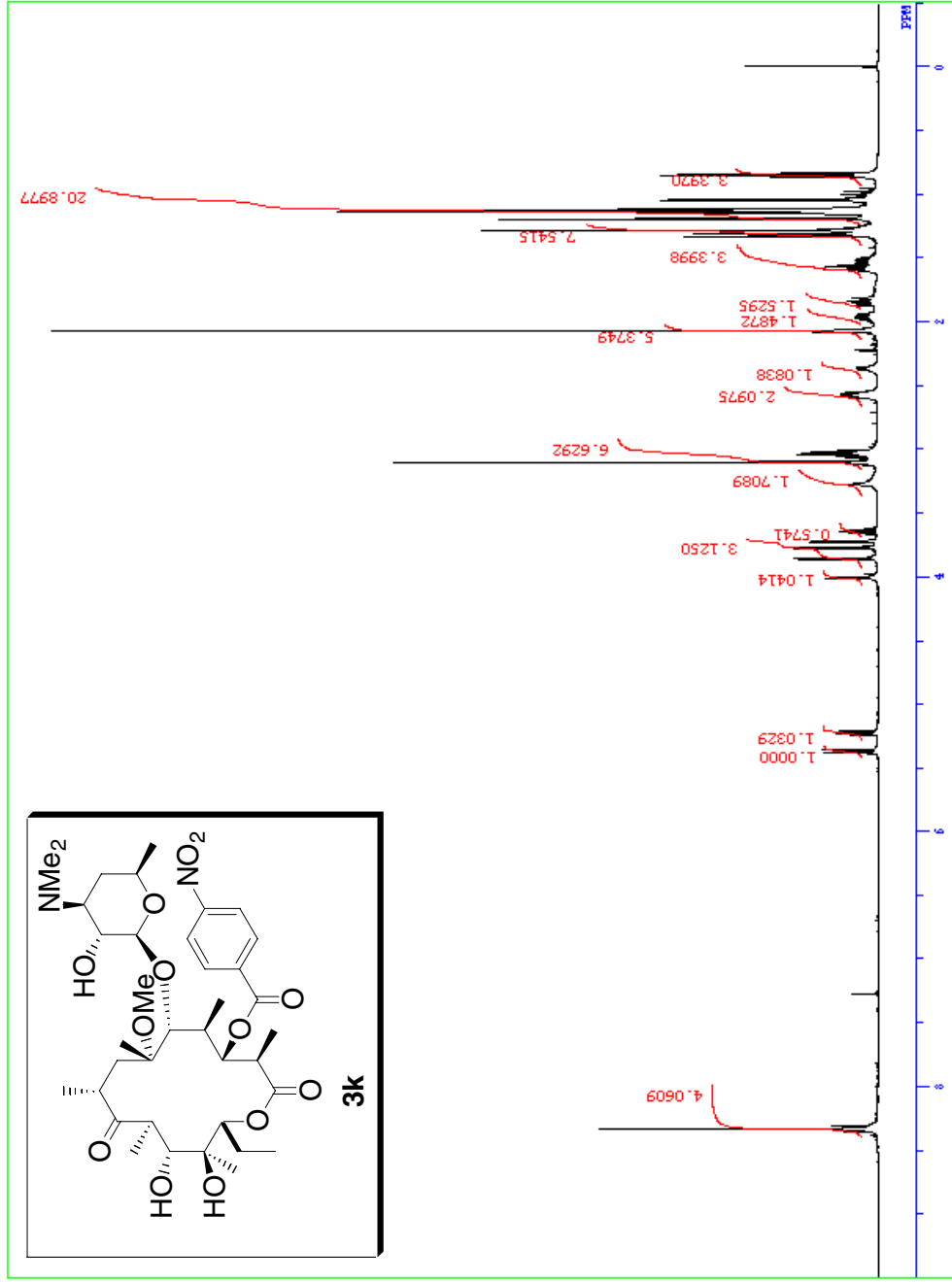


Table 13. Proton and carbon assignments of compound **3k**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	3.01 – 3.13	m	-	C ₁	173.4
2-Me	1.12 – 1.17	m	-	C ₂	42.9
C ₃ -H	5.38	d	11.0	C ₂ -Me	15.3
C ₄ -H	2.37	m	-	C ₃	80.1
4-Me	1.32	d	7.9	C ₄	36.4
C ₅ -H	3.73	d	3.1	C ₄ -Me	9.1
6-Me	1.29	s	-	C ₅	81.3
6-OMe	3.10	s	-	C ₆	77.9
C ₇ -H	1.84	m	-	C ₆ -Me	19.3
	1.49 – 1.61	m	-	C ₆ -OMe	50.1
C ₈ -H	2.54 – 2.59	m	-	C ₇	38.7
8-Me	1.12 – 1.17	m	-	C ₈	45.4
C ₁₀ -H	3.01 – 3.13	m	-	C ₈ -Me	17.9
10-Me	1.12 – 1.17	m	-	C ₉	220.6
C ₁₁ -H	3.87	d	1.2	C ₁₀	37.3
12-Me	1.20	s	-	C ₁₀ -Me	12.5
C ₁₃ -H	5.23	dd	11.0, 1.8	C ₁₁	69.5
C ₁₄ -H	1.97	m	-	C ₁₂	74.2
	1.49 – 1.61	m	-	C ₁₂ -Me	16.1
C ₁₅ -H	0.85	t	7.3	C ₁₃	77.3
C ₁ '-H	3.78	d	7.3	C ₁₄	21.2
C ₂ '-H	3.01 – 3.13	m	-	C ₁₅	10.4
C ₃ '-H	1.49 – 1.61	m	-	C ₁ '	103.0
C ₃ '-NMe ₂	2.08	s	-	C ₂ '	70.1
C ₄ '-H	1.26 – 1.33	m	-	C ₃ '	65.8
	0.96 – 1.05	m	-	C ₃ '-NMe ₂	40.1
C ₅ '	2.54 – 2.59	m	-	C ₄ '	28.0
C ₅ '-Me	1.05	d	6.1	C ₅ '	69.5
OH	3.28	br	-	C ₅ '-Me	20.9
	4.01	br	-	C ₃ -COPh	123.6
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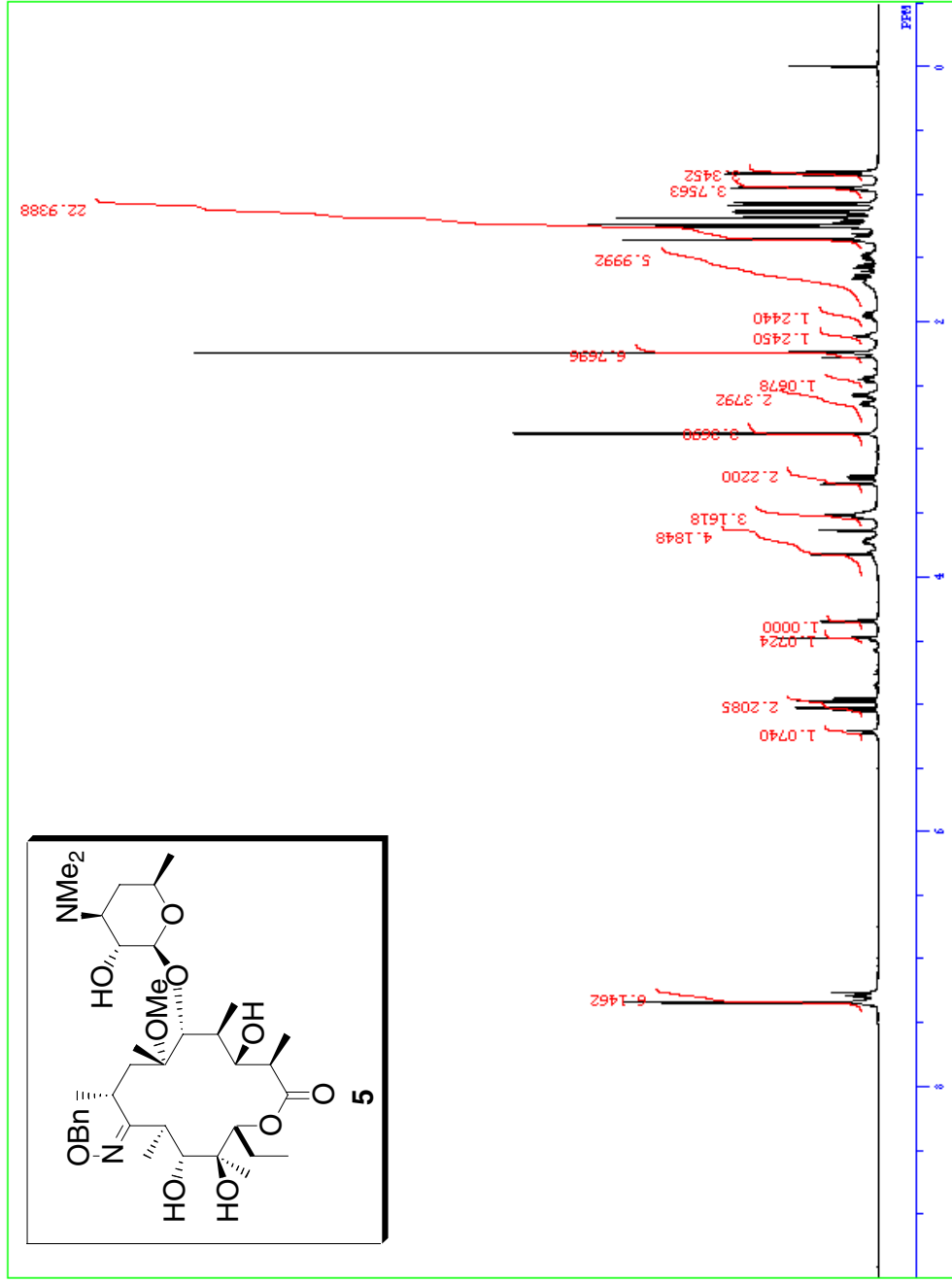


Table 14. Proton and carbon assignments of compound **5**.

500MHz ¹ H NMR data				75MHz ¹³ C NMR data	
	δ (ppm)	peak	<i>J</i> (Hz)		δ (ppm)
C ₂ -H	2.65	m	-	C ₁	175.0
2-Me	1.25	d	5.8	C ₂	44.6
C ₃ -H	3.50 – 3.55	m	-	C ₂ -Me	15.2
C ₄ -H	2.12	m	-	C ₃	79.0
4-Me	1.08	d	7.3	C ₄	36.0
C ₅ -H	3.64	d	1.2	C ₄ -Me	8.2
6-Me	1.36	s	-	C ₅	88.2
6-OMe	2.88	s	-	C ₆	78.4
C ₇ -H	1.65	m	-	C ₆ -Me	18.8
	1.19 – 1.24	m	-	C ₆ -OMe	49.5
C ₈ -H	3.73	m	-	C ₇	28.1
8-Me	0.95	d	7.0	C ₈	26.4
C ₁₀ -H	2.58	m	-	C ₈ -Me	18.3
10-Me	1.14	d	7.0	C ₉	170.2
C ₁₁ -H	3.83	d	1.5	C ₁₀	33.1
12-Me	1.19	s	-	C ₁₀ -Me	15.4
C ₁₃ -H	5.22	dd	11.0, 2.4	C ₁₁	70.6
C ₁₄ -H	1.49	m	-	C ₁₂	74.0
	1.96	m	-	C ₁₂ -Me	16.2
C ₁₅ -H	0.84	t	7.3	C ₁₃	76.9
C ₁ '-H	4.35	d	7.3	C ₁₄	21.7
C ₂ '-H	3.22	dd	10.4, 7.3	C ₁₅	10.5
C ₃ '-H	2.45	m	-	C ₁ '	106.8
C ₃ '-NMe ₂	2.24	s	-	C ₂ '	70.7
C ₄ '-H	1.58	m	-	C ₃ '	65.7
	1.32 – 1.37	m	-	C ₃ '-NMe ₂	40.3
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C ₅ '-Me	1.25	d	5.8	C ₅ '	70.3
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	5.05	d		-CH ₂ Ph	128.3
-CH ₂ Ph	7.26 – 7.37	m	-		137.9

Table 15. Proton and carbon assignments of compound **7**.500MHz ^1H NMR data

	δ (ppm)	peak	J (Hz)
C ₂ -H	2.84 – 2.95	m	-
2-Me	1.21	d	6.7
C ₃ -H	3.54 – 3.60	m	-
C ₄ -H	1.87 – 1.99	m	-
4-Me	1.03	d	7.3
C ₅ -H	4.22	d	3.7
6-Me	1.30	s	-
6-OMe	2.92	s	-
C ₇ -H	1.30 – 1.53	m	-
C ₈ -H	3.70	m	-
8-Me	0.93	d	6.7
C ₁₀ -H	2.57	m	-
10-Me	1.13	d	6.7
C ₁₁ -H	3.78	d	1.8
12-Me	1.15	s	-
C ₁₃ -H	5.19	dd	11.0, 1.8
C ₁₄ -H	1.87 – 1.99	m	-
C ₁₅ -H	0.84	t	7.3
C ₁ '-H	4.50	d	7.3
C ₂ '-H	3.20	dd	9.8, 7.3
C ₃ '-H	2.48	m	-
C ₃ '-NMe ₂	2.30	s	-
C ₄ '-H	1.65	m	-
C ₅ '	3.54 – 3.60	m	-
C ₅ '-Me	1.22	d	6.1
OH	3.15	br	-
	3.24	s	-
	4.54	s	-
-CH ₂ Ph	4.98	d	11.6
	5.04	d	
-CH ₂ Ph	7.28 – 7.35	m	-
THP(C ₁ -H)	4.41	dd	9.2, 1.2
THP(C ₂ -H)	3.44	m	-
THP(C ₃ -H)	1.30 – 1.53	m	-
THP(C ₄ -H)	1.30 – 1.53	m	-
THP(C ₅ -H)	1.30 – 1.53	m	-

75MHz ¹³C NMR data

	δ (ppm)
C ₁	175.1
C ₂	44.5
C ₂ -Me	15.4
C ₃	80.9
C ₄	36.7
C ₄ -Me	9.1
C ₅	78.9
C ₆	78.3
C ₆ -Me	196
C ₆ -OMe	50.0
C ₇	37.2
C ₈	26.4
C ₈ -Me	18.6
C ₉	170.4
C ₁₀	33.0
C ₁₀ -Me	15.3
C ₁₁	70.4
C ₁₂	74.0
C ₁₂ -Me	16.1
C ₁₃	76.9
C ₁₄	214
C ₁₅	10.5
C ₁ '	101.9
C ₂ '	70.9
C ₃ '	65.9
C ₃ '-NMe ₂	40.4
C ₄ '	29.2
C ₅ '	68.6
C ₅ '-Me	21.2
-CH ₂ Ph	75.7
-CH ₂ Ph	127.6, 128.1, 128.3, 137.9
THP(C ₁)	102.6
THP(C ₂)	66.3
THP(C ₃)	22.7
THP(C ₄)	25.4
THP(C ₅)	31.4