

# **Iodine Monochloride (ICl) as a Highly Efficient, Green Oxidant for the Oxidation of Alcohols to Corresponding Carbonyl Compounds**

**Peng Wei,<sup>1,2</sup> Datong Zhang,<sup>1</sup> Zhigang Gao,<sup>1,2</sup> Wenqing Cai,<sup>2</sup> Weiren Xu,<sup>2</sup> Lida  
Tang,<sup>2</sup> Guilong Zhao<sup>1,2</sup>**

<sup>1</sup>*School of Chemistry and Pharmaceutical Engineering, Qilu University of Technology,  
Jinan 250353, P. R. China*

<sup>2</sup>*Tianjin Key Laboratory of Molecular Design and Drug Discovery, Tianjin Institute of  
Pharmaceutical Research, Tianjin 300193, P. R. China*

Corresponding authors:

Guilong Zhao, E-mail: zhao\_guilong@126.com

Datong Zhang, E-mail: dtzhang@qlu.edu.cn

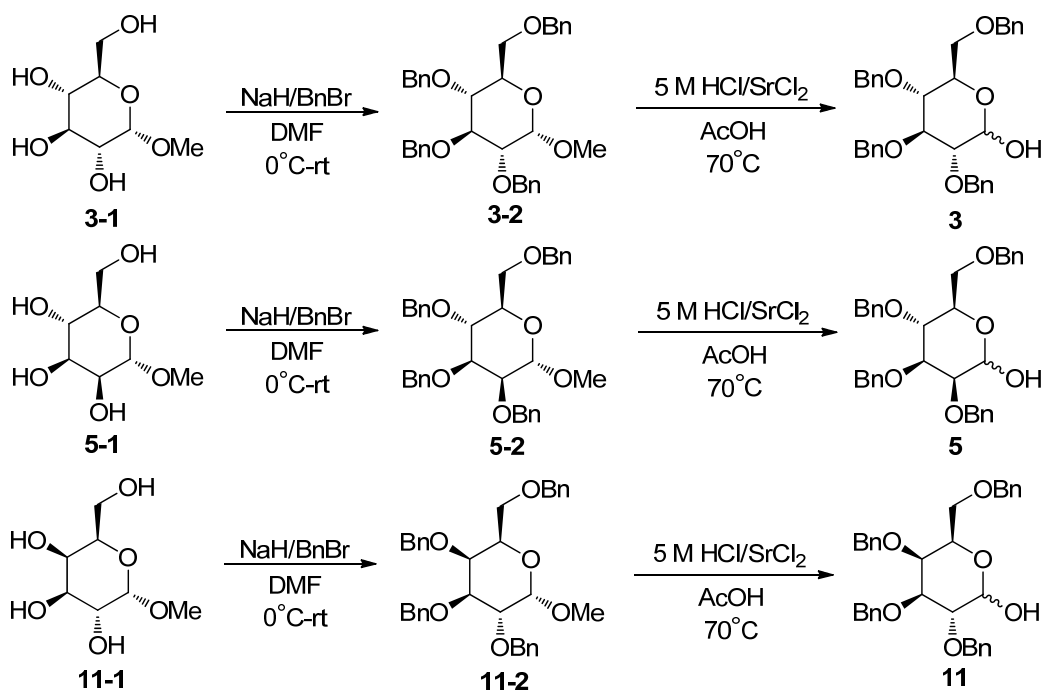
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**Supplementary Information**

## Supplementary Information

Description of synthesis and  $^1\text{H}$  NMR data/melting points of compounds **3**, **5-7**, **9-17** and the non-glycoside products listed in Table 3 except **18** and **44**.

### General Procedure for the Synthesis of Aldose Hemiacetals **3**, **5** and **11**



#### A. Synthesis of Methyl 2,3,4,6-Tetra-O-benzyl- $\alpha$ -D-glycopyranosides **3-2**, **5-2** and **11-2**

To a stirred solution of methyl  $\alpha$ -D-glycopyranosides **3-1**, **5-1** or **11-1** (1.94 g, 10 mmol) in 30 mL of dried DMF cooled with an ice-water bath was added portionwise NaH (2.40 g, 60 mmol, 60% in mineral oil). After addition, the reaction mixture was stirred at this temperature until the evolution of gas subsided (typically within 30 min). Benzyl bromide (10.26 g, 60 mmol) was added portionwise to the reaction mixture over 5 min. After addition, the reaction mixture was stirred at this temperature for 30 min and then at room temperature overnight, when TLC analysis indicated that the

reaction completed.

The reaction mixture was *carefully* poured into 300 mL of ice-water while stirring, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed with brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oily residue, which was purified by column chromatography to yield the pure methyl 2,3,4,6-tetra-*O*-benzyl- $\alpha$ -D-glycopyranosides **3-2**, **5-2** and **11-2**.

**Methyl 2,3,4,6-tetra-*O*-benzyl- $\alpha$ -D-glucopyranoside (3-2)**

Colorless oil, 4.46 g, 96%.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$ : 7.23-7.35 (m, 18H), 7.16-7.18 (m, 2H), 4.82-4.85 (m, 2H), 4.69-4.74 (m, 2H), 4.66 (d, 1H,  $J = 12.0$  Hz), 4.62 (d, 1H,  $J = 12.0$  Hz), 4.44-4.53 (m, 3H), 3.76 (t, 1H,  $J = 9.2$  Hz), 3.62 (s, 3H), 3.43-3.49 (m, 2H), 3.32 (s, 3H). The  $^1\text{H}$  NMR data are in good agreement with those reported.<sup>[1]</sup>

**Methyl 2,3,4,6-tetra-*O*-benzyl- $\alpha$ -D-mannopyranoside (5-2)**

Colorless oil, 4.55 g, 98%.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$ : 7.24-7.39 (m, 18H), 7.16-7.18 (m, 2H), 4.88 (d, 1H,  $J = 10.8$  Hz), 4.50-4.78 (m, 8H), 3.98 (t, 1H,  $J = 9.0$  Hz), 3.89 (dd, 1H,  $J = 3.0$  Hz and 9.4 Hz), 3.73-3.80 (m, 4H), 3.33 (s, 3H). The  $^1\text{H}$  NMR data are in good agreement with those reported.<sup>[2]</sup>

**Methyl 2,3,4,6-tetra-*O*-benzyl- $\alpha$ -D-galactopyranoside (11-2)**

Colorless oil, 4.51 g, 97%.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$ : 7.22-7.39 (m, 20H), 4.61-4.80 (m, 6H), 4.42-4.51 (m, 3H), 4.03 (s, 1H), 3.81-3.84 (m, 3H), 3.48-3.54 (m, 2H), 3.26 (s, 3H). The  $^1\text{H}$  NMR data are in good agreement with those reported.<sup>[2]</sup>

**B. Synthesis of 2,3,4,6-Tetra-*O*-benzyl-D-glycopyranoses **3**, **5** and **11**.**

To a stirred solution of methyl 2,3,4,6-tetra-*O*-benzyl- $\alpha$ -D-glycopyranosides **3-2**,

**5-2** or **11-2** (4.99 g, 9 mmol) in 25 mL of glacial acetic acid containing SrCl<sub>2</sub>·6H<sub>2</sub>O (0.24 g, 0.9 mmol) heated at 70°C was added 4 mL of 5 M hydrochloric acid. The stirring was continued at 70°C until reaction completed as indicated by TLC analysis (typically within 2-3 h).

The reaction mixture was poured into 300 mL of ice-water while stirring immediately after the reaction completed, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed successively with saturated sodium bicarbonate and brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oily residue, which was purified by column chromatography to yield the pure 2,3,4,6-tetra-*O*-benzyl-D-glycopyranoses **3**, **5** and **11** as anomeric mixtures.

#### **2,3,4,6-tetra-*O*-benzyl-D-glucopyranose (3)**

White solid, 4.43 g, 91%. M.p., 148-149.5°C (literature value, 150-151°C<sup>[3]</sup>). A 20-fold scale-up was performed so that it could meet the need of quantity for the experiments in this study.

#### **2,3,4,6-tetra-*O*-benzyl-D-mannopyranose (5)**

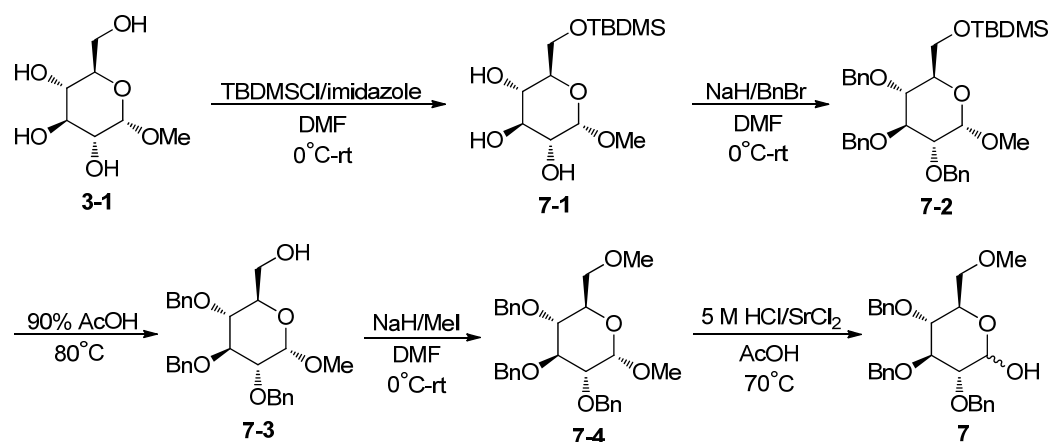
Colorless oil, 4.28 g, 88%. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ: 7.24-7.38 (m, 18H), 7.17-7.18 (m, 2H), 5.25-5.27 (m, 1H), 5.08 (d, 0.2H, *J* = 11.6 Hz), 4.85-4.90 (m, 1H), 4.69-4.76 (m, 2.5H), 4.49-4.65 (m, 5.3H), 4.03-4.07 (m, 0.8H), 3.94-3.98 (m, 0.8H), 3.79-3.92 (m, 1.6H), 3.65-3.74 (m, 1.8H). The <sup>1</sup>H NMR data are in good agreement with those reported.<sup>[4]</sup>

#### **2,3,4,6-tetra-*O*-benzyl- $\alpha$ -D-galactopyranose (11)**

Colorless oil, 4.23 g, 87%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz) δ: 7.22-7.40 (m, 20H), 6.86 (d, 0.4H, *J* = 7.2 Hz), 6.42 (d, 0.6H, *J* = 4.8 Hz), 5.17 (t, 0.60H, *J* = 4.0 Hz), 4.64-4.82 (m, 5H), 4.40-4.50 (m, 3.4H), 4.06 (t, 0.6H, *J* = 6.2 Hz), 4.01 (s, 0.6H),

3.96 (d, 0.4H,  $J = 2.8$  Hz), 3.89 (dd, 0.6H,  $J = 2.8$  Hz and 10.4 Hz), 3.76 (dd, 0.6H,  $J = 3.4$  Hz and 10.2 Hz), 3.68 (t, 0.4H,  $J = 6.2$  Hz), 3.59 (dd, 0.4H,  $J = 2.8$  Hz and 10.4 Hz), 3.51-3.54 (m, 1.4H), 3.41-3.48 (m, 1H). The  $^1\text{H}$  NMR data are in good agreement with those reported.<sup>[5]</sup>

### Procedure for the Synthesis of 2,3,4-Tri-*O*-benzyl-6-*O*-methyl-D-glucopyranose 7



#### A. Synthesis of Methyl 6-*O*-*t*-Butyldimethylsilyl- $\alpha$ -D-glucopyranoside (7-1)

To a stirred solution of **3-1** (15.53 g, 80 mmol) and imidazole (16.34 g, 240 mmol) in 150 mL of dried DMF cooled with an ice-water bath was added dropwise a solution of TBDMSCl (15.07 g, 100 mmol) in 15 mL of dried DMF and 15 mL of dried dichloromethane. After addition, the resulting mixture was stirred at room temperature overnight, when TLC analysis found **3-1** was consumed completely.

The reaction mixture was concentrated on a rotary evaporator at 60°C under highly reduced pressure (provided with an oil vacuum pump) to remove most of the DMF, and the residue was diluted with 600 mL of ethyl acetate. The solution thus obtained was washed with brine (5  $\times$ ) to remove imidazole and residual DMF, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford a yellow oil (crude **7-1**), which was co-evaporated with THF (100 mL  $\times$  3) to remove

any residual ethyl acetate. The residue thus obtained was directly used in the next step without further purification. A very small aliquot of the crude **7-1** was purified by column chromatography to give a pure sample for collection of spectroscopic and physical data.

White waxy solid. M.p., 88-89°C (literature value, 88-90°C.<sup>[6]</sup>). <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz) δ: 4.85 (d, 1H, *J* = 5.6 Hz), 4.73 (d, 1H, *J* = 4.8 Hz), 4.66 (d, 1H, *J* = 6.4 Hz), 4.50 (d, 1H, *J* = 3.6 Hz), 3.82 (dd, 1H, *J* = 1.6 Hz and 11.2 Hz), 3.60 (dd, 1H, *J* = 6.2 Hz and 11.0 Hz), 3.32-3.39 (m, 2H), 3.25 (s, 3H), 3.13-3.18 (m, 1H), 2.97-3.03 (m, 1H), 0.85 (s, 9H), 0.02 (s, 6H).

## **B. Synthesis of Methyl 2,3,4-Tri-*O*-benzyl-6-*O*-*t*-butyldimethylsilyl- $\alpha$ -D-glucopyranoside (7-2)**

To a stirred solution of the crude **7-1** (deemed to be 80 mmol) prepared above in 600 mL of dried DMF cooled with an ice-water bath was added portionwise NaH (16.00 g, 400 mmol, 60% in mineral oil) at such a rate that the evolution of gas could be controlled. After addition, the resulting slurry was stirred at this temperature until the evolution of gas subsided (typically within 1h). Benzyl bromide (68.41 g, 400 mmol) was added portionwise over 5 min. After addition, the reaction mixture was stirred at room temperature overnight, when TLC analysis found the reaction completed.

The reaction mixture was *carefully* poured into 3000 mL of ice-water while stirring, and the resulting mixture was extracted with three 500-mL portions of dichloromethane. The combined extracts were washed with brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an yellow oil (crude **7-2**), which was directly used in the next step without further purification. A very small aliquot of the crude **7-2** was purified by column chromatography to give a

pure sample for collection of spectroscopic and physical data.

White solid. M.p., 81-82°C (literature value, 77-78°C.<sup>[7]</sup>). <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz) δ: 7.24-7.33 (m, 15H), 4.81-4.84 (m, 2H), 4.82 (d, 1H, *J* = 3.2 Hz), 4.68-4.79 (m, 3H), 4.64-4.65 (m, 2H), 4.54-4.60 (m, 1H), 3.70-3.78 (m, 3H), 3.37-3.45 (m, 3H), 3.28 (s, 3H), 0.85 (s, 9H), 0.01 (s, 6H).

### C. Synthesis of Methyl 2,3,4-Tri-*O*-benzyl- $\alpha$ -D-glucopyranoside (7-3)

To a stirred solution of the crude **7-2** (deemed to be 80 mmol) prepared above in 180 mL of glacial acetic acid was added 20 mL of water at room temperature. The resulting mixture was stirred at 80°C until TLC analysis indicated that the reaction completed (typically within 3 h).

On cooling, the reaction mixture was poured into 800 mL of ice-water while stirring, and the resulting mixture was extracted with three 200-mL portions of dichloromethane. The combined extracts were washed successively with saturated sodium bicarbonate and brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford a red oil, which was purified by column chromatography to yield the pure **7-3**.

White solid, 24.90 g, 67% (overall from **3-1** to **7-3**). M.p., 68-69°C (literature value, 66.5-67°C.<sup>[8]</sup>). <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz) δ: 7.25-7.35 (m, 15H), 4.83 (d, 1H, *J* = 11.2 Hz), 4.78 (d, 1H, *J* = 3.6 Hz), 4.61-4.75 (m, 5H), 4.59 (d, 1H, *J* = 11.2 Hz), 3.73-3.78 (m, 1H), 3.62 (dd, 1H, *J* = 5.0 Hz and 11.4 Hz), 3.51-3.55 (m, 1H), 3.45 (d, 1H, *J* = 3.6 Hz), 3.41-3.43 (m, 2H), 3.28 (s, 3H).

### D. Synthesis of Methyl 2,3,4-Tri-*O*-benzyl-6-*O*-methyl- $\alpha$ -D-glucopyranoside (7-4)

To a stirred solution of **7-3** (6.04 g, 13 mmol) in 60 mL of dried DMF cooled with an ice-water bath was added portionwise NaH (0.80 g, 20 mmol, 60% in mineral oil). After addition, the resulting slurry was stirred at this temperature until the

evolution of gas subsided (typically within 30 min). Methyl iodide (2.84 g, 20 mmol) was added portionwise over 1 min. After addition, the reaction mixture was stirred at room temperature overnight, when TLC analysis found that the reaction completed.

The reaction mixture was *carefully* poured into 300 mL of ice-water while stirring, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed with brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oil (crude **7-4**), which was purified by column chromatography to give the pure product **7-4**.

Colorless oil, 5.97 g, 96%.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$ : 7.24-7.35 (m, 15H), 4.96 (d, 1H,  $J = 11.2$  Hz), 4.86 (d, 1H,  $J = 11.2$  Hz), 4.82 (d, 1H,  $J = 10.8$  Hz), 4.77 (d, 1H,  $J = 12.0$  Hz), 4.64 (d, 1H,  $J = 12.0$  Hz), 4.57-4.59 (m, 2H), 3.97 (t, 1H,  $J = 9.4$  Hz), 3.68-3.71 (m, 1H), 3.50-3.60 (m, 4H), 3.36 (s, 3H), 3.32 (s, 3H). The  $^1\text{H}$  NMR data are in good agreement with those reported.<sup>[9]</sup>

#### **E. Synthesis of 2,3,4-Tri-*O*-benzyl-6-*O*-methyl-D-glucopyranose (**7**)**

To a stirred solution of **7-4** (4.79 g, 10 mmol) in 29 mL of glacial acetic acid containing  $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$  (0.27 g, 1 mmol) heated at  $70^\circ\text{C}$  was added 5 mL of 5 M hydrochloric acid. The stirring was continued at  $70^\circ\text{C}$  until reaction completed as indicated by TLC analysis (typically within 2-3 h).

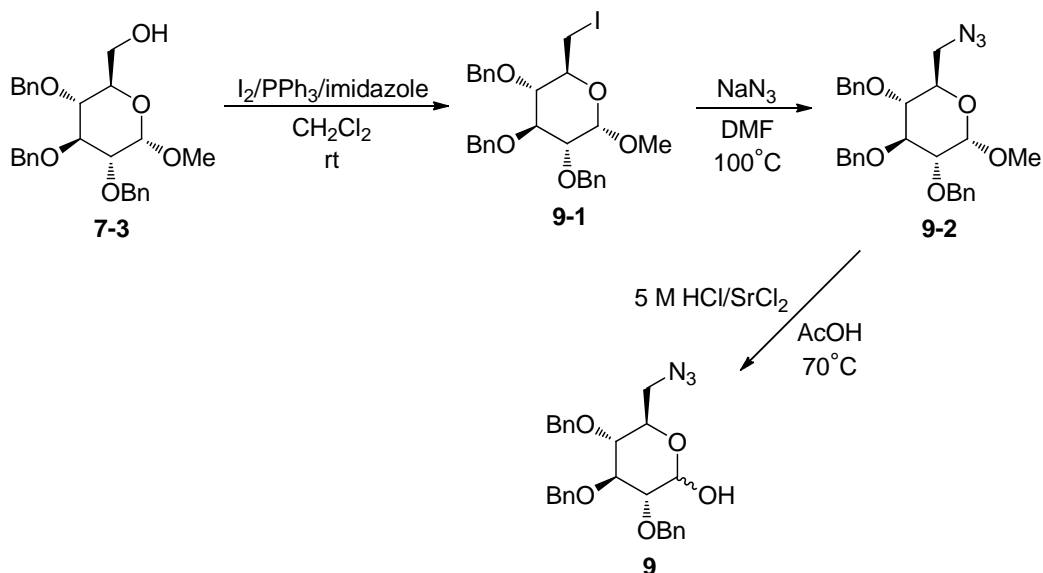
The reaction mixture was poured into 300 mL of ice-water while stirring immediately after the reaction completed, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed successively with saturated sodium bicarbonate and brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oily residue, which was purified by column chromatography to yield the pure **7** as an anomeric mixture.



White solid, 4.18 g, 90%. M.p., 120.5-122°C (literature value, 128-129°C<sup>[3]</sup>).

## Procedure for the Synthesis of

### 6-Azido-2,3,4-tri-*O*-benzyl-6-deoxy-D-glucopyranoside **9**



#### A. Synthesis of Methyl 2,3,4-Tri-*O*-benzyl-6-deoxy-6-iodo- $\alpha$ -D-glucopyranoside (**9-1**)

Iodine (18.27 g, 72 mmol) was stirred in dried dichloromethane (200 mL) on an ice-water bath, followed by portionwise addition of triphenylphosphine (19.67 g, 75 mmol). After addition, the resulting mixture was stirred for 30 min at this temperature, followed by portionwise addition of imidazole (14.71 g, 216 mmol). The mixture thus obtained was stirred at this temperature for 30 min, followed by addition of **7-3** (22.30 g, 48 mmol). The reaction mixture was stirred at room temperature until **7-3** was consumed completely (typically within 12 h).

The reaction mixture was poured into ice-water (500 mL). The mixture thus obtained was stirred and the organic phase was separated. The aqueous phase was back-extracted with dichloromethane (100 mL  $\times$  2). The combined organic phases

were washed successively with aqueous sodium thiosulfate and brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford a residue. The residue was triturated with EtOAc/petroleum ether (1/1 by v/v; 300 mL) for 30 min and filtered off, and the filtrate was evaporated on a rotary evaporator to afford the crude **9-1** as a yellow solid, which was purified by column chromatography to produce pure **9-1**.

White solid, 23.71 g, 86%. M.p., 57°C (literature value, 61-62°C.<sup>[10]</sup>). <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz) δ: 7.24-7.35 (m, 15H), 4.85 (d, 1H, *J* = 3.6 Hz), 4.84 (d, 1H, *J* = 11.2 Hz), 4.79 (d, 1H, *J* = 11.2 Hz), 4.69 (d, 1H, *J* = 11.2 Hz), 4.61-4.65 (m, 3H), 3.78 (t, 1H, *J* = 9.0 Hz), 3.46-3.52 (m, 2H), 3.37 (s, 3H), 3.25-3.34 (m, 4H).

#### **B. Synthesis of Methyl 6-Azido-2,3,4-tri-*O*-benzyl-6-deoxy- $\alpha$ -D-glucopyranoside (**9-2**)**

A suspension of **9-1** (11.49 g, 20 mmol) and sodium azide (6.50 g, 100 mmol) in 70 mL of dried DMF was stirred at 100°C under nitrogen atmosphere until **9-1** was consumed completely as indicated by TLC analysis (typically within 10 h).

On cooling, the reaction mixture was poured into 500 mL of ice-water while stirring, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed with brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford a deep yellow oil, which was purified by column chromatography to yield the pure **9-2**.

White waxy solid, 8.71 g, 89%. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ: 7.23-7.36 (m, 15H), 4.98 (d, 1H, *J* = 10.8 Hz), 4.89 (d, 1H, *J* = 10.8 Hz), 4.80 (d, 1H, *J* = 10.8 Hz), 4.78 (d, 1H, *J* = 12.0 Hz), 4.65 (d, 1H, *J* = 12.0 Hz), 4.60 (d, 1H, *J* = 3.6 Hz), 4.56 (d, 1H, *J* = 10.8 Hz), 3.97 (t, 1H, *J* = 9.4 Hz), 3.75-3.79 (m, 1H), 3.52 (dd, 1H, *J* = 3.4 Hz and 9.8 Hz), 3.43 (dd, 1H, *J* = 2.8 Hz and 12.4 Hz), 3.39 (s, 3H), 3.31 (dd, 1H, *J* = 5.8

Hz and 13.0 Hz). The  $^1\text{H}$  NMR data are in good agreement with those reported.<sup>[11]</sup>

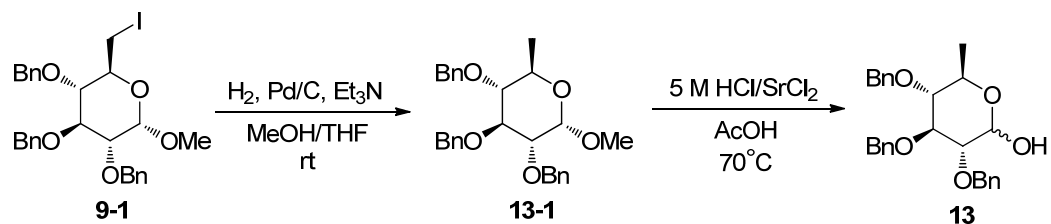
### C. Synthesis of 6-Azido-2,3,4-tri-*O*-benzyl-6-deoxy-D-glucopyranoside (9)

To a stirred solution of **9-2** (4.90 g, 10 mmol) in 29 mL of glacial acetic acid containing  $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$  (0.27 g, 1 mmol) heated at  $70^\circ\text{C}$  was added 5 mL of 5 M hydrochloric acid. The stirring was continued at  $70^\circ\text{C}$  until reaction completed as indicated by TLC analysis (typically within 2-3 h).

The reaction mixture was poured into 300 mL of ice-water while stirring immediately after the reaction completed, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed successively with saturated sodium bicarbonate and brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oily residue, which was purified by column chromatography to yield the pure **9** as an anomeric mixture.

White waxy solid, 4.09 g, 86%.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$ : 7.23-7.38 (m, 15H), 5.21 (d, 1H,  $J = 3.6$  Hz), 4.56-4.97 (m, 6.3H), 4.02-4.06 (m, 0.7H), 3.96 (t, 0.7H,  $J = 9.2$  Hz), 3.75 (t, 0.3H,  $J = 9.2$  Hz), 3.65 (t, 0.3H,  $J = 8.8$  Hz), 3.57 (dd, 0.7H,  $J = 3.6$  Hz and 9.2 Hz), 3.33-3.54 (m, 3.3H), 2.92 (bs, 1H). IR (thin film),  $\nu$  3365 (s), 3087 (w), 3062 (w), 3030 (w), 2103 (s), 1497 (w), 1449 (m). HR-MS, calcd for  $\text{C}_{27}\text{H}_{33}\text{N}_4\text{O}_5$  ( $[\text{M}+\text{NH}_4]^+$ ) 493.2451, found 493.2442; calcd for  $\text{C}_{27}\text{H}_{29}\text{N}_3\text{NaO}_5$  ( $[\text{M}+\text{Na}]^+$ ) 498.2005, found 498.1995.

### Procedure for the Synthesis of 2,3,4-Tri-*O*-benzyl-6-deoxy-D-glucopyranose **13**



### A. Synthesis of Methyl 2,3,4-Tri-*O*-benzyl-6-deoxy- $\alpha$ -D-glucopyranoside (**13-1**)

A 250-mL round-bottomed flask was charged with **9-1** (11.49 g, 20 mmol), triethylamine (6.07 g, 60 mmol), 10% Pd/C (0.60 g), 100 mL of methanol and 50 mL of THF, and the resulting mixture was subjected to hydrogenolysis with a hydrogen balloon at room temperature until **9-1** was consumed completely as indicated by TLC (typical within 10 h).

The reaction mixture was filtered off and the filtrate was concentrated on a rotary evaporator to half its original volume and poured into 300 mL of ice-water. The mixture thus obtained was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed successively with 100 mL of 2% diluted hydrochloric acid and saturated brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford the crude product **13-1** as a pale yellow oil, which was purified by column chromatography to yield the pure product **13-1**.

Colorless oil, 7.89 g, 88%.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$ : 7.26-7.34 (m, 15H), 4.83 (d, 1H,  $J = 11.2$  Hz), 4.76 (d, 1H,  $J = 11.2$  Hz), 4.75 (d, 1H,  $J = 3.6$  Hz), 4.69 (d, 1H,  $J = 11.2$  Hz), 4.63-4.67 (m, 2H), 4.59 (d, 1H,  $J = 11.2$  Hz), 3.71 (t, 1H,  $J = 9.2$  Hz), 3.53-3.57 (m, 1H), 3.47 (dd, 1H,  $J = 3.2$  Hz and 9.6 Hz), 3.28 (s, 3H), 3.10 (t, 1H,  $J = 9.2$  Hz), 1.16 (d, 3H,  $J = 6.4$  Hz). The  $^1\text{H}$  NMR data are in good agreement with those reported.<sup>[12]</sup>

### B. Synthesis of 2,3,4-Tri-*O*-benzyl-6-deoxy-D-glucopyranose (**13**)

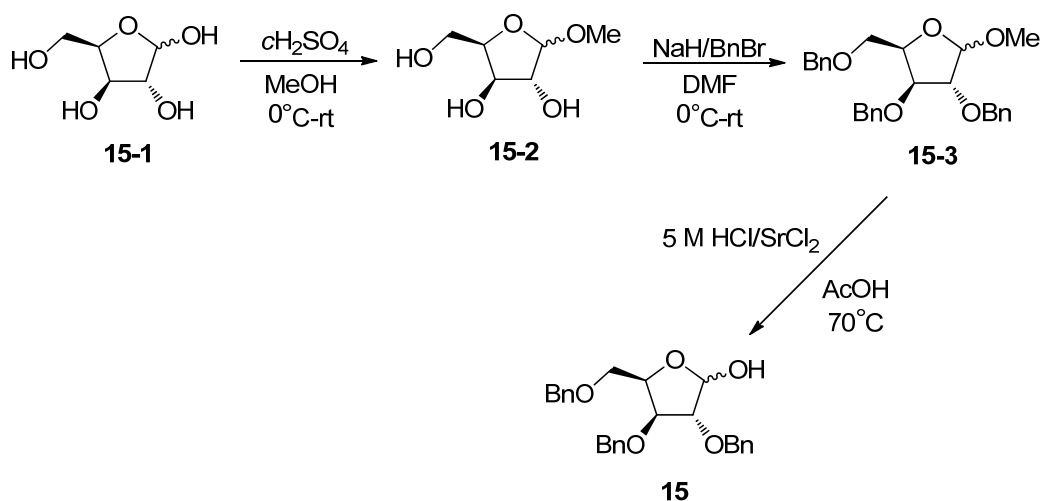
To a stirred solution of **13-1** (4.49 g, 10 mmol) in 27 mL of glacial acetic acid containing  $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$  (0.27 g, 1 mmol) heated at 70°C was added 5 mL of 5 M hydrochloric acid. The stirring was continued at 70°C until reaction completed as indicated by TLC analysis (typically within 2-3 h).

The reaction mixture was poured into 300 mL of ice-water while stirring

immediately after the reaction completed, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed successively with saturated sodium bicarbonate and brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oily residue, which was purified by column chromatography to yield the pure **13** as an anomeric mixture.

White solid, 3.78 g, 87%. M.p., 92-94°C (literature value, 92-94°C.<sup>[12]</sup>).

### Procedure for the Synthesis of 2,3,5-Tri-*O*-benzyl-D-xylofuranose **15**



To a stirred suspension of **15-1** (3.00 g, 20 mmol) in 30 mL of methanol cooled with an ice-water bath was added dropwise 3 mL of concentrated sulfuric acid, and the resulting mixture was stirred at room temperature until **15-1** was consumed completely as indicated by TLC analysis (typically within 12 h).

The reaction mixture was diluted with 70 mL of methanol and cooled with an ice-water bath. Sodium carbonate powder was added portionwise until pH > 7. The slurry thus obtained was filtered with suction and the filtrate was evaporated on a rotary evaporator to afford a residue, which was co-evaporated with THF (50 mL × 3) to remove any residual methanol to give the crude **15-1** as a yellow oil, which was

used directly in the next step without further purification.

To a stirred solution of the crude **15-1** prepared above (deemed to be 20 mmol) in 100 mL of dried DMF cooled with an ice-water bath was added portionwise NaH (4.00 g, 100 mmol, 60%). After addition, the mixture was stirred until the evolution of gas subsided (typically within 30 min), followed by portionwise addition of benzyl bromide (17.10 g, 100 mmol) over 5 min. After addition, the reaction mixture was stirred at room temperature until **15-2** was consumed completely as indicated by TLC analysis (typically within 5 h).

The reaction mixture was *carefully* poured into 400 mL of ice-water while stirring, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed with brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oily residue (crude **15-3**). The crude sample was purified by column chromatography to yield the pure **15-3** as an anomeric mixture, which was directly used in the next step.

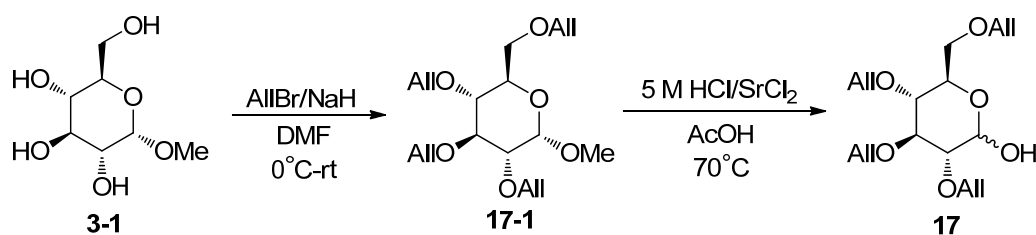
To a stirred solution of **15-3** (4.34 g, 10 mmol) in 26 mL of glacial acetic acid containing  $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$  (0.27 g, 1 mmol) heated at 70°C was added 5 mL of 5 M hydrochloric acid. The stirring was continued at 70°C until reaction completed as indicated by TLC analysis (typically within 2-3 h).

The reaction mixture was poured into 300 mL of ice-water while stirring immediately after the reaction completed, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed successively with saturated sodium bicarbonate and brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oily residue, which was purified by column chromatography to yield the pure **15** as an anomeric mixture.

Colorless oil, 3.66 g, 87%.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$ : 7.24-7.36 (m,

15H), 5.48 (d, 1H,  $J = 3.6$  Hz), 5.26 (d, 1H,  $J = 8.8$  Hz), 4.45-4.64 (m, 6.3H), 4.37-4.42 (m, 1H), 4.09-4.11 (m, 0.8H), 4.03-4.04 (m, 0.4H), 4.01 (s, 0.8H), 3.94-3.95 (m, 0.4H), 3.66-3.79 (m, 2.3H). The  $^1\text{H}$  NMR data are in agreement with those reported.<sup>[13]</sup>

### Procedure for the Synthesis of 2,3,4,6-Tetra-*O*-allyl- $\alpha$ -D-glucopyranose **17**



#### A. Synthesis of Methyl 2,3,4,6-Tetra-*O*-allyl- $\alpha$ -D-glucopyranoside (**17-1**)

To a stirred solution of **3-1** (1.94 g, 10 mmol) in 40 mL of dried DMF cooled with an ice-water bath was added portionwise NaH (2.40 g, 60 mmol, 60% in mineral oil). After addition, the reaction mixture was stirred at this temperature until the evolution of gas subsided (typically within 30 min). Allyl bromide (7.26 g, 60 mmol) was added portionwise to the reaction mixture over 5 min. After addition, the reaction mixture was stirred at this temperature for 30 min and then at room temperature overnight, when TLC analysis indicated that the reaction completed.

The reaction mixture was *carefully* poured into 300 mL of ice-water while stirring, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed with brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oily residue, which was purified by column chromatography to yield the pure **17-1**.

White waxy solid, 3.37 g, 95%.  $^1\text{H}$  NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 5.82-5.97 (m, 4H), 5.08-5.25 (m, 8H), 4.72 (d, 1H,  $J = 3.6$  Hz), 3.93-4.33 (m, 8H), 3.56-3.69 (m,

4H), 3.33-3.40 (m, 5H). The  $^1\text{H}$  NMR data are in good agreement with those reported.<sup>[14]</sup>

### **B. Synthesis of 2,3,4,6-Tetra-*O*-allyl- $\alpha$ -D-glucopyranose (17)**

To a stirred solution of **17-1** (2.84 g, 8 mmol) in 17 mL of glacial acetic acid containing  $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$  (0.21 g, 0.8 mmol) heated at  $70^\circ\text{C}$  was added 5 mL of 5 M hydrochloric acid. The stirring was continued at  $70^\circ\text{C}$  until reaction completed as indicated by TLC analysis (typically within 2-3 h).

The reaction mixture was poured into 300 mL of ice-water while stirring immediately after the reaction completed, and the resulting mixture was extracted with three 100-mL portions of dichloromethane. The combined extracts were washed successively with saturated sodium bicarbonate and brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford an oily residue, which was purified by column chromatography to yield the pure **17** as an anomeric mixture.

White waxy solid, 2.18 g, 80%.  $^1\text{H}$  NMR ( $\text{DMSO-d}_6$ , 400 MHz)  $\delta$ : 6.86 (d, 0.2H,  $J = 6.8$  Hz), 6.47 (dd, 0.8H,  $J = 0.6$  Hz and 4.6 Hz), 5.81-5.95 (m, 3.5H), 5.23-5.29 (m, 3.5H), 5.20-5.22 (m, 0.5H), 5.07-5.18 (m, 4.5H), 4.43 (dd, 0.2H,  $J = 6.8$  Hz and 7.6 Hz), 3.87-4.28 (m, 8.5H), 3.71-3.75 (m, 0.8H), 3.34-3.57 (m, 3H), 3.32-3.36 (m, 0.3H), 3.25-3.28 (m, 0.2H), 3.13-3.19 (m, 1.8H), 2.93 (dd, 1H,  $J = 8.0$  Hz and 9.2 Hz). The  $^1\text{H}$  NMR data are in good agreement with those reported.<sup>[5]</sup>

### **General Procedure for the Study of Scope of ICl as Oxidant (Table 3)**

Starting alcohols specified in Table 2 and Table 3 (1.00 g, 1.0 eq) was dissolved in 10 mL of dried dichloromethane (for substrates in entries 8-20, 30 mL of dried  $\text{CH}_2\text{Cl}_2$  was used). The resulting mixture was stirred in an ice-water bath, followed by addition of  $\text{Cs}_2\text{CO}_3$  (3.0 eq). The suspension was stirred at this temperature, and ICl



(1.5 eq) dissolved in 2 mL of dichloromethane was added dropwise. After addition, the reaction mixture was stirred at room temperature until the reaction completed as indicated by TLC analysis. The TLC analysis was carried out at 0.5-h intervals.

The reaction mixture was poured into 50 mL of ice-water while stirring, and the resulting mixture was extracted with dichloromethane (50 mL  $\times$  3). The combined extracts were washed successively with aqueous sodium thiosulfate and brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford a residue, which was purified by column chromatography to yield the pure products specified in Table 2 and Table 3.

Compounds **4** and **8**, which have already been described in EXPERIMENTAL, are not included here.

**2,3,4,6-Tetra-*O*-benzyl-D-mannonolactone (6)**

White solid, 0.936 g, 94%. M.p., 83-84°C (literature value, 83°C<sup>[15]</sup>).

**6-Azido-2,3,4-tri-*O*-benzyl-6-deoxy-D-gluconolactone (10)**

Colorless oil, 0.826 g, 83%. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 7.20-7.38 (m, 15H), 4.91 (d, 1H,  $J$  = 11.2 Hz), 4.61-4.70 (m, 3H), 4.49-4.54 (m, 3H), 4.10 (d, 1H,  $J$  = 5.2 Hz), 3.90 (t, 1H,  $J$  = 5.6 Hz), 3.77 (dd, 1H,  $J$  = 6.0 Hz and 9.6 Hz), 3.63 (dd, 1H,  $J$  = 2.6 Hz and 13.4 Hz), 3.44 (dd, 1H,  $J$  = 4.2 Hz and 13.4 Hz). The <sup>1</sup>H NMR data are in good agreement with those reported.<sup>[16]</sup>

**2,3,4,6-Tetra-*O*-benzyl-D-galactonolactone (12)**

Colorless oil, 0.946 g, 95%. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 7.40-7.42 (m, 2H), 7.22-7.35 (m, 18H), 5.17 (d, 1H,  $J$  = 11.2 Hz), 4.93 (d, 1H,  $J$  = 11.2 Hz), 4.66-4.79 (m, 3H), 4.60 (d, 1H,  $J$  = 11.2 Hz), 4.43-4.51 (m, 3H), 4.33 (t, 1H,  $J$  = 6.8 Hz), 4.15 (s, 1H), 3.88 (dd, 1H,  $J$  = 2.0 Hz and 9.6 Hz), 3.63-3.73 (m, 2H). The <sup>1</sup>H NMR data are in good agreement with those reported.<sup>[15]</sup>

**2,3,4-Tri-*O*-benzyl-6-deoxy-D-gluconolactone (14)**

White solid, 0.906 g, 91%. M.p., 64-66°C (literature value, 66-67°C<sup>[12]</sup>).

**2,3,5-Tri-*O*-benzyl-D-xylonolactone (16)**

White waxy solid, 0.955 g, 96%. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ: 7.23-7.35 (m, 15H), 5.04 (d, 1H, *J* = 11.6 Hz), 4.69 (d, 1H, *J* = 11.6 Hz), 4.65 (d, 1H, *J* = 12.0 Hz), 4.50-4.59 (m, 5H), 4.36 (t, 1H, *J* = 7.2 Hz), 3.76 (dd, 1H, *J* = 2.8 Hz and 10.8 Hz), 3.70 (dd, 1H, *J* = 3.2 Hz and 10.8 Hz). The <sup>1</sup>H NMR data are in good agreement with those reported.<sup>[17]</sup>

**Benzophenone (20)**

White solid, 0.930 g, 94%. M.p., 47-49°C (literature value, 46-48°C<sup>[18]</sup>).

**9-Fluorenone (22)**

White solid, 0.821 g, 83%. M.p., 82-83°C (literature value, 82-84°C<sup>[19]</sup>).

**9-Xanthenone (24)**

White solid, 0.891 g, 90%. M.p., 174-175°C (literature value, 176-177°C<sup>[20]</sup>).

**Acetophenone (26)**

Colorless oil, 0.944 g, 96%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz) δ: 7.95 (d, 2H, *J* = 7.6 Hz), 7.63 (t, 1H, *J* = 7.4 Hz), 7.51 (d, 2H, *J* = 7.6 Hz), 2.57 (s, 3H). The <sup>1</sup>H NMR data are in good agreement with those reported.<sup>[21]</sup>

**4-Bromoacetophenone (28)**

White solid, 0.901 g, 91%. M.p., 50-51°C (literature value, 50-52°C<sup>[22]</sup>).

**4-Nitroacetophenone (30)**

White solid, 0.810 g, 82%. M.p., 79-80°C (literature value, 79-80°C<sup>[23]</sup>).

**4-Methoxyacetophenone (32)**

White solid, 0.809 g, 82%. M.p., 37-38°C (literature value, 38-39°C<sup>[24]</sup>).

**Indan-1-one (34)**

White solid, 0.965 g, 98%. M.p., 37-38°C (literature value, 39-40°C<sup>[25]</sup>).

#### **2-Tridecanone (36)**

White waxy solid, 0.901 g, 91%. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ: 2.39 (t, 2H, *J* = 7.6 Hz), 2.11 (s, 3H), 1.52-1.57 (m, 2H), 1.23 (s, 16H), 0.86 (t, 3H, *J* = 6.8 Hz). The <sup>1</sup>H NMR data are in good agreement with those reported.<sup>[26]</sup>

#### **Adamantan-2-one (38)**

White solid, 0.868 g, 88%. M.p., 253-255°C (literature value, 255-259°C<sup>[18]</sup>).

#### **Benzaldehyde (40)**

Colorless oil, 0.314 g, 32%. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz) δ: 10.01 (s, 1H), 7.89-7.92 (m, 2H), 7.71 (t, 1H, *J* = 7.4 Hz), 7.60 (t, 2H, *J* = 7.6 Hz). The <sup>1</sup>H NMR data are in good agreement with those reported.<sup>[27]</sup>

#### **4-Bromobenzaldehyde (42)**

White solid, 0.485 g, 49%. M.p., 59-60°C (literature value, 58-59°C<sup>[28]</sup>).

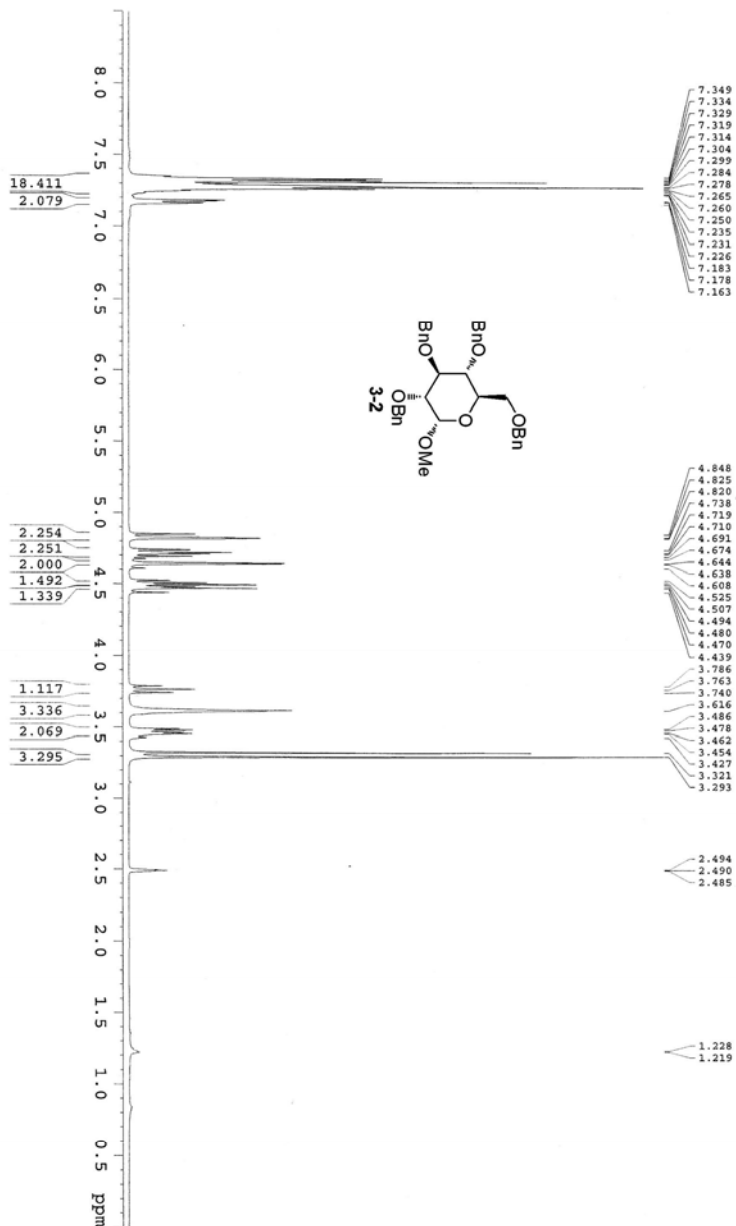
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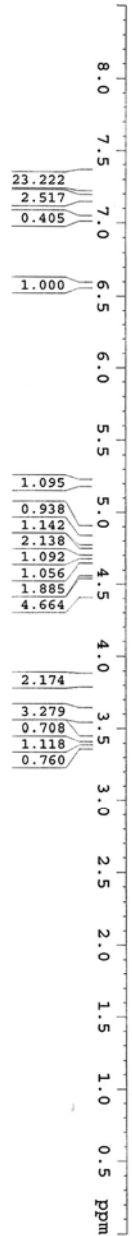
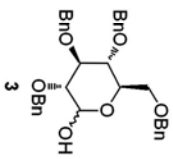
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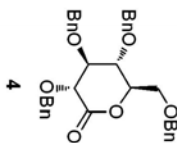
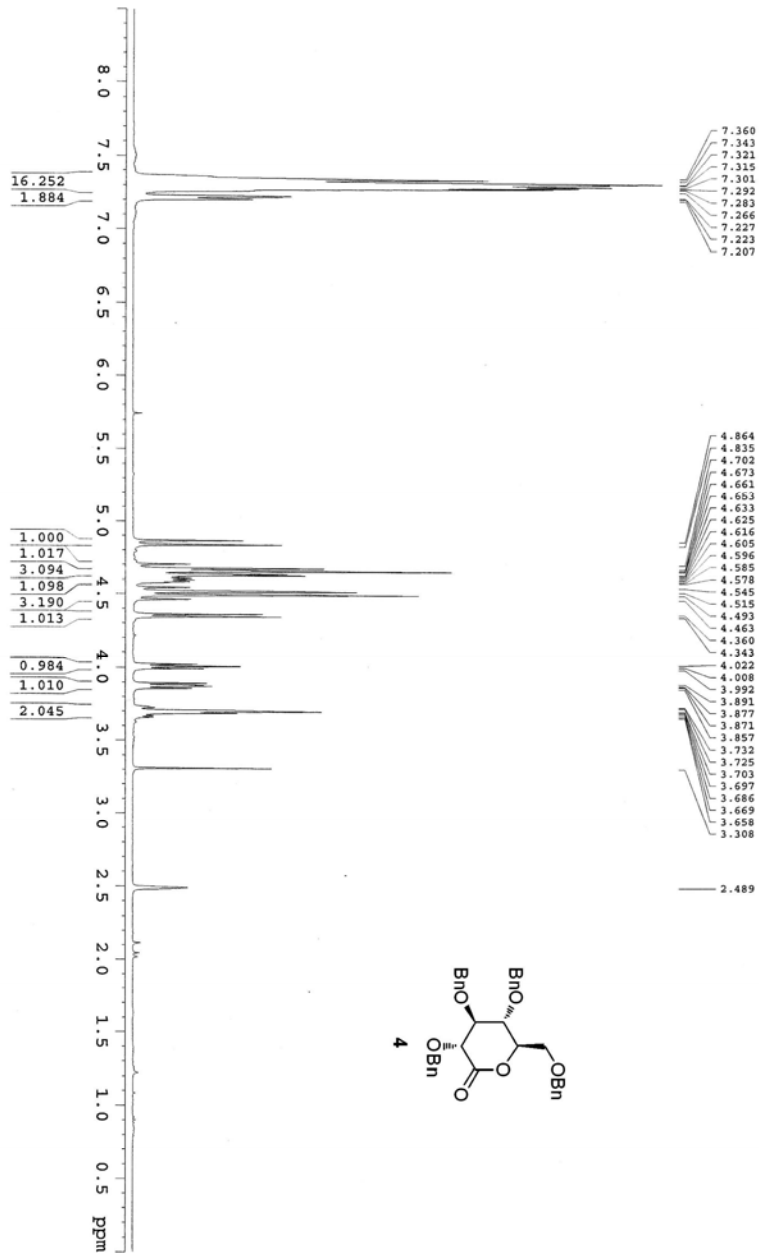
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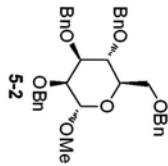
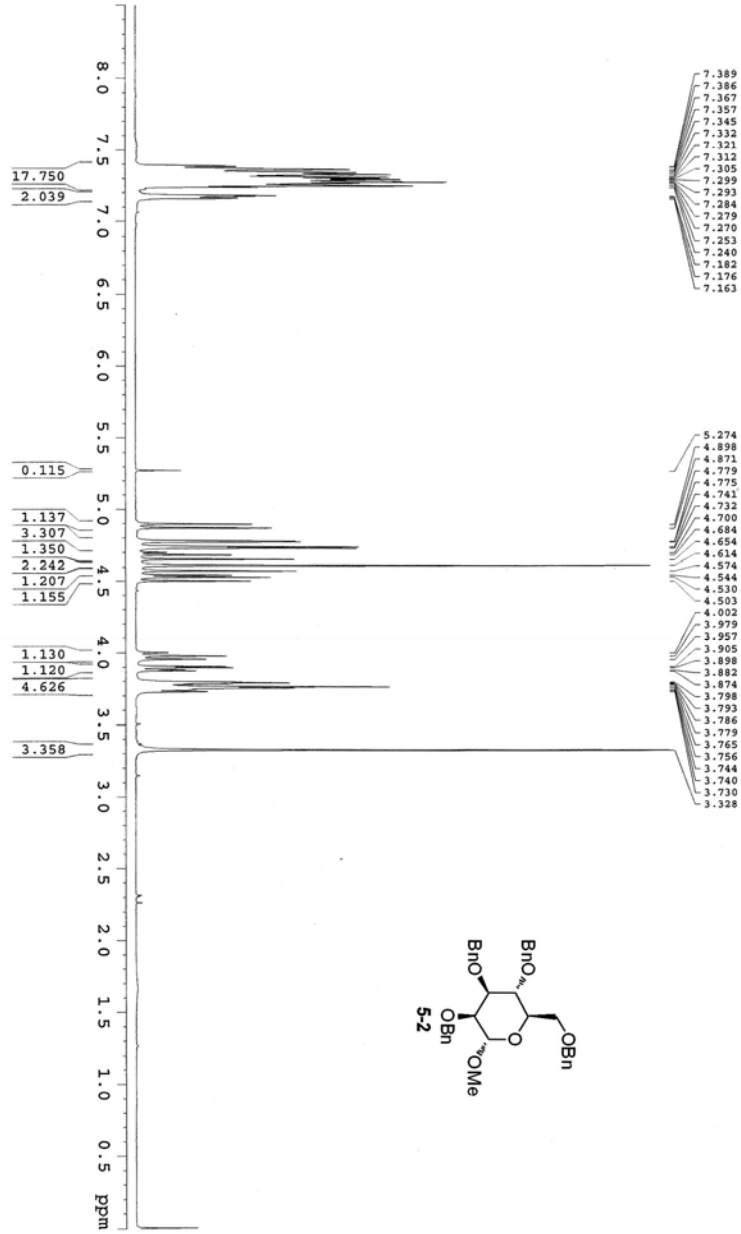
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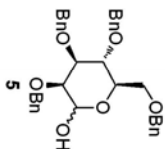
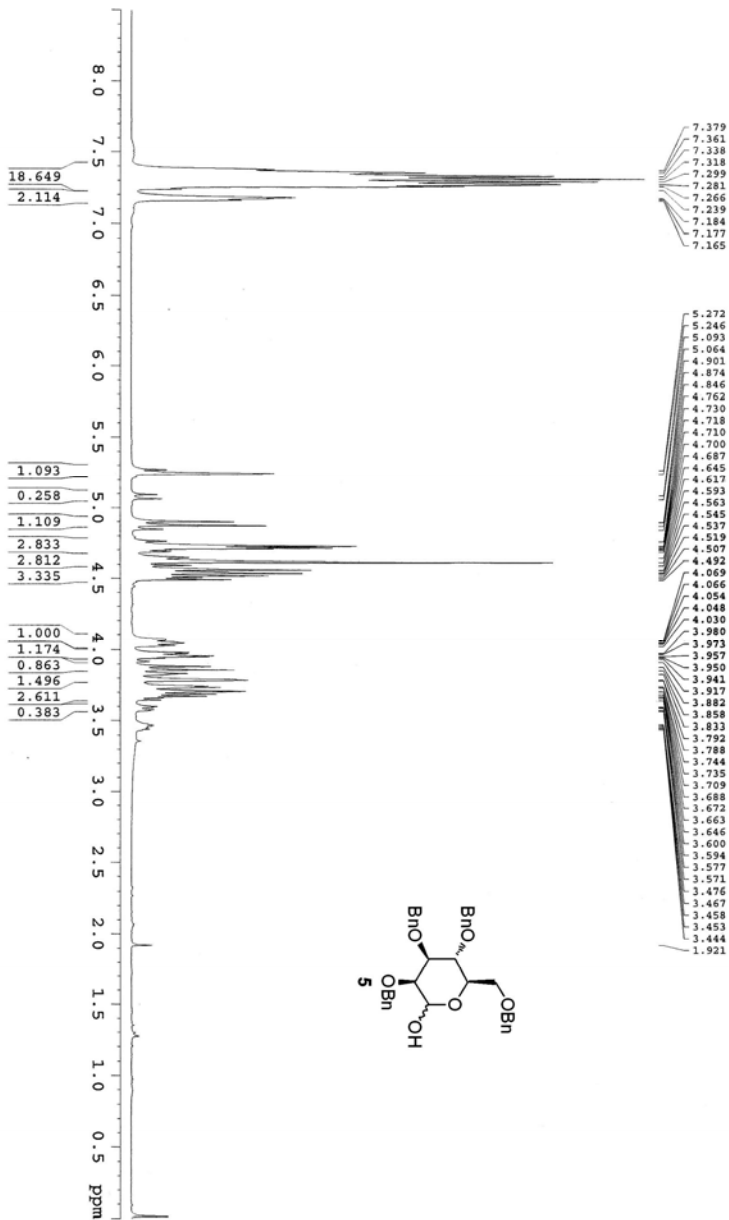
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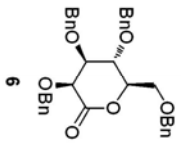
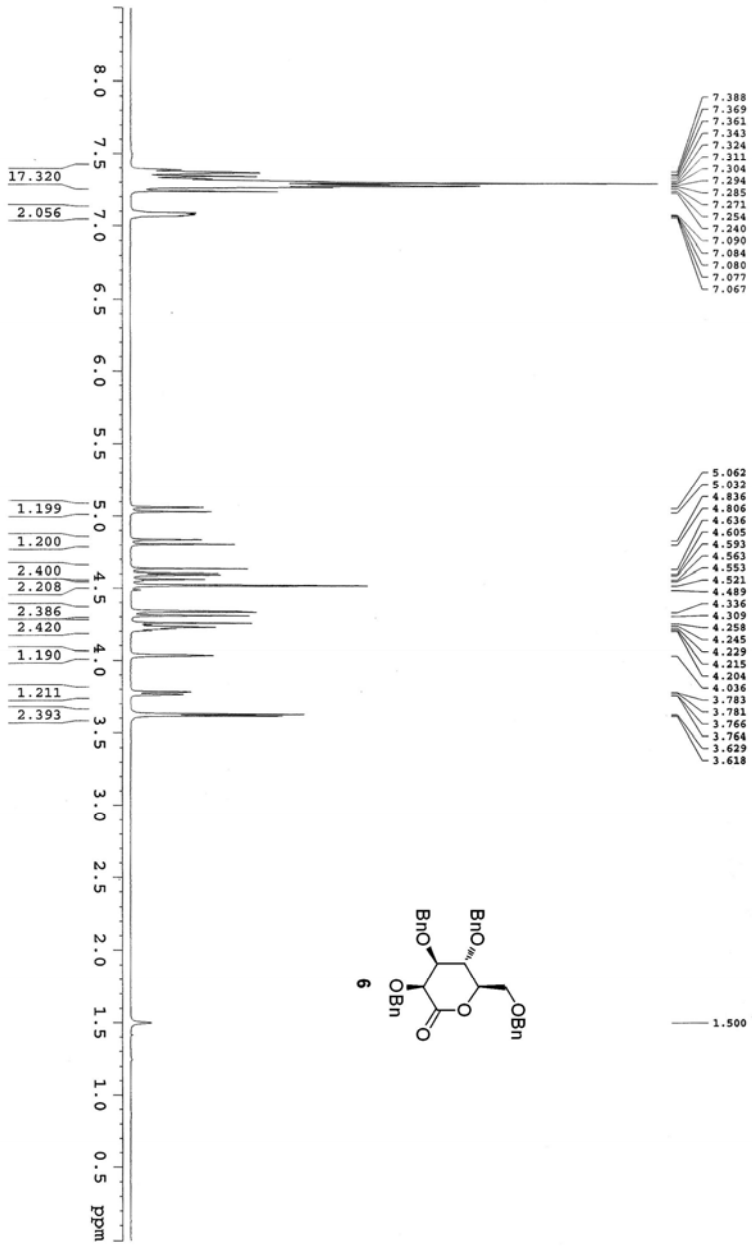


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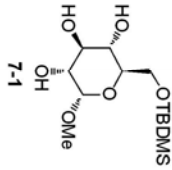
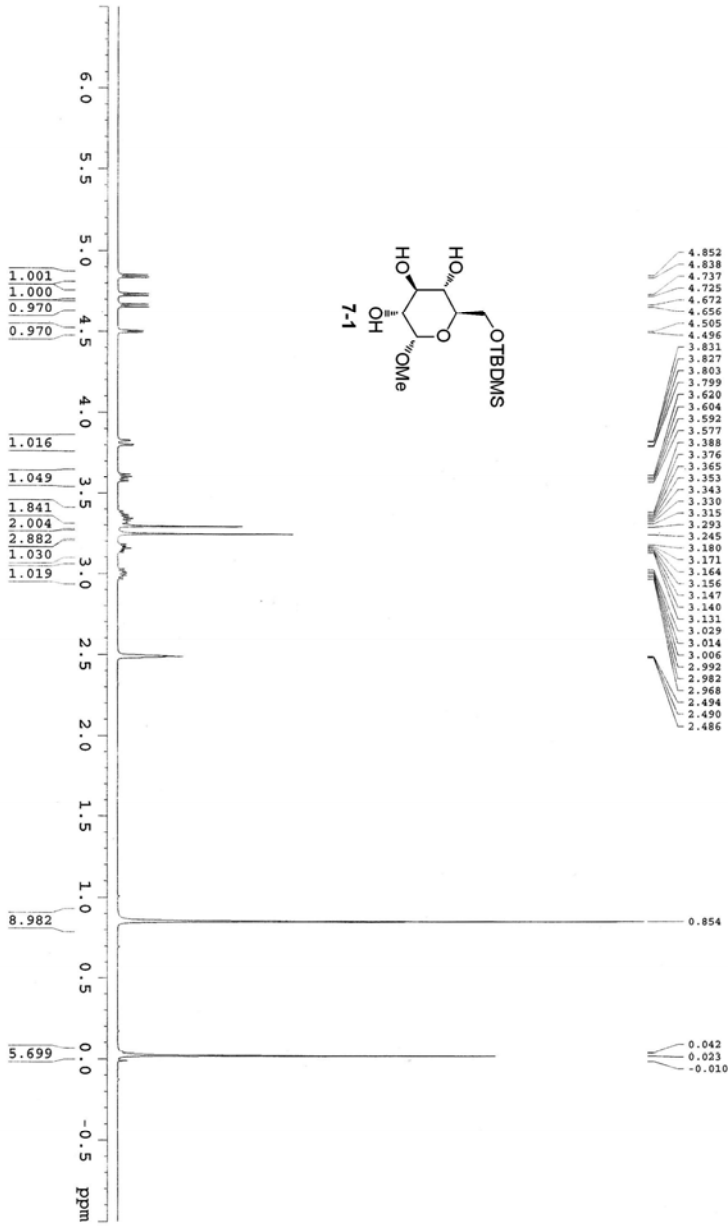
Current Data Parameters
NAME      WP_140526-OSA-4
EXPTNO   1
PROCNO    1
P2 - Acquisition Parameters
Date_     20140526
Time      11.44
INSTRUM   av400
PROBHD    5 mm DDL 1H-1
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         16
DS         4
SWH        6377.551 Hz
FIDRES     0.124627 Hz
AQ         2.526627 sec
RG         65.4
WDW         78.400 usec
SSB         380.0 X
TE         300.0 K
D1         2.00000000 sec
***** CHANNEL f1 *****
NUC1       1H
P1         11.48 usec
PL1        0.00 dB
SFO1       400.1320799 MHz
P2 - Processing parameters
SI         16384
SF         400.1300175 MHz
RG         65.4
WDW         78.400 usec
SSB         380.0 X
TE         300.0 K
D1         2.00000000 sec
PC         1.00
  
```



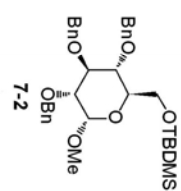
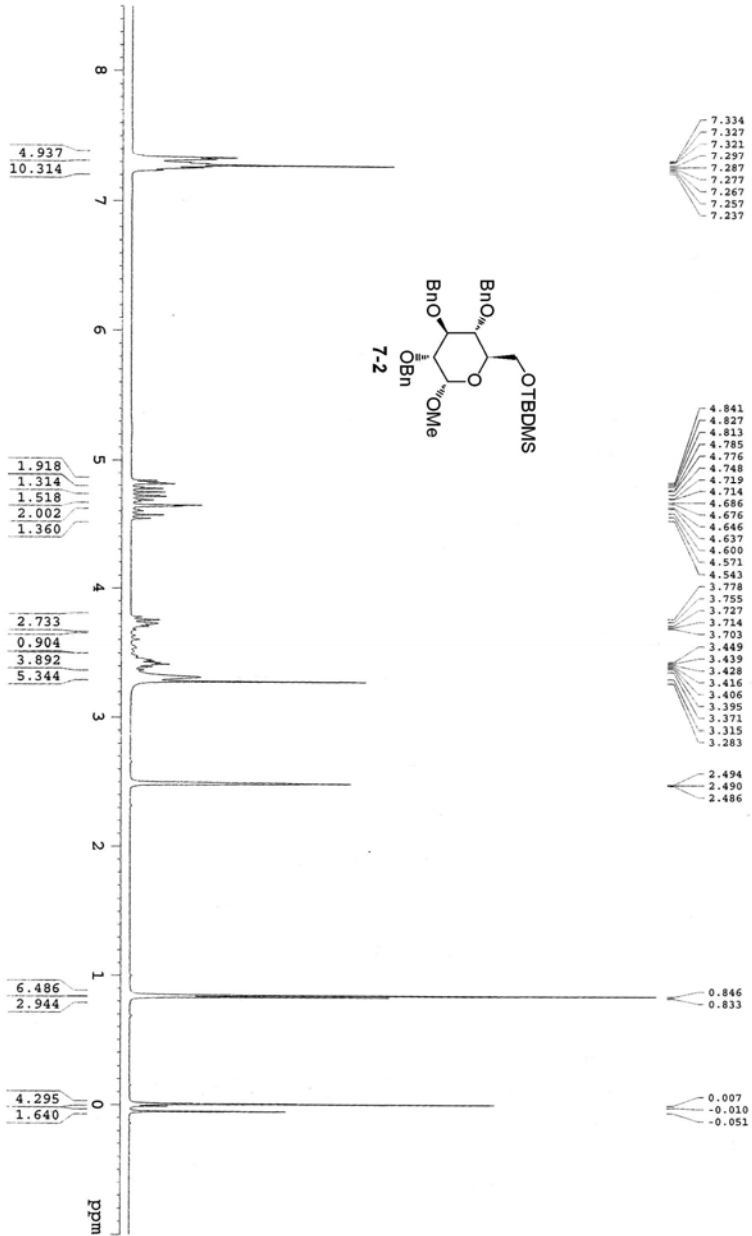
Current Data Parameters  
 EXPNO 2  
 F2 - Acquisition Parameters  
 Date\_ 20140513  
 Time 16:08  
 INSTRUM spect  
 PROBRID 5 mm DUL 13C-1  
 PULPROG zgpg30  
 SOLVENT CDCl3  
 NS 16  
 DS 4  
 SFO 114.94, 25.3 Hz  
 FIDRES 0.150777 Hz  
 AQ 1.4254597 sec  
 RG 4096  
 DW 43.500 usec  
 DE 6.00 usec  
 TE 300.2 K  
 D1 2.00000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 1H  
 P1 11.85 usec  
 PL 0.00 dB  
 SFO1 400.132607 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 400.1305177 MHz  
 ACQ 32768  
 PC 1.00



Current Data Parameters  
 NAME: 20140513-08N1  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20140513  
 Time: 16:20  
 INSTRUM: spect  
 PROBMW: 5 mm DUL 13C-1  
 PULPROG: zgpg30  
 SOLVENT: CDCl3  
 NS: 16  
 DS: 4  
 SWH: 11261.260 Hz  
 FIDRES: 0.343666 Hz  
 AQ: 1.454491 sec  
 SFO1: 400.13082 MHz  
 DM: 44.460 usec  
 DE: 6.00 usec  
 DI: 2.0000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1: 13C  
 P1: 11.85 usec  
 PL1: -2.00 dB  
 SFO1: 400.13082 MHz  
 F2 - Processing parameters  
 SI: 32768  
 SF: 400.13082 MHz  
 KW: 1024  
 SSB: 0  
 CB: 0  
 GB: 0  
 PC: 1.00



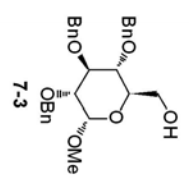
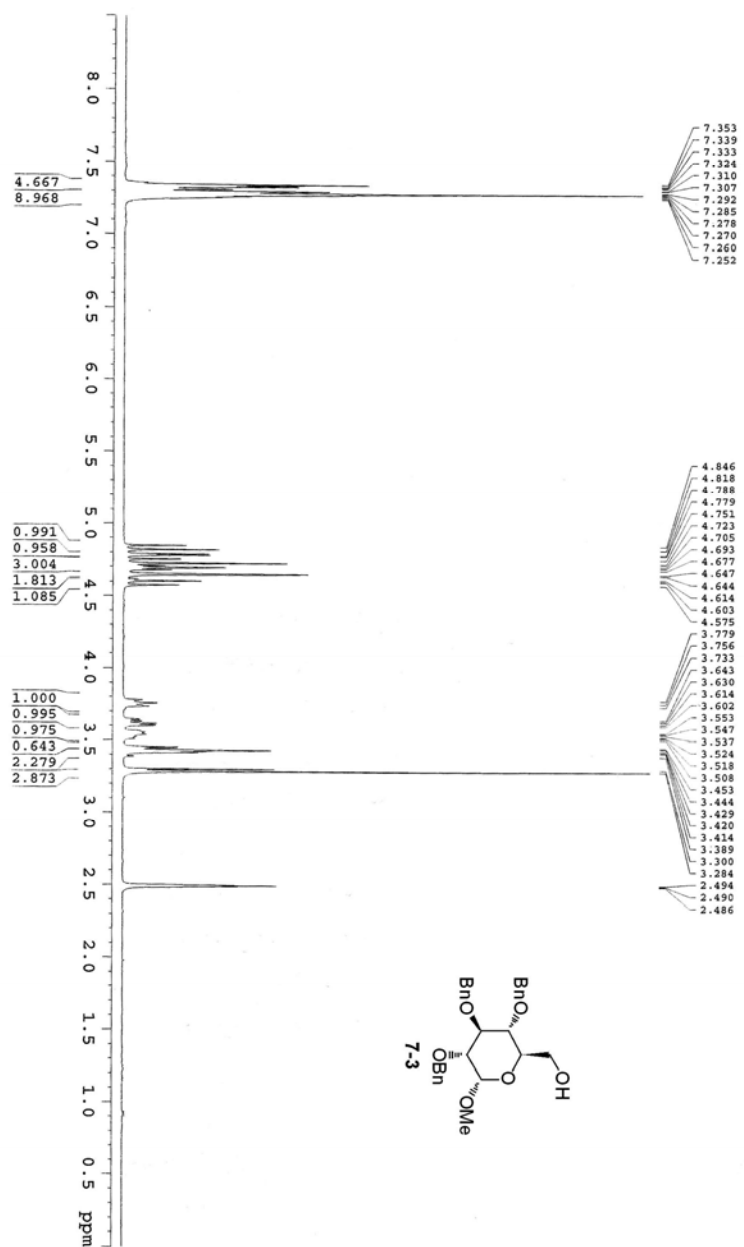
Current Data Parameters  
 NAME: 17102-M-21-201  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 2013-04-08  
 Time: 9:02  
 Instrument: av400  
 PROBRD: 5 mm DUL 13C-1  
 PULPROG: zgpg30  
 SOLVENT: DMSO  
 NS: 16  
 DS: 4  
 SWH: 4844.961 Hz  
 FIDRES: 0.147856 Hz  
 AQ: 3.351717 sec  
 INJ: 2.00  
 DE: 103.200 uS  
 TE: 300.2 K  
 D1: 6.00 uS  
 DI: 2.0000000 sec  
 ===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 11.85 uS  
 PL1: 0.00 dB  
 SFO1: 400.118850 MHz  
 F2 - Processing parameters  
 SI: 32768  
 SF: 400.130069 MHz  
 WDW: HO  
 SSB: 0  
 LB: 0.00 Hz  
 GB: 0  
 PC: 1.00



**BRUKER**  
AV400 NMR

```

Current Data Parameters
NAME      N-3(130403M-3P)
EXPNO     1
PROCNO    1
P2 - Acquisition Parameters
Date_     20150808
Time      8.41
INSTRUM   5 mm DUL AV400
PROBHD    zgpg30
PULPROG   zgpg30
TD         32768
SFO        400.1300000
AQ         0.327253
RG         1024
DE         6.00
TE         300.2 K
D1         2.00000000 sec
***** CHANNEL f1 *****
NUC1       13C
P1         12.00 usec
PL1        0.00 dB
SFO1       400.1181355 MHz
P2 - Processing parameters
SI         16384
SF         400.1300071 MHz
WDW        EM
SSB        0
GB         0
PC         1.00
  
```

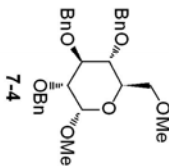
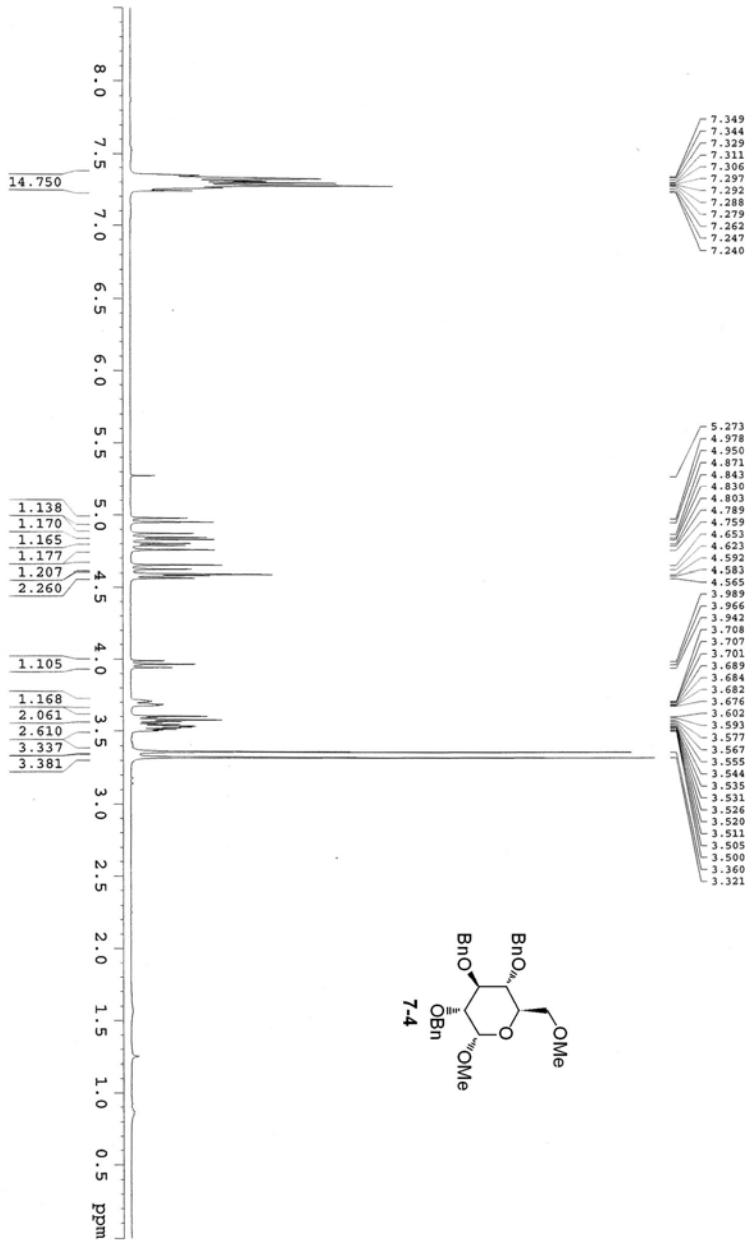


**BRUKER**  
AV400 NMR

```

Current Data Parameters
NAME      H-130908-1
EXPNO    1
PROCNO   1
F2 - Acquisition Parameters
Date_    20130408
Time     04:06
INSTRUM  spect
PROBHD   5 mm DUL 13C-1
PULPROG  zgpg30
AQ       3.2749
SOLVENT  DMSO
NS       16
DS       4
SWH       4901.961 Hz
FIDRES   0.144956 Hz
AQ       3.3423860 sec
RG       655
DM       102.000 usec
DE       6.00 usec
TE       300.2
D1       2.00000000 sec

***** CHANNEL f1 *****
NUC1      13C
P1       11.85 usec
PL1      -2.00 dB
SFO1     400.132629 MHz
F2 - Processing parameters
SI       32768
SF       400.130069 MHz
WDW      EM
SSB      0.00 Hz
LB       0
GB       0
PC       1.00
  
```



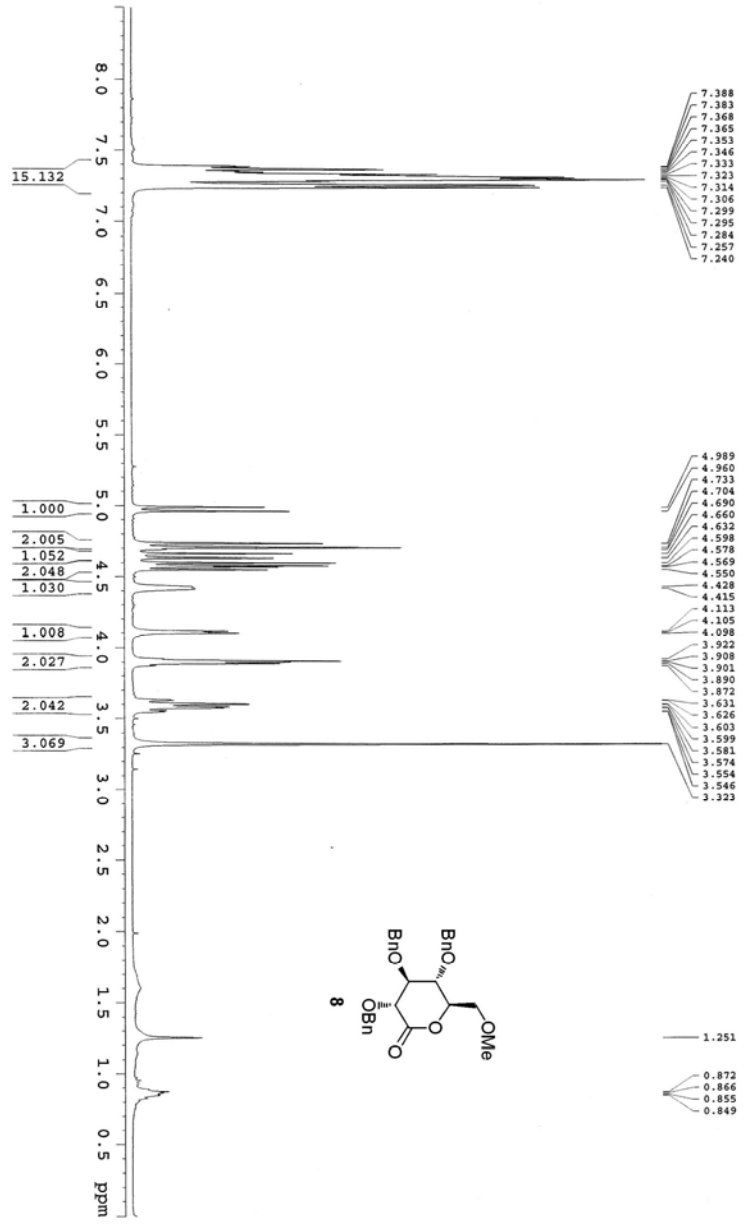
Current Data Parameters  
 EXPNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20140526  
 Time 8:46  
 F2 - Processing parameters  
 SI 16384  
 SF 400.1309354 MHz  
 MD no  
 SSB 0 Hz  
 CB 0 Hz  
 PC 1.00

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 13C  
 PULP 11.85 usec  
 PL1 -2.00 dB  
 SFO1 400.132909 MHz

F2 - Acquisition Parameters  
 Date\_ 20140526  
 Time 8:46  
 F2 - Processing parameters  
 SI 16384  
 SF 400.1309354 MHz  
 MD no  
 SSB 0 Hz  
 CB 0 Hz  
 PC 1.00

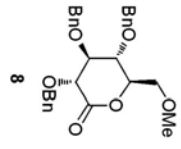
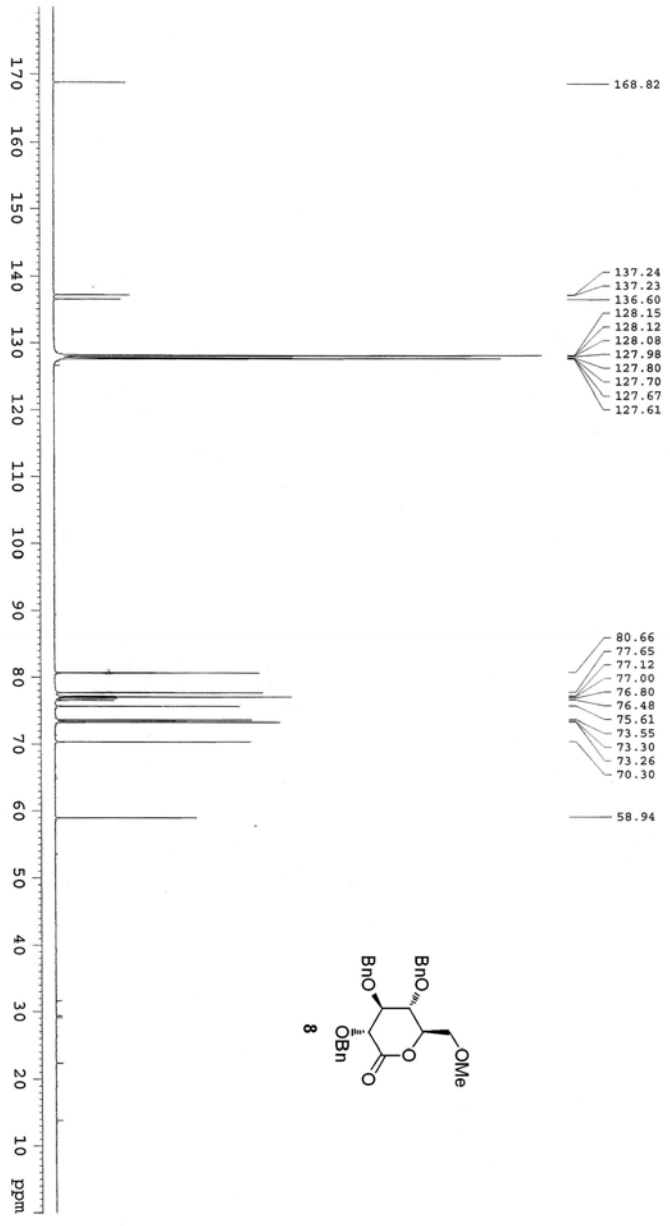






**BRUKER**  
AV400 NMR

Current Data Parameters  
 Date\_ 20140526  
 Time\_ 11:45:56  
 EXNO 1  
 PROCNO 1  
 P2 - Acquisition Parameters  
 Date\_ 20140526  
 Time\_ 11:45:56  
 INSTRUM av400  
 PROBHD 5 mm DDL 13C-1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 4  
 SWH 6578.947 Hz  
 FIDRES 0.200774 Hz  
 AQ 2.1904138 sec  
 RG 327.8  
 DW 76.000 usec  
 DE 3.000 usec  
 TE 300.2 K  
 D1 2.00000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 1H  
 P1 11.85 usec  
 PL 0.00 dB  
 SFO1 400.118550 MHz  
 P2 - Processing parameters  
 SI 16384  
 SF 400.1300174 MHz  
 DS 2  
 SW 5.00 Hz  
 ZS 0  
 GB 0  
 PC 1.00



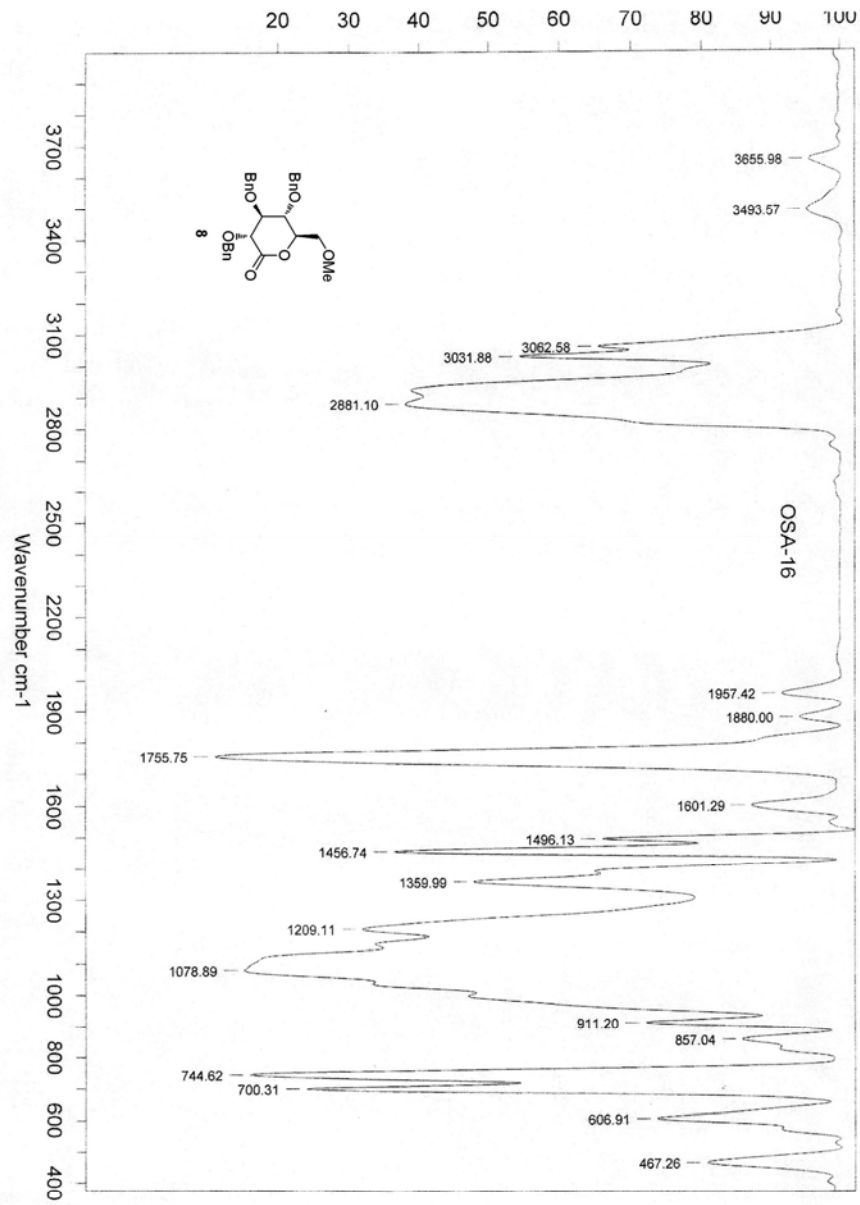
**BRUKER**  
**AV400 NMR**

Current Data Parameters  
 EXPNO 2  
 F2 - Acquisition Parameters  
 Date\_ 20140527  
 Time 18:16  
 Time2 18:16  
 PROBRD 5 mm DUL 13C-1  
 PULPROG zgpg30  
 SFO1 100.6218241 MHz  
 SOLVENT CDCl3  
 NS 4096  
 DS 4  
 SWH 30120.485 Hz  
 FIDRES 0.4839602 Hz  
 AQ 1.0979476 sec  
 DE 16.600 usec  
 TE 300.2 K  
 D1 2.0000000 sec  
 d11 0.0300000 sec

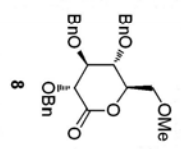
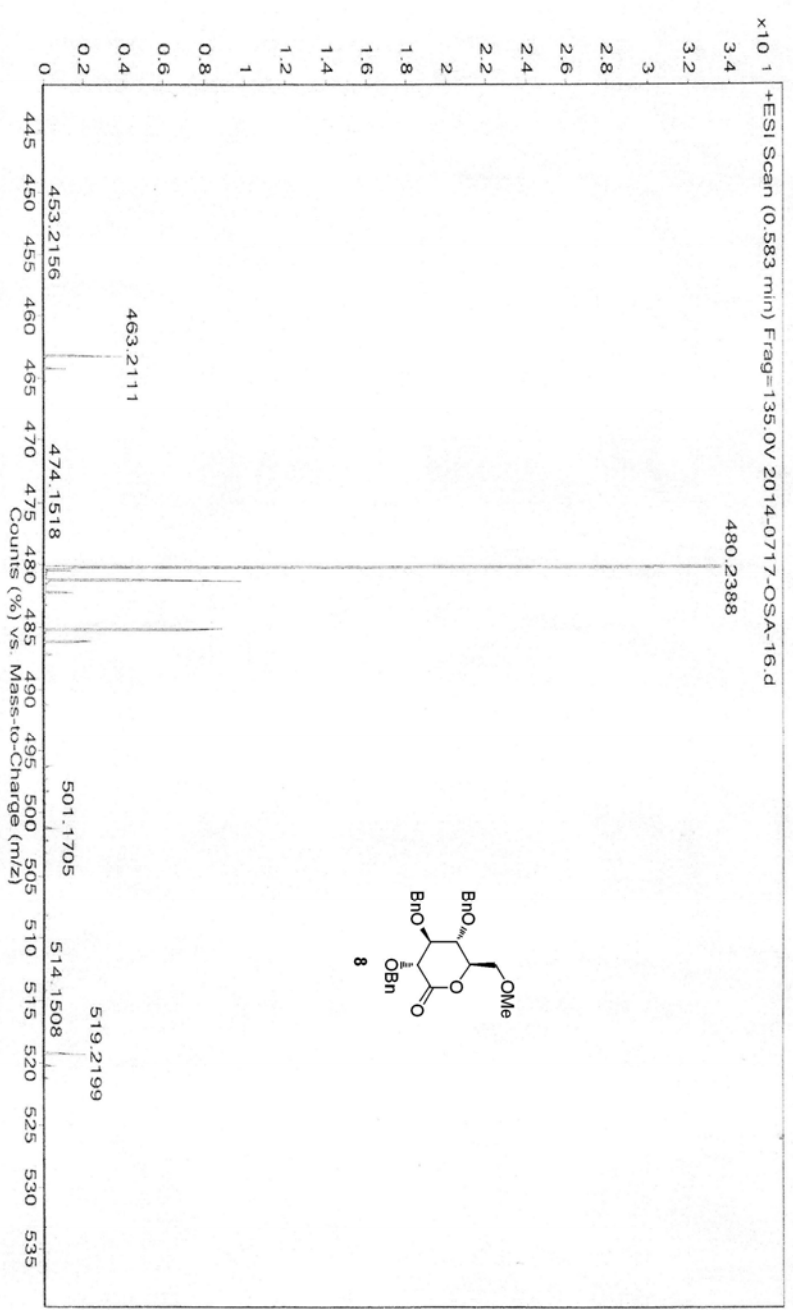
\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 13C  
 P1 13.05 usec  
 PL 0.00 dB  
 SFO1 100.6218241 MHz

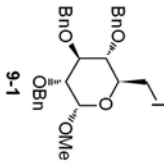
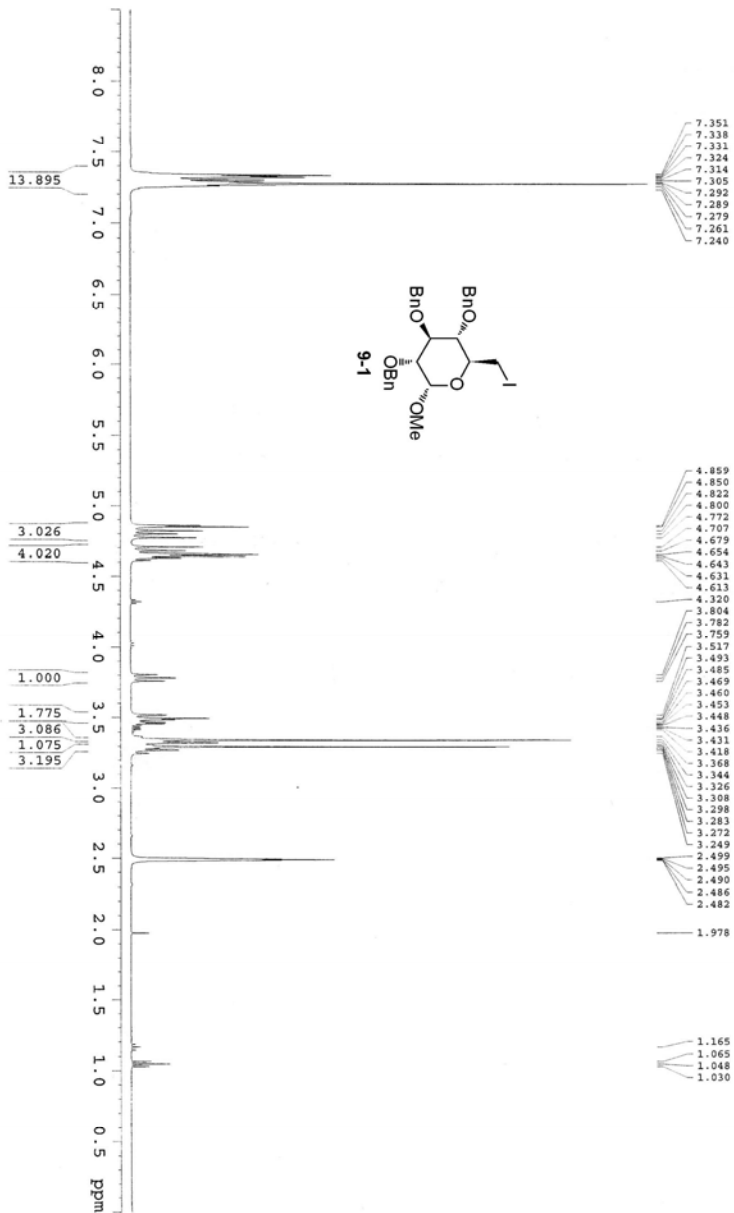
\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 CPDPRG2 waltz16  
 NUC2 1H  
 P2 190.00 usec  
 PL2 12.00 dB  
 PL12 16.53 dB  
 SFO2 400.1320007 MHz

F2 - Processing parameters  
 SI 32768  
 SF 100.6218241 MHz  
 WDW EM  
 SSB 0  
 GB 1.00 Hz  
 PC 1.40



Sample Name 2014-0717-OSA-16 Position P1-F1 Instrument Name Instrument 1 User Name  
 Inj Vol -1 InjPosition SampleType Sample IRM Calibration Status Success  
 Data Filename 2014-0717-OSA-16.d ACQ Method 0103.m Comment Acquired Time 7/17/2014 10:53:43

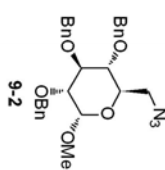
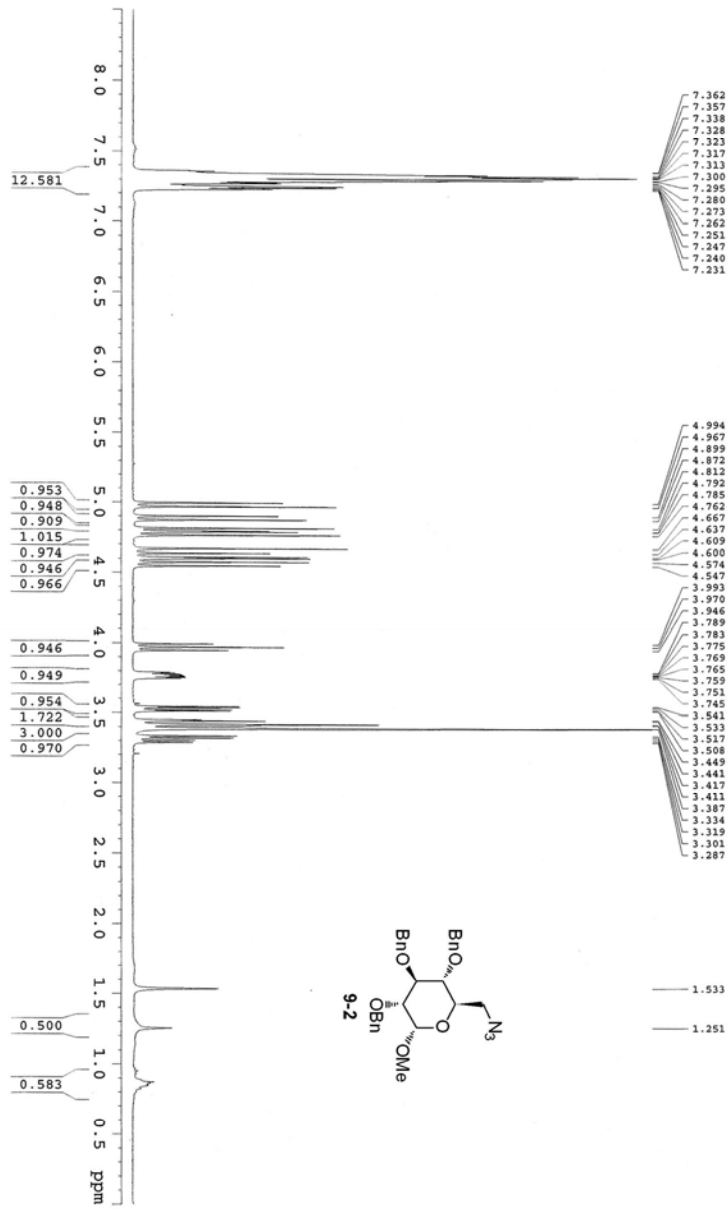




  
**AV400 NMR**

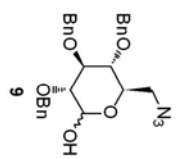
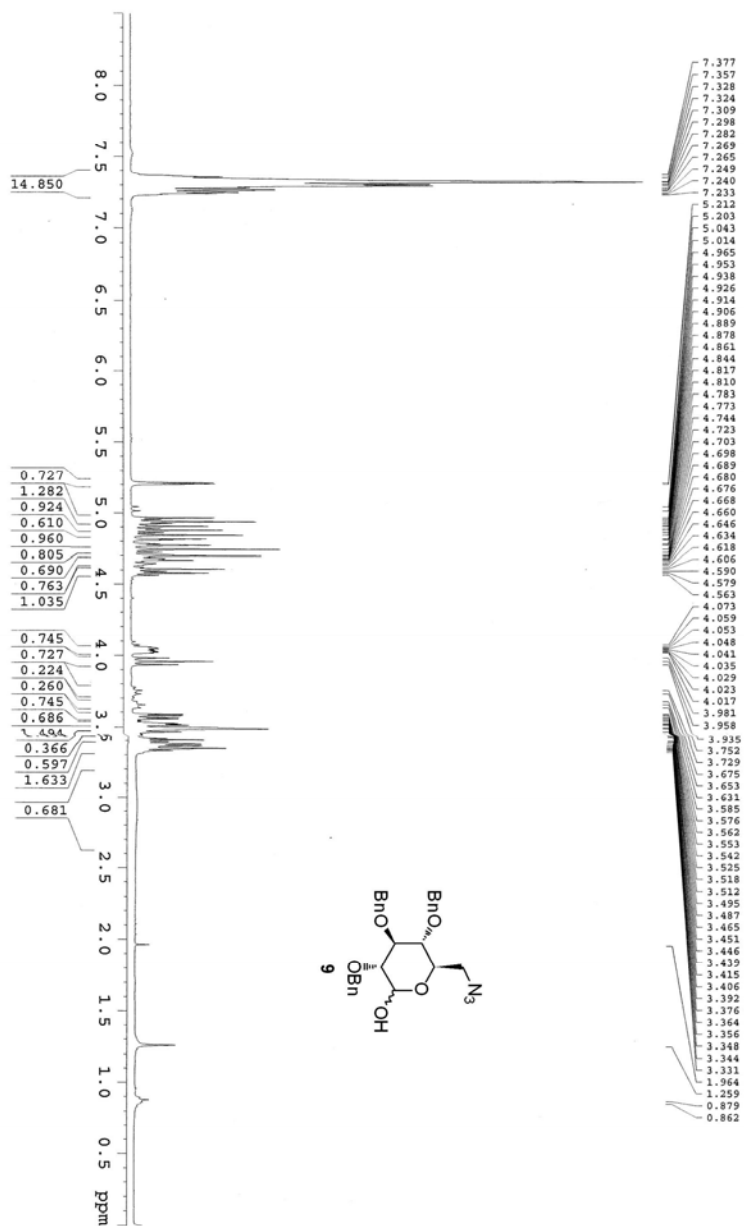
```

Current Data Parameters
NAME      N-5(130417H-5)
PROCNO    1
P2 - Acquisition Parameters
Date_     20130828
Time      13.39
INSTRUM   5 mm DNP AV400
PROBHD    zgpg30
PULPROG   zgpg30
TD         32768
SFO        400.146
AQ         1.00
RG         1024
DS         4
SSB        0
P1         0.156000 Hz
PC         0.156000 Hz
AQ         3.2047603 sec
RG         97.420
DE         6.00 usec
TE         300.2 K
D1         2.00000000 sec
***** CHANNEL f1 *****
NUC1       13C
P1         11.85 usec
PL1        -2.00 dB
SFO1       400.119260 MHz
P2 - Processing parameters
SI         32768
SF         400.130838 MHz
WDW        ms
SSB        0
CB         0
PC         1.00
  
```



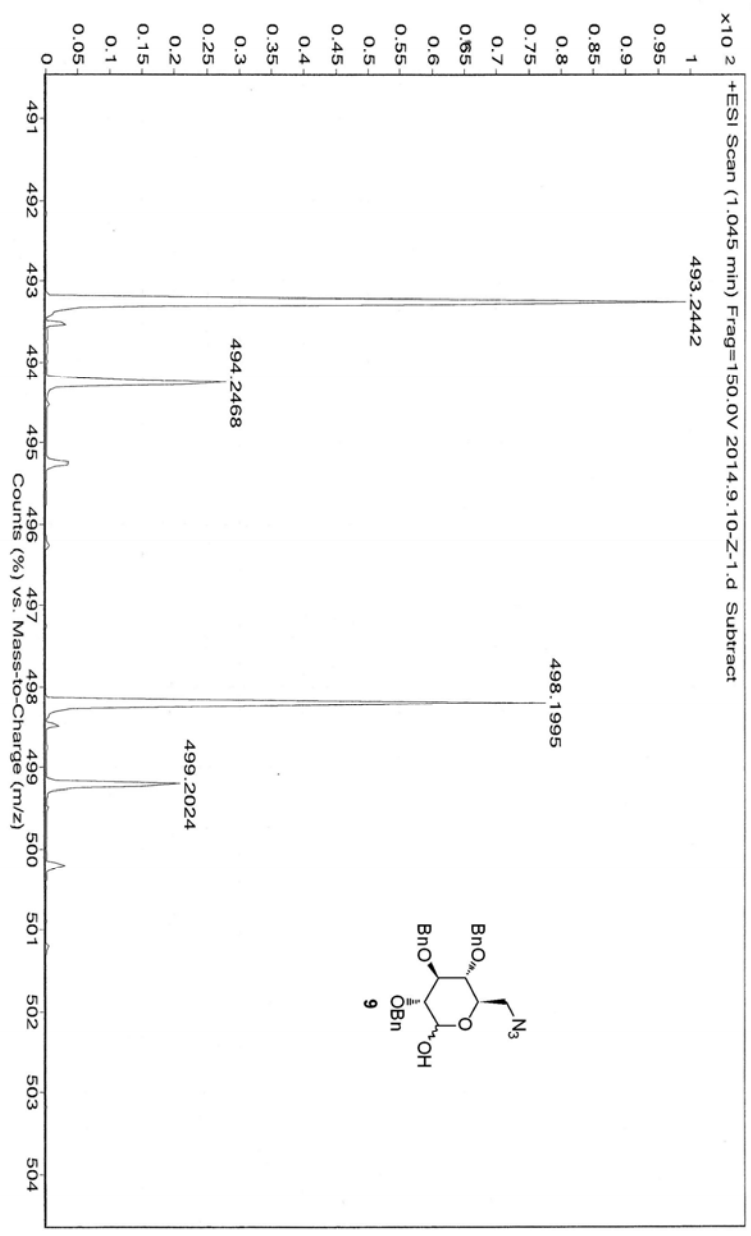
**BRUKER**  
AV400 NMR

Current Data Parameters  
 EXPNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20140923  
 Time 15:27  
 INSTRUM spect  
 PROBHD 5 mm DUL 13C-1  
 PULPROG zgpg30  
 SOLVENT CDCl3  
 NS 16  
 DS 4  
 SWH 5376.344 Hz  
 FIDRES 0.1644973 Hz  
 AQ 3.04437 sec  
 RG 143.7  
 DW 93.000 usec  
 DE 30.0 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 13C  
 P1 11.85 usec  
 PL 0.00 dB  
 SFO1 400.137426 MHz  
 F2 - Processing parameters  
 SI 16384  
 SF 400.1300173 MHz  
 NO 4096  
 SSB NO  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

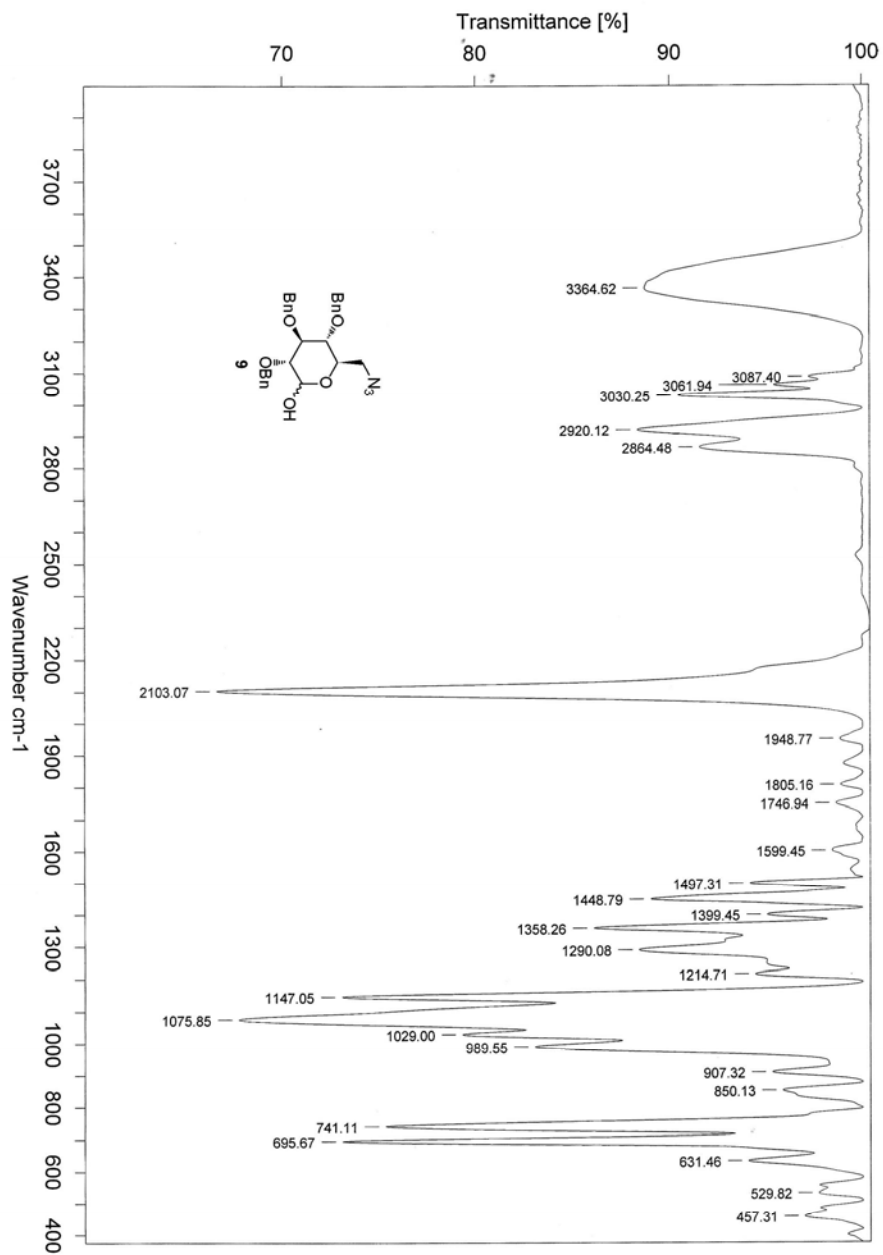


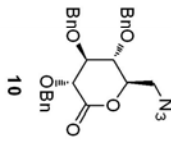
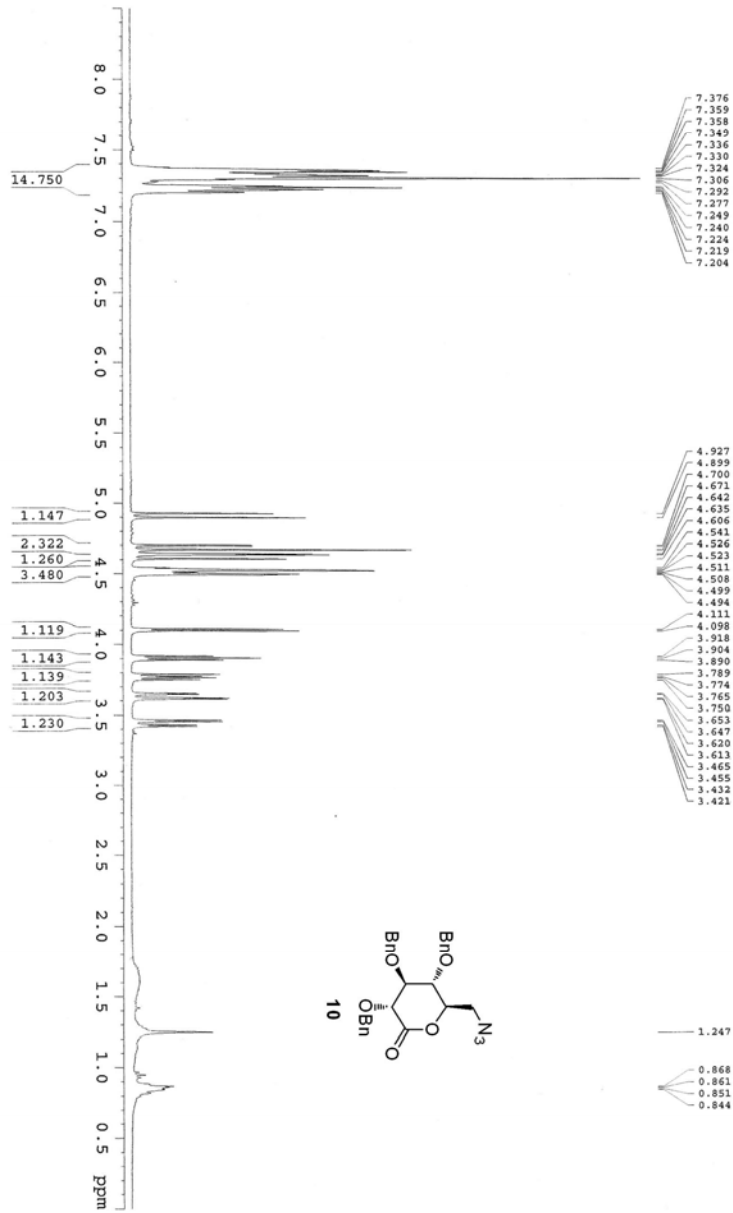
Current Data Parameters  
 NAME MP-140526-05A-19  
 PROCNO 1  
 P2 - Acquisition Parameters  
 Date\_ 20140526  
 Time\_ 9.11  
 INSTRUM 5 mm DUL 400K  
 PULPROG zgpg30  
 TD 32768  
 SFO 400.136344  
 F2 - Processing parameters  
 SI 16384  
 SF 400.136344 MHz  
 WDW no  
 SSB 0 Hz  
 GB 0 Hz  
 PC 1.00

Sample Name 2014.9.10-Z-1 Position P1-A7 Instrument Name Instrument 1 User Name  
 Inj Vol -1 InjPosition ACQ Method 0103.m Sample Type Sample IRM Calibration Status  
 Data Filename 2014.9.10-Z-1.d Comment  
 Success  
 Acquired Time 9/10/2014 12:25:01 PM







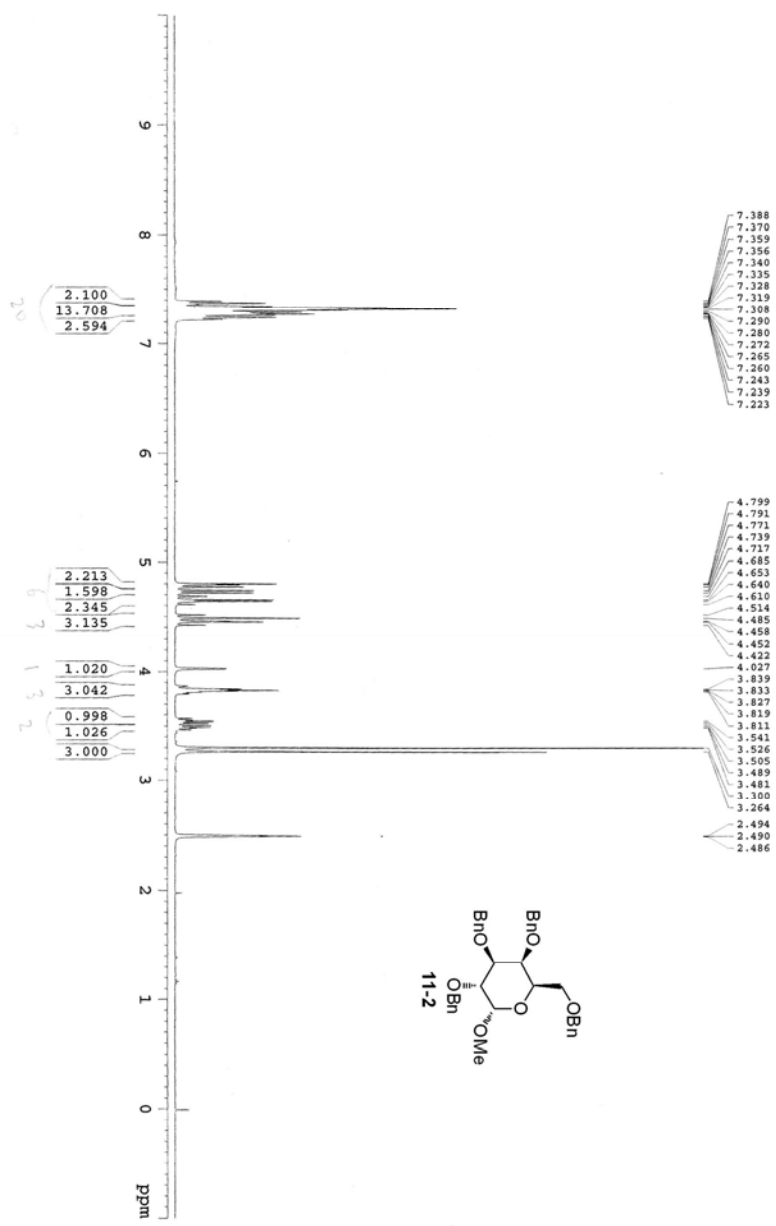
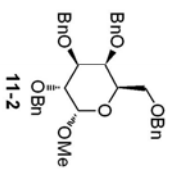


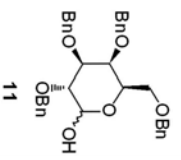
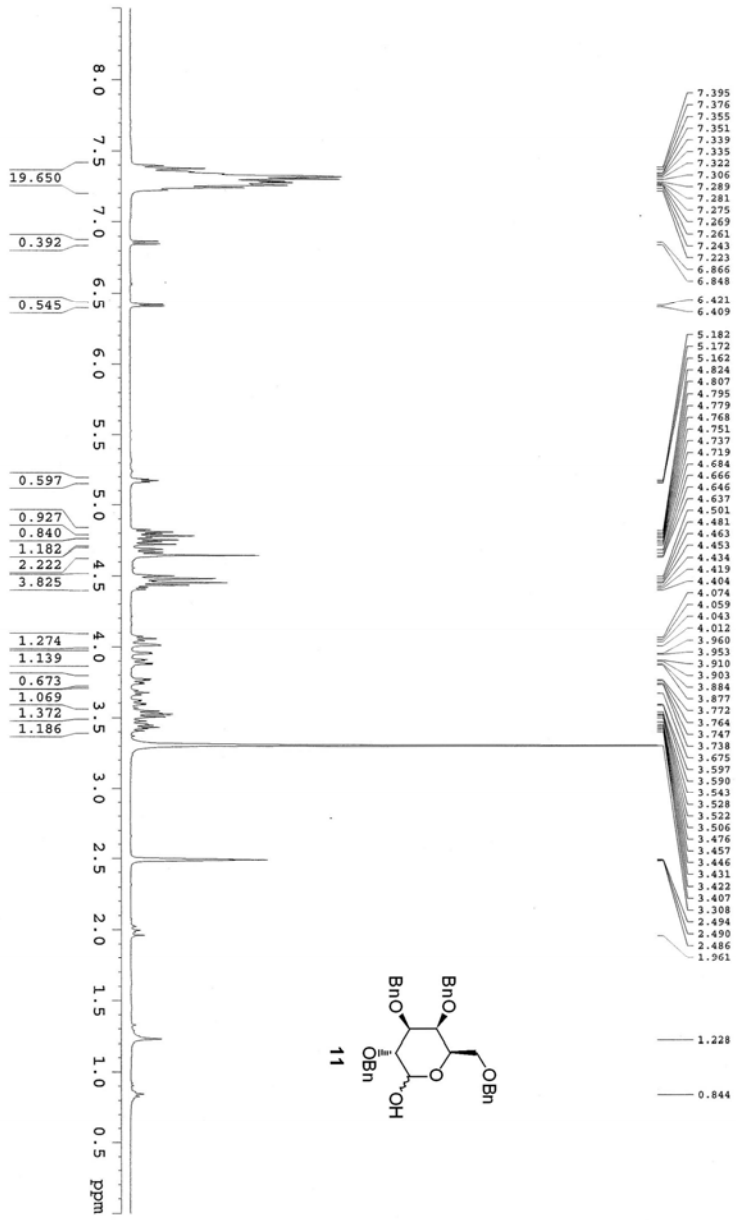
Current Data Parameters  
 Name: NP-140526-OSN-20  
 ExpNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_Time: 20140526 9:20  
 Instrument: AV400  
 Processor: 32  
 PULPROG: zgpg30  
 TOU: 1.00  
 SFO: 400.130175  
 AQ: 2.2544885 sec  
 TM: 68.800 usec  
 DE: 6.90 usec  
 TE: 2.00000000 K  
 D1: 2.00000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1: 13C  
 P1: 11.85 usec  
 PL1: 0.00 dB  
 SFO1: 400.130175 MHz  
 F2 - Processing parameters  
 SI: 32768  
 SF: 400.130175 MHz  
 MDW: no  
 SSB: 0.00 Hz  
 ICB: 0  
 PC: 1.00





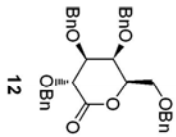
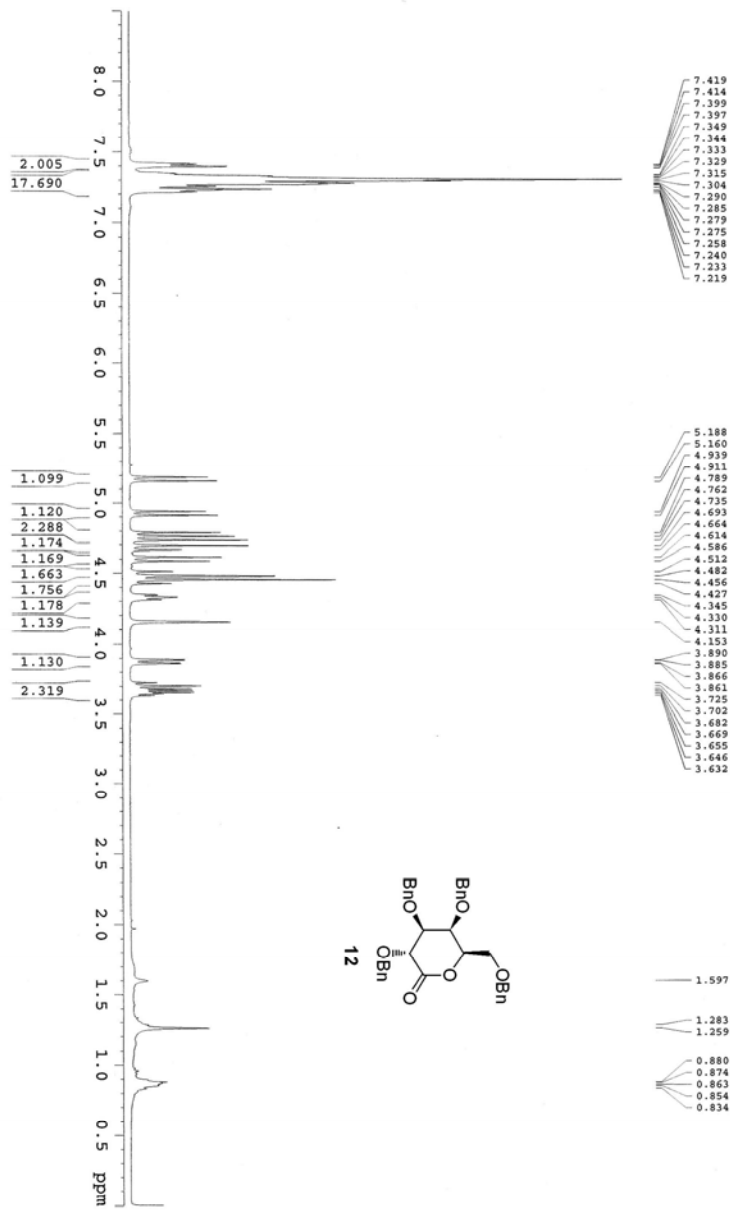
Current Data Parameters  
 NAME TT702-B41-3  
 PROBHD 1  
 P2 - Acquisition Parameters  
 INSTRUM 5 mm BBO 1H/2  
 Time 15:57  
 PULPROG zgpg30  
 TOUVENT 32768  
 NS 0  
 DS 0  
 FIDRES 0.131141 Hz  
 AQ 0.132461 Hz  
 RG 2.9527256 sec  
 DE 79.128 usec  
 RE 6.90 usec  
 TE 300.0 K  
 DI 2.0000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 13C  
 P1 11.85 usec  
 PL1 -2.00 dB  
 SFO1 400.1321206 MHz  
 P2 - Processing parameters  
 SI 16384  
 SF 400.1300000 MHz  
 WDW no  
 SSB 0  
 GB 0  
 PC 1.00





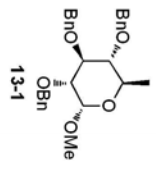
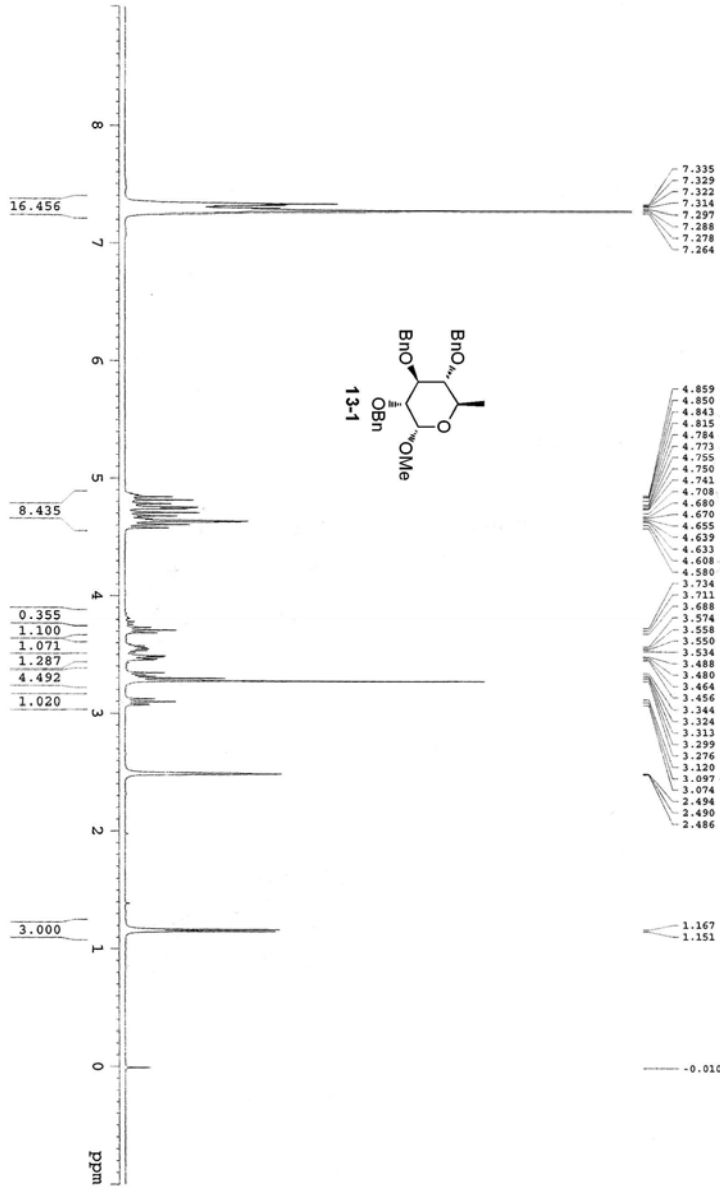
```

Current Data Parameters
NAME      NP-140725-08A_22
EXPNO     1
PROCNO    1
Date_     20140724
Time      14:42:42
INSTRUM   AV400
PROBHD    5 mm DDL 13C-1
PULPROG   zgpg30
TD         32768
SOLVENT   DMSO
NS        16
DS         1
SWH        7225.434 Hz
FIDRES    0.225082 Hz
AQ         2.225082 sec
RG         143.7
WDW        EM
SSB        0
LB         69.200 ussec
GB         0
PC         300.0 K
TE         300.2
D1         2.00000000 sec
***** CHANNEL f1 *****
NUC1       13H 100%
P1         12.00 usec
PL1        -2.00 dB
SFO1       400.115641 MHz
F2 - Processing parameters
SI         400.130068 MHz
SF         400.130068 MHz
WDW        EM
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
  
```

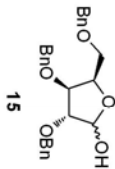
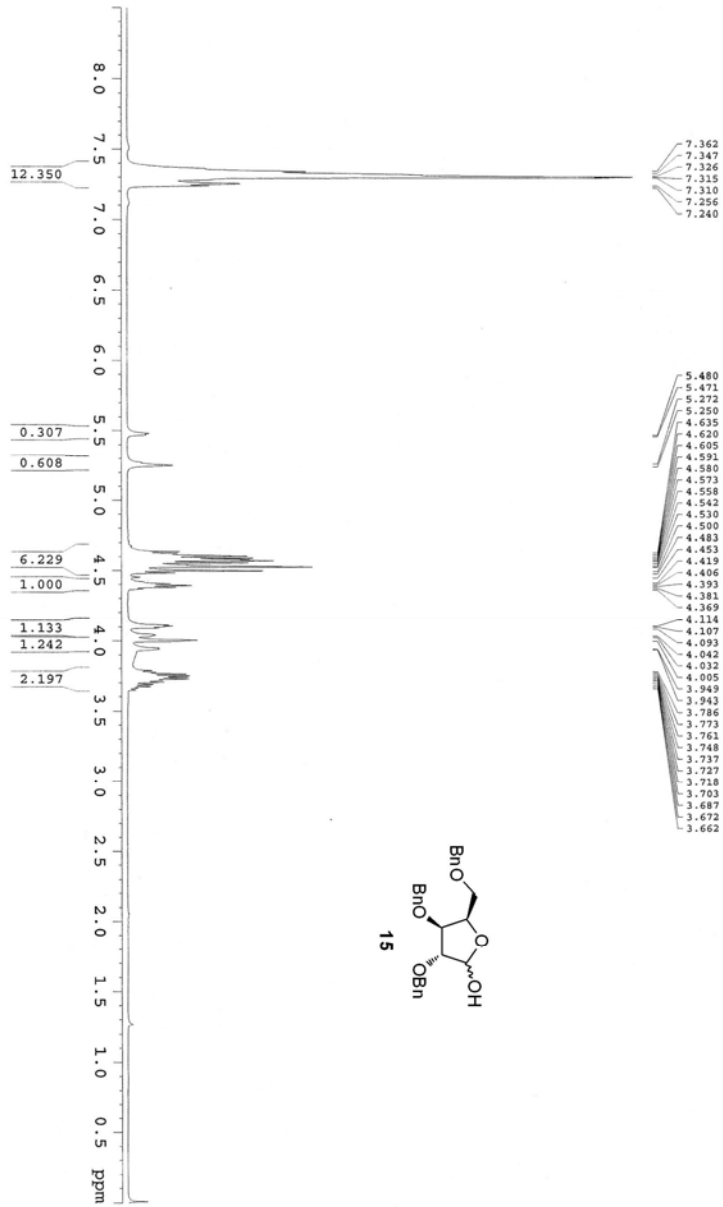


Current Data Parameters  
 NAME WF-140515-02A-23  
 EXPNO 1  
 PROCNO 1  
 P2 - Acquisition Parameters  
 Date\_ 20140515  
 Time 0:16  
 INSTRUM spect  
 PROBUFG 5 mm DUL 13C-1  
 PULPROG zgpg30  
 SOLVENT CDCl3  
 NS 16  
 DS 4  
 SWH 7022.472 Hz  
 FIDRES 0.214309 Hz  
 AQ 2.312016 sec  
 DM 71.700 usec  
 DE 170.0 usec  
 DI 2.0000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUCL1 1H  
 P1 11.85 usec  
 SFO1 400.117090 MHz  
 P2 - Processing parameters  
 SI 16384  
 SF 400.1300171 MHz  
 NS 16  
 ISF 0.00 Hz  
 GB 0  
 PC 1.00





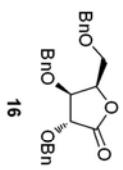
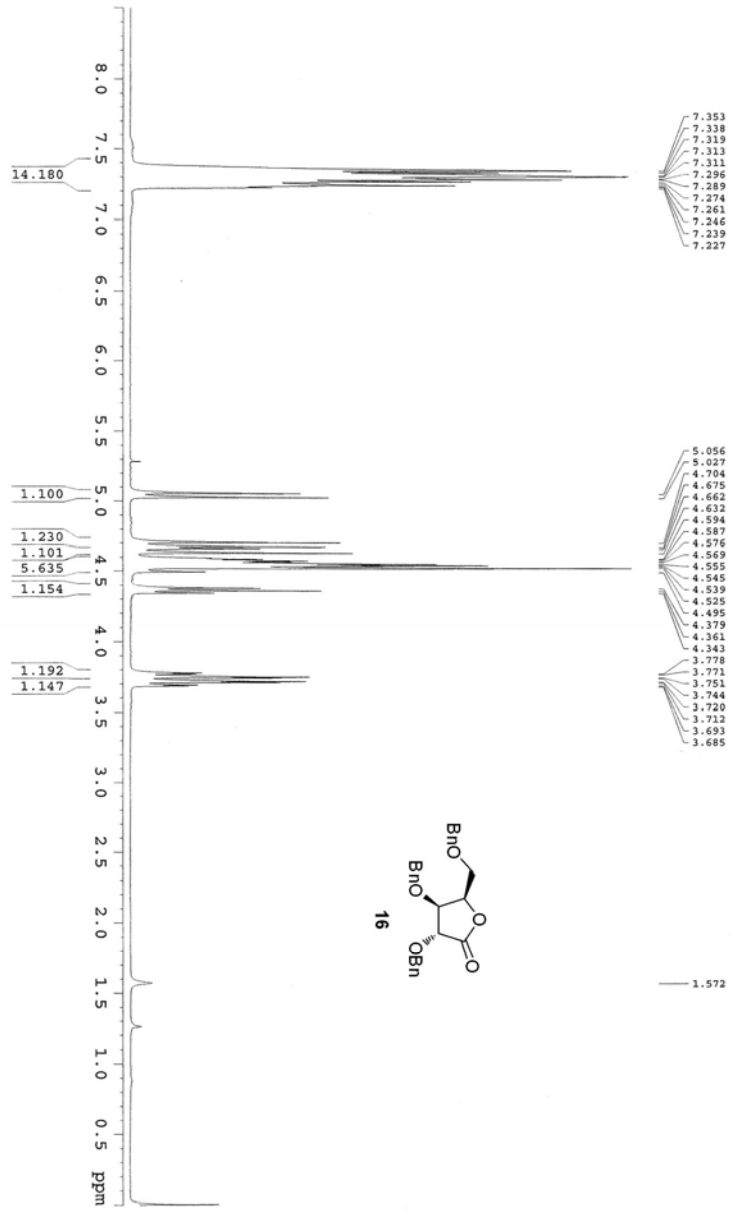
Current Data Parameters  
 NAME N-6(130425N-6P)  
 EXPNO 1  
 PROCNO 1  
 P2 - Acquisition Parameters  
 Date\_ 2011.07  
 Time 11:07  
 INSTRUM 5 mm DNP AV400  
 PULPROG zgpg30  
 TOUVENT 32768  
 NS 16  
 DSF 5376.348 Hz  
 SFO 400.1460713 MHz  
 AQ 0.1660713 sec  
 FIDRES 3.0474733 sec  
 TD 93,000  
 DE 6.00 usec  
 RE 2.0000000 Ksec  
 D1 2.0000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 13C  
 P1 11.85 usec  
 PL1 0.00 dB  
 SFO1 400.116493 MHz  
 P2 - Processing parameters  
 SI 32768  
 SF 400.1300070 MHz  
 DSF 5376.348 Hz  
 SFO 400.1300070 MHz  
 DE 6.00 Hz  
 GB 1.00  
 PC 1.00



**BRUKER**  
**AV400 NMR**

Current Data Parameters  
 Name: WP-140515-OSA-9  
 ExpNO: 1  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_Time: 20140516 9:14  
 Name: 15  
 PULPROG: zgpg30  
 F2 - Processing parameters  
 Date\_Time: 20140516 9:14  
 Name: 15  
 PULPROG: zgpg30  
 F2 - Processing parameters  
 Date\_Time: 20140516 9:14  
 Name: 15  
 PULPROG: zgpg30



7.353  
7.338  
7.319  
7.313  
7.311  
7.296  
7.289  
7.274  
7.261  
7.246  
7.239  
7.227

5.056  
5.027  
4.704  
4.675  
4.662  
4.632  
4.594  
4.587  
4.576  
4.569  
4.555  
4.545  
4.539  
4.525  
4.495  
4.379  
4.361  
4.343  
3.778  
3.771  
3.751  
3.744  
3.720  
3.712  
3.691  
3.685

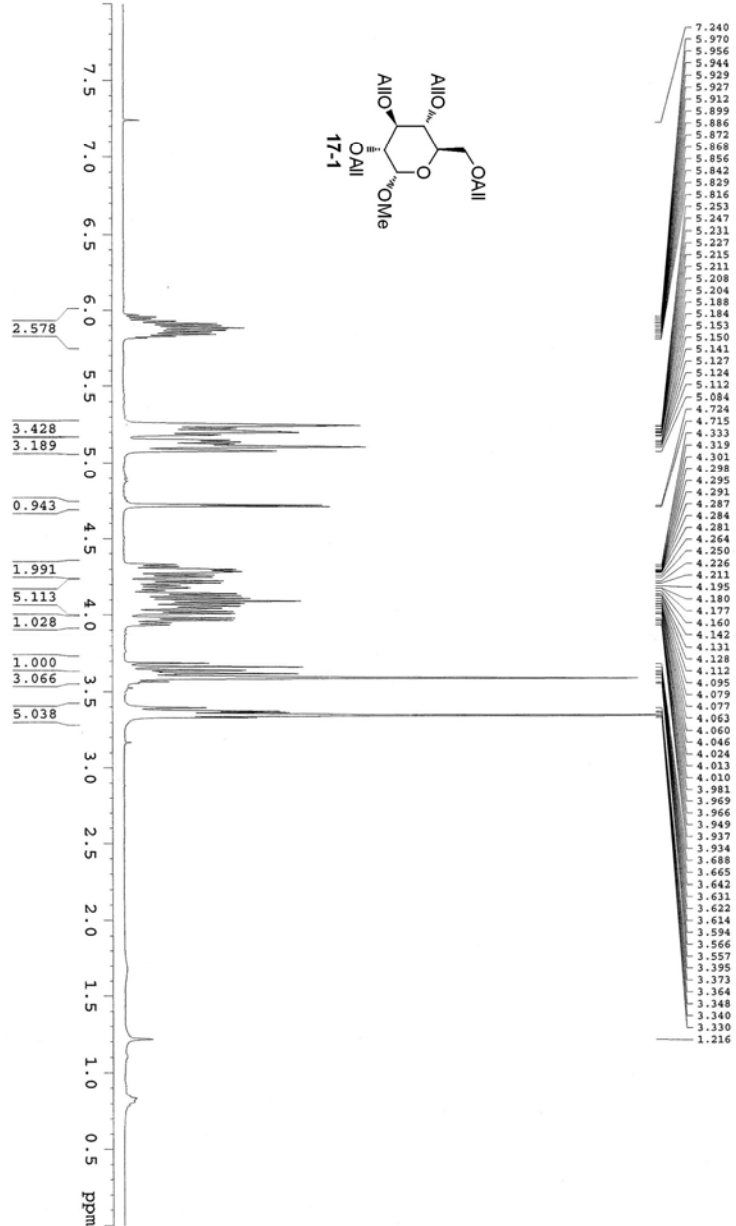
1.572



```

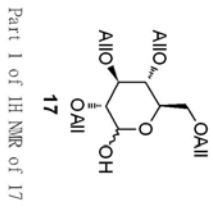
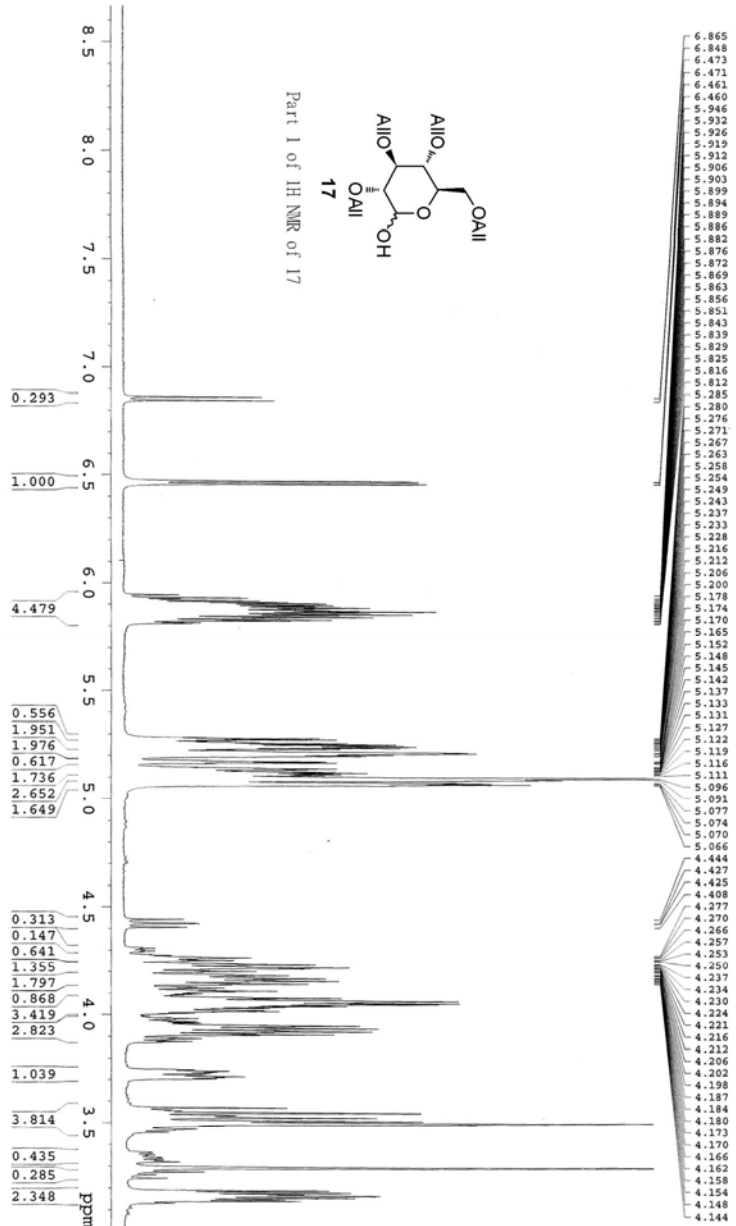
Current Data Parameters
NAME      HP-140515-OSB-10
EXPNO     1
PROCNO    1
Date_     20140516
Time      12.42
INSTRUM   AV400
PROBHD    5 mm DUL 1H-1
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
DS         1
SWH        7062.147 Hz
AQ         2.1205245 sec
RG         70.800 umsec
DM         300.0 K
TE         2.00000000 sec
D1         2.00000000 sec
***** CHANNEL f1 *****
NUC1       1H
P1         11.00 usec
PL1        -1.00 dB
SFO1       400.113981 MHz
F2 - Processing parameters
SI         16384
SF         400.1130145 MHz
WDW        EM
SSB        0
GB         0.00 Hz
PC         1.00
  
```



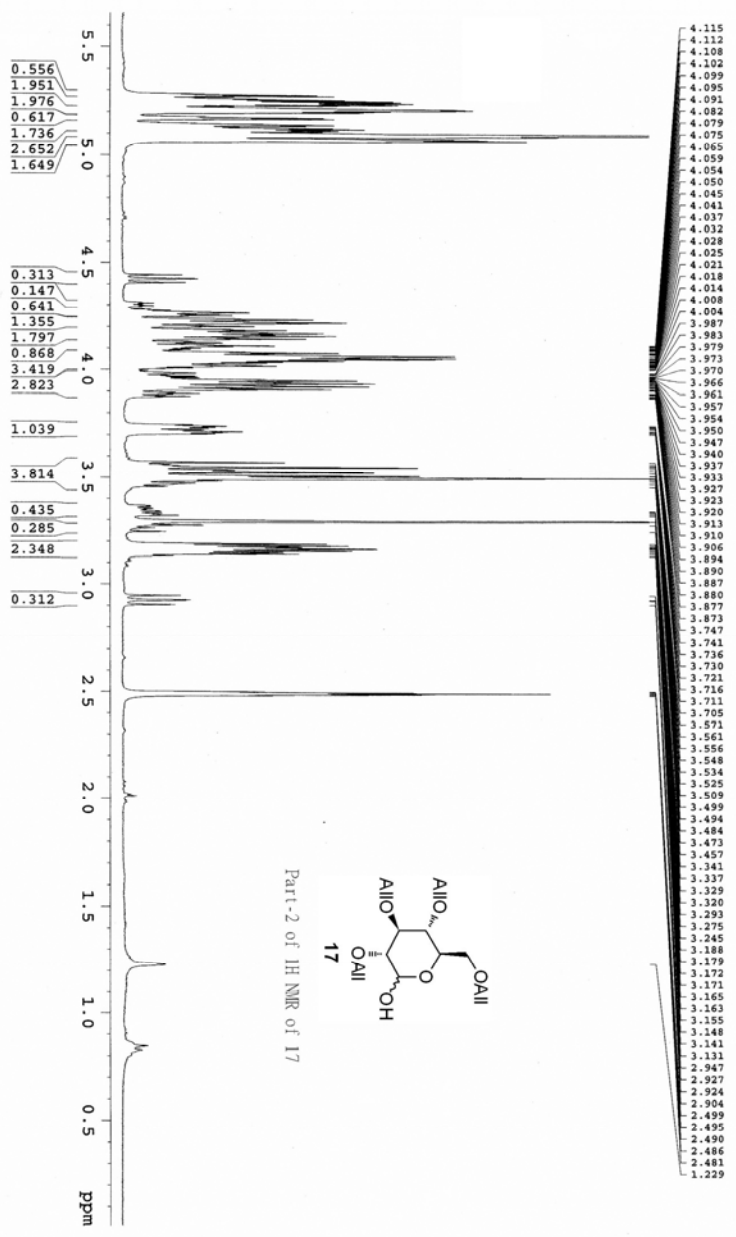


**BRUKER**  
AV400 NMR

Current Data Parameters  
 Name: 17-1\_400MHz\_24  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20140523  
 Time: 11:00:00  
 INSTRUM: av400  
 PROBMOD: 5 mm DUL 13C-1  
 PULPROG: zgpg30  
 D2: 32.748  
 SOLVENT: CDCl3  
 NS: 16  
 DS: 4  
 SWH: 4930.366 Hz  
 FIDRES: 0.150481 Hz  
 AQ: 0.0324033 sec  
 RG: 3.327403  
 DW: 101.400 usec  
 DE: 8.00 usec  
 TE: 300.2 K  
 D1: 2.00000000 sec  
 D11:   
 ===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 11.85 usec  
 SFO1: 400.114973 MHz  
 F2 - Processing parameters  
 SI: 32768  
 SF: 400.1100173 MHz  
 MDW: 60  
 LB: 0.00 Hz  
 GB: 0  
 PC: 1.00

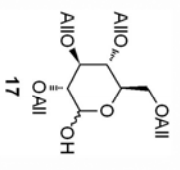


Current Data Parameters  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20140722  
 Time 1.30  
 INSTRUM spect  
 PROBRD 5 mm DUL 13C-1  
 PULPROG zgpg30  
 SOLVENT DMSO  
 NS 16  
 DS 4  
 SWH 5518.760 Hz  
 FIDRES 0.168419 Hz  
 AQ 2.9688108 sec  
 SFO1 400.130070 MHz  
 DM 90.000 usec  
 DE 6.00 usec  
 DI 2.00000000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.85 usec  
 PL1 -2.00 dB  
 SFO1 400.130070 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 400.130070 MHz  
 HF 0  
 SSB 0.00 Hz  
 GB 0  
 PC 1.00

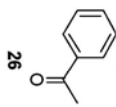
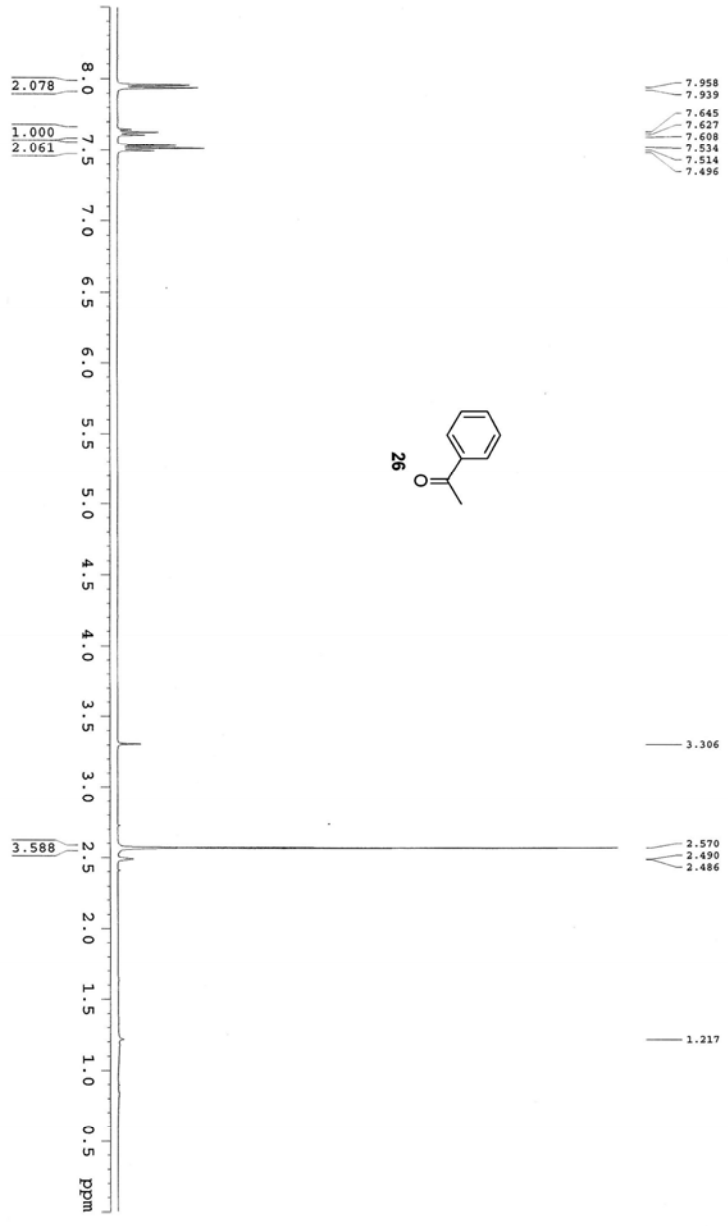


4.115  
4.112  
4.108  
4.102  
4.099  
4.095  
4.091  
4.082  
4.079  
4.075  
4.065  
4.059  
4.054  
4.050  
4.045  
4.041  
4.037  
4.032  
4.028  
4.025  
4.021  
4.018  
4.014  
4.008  
4.004  
3.987  
3.983  
3.979  
3.973  
3.970  
3.966  
3.961  
3.957  
3.954  
3.950  
3.947  
3.940  
3.937  
3.933  
3.927  
3.923  
3.920  
3.913  
3.910  
3.906  
3.894  
3.890  
3.887  
3.880  
3.877  
3.873  
3.847  
3.741  
3.736  
3.730  
3.721  
3.716  
3.711  
3.705  
3.671  
3.561  
3.556  
3.548  
3.534  
3.525  
3.509  
3.499  
3.494  
3.484  
3.473  
3.457  
3.341  
3.337  
3.325  
3.320  
3.293  
3.275  
3.270  
3.179  
3.172  
3.171  
3.145  
3.141  
3.131  
2.947  
2.927  
2.924  
2.904  
2.499  
2.495  
2.490  
2.486  
2.483  
1.229

Part 2 of 1H NMR of 17



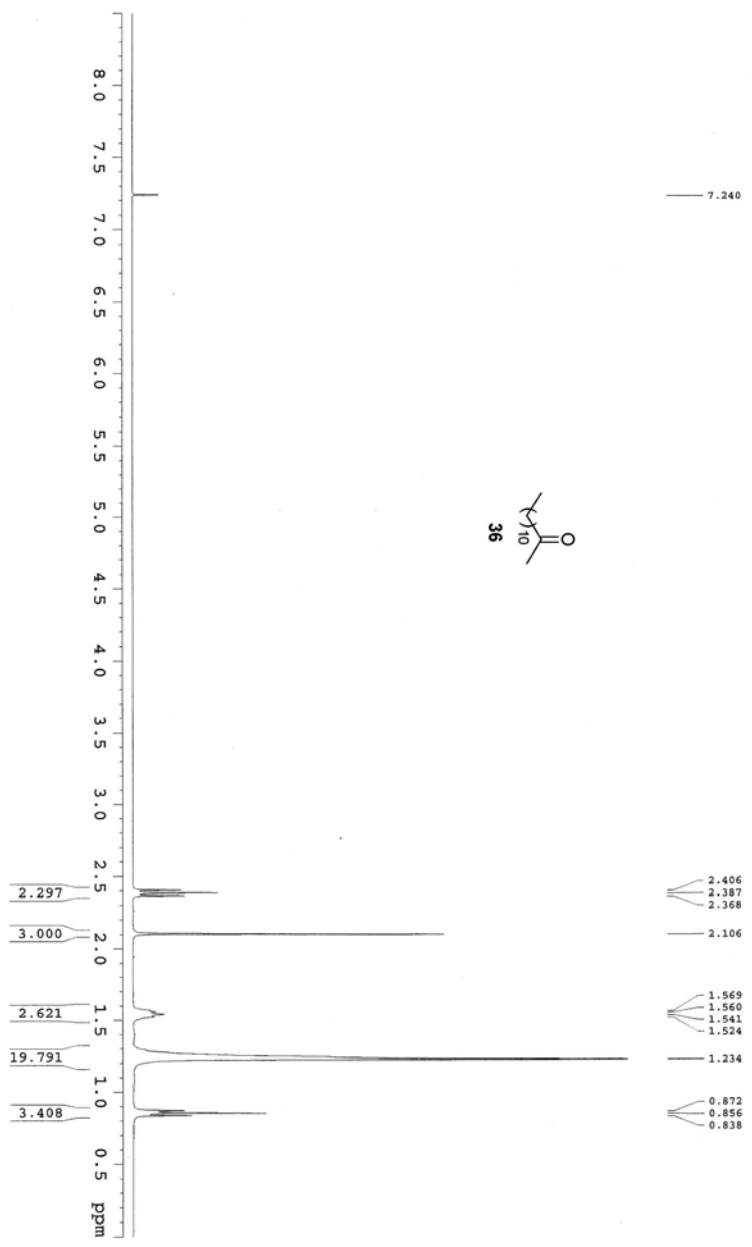
Current Data Parameters  
 NAME WP-140722-02A-25  
 PROCNO 1  
 P2 - Acquisition Parameters  
 Date\_ 20140723  
 Time\_ 1.30  
 INSTRUM spect  
 PROBHD 5 mm DPL 1H/1  
 PULPROG zgpg30  
 TD 32768  
 SFO 400.130400  
 AQ 16  
 DS 512  
 FIDRES 0.168419 Hz  
 AQ 2.9688108 sec  
 DE 90.400 usec  
 TE 300.2 K  
 D1 2.0000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 13C  
 P1 11.85 usec  
 PL1 -2.00 dB  
 SFO1 400.111868 MHz  
 P2 - Processing parameters  
 SI 32768  
 SF 400.130400 MHz  
 WBW 0  
 SSB 0  
 CB 0  
 PC 1.00



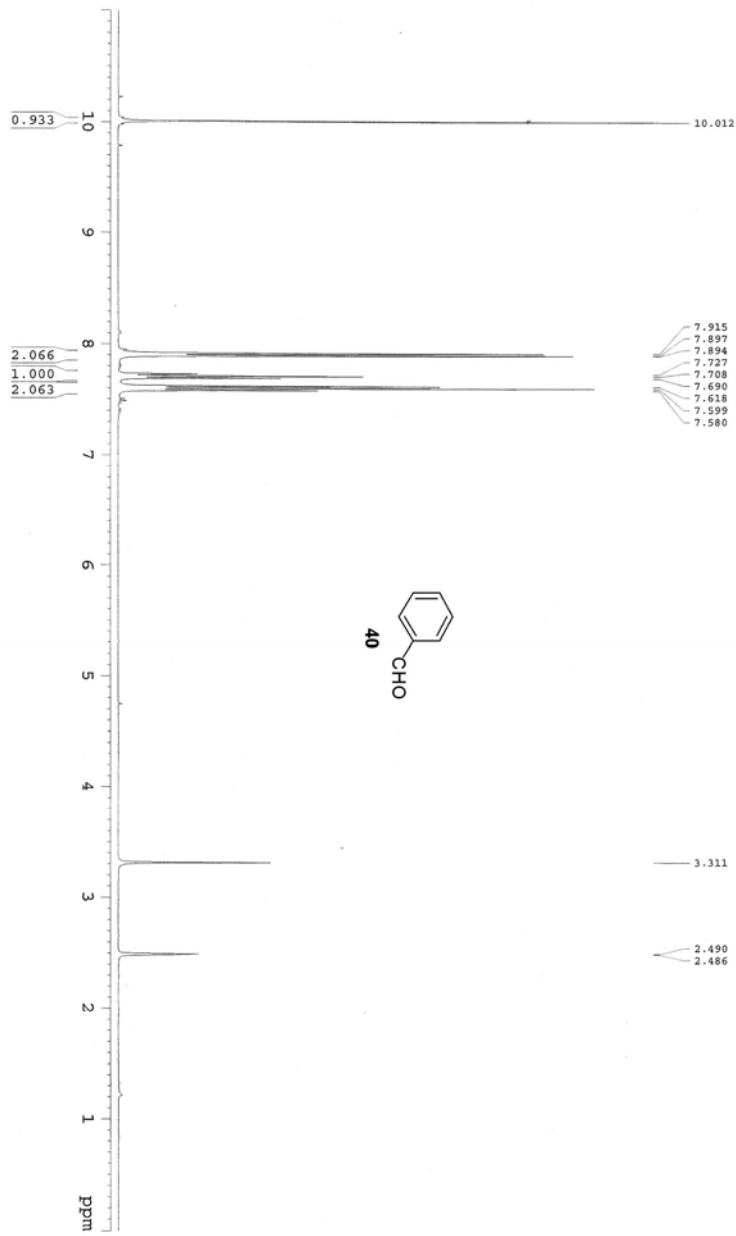
**BRUKER**  
AV400 NMR

```

Current Data Parameters
Date_ 20140627
Time 8:20
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
SOLVENT DMSO
NS 16
DS 4
SWH 6944.444 Hz
FIDRES 0.211928 Hz
AQ 2.35292114 sec
RG 114
DM 72.000 usec
DE 360.0 X
TE 30.0 X
D1 2.00000000 sec
***** CHANNEL f1 *****
NUC1 1H
P1 11.85 usec
PL 0.00 dB
SFO1 400.1318639 MHz
P2 - Processing parameters
SI 16384
SF 400.1300071 MHz
RG 114
AQ 2.35292114 sec
DE 360.0 X
TE 30.0 X
D1 2.00000000 sec
  
```



Current Data Parameters  
 NAME WP-140630-GSN-47  
 EXPNO 1  
 PROCNO 1  
 P2 - Acquisition Parameters  
 Date\_ 20140630  
 Time 11:11:11  
 INSTRUM av400  
 PROBRD 5 mm DUL 13C-1  
 TD 65536  
 SFO 125.760  
 AQ 1.00000000  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 4  
 SWH 6410.286 Hz  
 FIDRES 0.135625 Hz  
 AQRES 2.352500 sec  
 RG 655  
 DM 78.000 usec  
 DE 1.000 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 \*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 13C  
 P1 11.80 usec  
 PL 0.00 dB  
 SFO1 400.1318432 MHz  
 P2 - Processing parameters  
 SI 16384  
 SF 400.1300175 MHz  
 MD 65536  
 AS 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00



**BRUKER**  
AV400 NMR

```

Current Data Parameters
NAME      WP-140811-054-50-D
EXPNO    1
PROCNO   1
P1 - Acquisition Parameters
Date_    20140812
Time     15:50
INSTRUM  spect
PROBHD   5 mm BBL 1H-1
PULPROG  zgpg30
SOLVENT  DMSO
NS       16
DS       4
SSB      0
AQ       0.957971 Hz
FIDRES   0.276427 Hz
AQ       1.8098436 sec
RG        55.200 usec
DE       3.00 usec
DI       2.0000000 sec
***** CHANNEL f1 *****
NUC1     13C
P1L1     11.85 usec
PL1      2.00 dB
SFO1     400.131798 MHz
P2 - Processing parameters
SI       400.130096 MHz
SF        400.130096 MHz
WDW      hc
SSB      0
GB       0
PC       1.00
  
```