Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

Supporting Information Part 2/2 (SI2/2) for

Accessing Distributions of Exchange and Dipolar Couplings in Stiff Molecular Rulers with Cu(II) centres

K. Keller,[a] I. Ritsch,H. Hintz,[b] M. Hülsmann,[b] M. Qi,[b] F. Breitgoff,[a] D. Klose,[a] Y. Polyhach,[a,*] M. Yulikov,[a,*] A. Godt,[b,*] G. Jeschke[a]

[a]*Laboratory of Physical Chemistry, Department of Chemistry and Applied Biosciences, ETH Zurich, Vladimir-Prelog-Weg 2, 8093 Zurich, Switzerland.*
[b]*Faculty of Chemistry and Center for Molecular Materials (CM_2), Bielefeld University, Universitätsstraße 25, 33615 Bielefeld, Germany*

NMR spectra

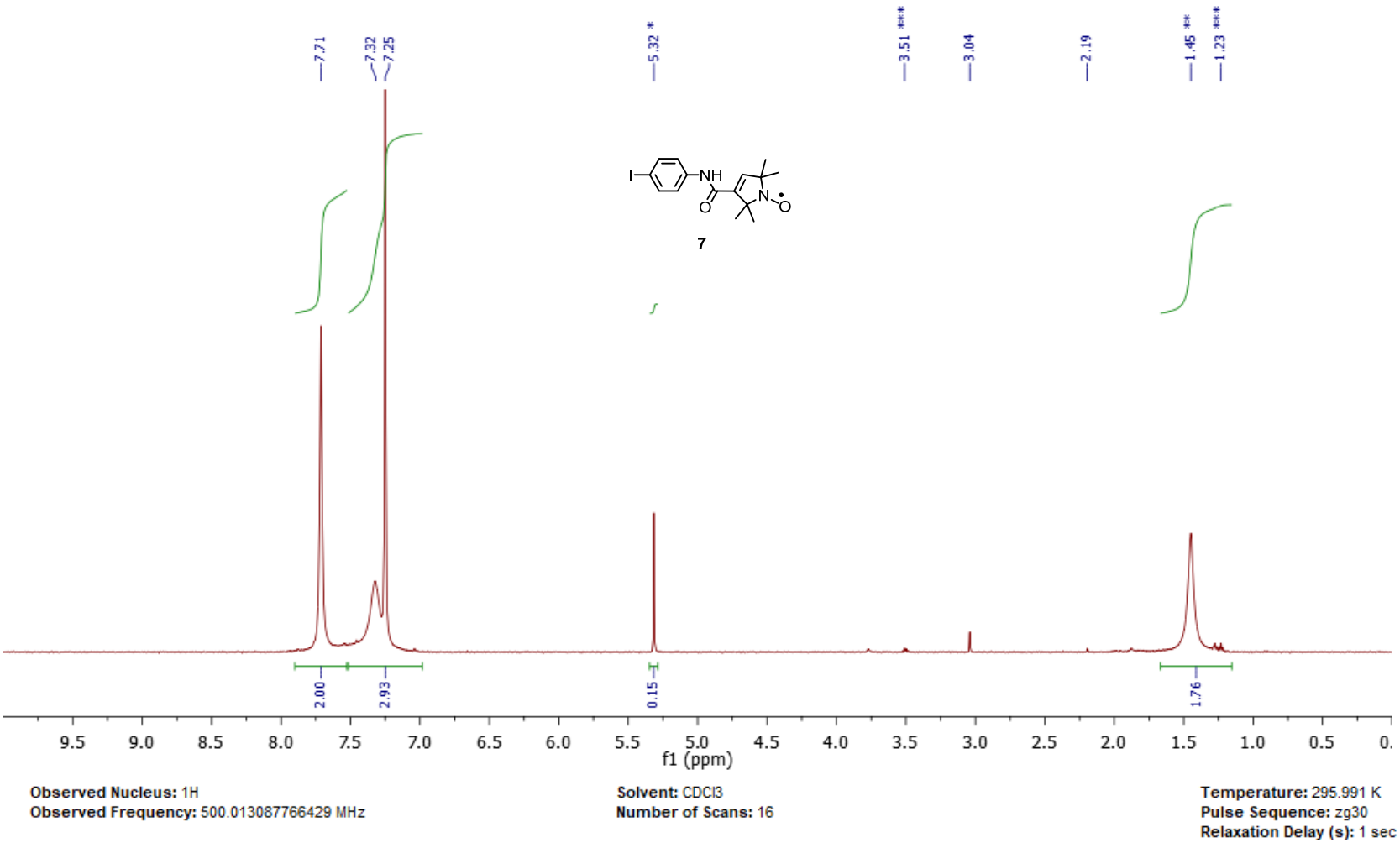

Figure S5. ^{1}H NMR spectrum of I-P-NO• **7**. *CH_2Cl_2, **water, ***Et_2O.

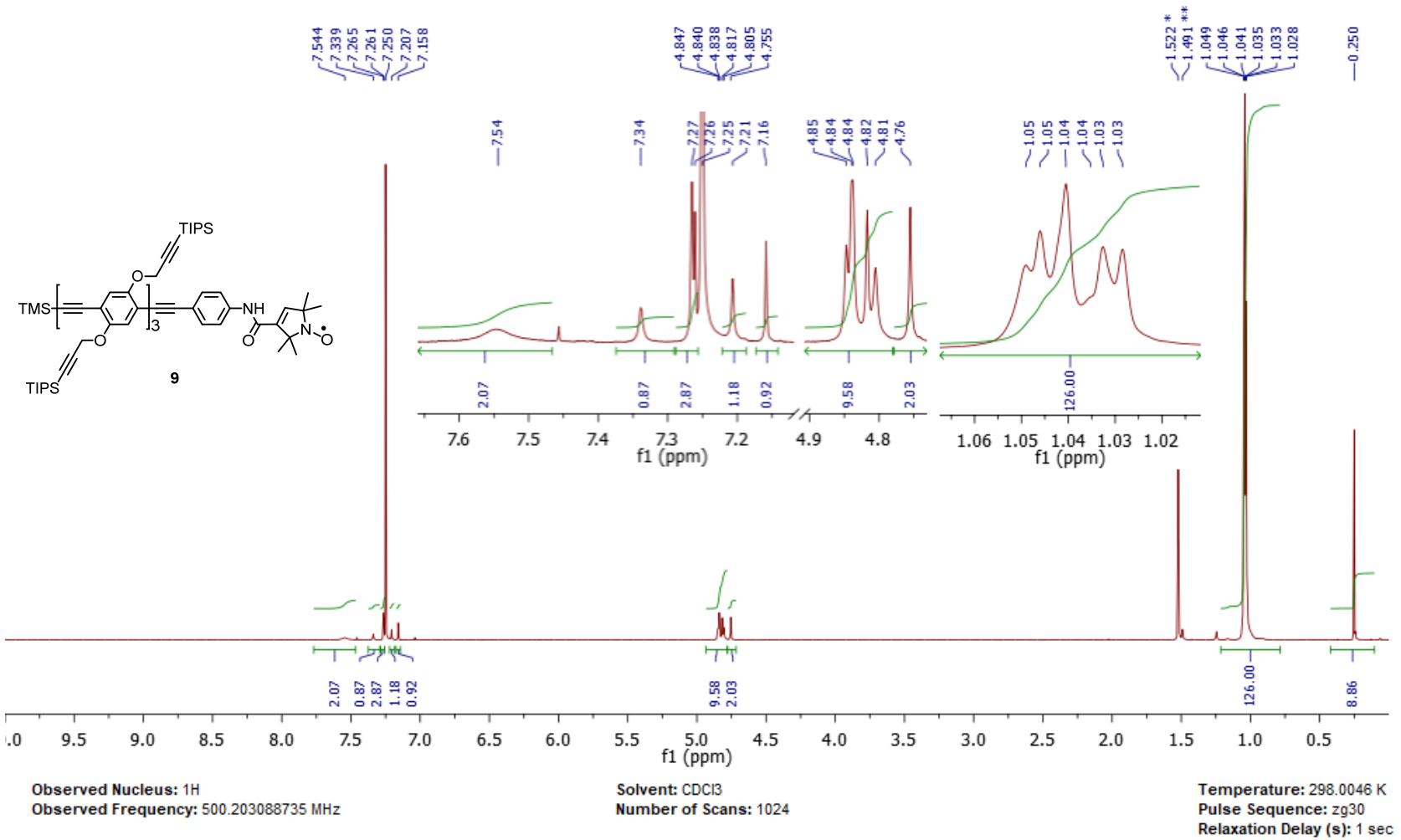

Figure S6. ^{1}H NMR spectrum of TMS-(EP)$_4$-NO• **9**. *H_2O, **HDO.

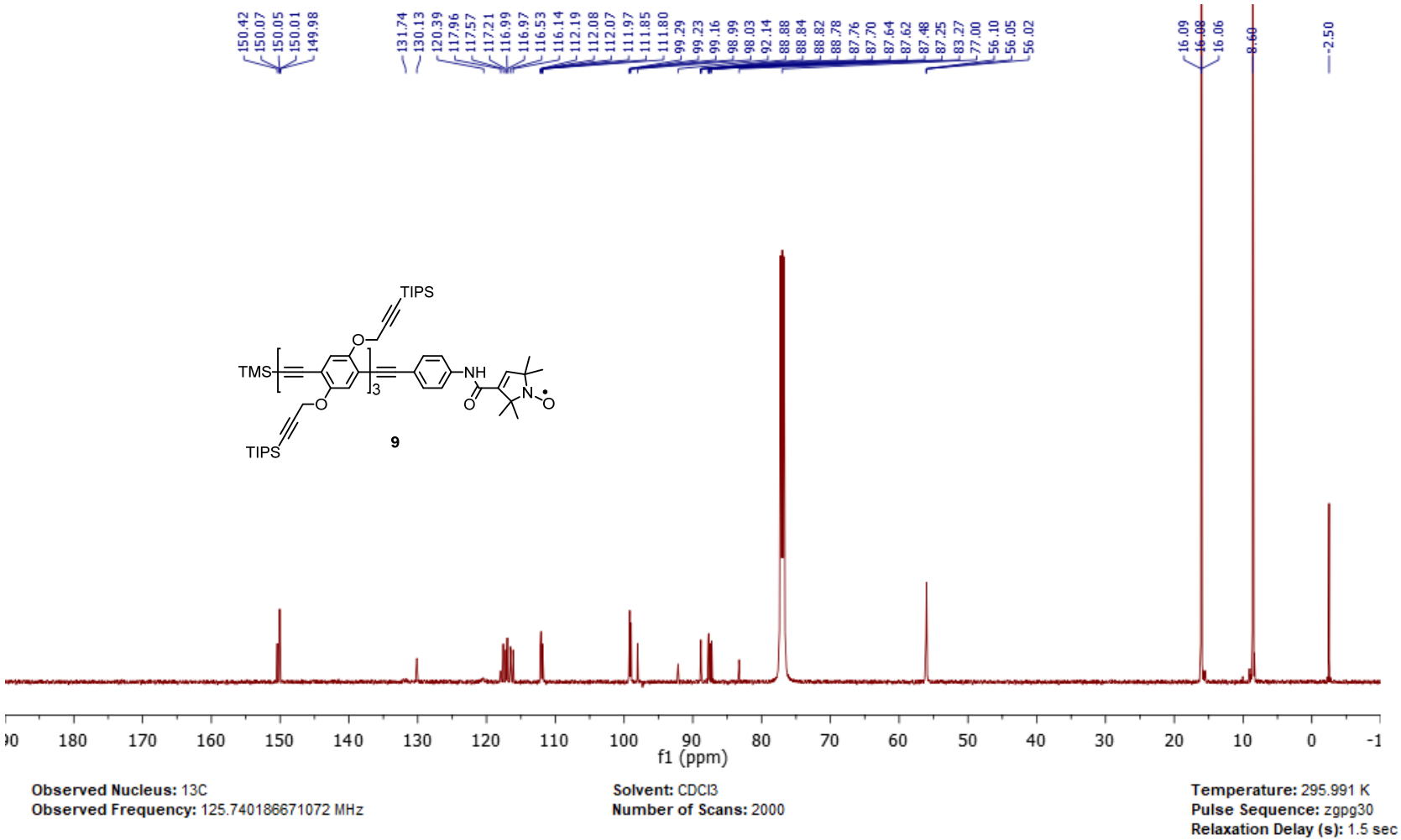

Figure S7. ^{13}C NMR spectrum of TMS-$(EP)_4$-NO• **9**.

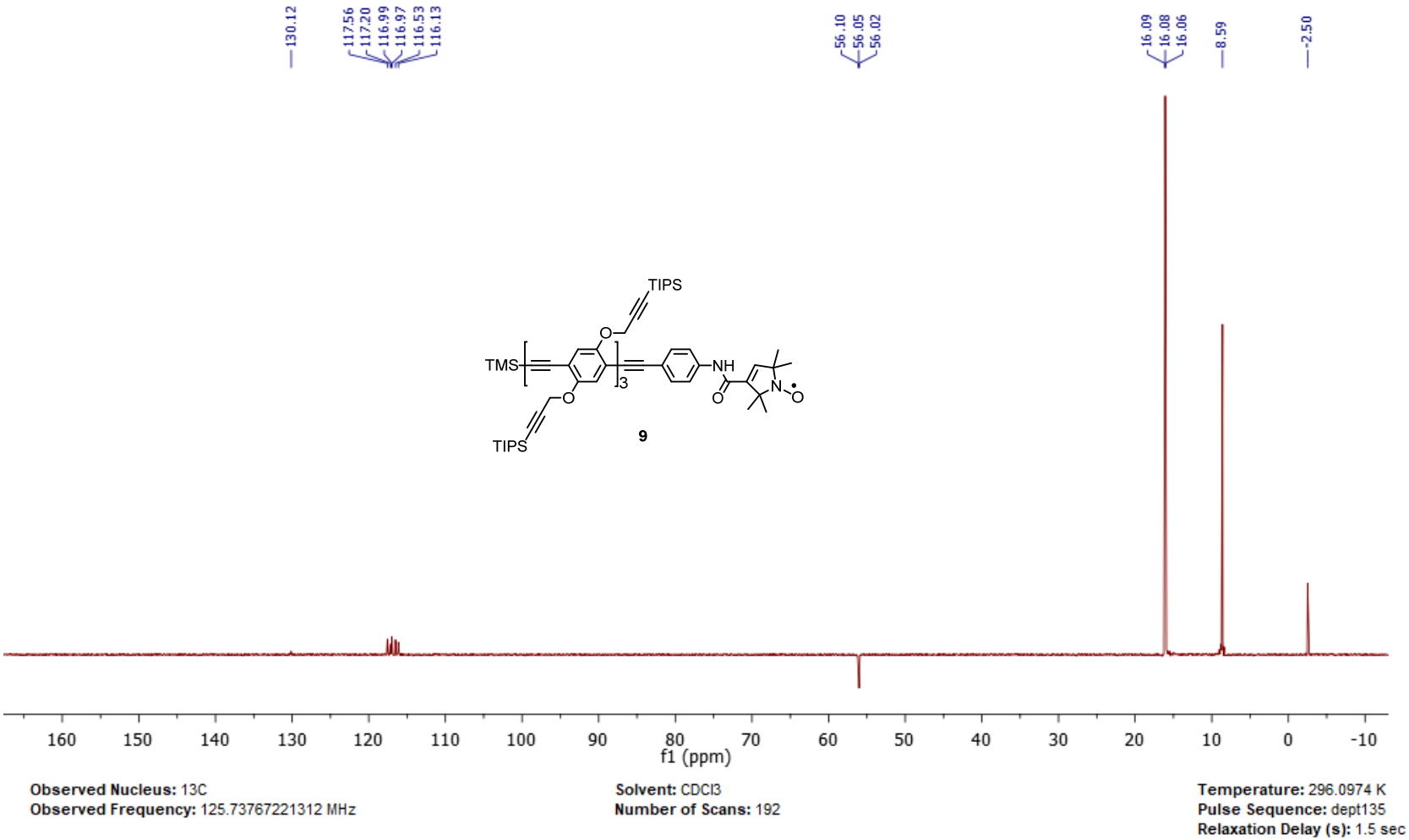

Figure S8. DEPT 135 NMR spectrum of TMS-(EP)$_4$-NO• **9**.

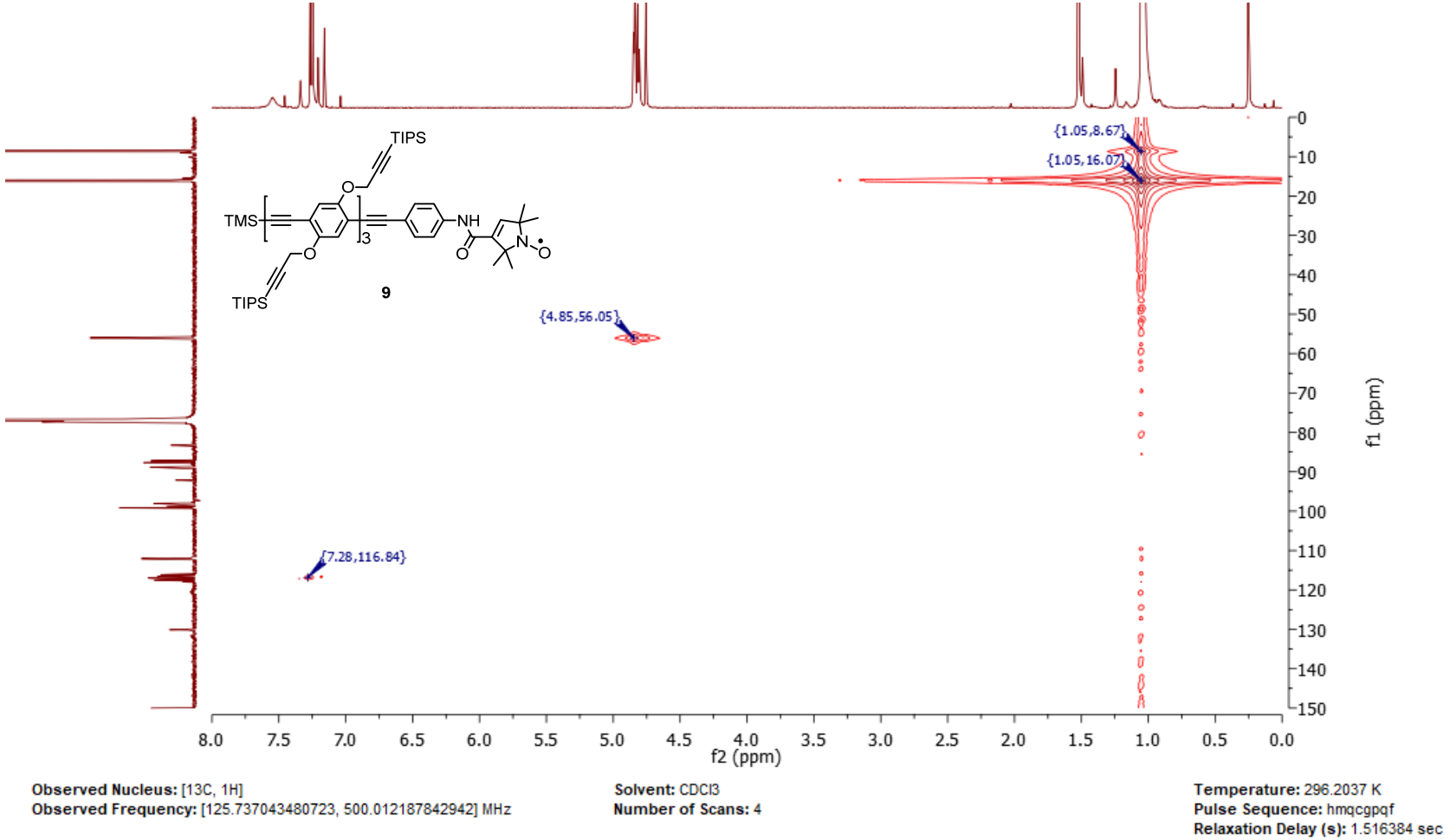

Figure S9. HMQC NMR spectrum of TMS-(EP)$_4$-NO• **9**.

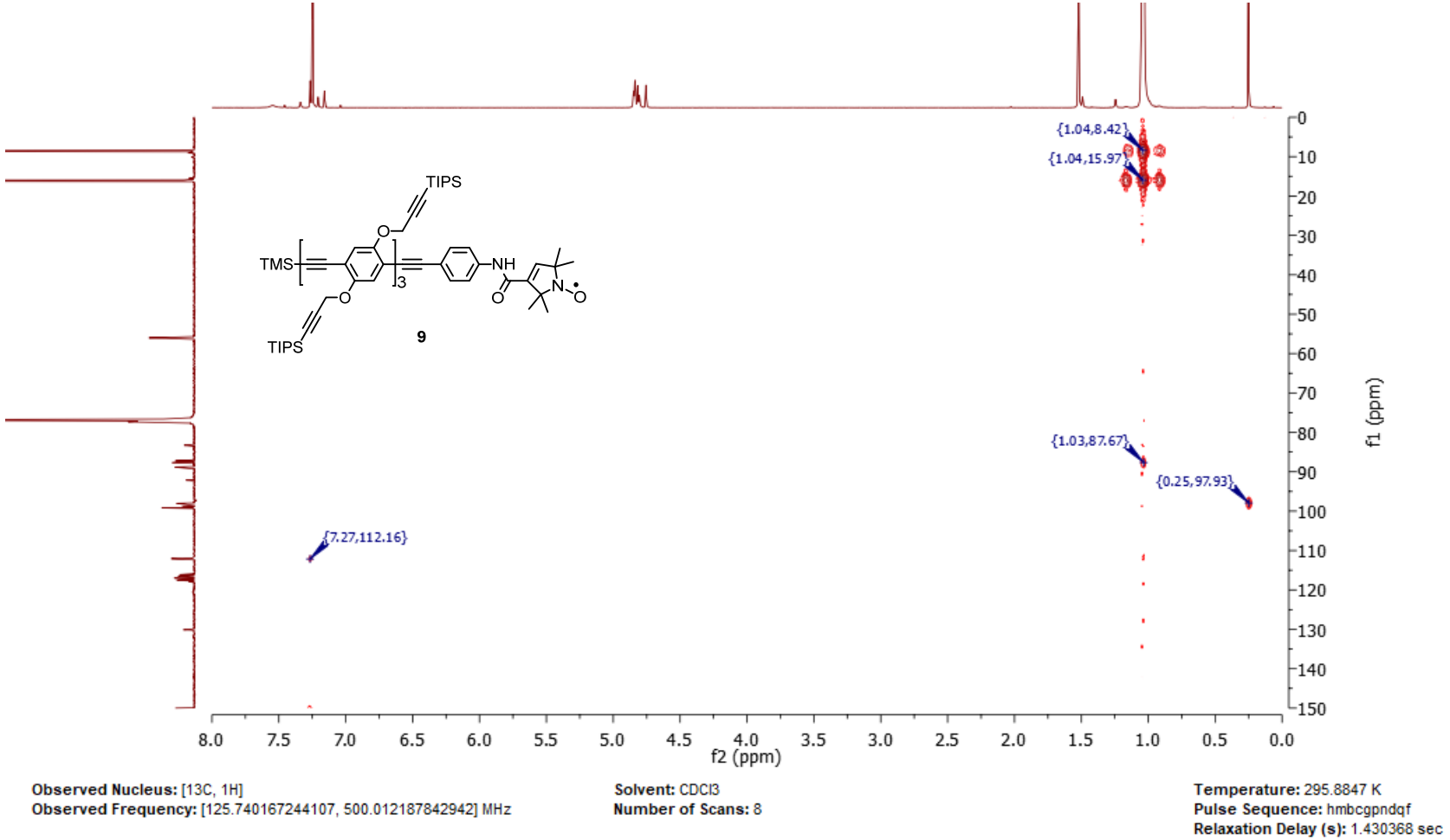

Figure S10. HMBC NMR spectrum of TMS-(EP)$_4$-NO• **9**.

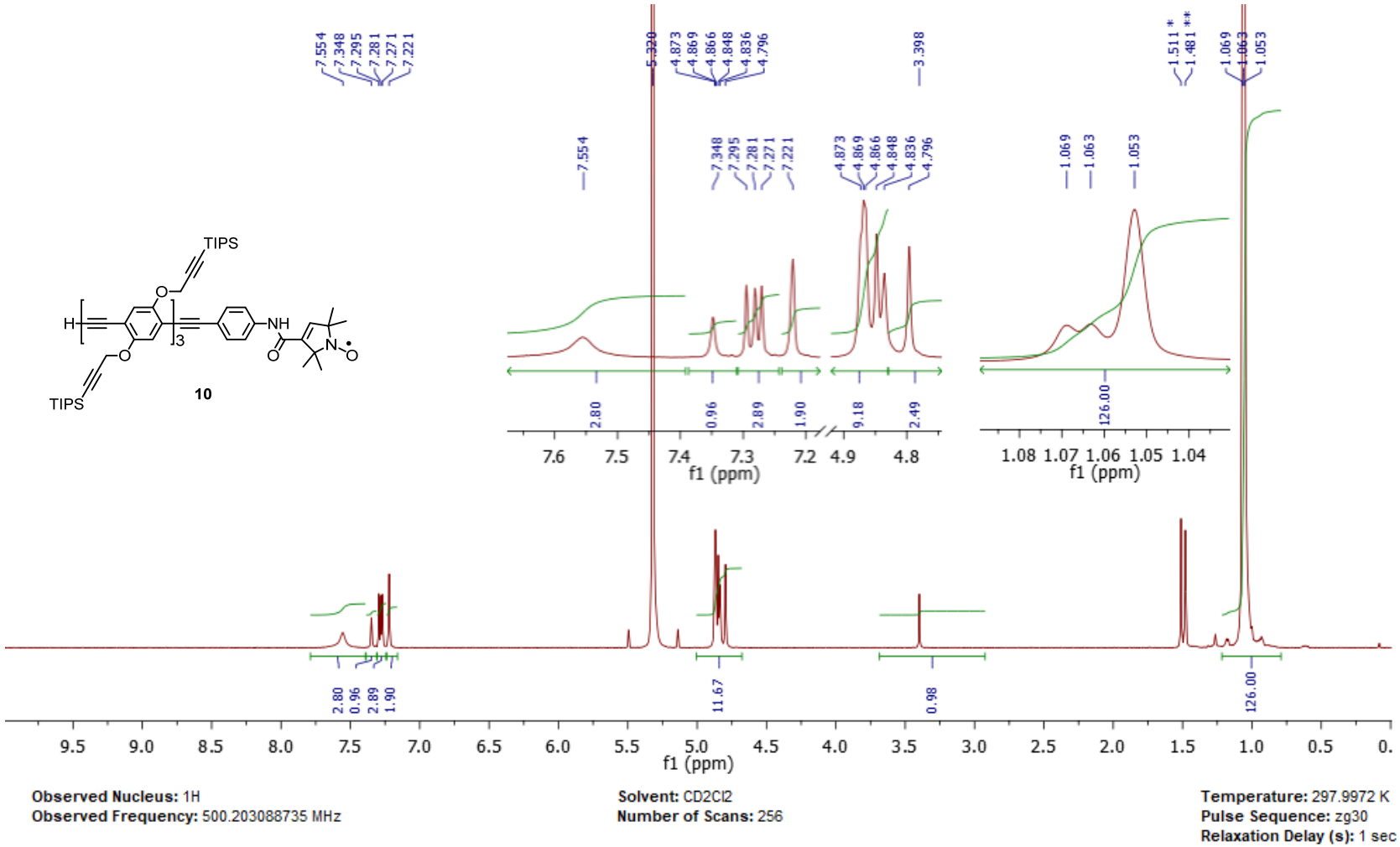

Figure S11. ^{1}H NMR spectrum of H-(EP)$_4$-NO• **10**. *H_2O, **HDO.

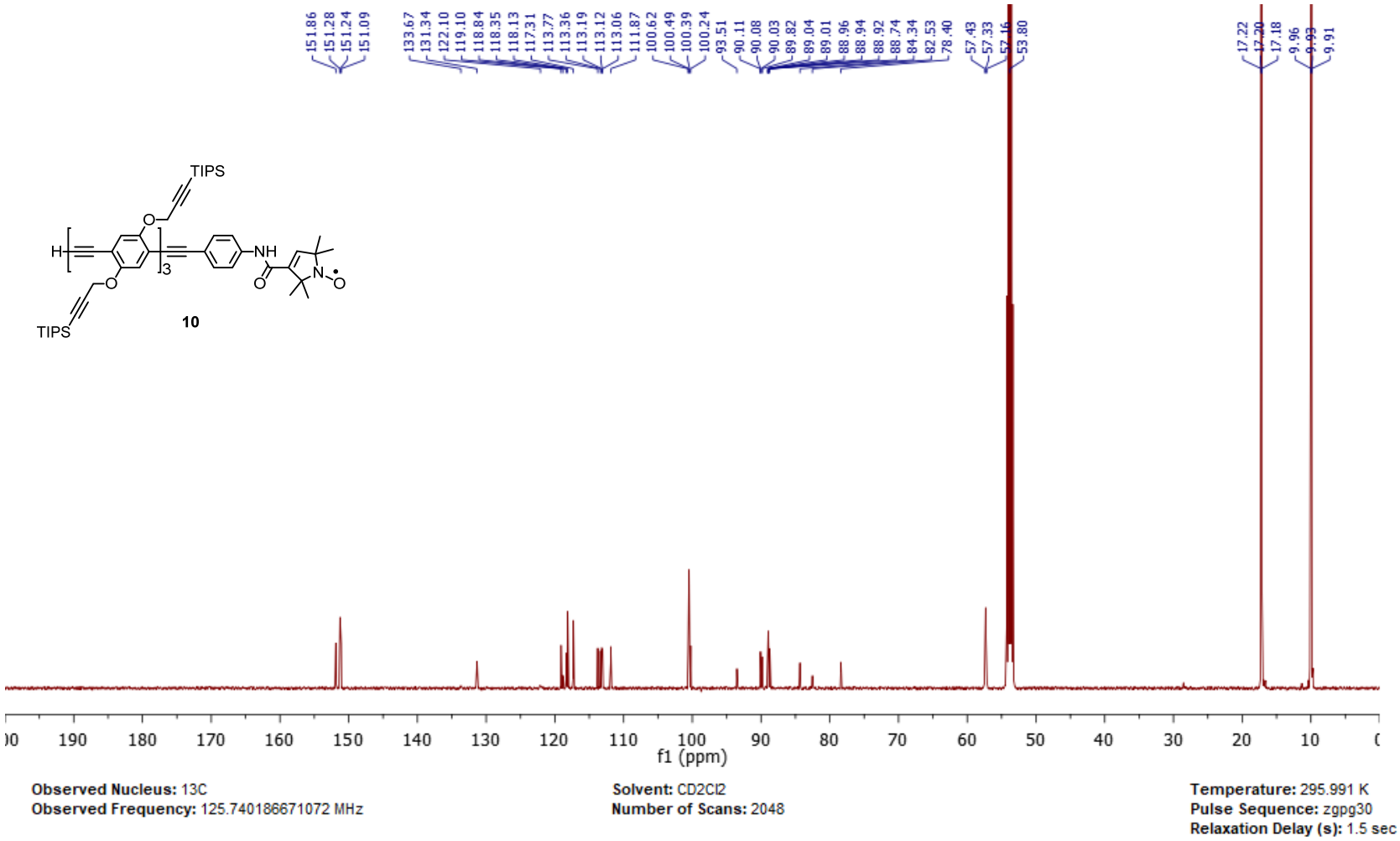

Figure S12. ^{13}C NMR spectrum of H-(EP)$_4$-NO• **10**.

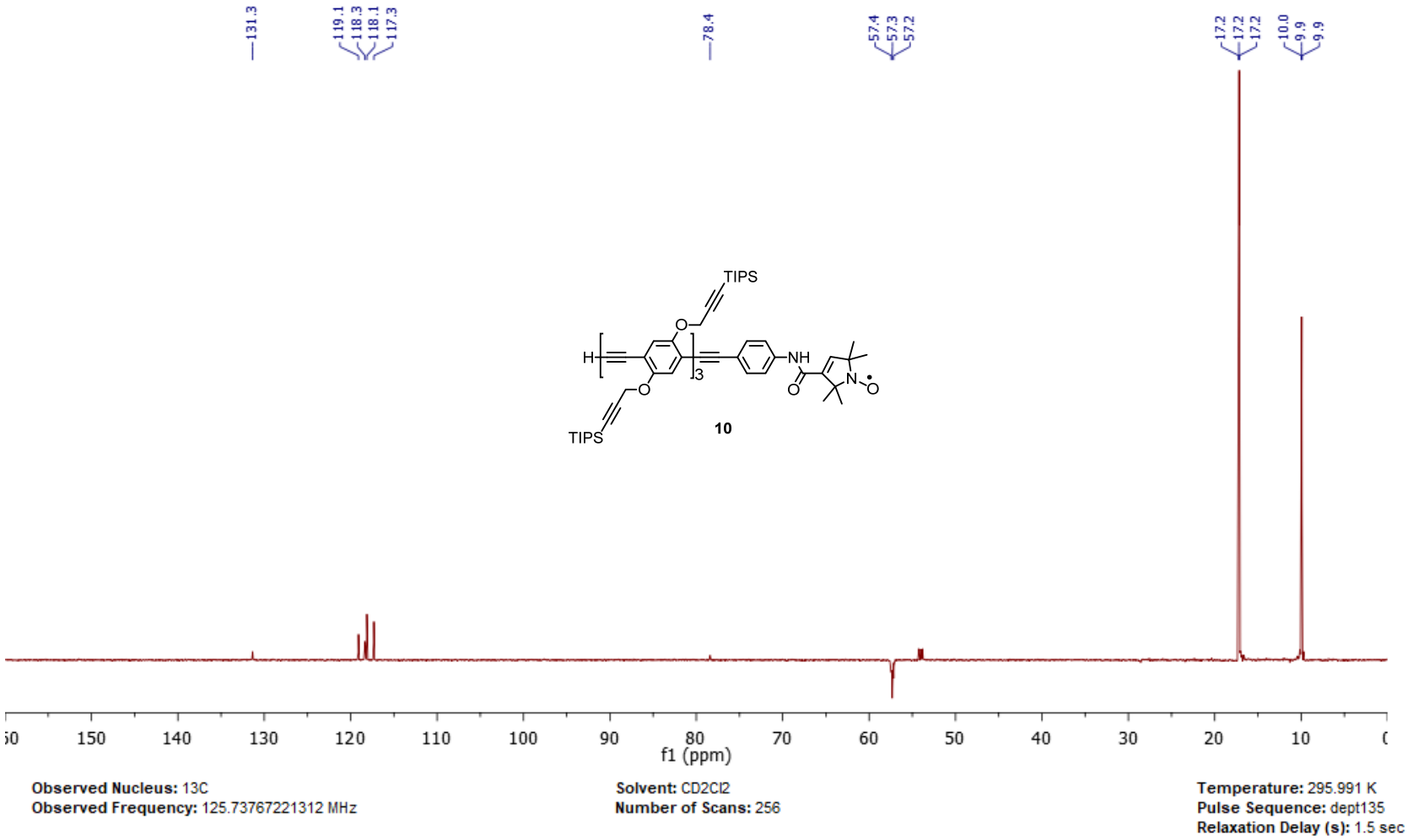

Figure S13. DEPT-135 NMR spectrum of H-(EP)$_4$-NO• **10**.

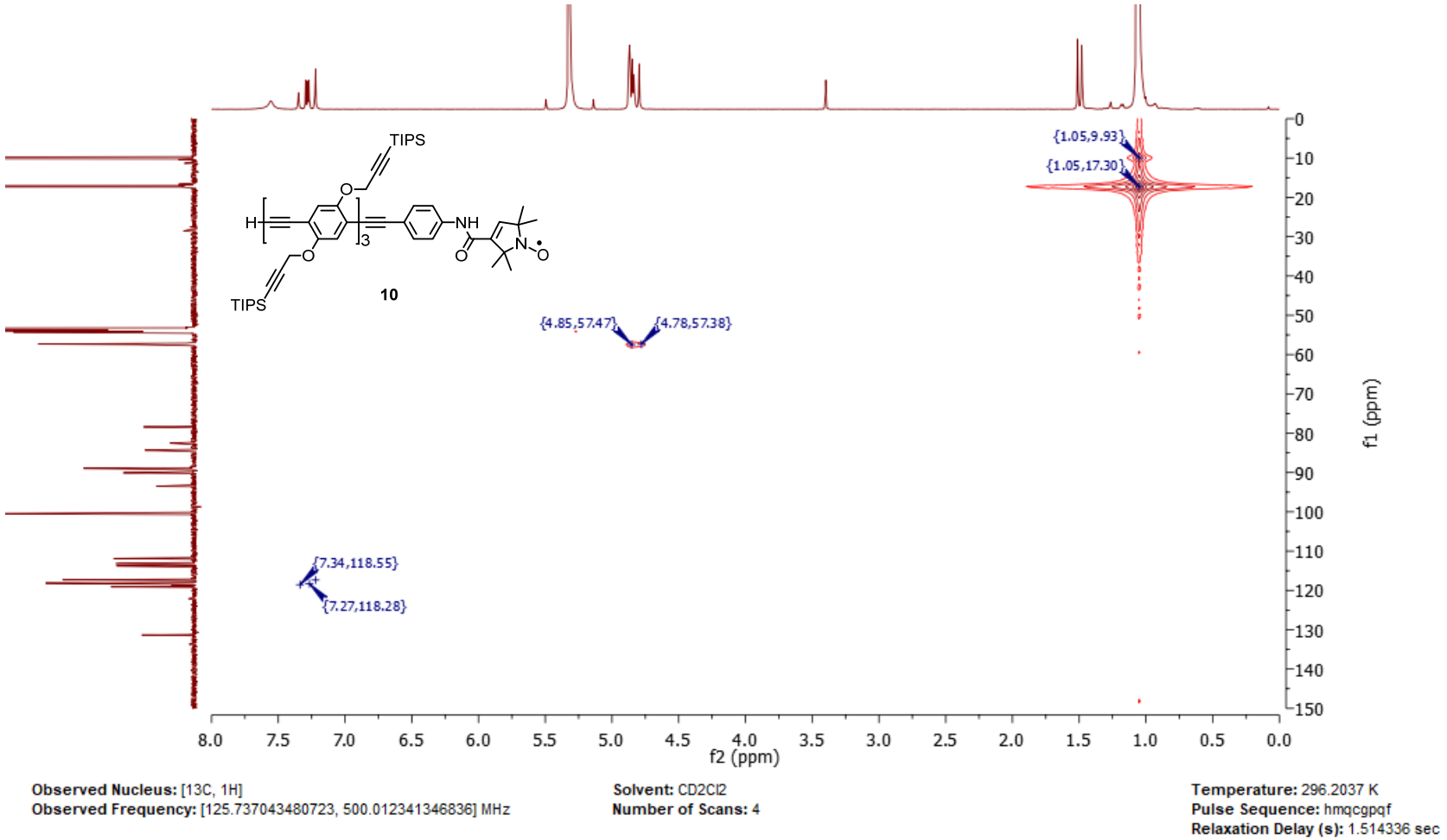

Figure S14. HMQC NMR spectrum of H-(EP)$_4$-NO• **10**.

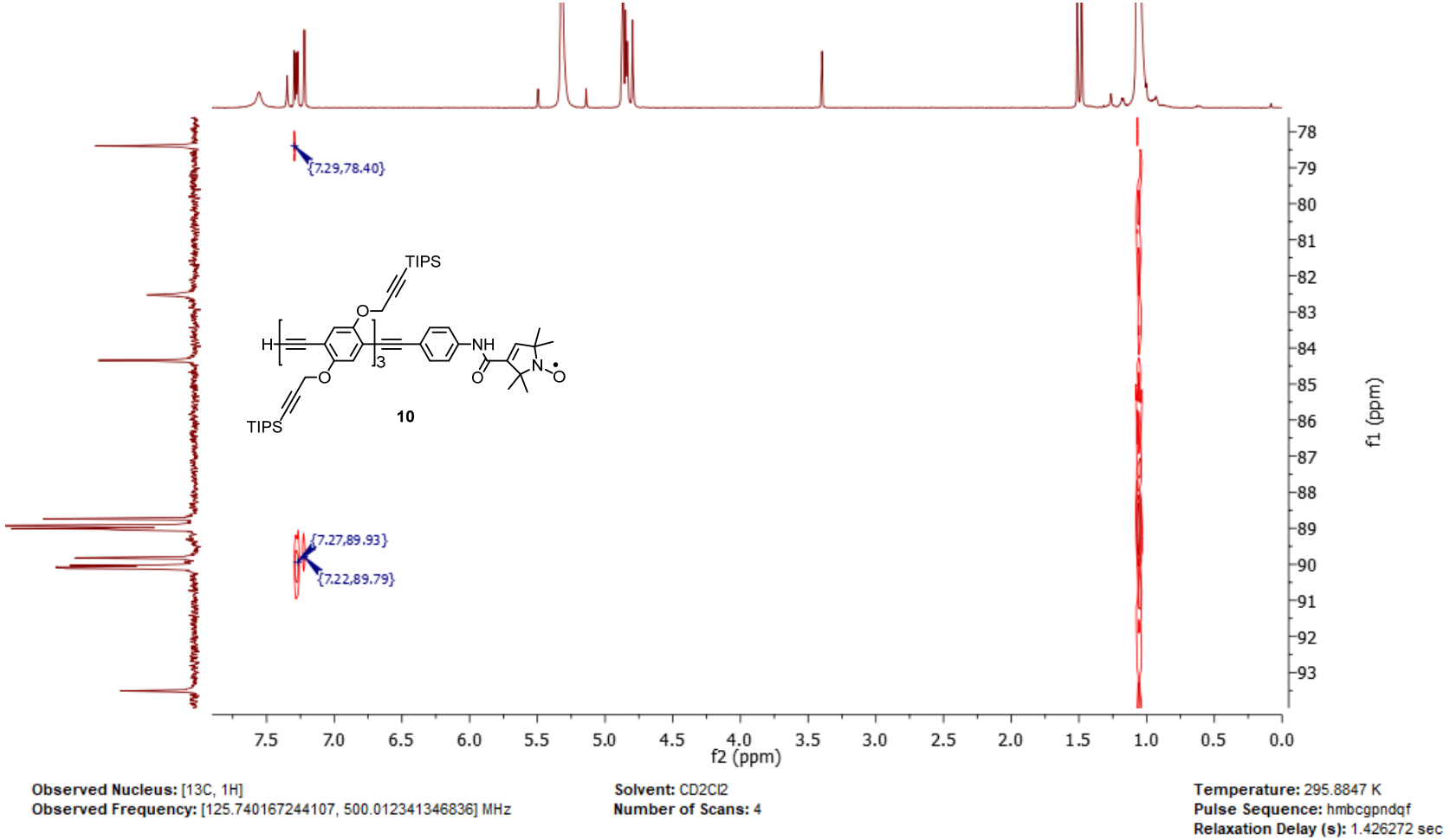

Figure S15. HMBC NMR spectrum of H-(EP)$_4$-NO• **10**.

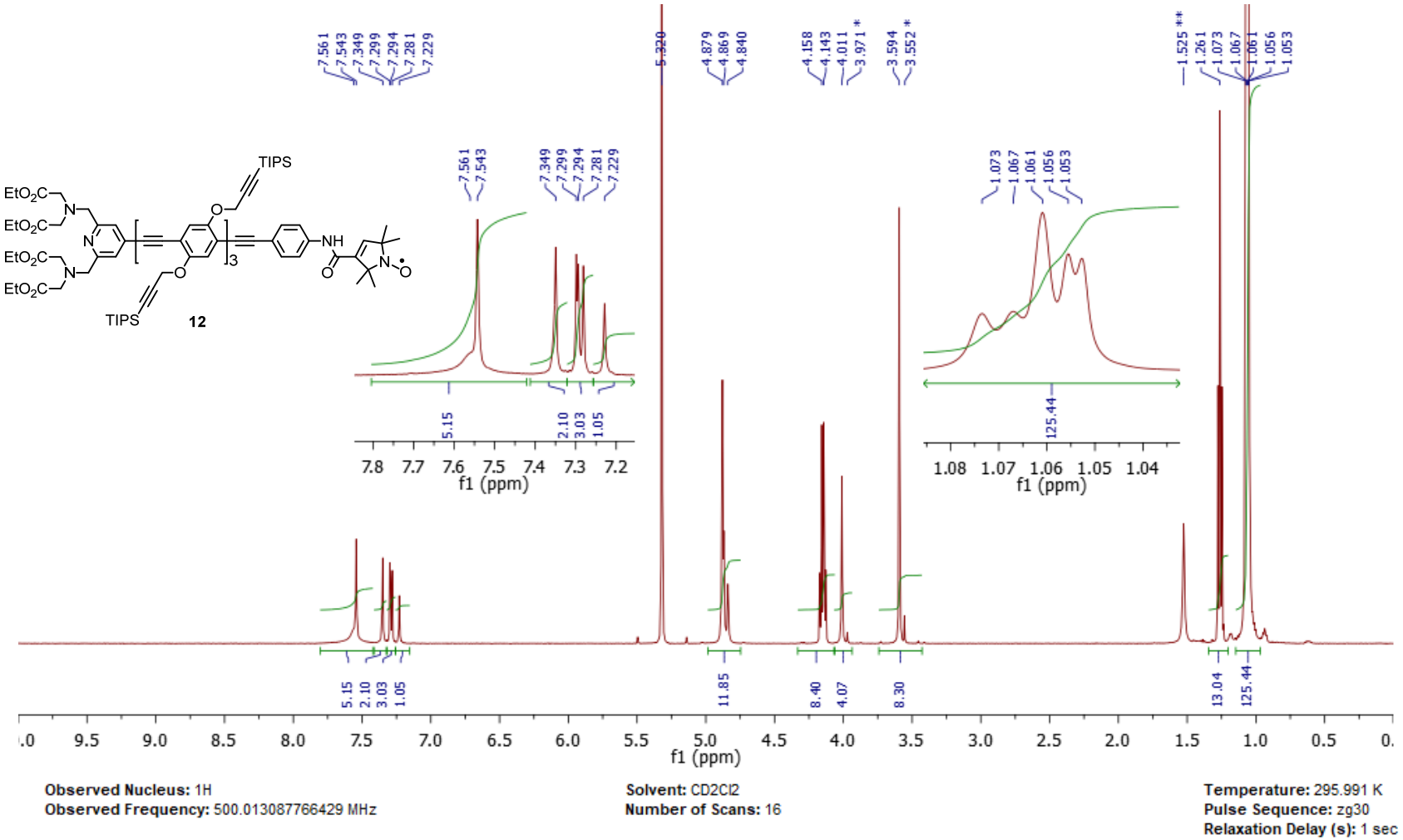

Figure S16. ^{1}H NMR spectrum of PyMTAester-(EP)$_4$-NO• **12**. *Signals of a compound containing the PyMTA ethyl ester moiety (about 5 mol%), possibly 4-chloro-PyMTA ethyl ester; **water.

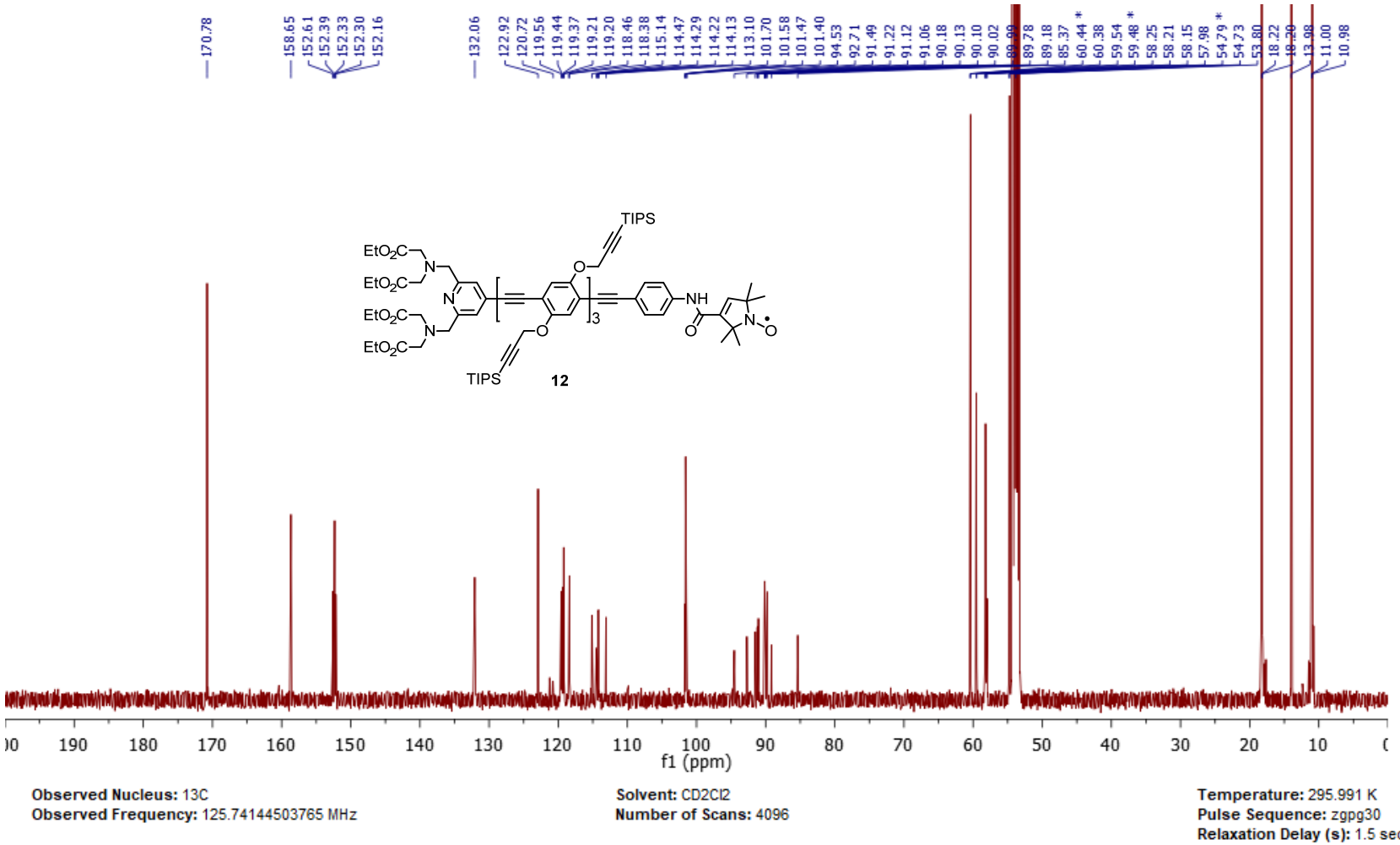

Figure S17. ^{1}H NMR spectrum of PyMTAester-(EP)$_4$-NO• **12**. *Signals of a compound containing the PyMTA ethyl ester moiety, possibly 4-chloro-PyMTA ethyl ester.

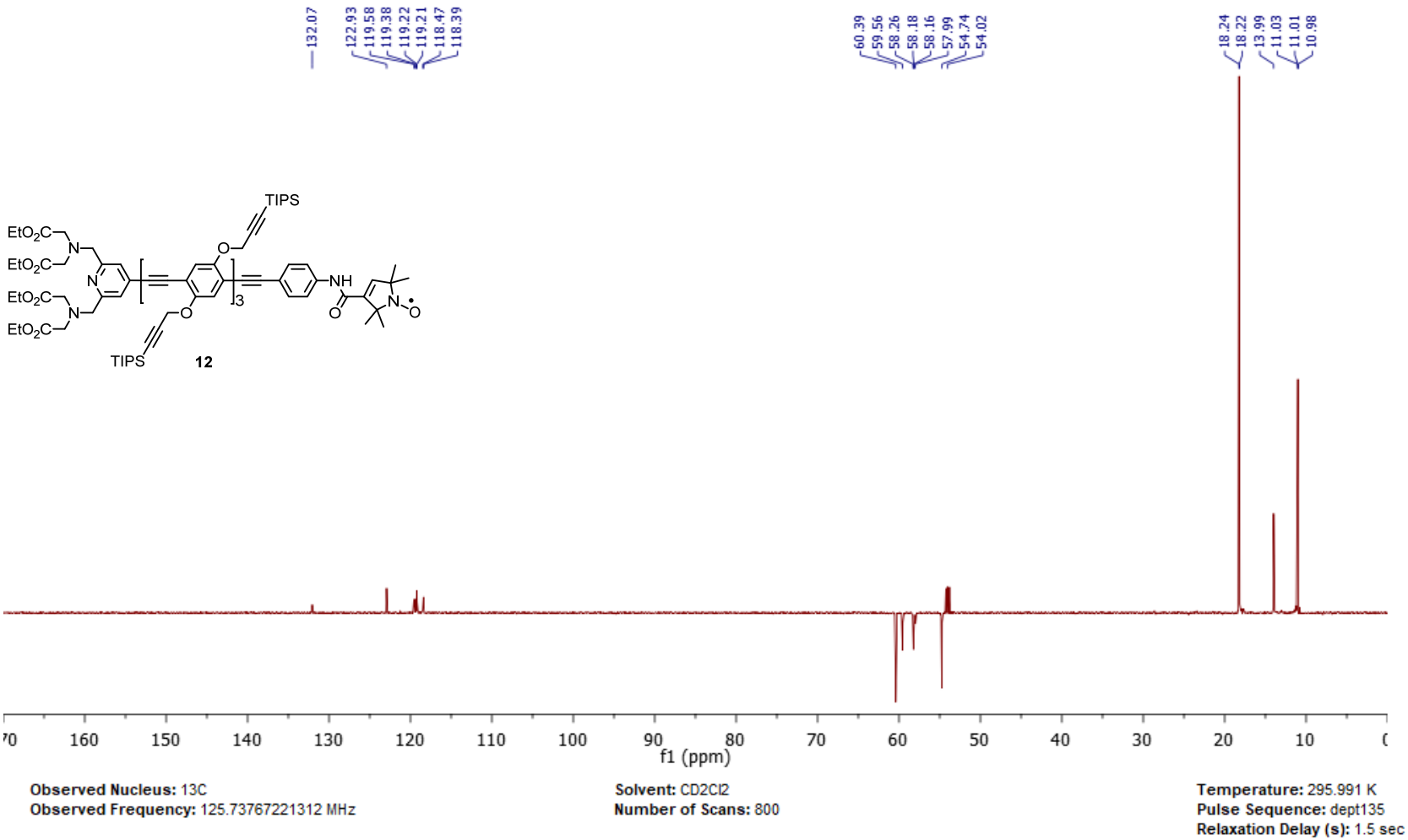

Figure S18. DEPT-135 NMR spectrum of PyMTAester-$(EP)_4$-NO• **12**.

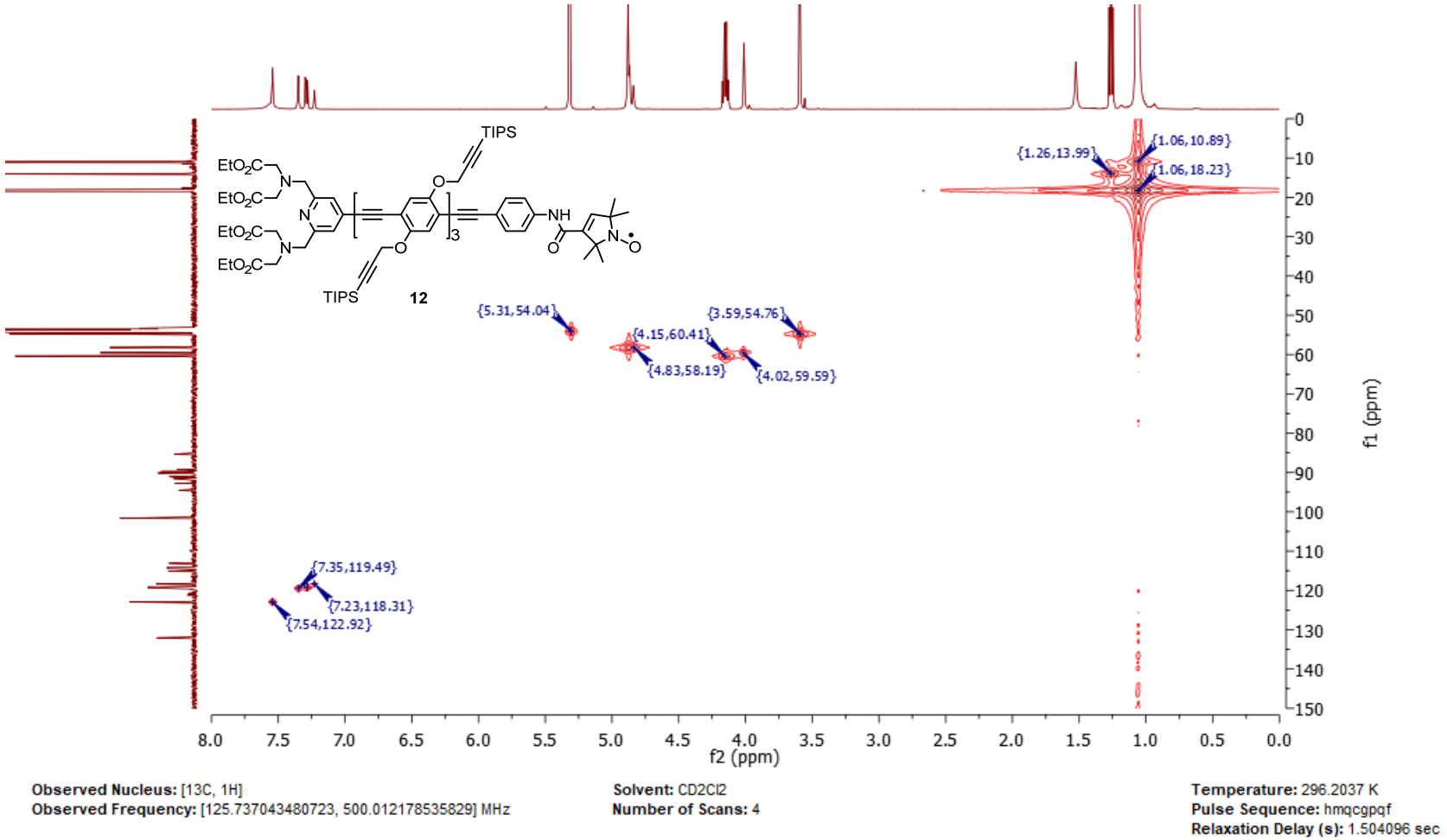

Figure S19. HMQC NMR spectrum of PyMTAester-$(EP)_4$-NO• **12**.

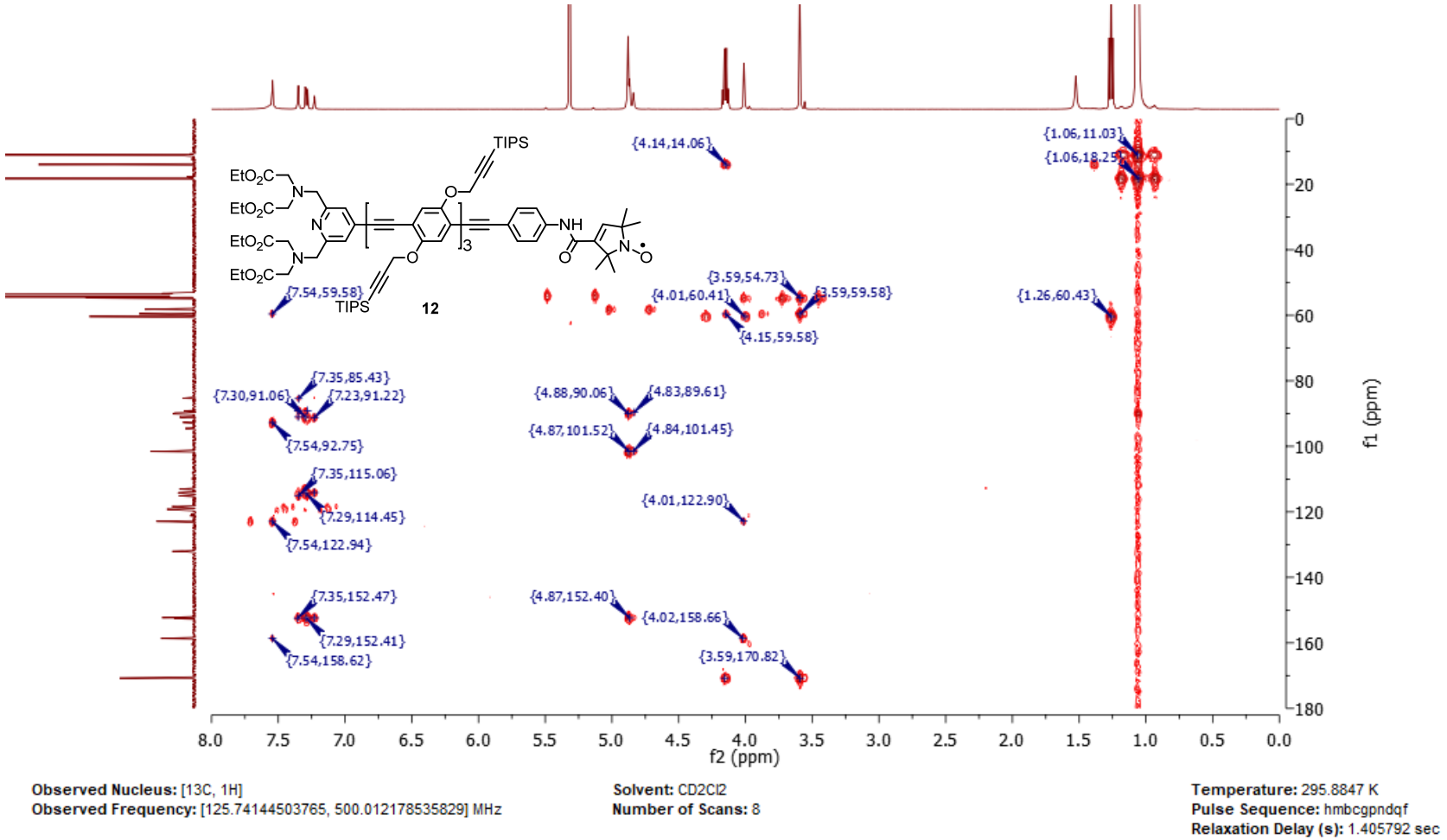

Figure S20. HMBC NMR spectrum of PyMTAester-$(EP)_4$-NO• **12**.

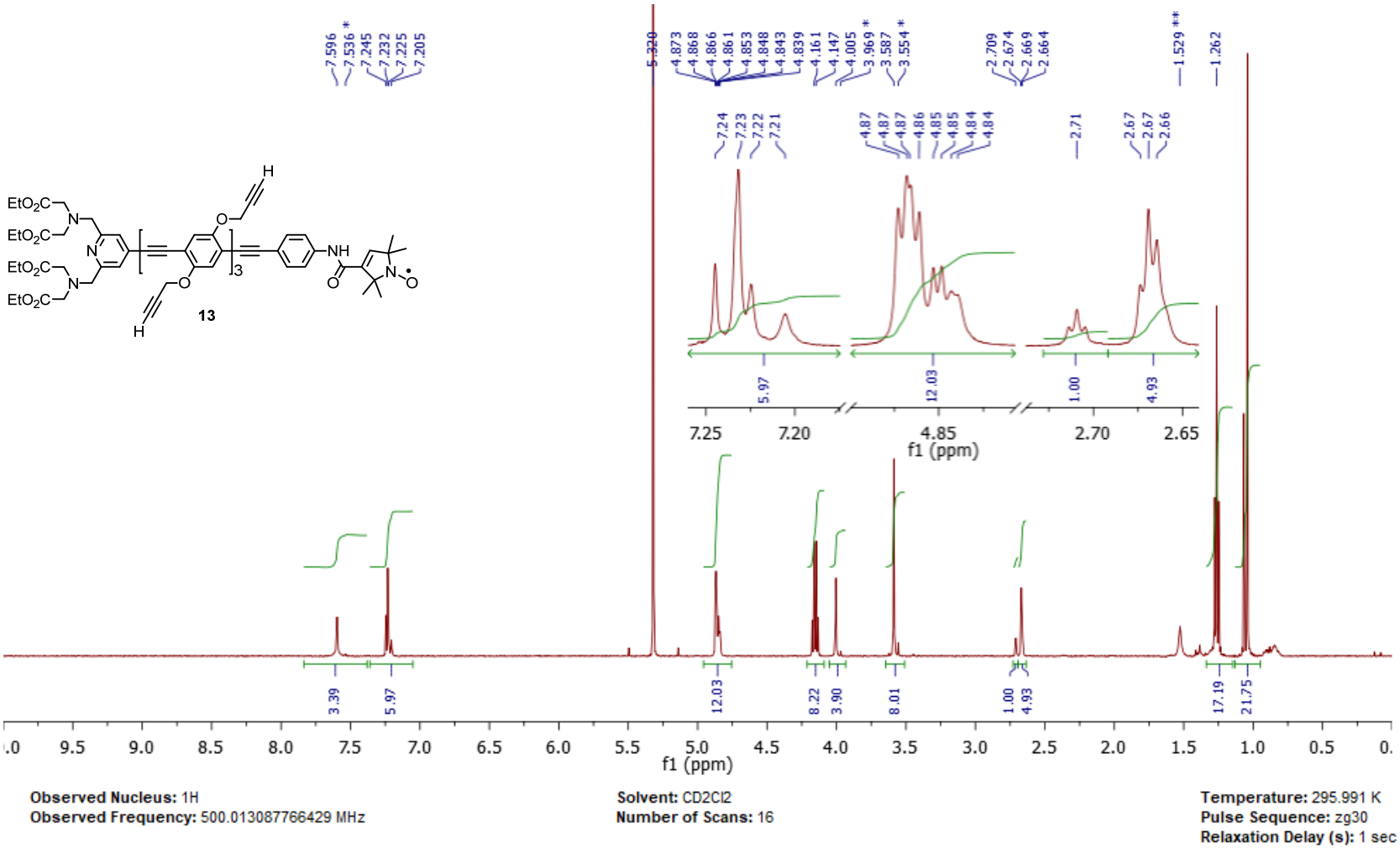

Figure S21. ^{1}H NMR spectrum of desilylated PyMTAester-(EP)$_4$-NO• **13**. *Signals of a compound containing the PyMTA ethyl ester, possibly 4-chloro-PyMTA ethyl ester. **water

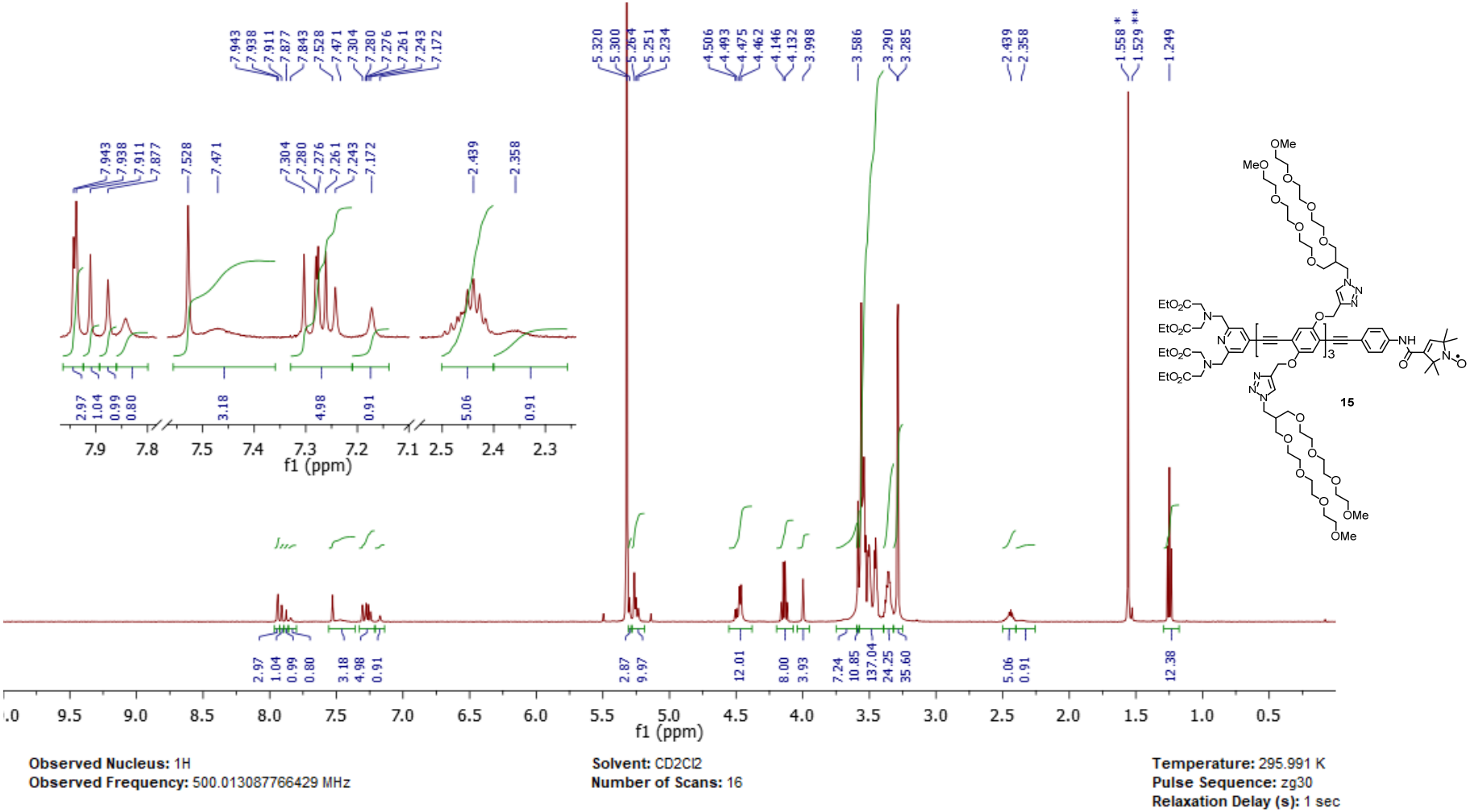

Figure S22. ^{1}H NMR spectrum of PEGylated PyMTAester-(EP)$_4$-NO• **15**. *H_2O, **HDO.

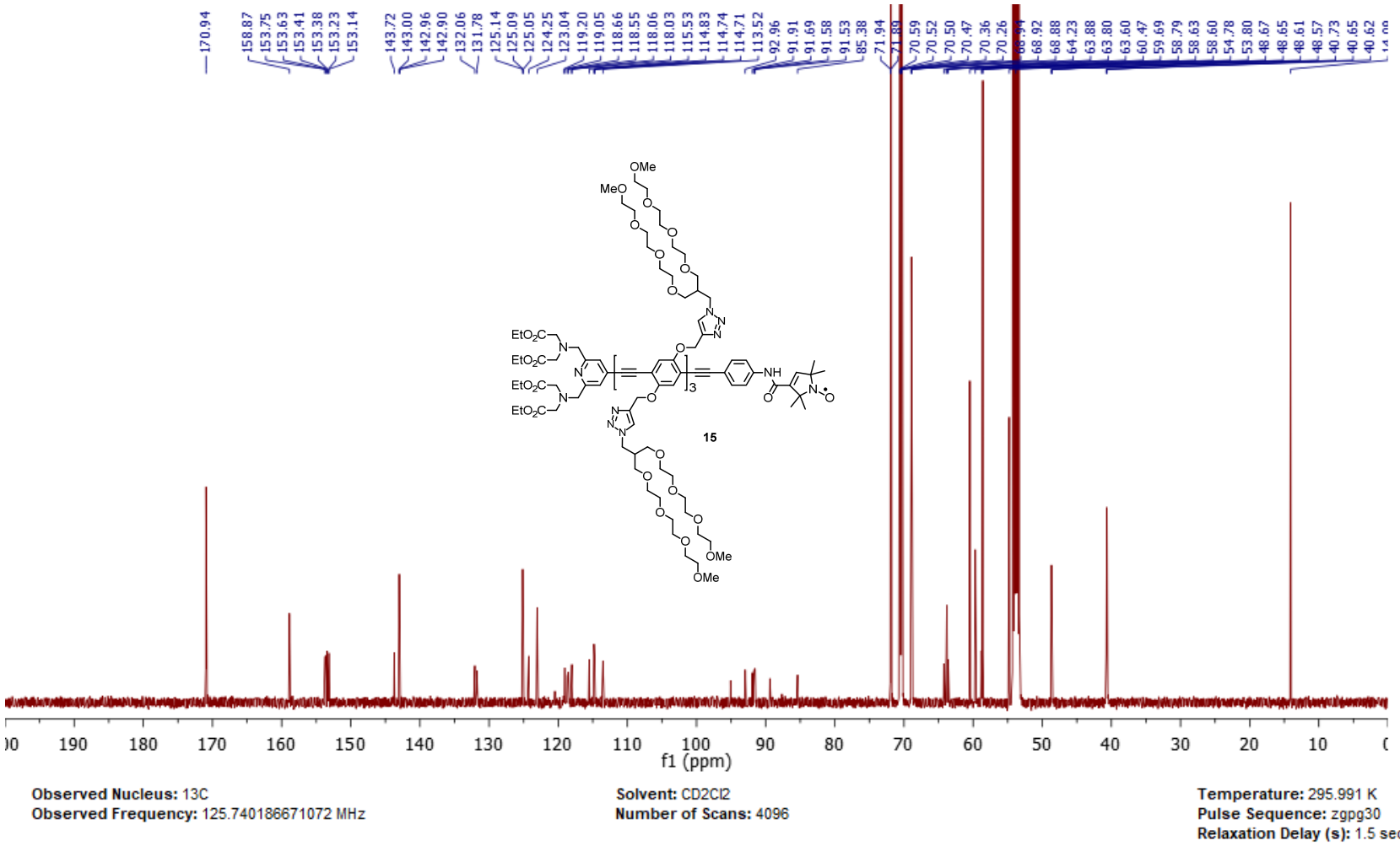

Figure S23. ^{13}C NMR spectrum of PEGylated PyMTAester-(EP)$_4$-NO• **15**.

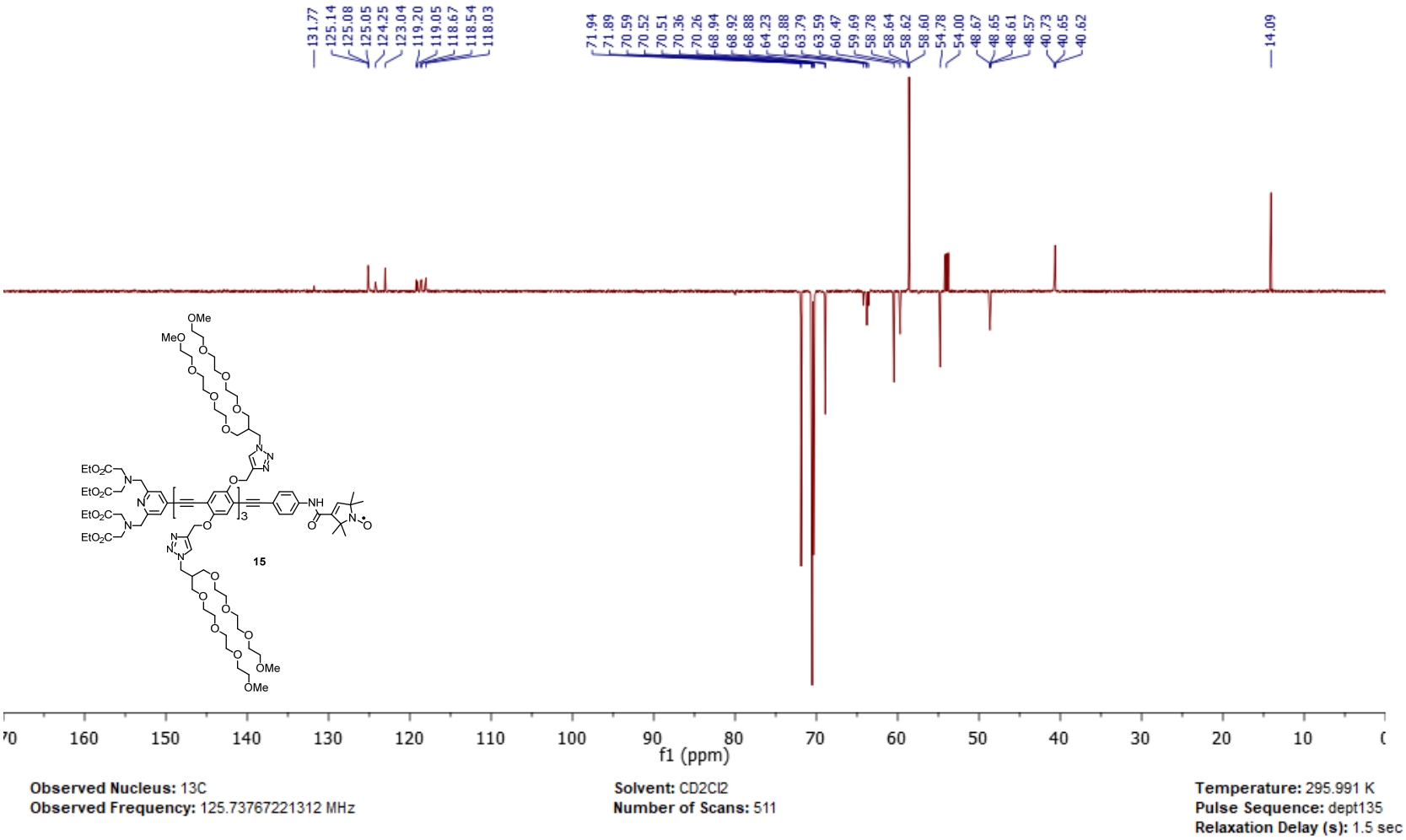

Figure S24. DEPT-135 NMR spectrum of PEGylated PyMTAester-$(EP)_4$-NO• **15**.

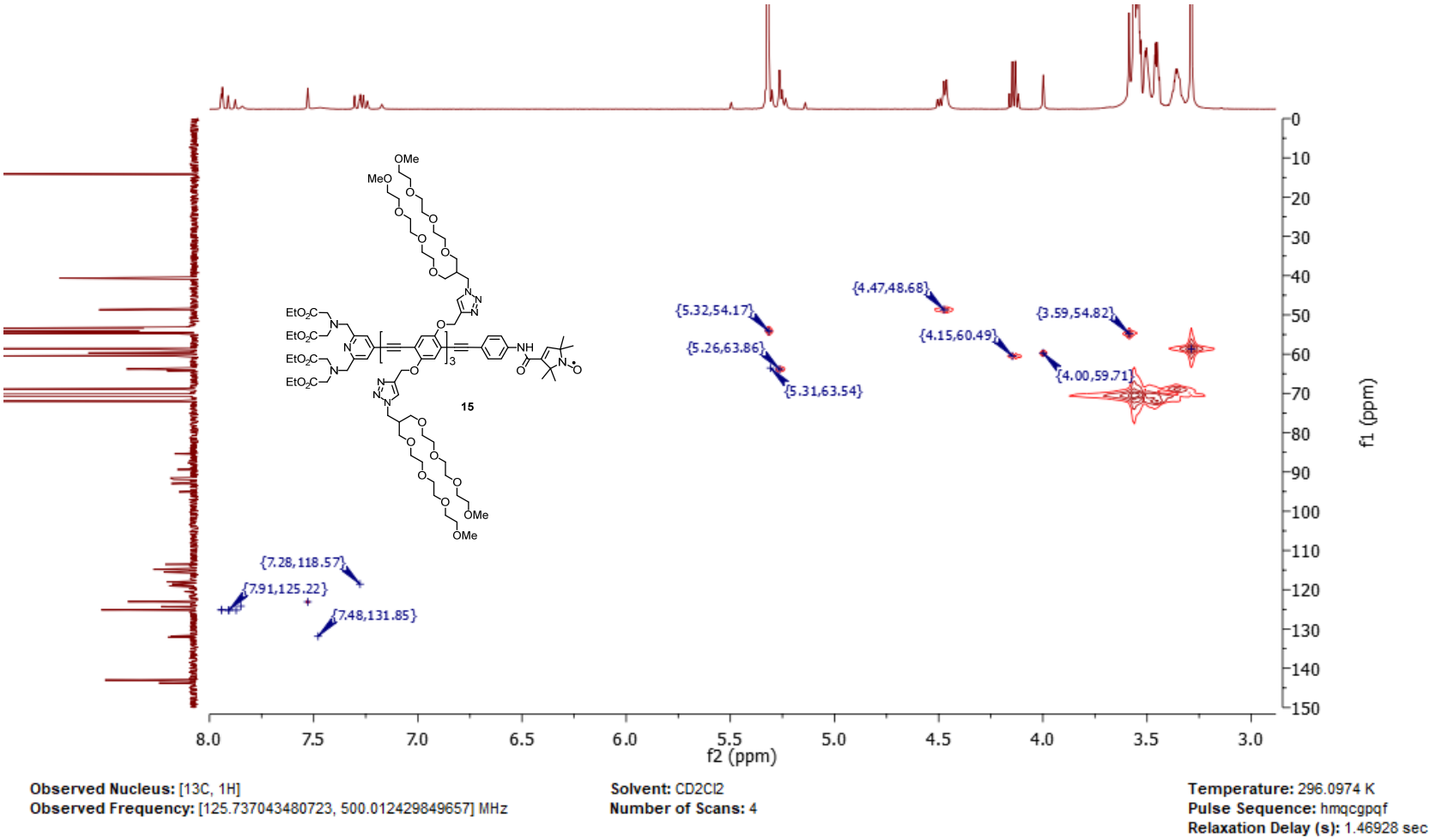

Figure S25. HMQC NMR spectrum of PEGylated PyMTAester-(EP)$_4$-NO• **15**.

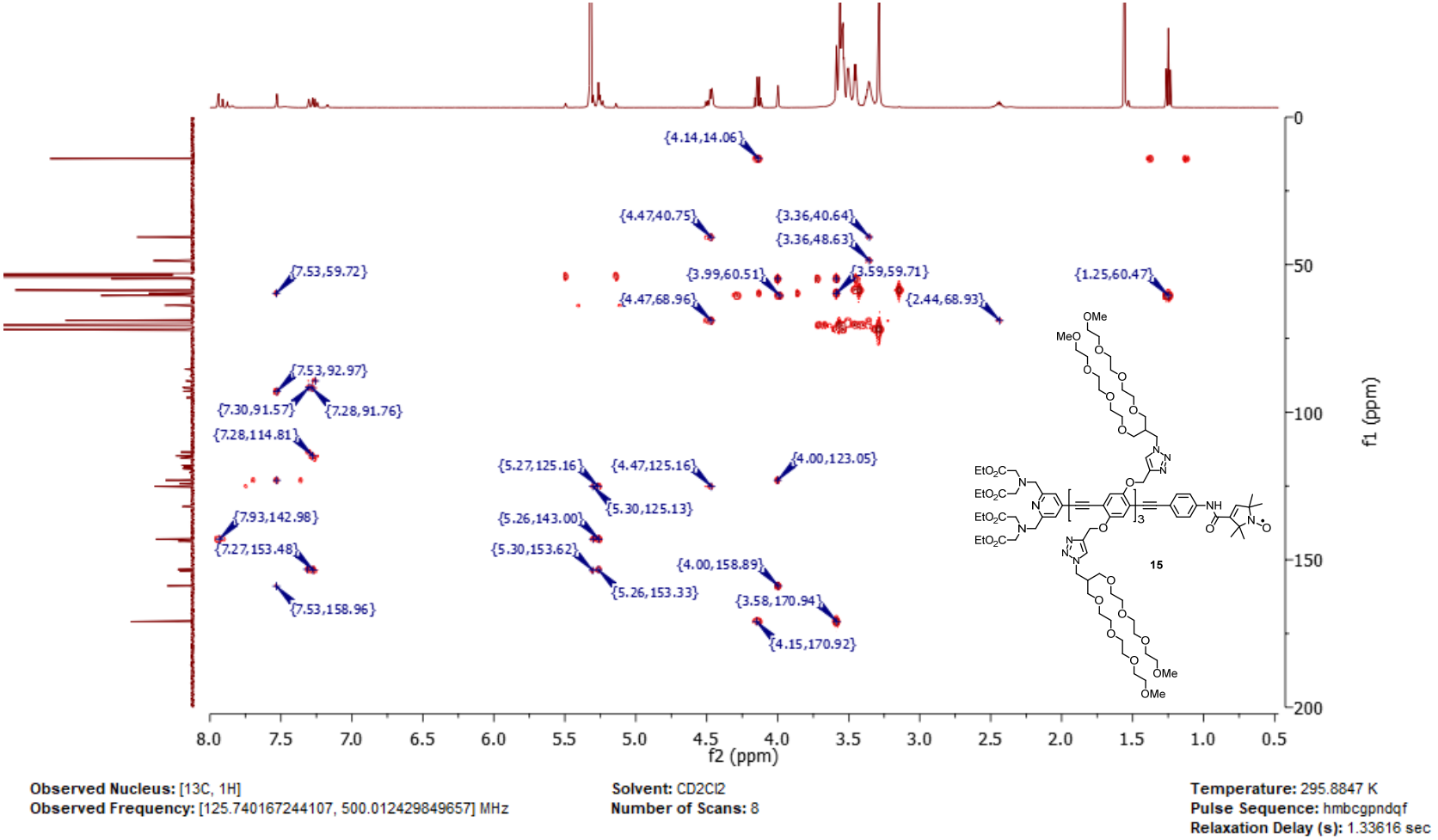

Figure S26. HMBC NMR spectrum of PEGylated PyMTAester-$(EP)_4$-NO• **15**.

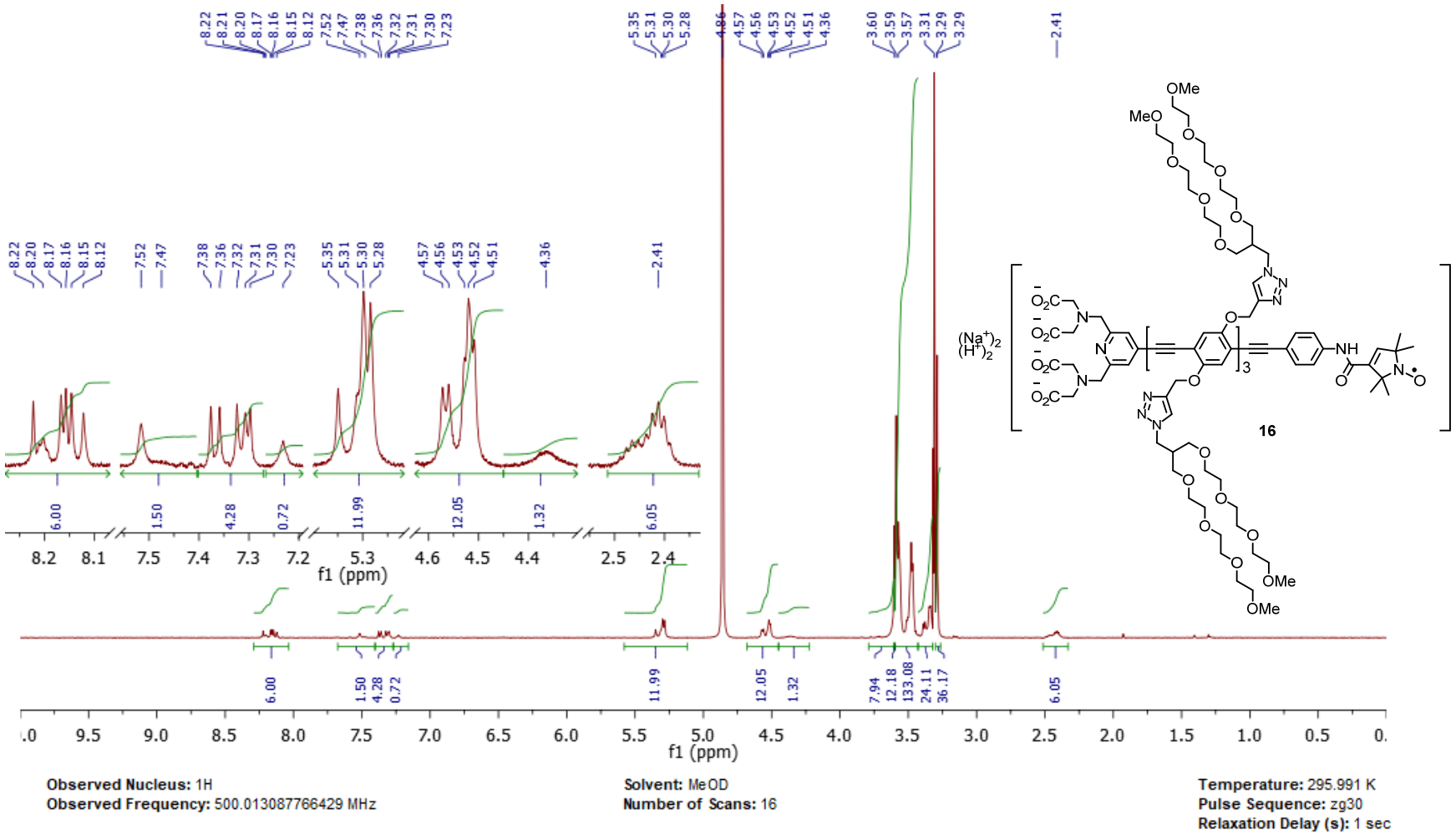

Figure S27. ^{1}H NMR spectrum of H_2Na_2[PyMTA-$(EP)_4$-NO•] **16**.

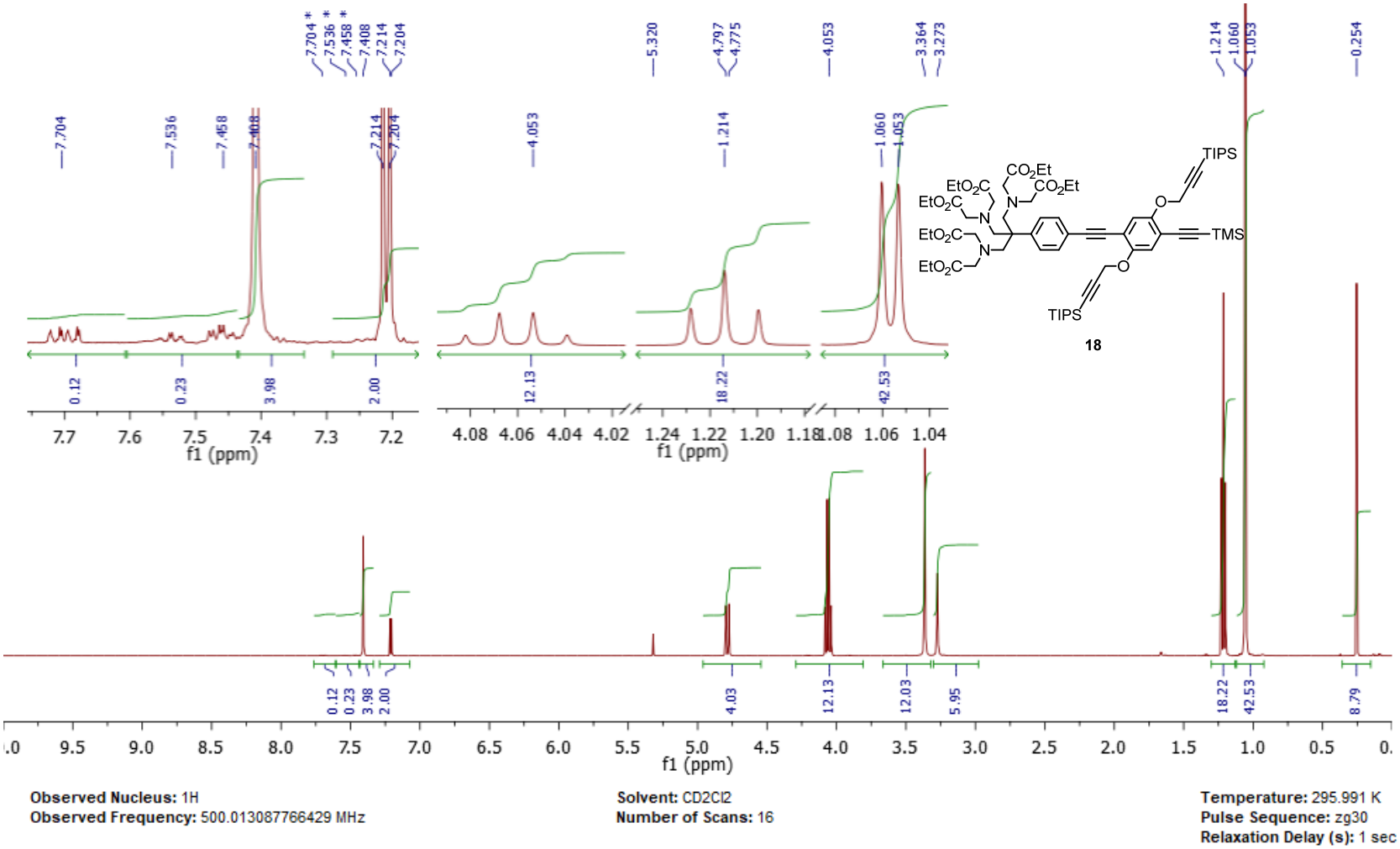

Figure S28. ^{1}H NMR spectrum of TAHAester-EPE-TMS **18**. *$Ph_3P=O$

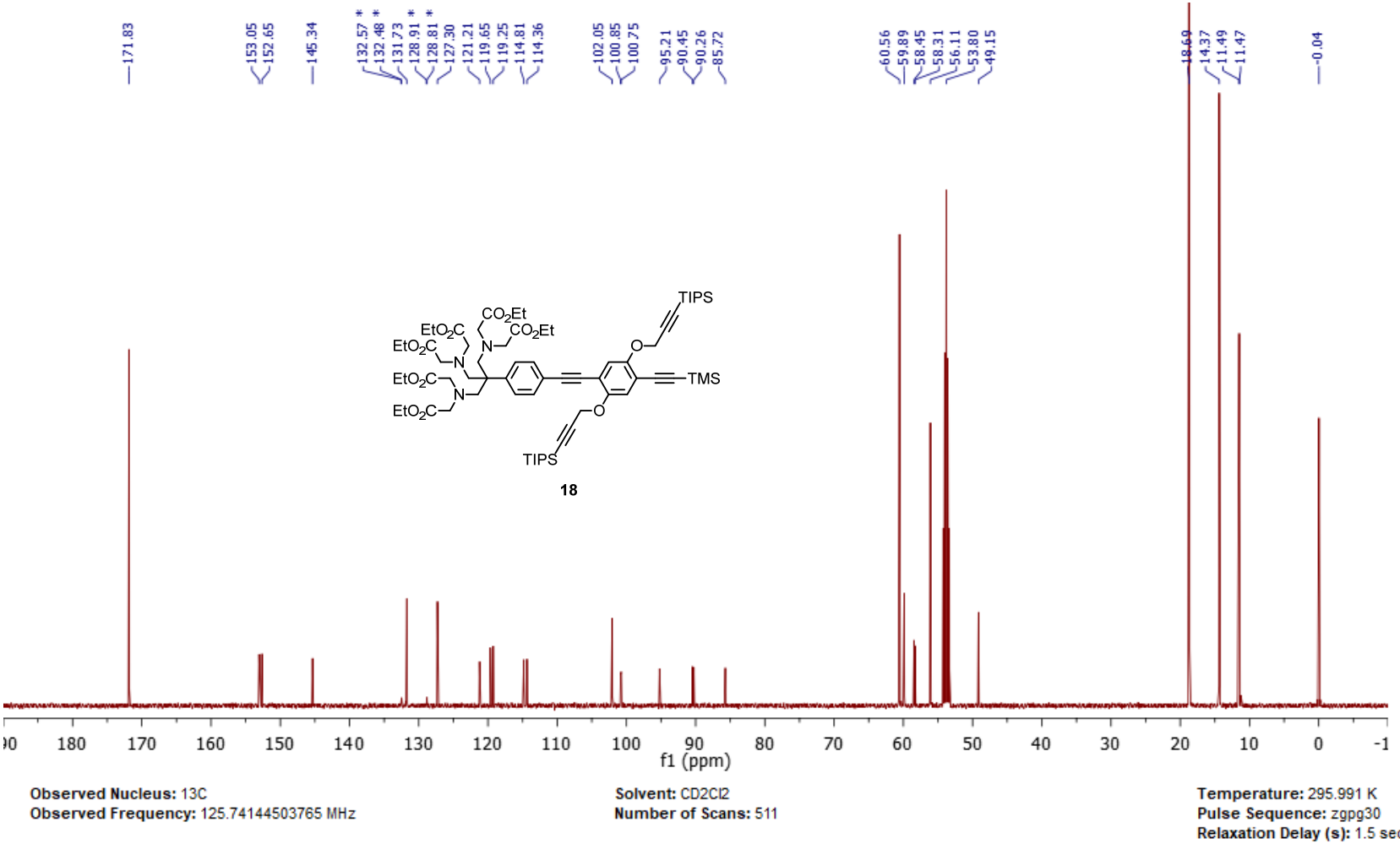

Figure S29. ^{13}C NMR spectrum of TAHAester-EPE-TMS **18**. *$Ph_3P=O$

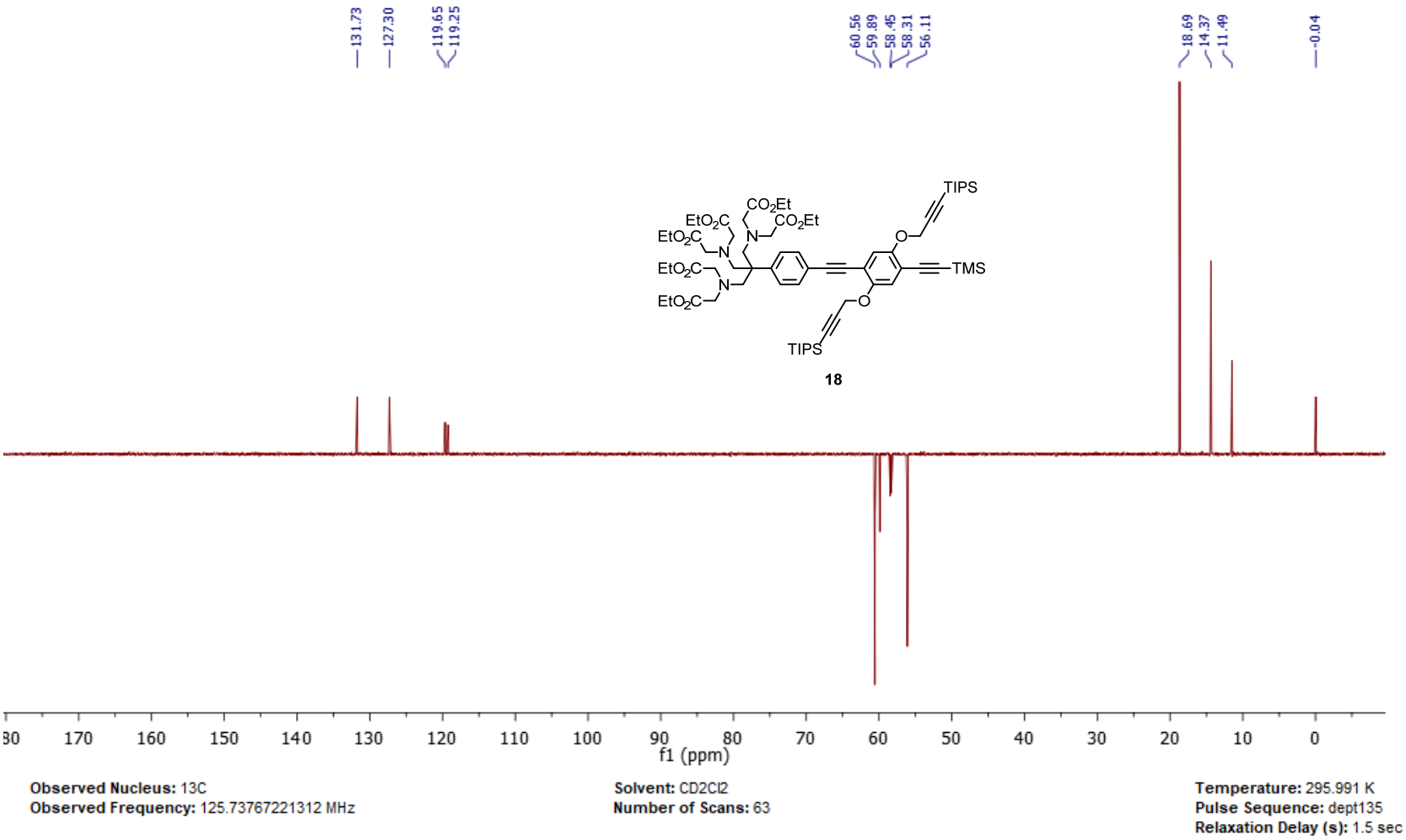

Figure S30. DEPT-135 NMR spectrum of TAHAester-EPE-TMS **18**.

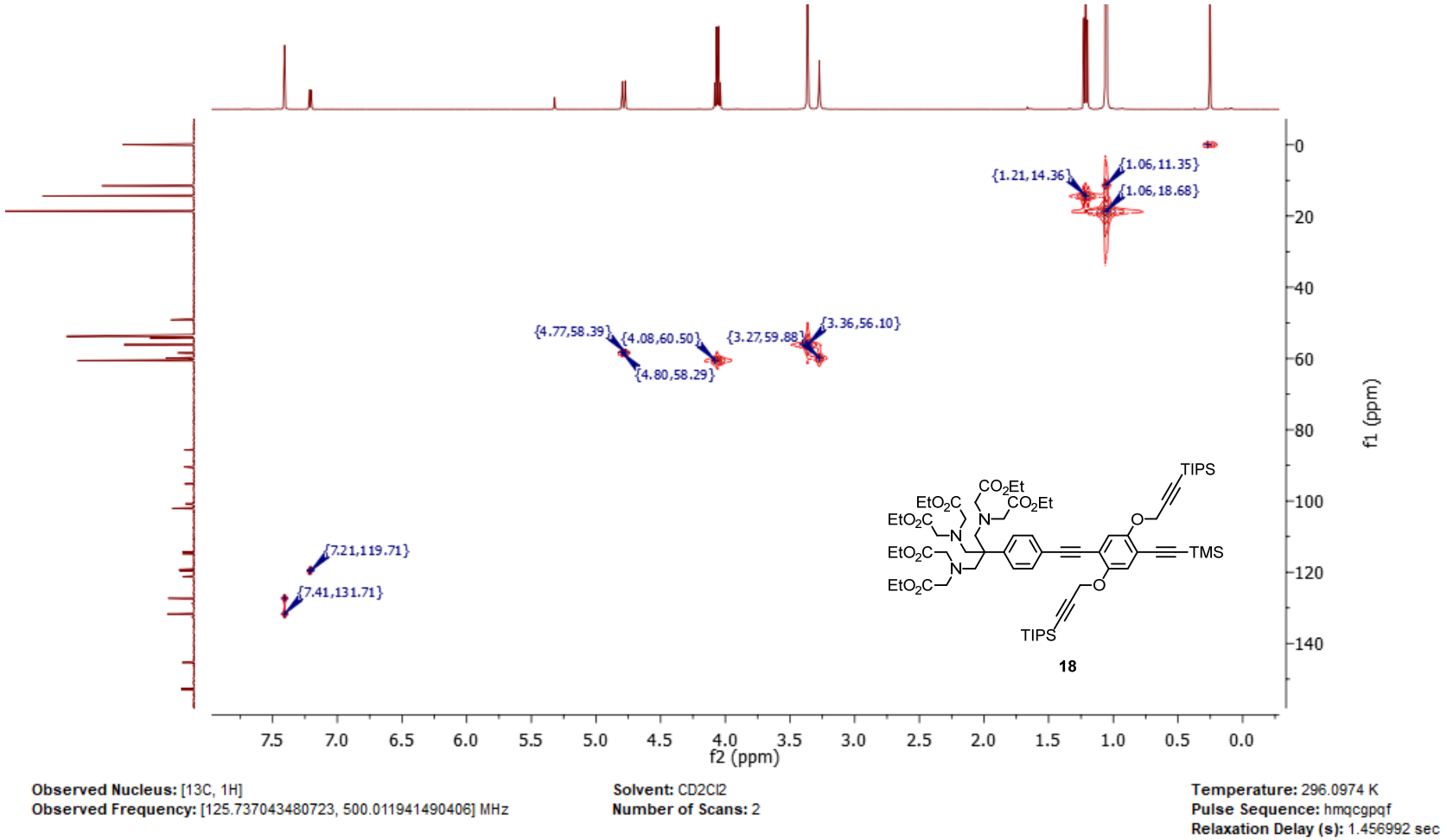

Figure S31. HMQC NMR spectrum of TAHAester-EPE-TMS **18**.

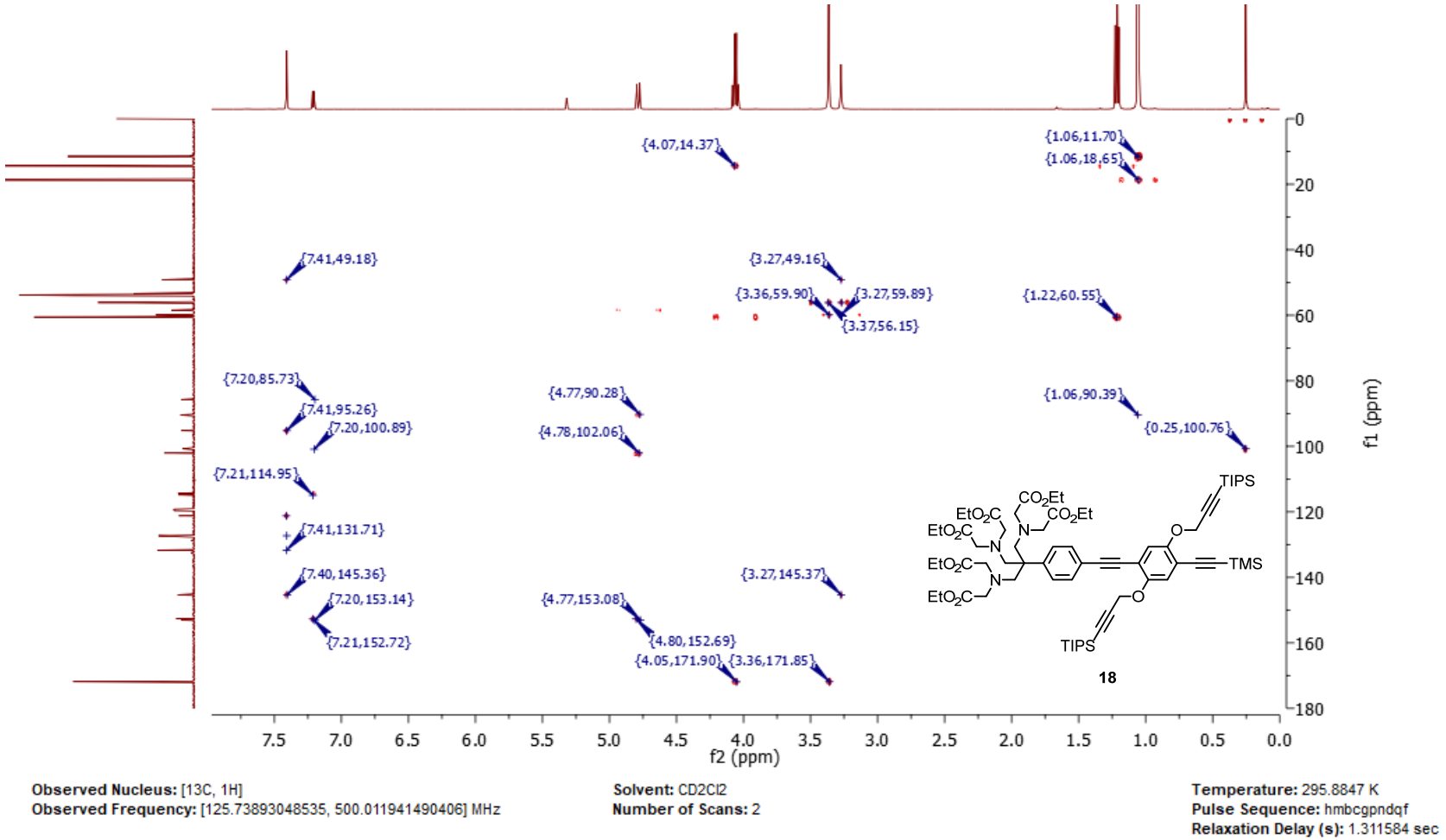

Figure S32. HMBC NMR spectrum of TAHAester-EPE-TMS **18**.

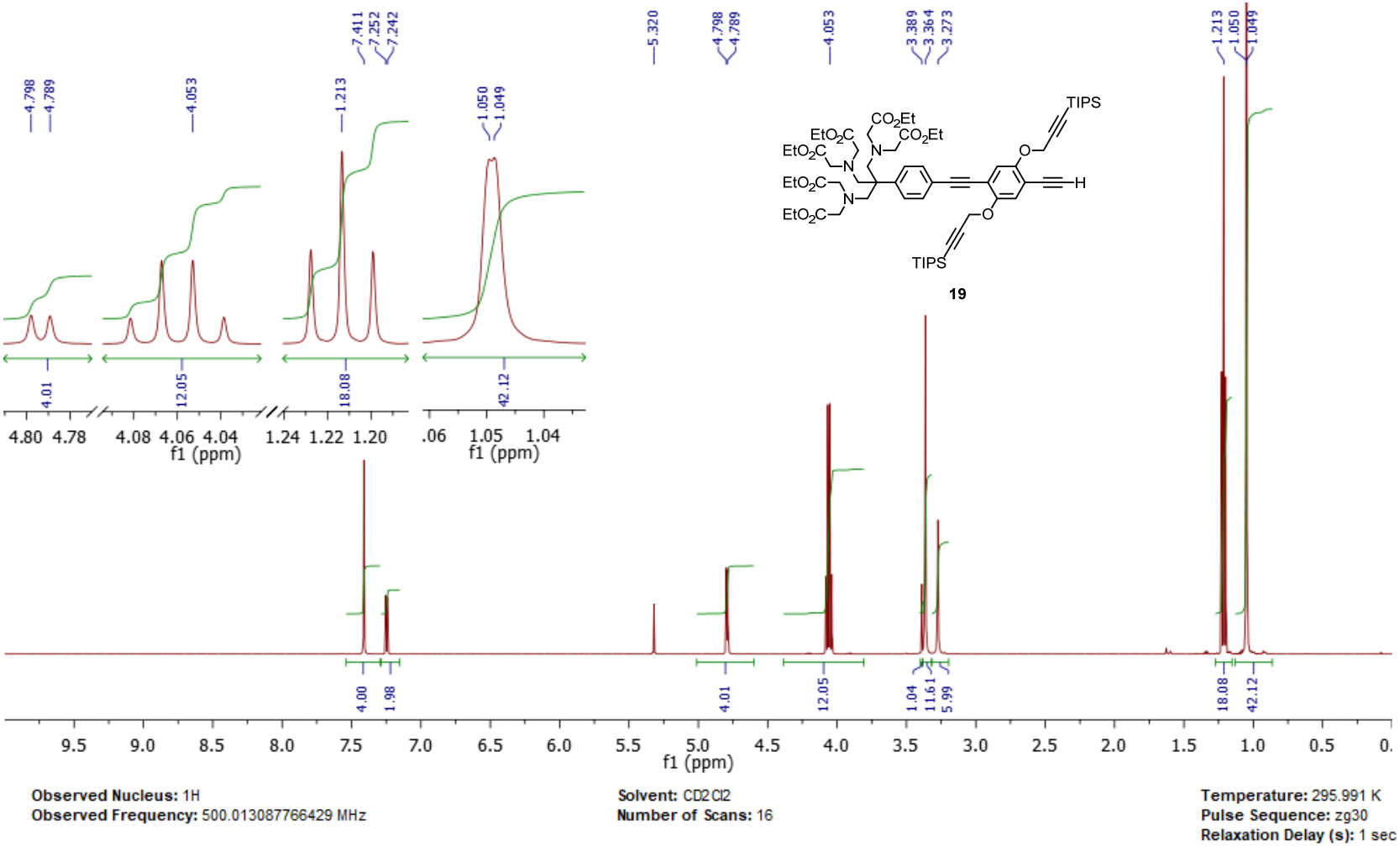

Figure S33. ^{1}H NMR spectrum of TAHAester-EPE-H **19**.

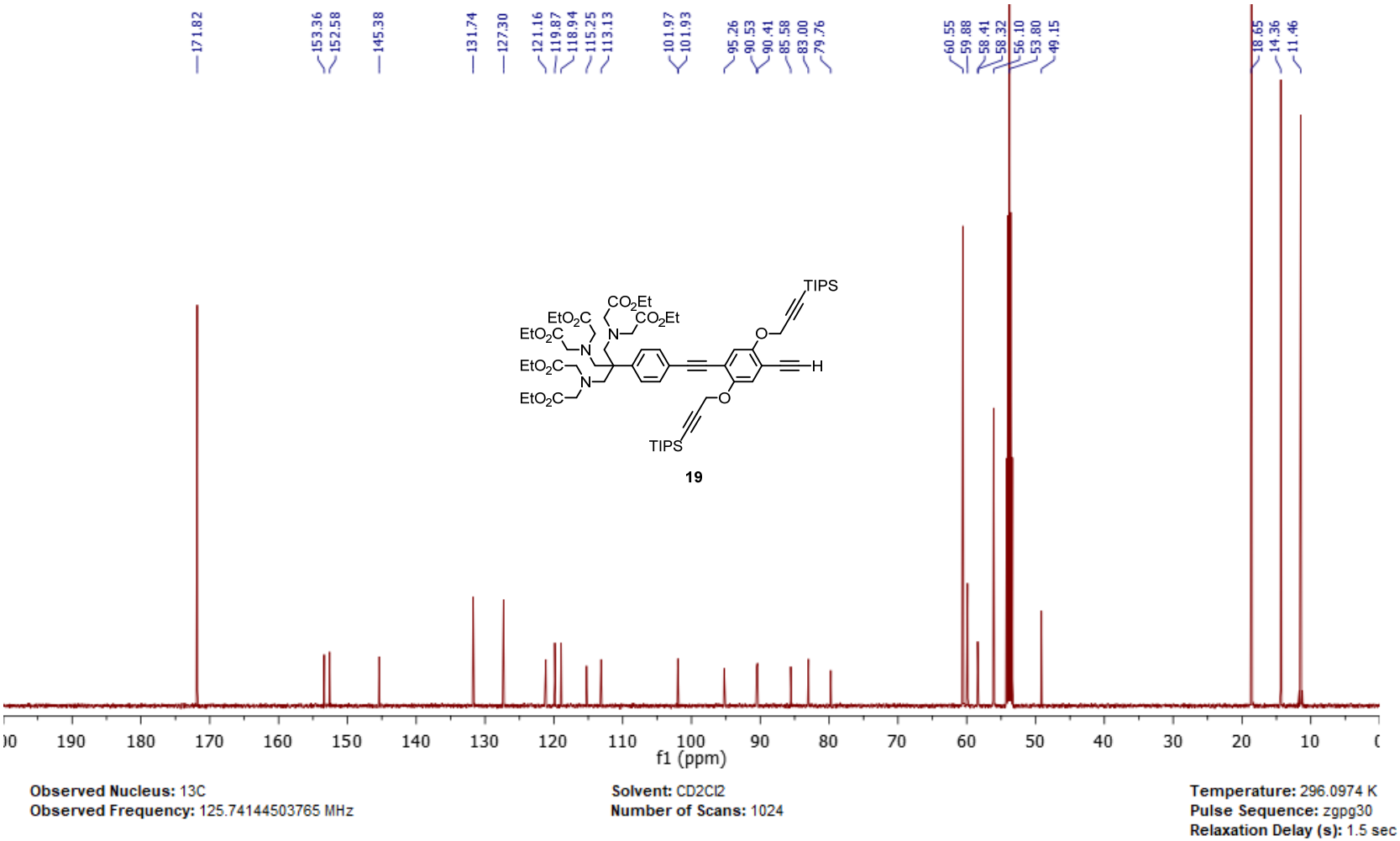

Figure S34. ^{13}C NMR spectrum of TAHAester-EPE-H **19**.

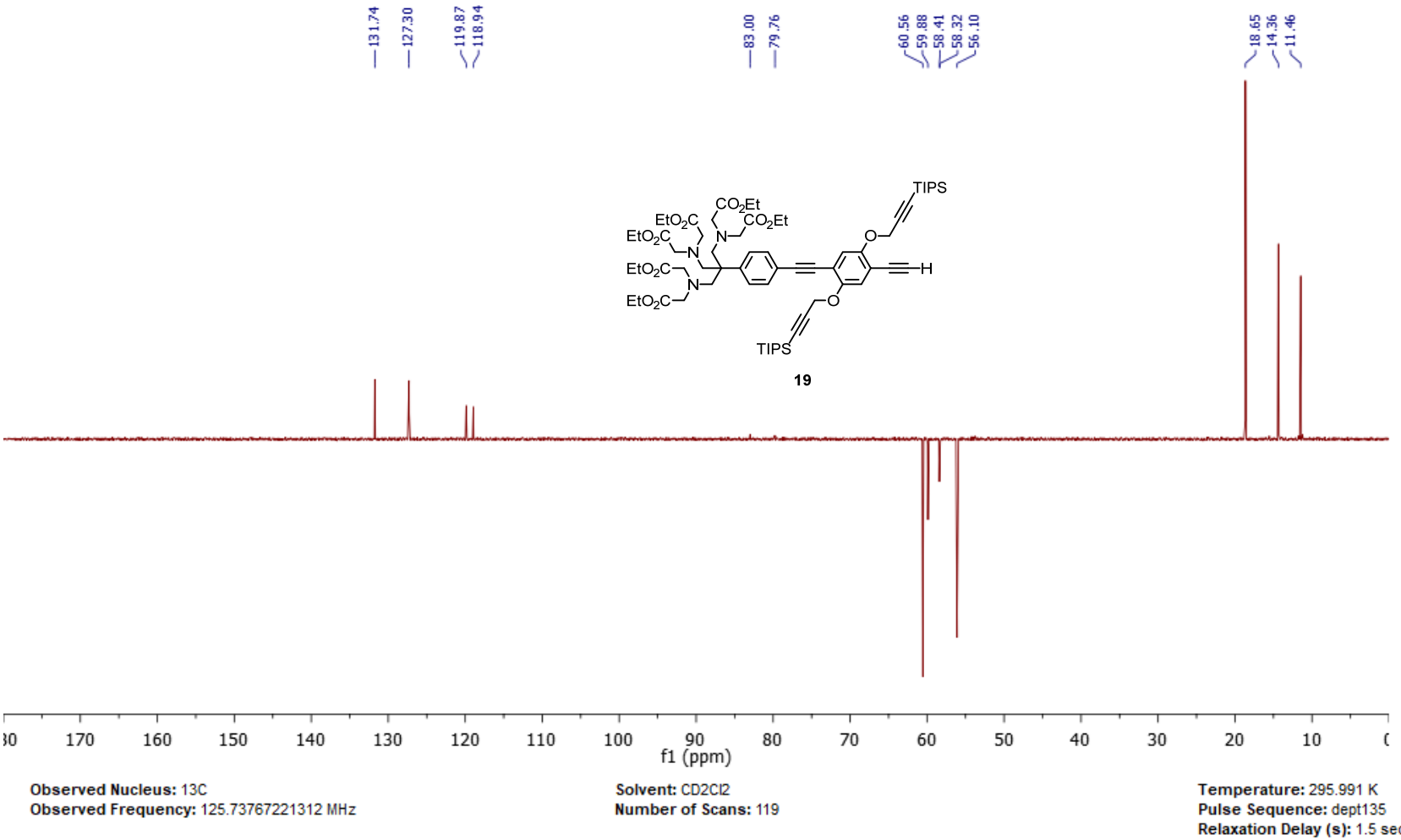

Figure S35. DEPT-135 NMR spectrum of TAHAester-EPE-H **19**.

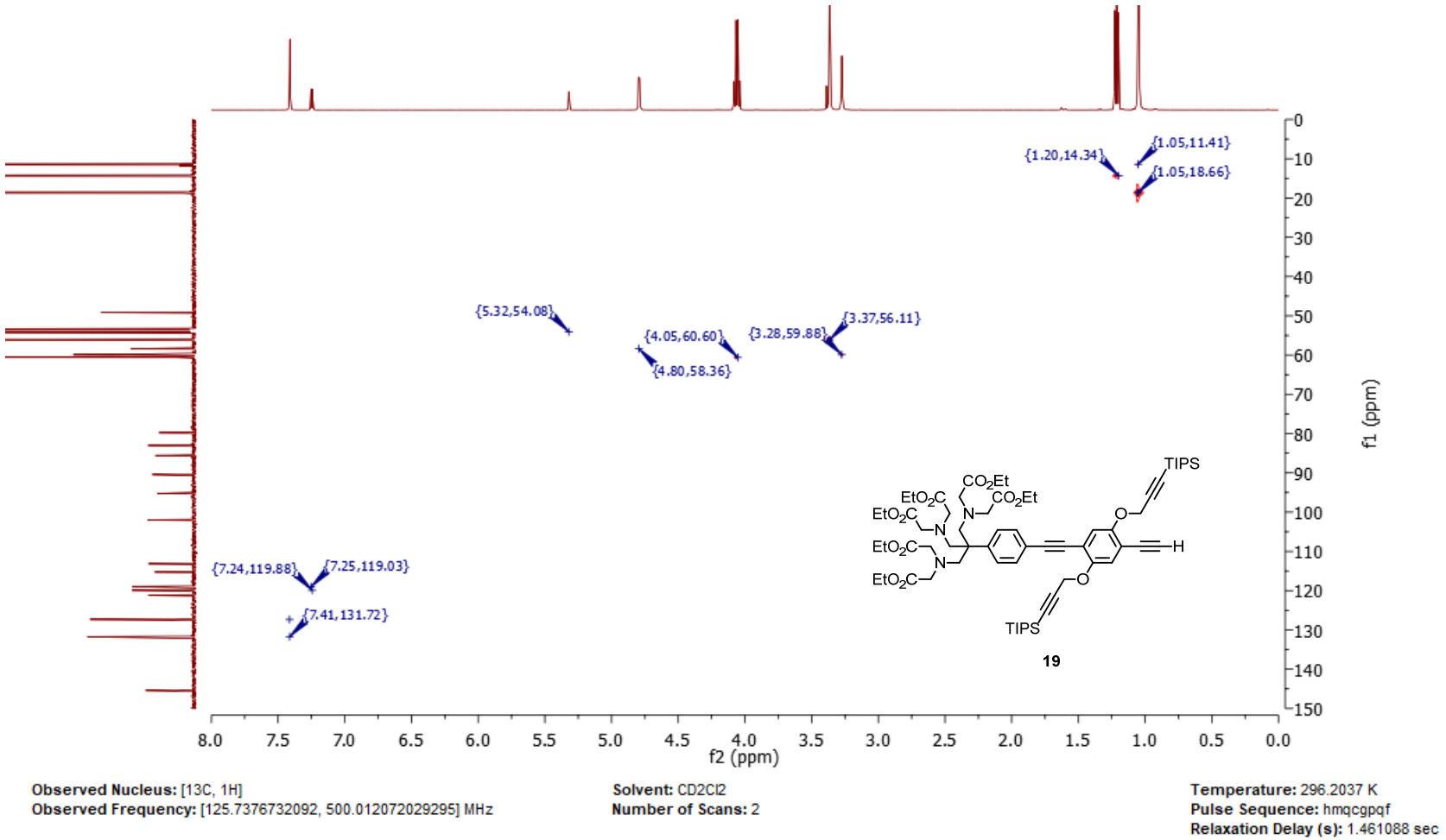

Figure S36. HMQC NMR spectrum of TAHAester-EPE-H **19**.

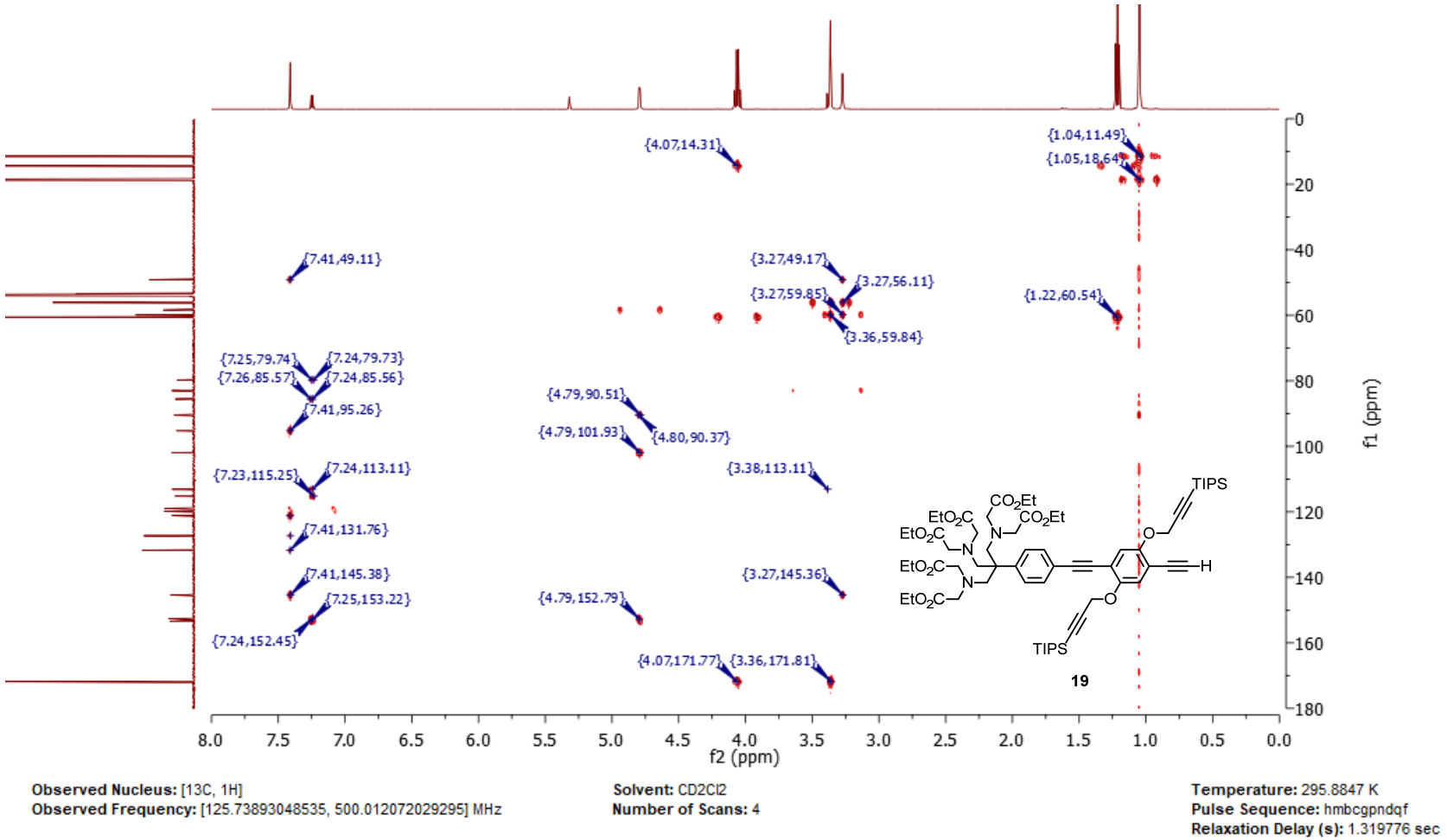

Figure S37. HMBC NMR spectrum of TAHAester-EPE-H **19**.

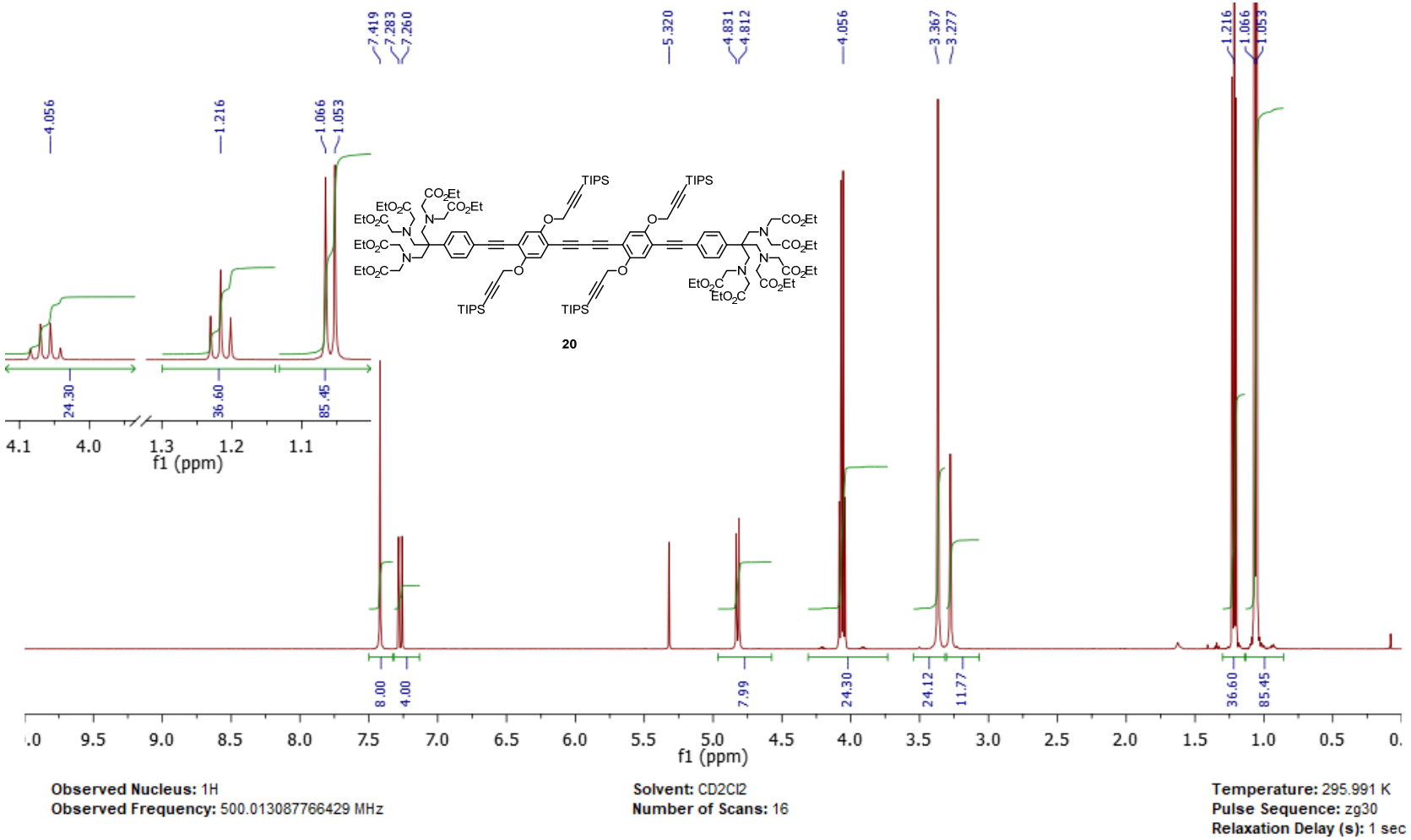

Figure S38. ^{1}H NMR spectrum of TAHAester-EPBPE-TAHAester **20**.

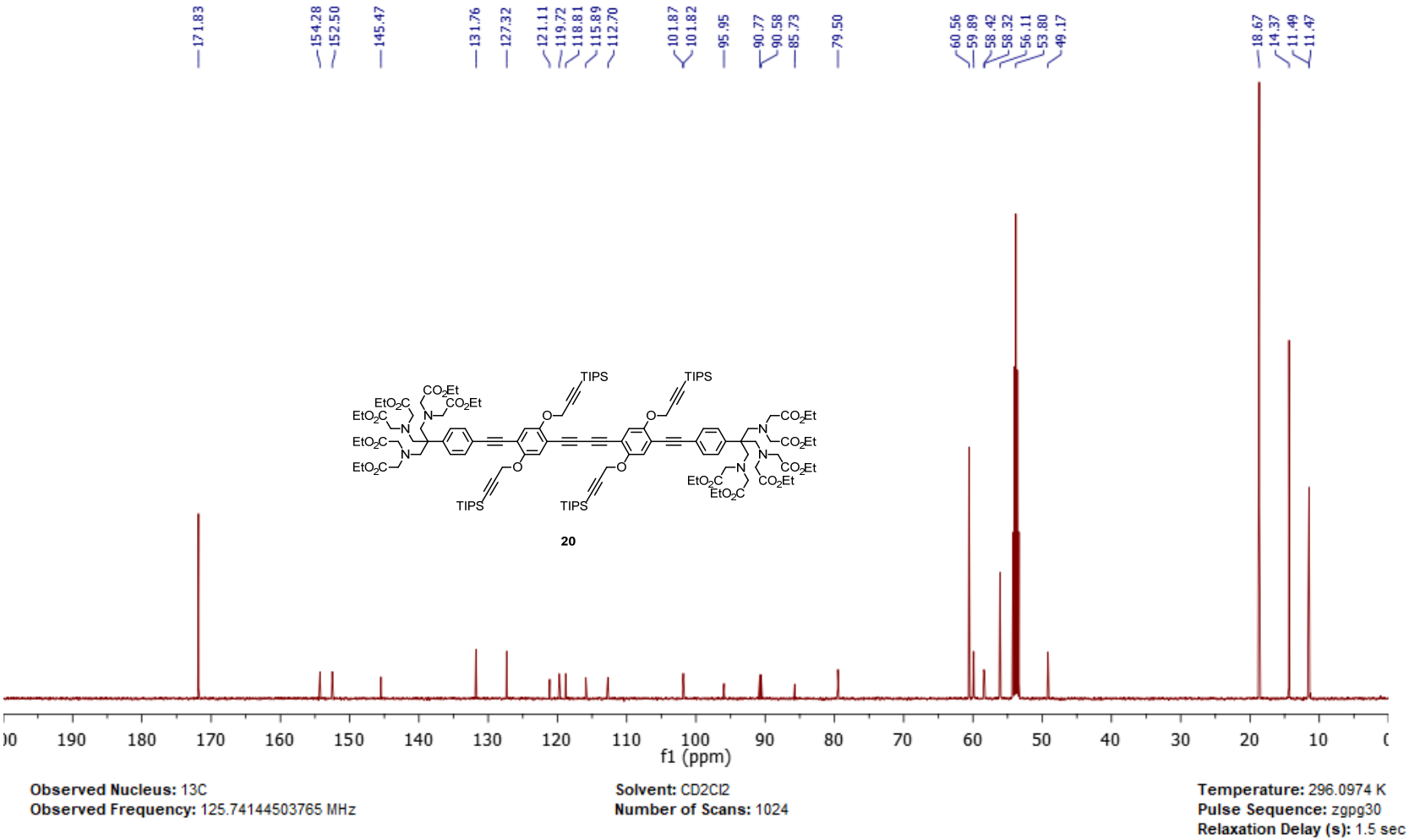

Figure S39. ^{13}C NMR spectrum of TAHAester-EPBPE-TAHAester **20**.

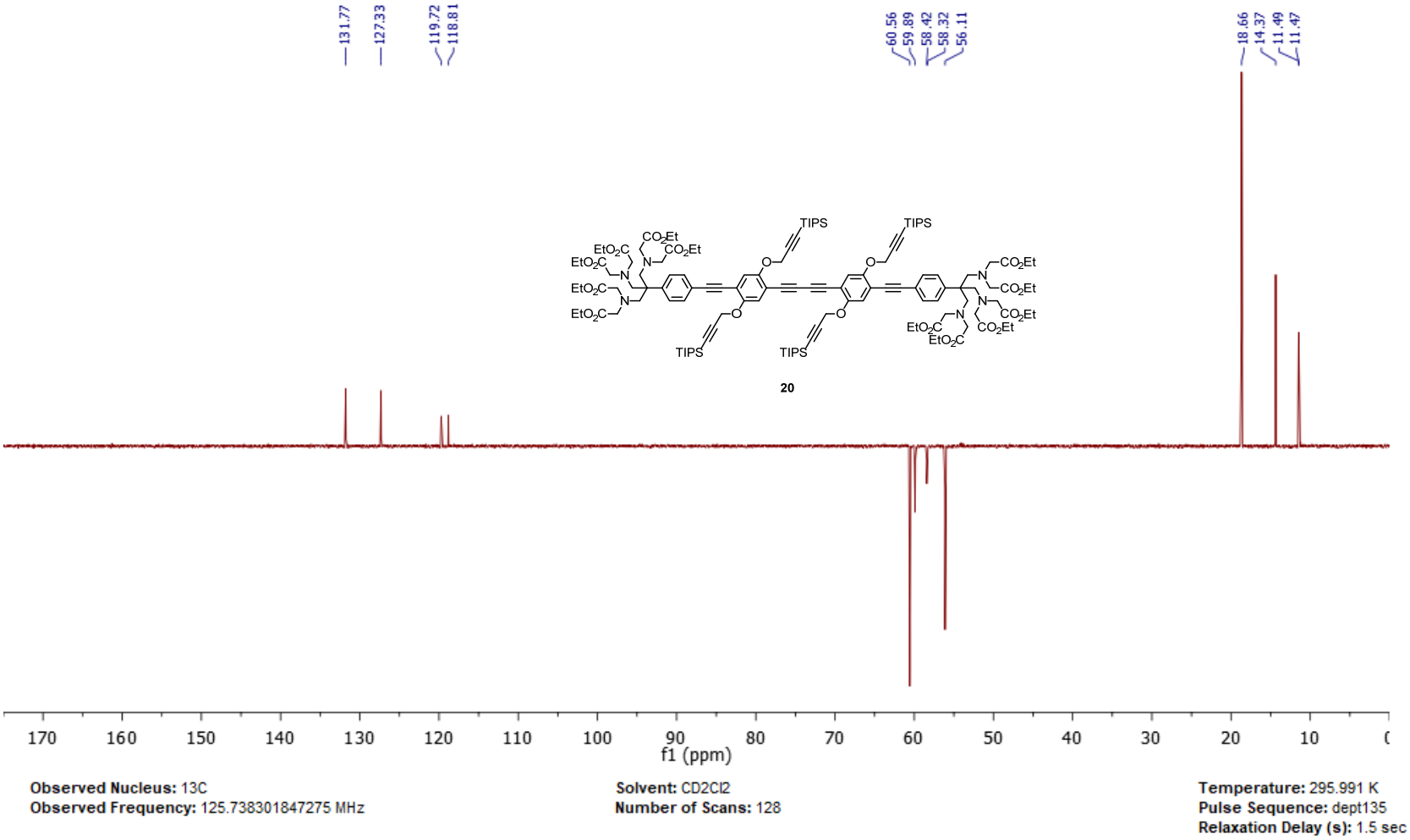

Figure S40. DEPT-135 NMR spectrum of TAHAester-EPBPE-TAHAester **20**.

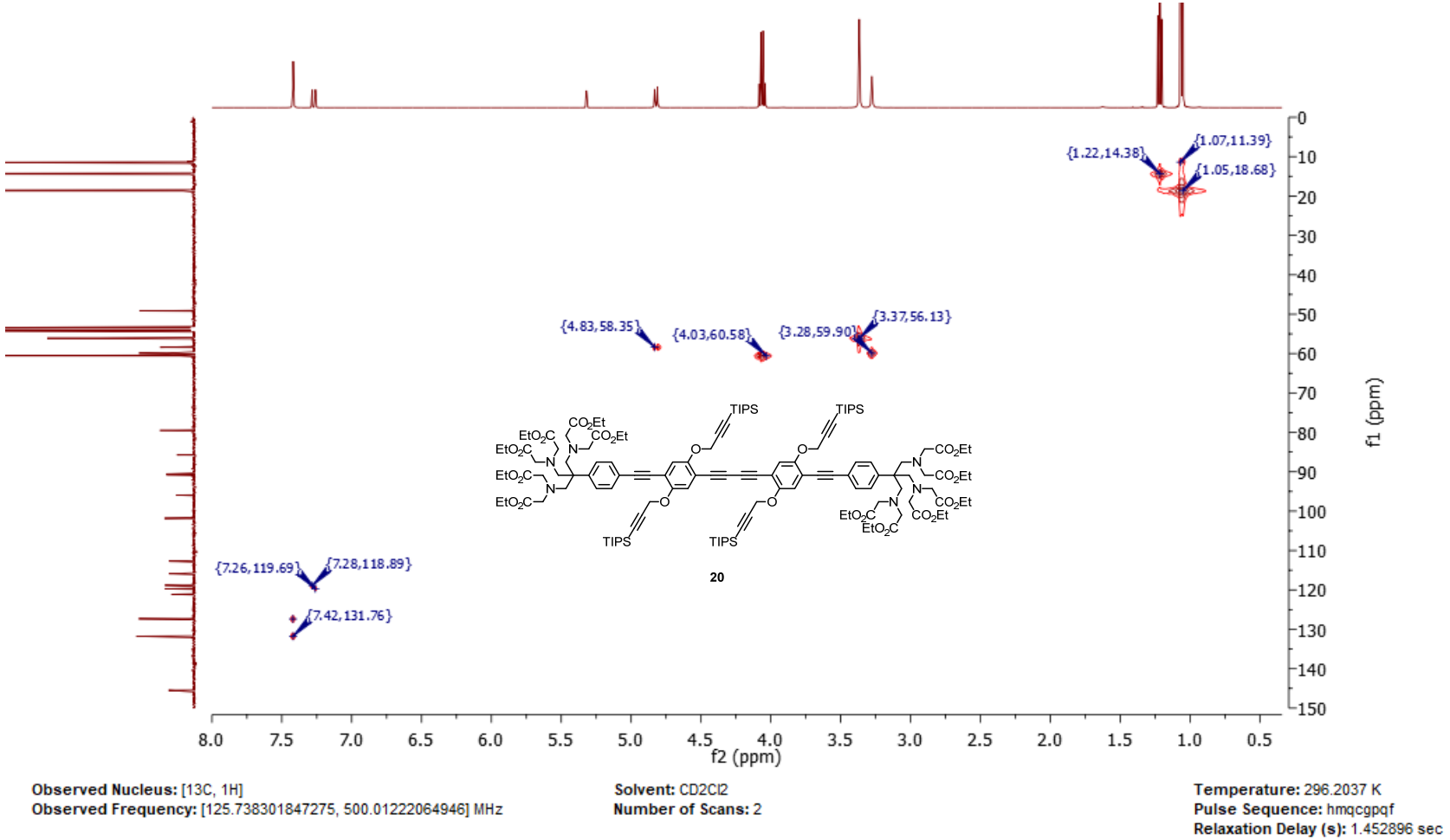

Figure S41. HMQC NMR spectrum of TAHAester-EPBPE-TAHAester **20**.

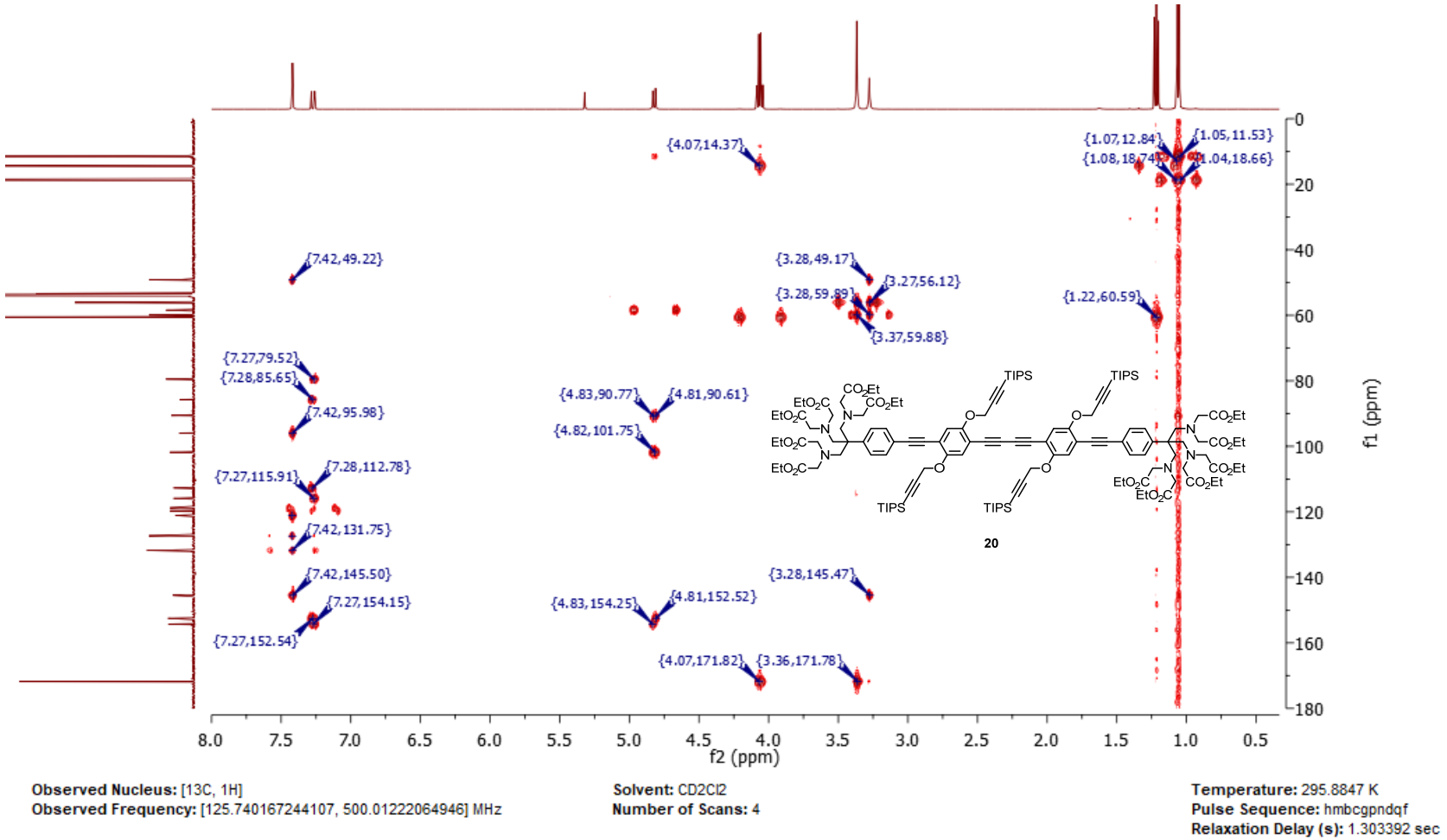

Figure S42. HMBC NMR spectrum of TAHAester-EPBPE-TAHAester **20**.

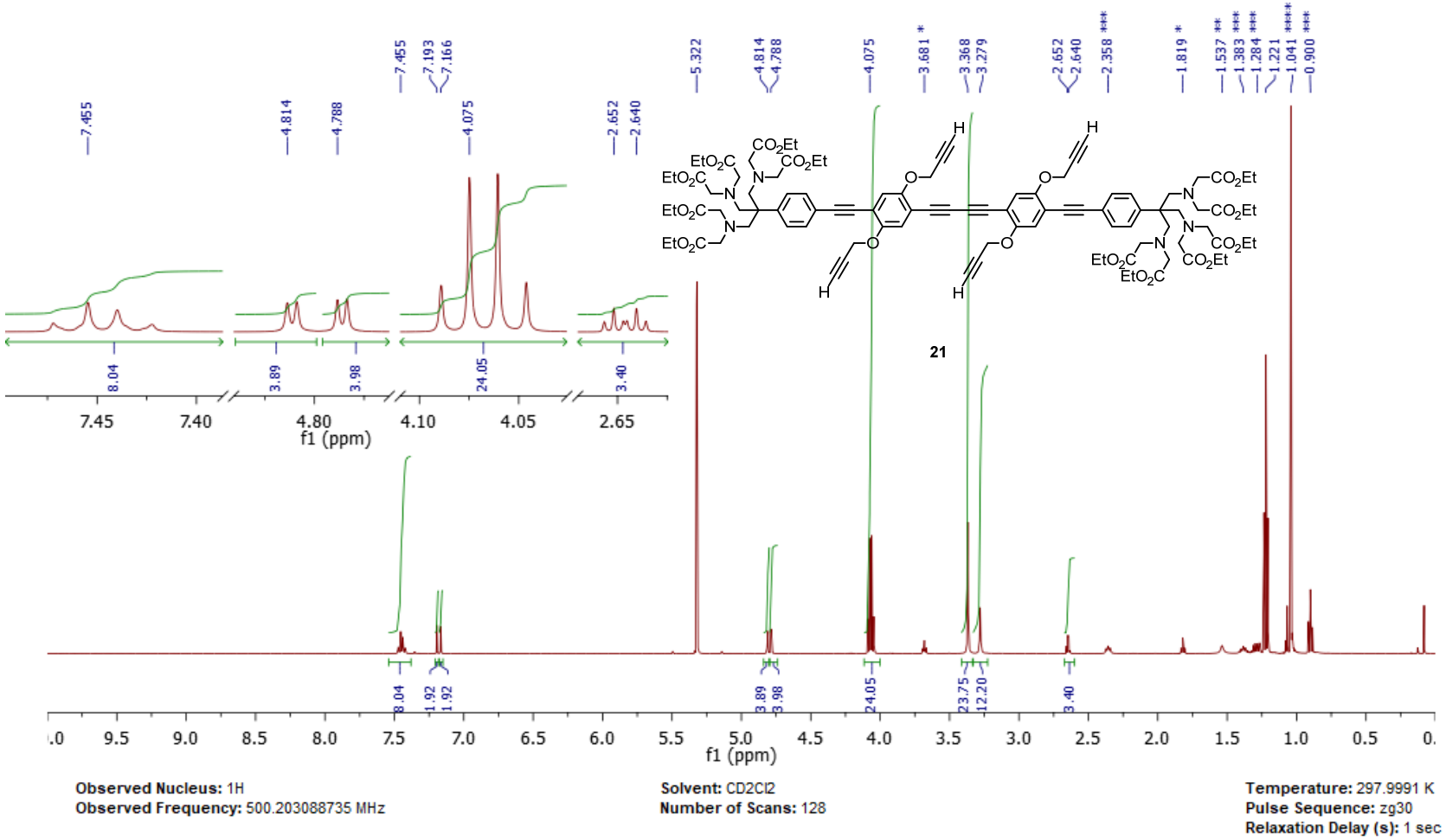

Figure S43. ^{1}H NMR spectrum of desilylated TAHAester-EPBPE-TAHAester **21**. *THF, **water, ***Bu_4N-salt, ****TIPS-OH and/or TIPS-F.

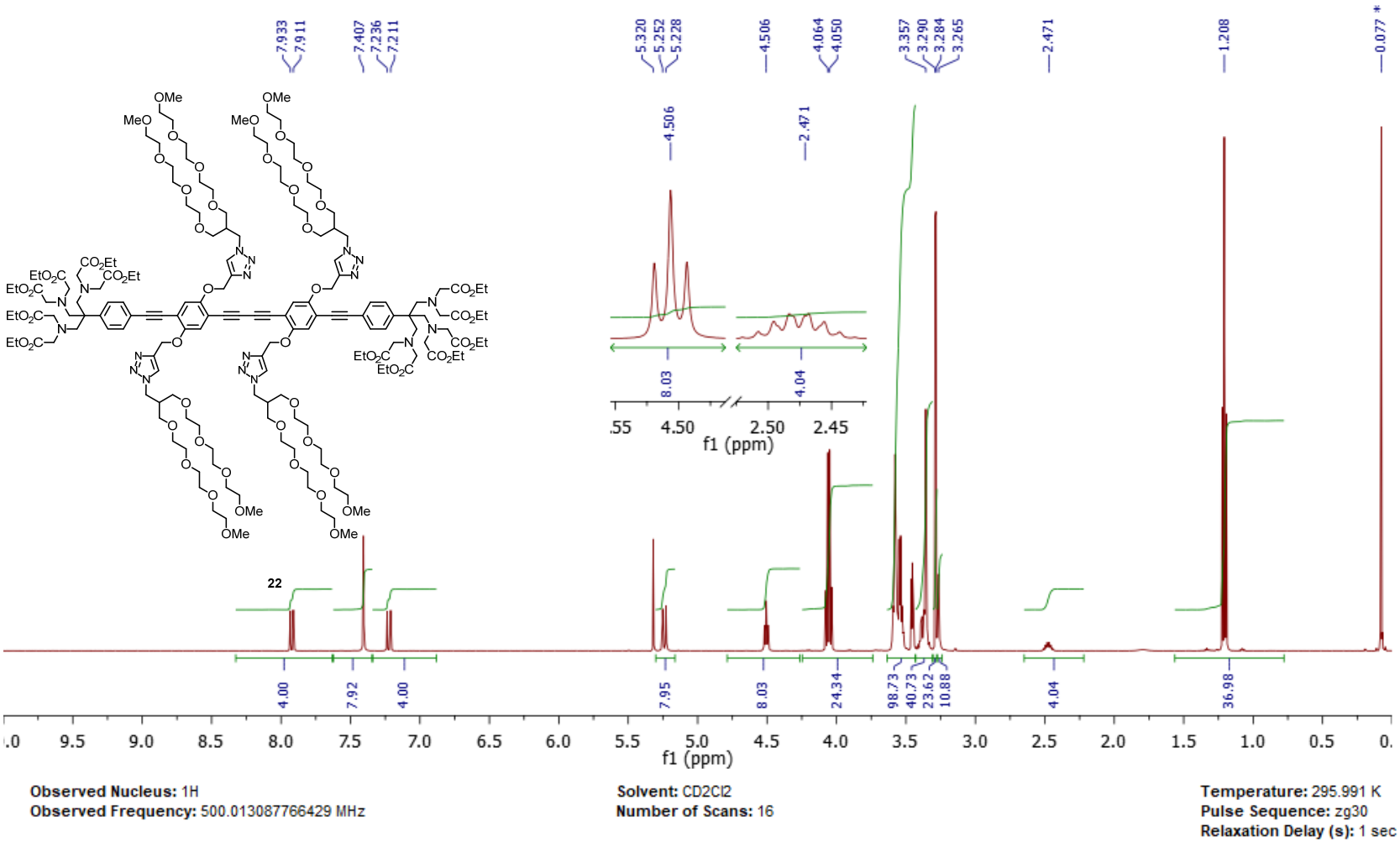

Figure S44. ^{1}H NMR spectrum of PEGylated TAHAester-EPBPE-TAHAester **22**. *grease.

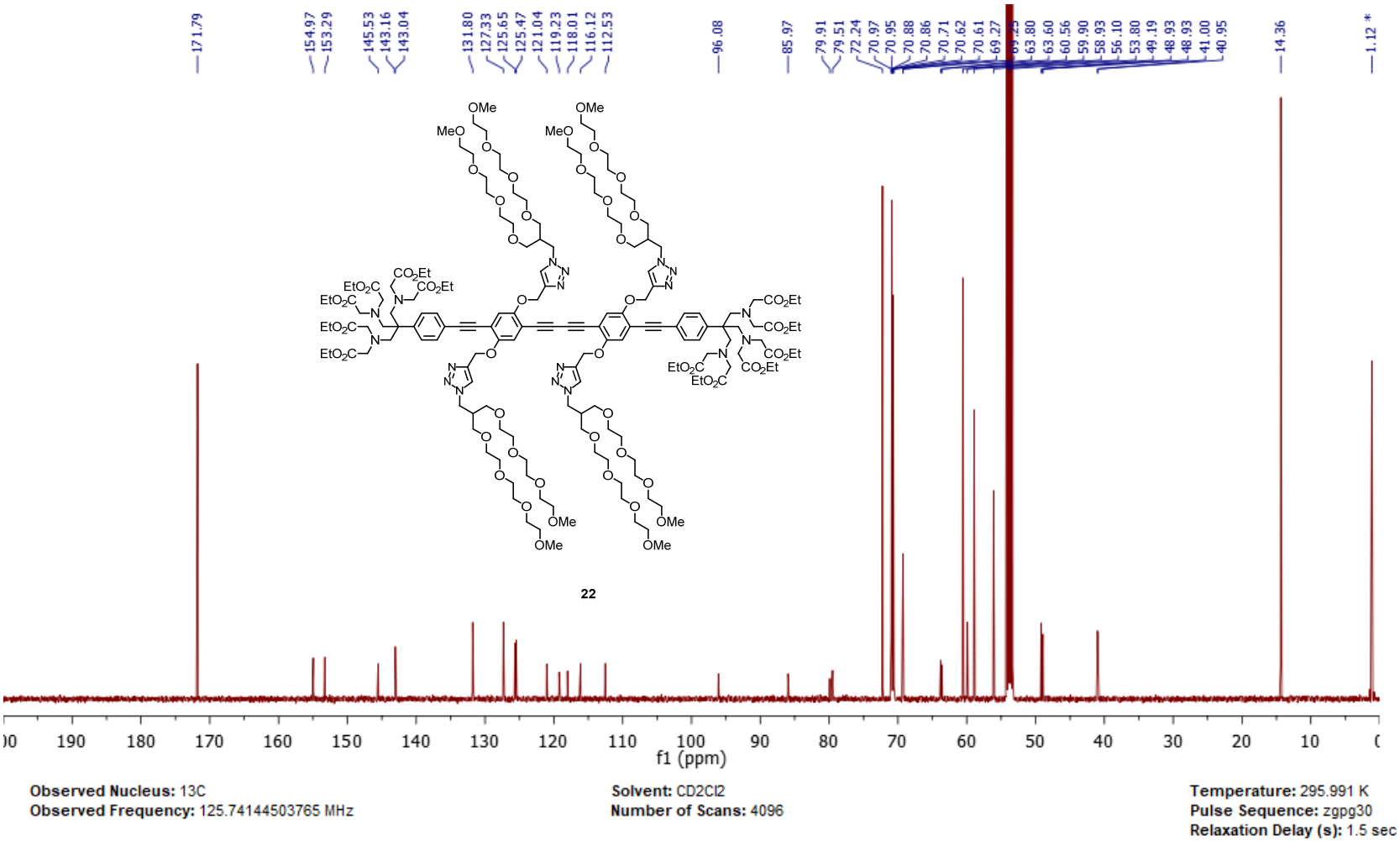

Figure S45. ^{13}C NMR spectrum of PEGylated TAHAester-EPBPE-TAHAester **22**. *grease.

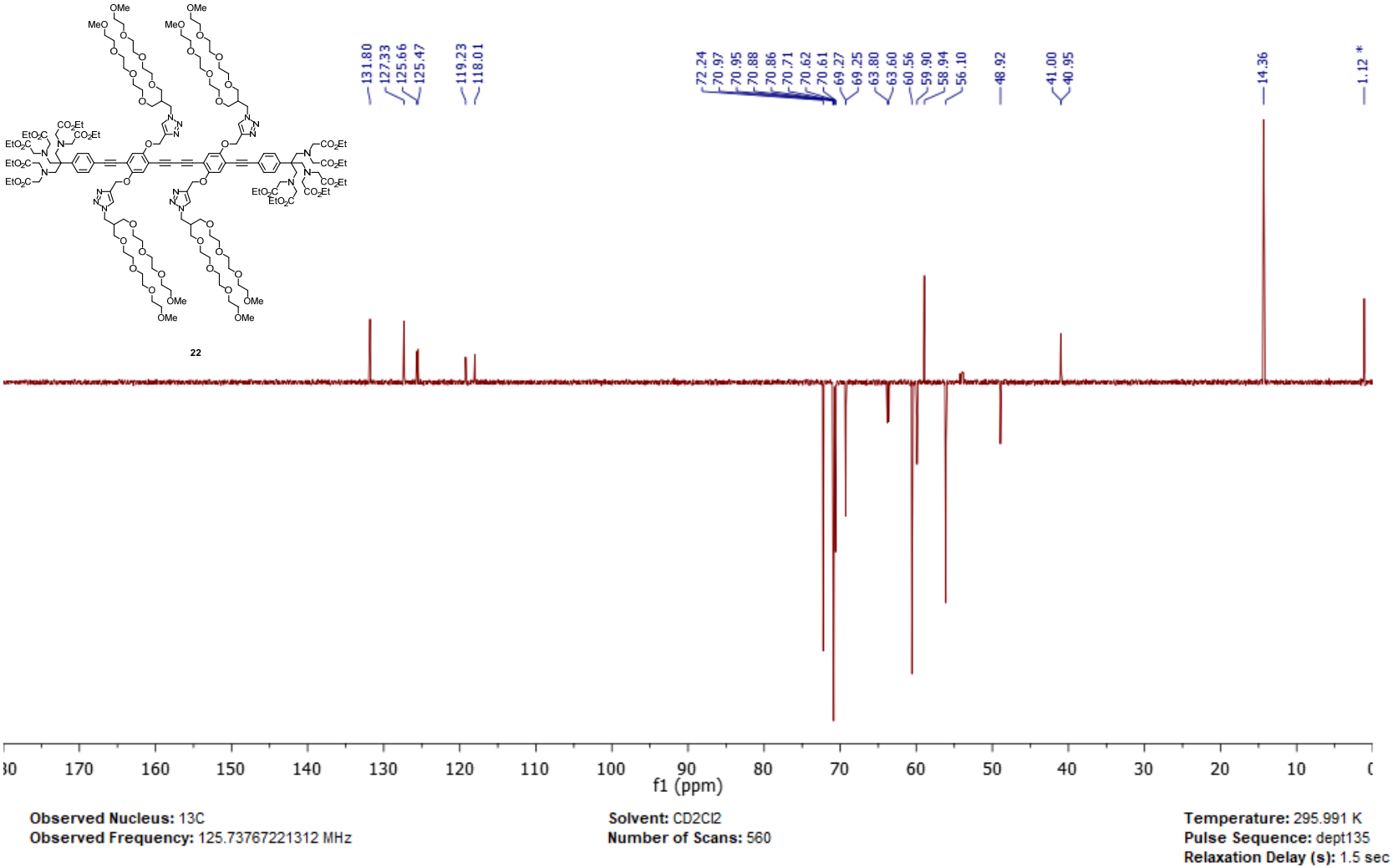

Figure S46. DEPT-135 NMR spectrum of PEGylated TAHAester-EPBPE-TAHAester **22**. *grease.

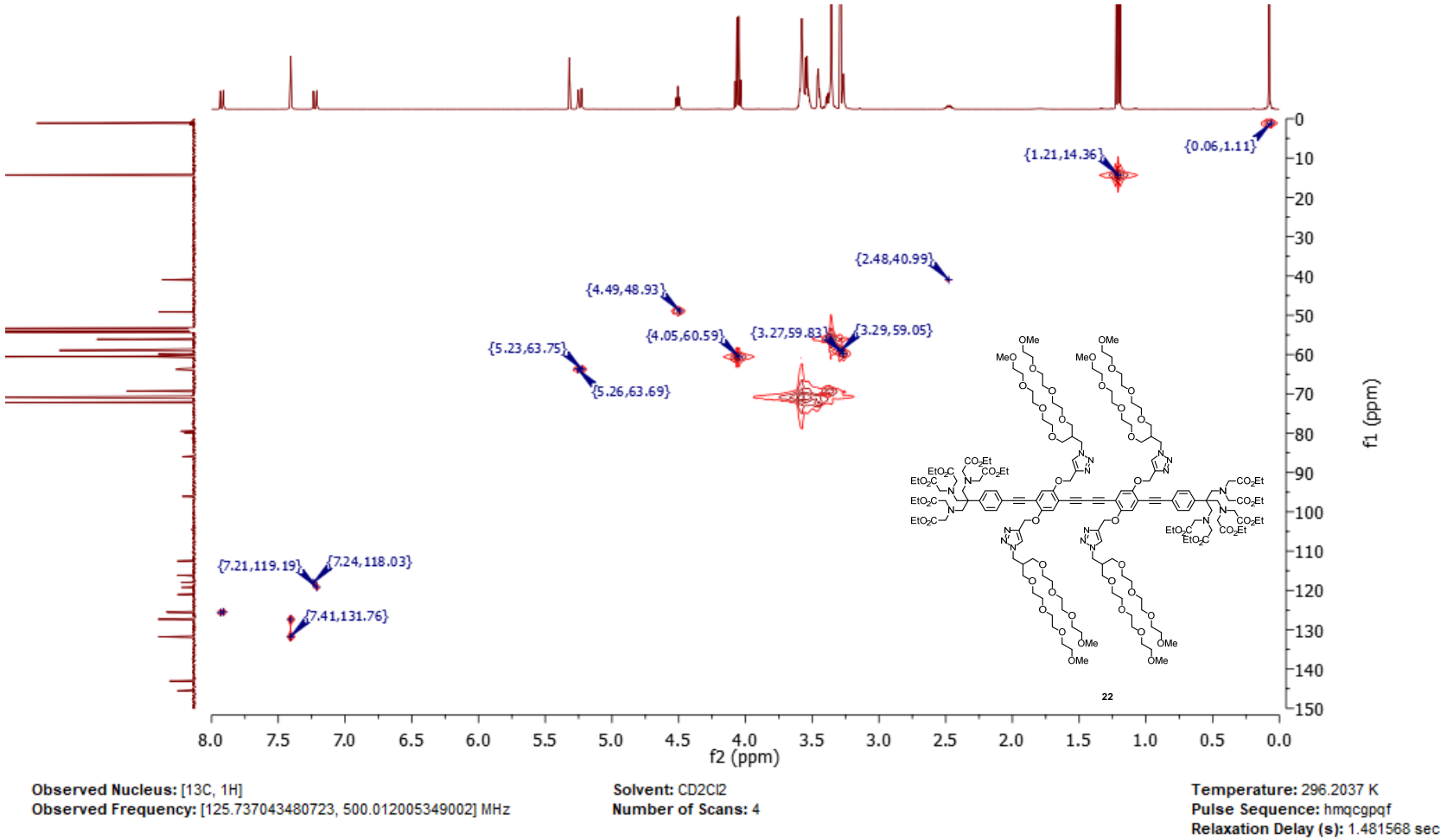

Figure S47. HMQC NMR spectrum of PEGylated TAHAester-EPBPE-TAHAester **22**.

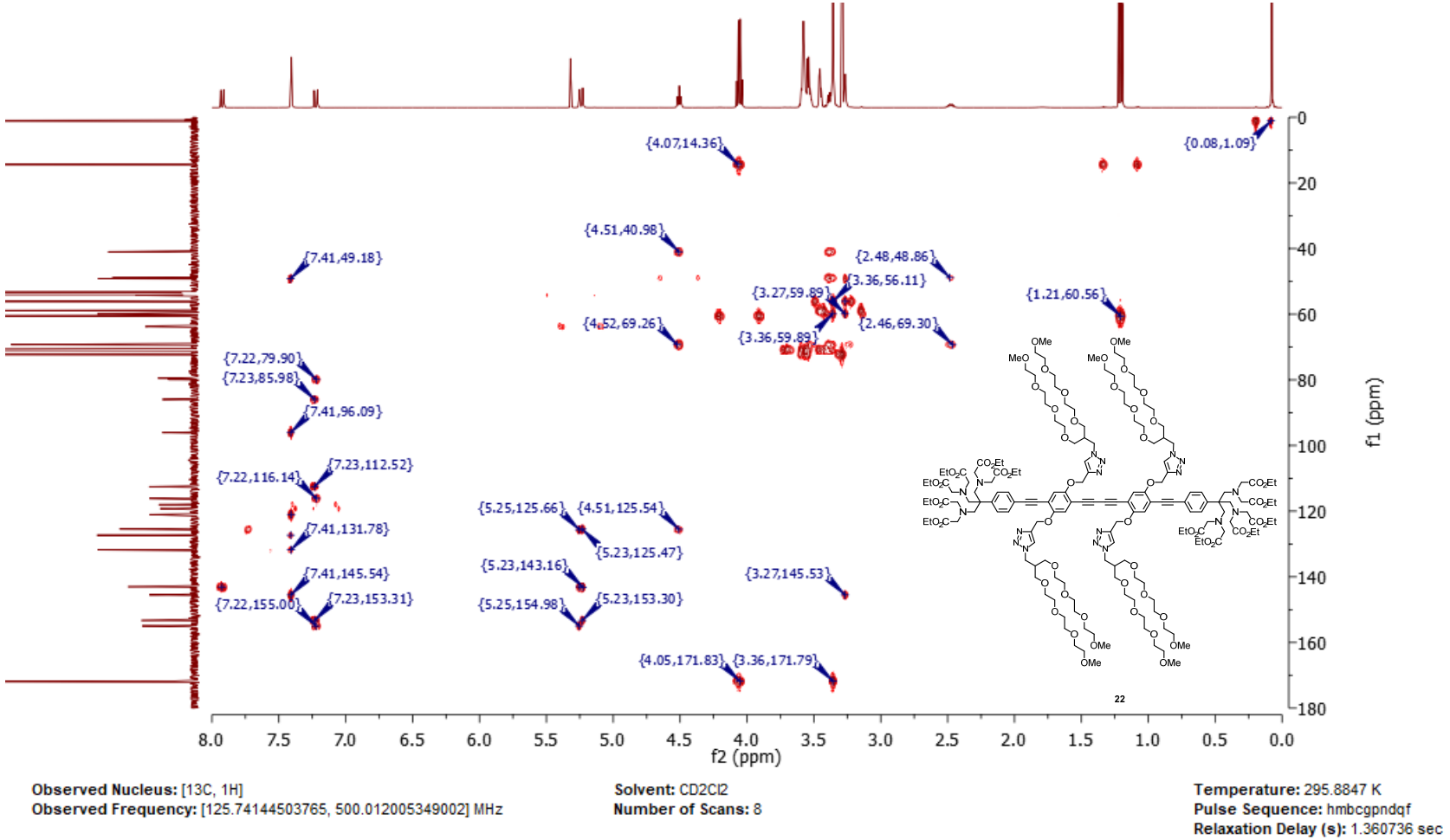

Figure S48. HMBC NMR spectrum of PEGylated TAHAester-EPBPE-TAHAester **22**.

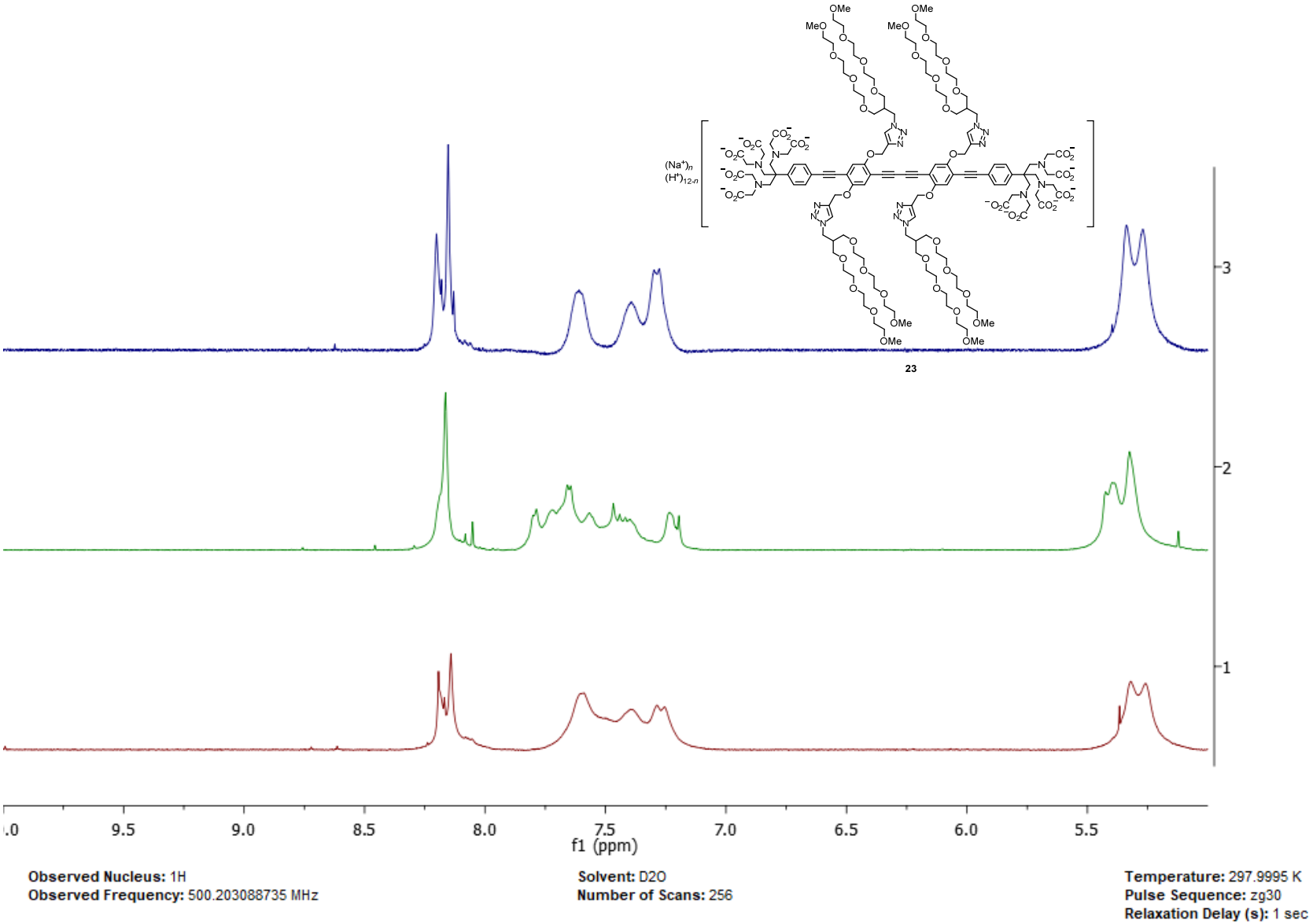

Figure S49. Cutout of ^{1}H NMR spectrum (10 ppm – 5 ppm) of H_xNa_{12-x}[TAHA-EPBPE-TAHA] **23** at pD 1.6 (top), pD 5.0 middle, and pD 1.3 (bottom).

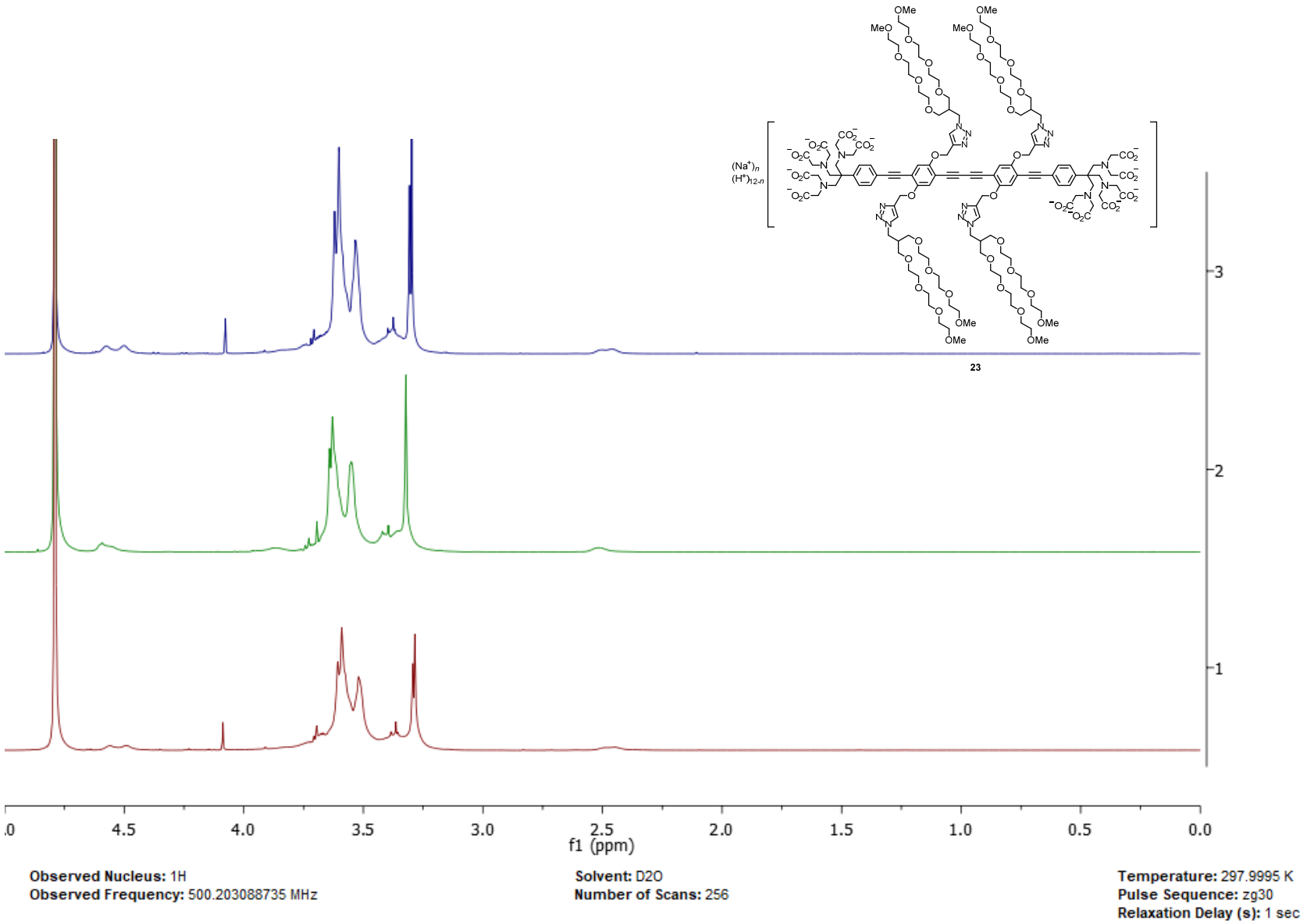

Figure S50. Cutout of ^{1}H NMR spectrum (5 ppm – 0 ppm) of H_xNa_{12-x}[TAHA-EPBPE-TAHA] **23** at pD 1.6 (top), pD 5.0 middle, and pD 1.3 (bottom).

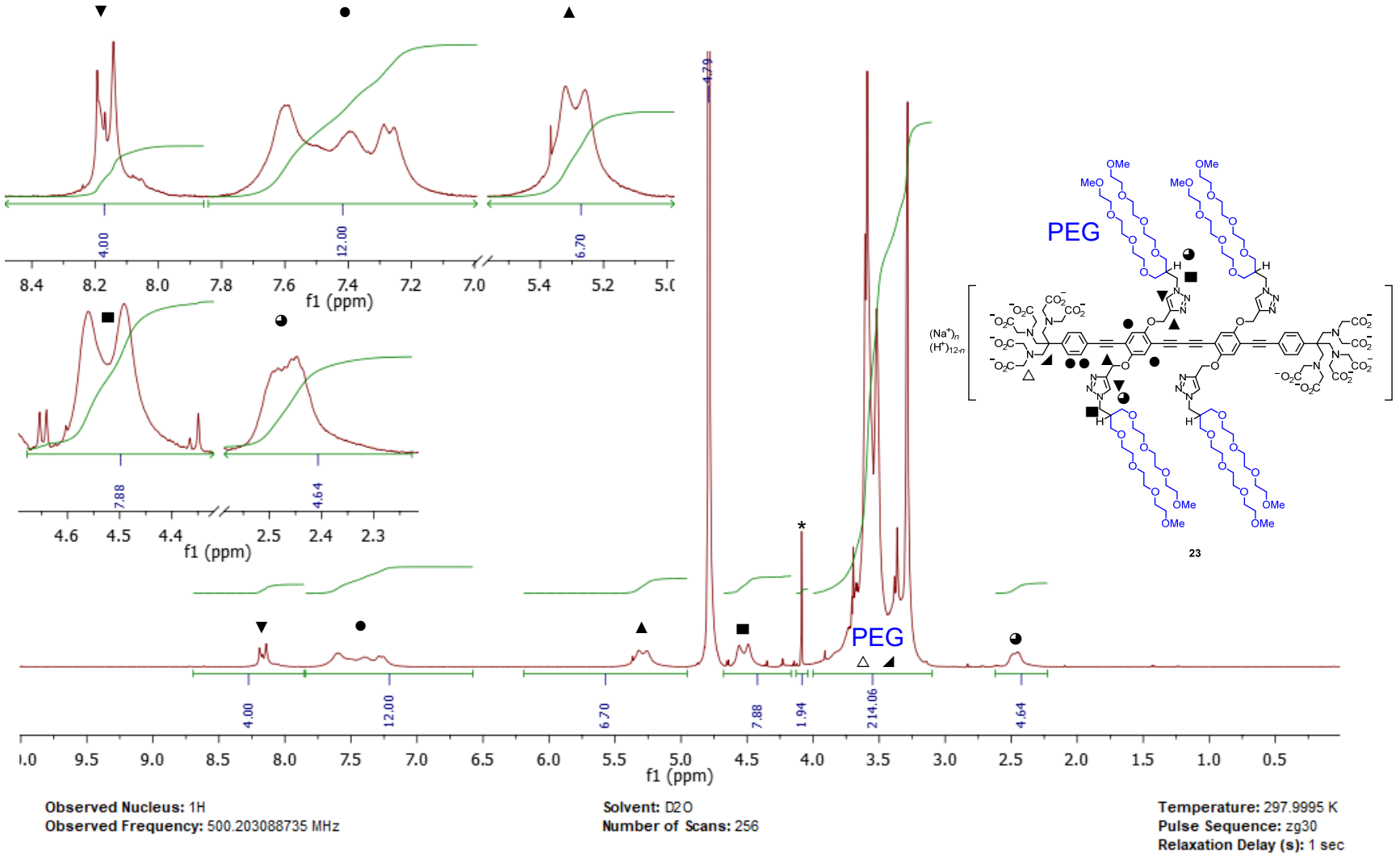

Figure S51. ^{1}H NMR spectrum of H_xNa_{12-x}[TAHA-EPBPE-TAHA] **23** at pD 1.6. *Signal of unidentified component which was only found in the spectra of solutions with pH 1.3 and 1.6, but not in the spectrum of a solution with pH 5.0.