

Synthesis, ^{123}I -radiolabeling optimization, and initial preclinical evaluation of novel urea-based PSMA inhibitors with a tributylstannyl prosthetic group in their structures

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Supplementary Materials

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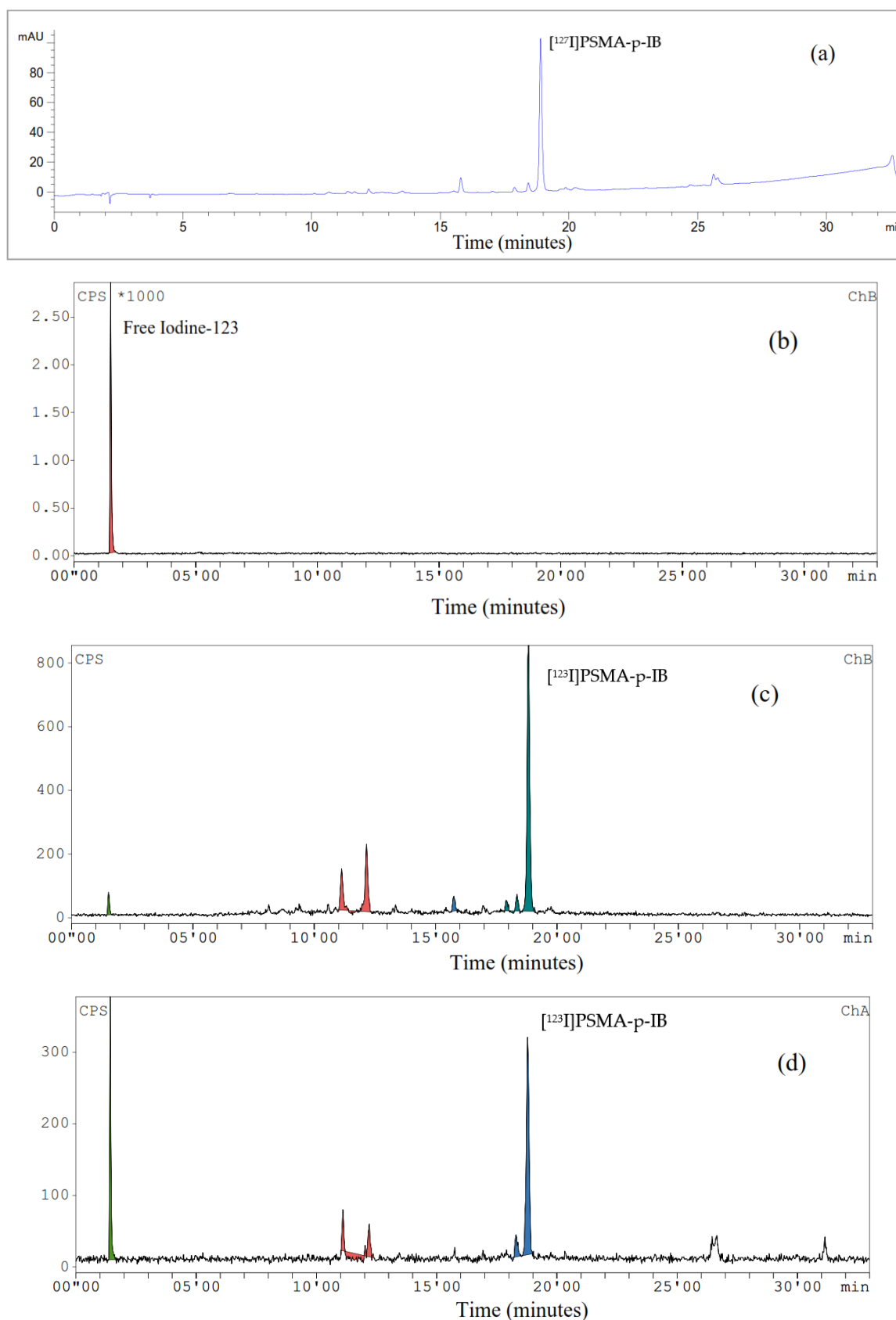


Figure S1. Radio-HPLC chromatograms of (a) $[^{127}\text{I}]\text{PSMA-p-IB}$ reference sample, (b) blank solution, (c) radiolabeling yield with $[^{123}\text{I}]\text{PSMA-p-IB}$ yield of 69.99%, and (d) radiolabeling yield with $[^{123}\text{I}]\text{PSMA-p-IB}$ yield of 65.73%.

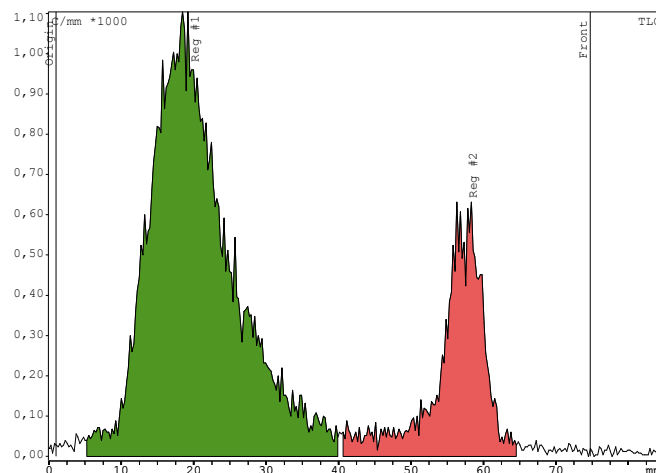


Figure S2. Radio-iTLC chromatogram of [^{123}I]PSMA-p-IB based on the results of radiolabeling optimization (the left peak: [^{123}I]PSMA-p-IB, the right peak: free Iodine-123). The chromatography was performed using iTLC-SG glass fiber sheet with a developing solution of $\text{CH}_3\text{CN}/\text{H}_2\text{O}$, 95/5 (v/v).

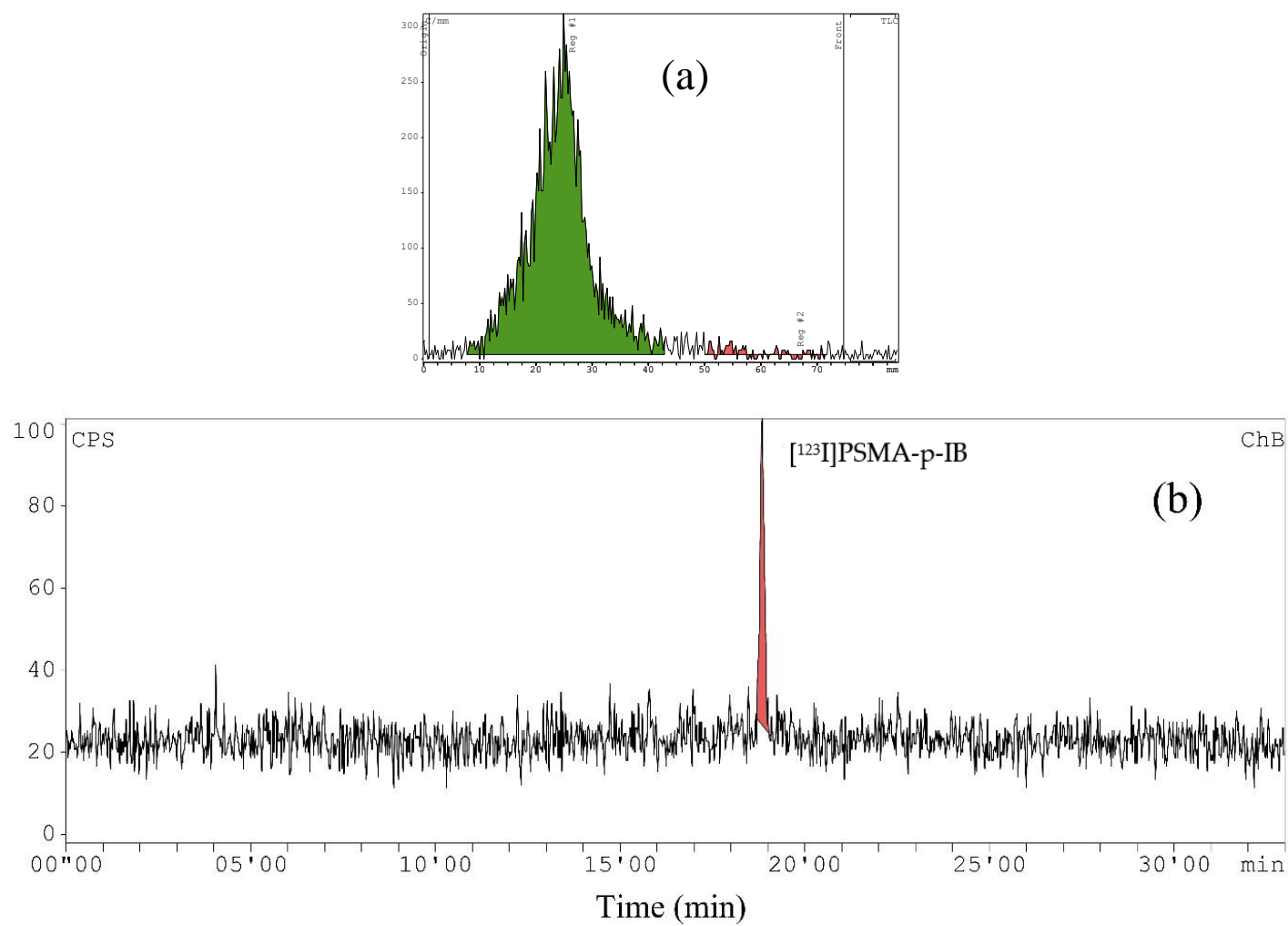


Figure S3. Chromatograms of $[^{123}\text{I}]\text{PSMA-p-IB}$ after purification analyzed by (a) Radio-TLC-SG 60 F254 and (b) Radio-HPLC

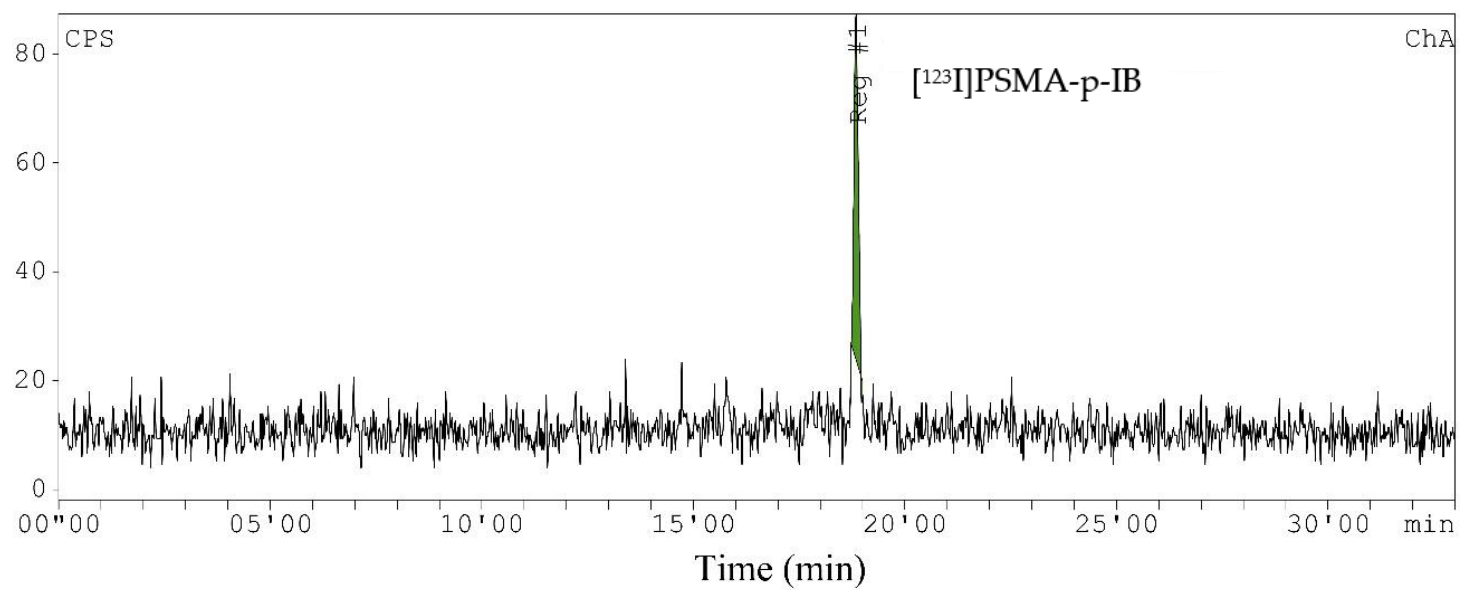


Figure S4. Radio-HPLC chromatogram of [^{123}I]PSMA-p-IB after 3 days storage period

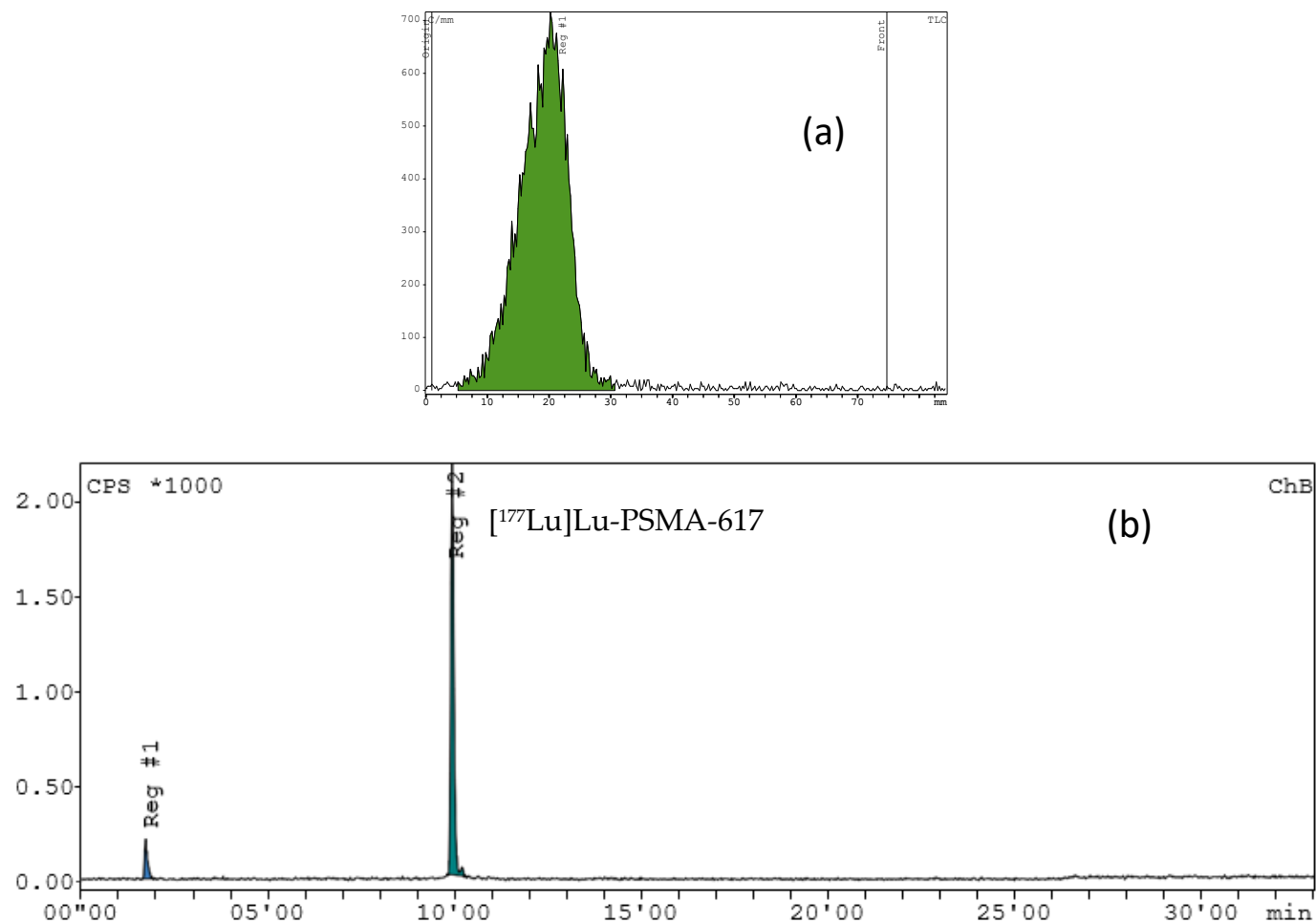


Figure S5. Chromatograms of $[^{177}\text{Lu}]\text{Lu-PSMA-617}$ by (a) Radio-iTLC and (b) Radio-HPLC

Compound 6

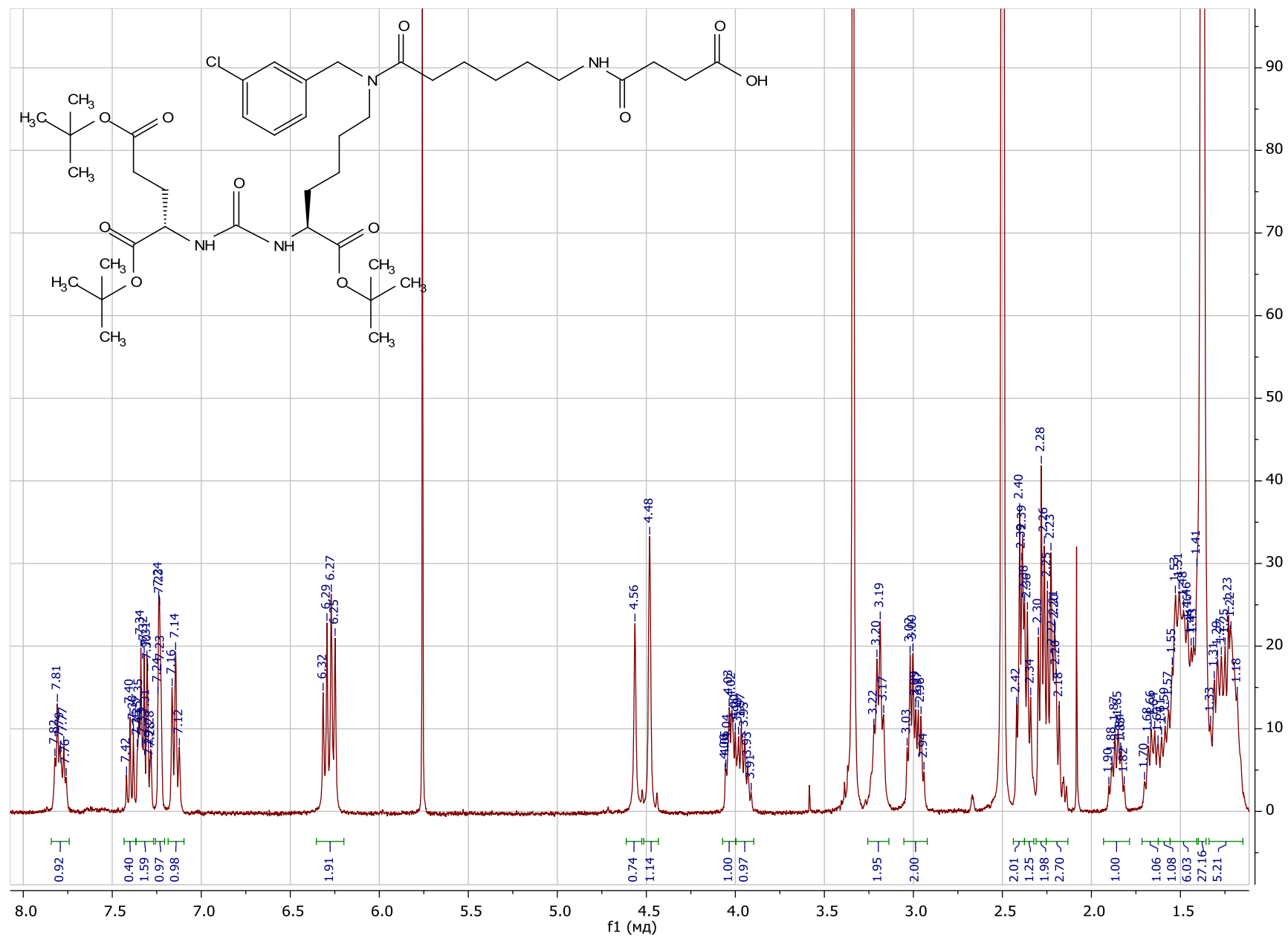


Figure S6. ¹H NMR spectrum of compound N^o 6 in DMSO-*d*₆.

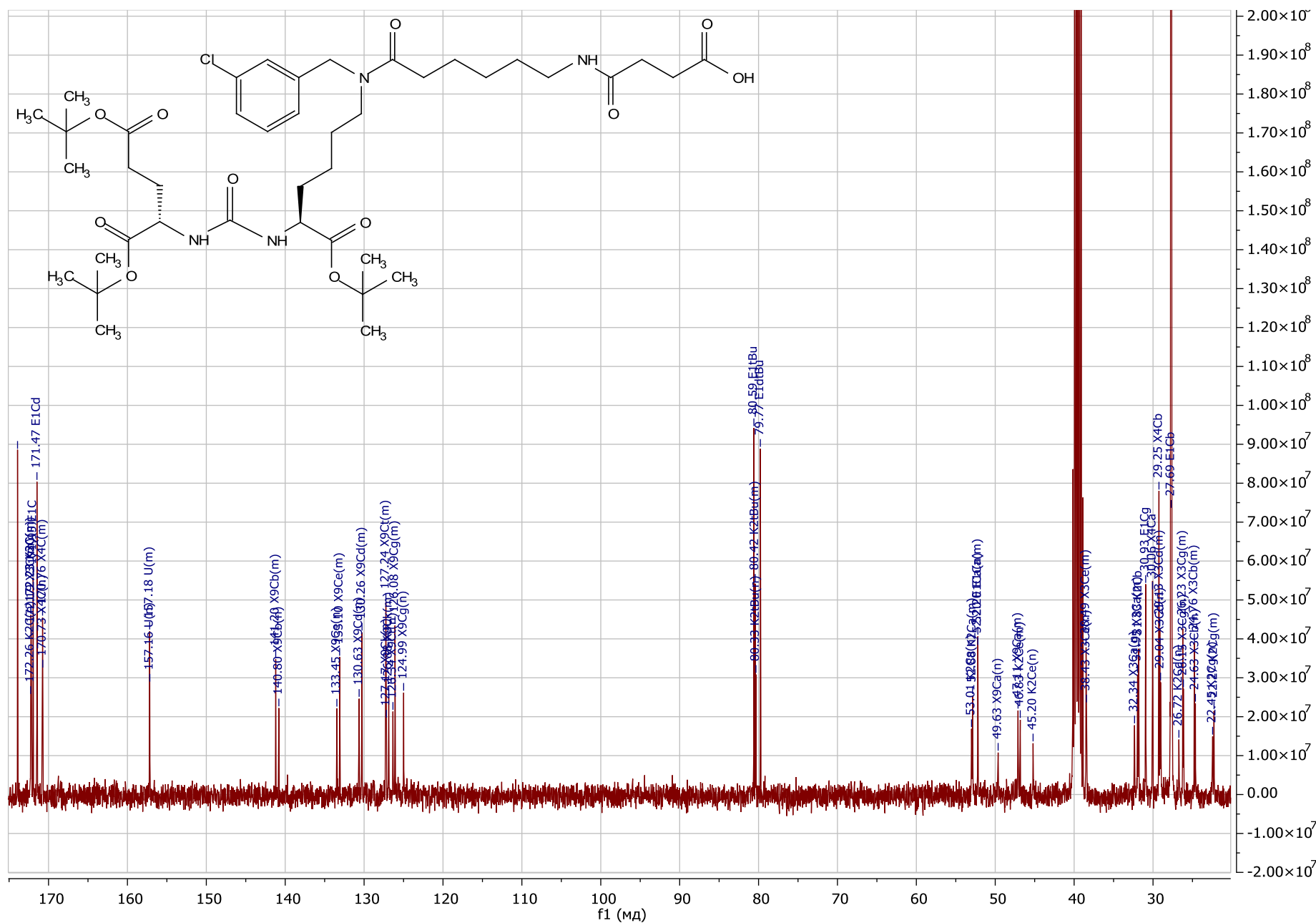


Figure S7. ¹³C NMR spectrum of compound № 6 in DMSO-*d*₆.

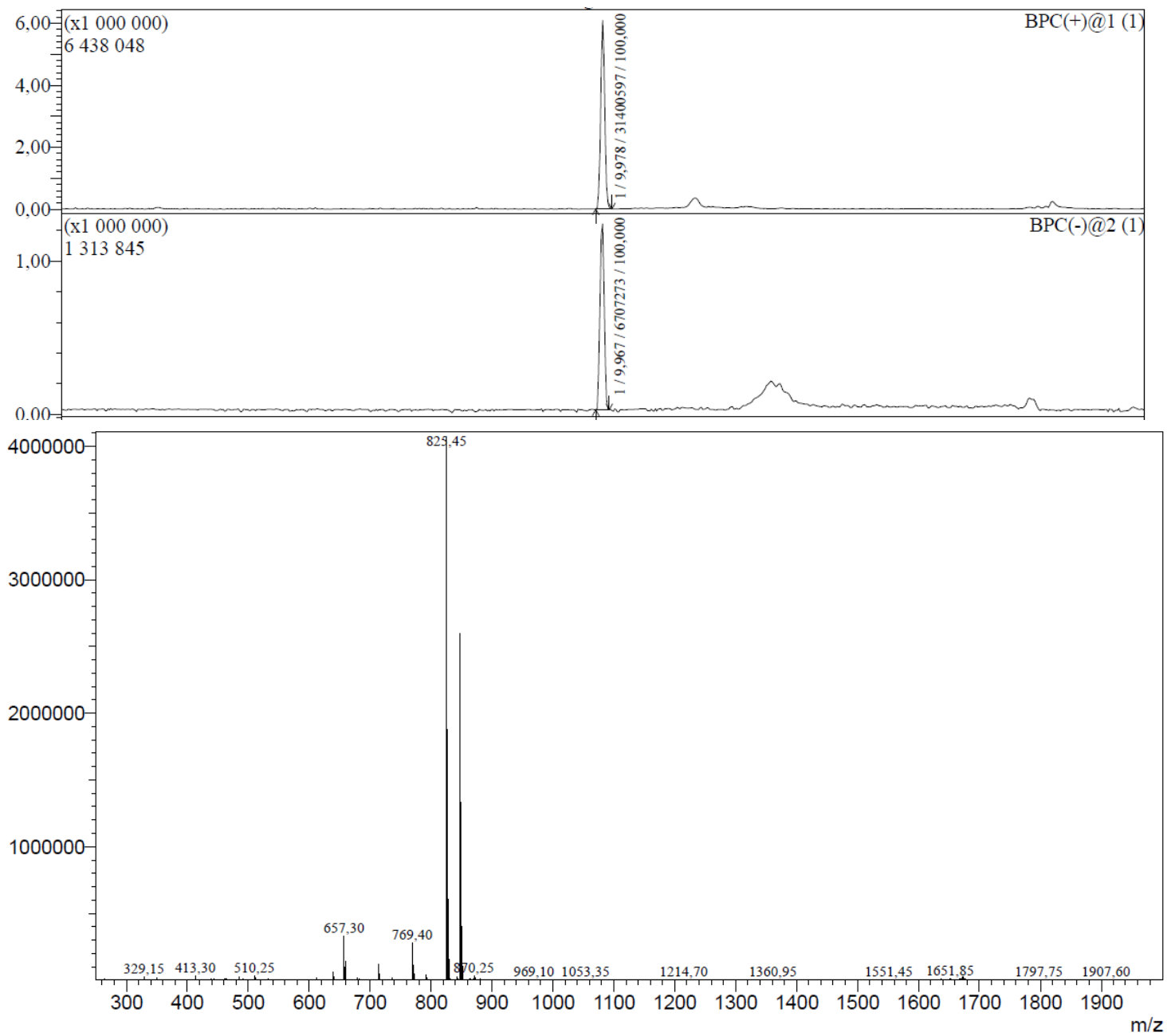


Figure S8. ESI-MS of compound № 6

Compound 9

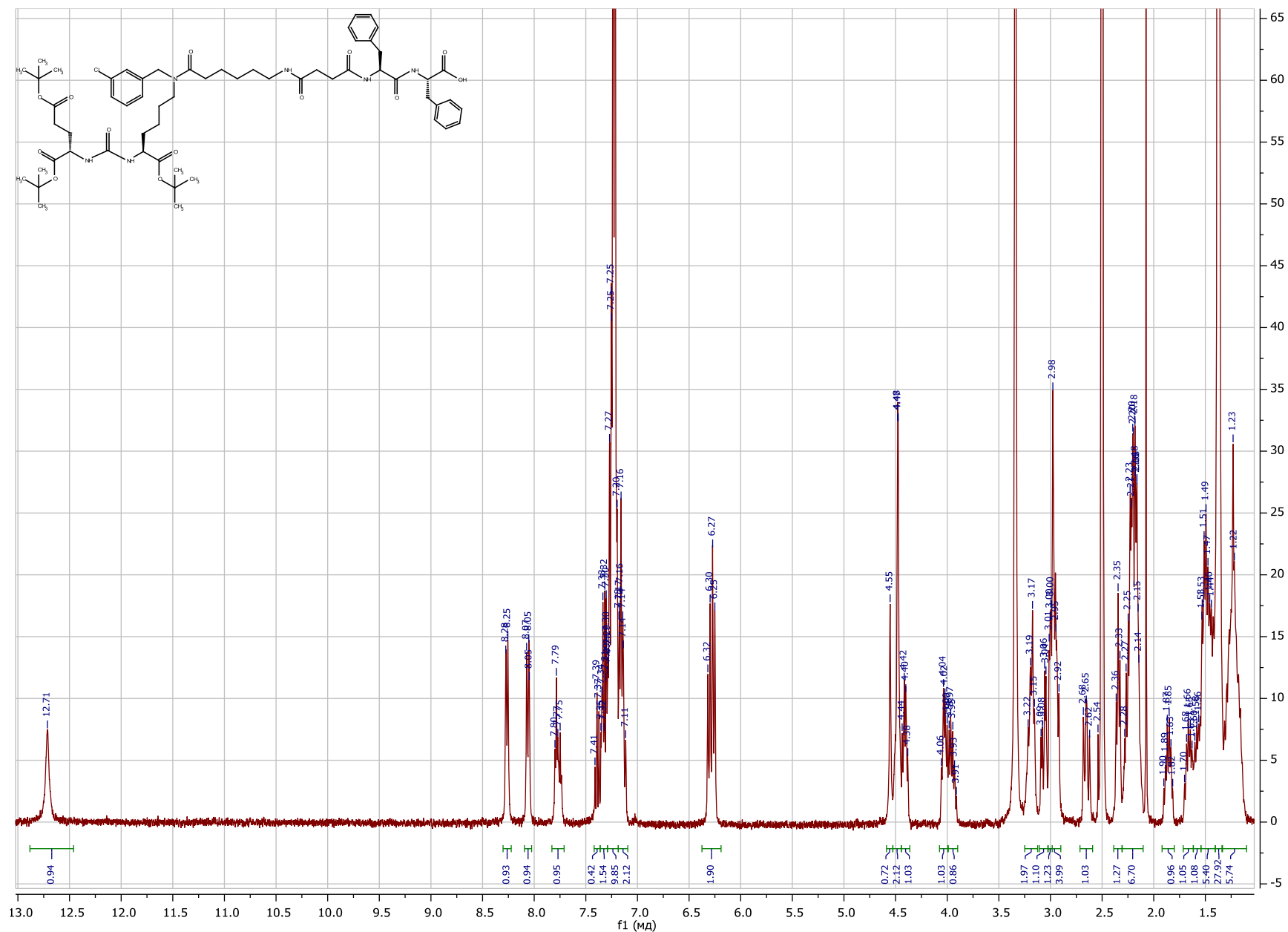
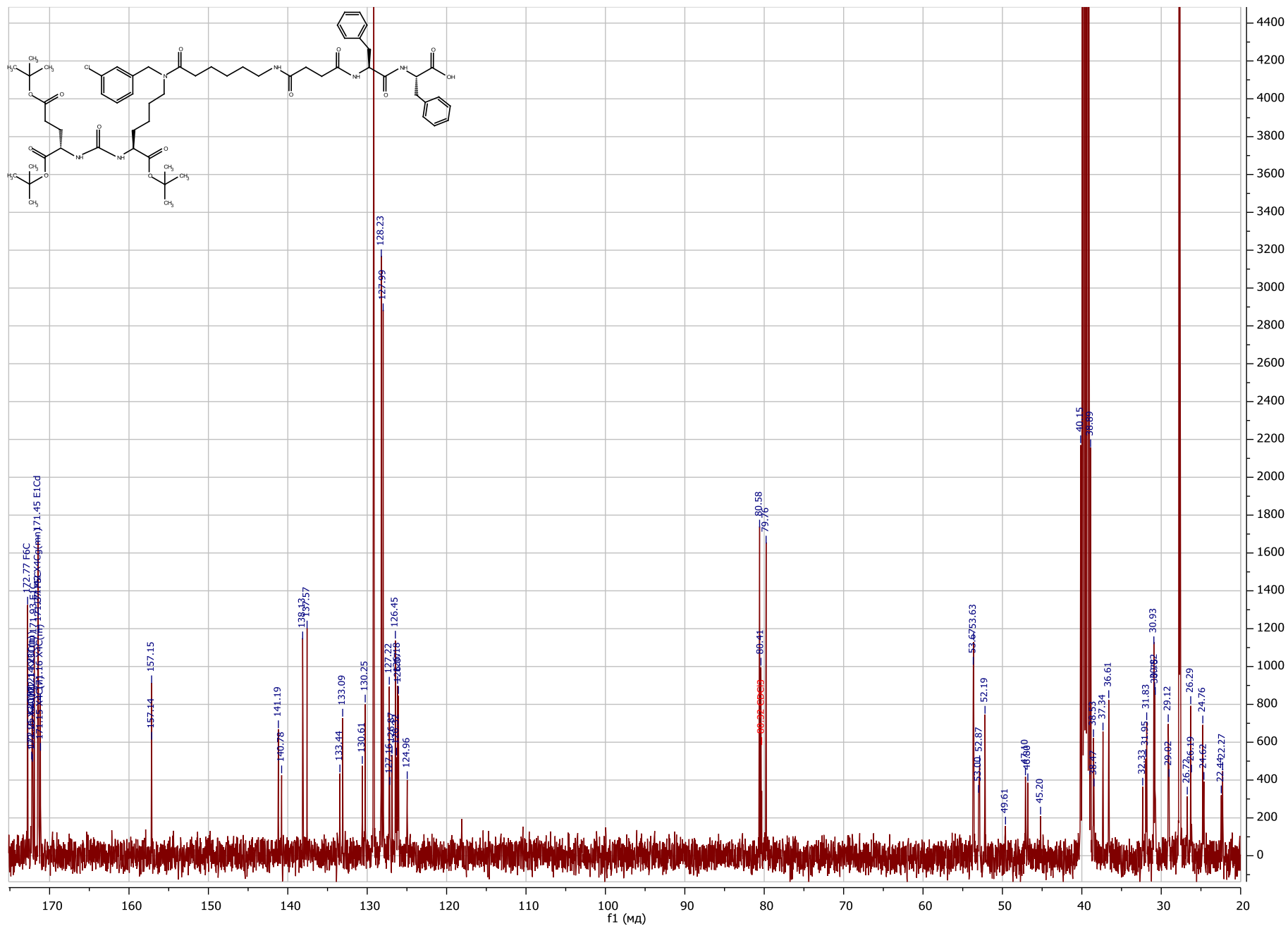


Figure S9. ¹H NMR spectrum of compound 9 in DMSO-*d*₆.



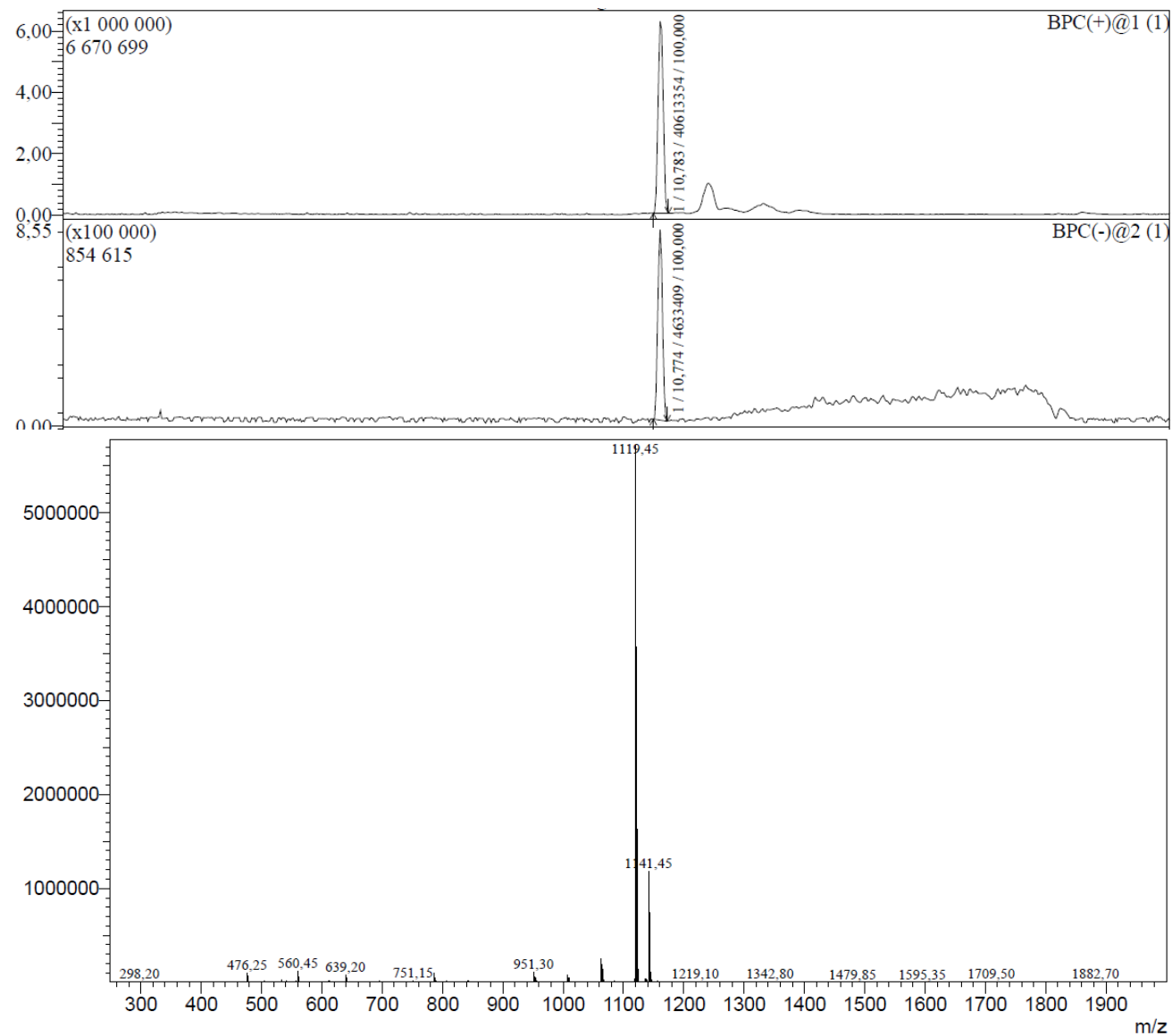


Figure S11. ESI-MS of compound № 9

Spectrum from 070320_POS.wiff (sample 39) - PS-114, +TOF MS (200 - 3000) from 0.167 to 0.209 min, subtracted by (Spectrum from 070320_POS.wiff (sample 39) - PS-114, +TOF MS (200 - 3000) from 0.079 to 0.125 min)

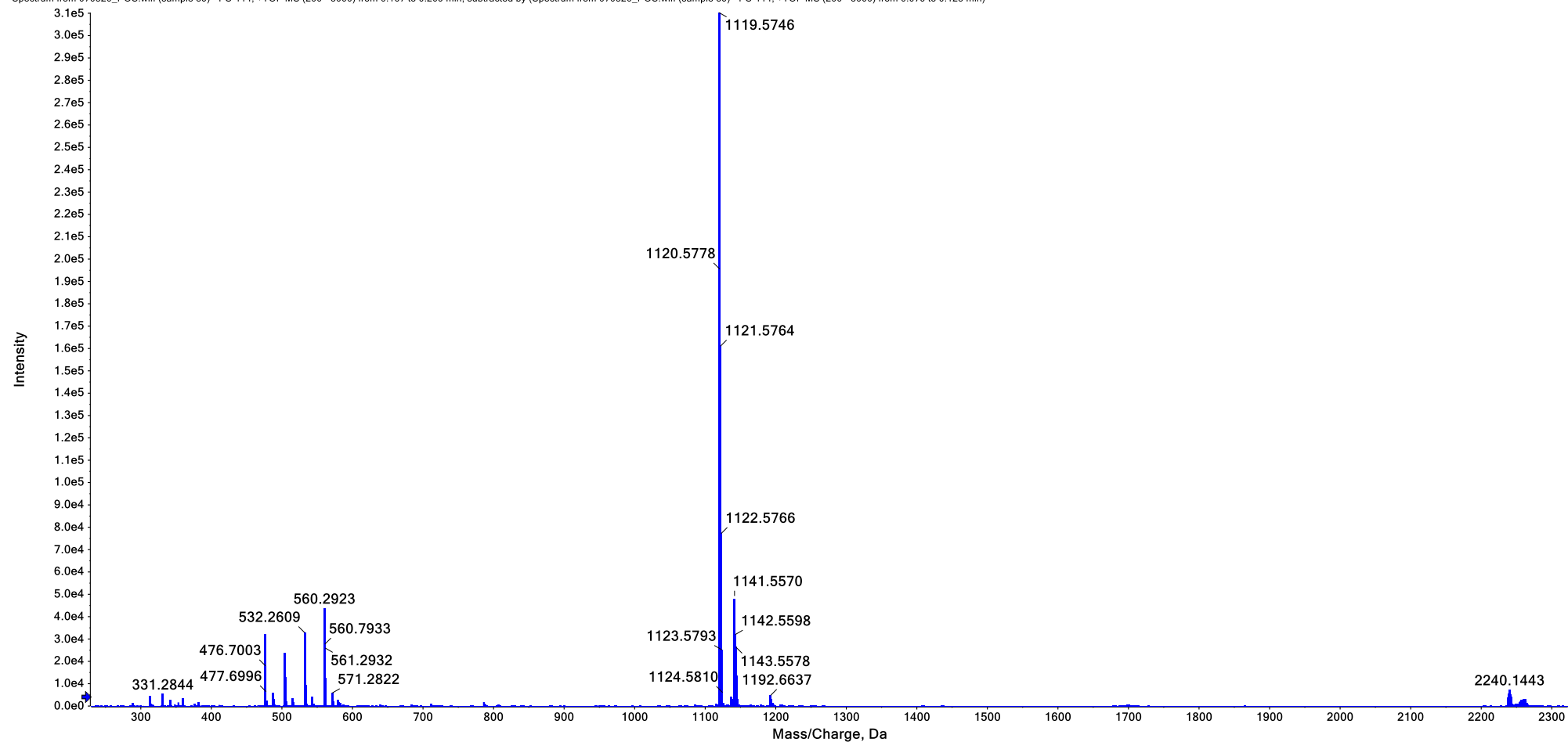


Figure S12. HRMS (m/z, ESI) of compound No 9

Compound 10

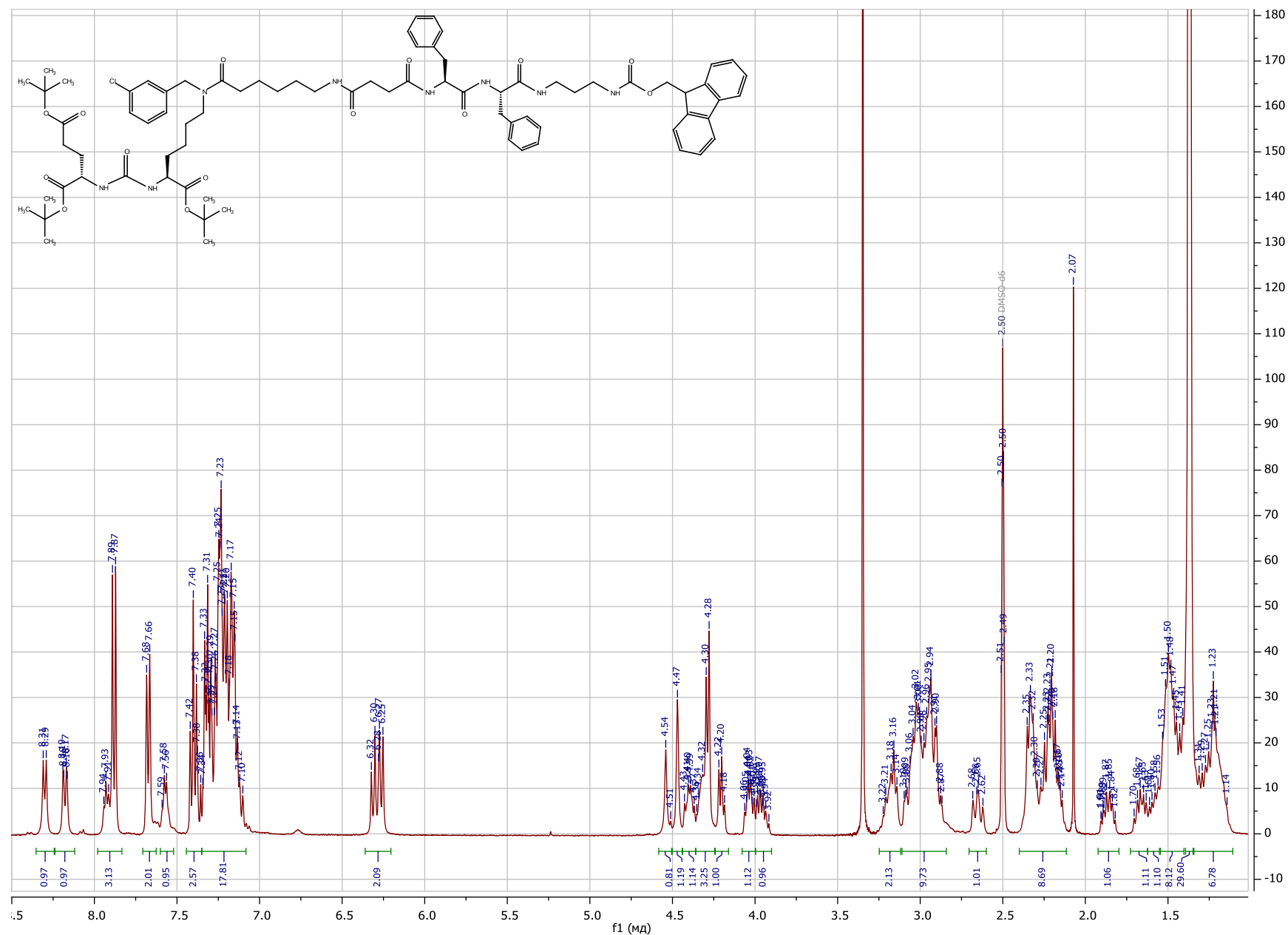


Figure S13. ¹H NMR spectrum of compound N^o 10 in DMSO-*d*₆.

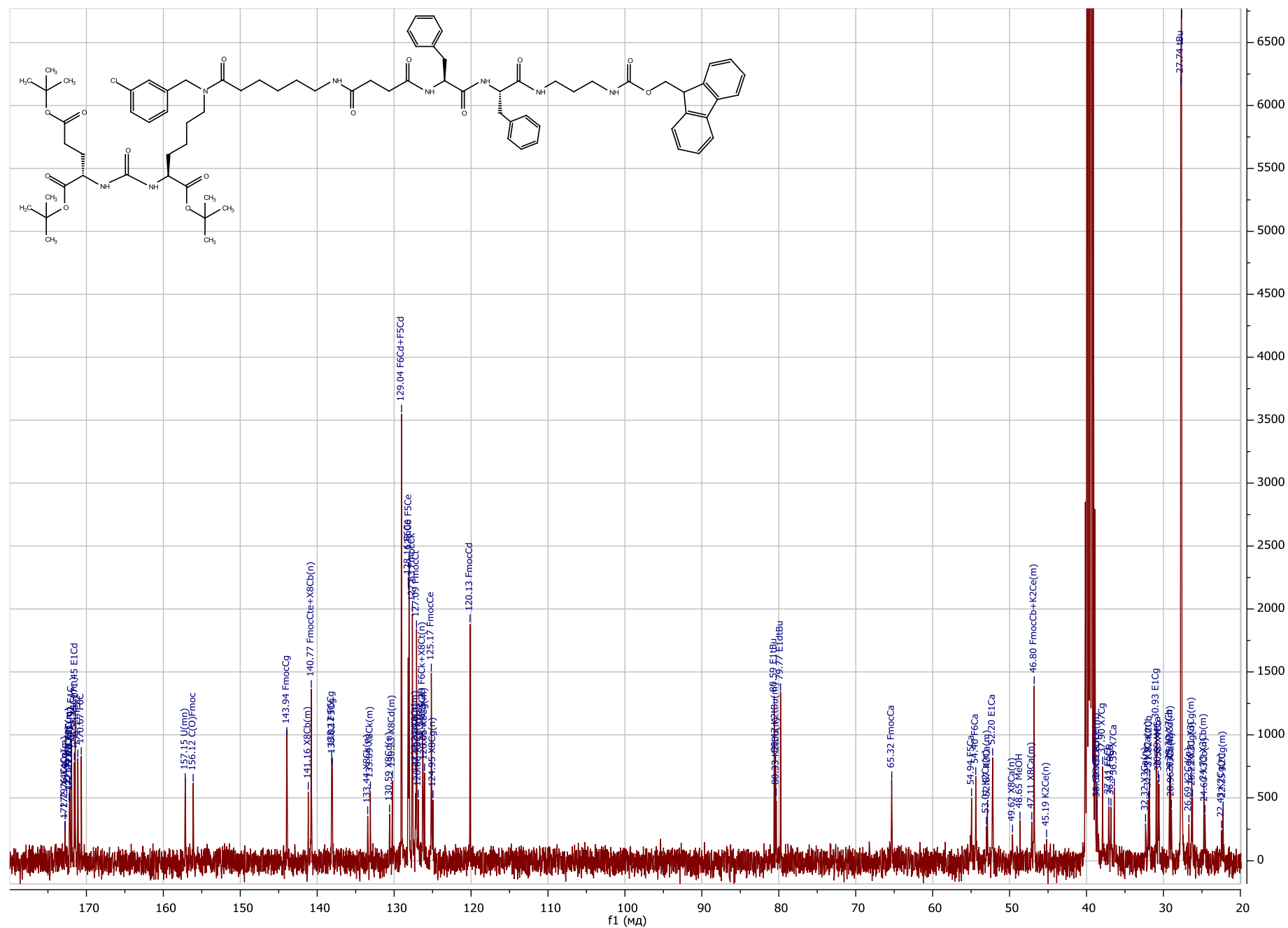


Figure S14. ^{13}C NMR spectrum of compound № 10 in $\text{DMSO}-d_6$.

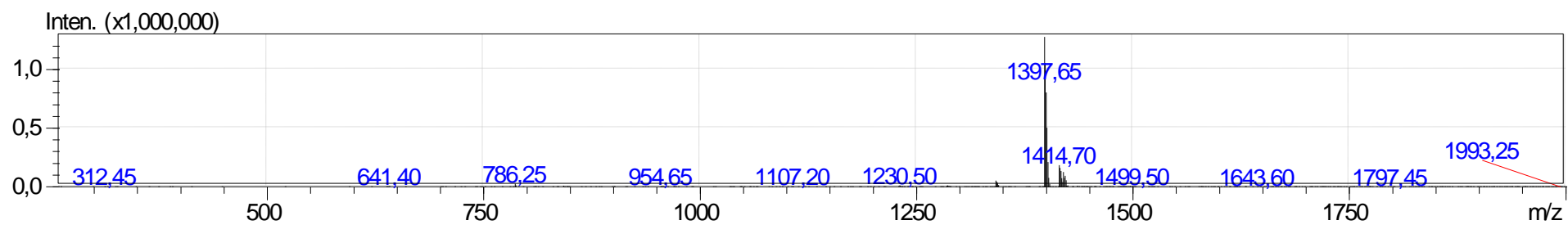
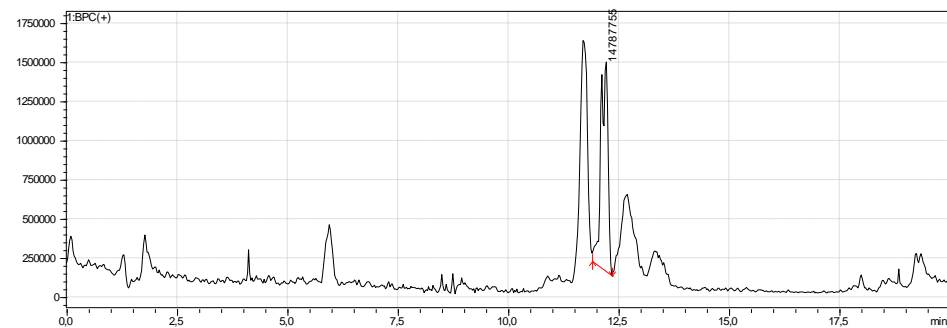
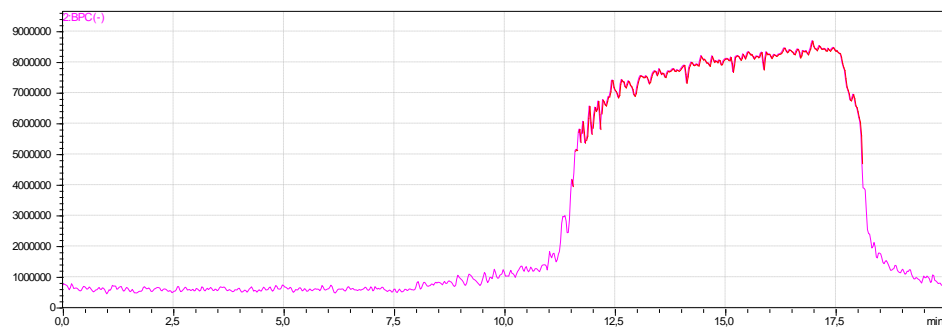


Figure S15. ESI-MS of compound № 10

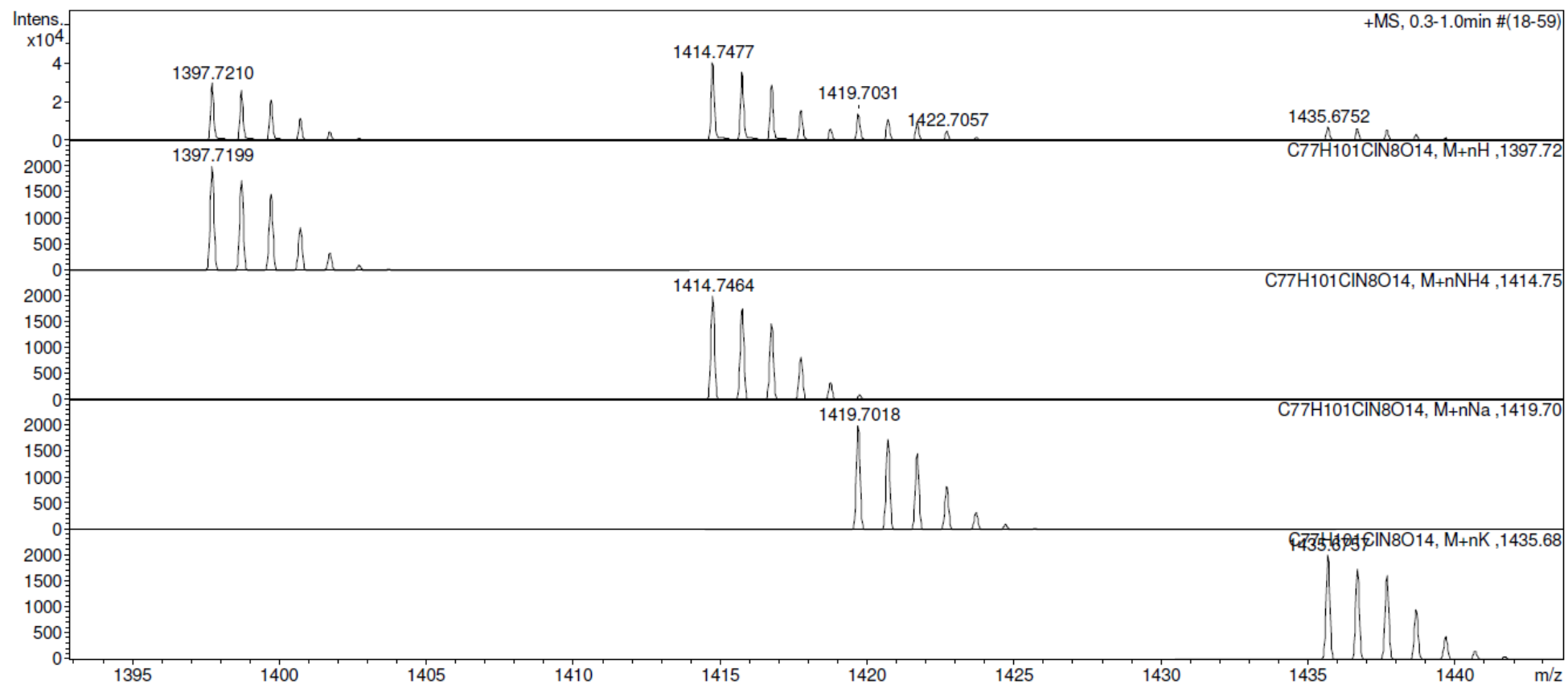


Figure S16. HRMS (m/z, ESI) of compound No 10

Compound 11

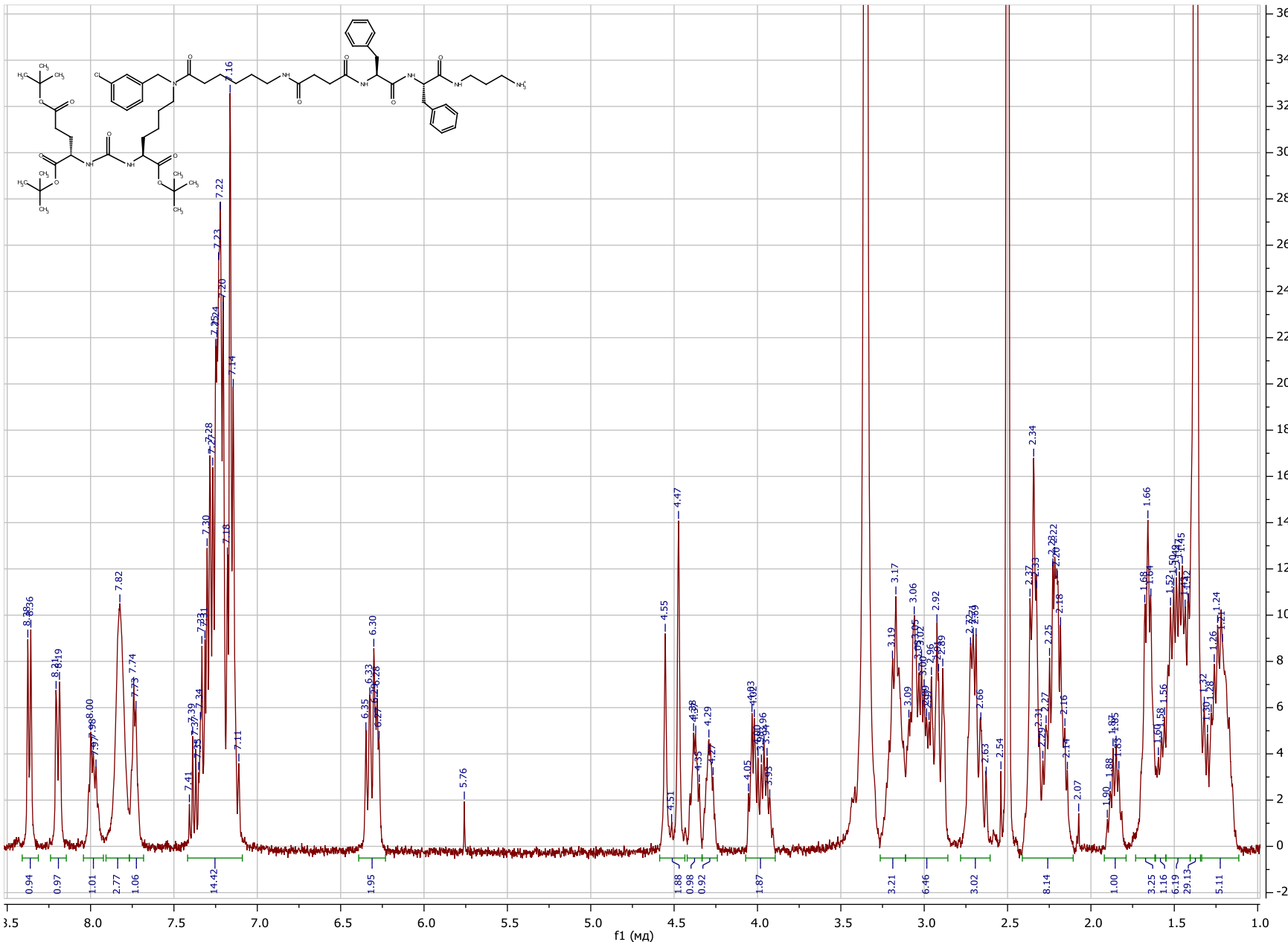


Figure S17. ^1H NMR spectrum of compound **11** in $\text{DMSO}-d_6$.

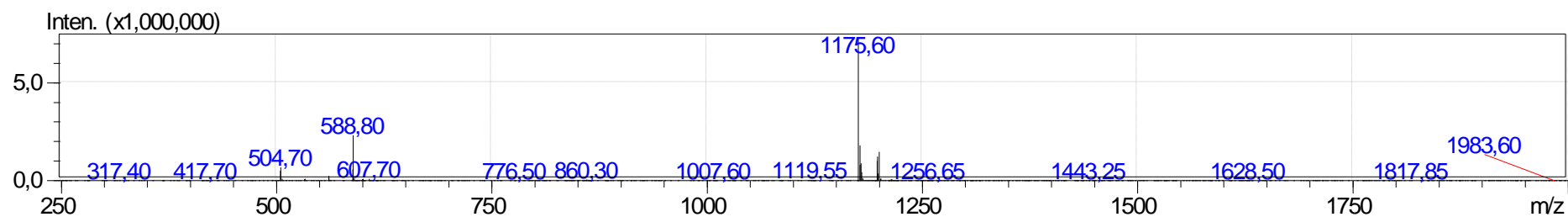
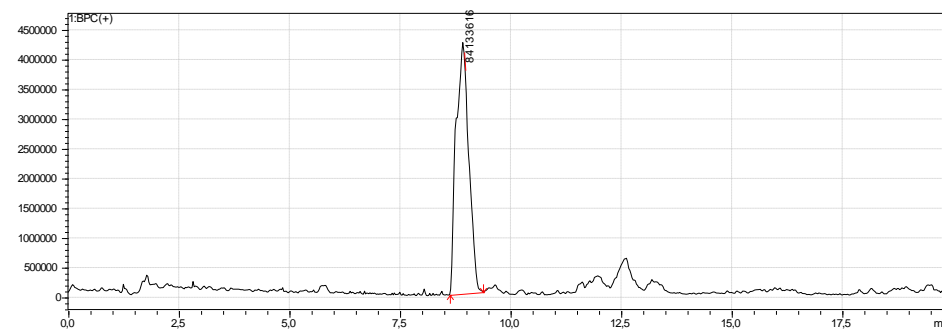
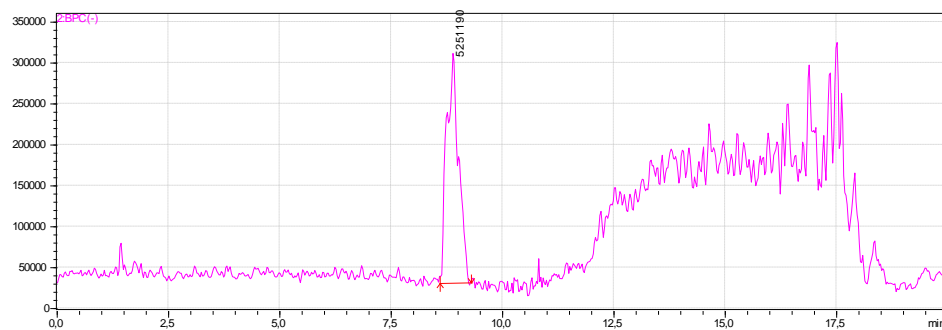


Figure S19. ESI-MS of compound № 11

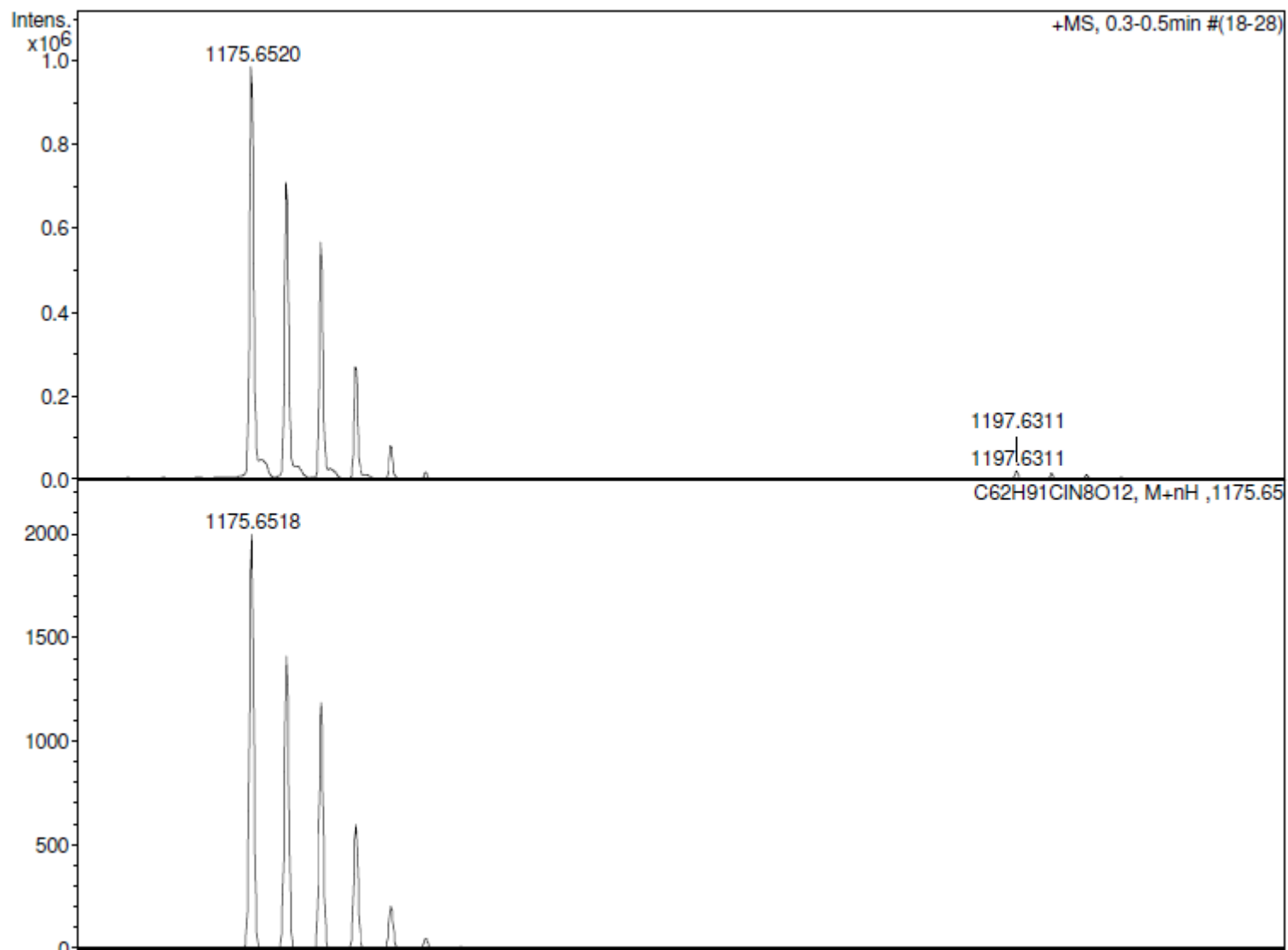


Figure S20. HRMS (m/z, ESI) of compound № 11

Compound 13

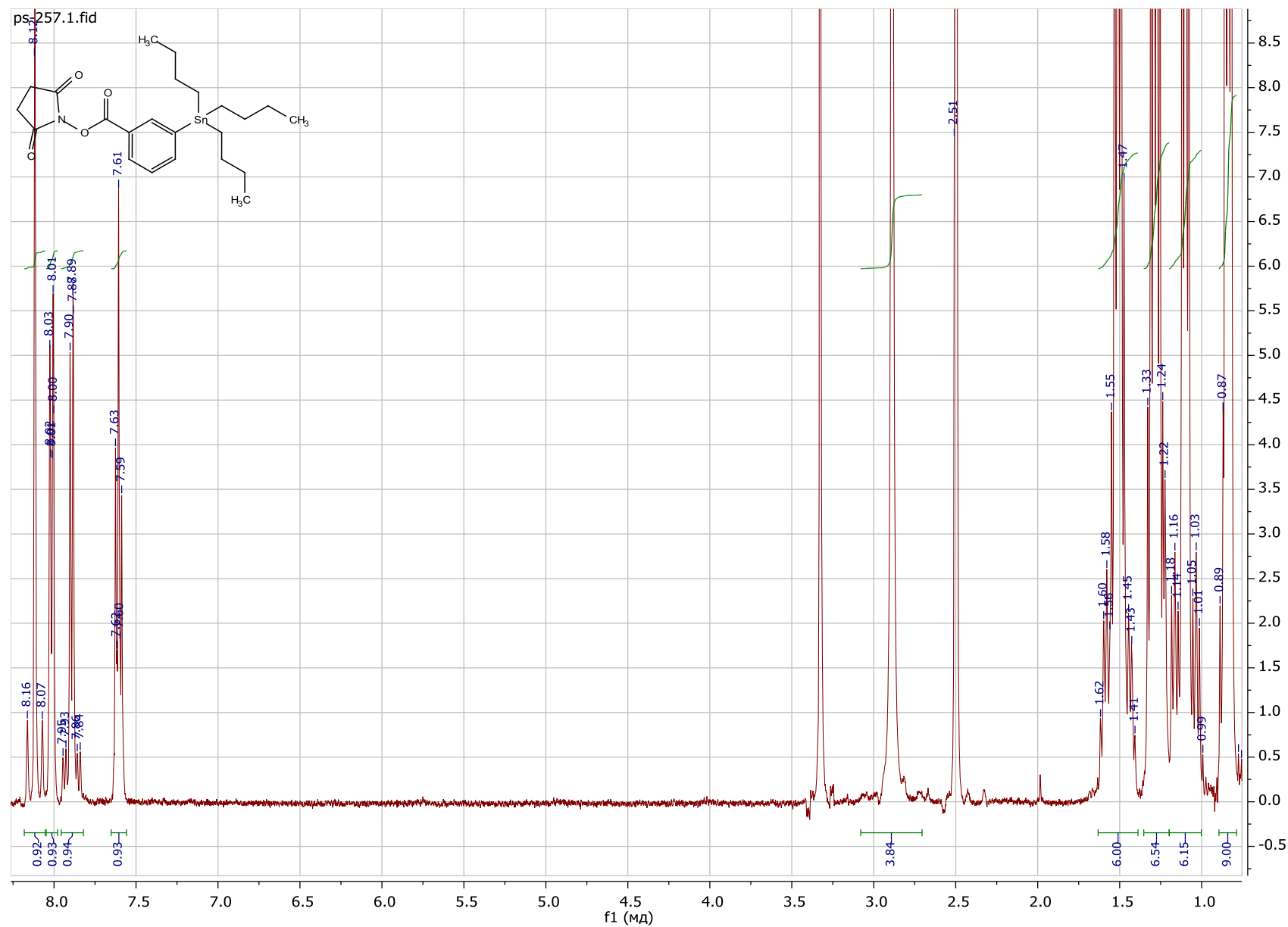


Figure S21. ¹H NMR spectrum of compound № 13 in DMSO-*d*₆.

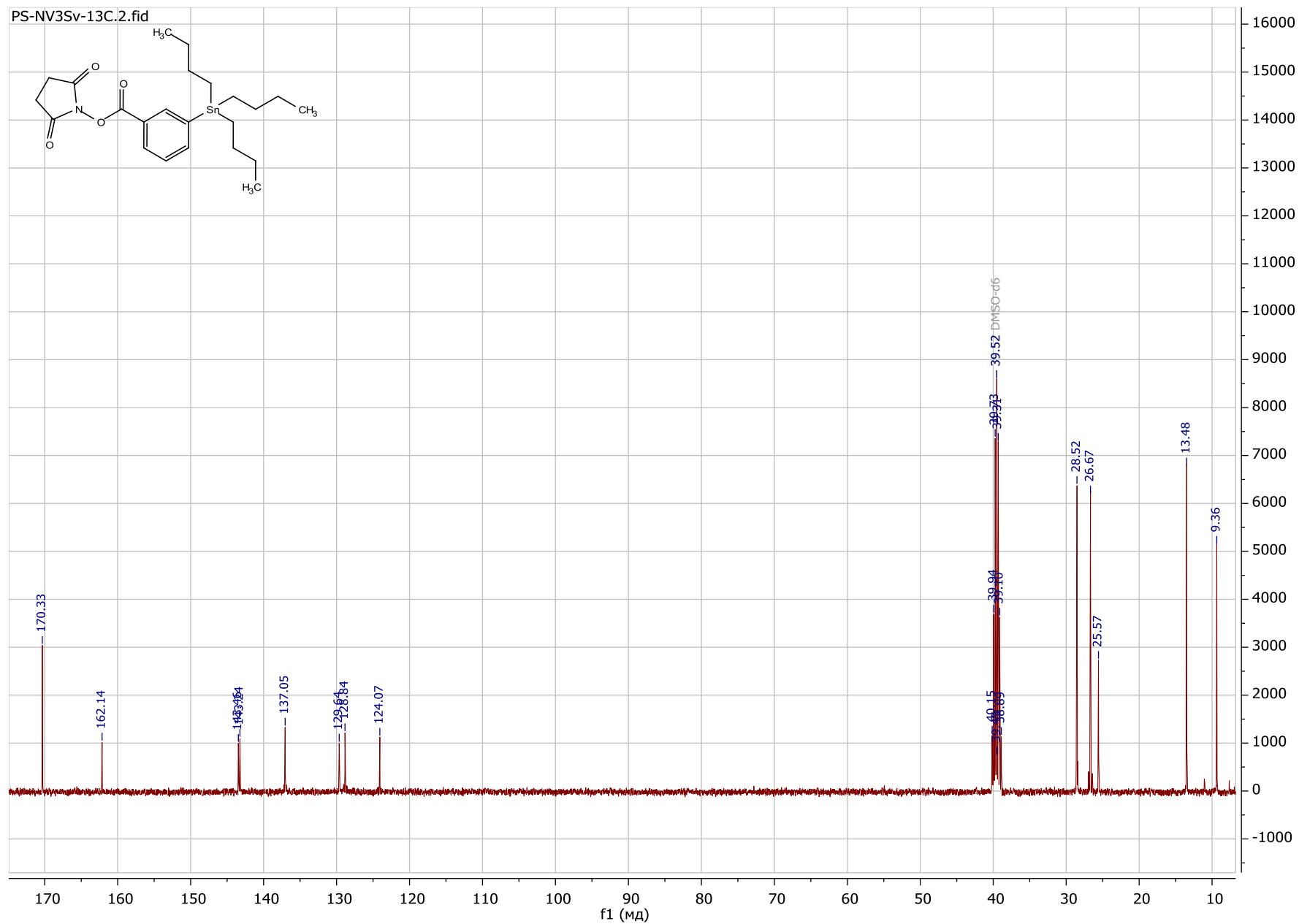


Figure S22. ^{13}C NMR spectrum of compound **13** in $\text{DMSO}-d_6$.

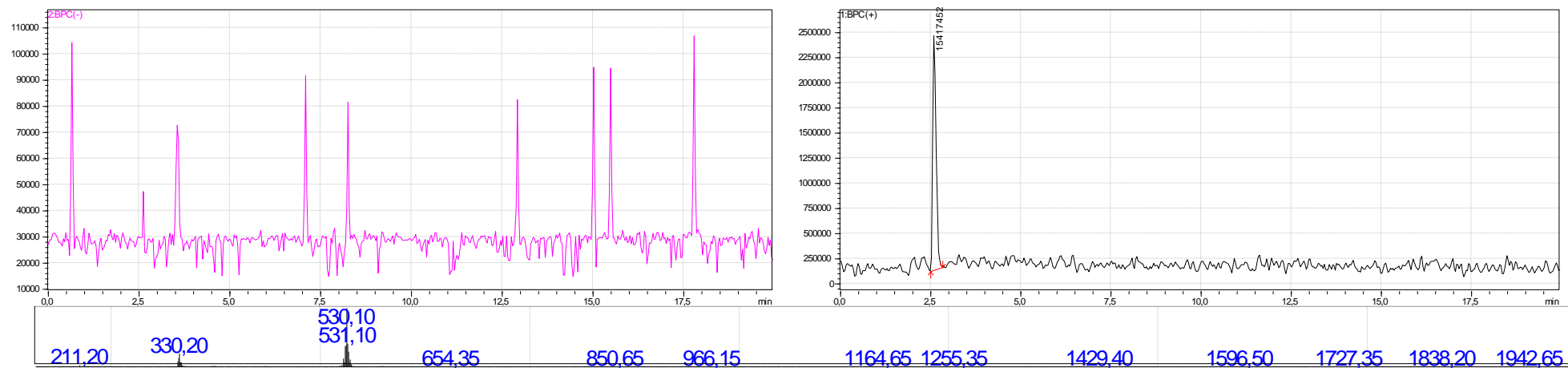


Figure S23. ESI-MS of compound № 13

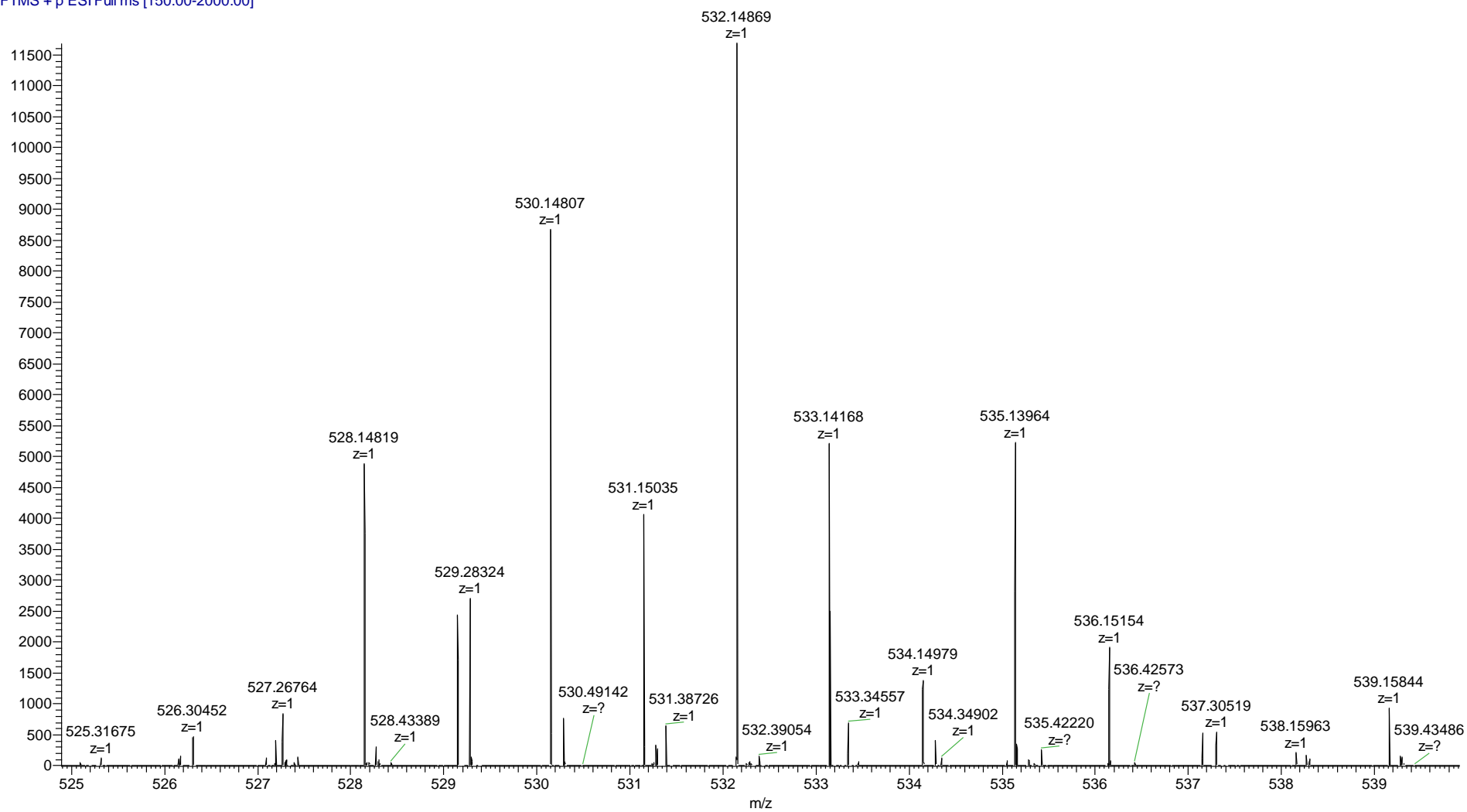


Figure S24. HRMS (m/z, ESI) of compound № 13

Compound 14

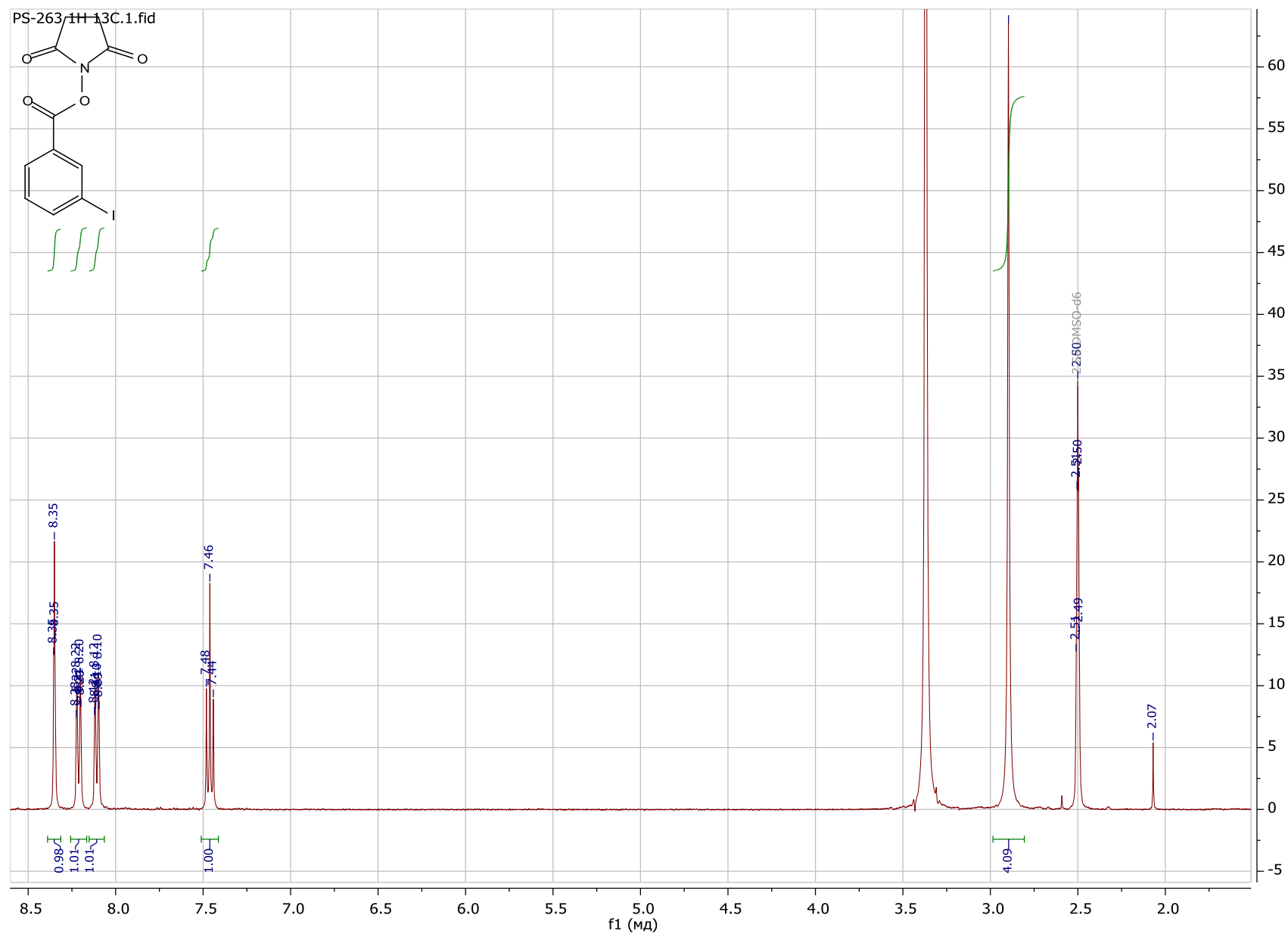


Figure S25. ¹H NMR spectrum of compound No 14 in DMSO-*d*₆.

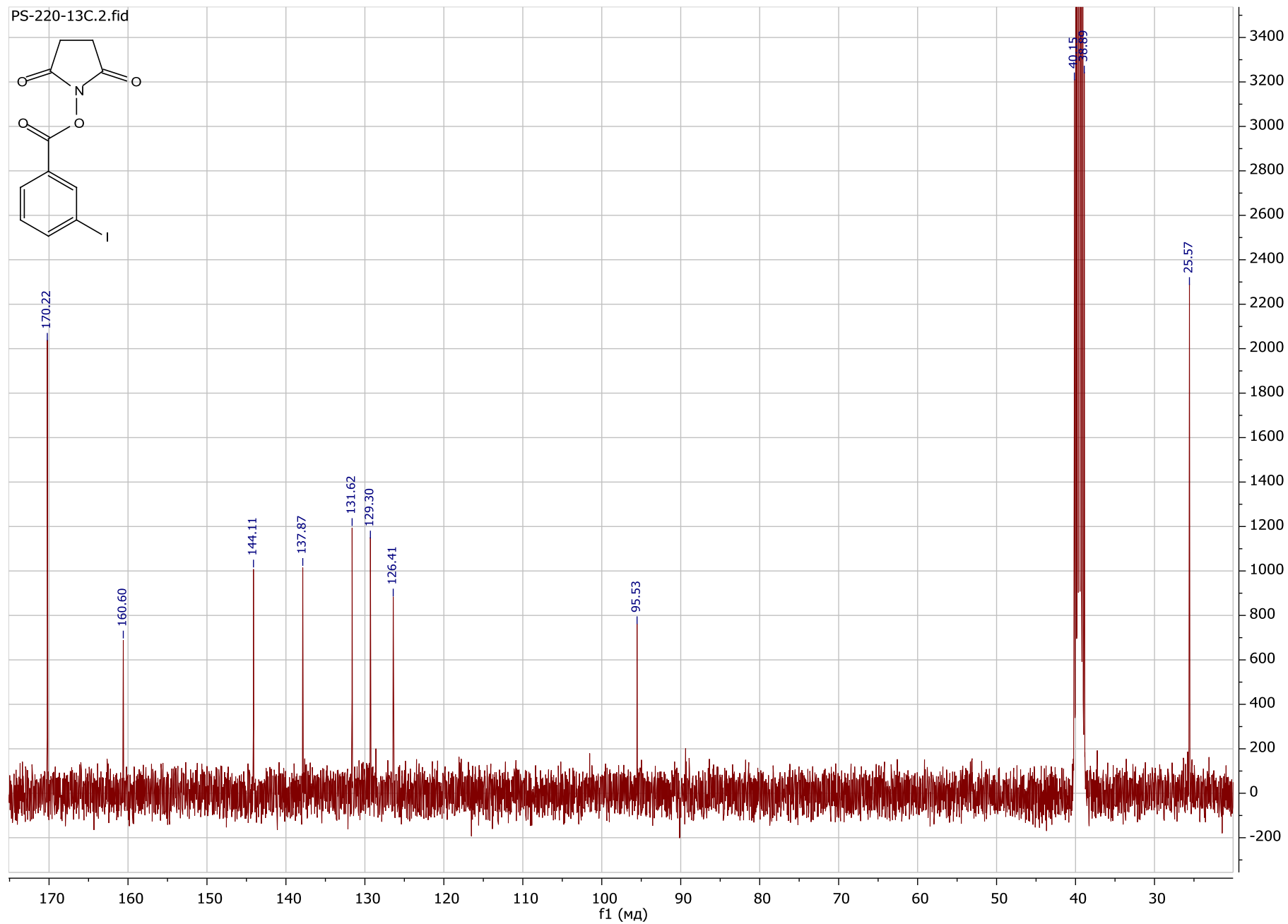


Figure S26. ^{13}C NMR spectrum of compound **14** in $\text{DMSO}-d_6$.

Compound 16

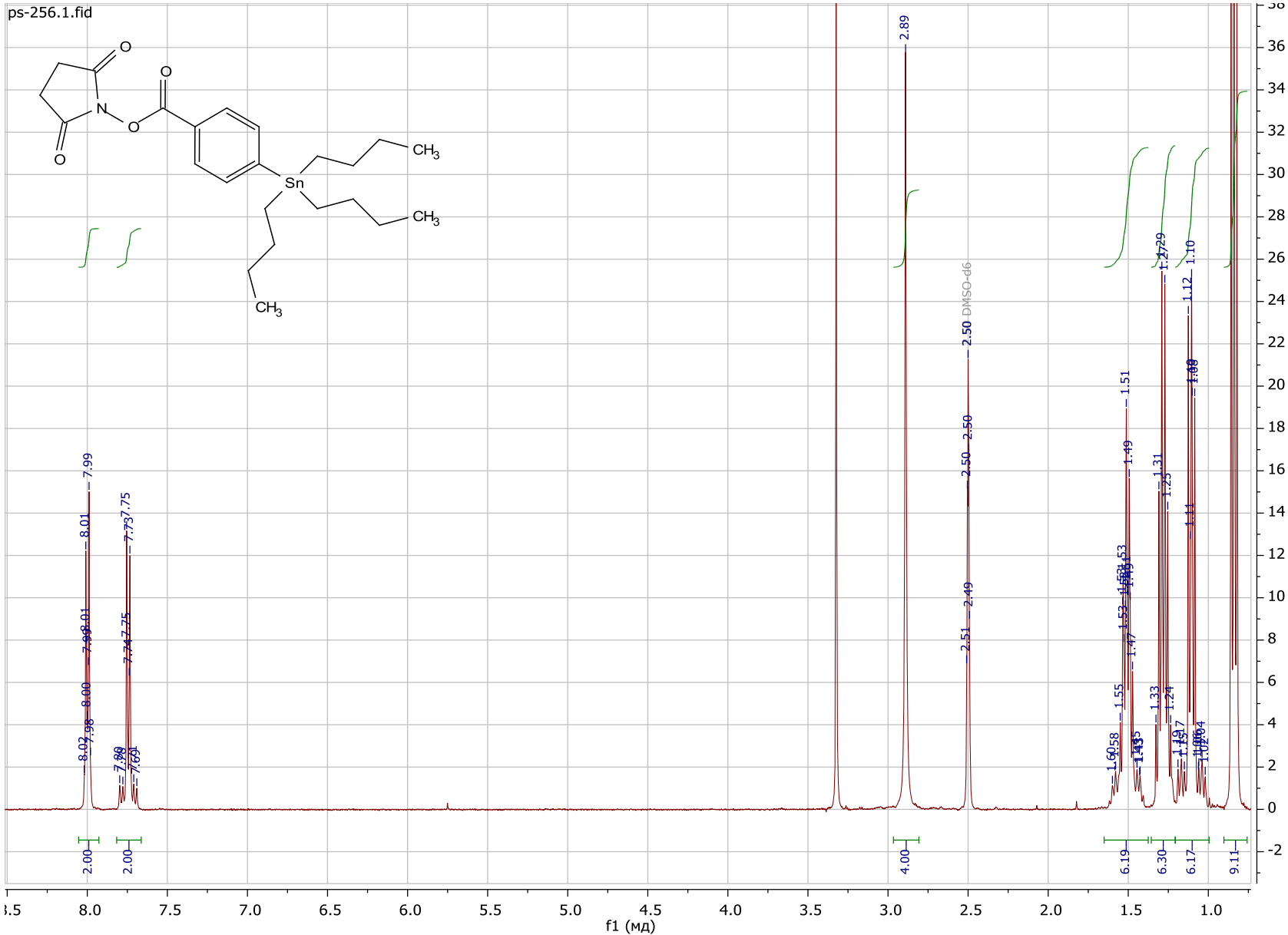


Figure S27. ^1H NMR spectrum of compound **16** in $\text{DMSO}-d_6$.

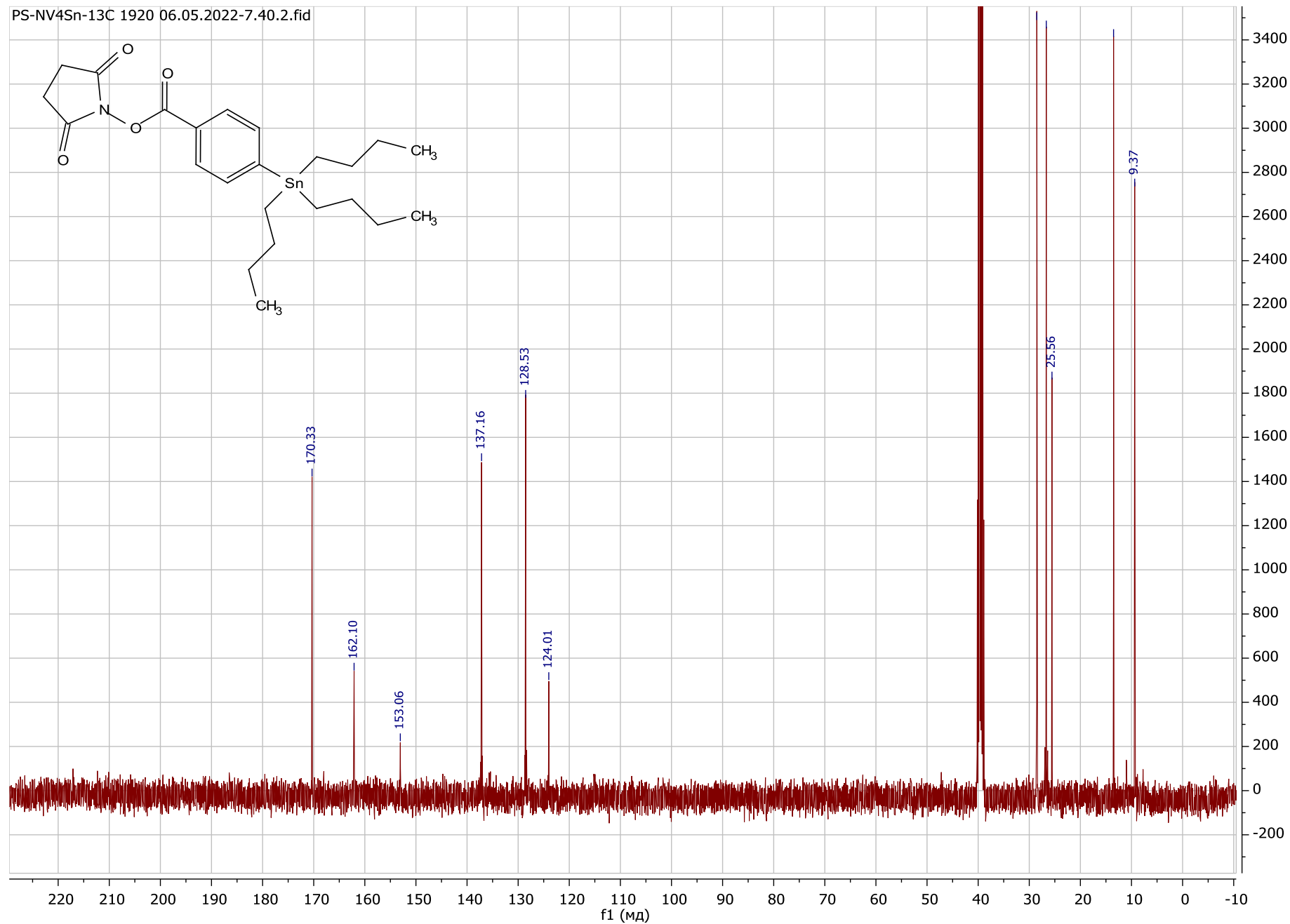


Figure S28. ^{13}C NMR spectrum of compound № 16 in $\text{DMSO}-d_6$.

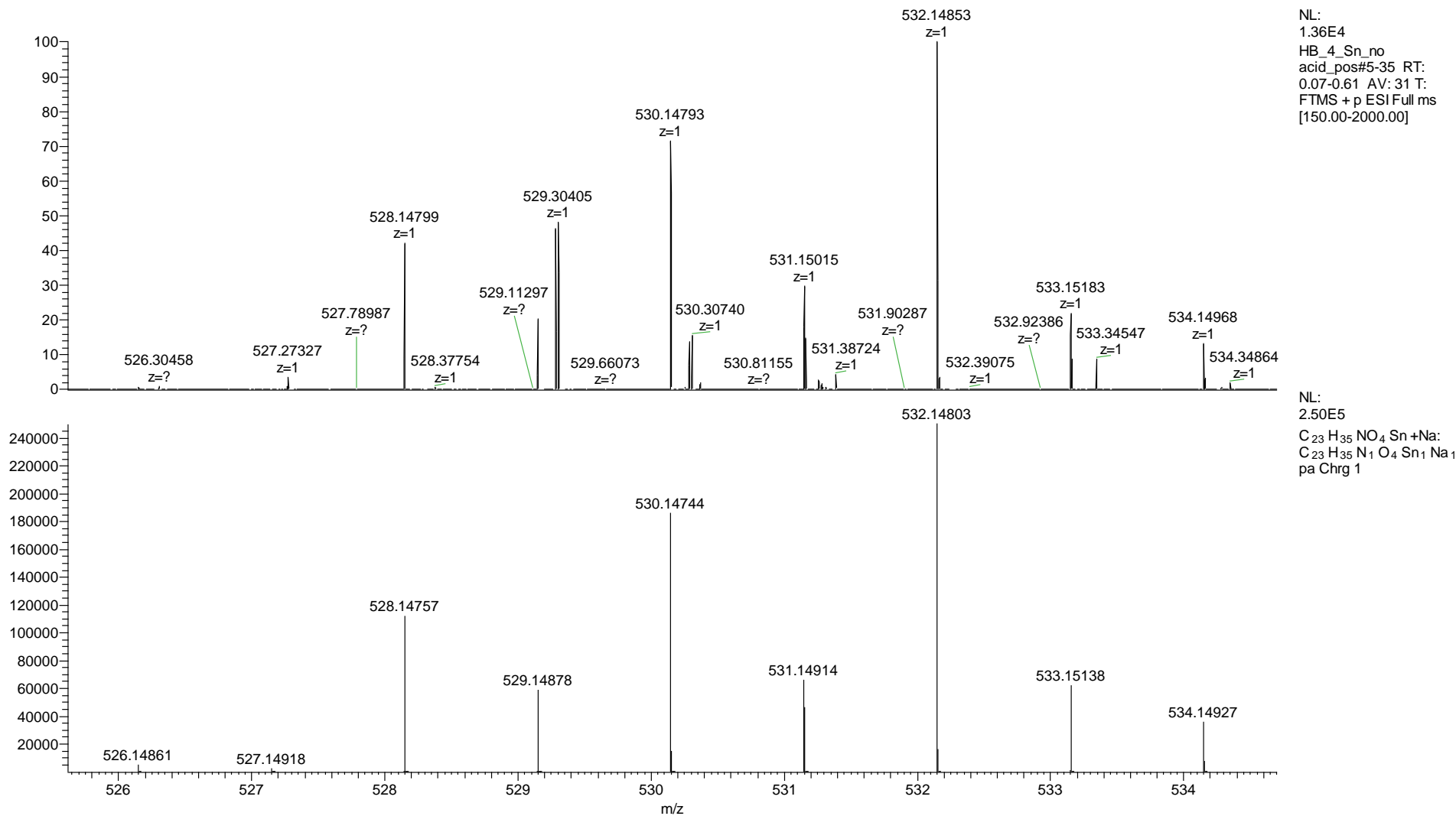


Figure S29. HRMS (m/z, ESI) of compound № 16

Compound 17

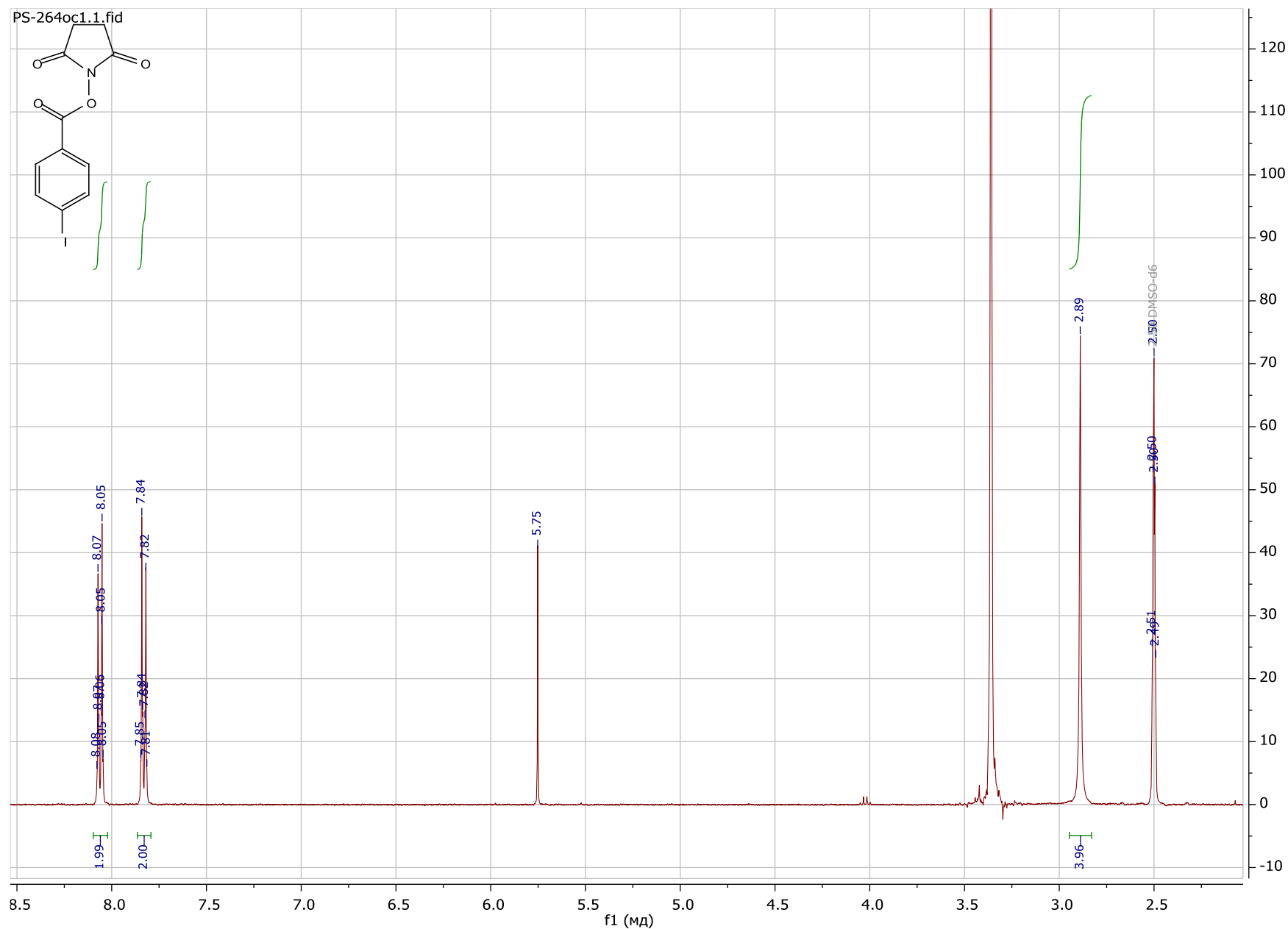
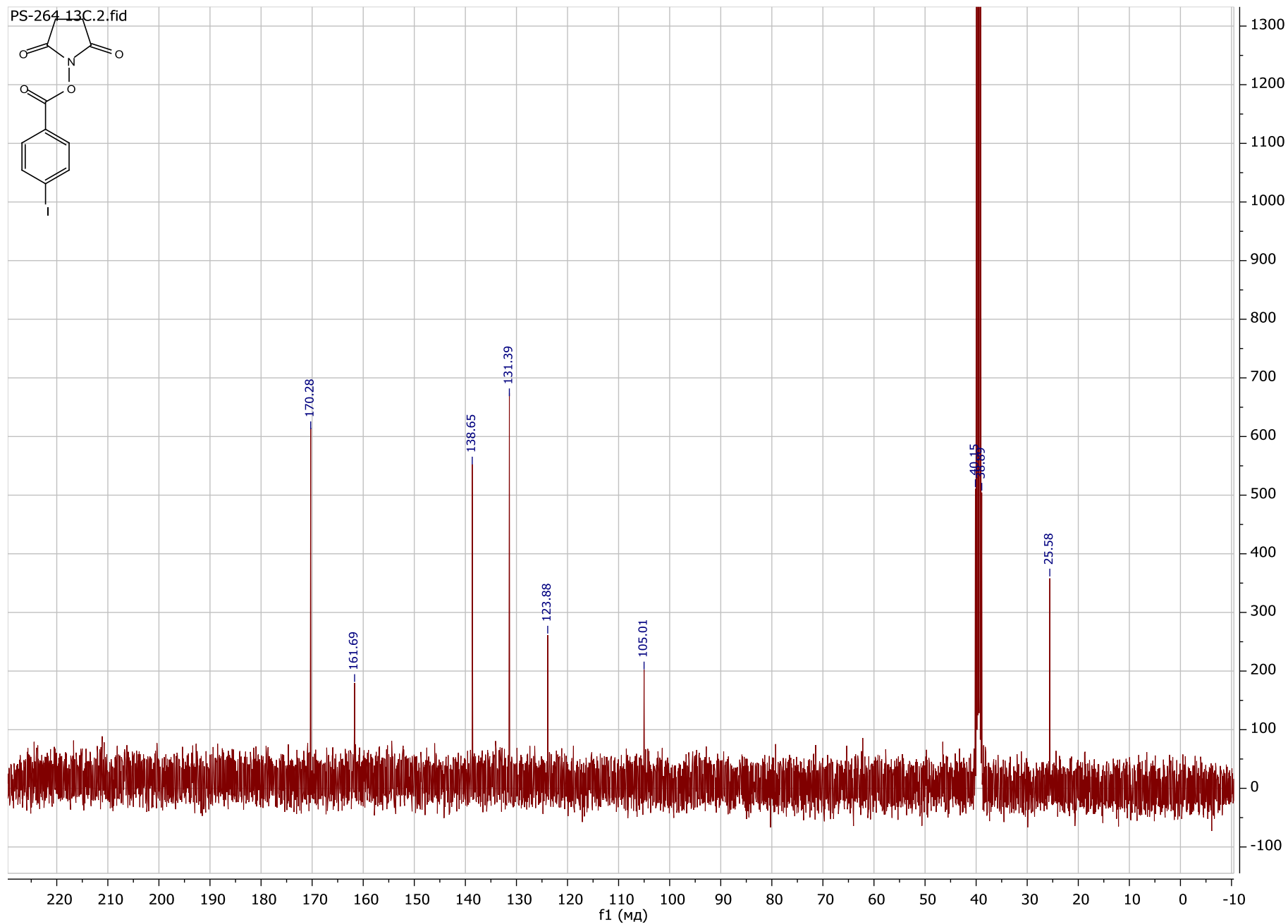


Figure S30. ¹H NMR spectrum of compound № 17 in DMSO-*d*₆.



Compound 18

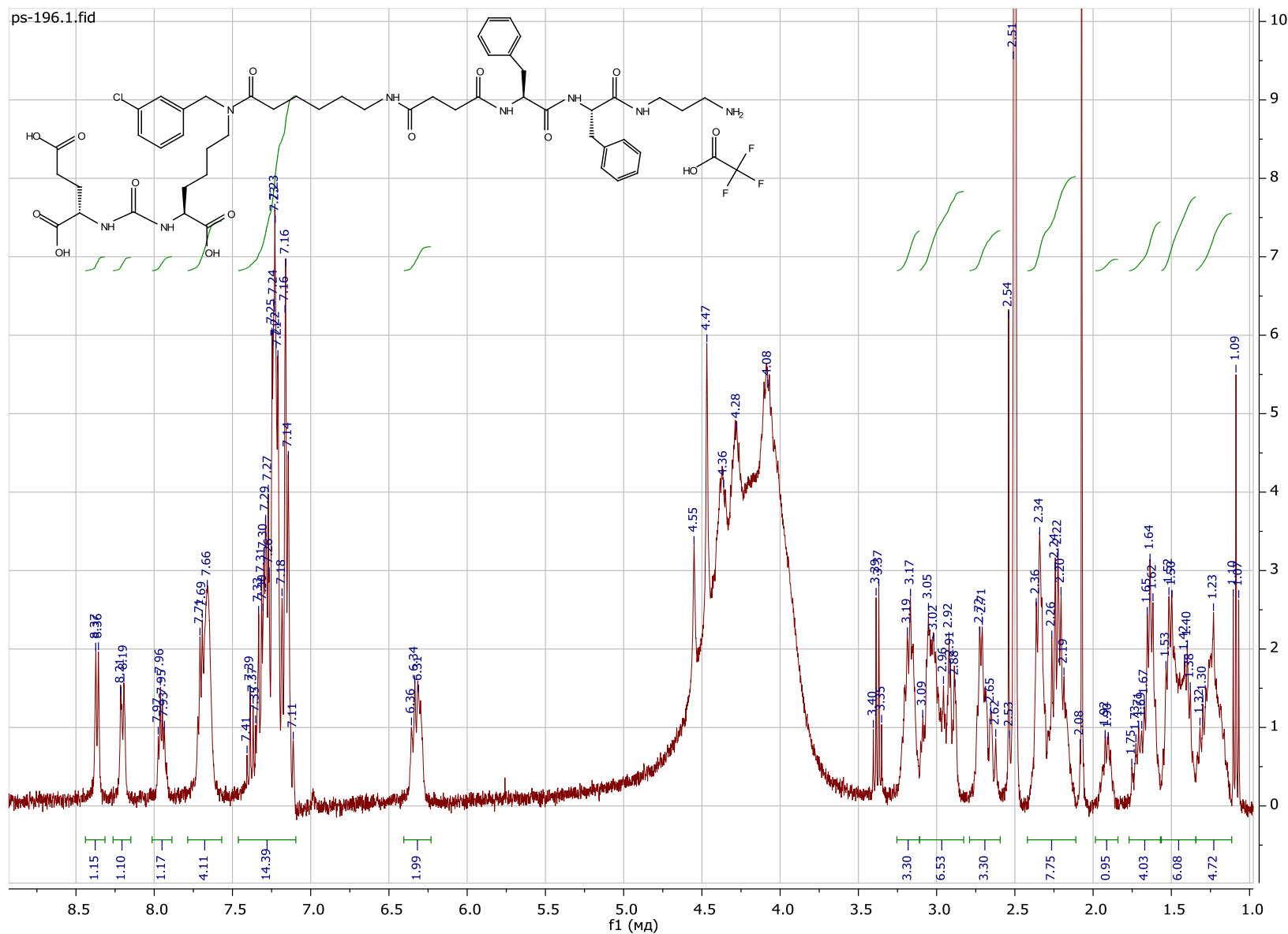


Figure S32. ¹H NMR spectrum of compound № 18 in DMSO-d₆.

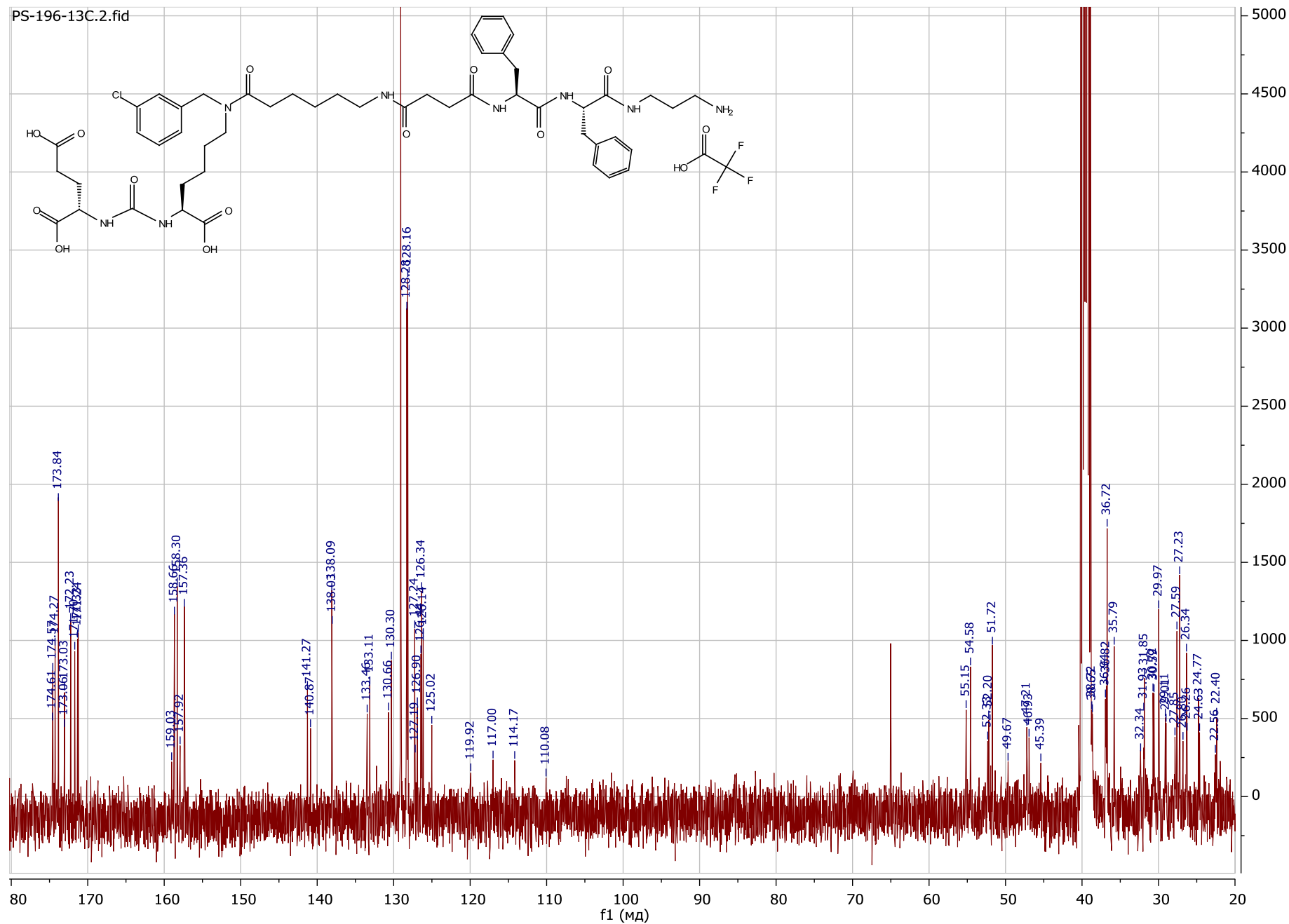


Figure S33. ¹³C NMR spectrum of compound № 18 in DMSO-*d*₆.

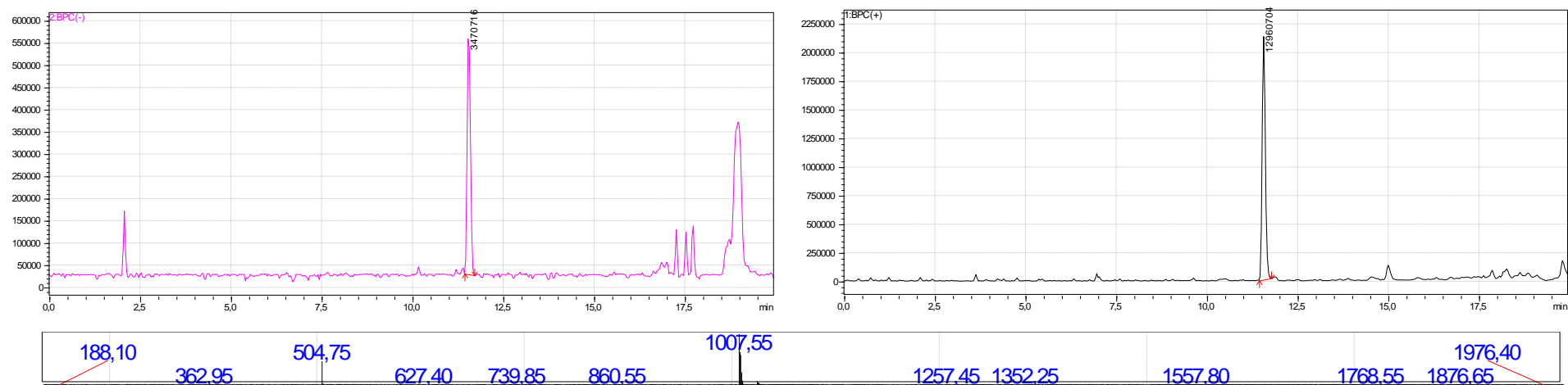


Figure S34. ESI-MS of compound № 18

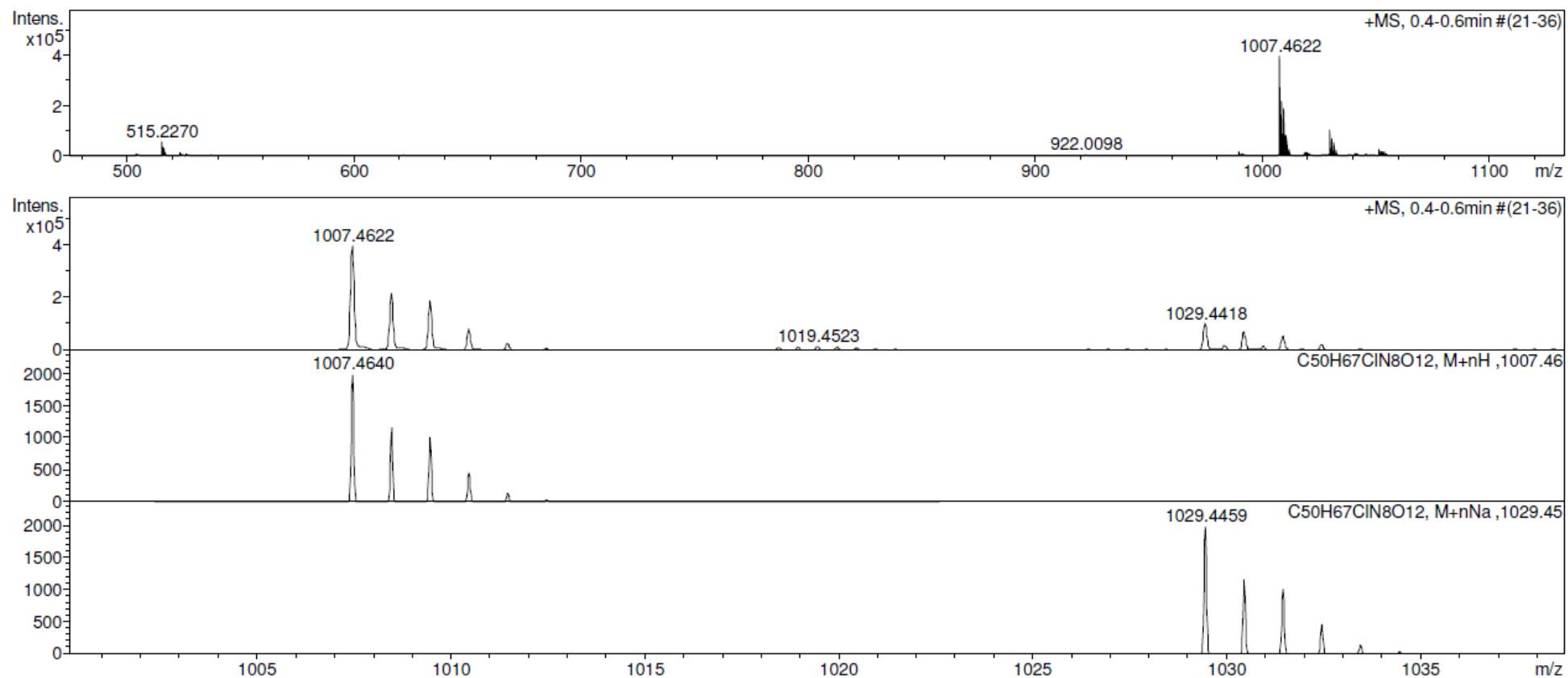


Figure S35. HRMS (m/z, ESI) of compound № 18

Compound 19

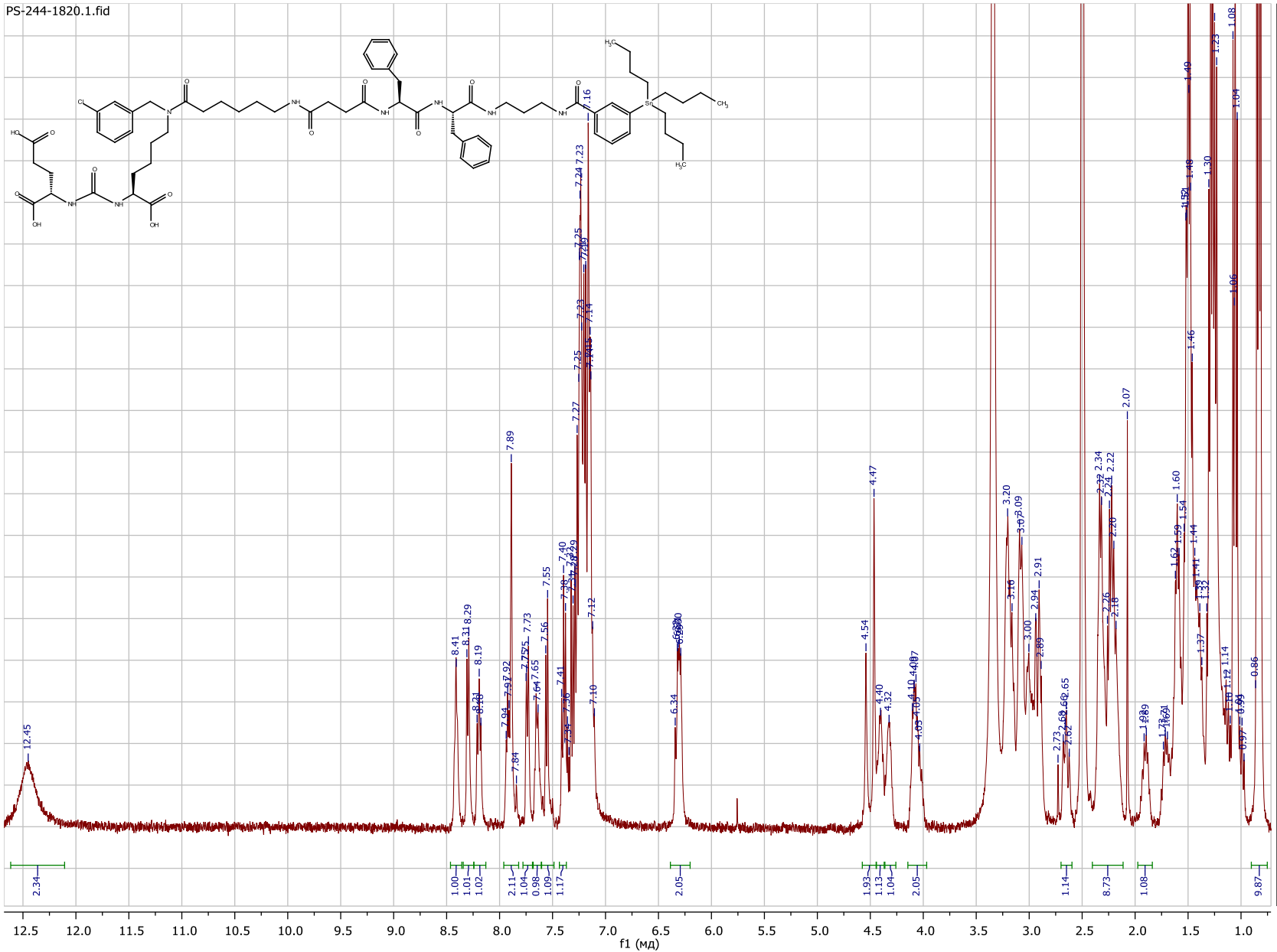


Figure S36. ^1H NMR spectrum of compound **19** in $\text{DMSO}-d_6$.

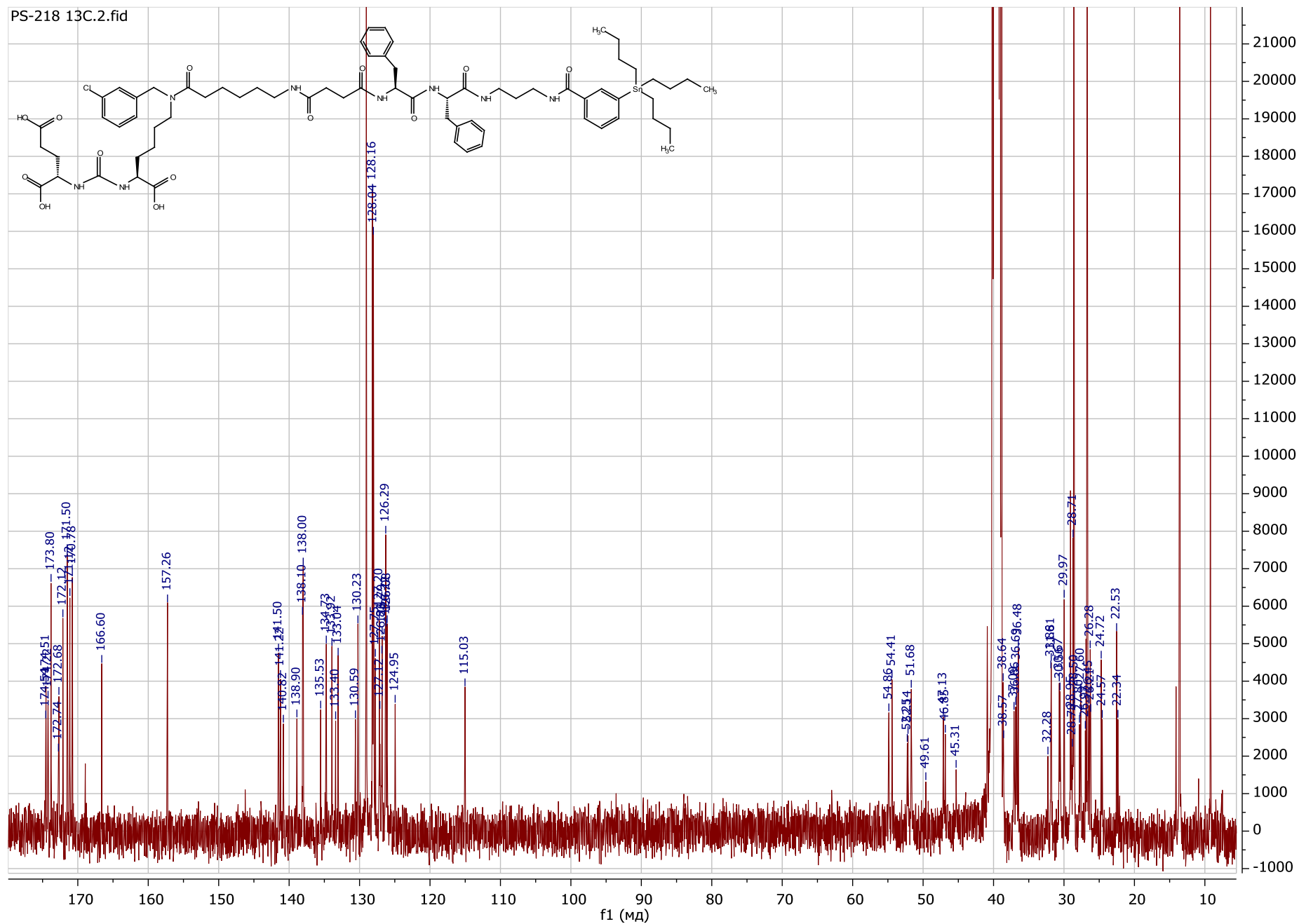


Figure S37. ^{13}C NMR spectrum of compound № 19 in $\text{DMSO}-d_6$.

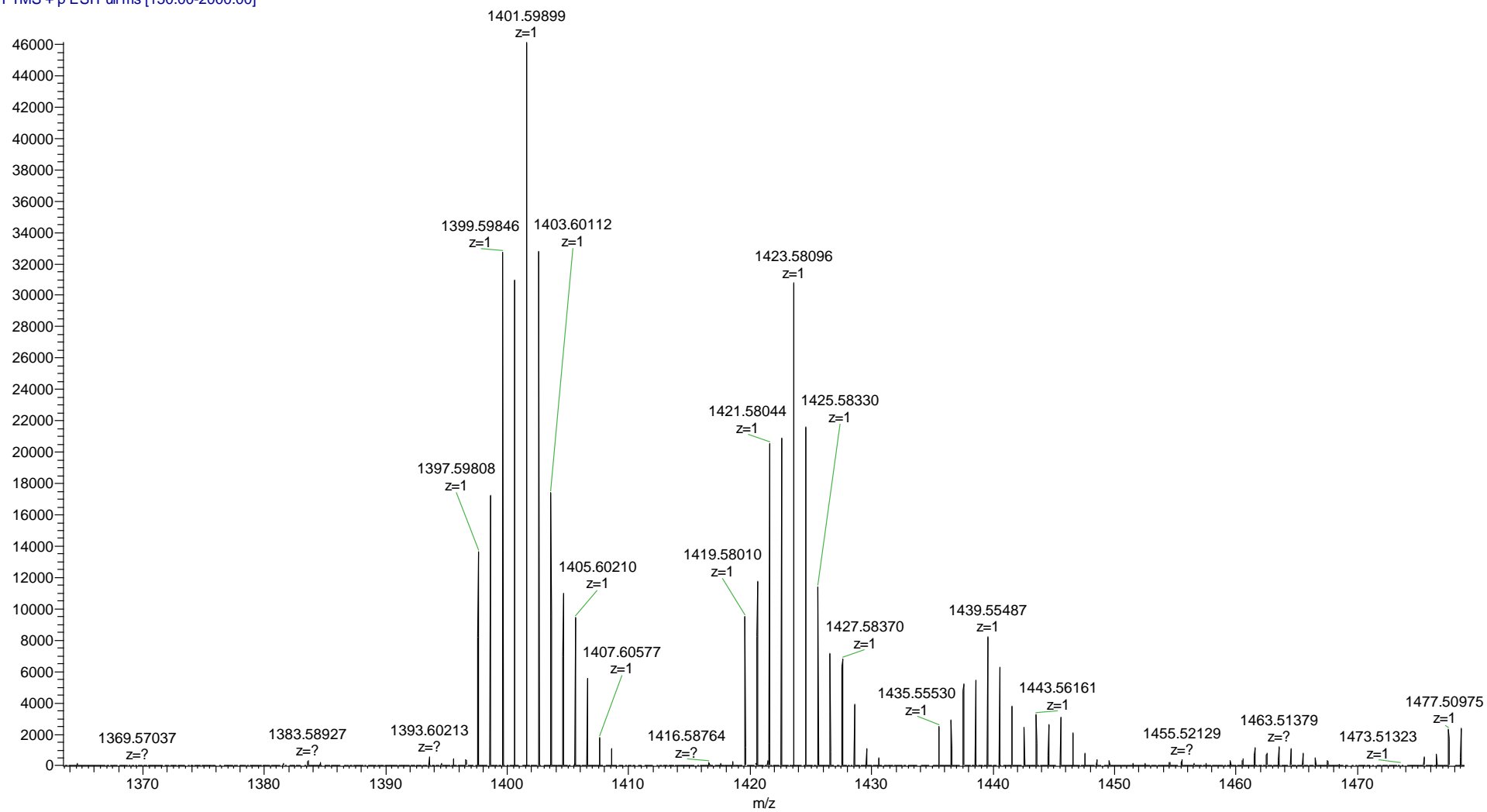


Figure S38. HRMS (m/z, ESI) of compound № 19

Compound 20

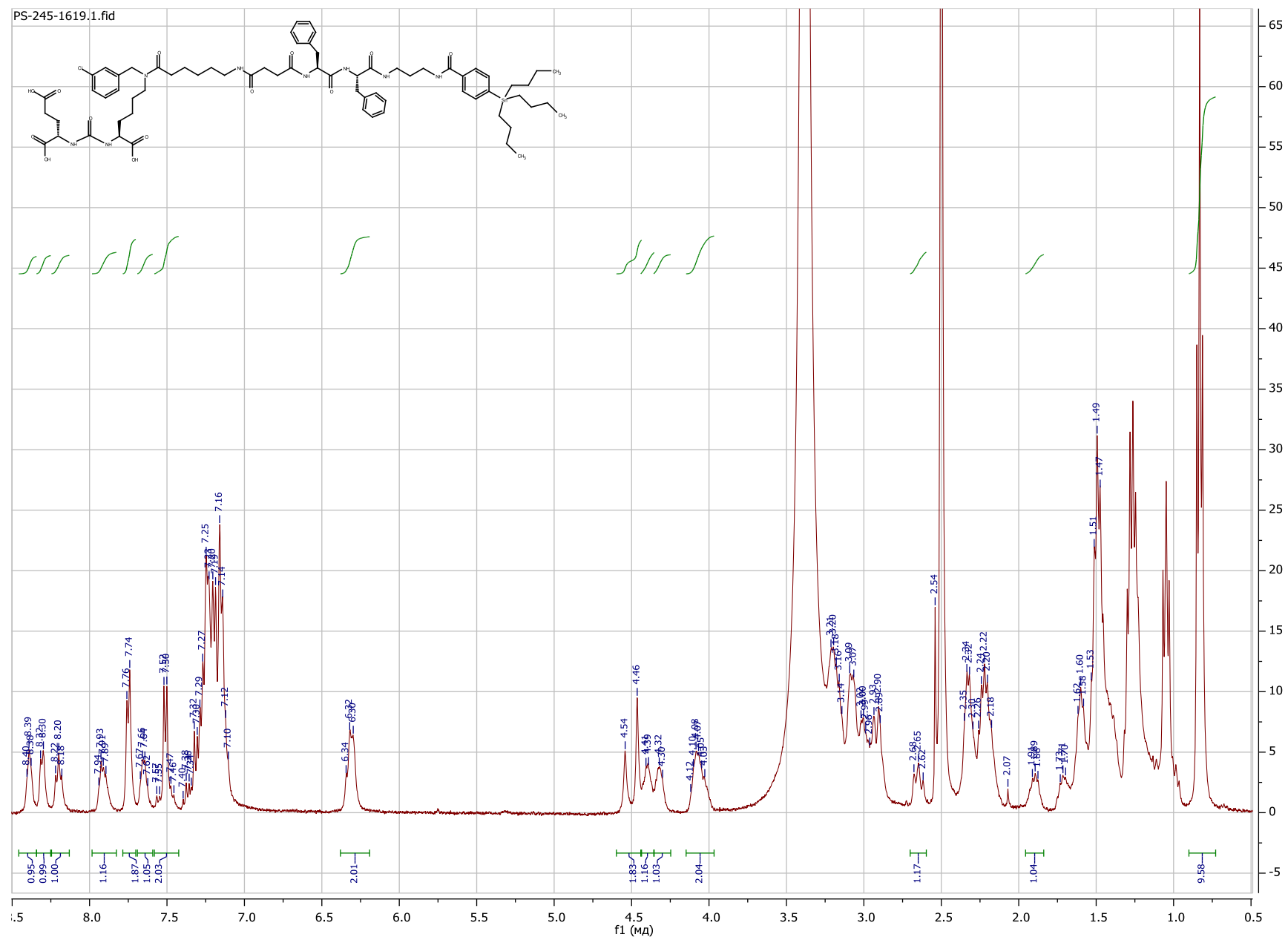


Figure S39. ¹H NMR spectrum of compound No 20 in DMSO-*d*₆.

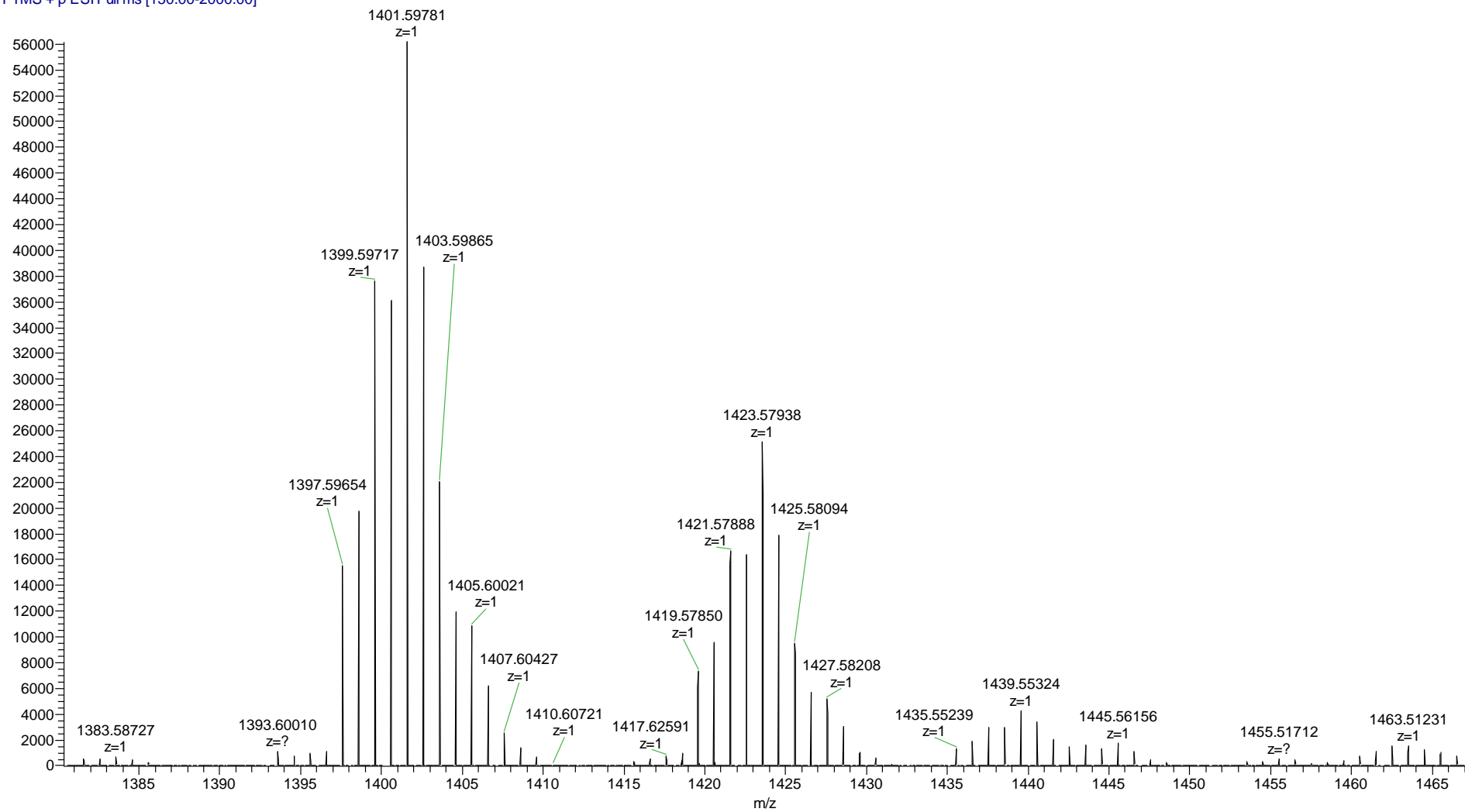


Figure S41. HRMS (m/z , ESI) of compound № 20

Compound 21

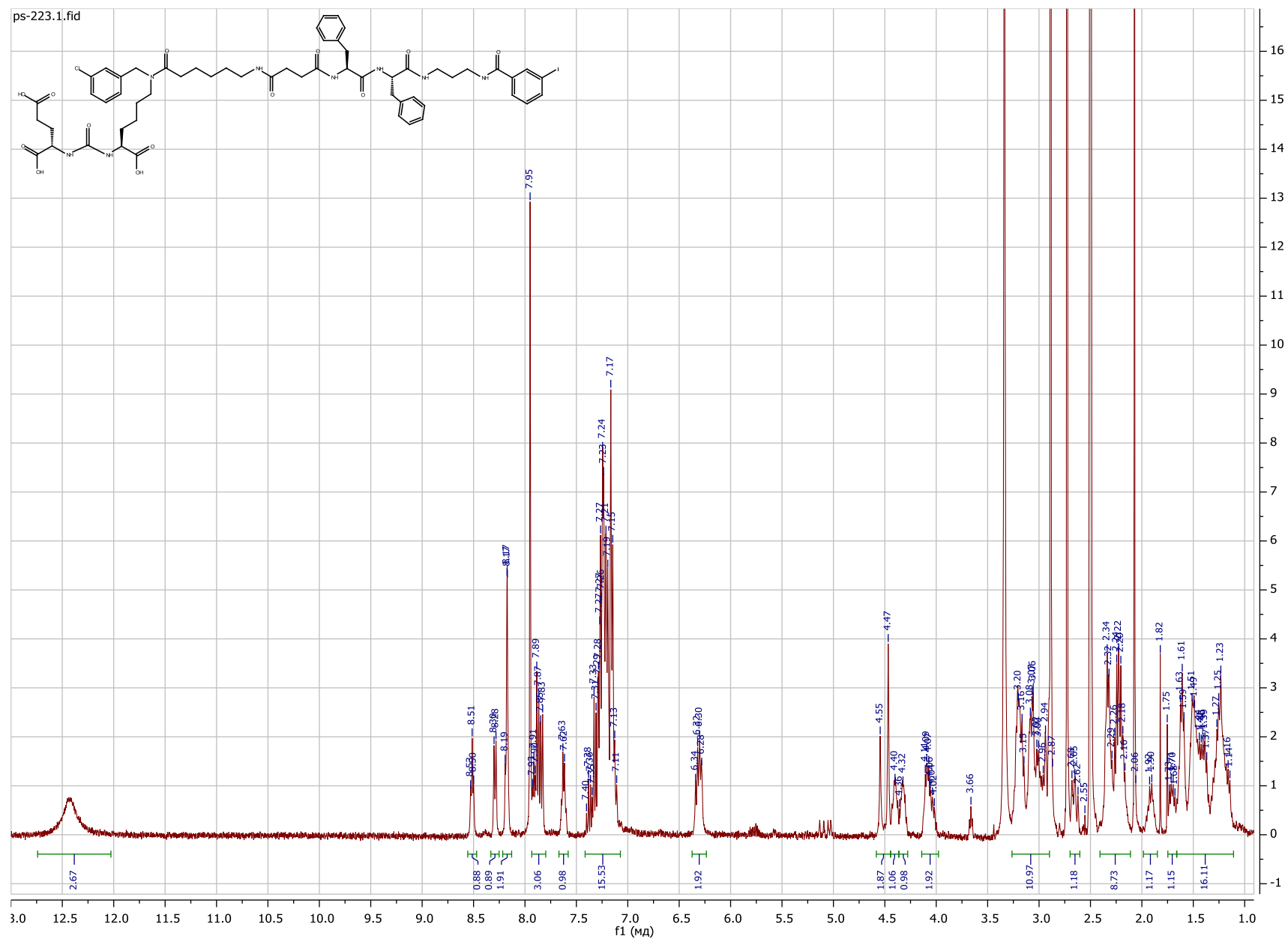


Figure S42. ¹H NMR spectrum of compound № 21 in DMSO-*d*₆.

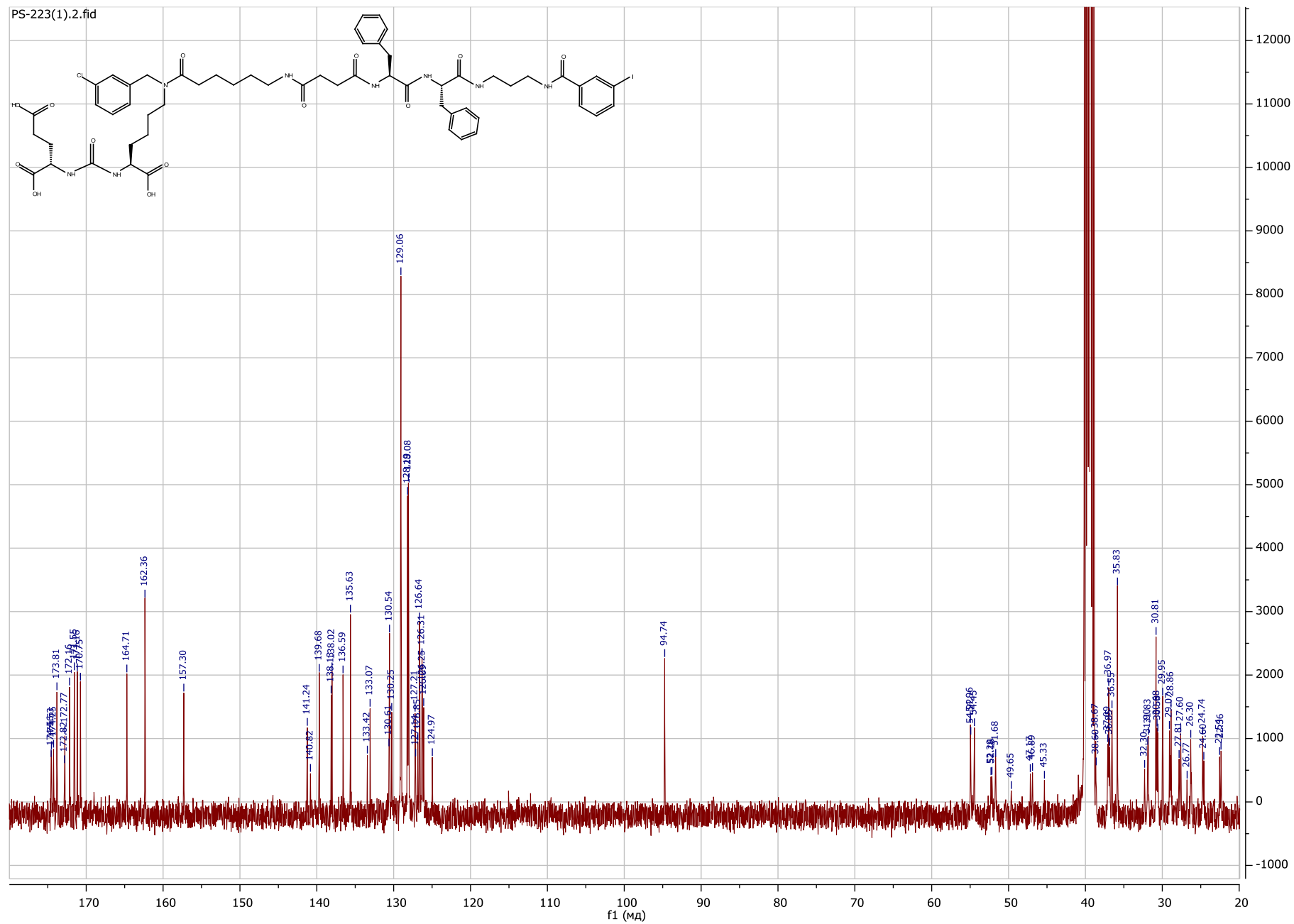


Figure S43. ¹³C NMR spectrum of compound № 21 in DMSO-*d*₆.

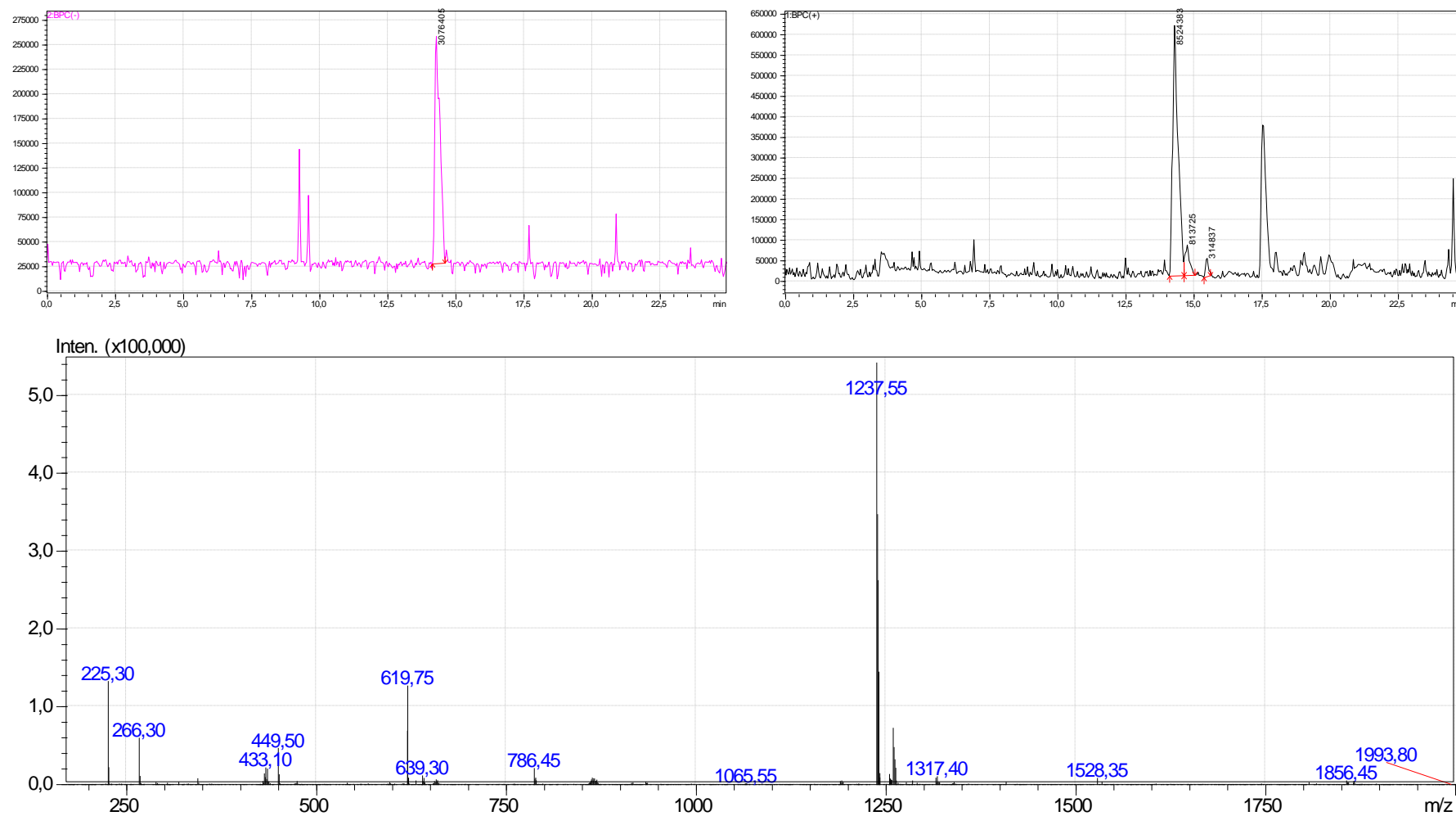


Figure S44. ESI-MS of compound № 21

Compound 22

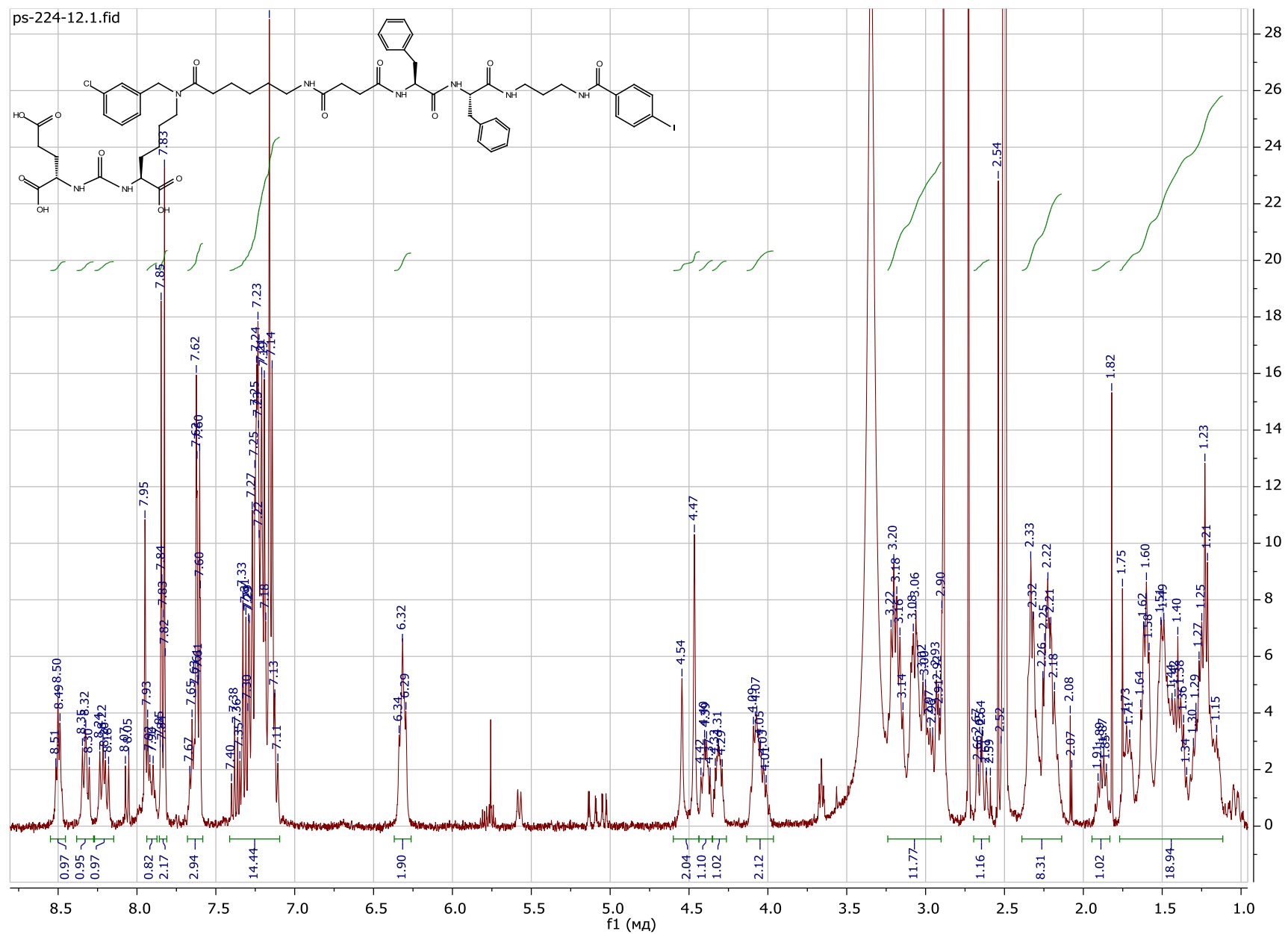


Figure S46. ^1H NMR spectrum of compound **22** in $\text{DMSO}-d_6$.

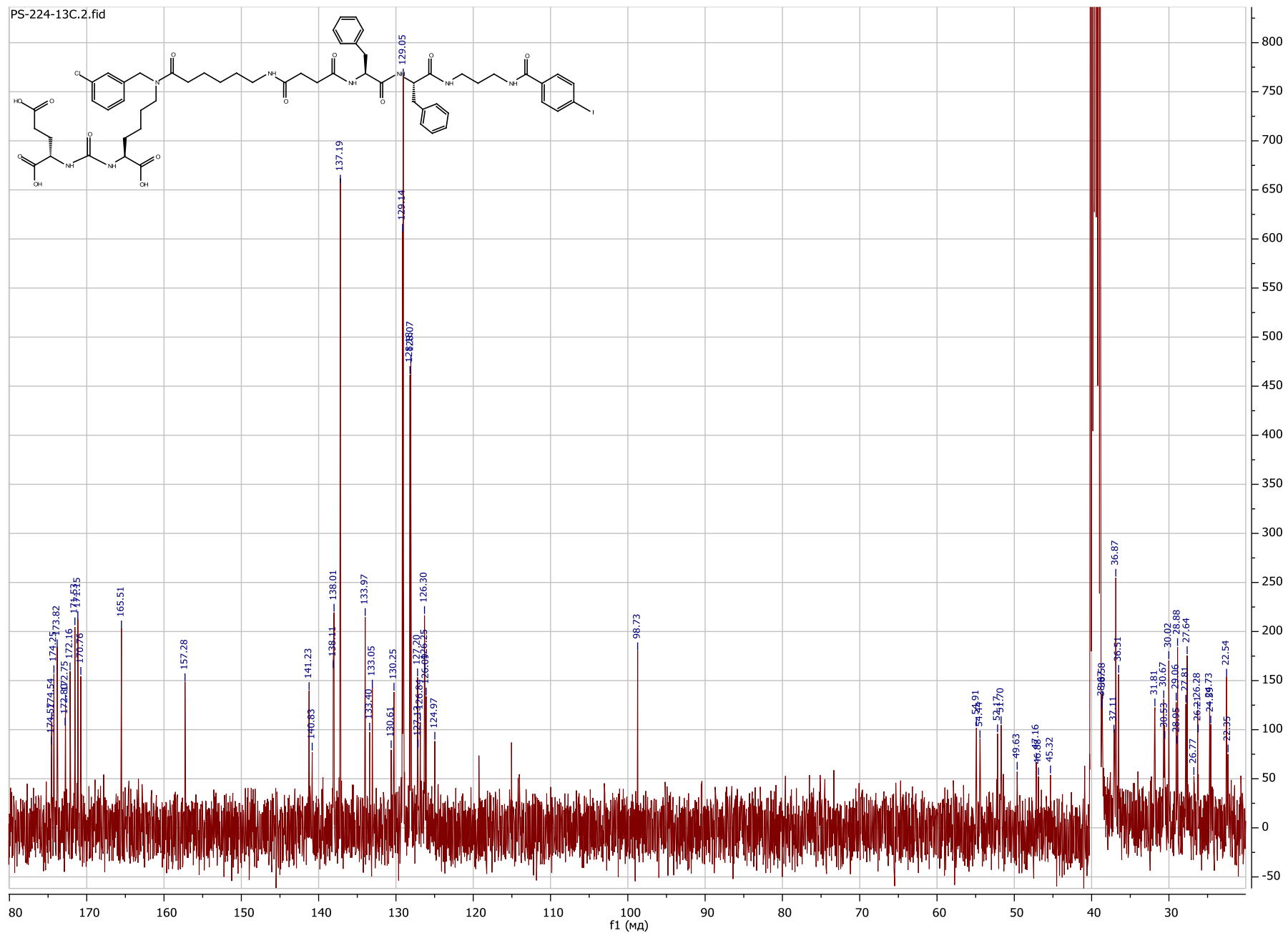


Figure S47. ¹³C NMR spectrum of compound № 22 in DMSO-*d*₆.

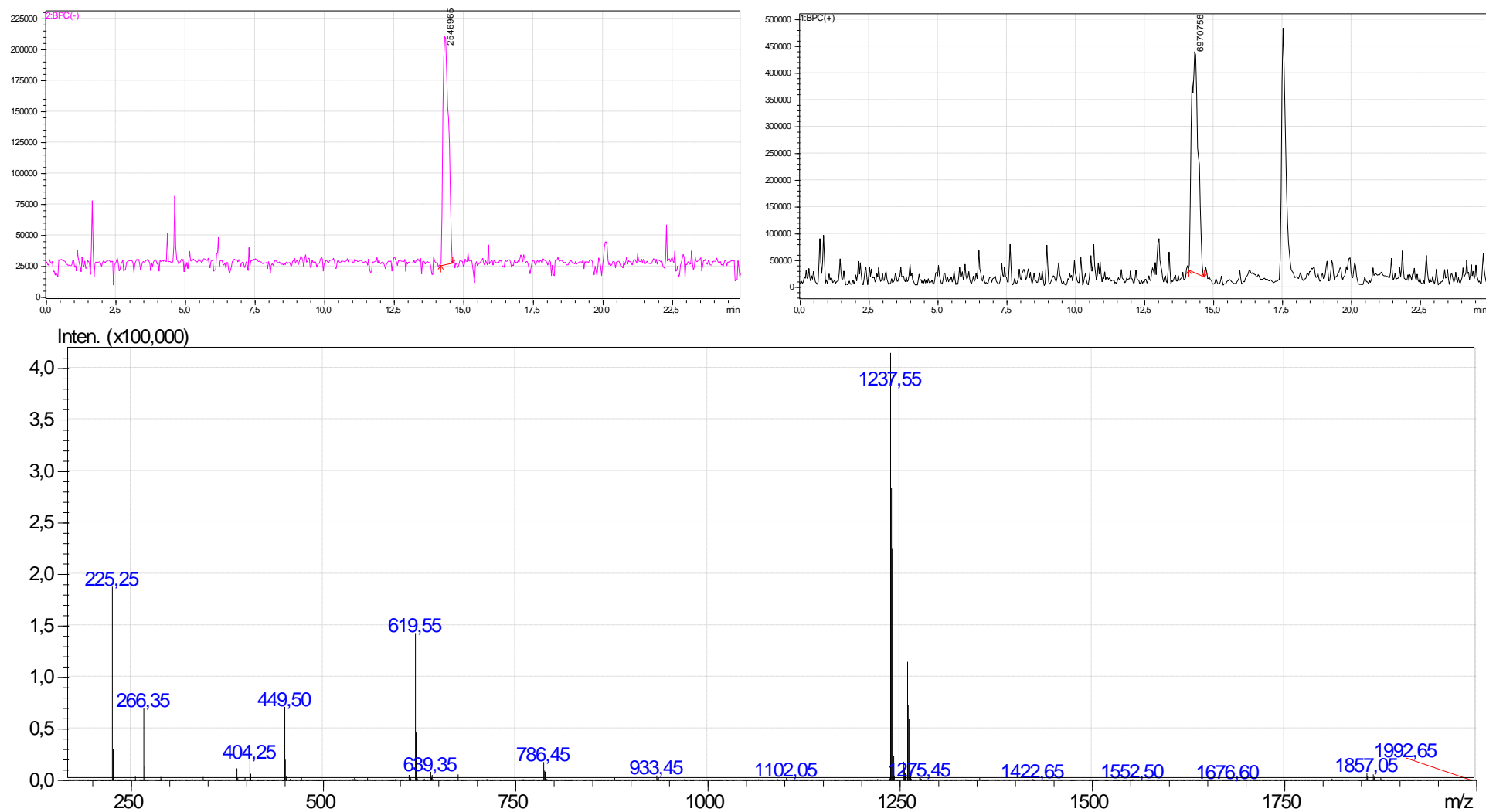
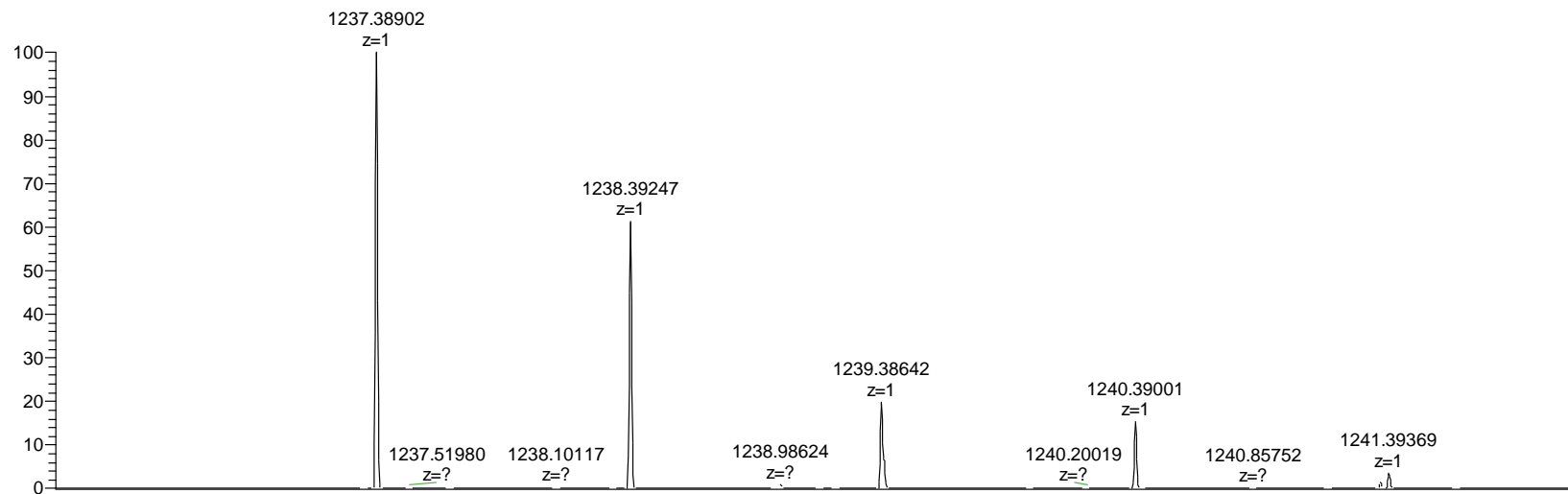


Figure S48. ESI-MS of compound № 22

NL:
1.24E4
PS_224_22042814194
9#34-58 RT: 1.01-1.38
AV: 25 T: FTMS + p ESI
Full ms
[150.00-2000.00]



NL:
3.83E5
C₅₇H₇₀ClIN₈O₁₃+H:
C₅₇H₇₁ClI₁N₈O₁₃
pa Chrg 1

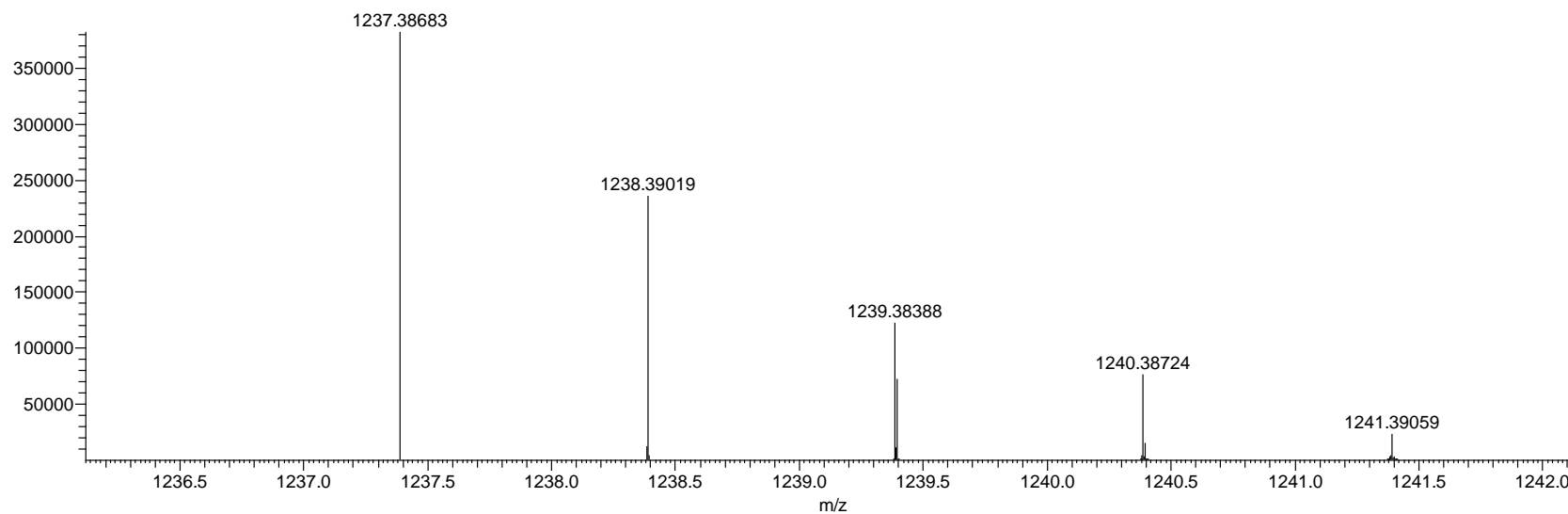


Figure S49. HRMS (m/z, ESI) of compound No 22