Pd-HPW/SiO_2 Bi-Functional catalyst: Sonochemical Synthesis, Characterization and effect on octahydroquinazolinone synthesis

Md. A. Bakht[1*], Mshari. Alotaibi[1], Abdulrahman I. Alharthi[1], Israf Ud Din[1], Abuzer Ali[2], Amena Ali[3], M.J. Ahsan[4]

[1]Department of Chemistry, College Science and Humanities, Prince Sattam Bin Abdulaziz University, PO. Box no. 83, Al-Kharj 11942, Saudi Arabia.

[2]Department of Pharmacognosy, College of Pharmacy, Taif University, P.O. Box 11099, Taif 21944 Saudi Arabia
[3]Department of Pharmaceutical Chemistry, College of Pharmacy, Taif University, P.O. Box 11099, Taif 21944 Saudi Arabia
[4]Department of Pharmaceutical Chemistry, Maharishi Arvind College of Pharmacy, Ambabari Circle, Jaipur, Rajasthan 302 039, India.
Correspondence m.bakht@psau.edu.sa; Tel.: +966-553753763

Supplemental Materials : NMR Spectral data

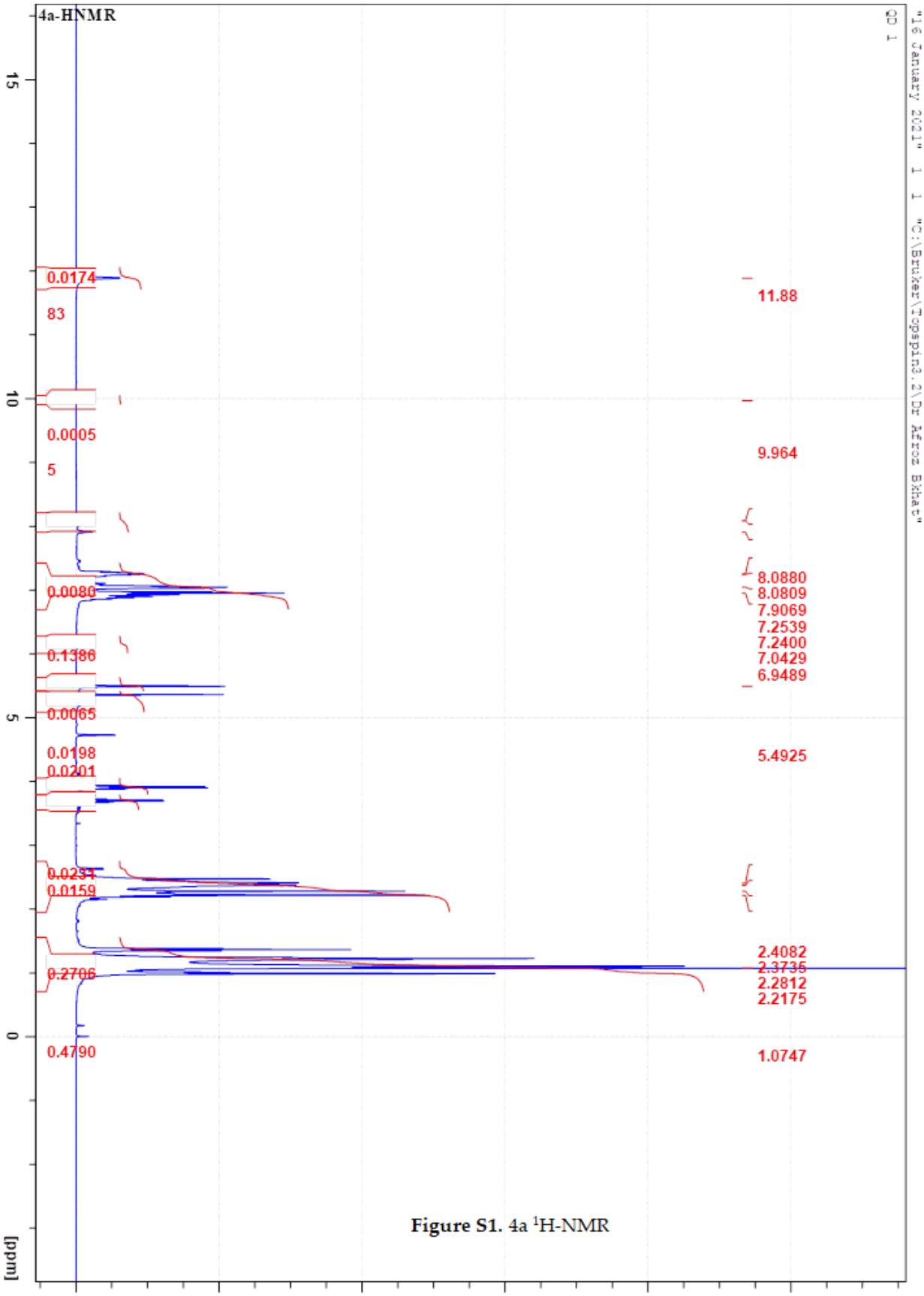

Figure S1. 4a ^{1}H-NMR

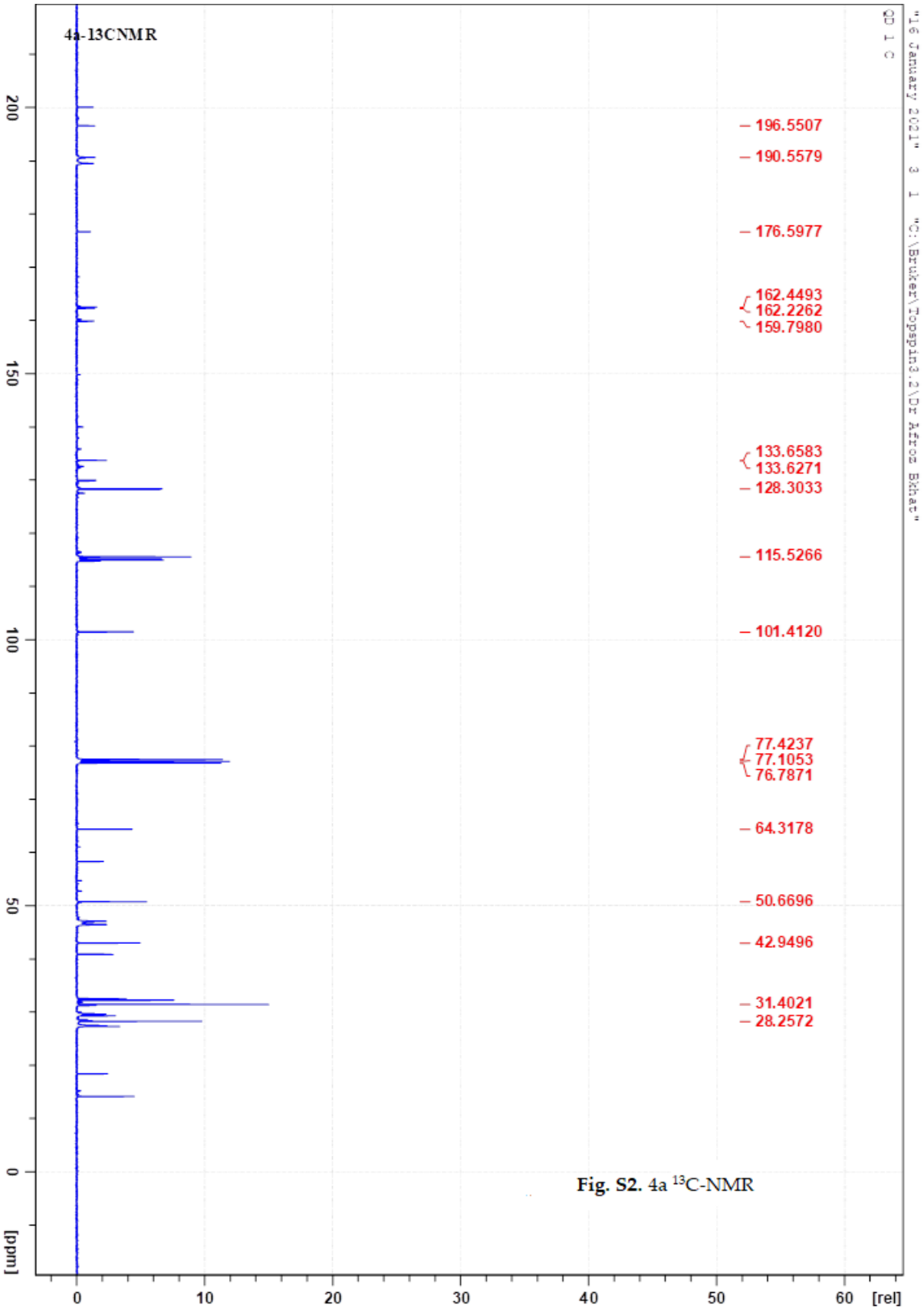

Fig. S2. 4a ^{13}C-NMR

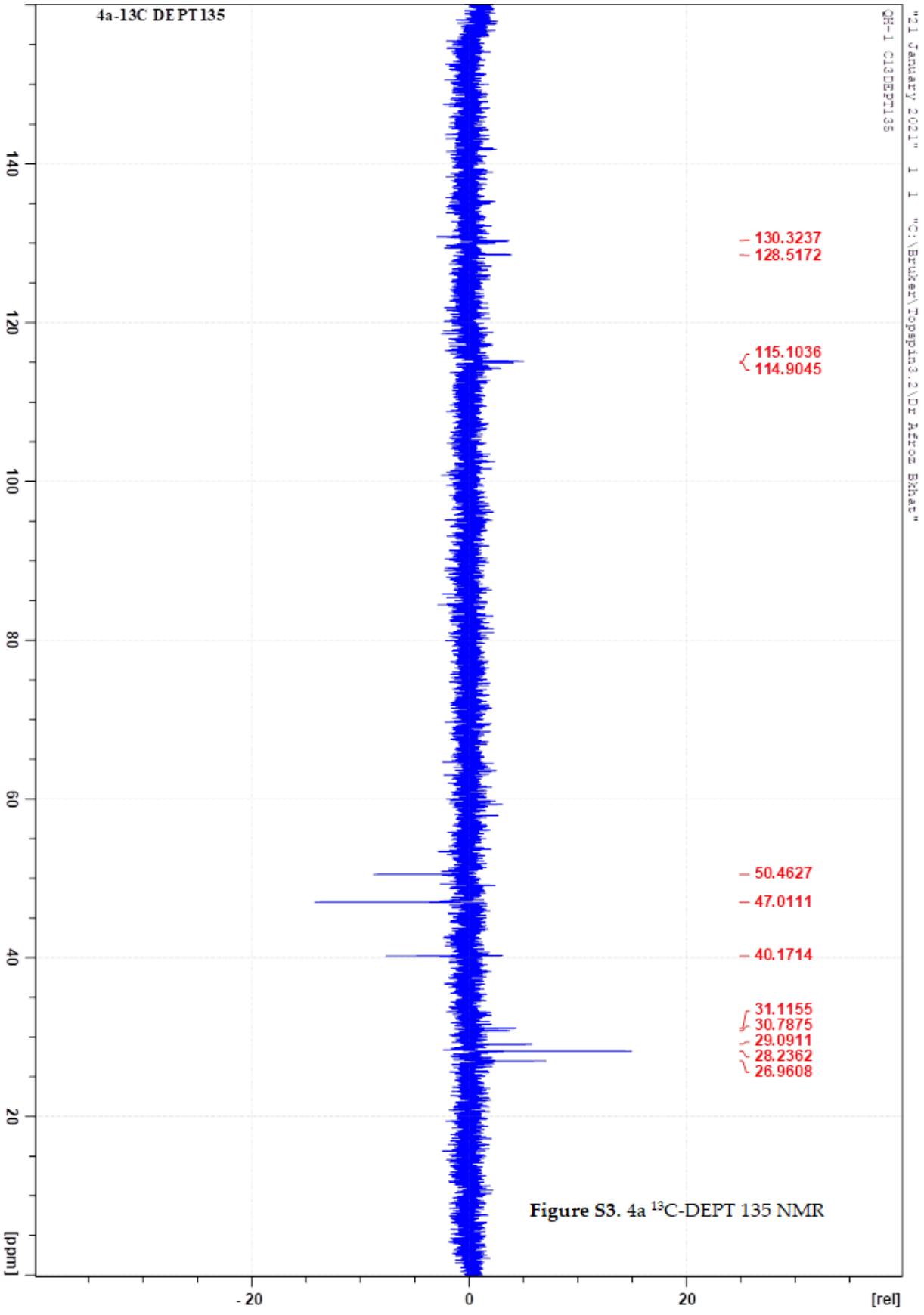

Figure S3. 4a ^{13}C-DEPT 135 NMR

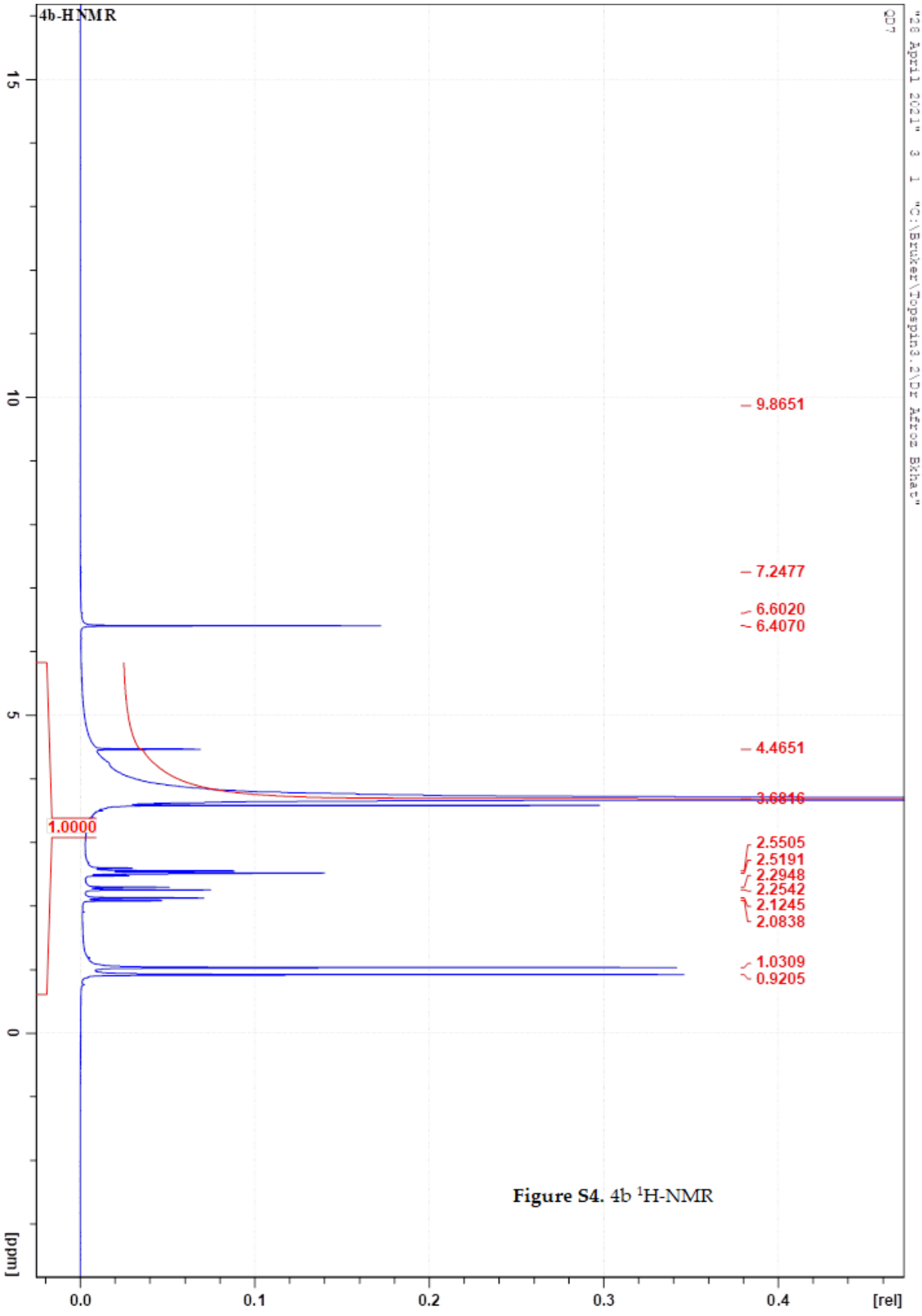

Figure S4. 4b ^{1}H-NMR

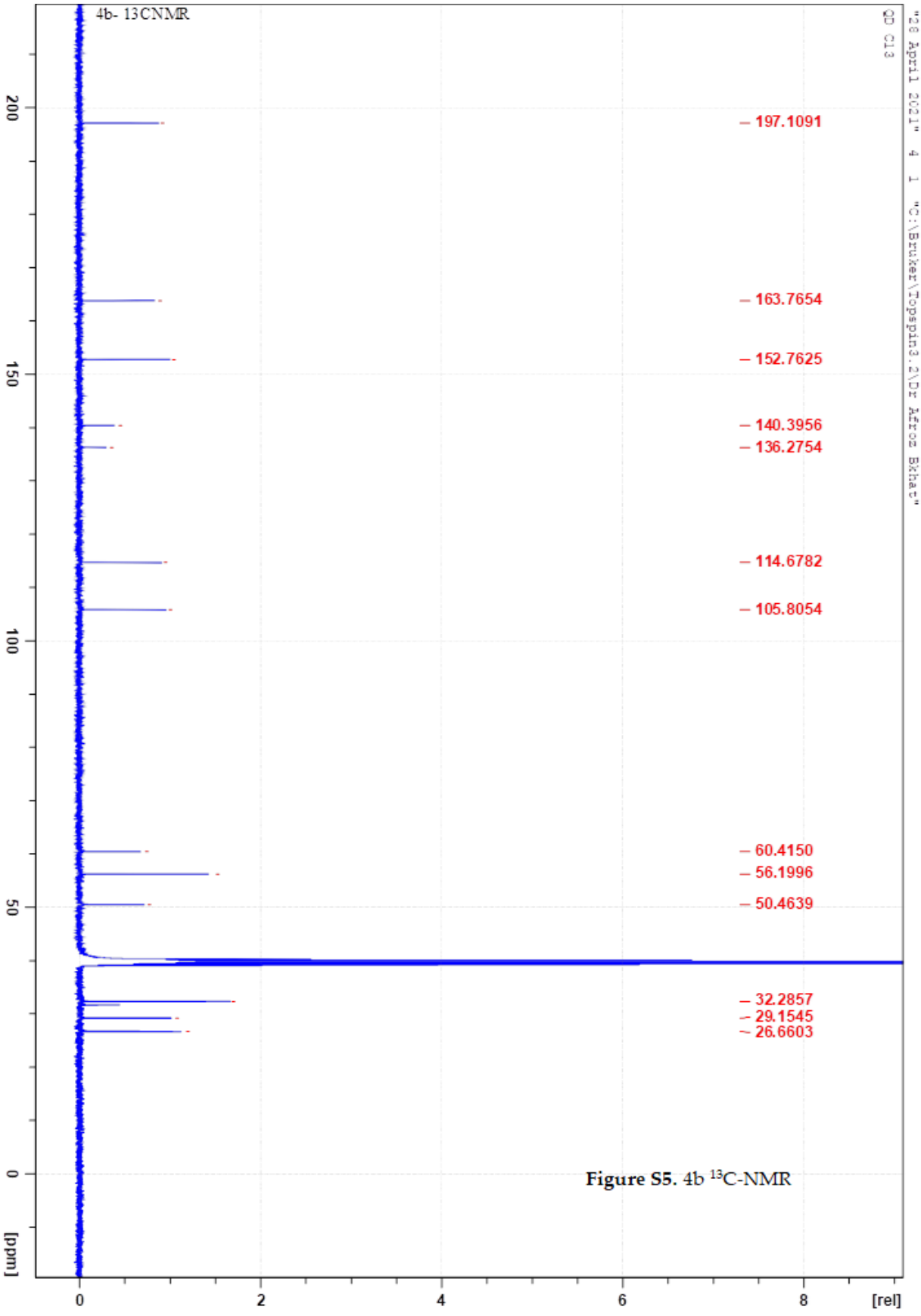

Figure S5. 4b ^{13}C-NMR

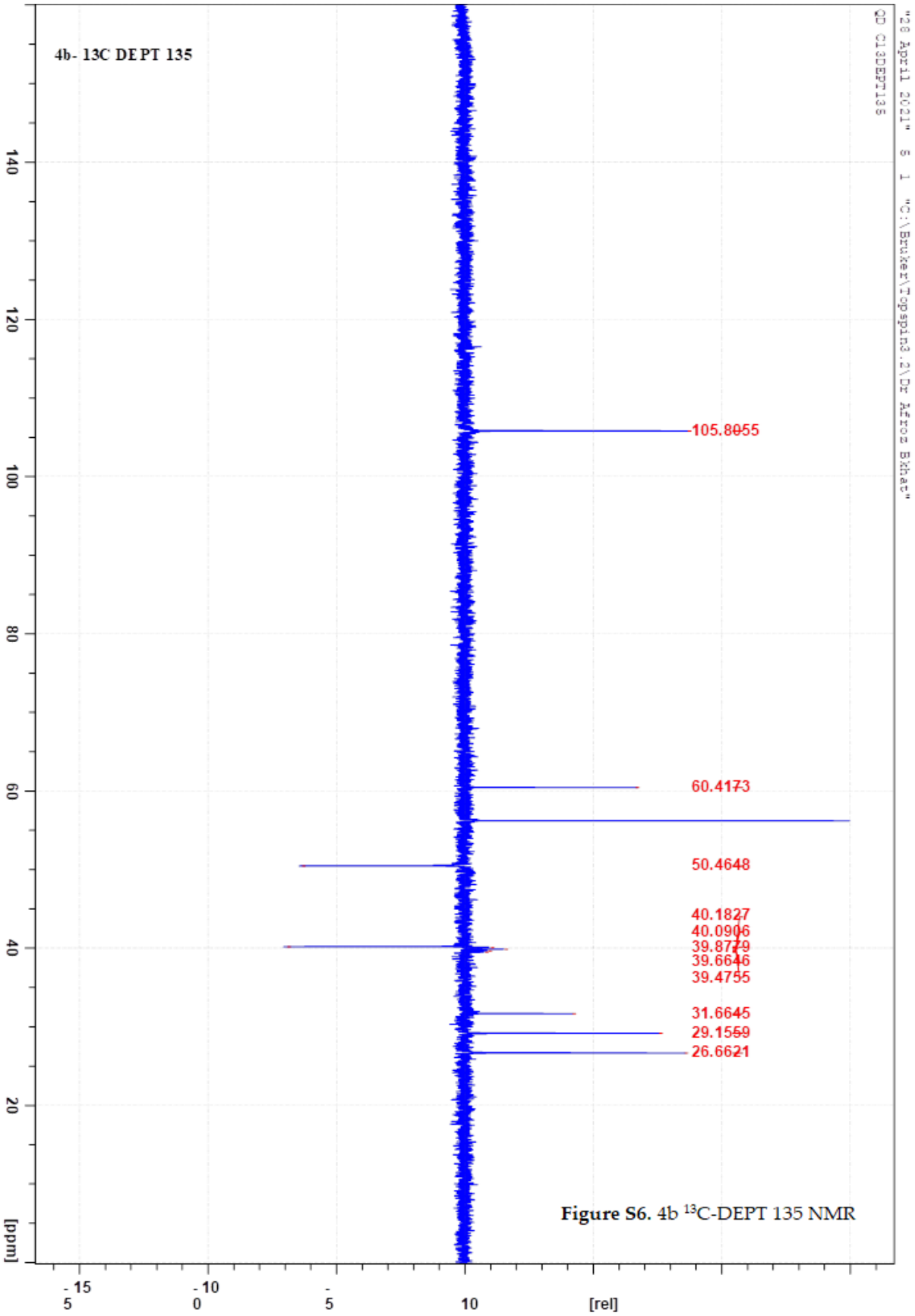

Figure S6. 4b ^{13}C-DEPT 135 NMR

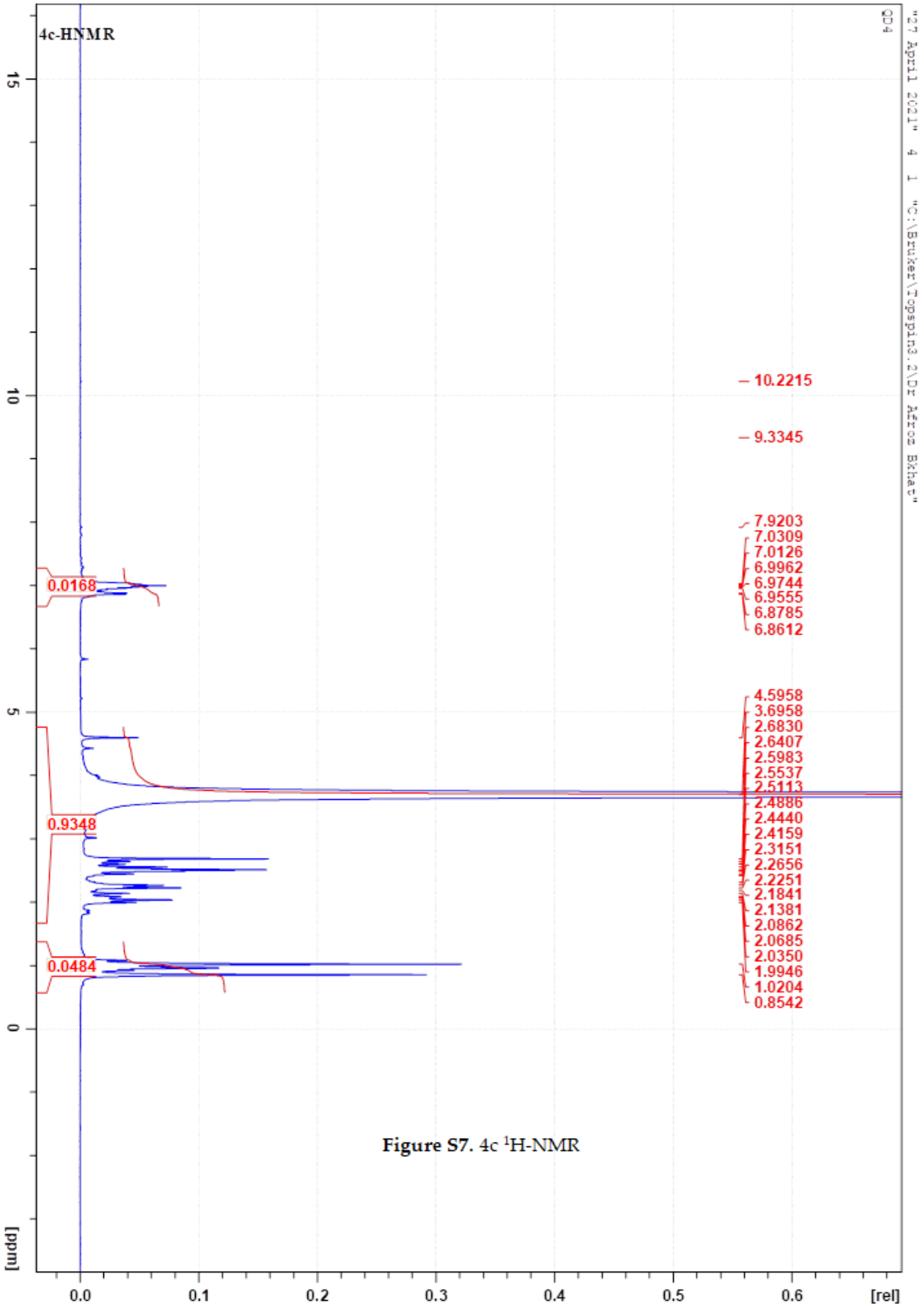

Figure S7. 4c ^{1}H-NMR

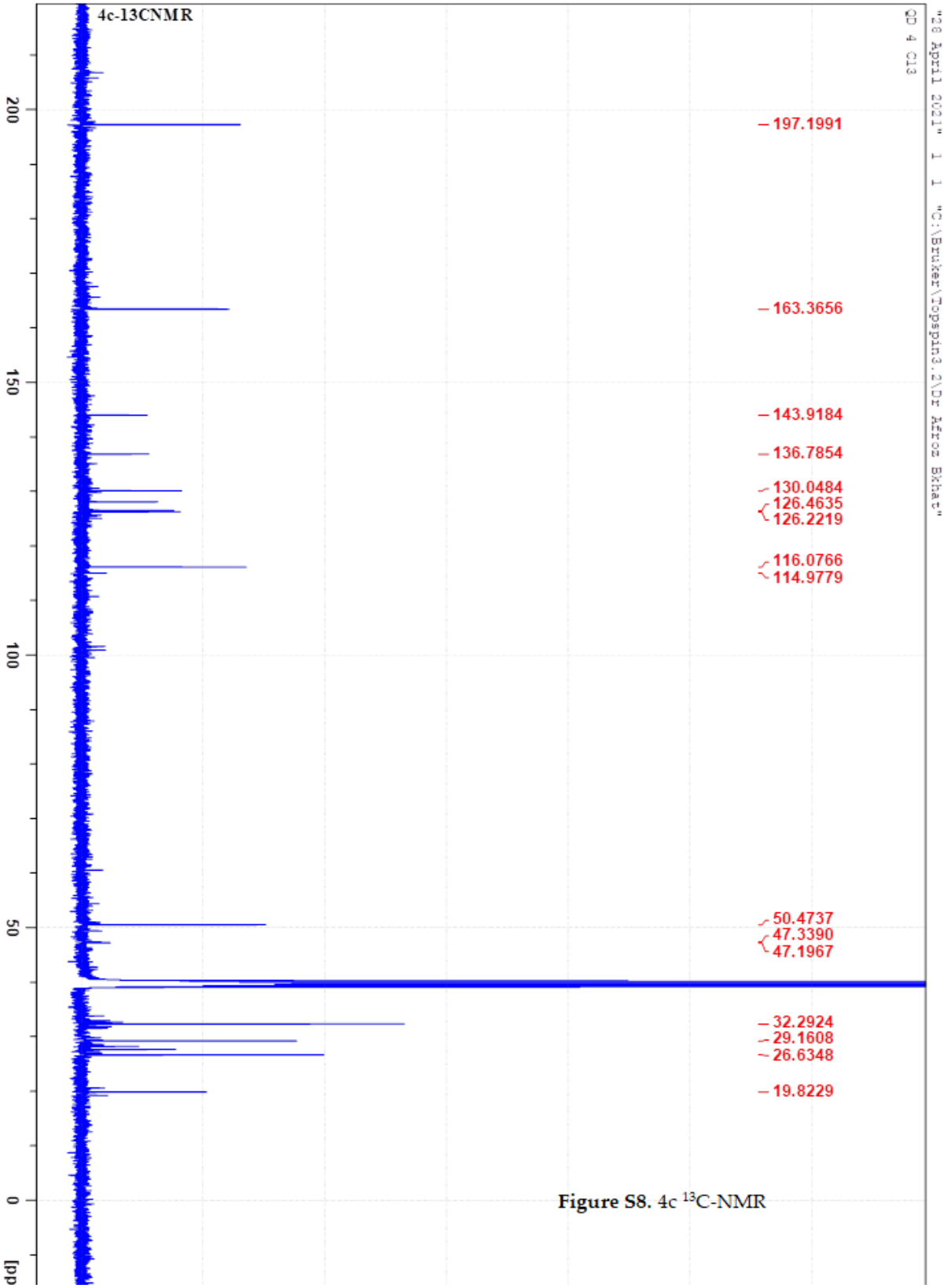

Figure S8. 4c ^{13}C-NMR

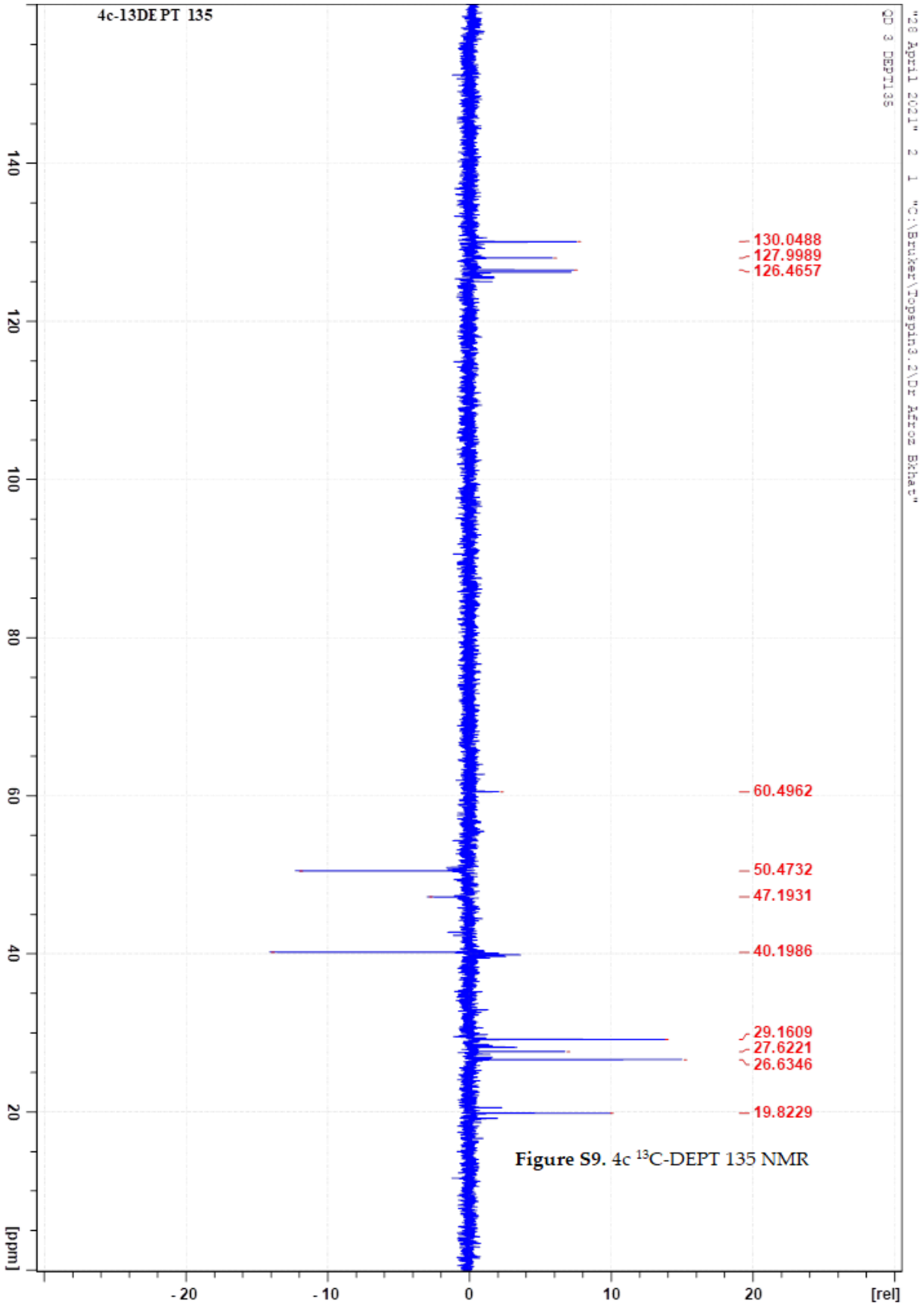

Figure S9. 4c ^{13}C-DEPT 135 NMR

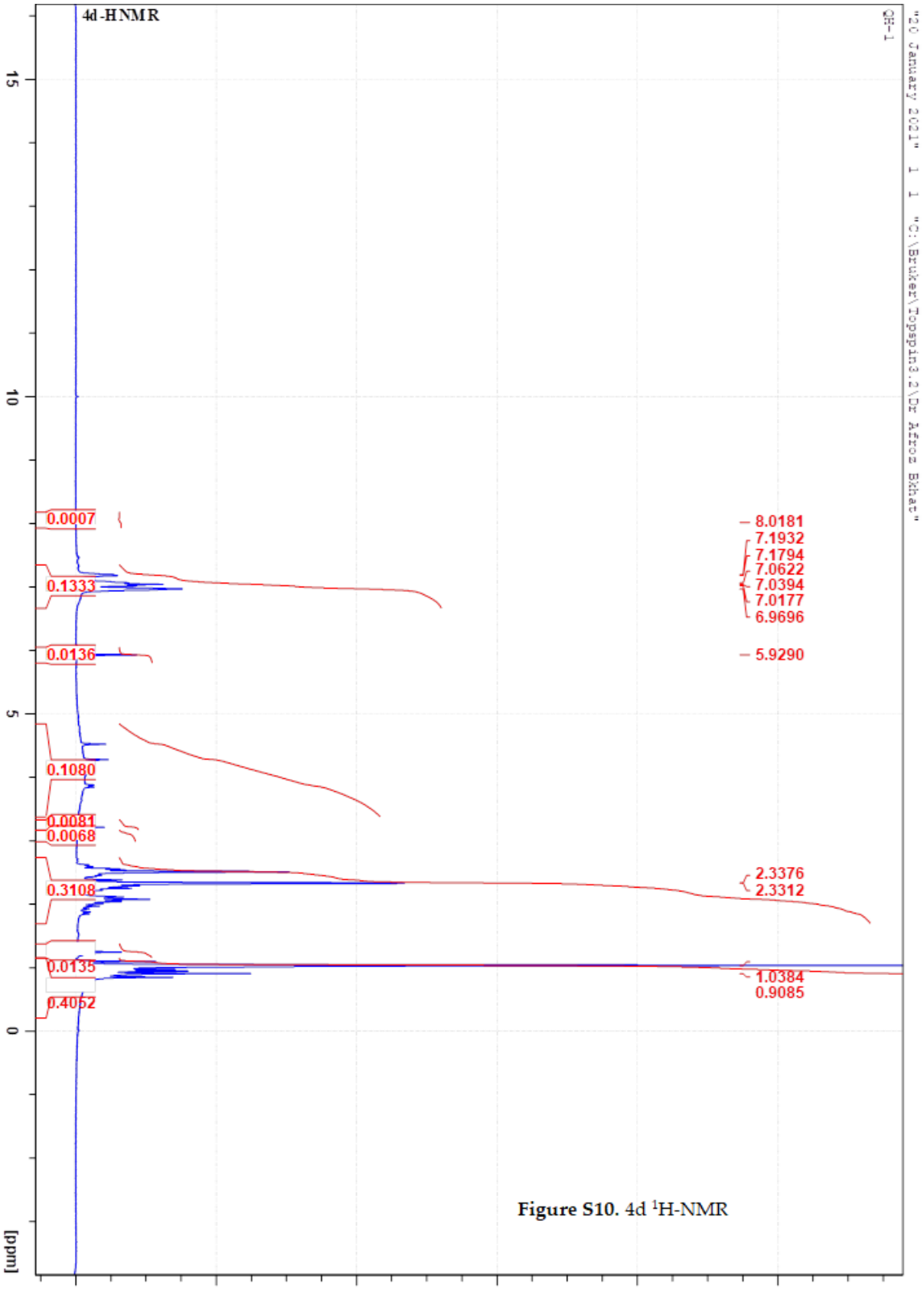

Figure S10. 4d ^{1}H-NMR

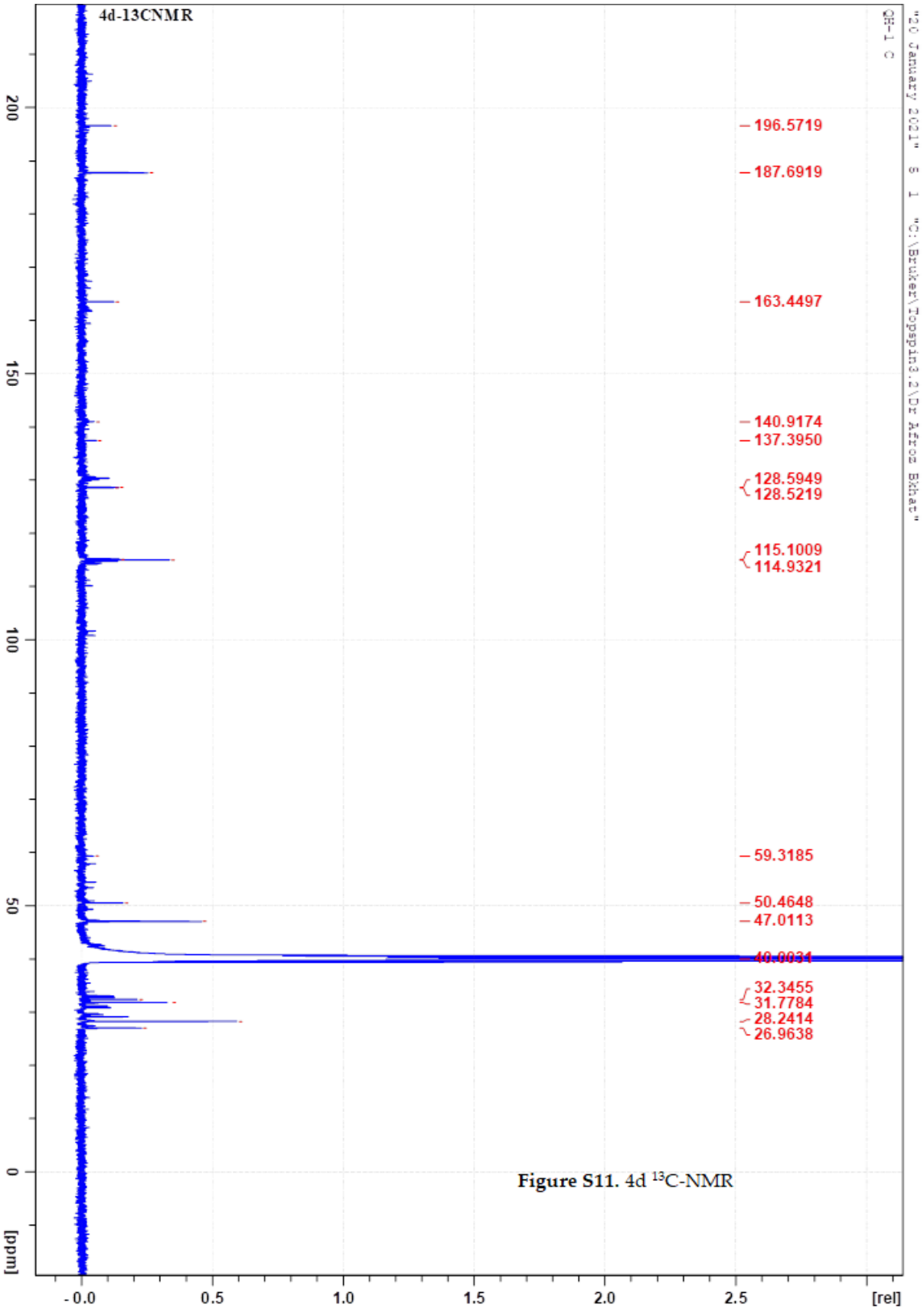

Figure S11. 4d ^{13}C-NMR

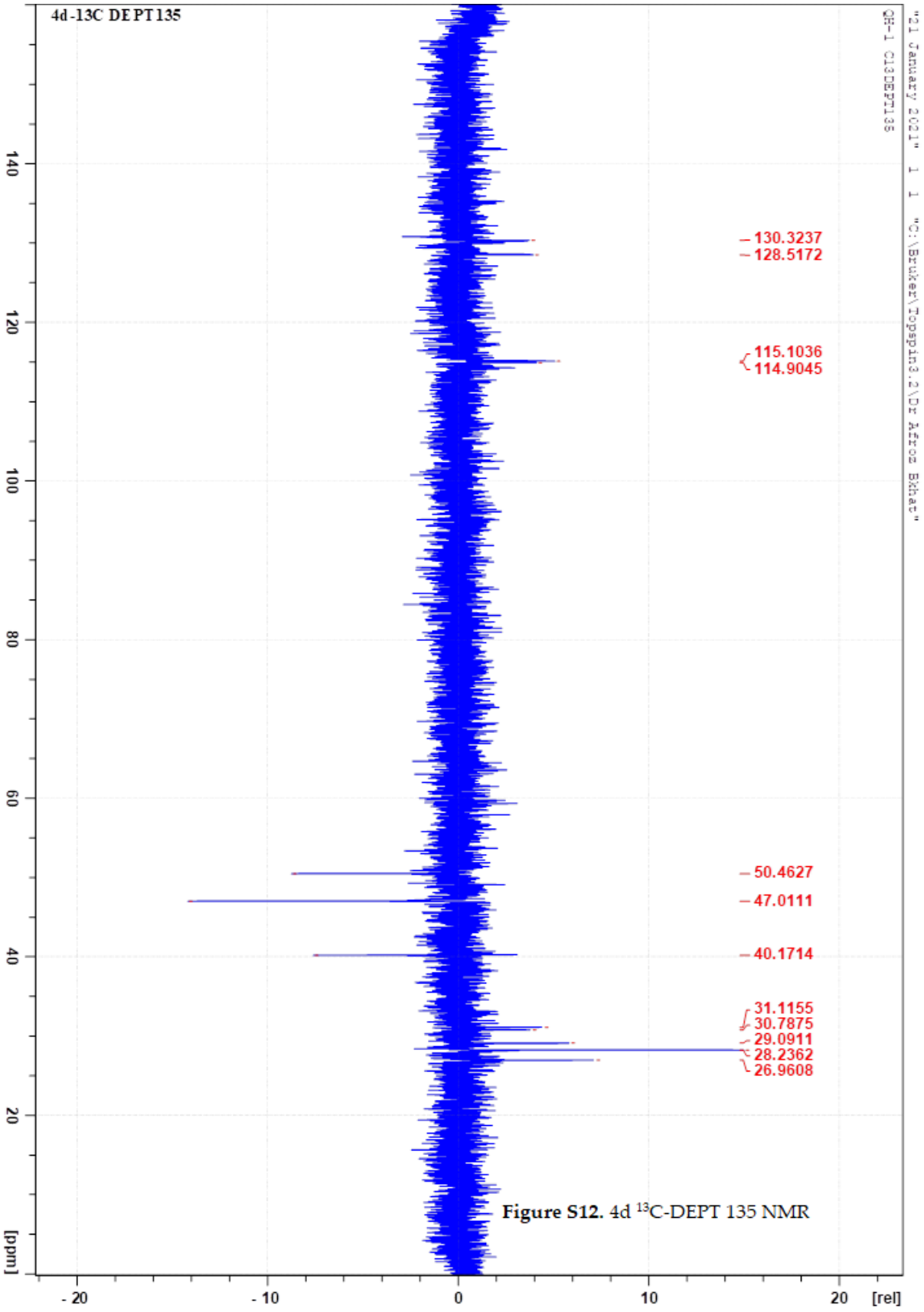

Figure S12. 4d ^{13}C-DEPT 135 NMR

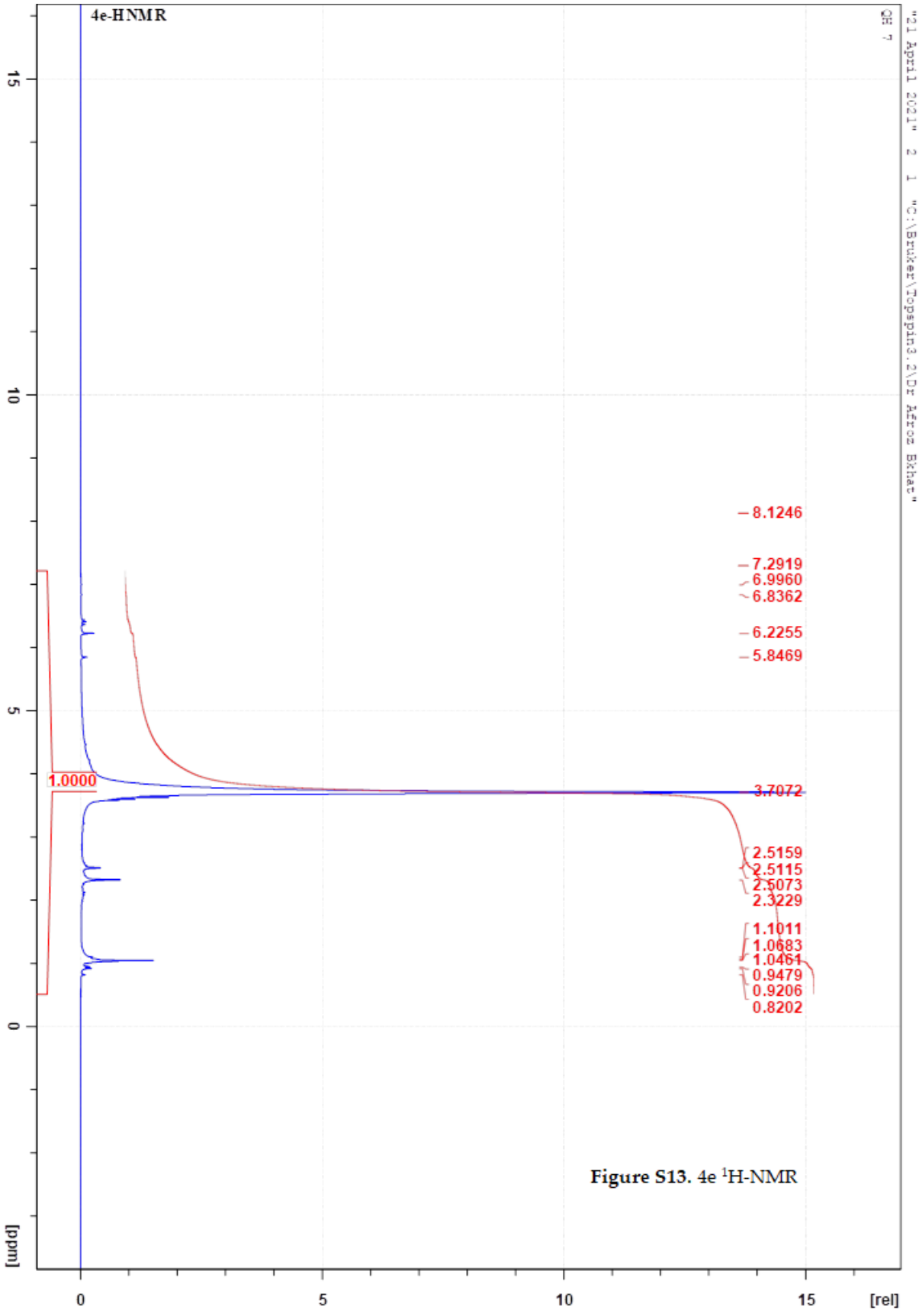

Figure S13. 4e ^{1}H-NMR

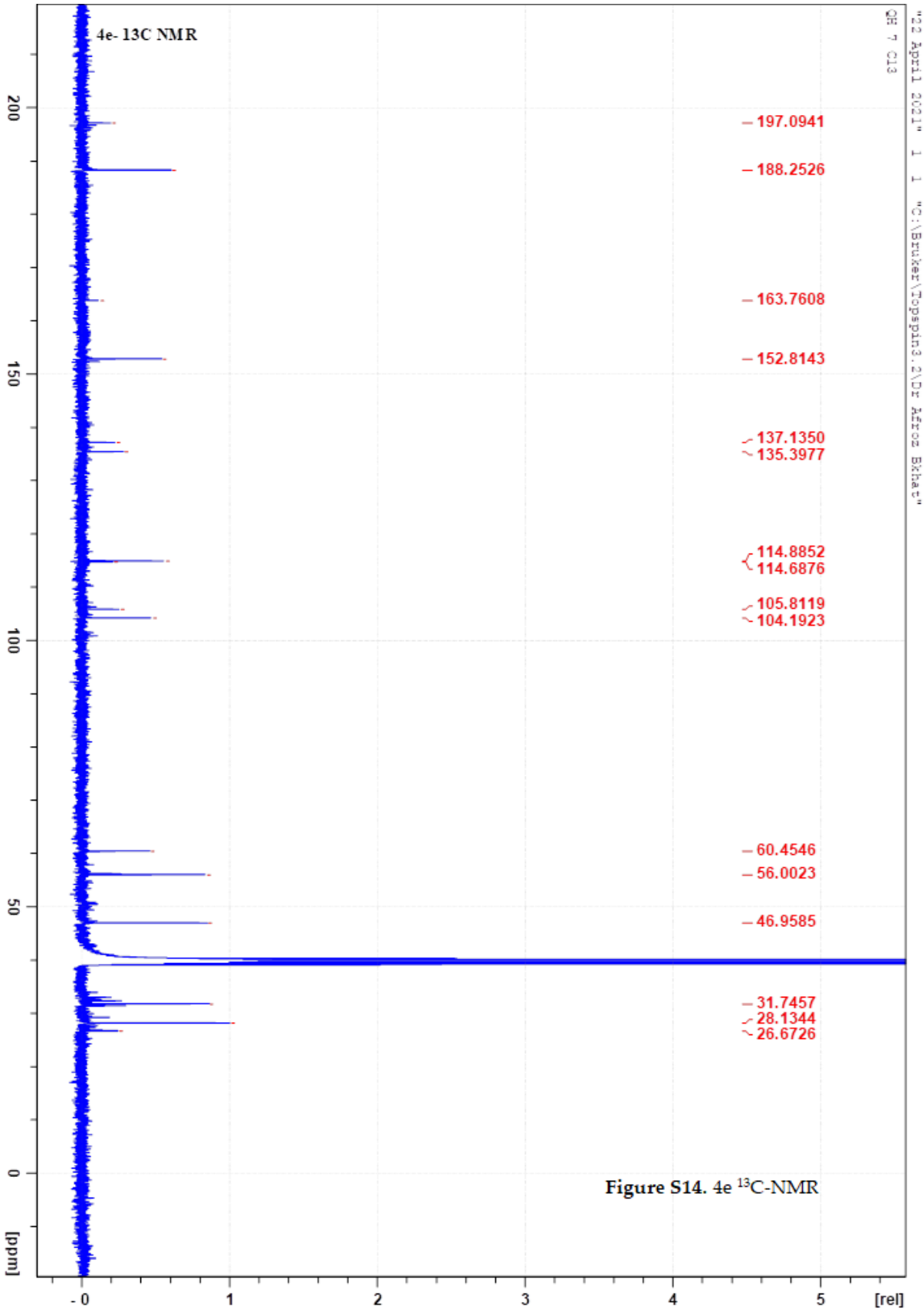

Figure S14. 4e ^{13}C-NMR

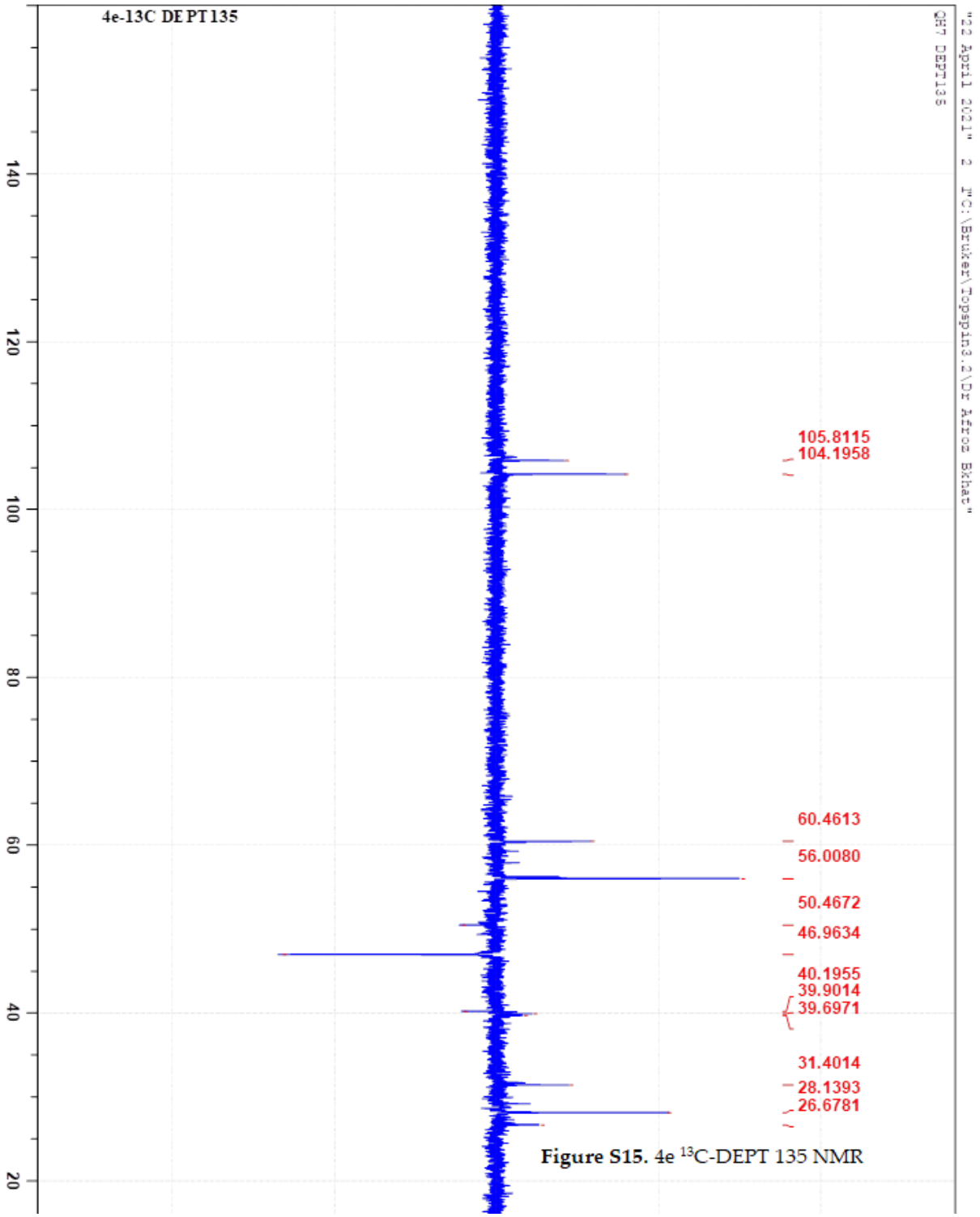

Figure S15. 4e ^{13}C-DEPT 135 NMR

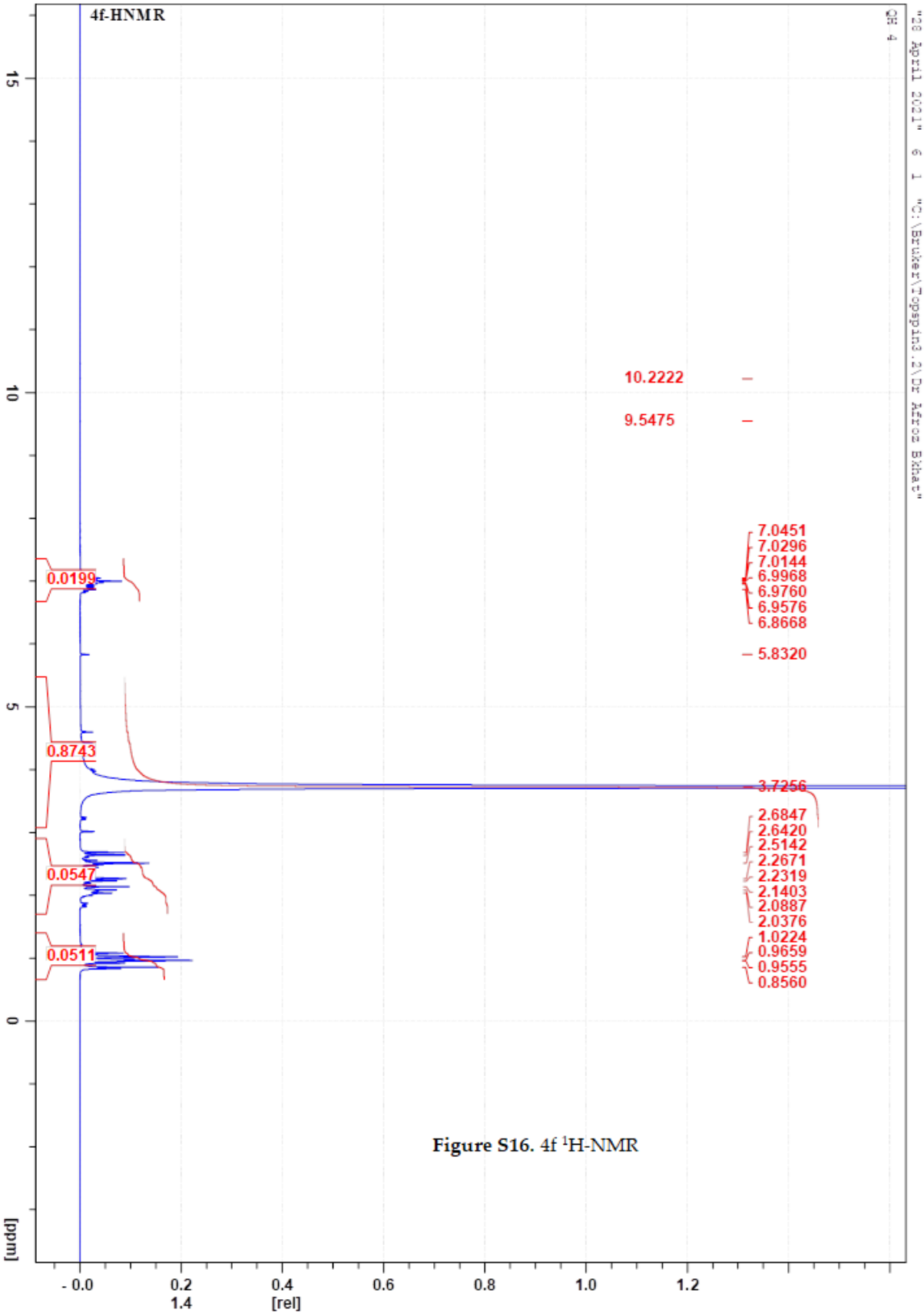

Figure S16. 4f ^{1}H-NMR

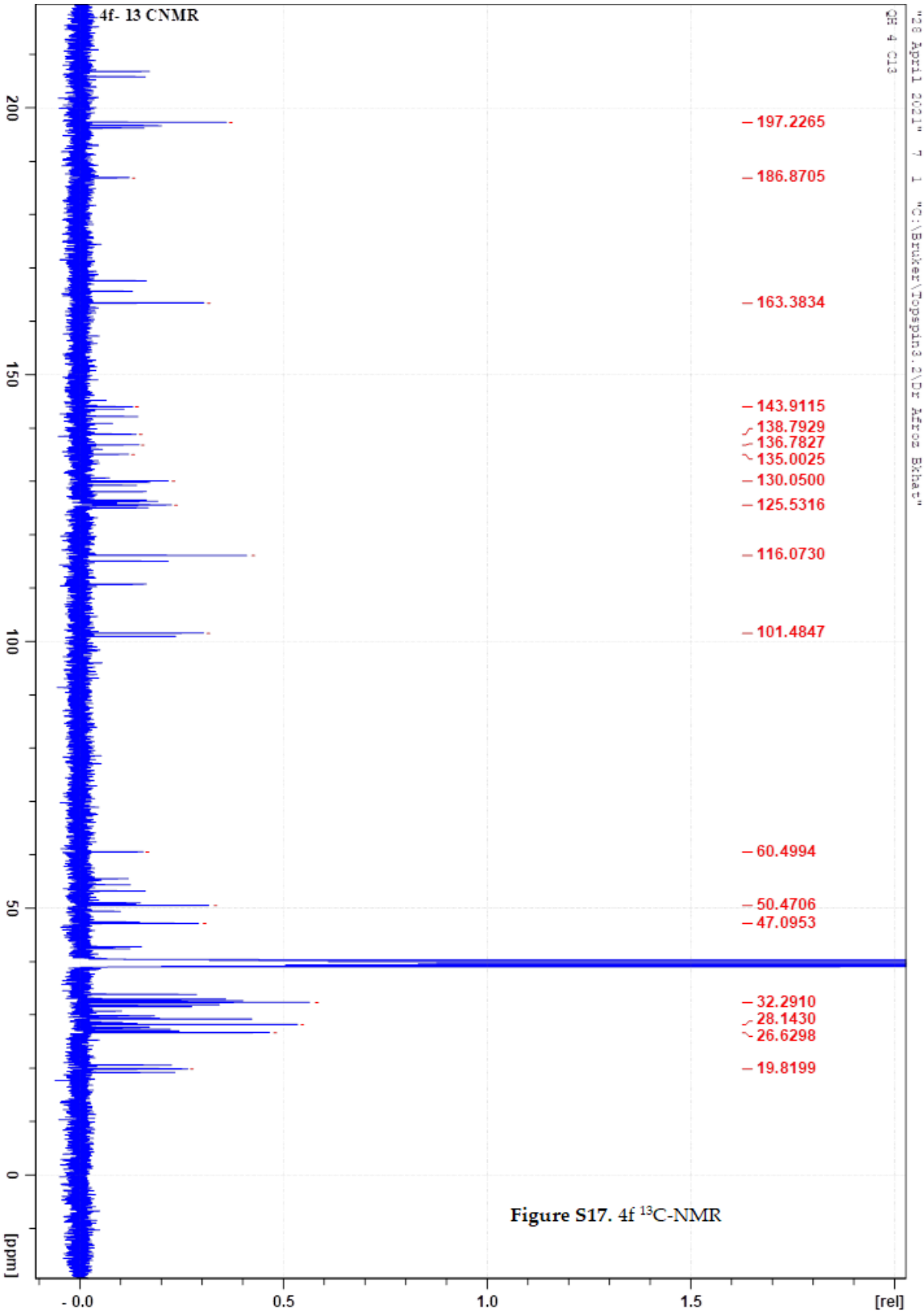

Figure S17. 4f ^{13}C-NMR

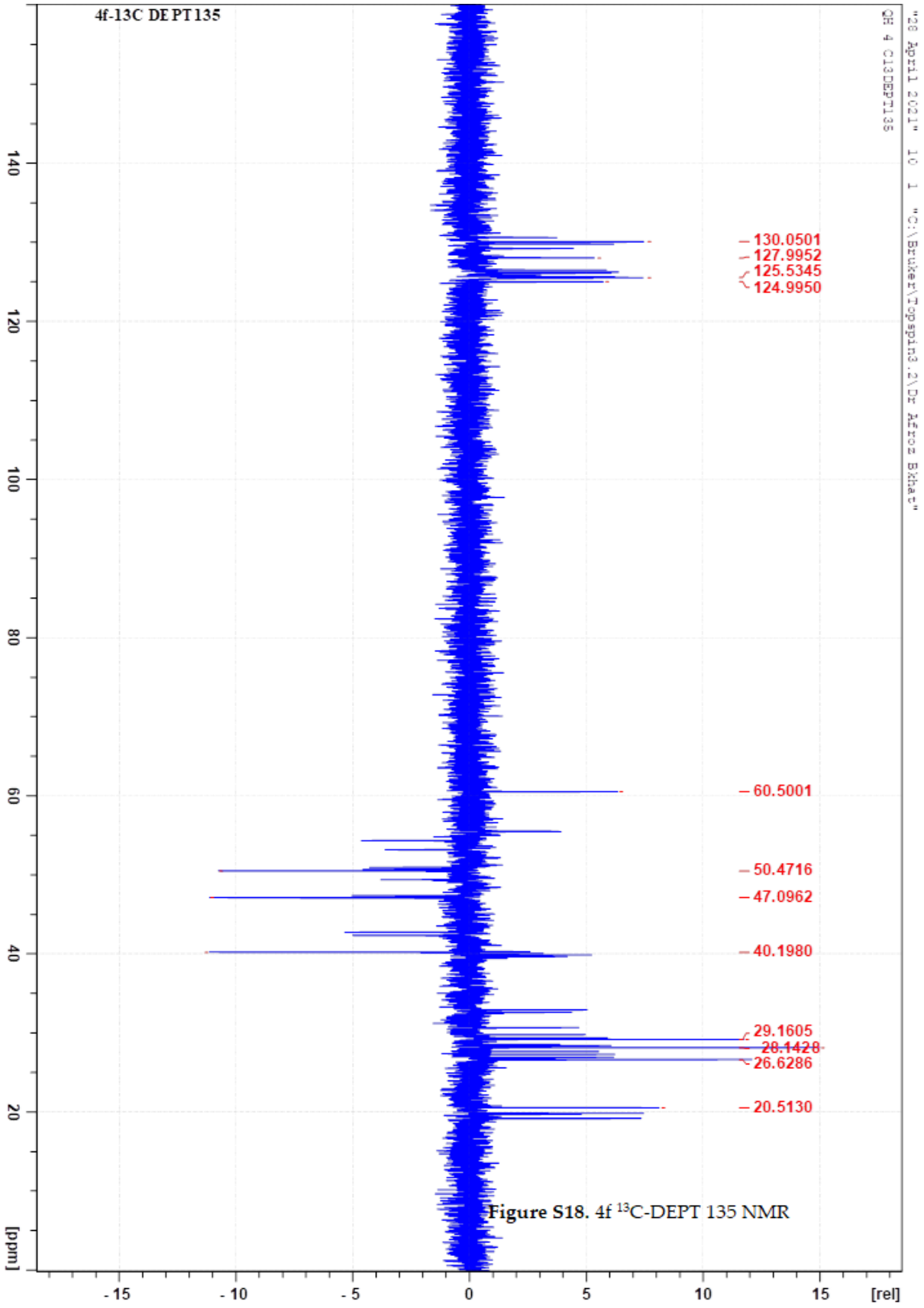

Figure S18. 4f ^{13}C-DEPT 135 NMR

Catalyst yield calculation

Yield of catalyst is simply calculated by formula

Yield (%) = Actual yield/Theoretical yield

Yield (%) considering non-ultrasonic method

- Quantity of material processed = Quantity of 25%HPW/SiO_2 + Quantity of 0.02 M $Pd(OAc)_2$
- 2.96(g) +4.48(g) = 7.44 (g) (Theoretical yield)

  Practical yield of catalyst after complete drying = 5.95 (g)

  Yield (%) = 5.95/7.44 x100

  = 79.97

  = 80%

Yield (%) considering ultrasonic method

Practical yield of catalyst after complete drying = 7.06 (g)

Yield (%) = 7.06/7.44x100

= 95 %

Effectiveness of energy consumption

Appendix 1

A.1. Energy calculations

1. Energy delivered during 20 min by ultrasonic method

- Energy delivered during sonication = Energy required to synthesized **catalyst**
- Electrical energy delivered during sonication using horn for 20 min (indicated by power meter) = 22.86 kJ.
- Efficiency of horn taken for calculation = 30% (estimated independently using calorimetric studies).
- Actual energy delivered by horn during sonication = Energy delivered during sonication using horn in 20 min x Efficiency of horn

  = 22.86 kJ x 30/100 = 6.85 kJ

  Quantity of material processed = Quantity of 25%HPW/SiO_2 + Quantity of 0.02 M $Pd(OAc)_2$

  2.96(g) +4.48(g) = 7.44 (g)

- Net energy supplied for processing of material using sonochemical method = Actual energy delivered by horn during sonication/Quantity of material processed

  6.85kJ/ 7.44(g) = 0.92 (kJ/g) ---------- (A1)

2. Energy Delivered During non-ultrasonic method

- Voltage input in magnetic stirrer (Fisher Scientific, Model C188618443308, Made in China) = 220 V.

- Current measured using digital multimeter (Model KYORITSU, Made in Japan) = 39 mA = 39 $x10^{-3}$ (A).

  Power input in magnetic stirrer = Voltage input x Current measured

  220(V) x 39 $x10^{-3}$ (A) = 8.5 W (J/s)

- Efficiency of magnetic stirrer taken for the calculation = 40% (estimated independently using calorimetric studies).
- Actual power input in overhead stirrer = Power input in magnetic stirrer (W) x 40/100

  8.5(W) x 40/100 = 3.4 W (J/s)
- Time required for completion of reaction = 1h (3600 s).
- Net energy delivered during non-ultrasonic method = Power input in magnetic stirrer x Time required for completion of reaction
- 3.4 J/s x 1h x 3600(s/h) = 12,240 J= 12.24 kJ ---------- (A2)
- Quantity of material processed = Quantity of 25%HPW/SiO_2 + Quantity of 0.02 M $Pd(OAc)_2$
- 2.96(g) +4.48(g) = 7.44 (g)
- Net energy supplied for processing of material using non-ultrasonic method = Net energy delivered during non-ultrasonic method /quantity of material processed

  12.24 kJ/ 7.44 = 1.64 (kJ/g) ---------- (A3)

3. Energy Saved

- Net Energy saved = (Net energy supplied for processing of material using non-ultrasonic method – (Net energy supplied for processing of material using ultrasonic method