

Supplementary Material
for

Reproducible Spontaneous Isolation and Systematic Characterization of an
 α -D-Glucopyranose-Rich Crystalline Solid from *Khalas* Date Fruit
(*Phoenix dactylifera* L.)

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Table of contents

Contents	Page
Figure S1. LC–MS Spectrum of the isolated α -D-glucopyranose-rich fraction (Batch 1).	S5
Figure S2. LC–MS Spectrum of the isolated α -D-glucopyranose-rich fraction (Batch 2).	S6-S7
Figure S3. LC–MS Spectrum of the isolated α -D-glucopyranose-rich fraction (Batch 3).	S8-S9
Figure S4. ¹ H-NMR Spectrum (DMSO- <i>d</i> ₆) of the isolated α -D-glucopyranose-rich fraction (Batch 1).	S10
Figure S5. Expanded ¹ H NMR Spectrum (DMSO- <i>d</i> ₆) of the isolated α -D-glucopyranose-rich fraction (Batch 1).	S11
Figure S6. ¹³ C NMR Spectrum (DMSO- <i>d</i> ₆) of the isolated α -D-glucopyranose-rich fraction (Batch 1).	S12

Figure S7. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 1).	S13
Figure S8. ^1H -NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 2).	S14
Figure S9. Expanded ^1H NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 2).	S15
Figure S10. ^{13}C NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 2).	S16
Figure S11. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 2).	S17
Figure S12. ^1H -NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 3).	S18
Figure S13. Expanded ^1H NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 3).	S19
Figure S14. ^{13}C NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 3).	S20
Figure S15. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 3).	S21
Figure S16. ^1H -NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).	S22
Figure S17. Expanded ^1H NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).	S23
Figure S18. ^{13}C NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).	S24

Figure S19. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).	S25
Figure S20. ^1H -NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).	S26
Figure S21. Expanded ^1H NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).	S27
Figure S22. ^{13}C NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).	S28
Figure S23. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).	S29
Figure S24. ^1H -NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).	S30
Figure S25. Expanded ^1H NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).	S31
Figure S26. ^{13}C NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).	S32
Figure S27. DEPT-135 Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0)	S33
Figure S28. ^1H -NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).	S34
Figure S29. Expanded ^1H NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).	S35
Figure S30. ^{13}C NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).	S36

Figure S31. DEPT-135 Spectrum (D ₂ O) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t ₂₄).	S37
Table S1. Optical rotation measurements of the isolated α -D-glucopyranose-rich fraction recorded over time during mutarotation in water at 20 °C.	S38
Figure S32. Representative polarimeter display images recorded immediately after dissolution (0 h), (1 h 50 min), (3 h) and after 18 h to monitor changes in optical rotation during mutarotation under the experimental conditions described in the main manuscript.	S39
Table S2. Reproducibility of the isolated α -D-glucopyranose-rich fraction across independent experimental batches.	S40

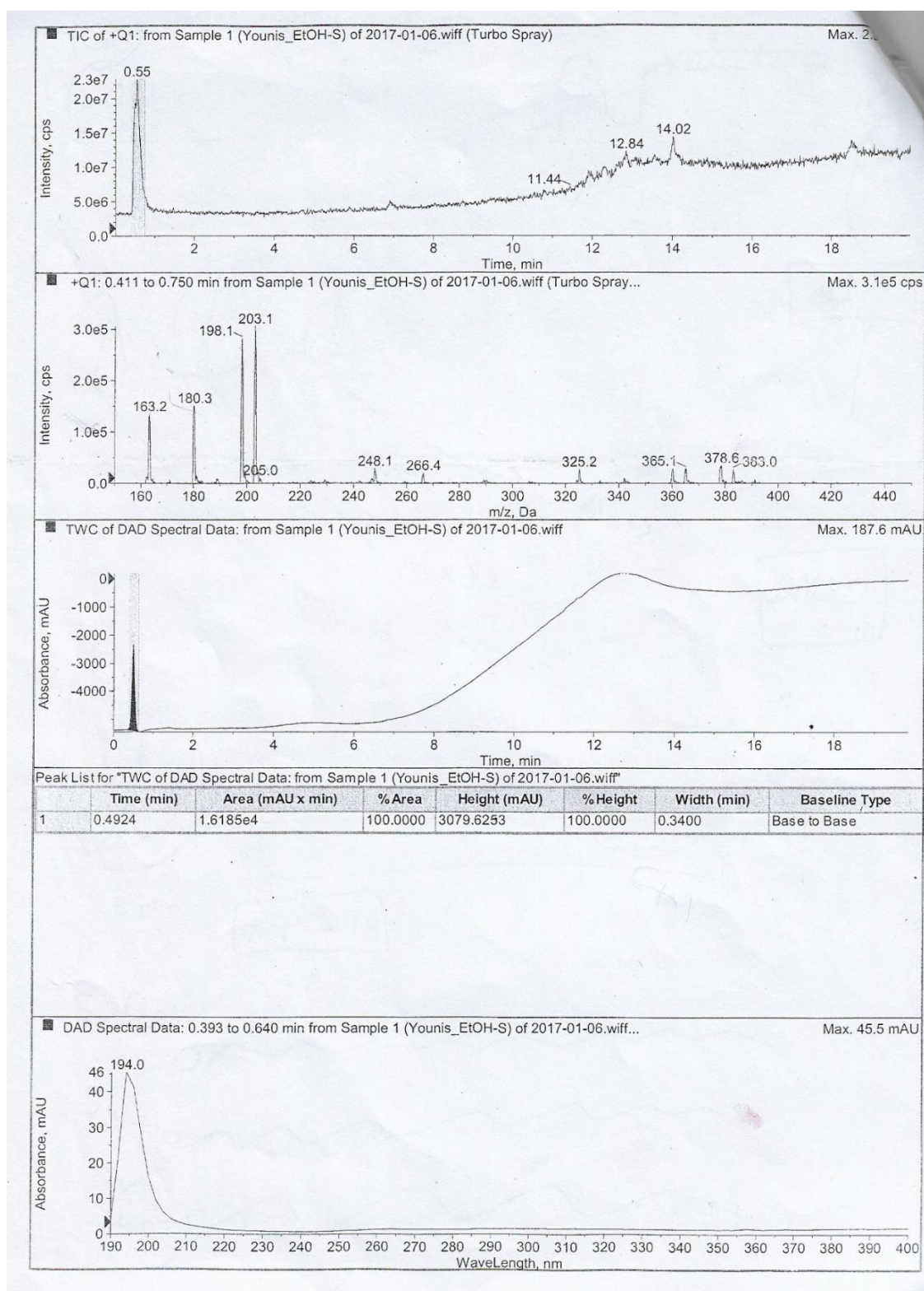
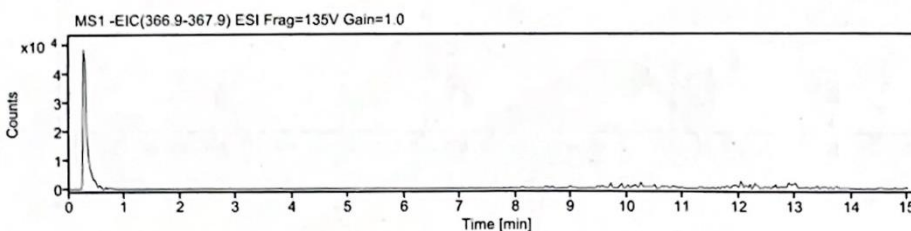
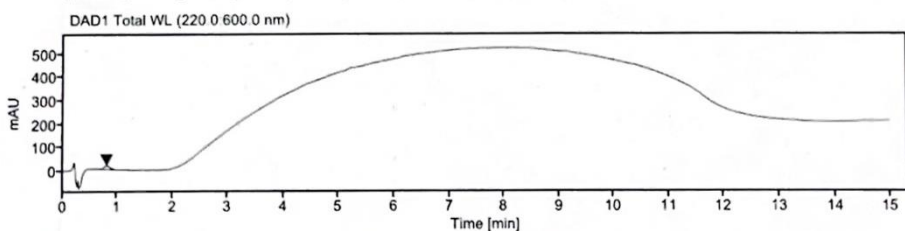
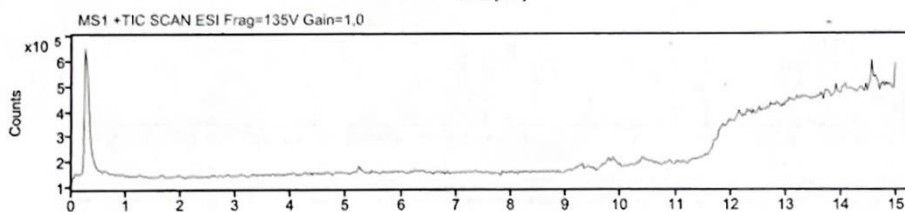
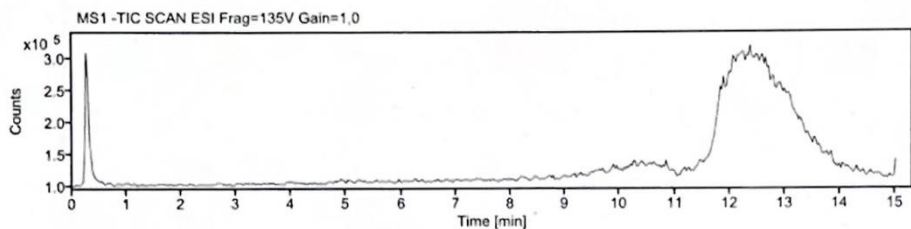


Figure S1. LC–MS Spectrum of the isolated α -D-glucopyranose-rich fraction (**Batch 1**).

Sequence Name: SingleSample
Data file: EtOH-WS.dx
Sample name: EtOH-WS
Instrument: Single Quad
Inj. volume: 2.000
Acq. method: 00-new_wasser-acn_standard_100-1500_BD-20min.amx
Processing method: MS_standard_plot.pmx
Project Name: Single Quad
Operator: SYSTEM
Acquired on: 2022-02-14 15:04:48+01:00
Location: P1-D11



Signal: DAD1 Total WL (220.0:600.0 nm)

RT [min]	Peak MS Base Peak m/z	Area	Area%	Max Peak%	Height
0.807		115.2264	100.0000	100.000	14.697
Sum		115.2264			

Signal: MS1 +TIC SCAN ESI Frag=135V Gain=1.0

RT [min]	Peak MS Base Peak m/z	Area	Area%	Max Peak%	Height
0.270	198.200	3107223.3643	100.0000	100.000	534342.906
Sum		3107223.3643			

Signal: MS1 -TIC SCAN ESI Frag=135V Gain=1,0

RT [min]	Peak MS Base Peak m/z	Area	Area%	Max Peak%	Height
0.281	367.200	989414.6288	100.0000	100.000	211132.496
Sum		989414.6288			

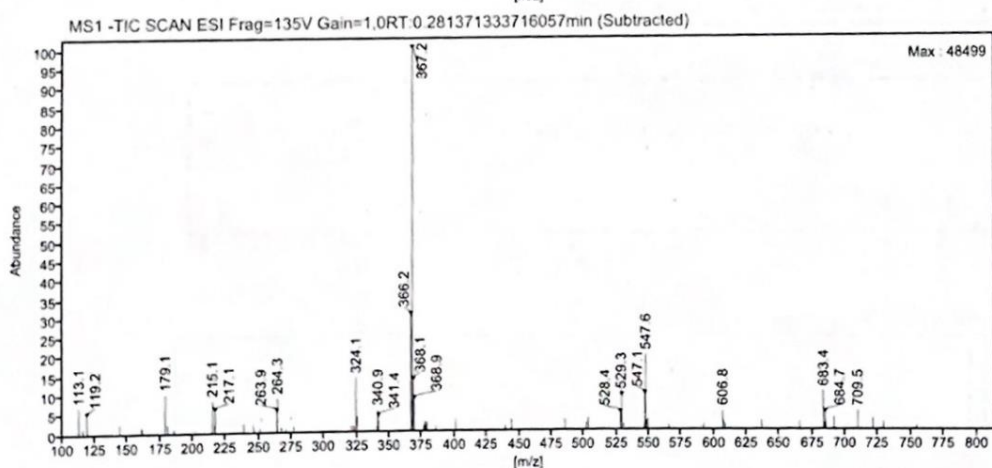
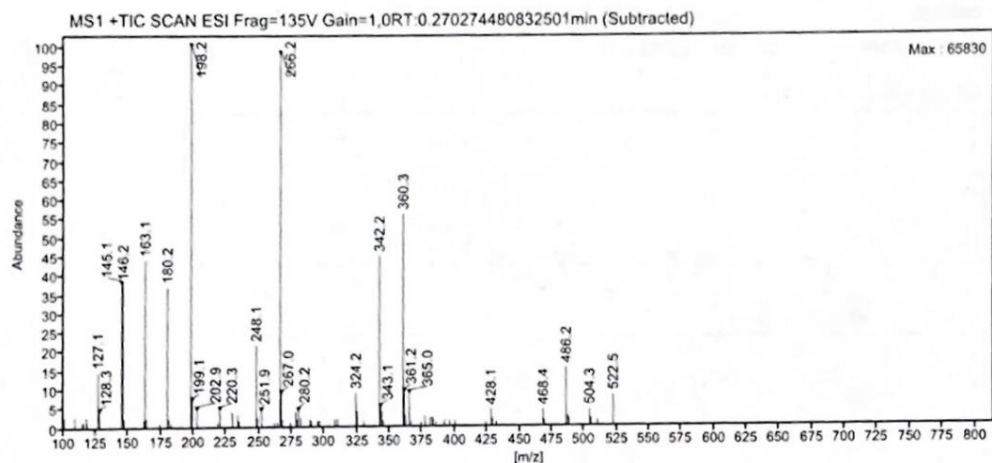
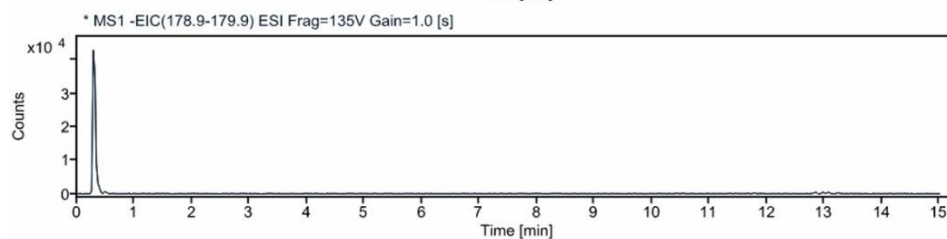
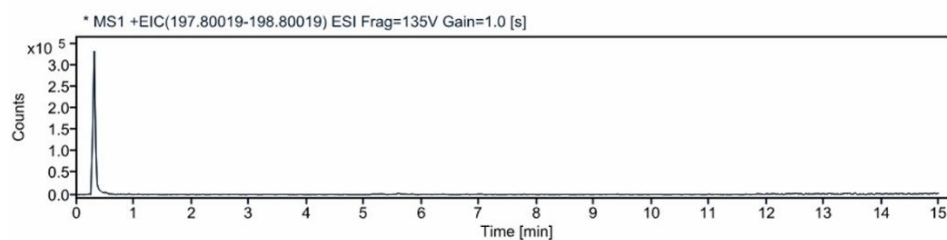
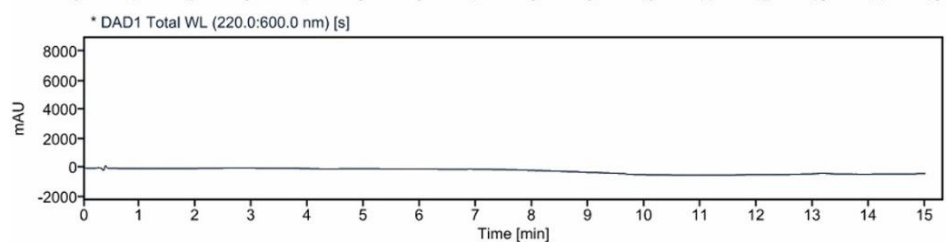
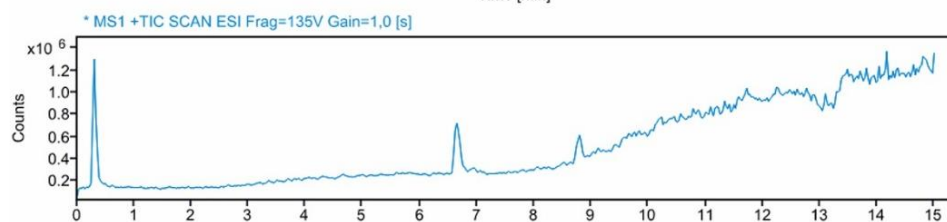
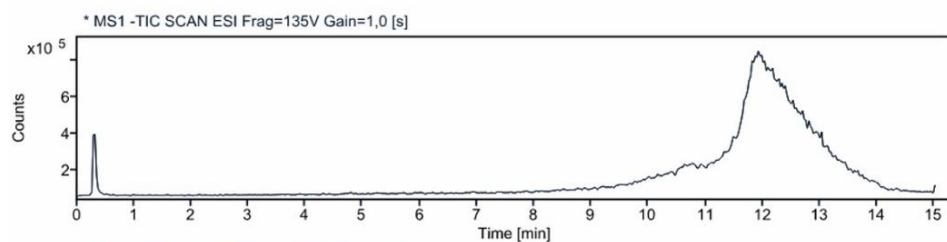


Figure S2. LC–MS Spectrum of the isolated α -D-glucopyranose-rich fraction (**Batch 2**).

Sequence Name:	SingleSample	Project Name:	Single Quad
Data file:	EtOH-s.dx	Operator :	SYSTEM
Sample name:	EtOH-s	Acquired on:	2025-09-10 12:48:15+02:00
Instrument:	Single Quad	Location:	P2-E9
Inj. volume:	2.000		
Acq. method:	00-new_wasser-acn_standard_100-1000_BD-20min.amx		
Processing method:	MS_standard_plot.pmx		



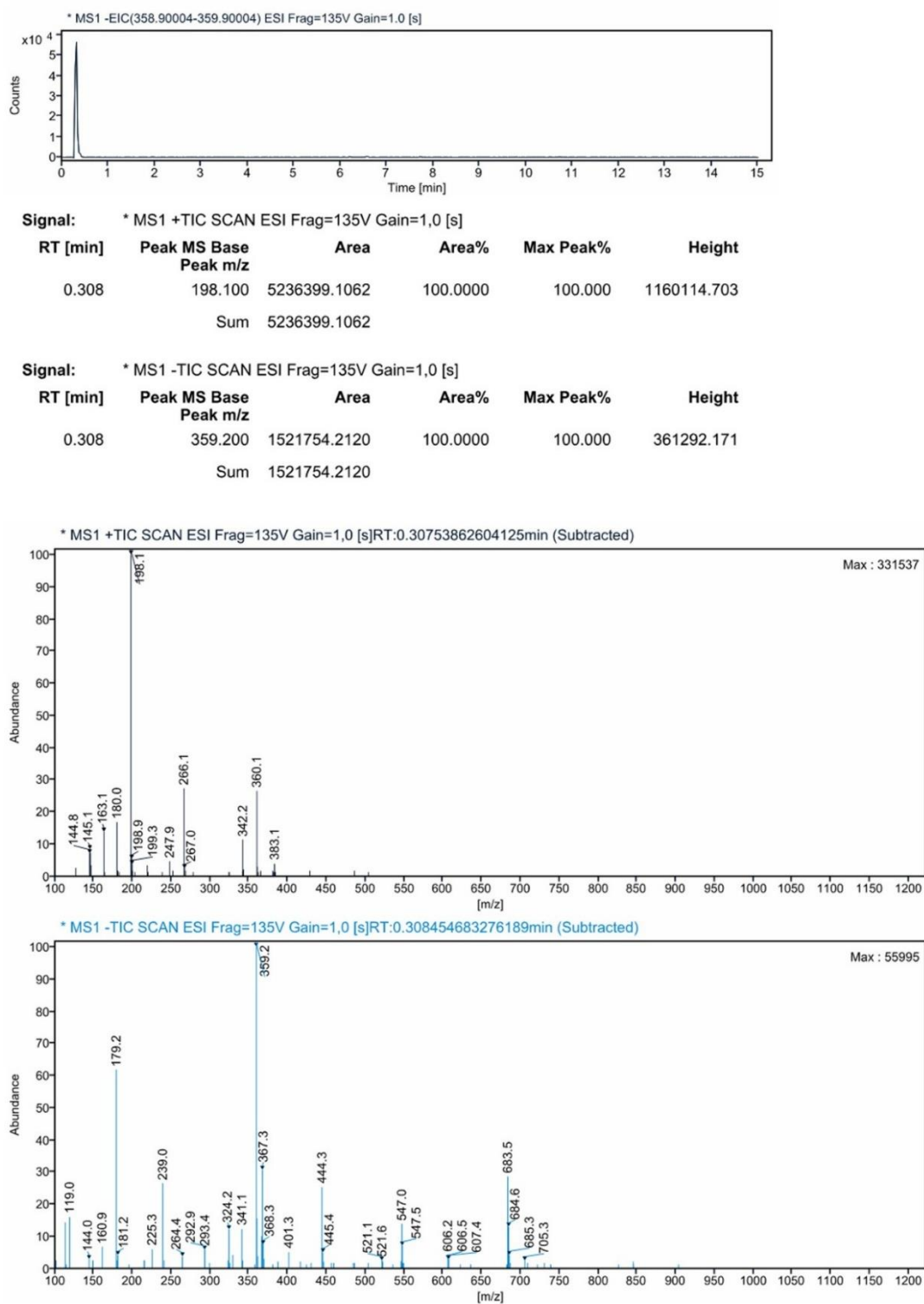


Figure S3. LC–MS Spectrum of the isolated α -D-glucopyranose-rich fraction (**Batch 3**).

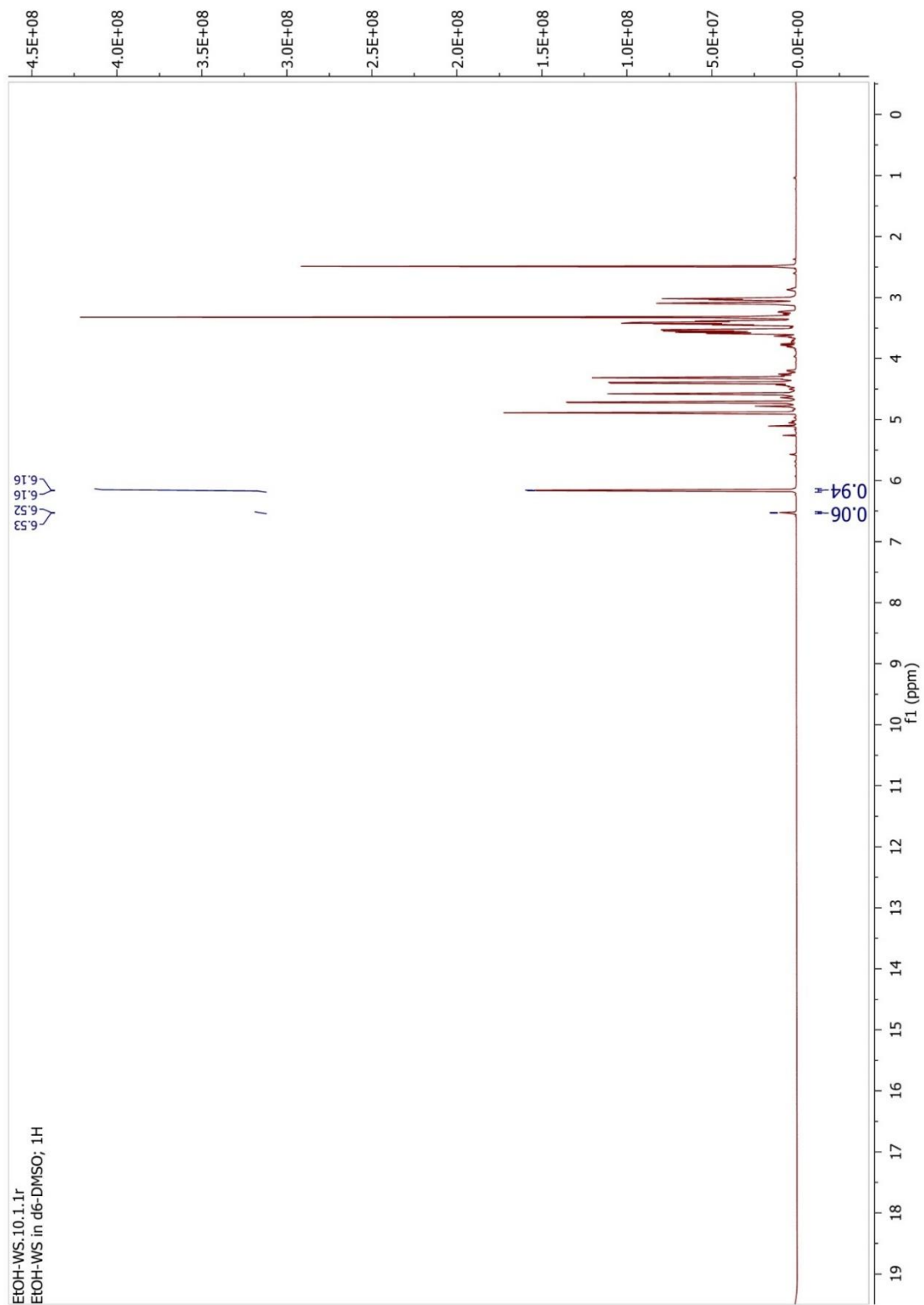


Figure S4. ^1H -NMR Spectrum ($\text{DMSO}-d_6$) of the isolated α -D-glucopyranose-rich fraction (**Batch 1**).

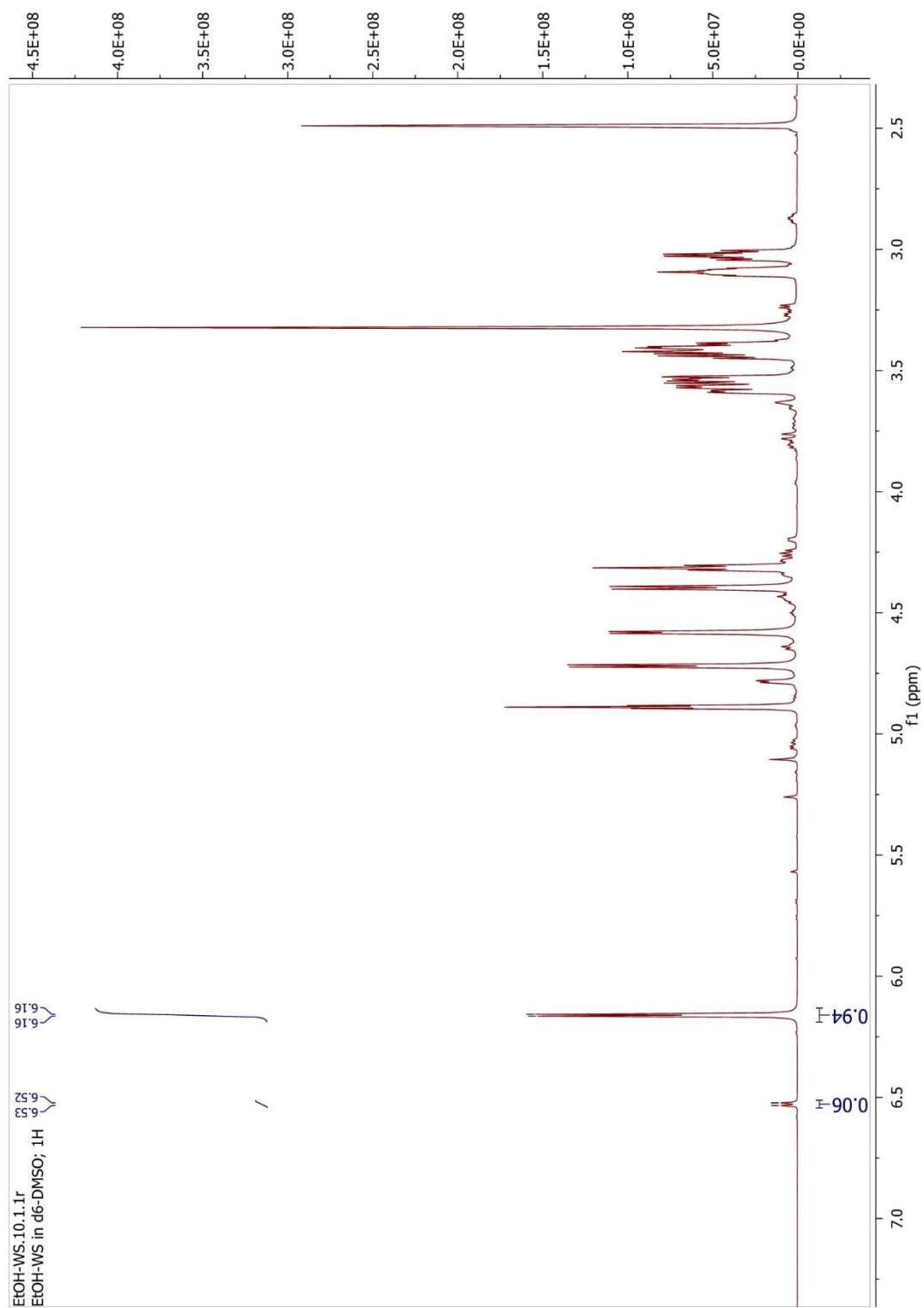


Figure S5. Expanded ^1H NMR Spectrum ($\text{DMSO}-d_6$) of the isolated α -D-glucopyranose-rich fraction (Batch 1).

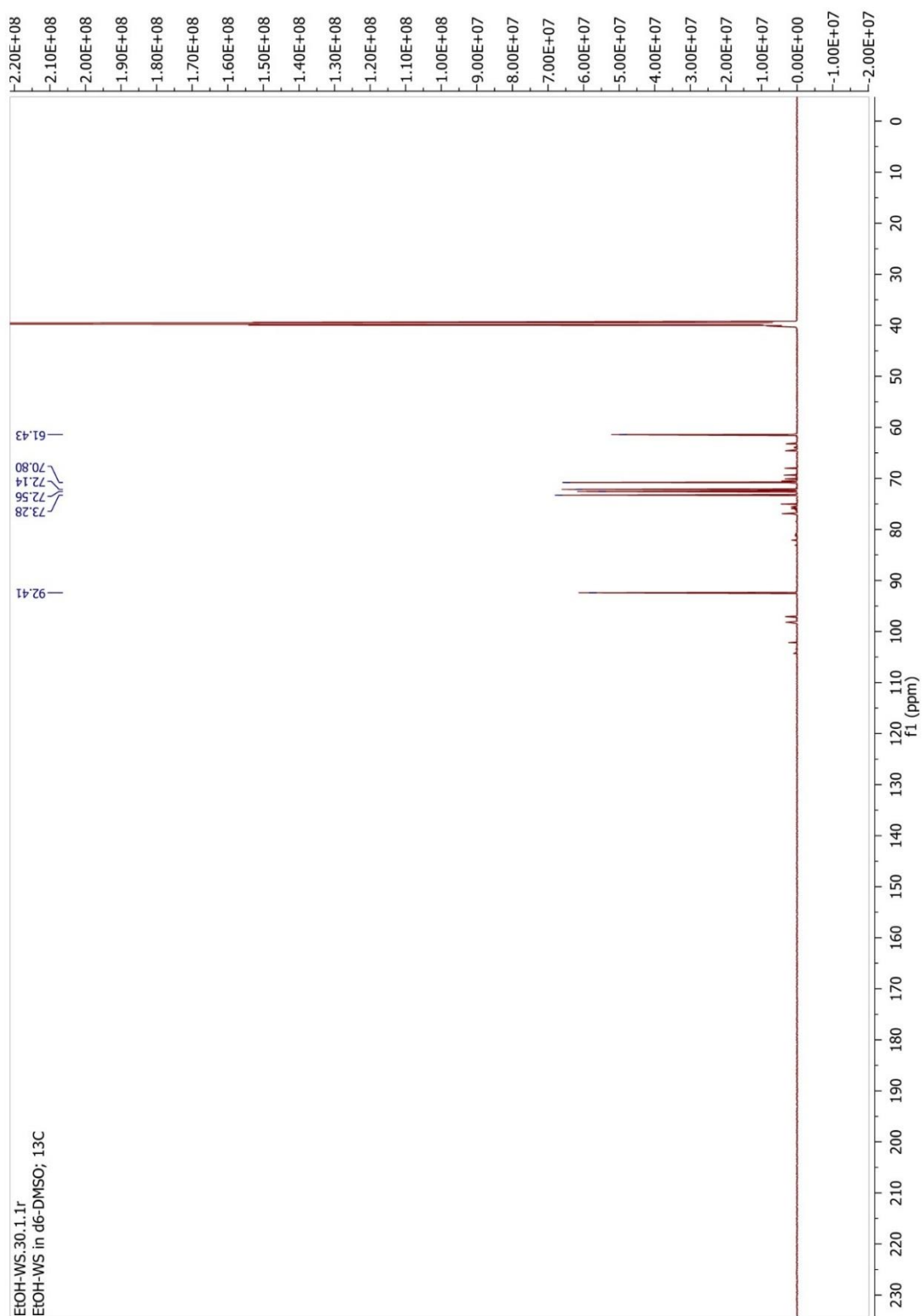


Figure S6. ^{13}C NMR Spectrum (DMSO-d_6) of the isolated α -D-glucopyranose-rich fraction (**Batch 1**).

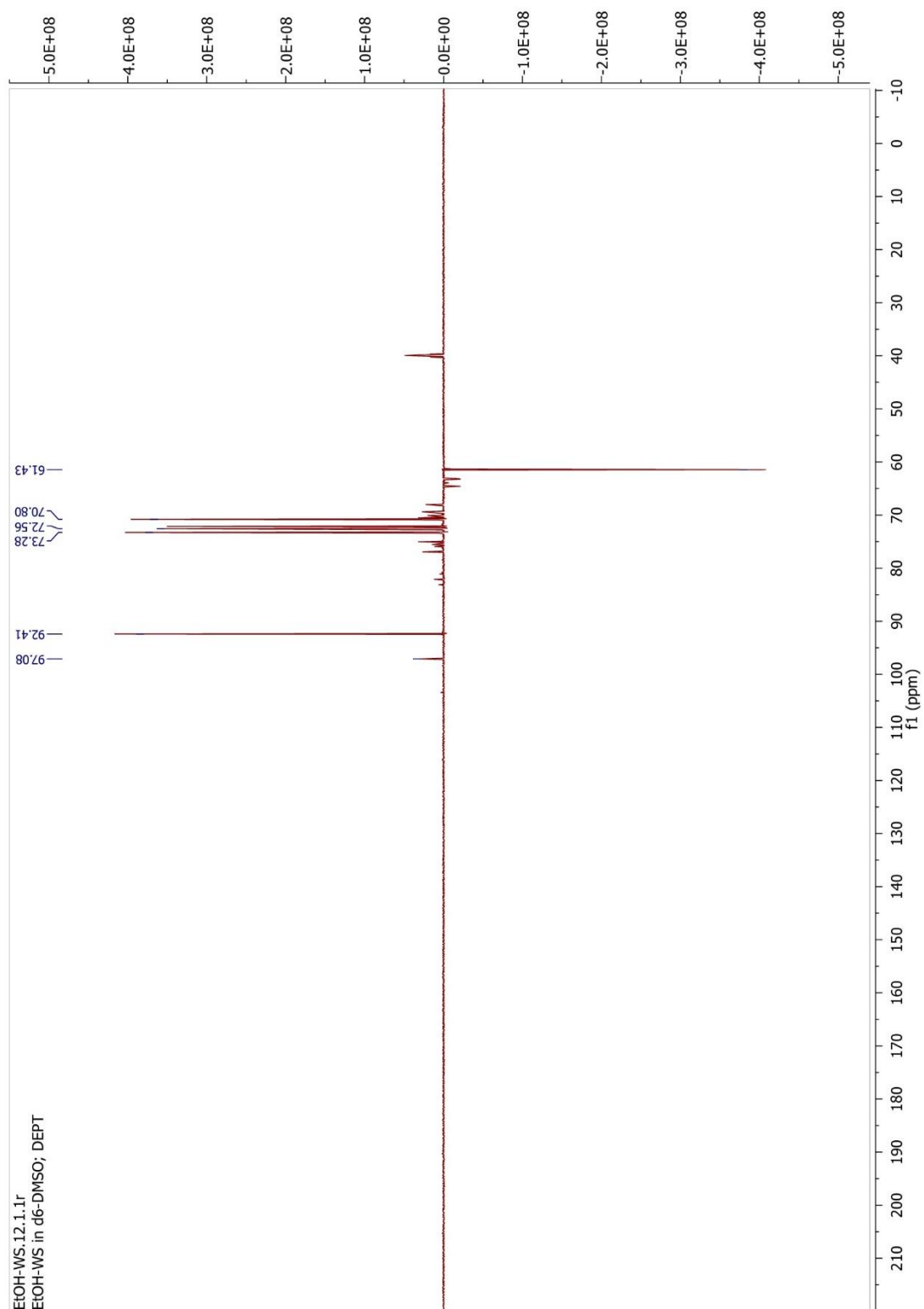


Figure S7. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (**Batch 1**).

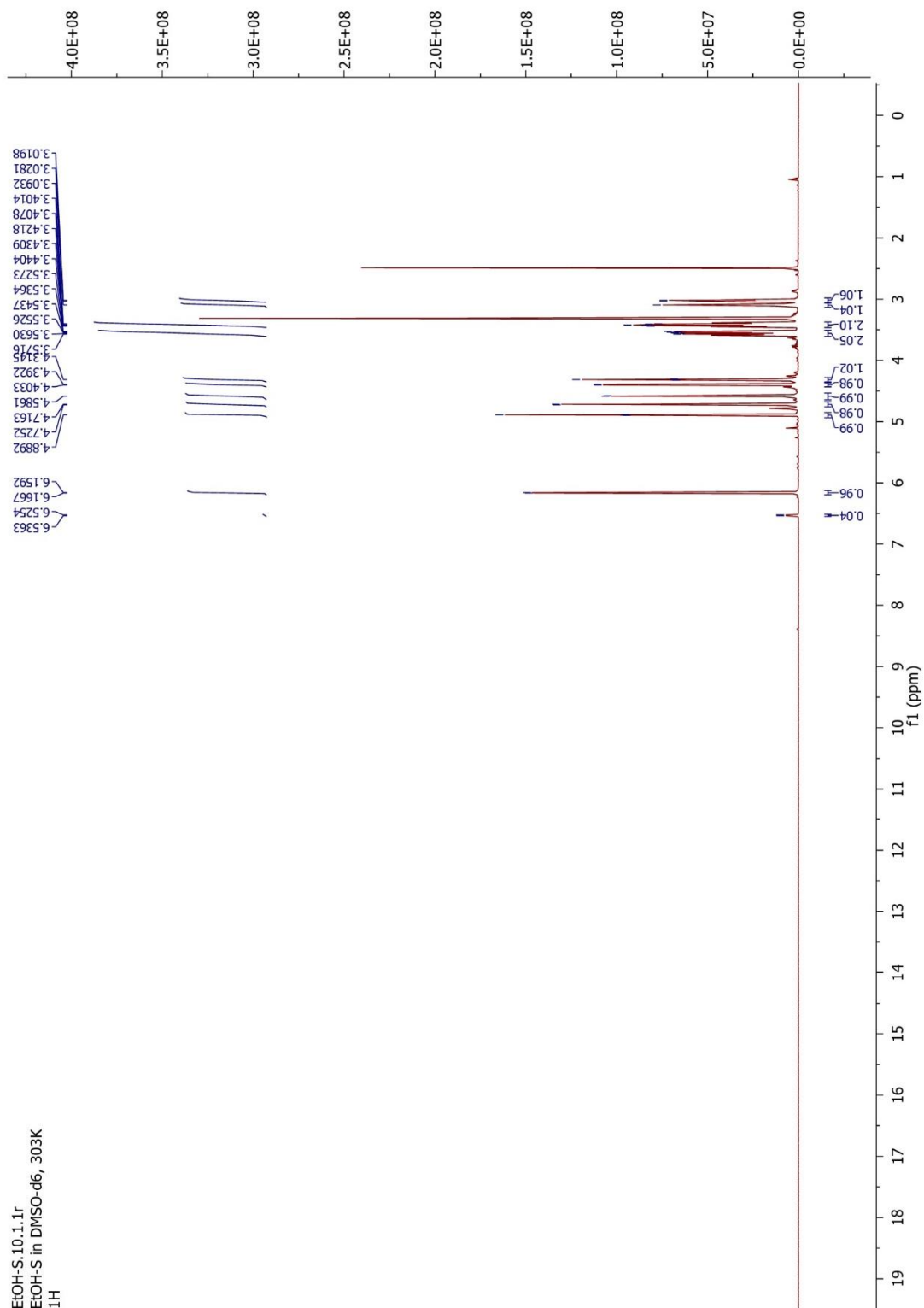


Figure S8. ¹H-NMR Spectrum (DMSO-d₆) of the isolated α -D-glucopyranose-rich fraction (**Batch 2**).

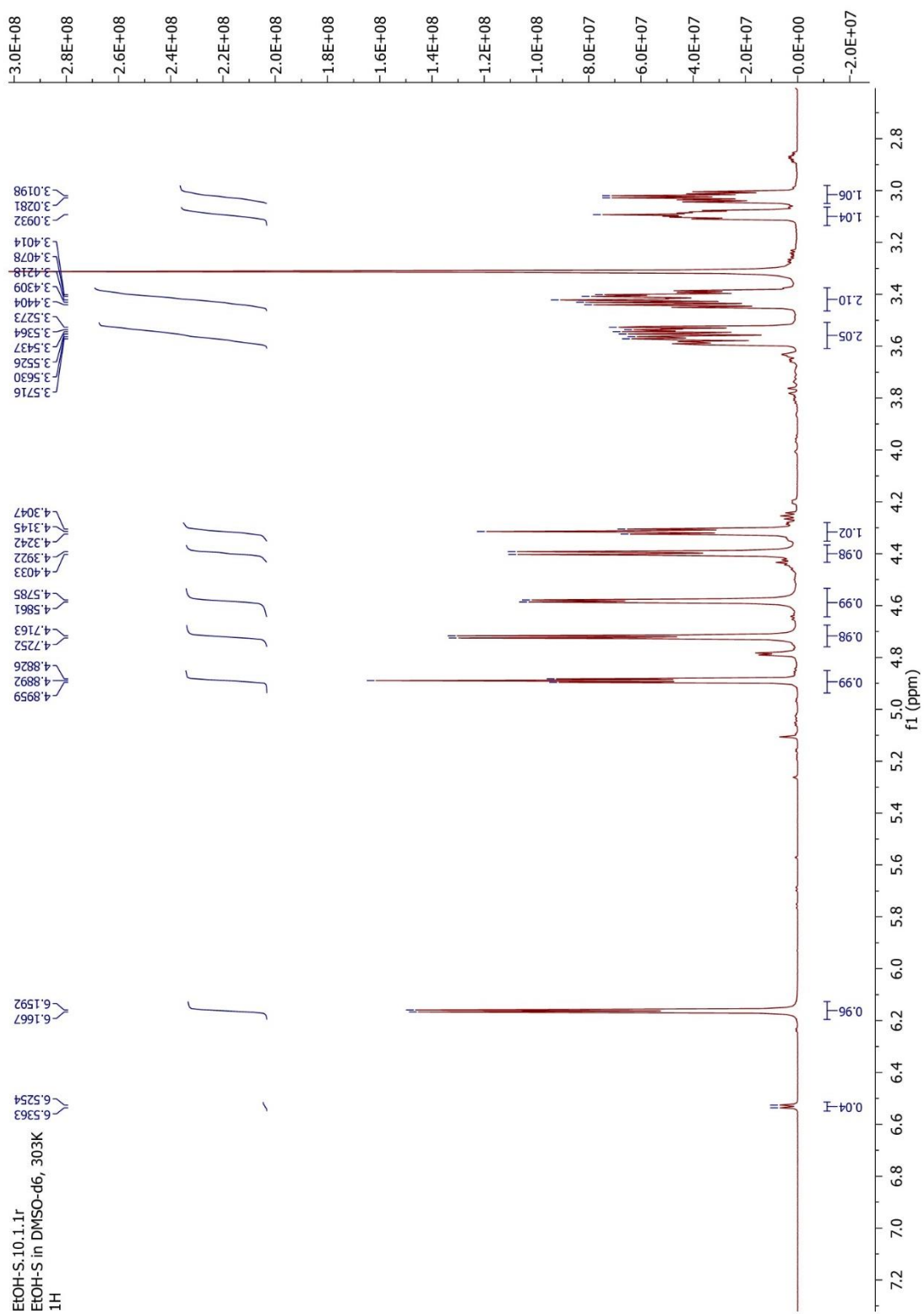


Figure S9. Expanded ¹H NMR Spectrum (DMSO-*d*₆) of the isolated α-D-glucopyranose-rich fraction (Batch 2).

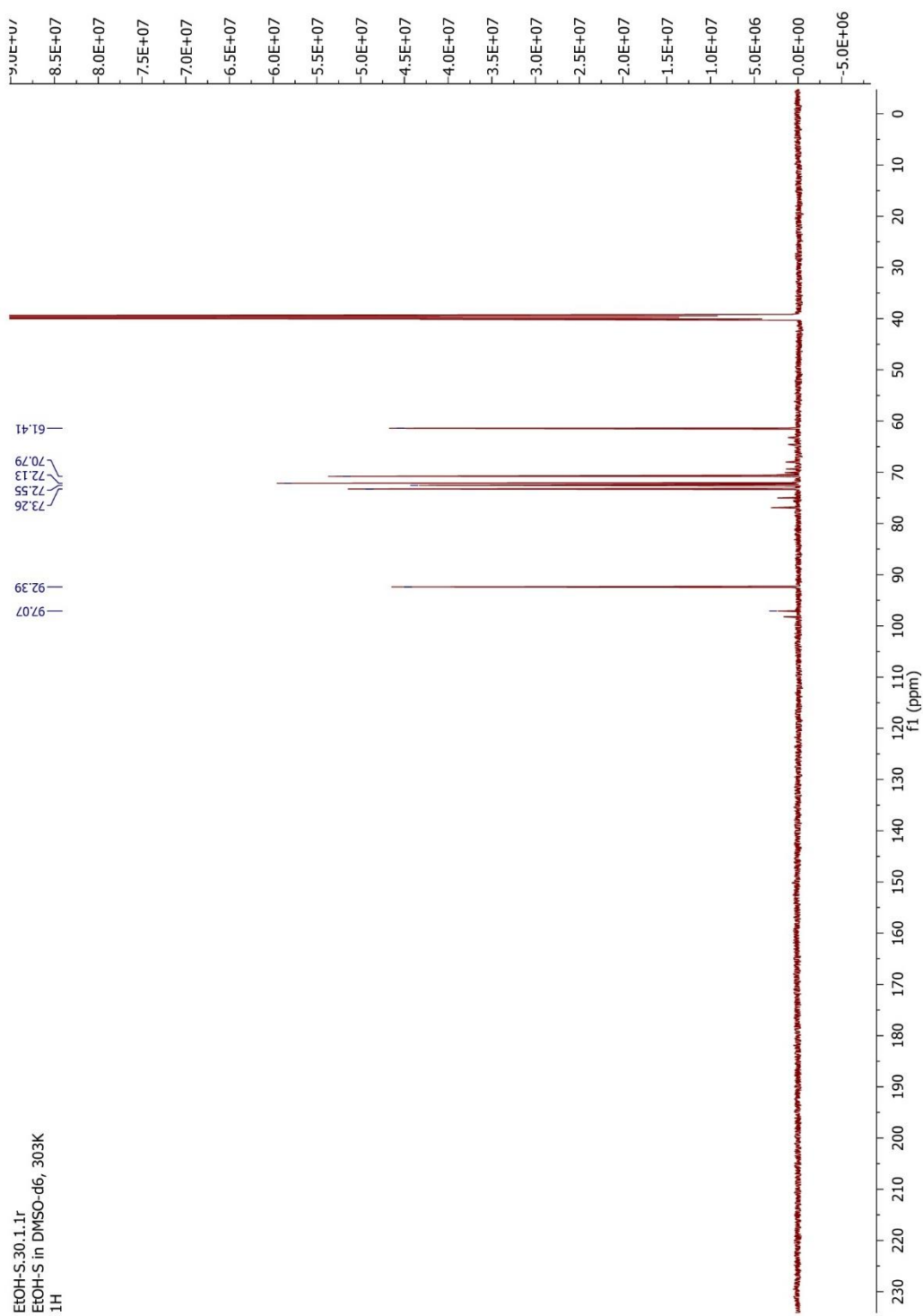


Figure S10. ¹³C NMR Spectrum (DMSO-*d*₆) of the isolated α -D-glucopyranose-rich fraction (**Batch 2**).

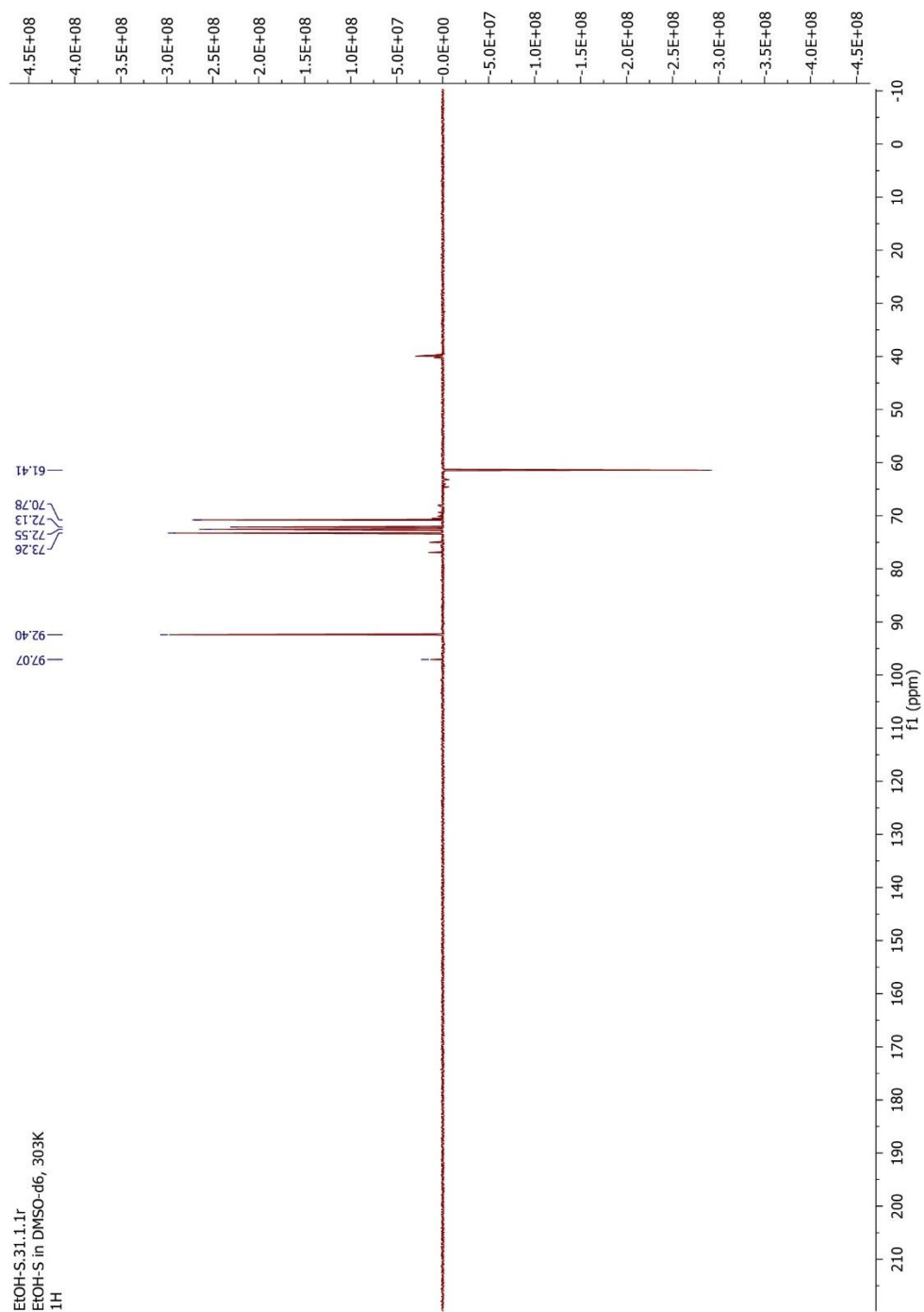


Figure S11. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 2).

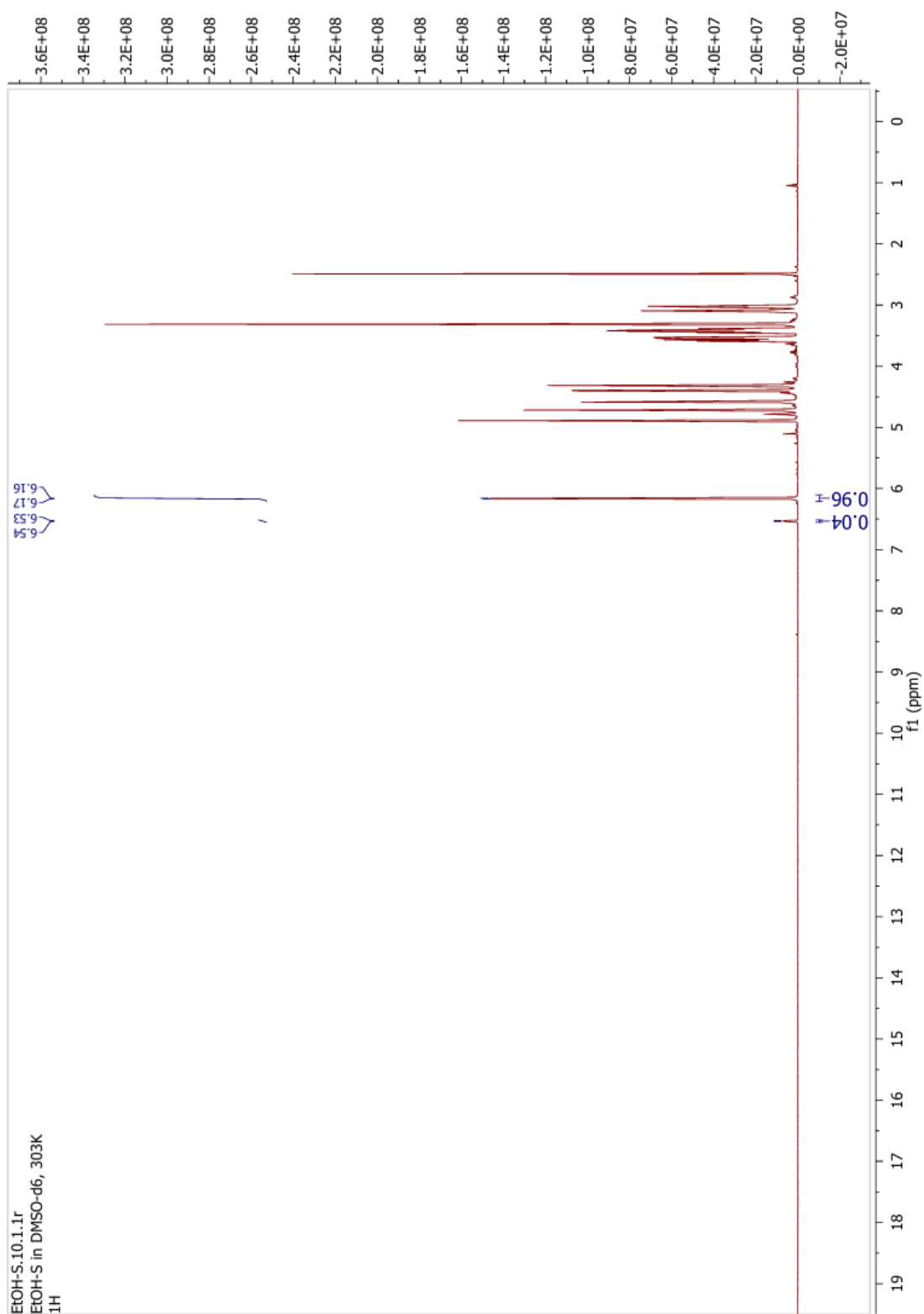


Figure S12. ^1H -NMR Spectrum ($\text{DMSO-}d_6$) of the isolated α -D-glucopyranose-rich fraction (**Batch 3**).

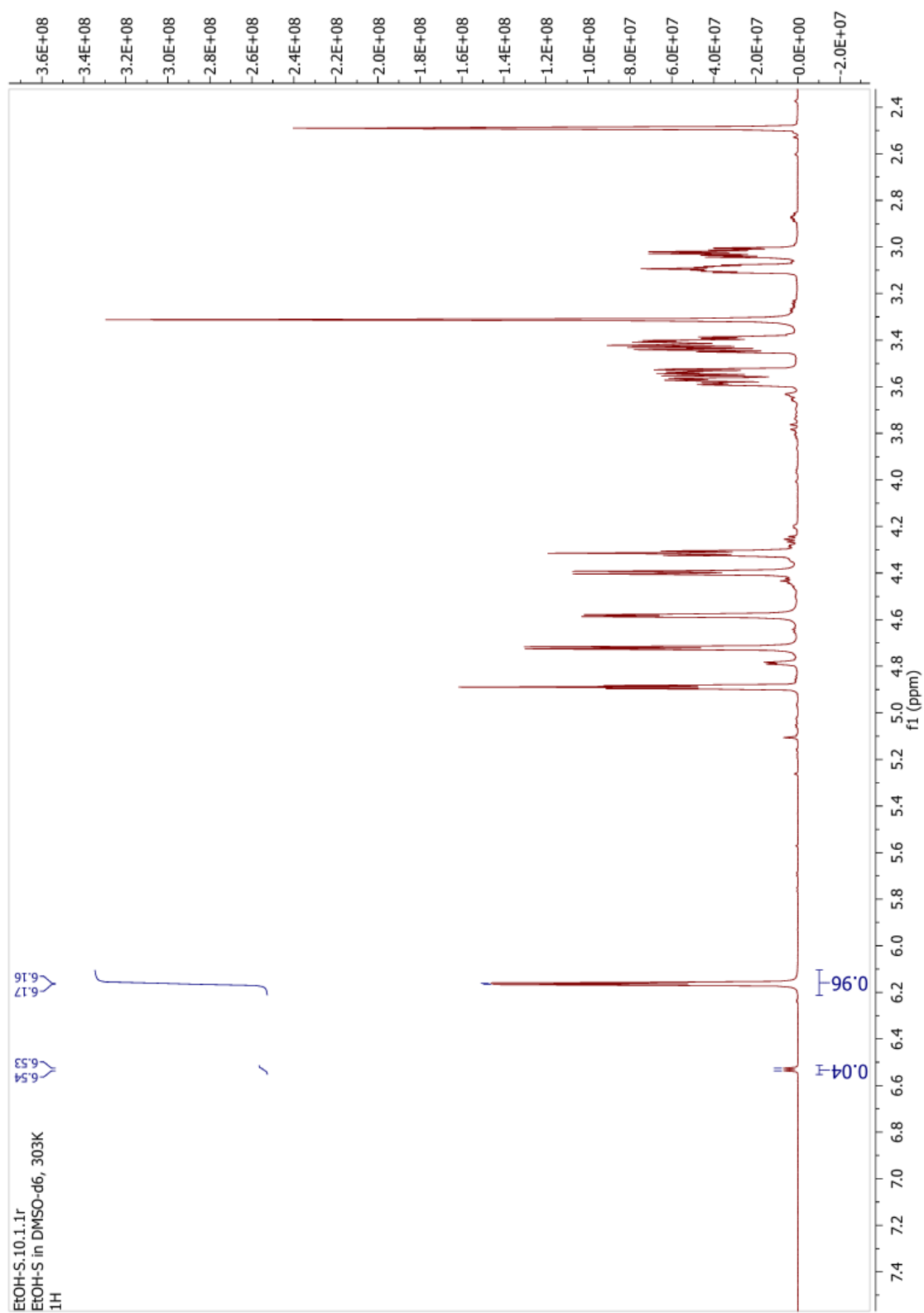


Figure S13. Expanded ¹H NMR Spectrum (DMSO-*d*₆) of the isolated α-D-glucopyranose-rich fraction (**Batch 3**).

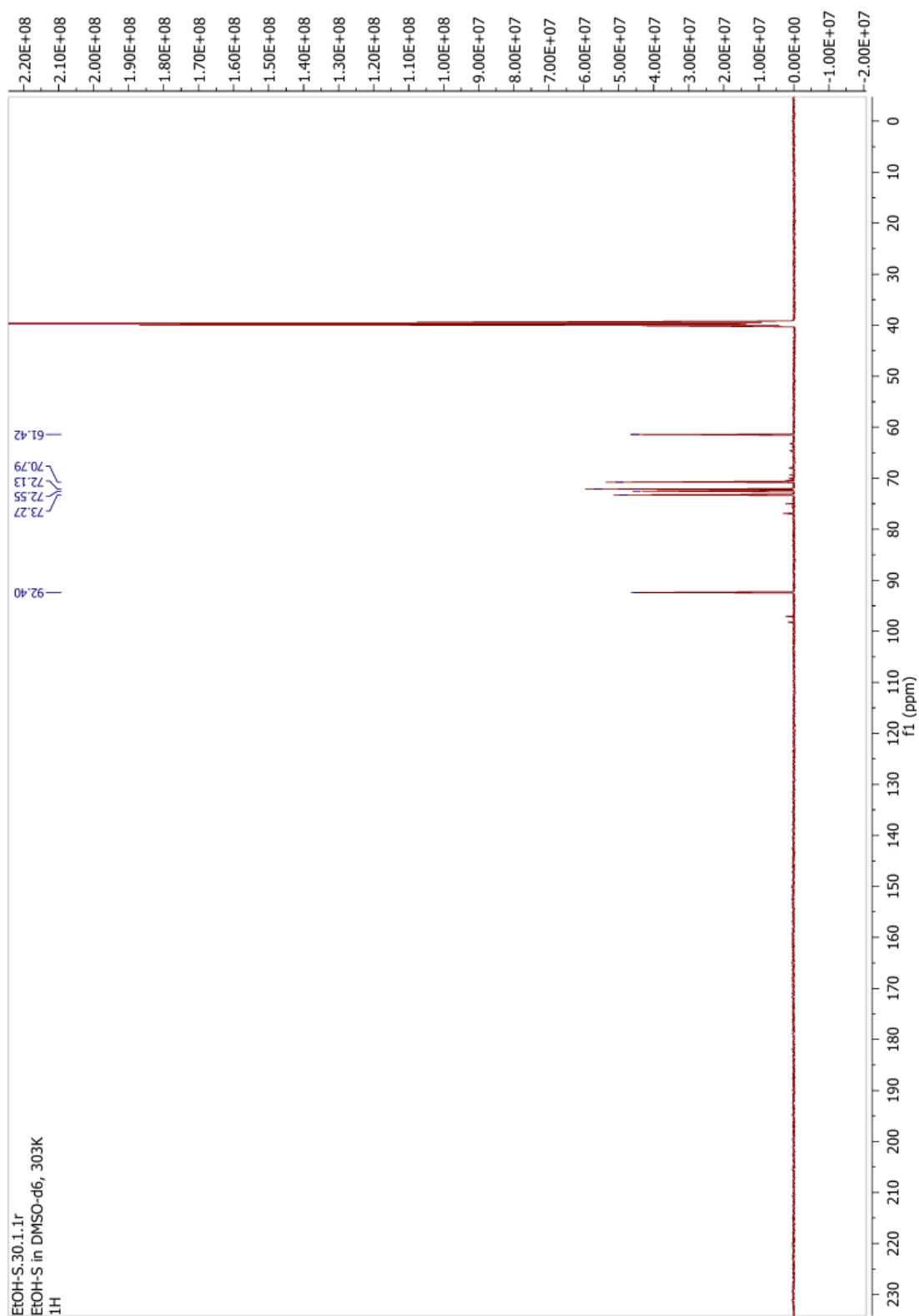


Figure S14. ^{13}C NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (**Batch 3**).

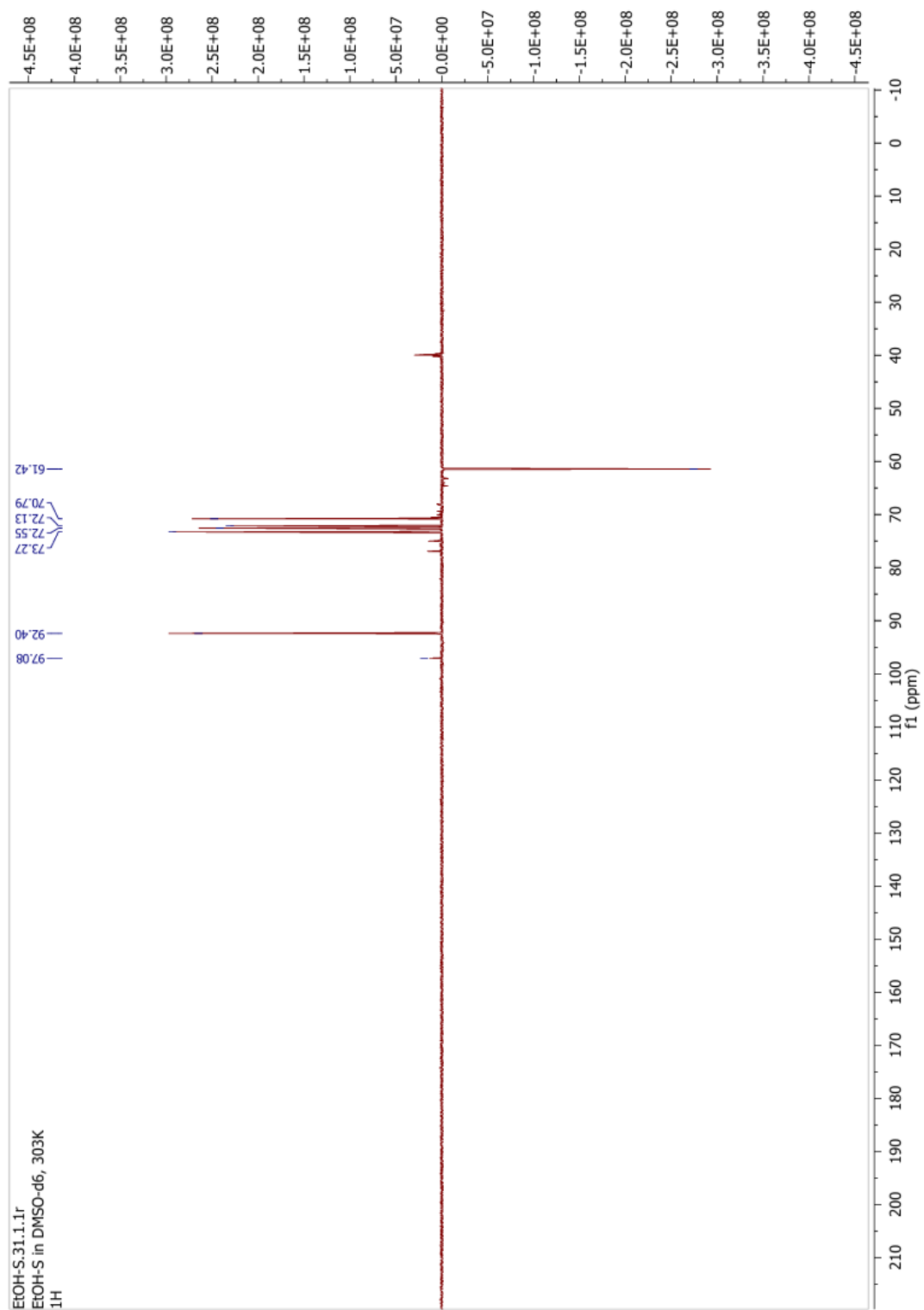


Figure S15. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction (Batch 3).

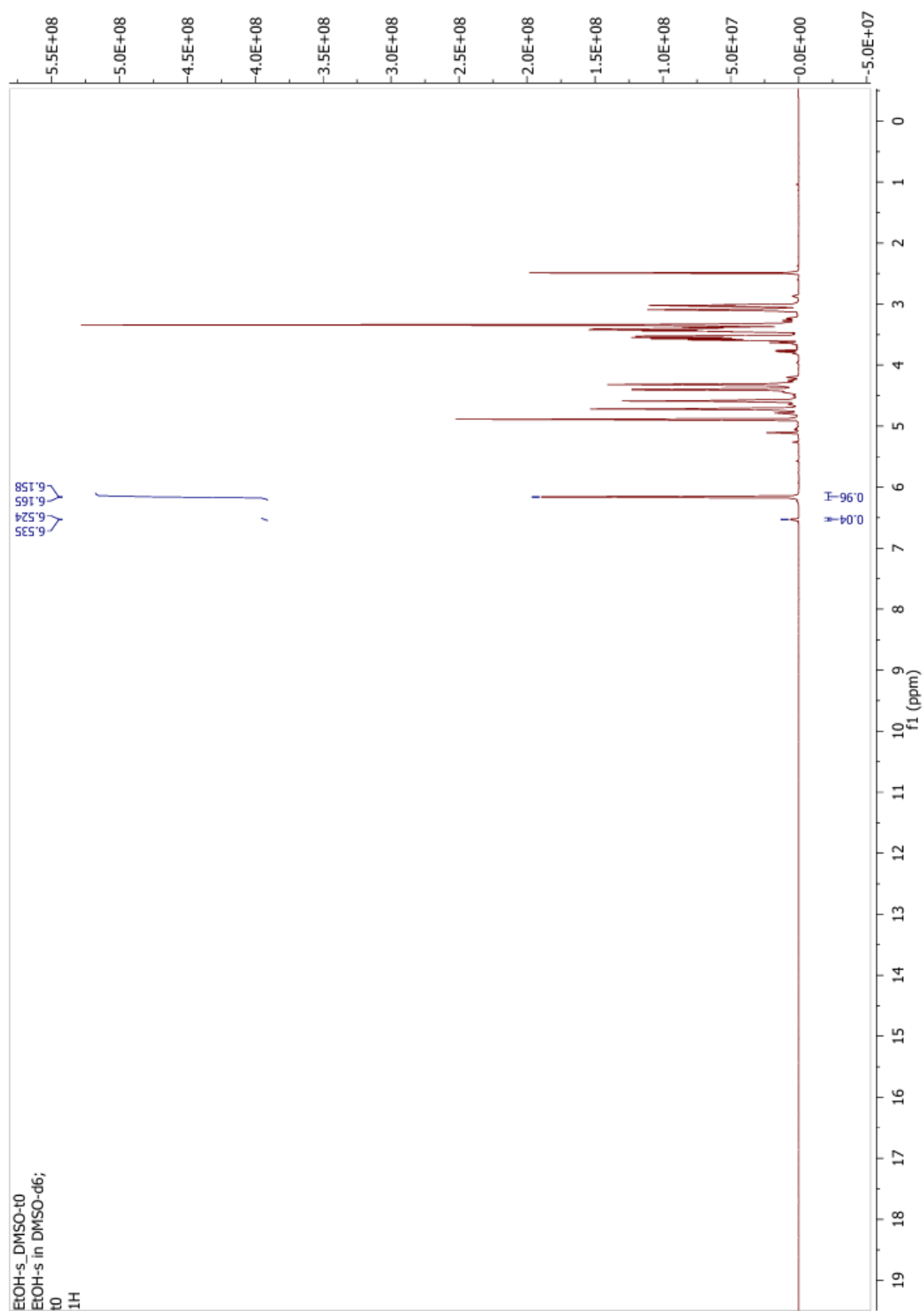


Figure S16. ^1H -NMR Spectrum ($\text{DMSO-}d_6$) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).

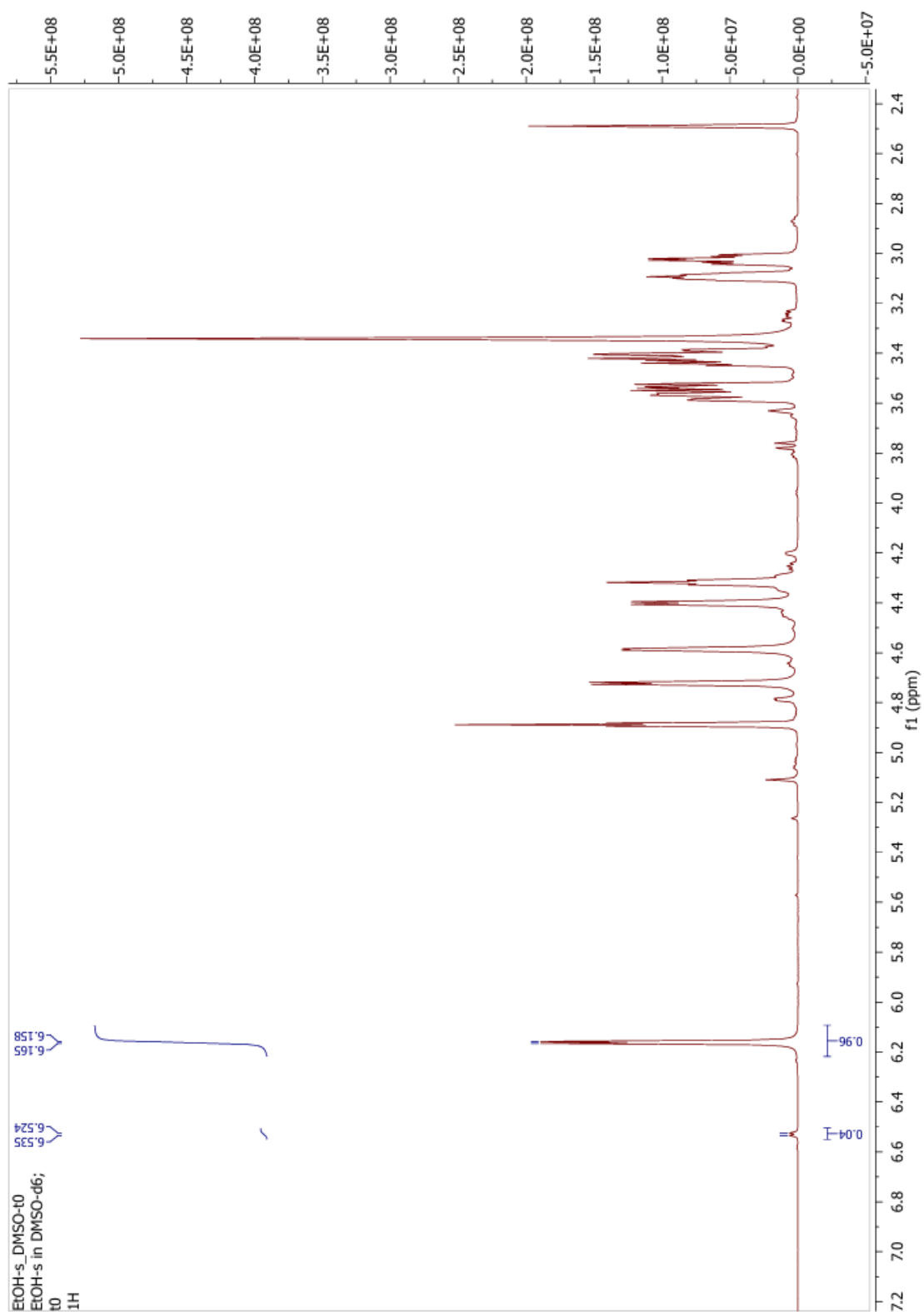


Figure S17. Expanded ^1H NMR Spectrum ($\text{DMSO-}d_6$) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).

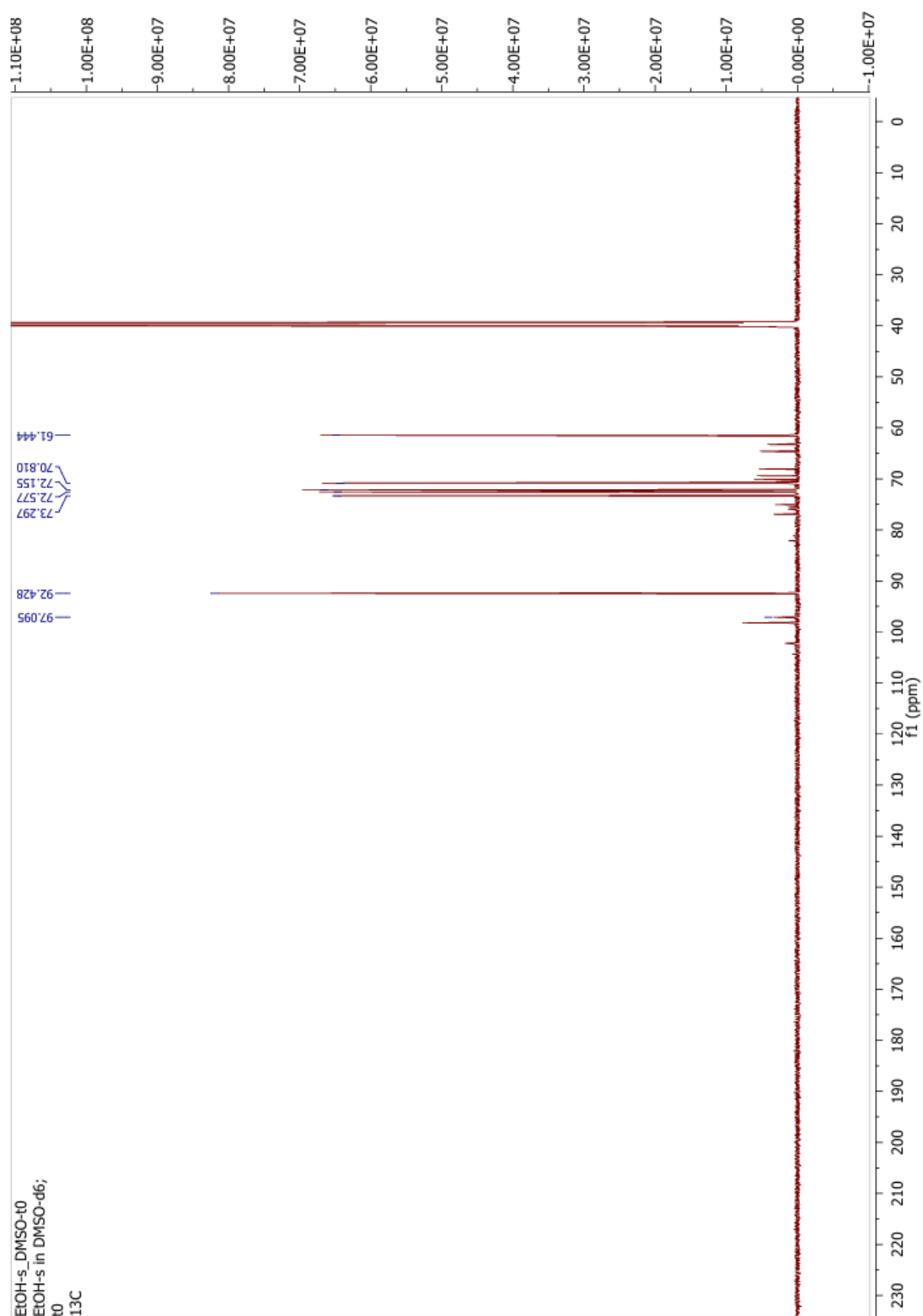


Figure S18. ^{13}C NMR Spectrum ($\text{DMSO-}d_6$) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).

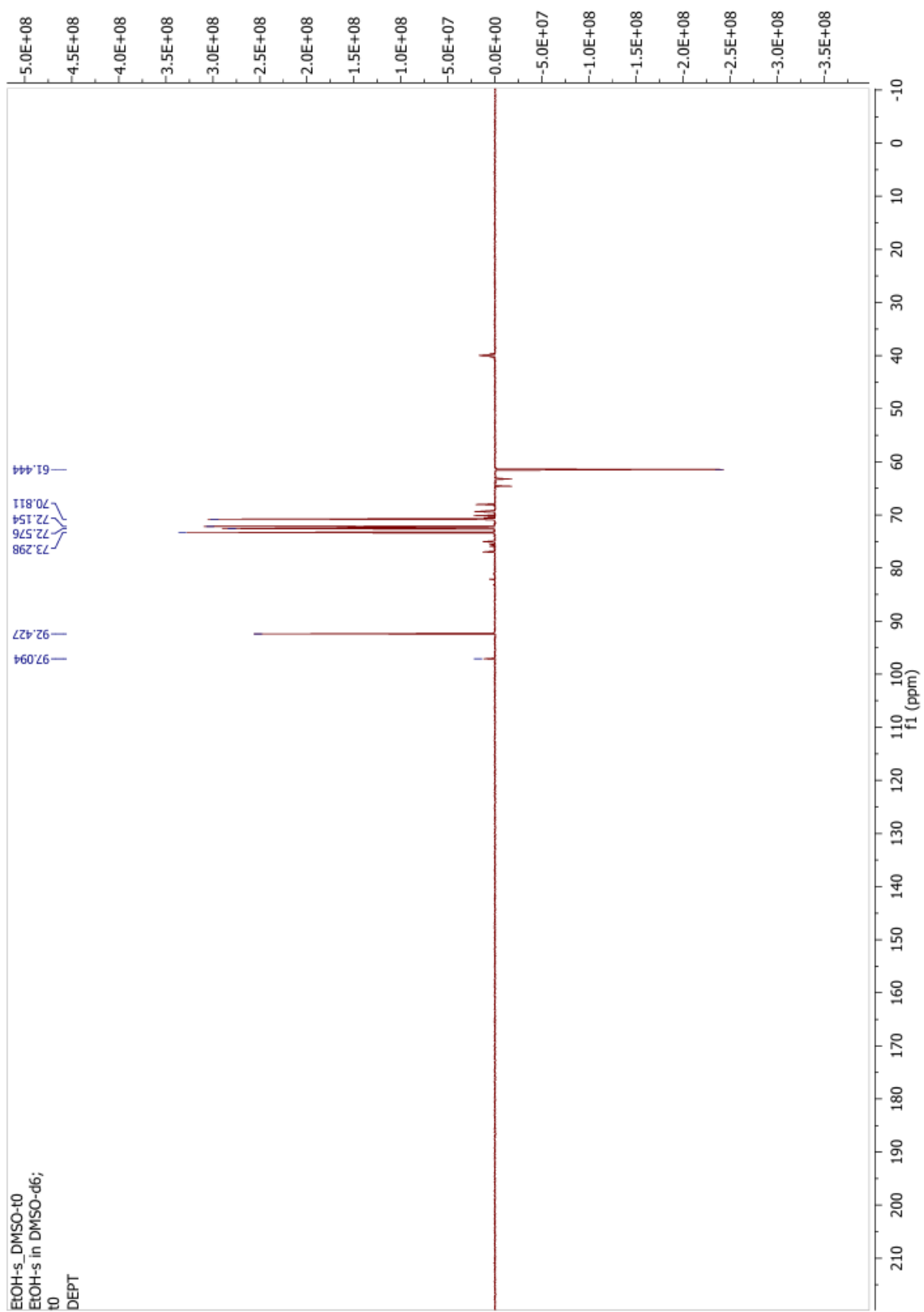


Figure S19. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).

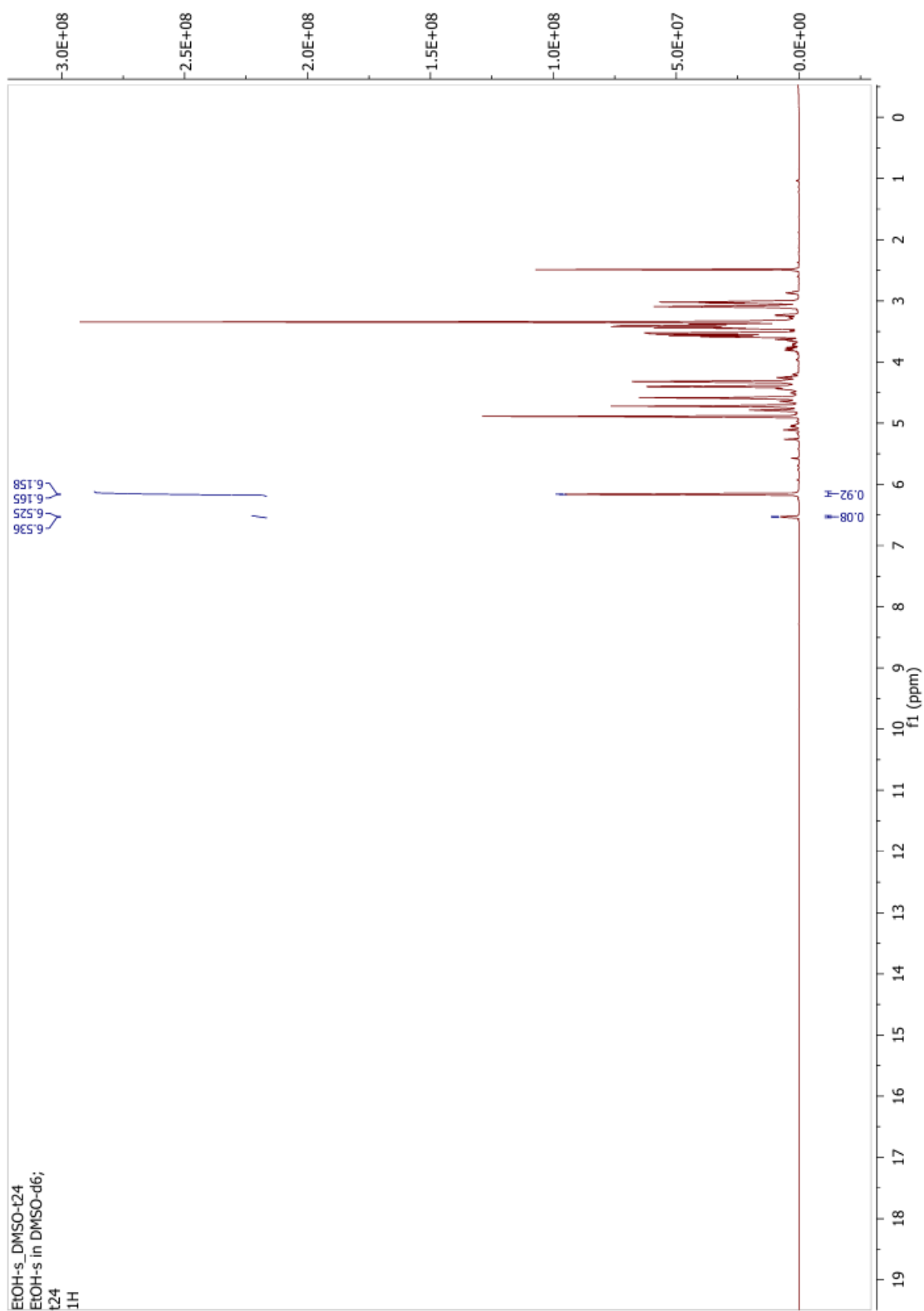


Figure S20. ^1H -NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).

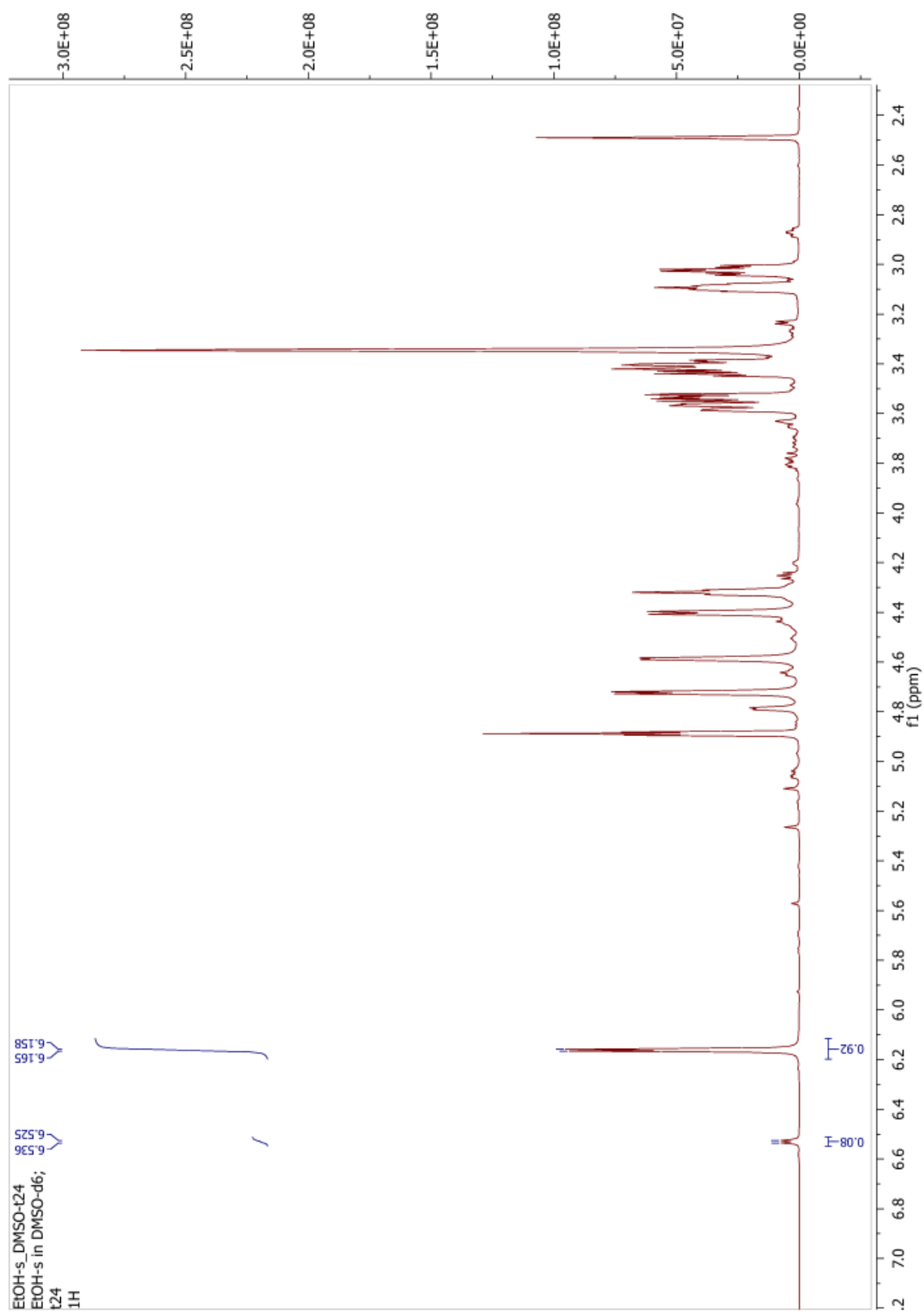


Figure S21. Expanded ^1H NMR Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).

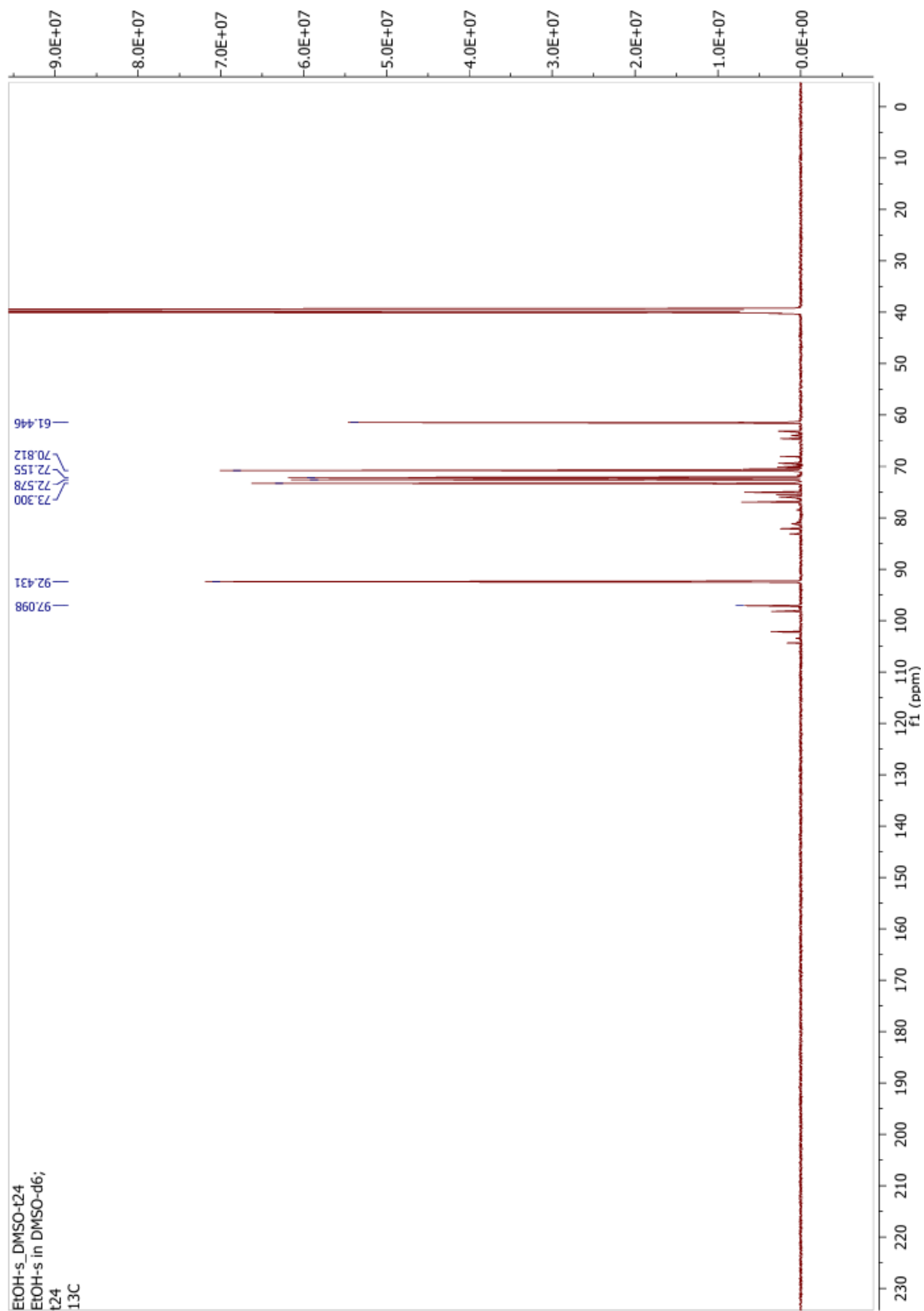


Figure S22. ^{13}C NMR Spectrum ($\text{DMSO-}d_6$) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).

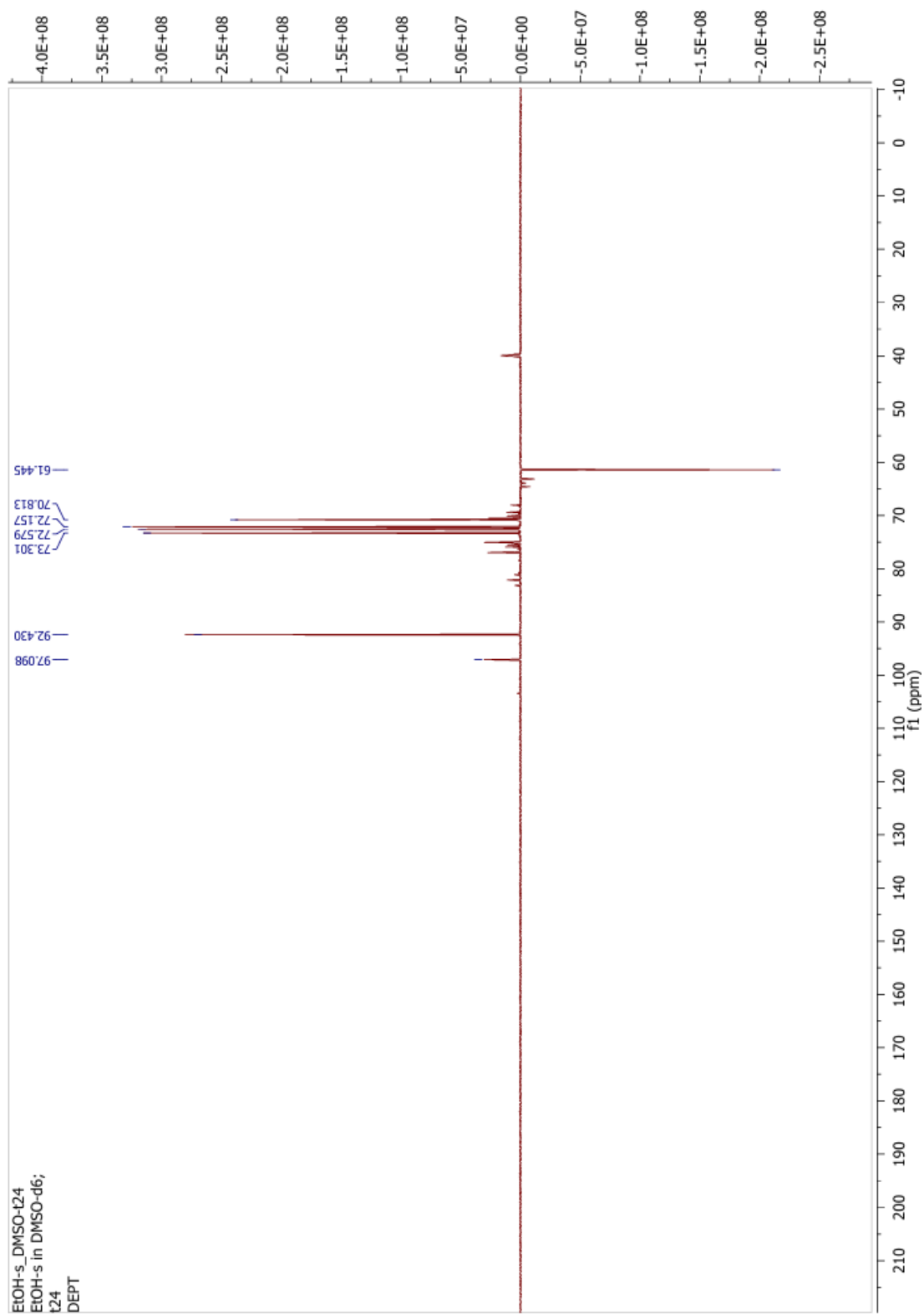


Figure S23. DEPT-135 Spectrum (DMSO- d_6) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).

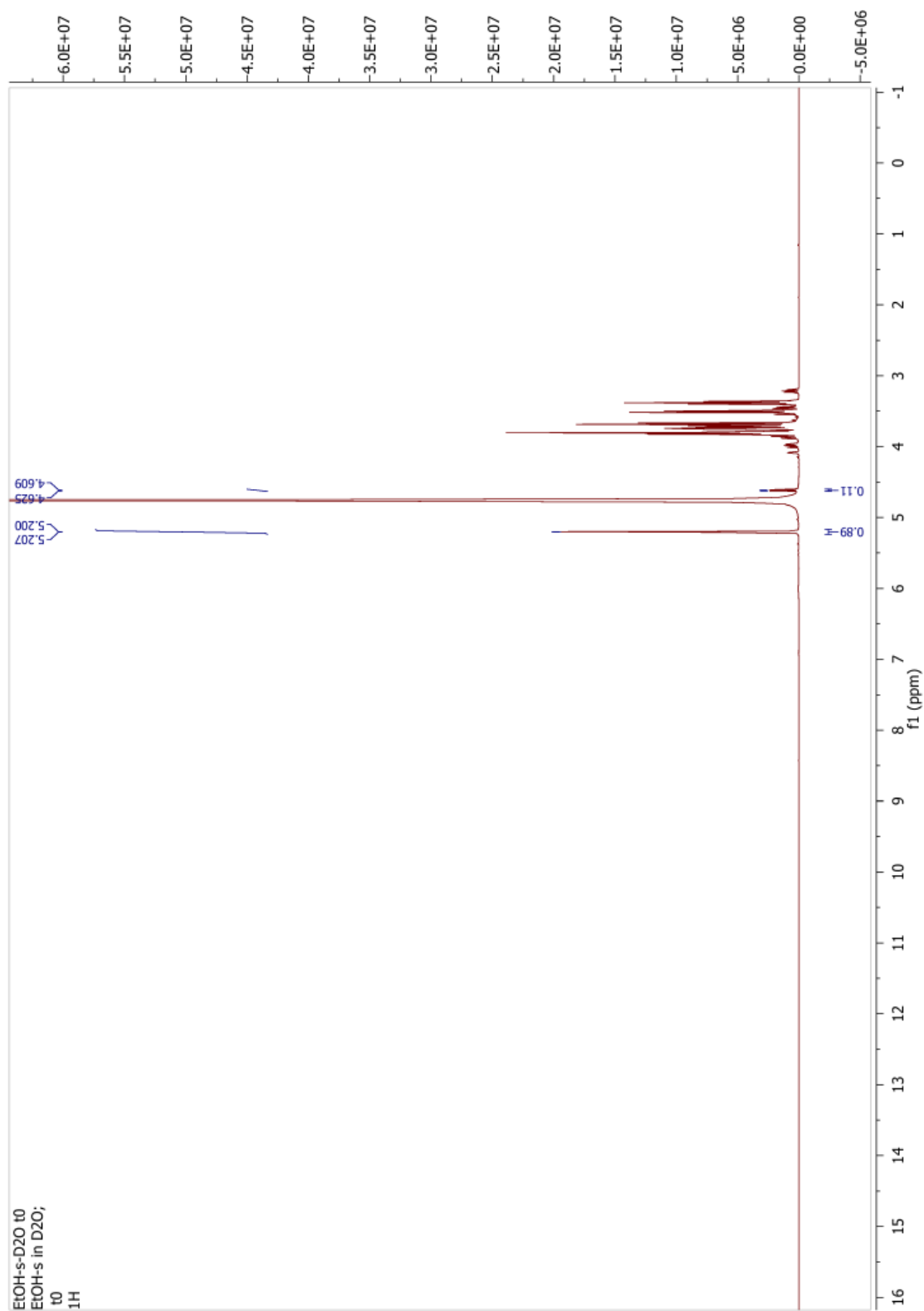


Figure S24. ^1H -NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).

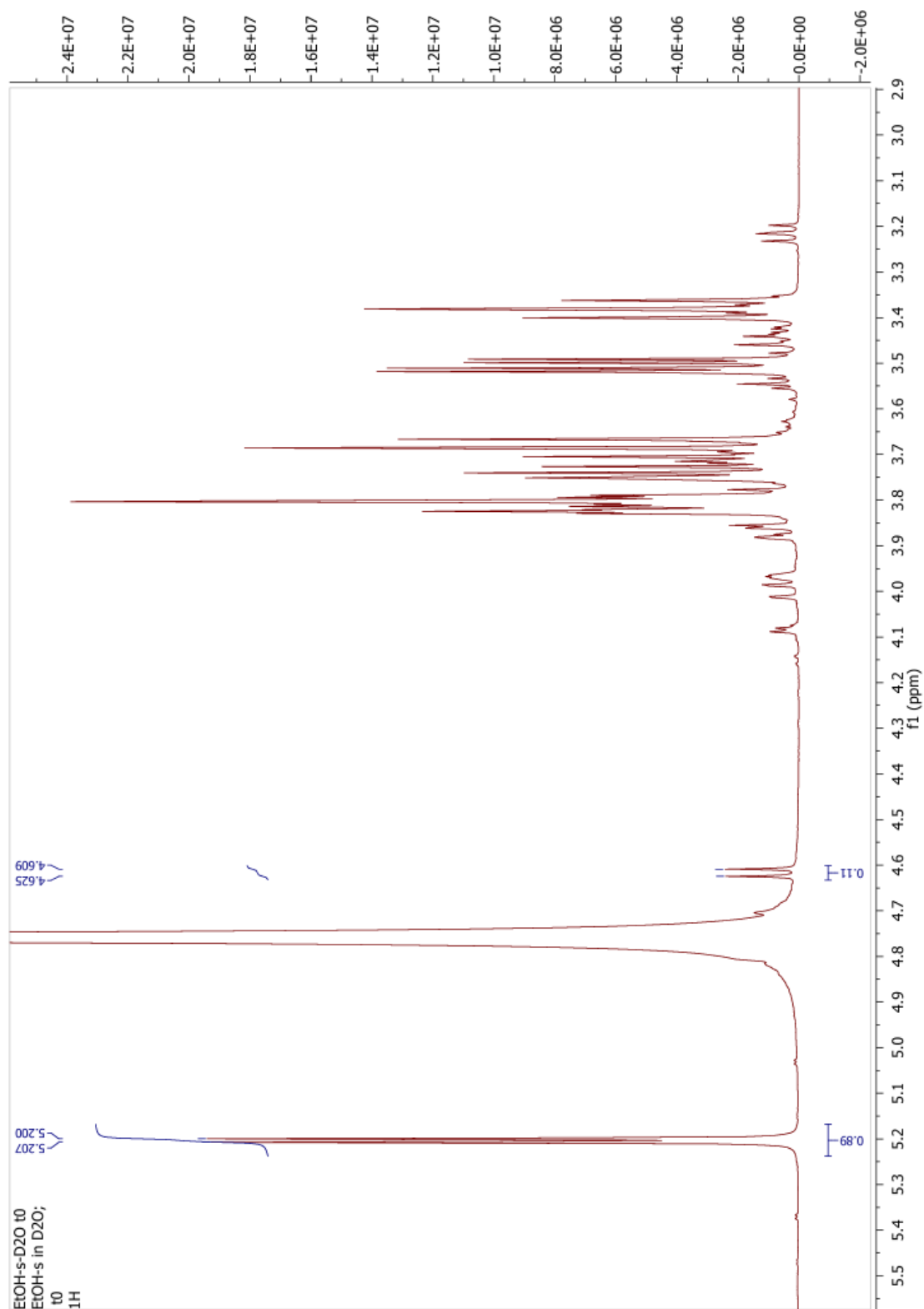


Figure S25. Expanded ^1H NMR Spectrum (D₂O) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).

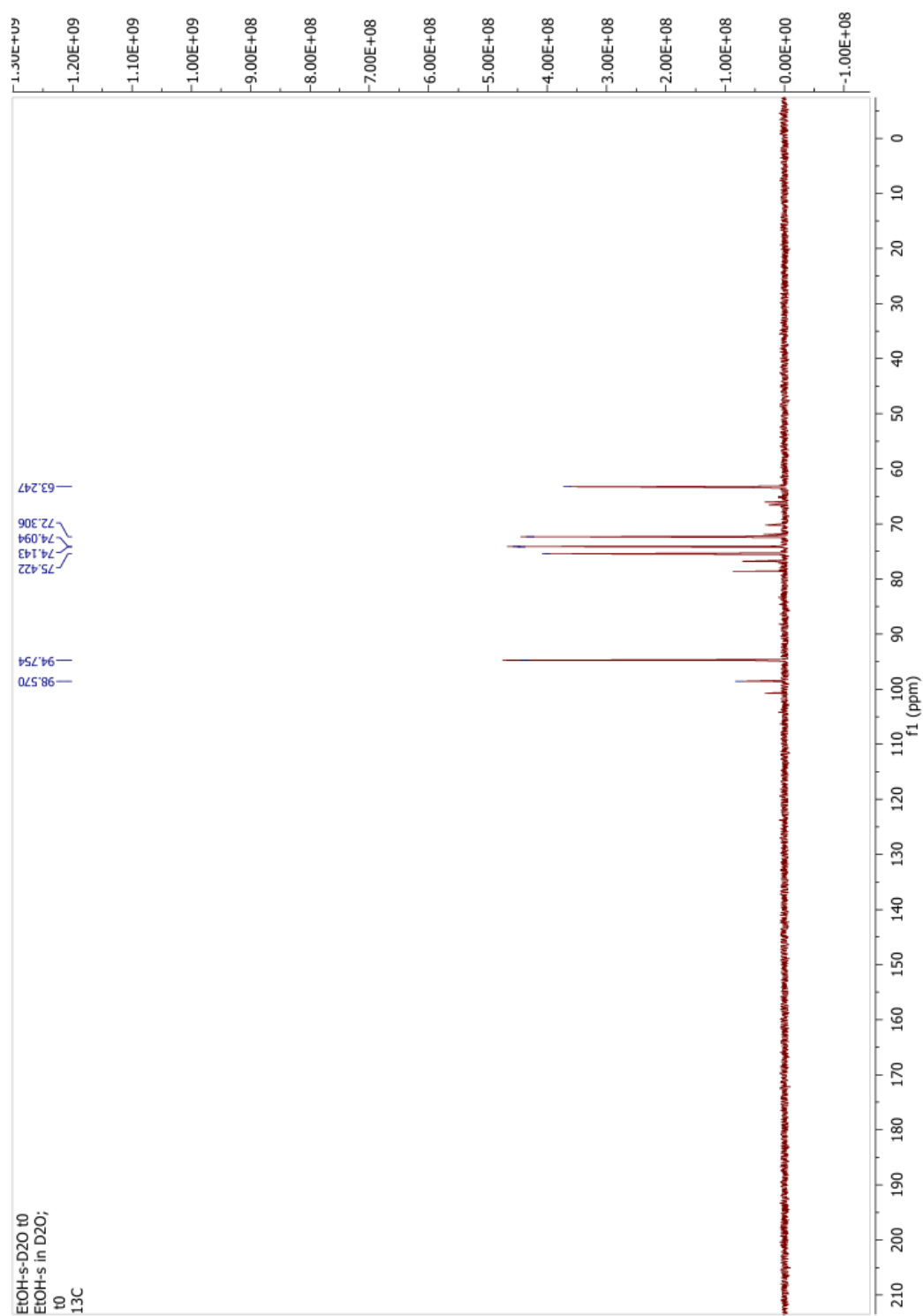


Figure S26. ^{13}C NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).

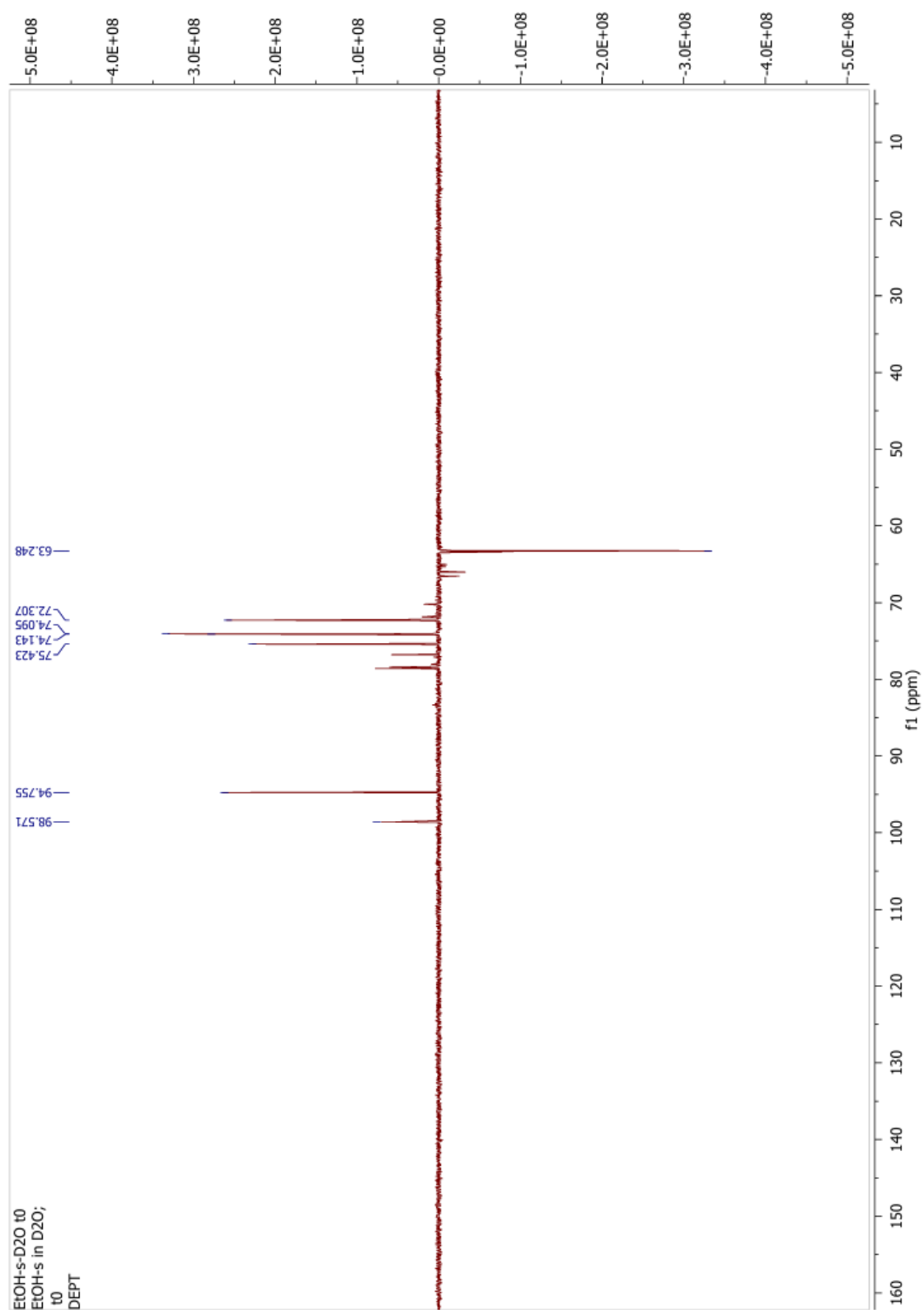


Figure S27. DEPT-135 Spectrum (D₂O) of the isolated α -D-glucopyranose-rich fraction at time zero (t_0).

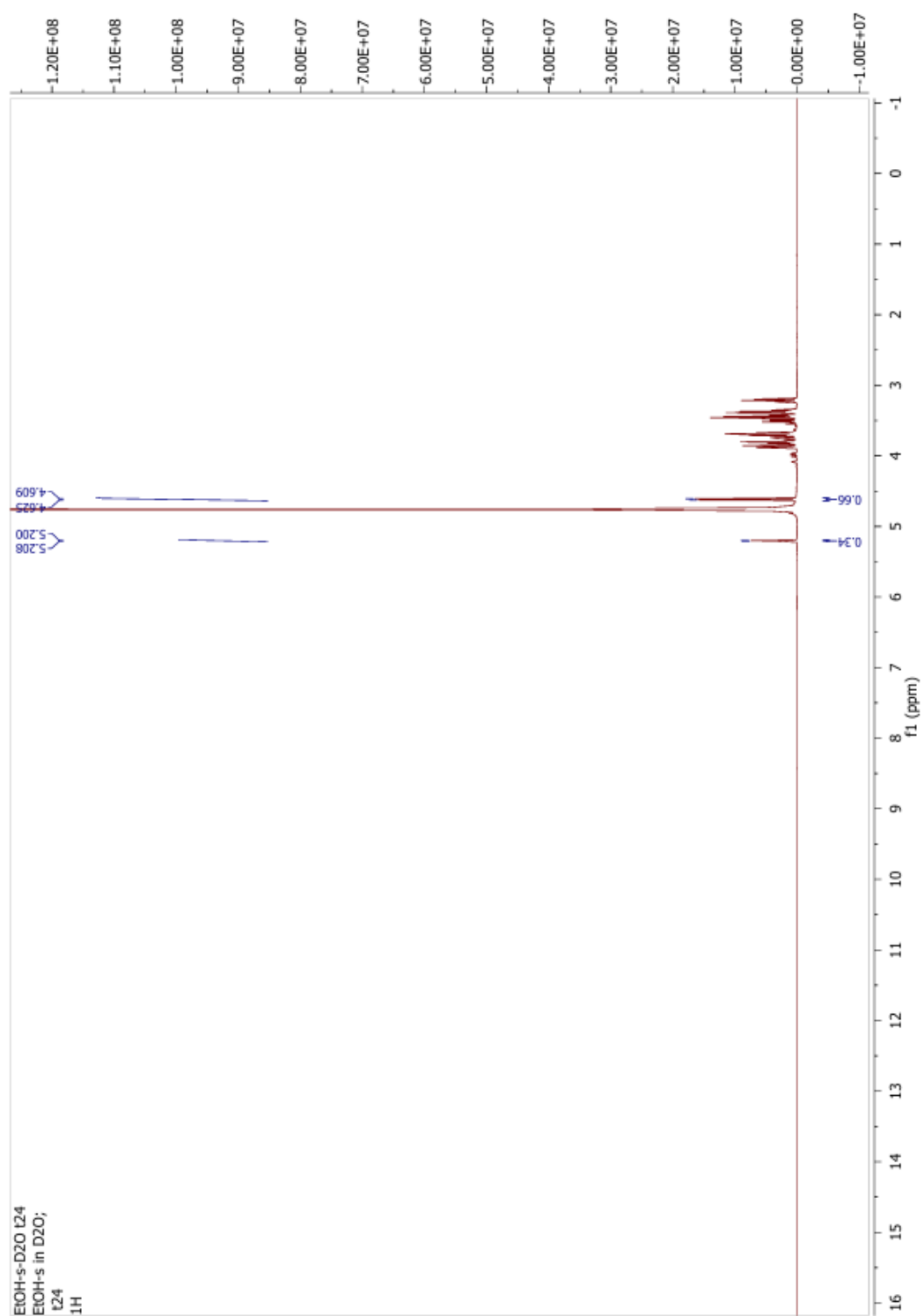


Figure S28. ^1H -NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).

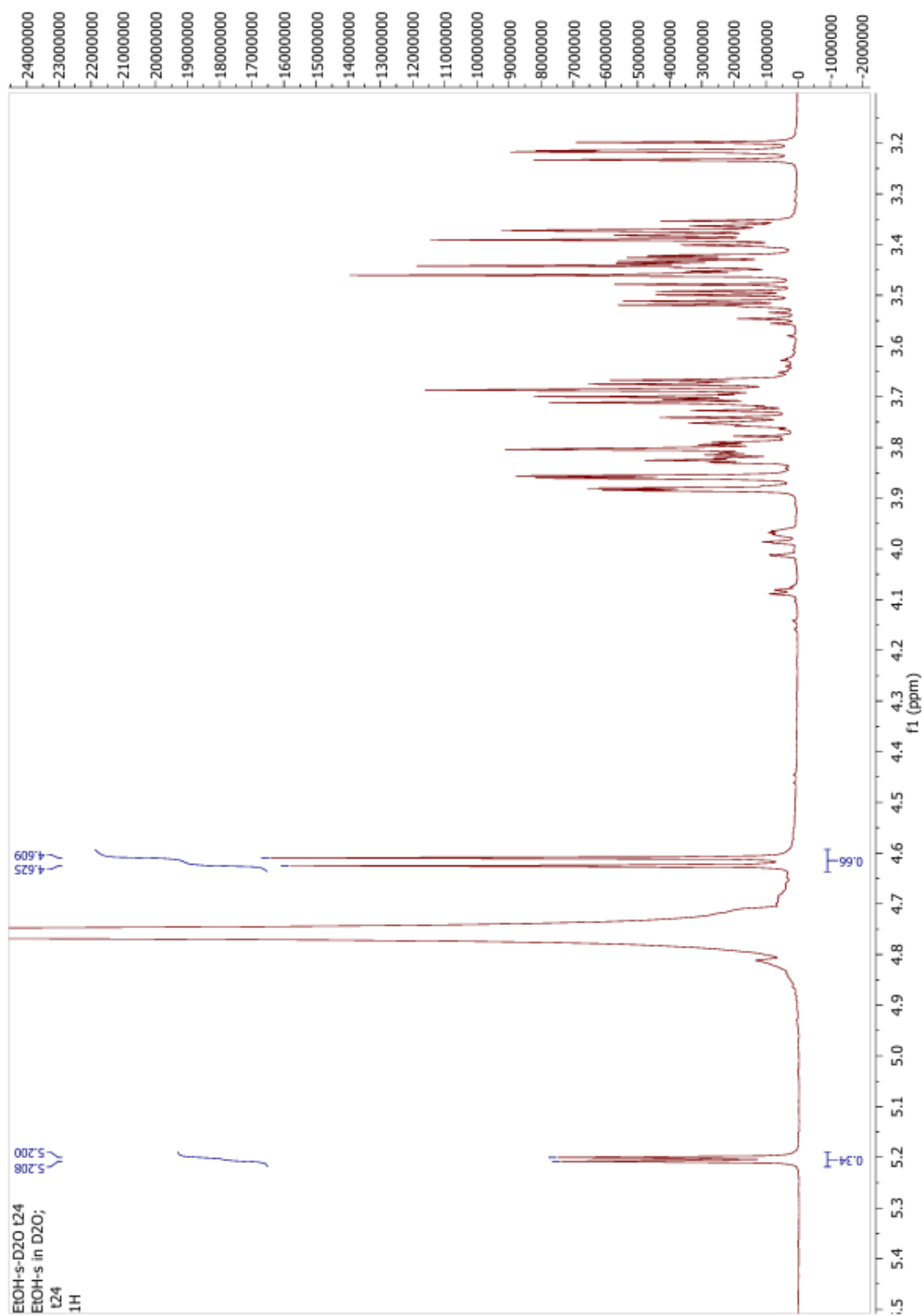


Figure S29. Expanded ^1H NMR Spectrum (D₂O) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).

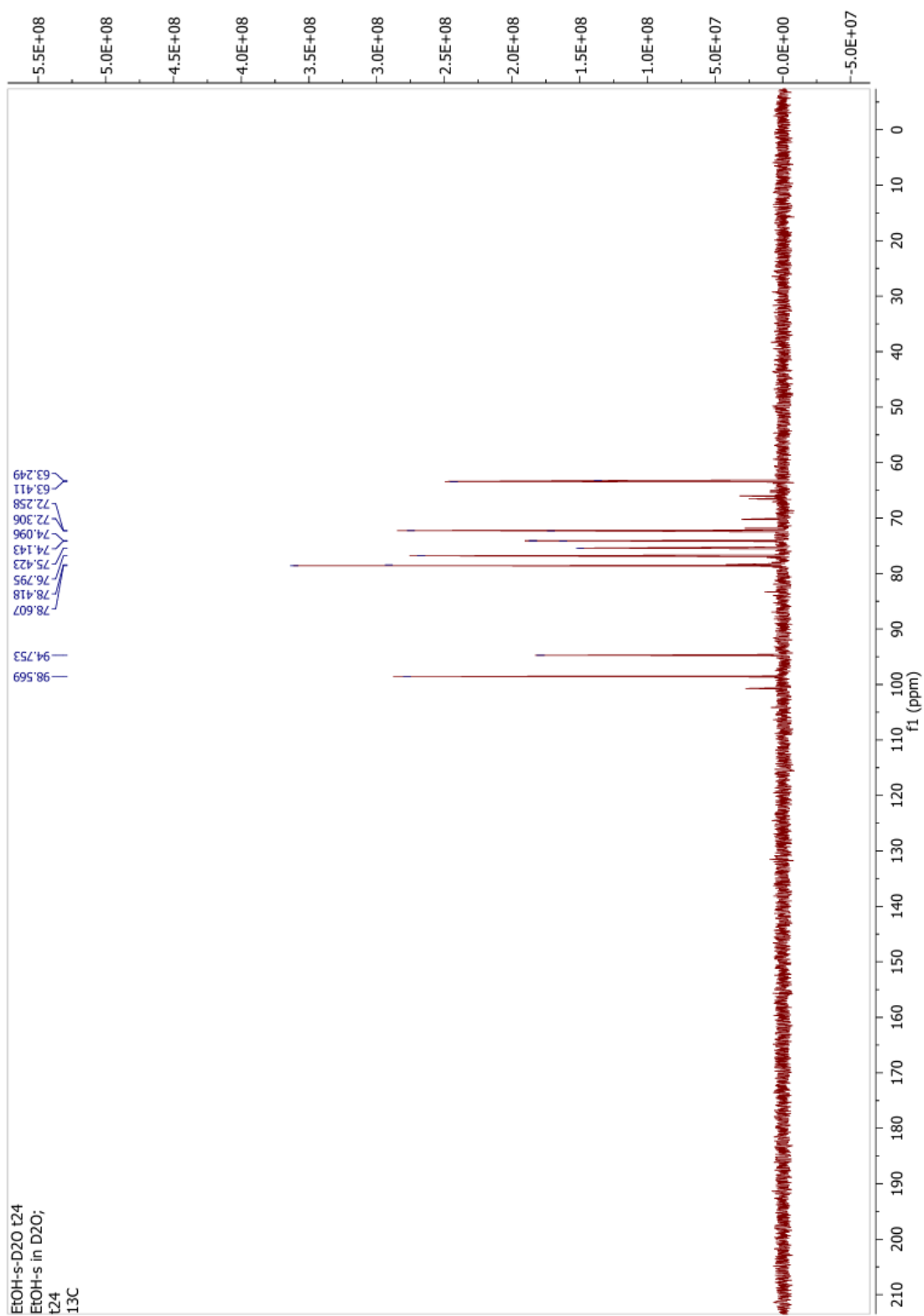


Figure S30. ^{13}C NMR Spectrum (D_2O) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t_{24}).

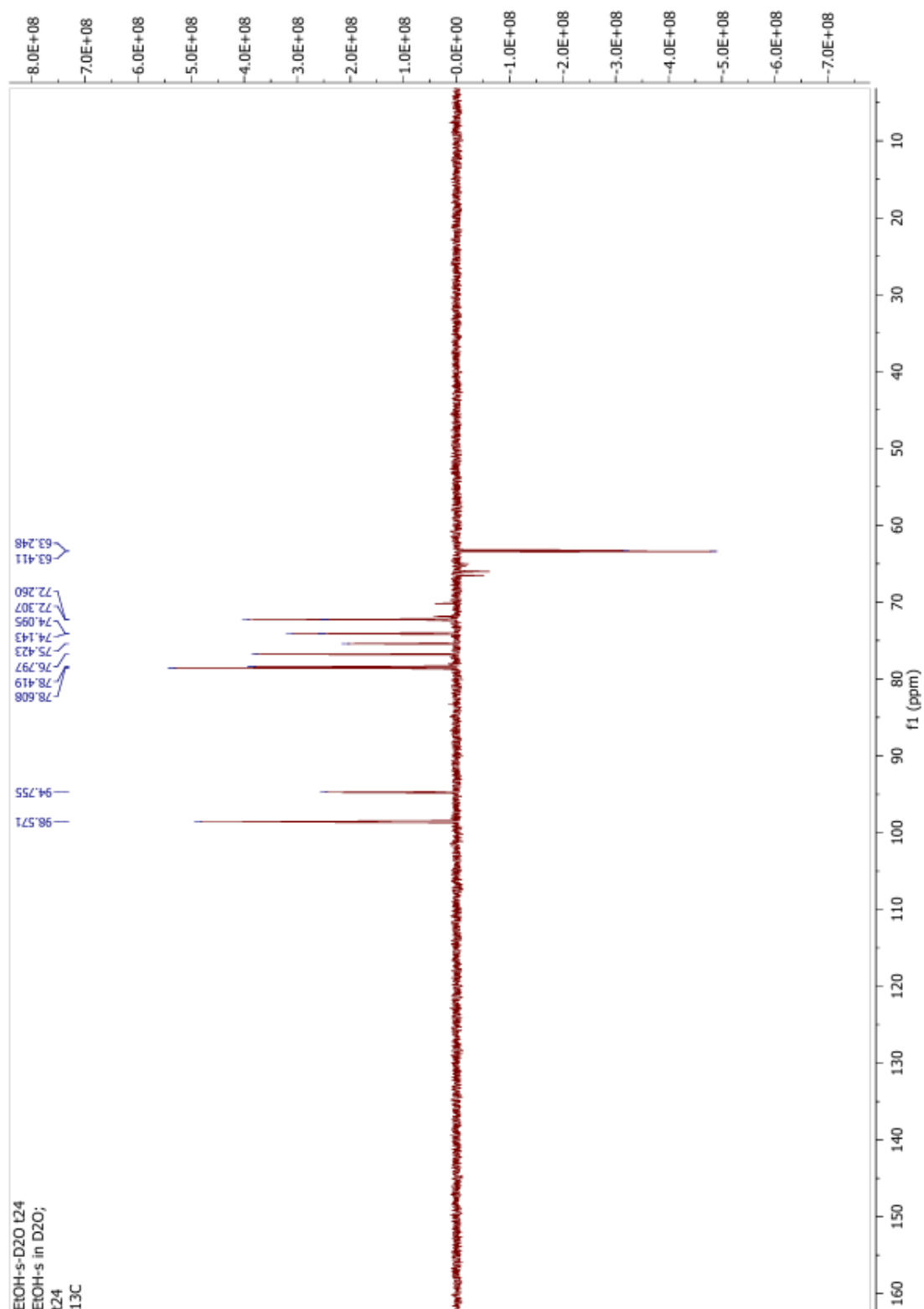


Figure S31. DEPT-135 Spectrum (D₂O) of the isolated α -D-glucopyranose-rich fraction after 24 hours (t₂₄).

Table S1. Optical rotation measurements of the isolated α -D-glucopyranose-rich fraction recorded over time during mutarotation in water at 20 °C.

Time (min)	Observed rotation, α (°)	Specific rotation $[\alpha]_D^{20}$ (°)
0	+0.923	+92.3
5	+0.882	+88.2
10	+0.848	+84.8
15	+0.815	+81.5
20	+0.785	+78.5
25	+0.758	+75.8
30	+0.733	+73.3
35	+0.710	+71.0
110 (1 h 50 min)	+0.518	+51.8
180 (3 h)	+0.465	+46.5
1080 (18 h, equilibrium)	+0.438	+43.8

Measurements were performed in water at 20 °C using a 1 dm polarimeter tube at 589 nm. Sample concentration was 1.0 g/100 mL (1% w/v).

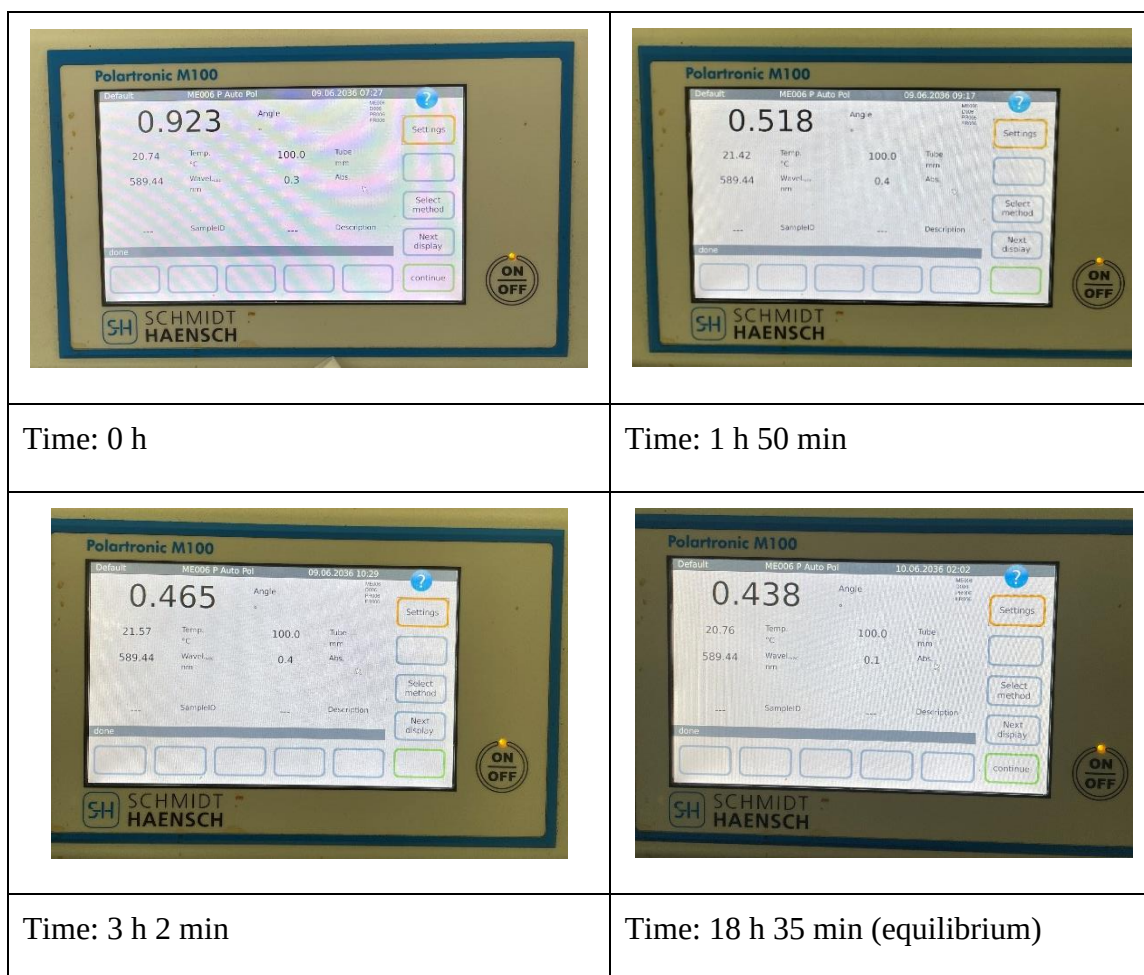


Figure S32. Representative polarimeter display images recorded immediately after dissolution (0 h), (1 h 50 min), (3 h 2 min) and after 18 h 35 min (equilibrium) to monitor changes in optical rotation during mutarotation under the experimental conditions described in the main manuscript.

Table S2. Reproducibility of the isolated α -D-glucopyranose-rich fraction across independent experimental batches.

Date fruit sample batch	Analysis performed
Batch 1	TLC, LC-MS, ^1H NMR, ^{13}C NMR, DEPT-135, specific rotation
Batch 2	TLC, LC-MS, ^1H NMR, ^{13}C NMR, DEPT-135
Batch 3	TLC, LC-MS, ^1H NMR, ^{13}C NMR, DEPT-135