

Towards macrocyclic ionic liquids: novel ammonium salts based on tetrasubstituted *p*-tert-butylthiacalix[4]arenes

Pavel L. Padnya,^{a,b} Elena A. Andreyko,^a Polina A. Gorbatova,^c Viktor V. Parfenov,^c Ildar Kh. Rizvanov^d and Ivan I. Stoikov*^a

^a*Kazan Federal University, A.M. Butlerov Chemical Institute, 420008 Kremlevskaya, 18, Kazan, Russian Federation*

^b*RUDN University, Faculty of Science, Organic Chemistry Department, 117198 Miklukho-Maklaya St., 6, Moscow, Russian Federation*

^c*Kazan Federal University, Institute of Physics 420008, Kremlevskaya, 18, Kazan, Russian Federation*

^d*A. E. Arbuzov Institute of Organic and Physical Chemistry, Kazan Scientific Centre of the Russian Academy of Sciences, 420088 Kazan, Russian Federation*

Electronic Supplementary Information

Table S1. Characteristic chemical shifts of protons in ¹H NMR spectra of compounds 5-72.

thiacalixarene	<i>t</i> -Bu	O-CH ₂ CO	Ar-H	CONH
5 ^a	1.11	4.99	7.35	8.49
6 ^a	1.11	4.99	7.35	8.53
7 ^a	1.10	4.91	7.38	9.13
8 ^a	1.11	4.99	7.36	8.75
9 ^a	1.08	4.82	7.39	8.56
10 ^a	1.12	5.03	7.36	8.68
11 ^b	1.08	4.84	7.40	8.54
12 ^a	1.07	4.82	7.39	8.53
13 ^a	1.10	4.81	7.33	8.63
14 ^a	1.11	4.97	7.34	8.74
15 ^a	1.11	4.99	7.34	8.82
16 ^a	1.10	5.03	7.32	9.08
17 ^a	1.10	4.97	7.33	9.03
18 ^a	1.09	4.93	7.30	9.01
19 ^a	1.12	4.98	7.35	8.94
20 ^b	1.08	4.86	7.40	8.94
21 ^b	1.08	4.87	7.40	8.81
22 ^b	1.21	3.98	7.60	8.01
23 ^b	1.21	3.99	7.60	8.03
24 ^b	1.20	4.01	7.60	8.10
25 ^b	1.20	4.00	7.60	8.05
26 ^b	1.20	3.98	7.60	8.06
27 ^a	1.20	3.99	7.60	8.04
28 ^b	1.19	3.96	7.59	8.04
29 ^b	1.20	3.99	7.59	8.06
30 ^b	1.20	3.99	7.60	8.05
31 ^b	1.22	4.05	7.61	8.18
32 ^b	1.22	4.03	7.61	8.13
33 ^b	1.19	4.20	7.60	8.38
34 ^b	1.22	4.04	7.60	8.27
35 ^b	1.21	4.08	7.60	8.29
36 ^b	1.21	4.08	7.60	8.27
37 ^b	1.21	4.05	7.60	8.23
38 ^b	1.22	4.09	7.60	8.27
39 ^b	1.08	4.82	7.40	8.49
40 ^b	1.08	4.82	7.40	8.49
41 ^a	1.10	4.91	7.35	8.29
42 ^a	1.11	4.89	7.35	8.23
43 ^b	1.08	4.79	7.39	8.47
44 ^a	1.11	4.89	7.35	8.23
45 ^b	1.08	4.80	7.40	8.50
46 ^b	1.08	4.80	7.39	8.50
47 ^b	1.07	4.80	7.38	8.50
48 ^a	1.11	4.91	7.36	8.38
49 ^a	1.11	4.91	7.36	8.40
50 ^a	1.10	4.95	7.35	8.51
51 ^a	1.11	4.90	7.36	8.36
52 ^b	1.08	4.85	7.40	8.78

53 ^b	1.07	4.83	7.39	8.76
54 ^b	1.07	4.83	7.45	8.87
55 ^b	1.08	4.84	7.40	8.81
56 ^b	1.21	4.00	7.60	8.03
57 ^b	1.21	4.00	7.60	8.03
58 ^b	1.20	4.03	7.60	8.07
59 ^b	1.20	4.00	7.59	8.02
60 ^b	1.20	3.99	7.61	8.06
61 ^b	1.20	3.99	7.59	8.03
62 ^b	1.20	3.97	7.59	8.02
63 ^b	1.20	4.01	7.59	8.03
64 ^b	1.21	3.99	7.60	8.04
65 ^b	1.22	4.05	7.60	8.17
66 ^b	1.21	4.04	7.60	8.14
67 ^b	1.19	4.20	7.61	8.34
68 ^b	1.21	4.08	7.61	8.22
69 ^b	1.21	4.07	7.69	8.22
70 ^b	1.21	4.05	7.60	8.22
71 ^b	1.21	4.04	7.59	8.21
72 ^b	1.21	4.07	7.60	8.23

^a CDCl₃, ^b DMSO-d₆

Fig. S1. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxy carbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 9), CDCl_3 , 298 K, 400 MHz

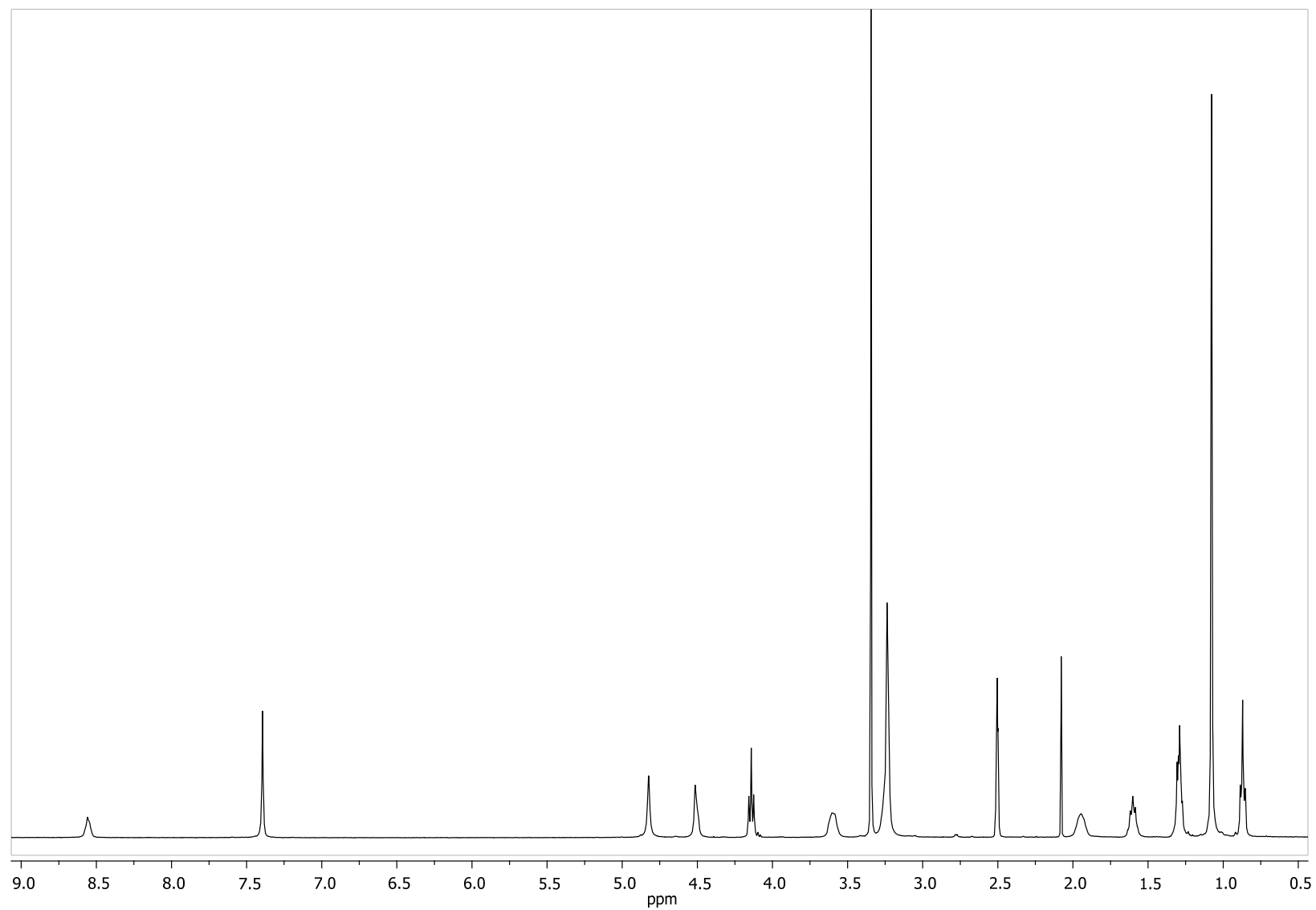


Fig. S2. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (*cone* 15), CDCl_3 , 298 K, 400 MHz

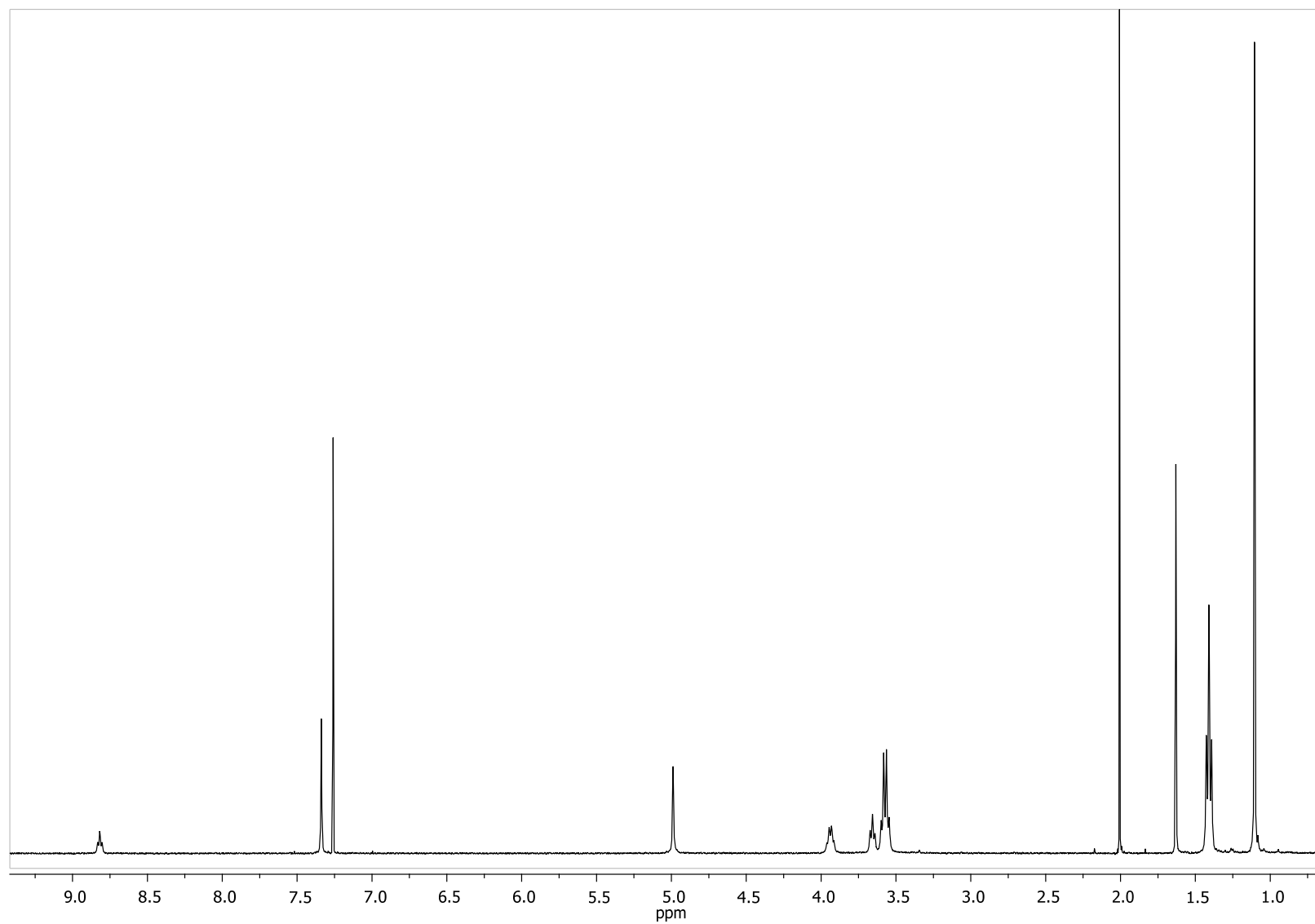


Fig. S3. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 16), CDCl_3 , 298 K, 400 MHz

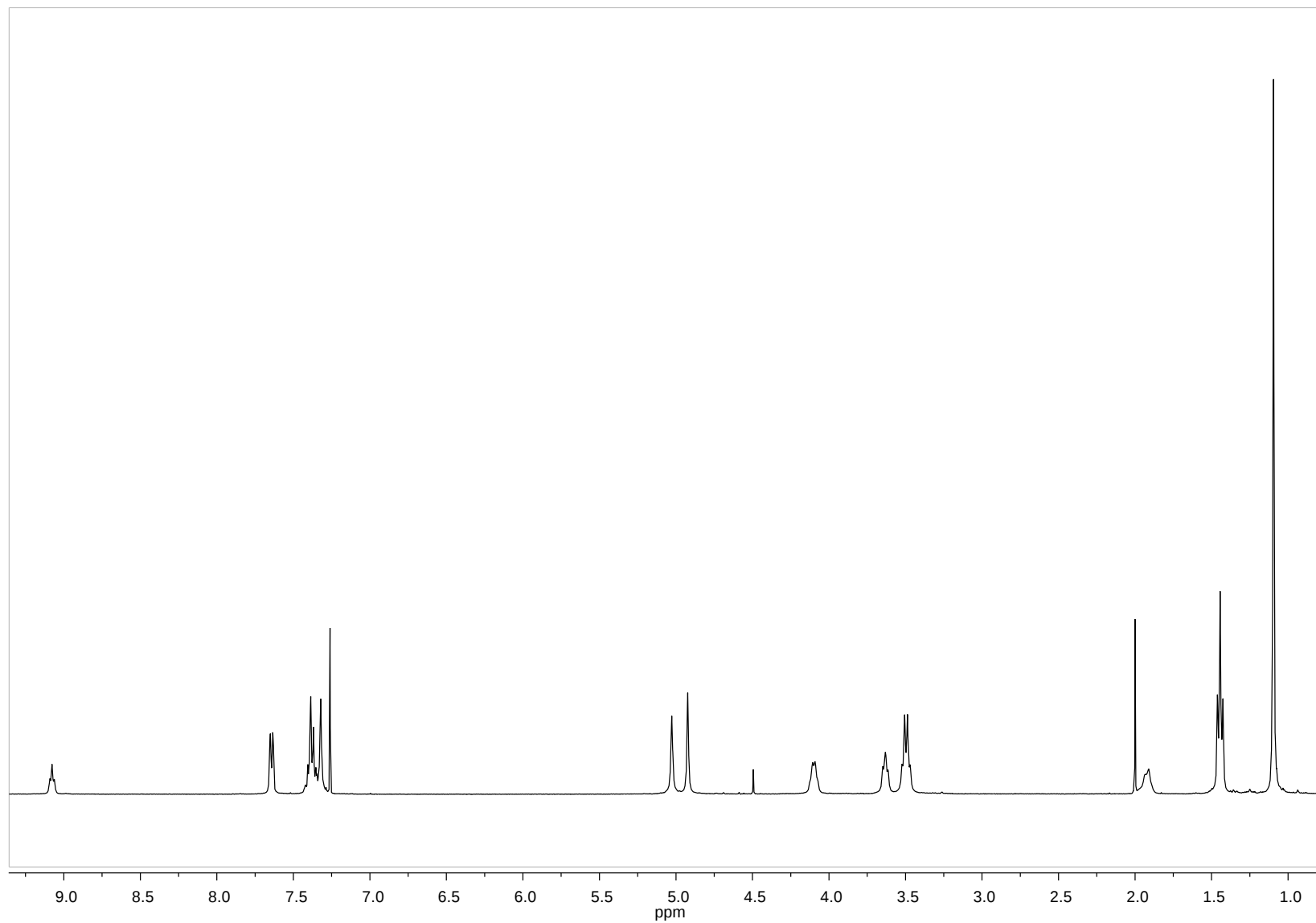


Fig. S4. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone 17*), CDCl_3 , 298 K, 400 MHz

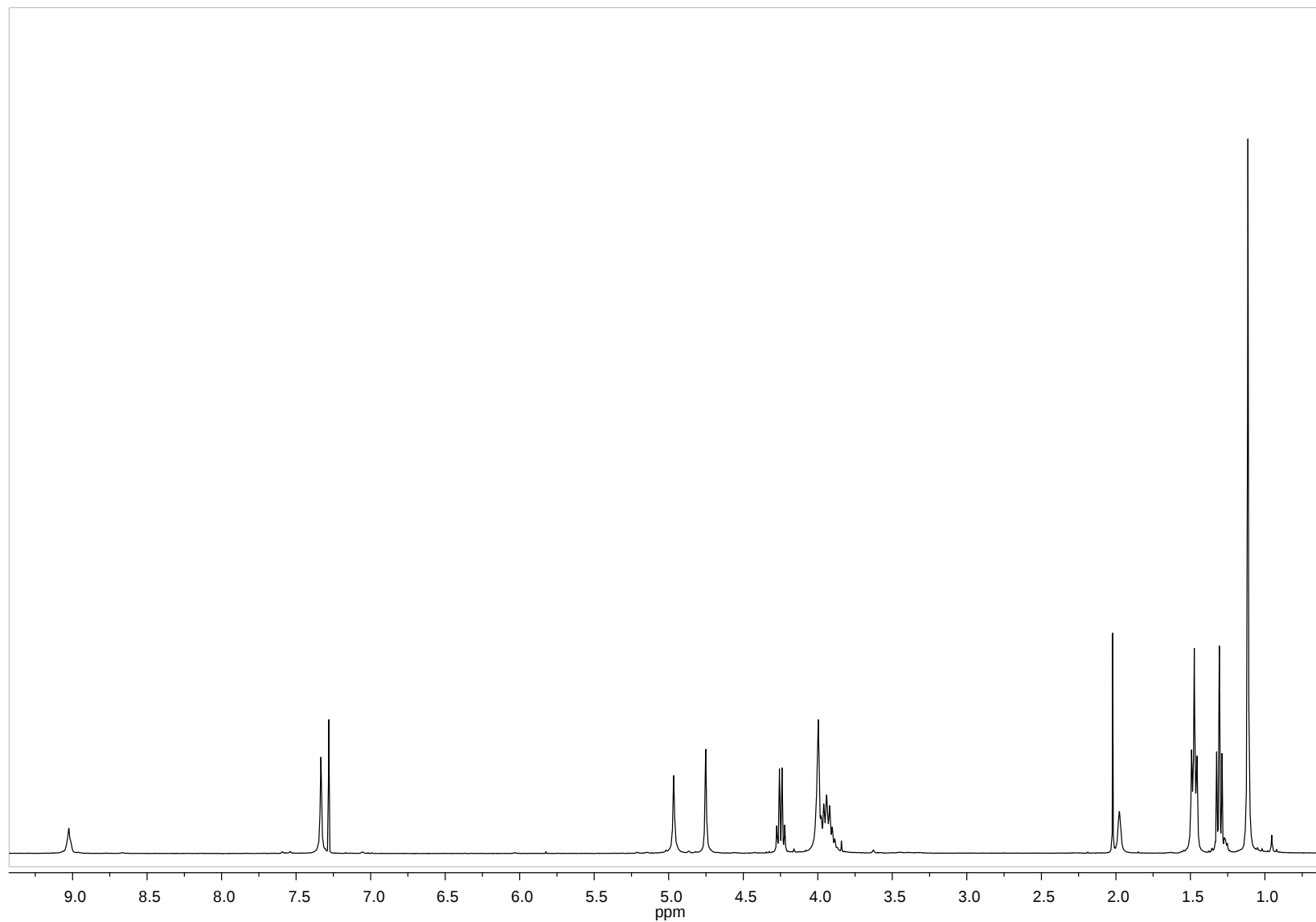


Fig. S5. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)-ammoniummethyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone 18*), CDCl_3 , 298 K, 400 MHz

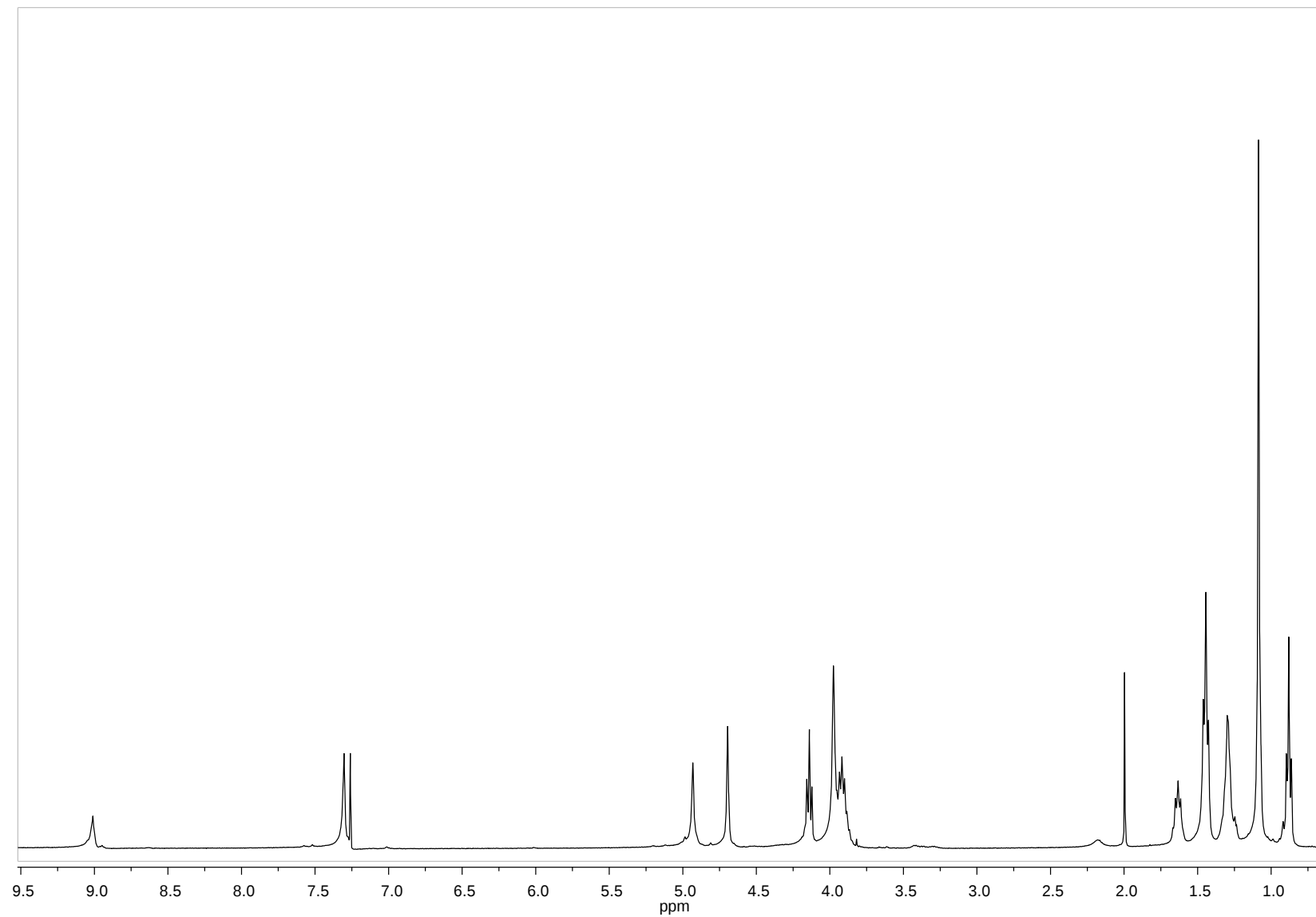


Fig. S6. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-*alternate* 22), DMSO- d_6 , 298 K, 400 MHz

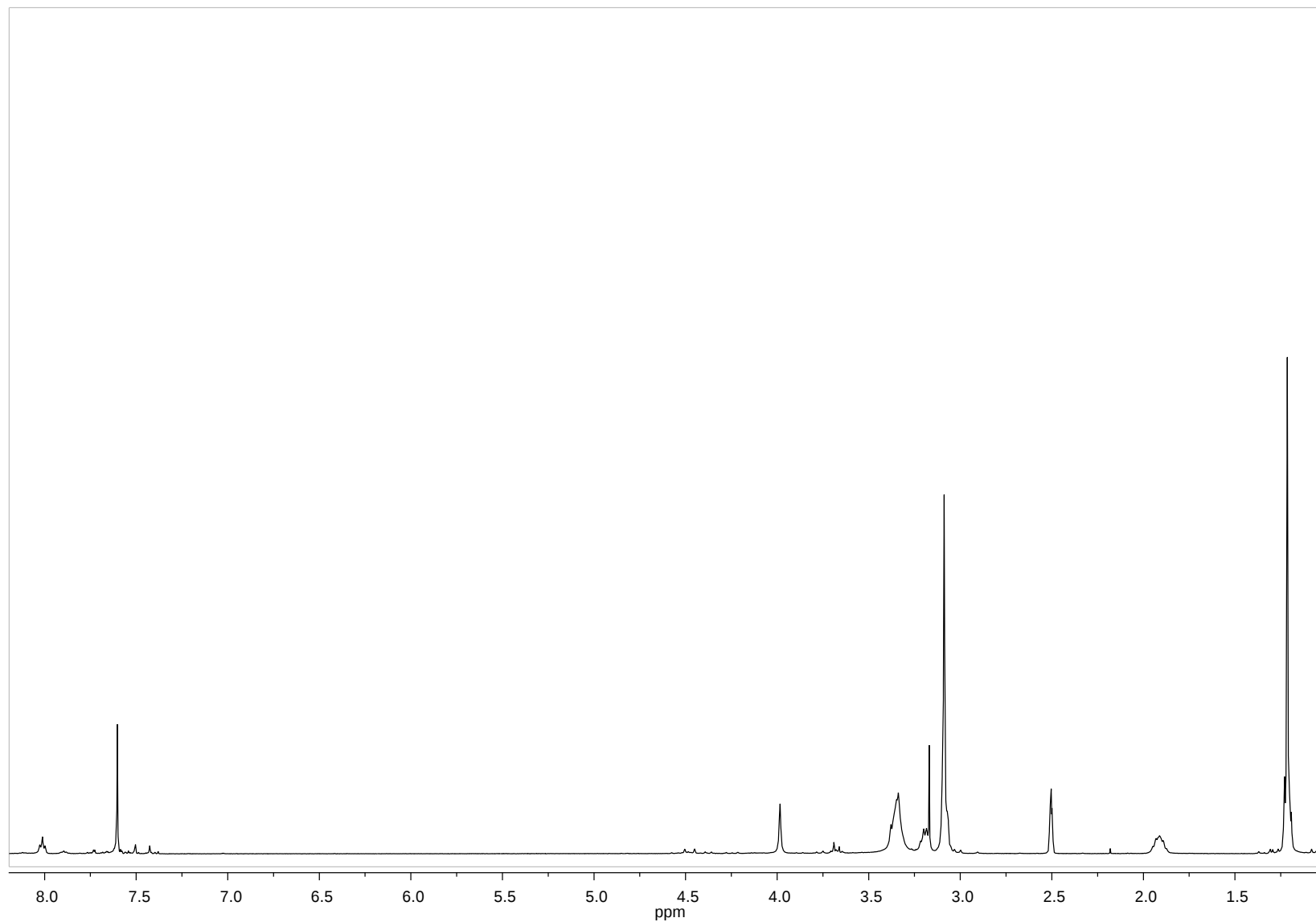


Fig. S7. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-alternate 23), DMSO- d_6 , 298 K, 400 MHz

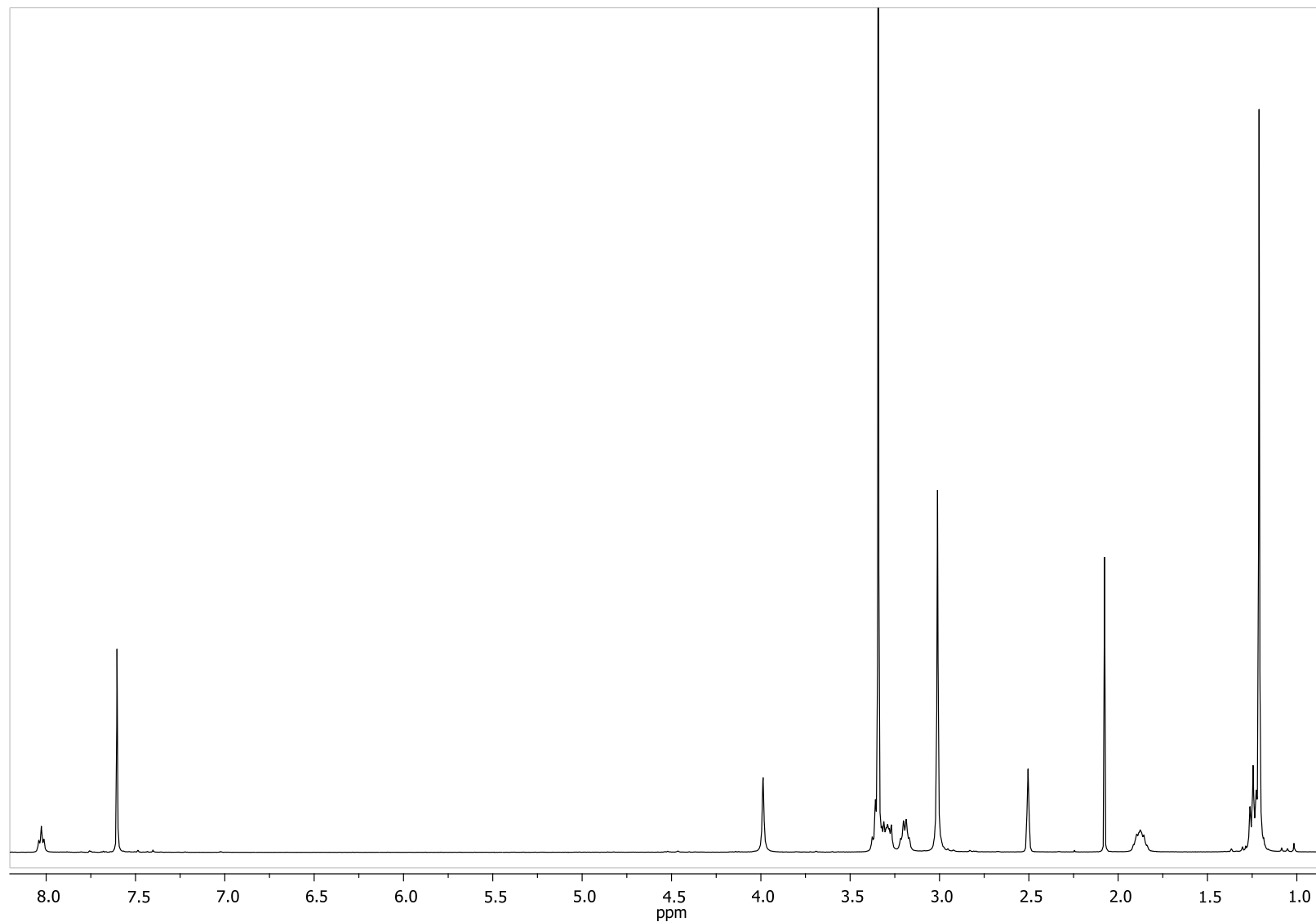


Fig. S8. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 24), DMSO- d_6 , 298 K, 400 MHz

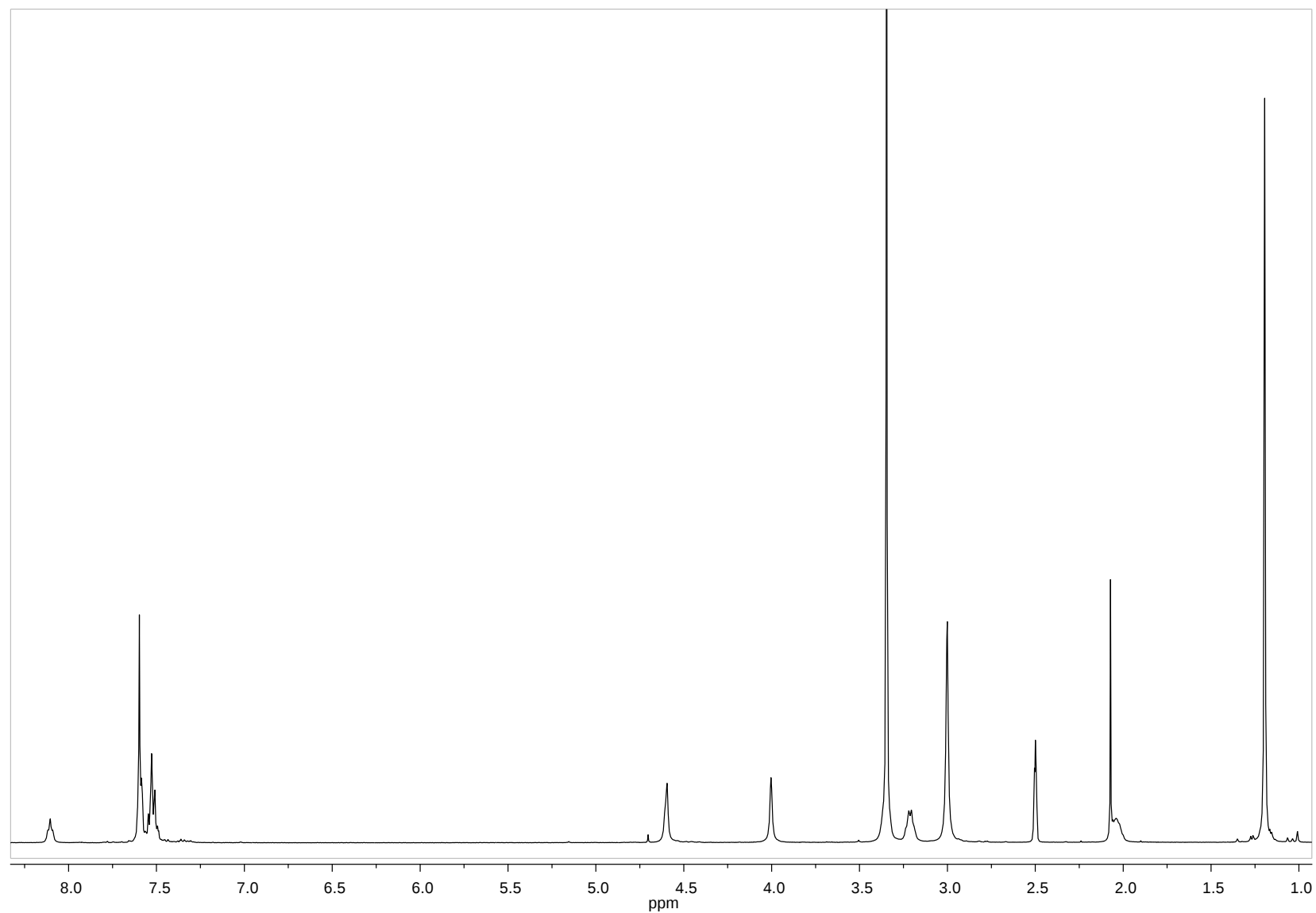


Fig. S9. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-alternate 25), DMSO- d_6 , 298 K, 400 MHz

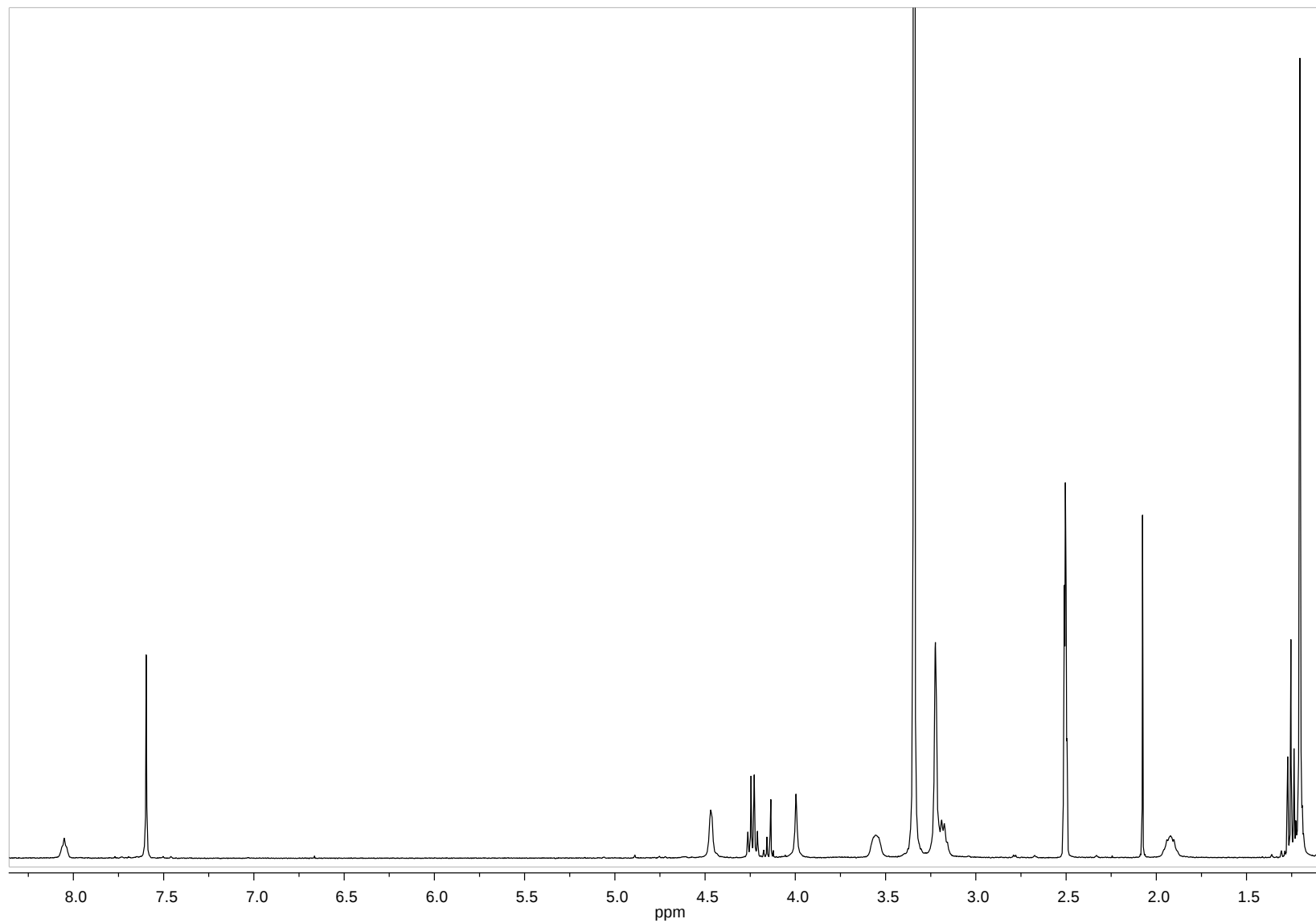


Fig. S10. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxy carbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 26), DMSO- d_6 , 298 K, 400 MHz

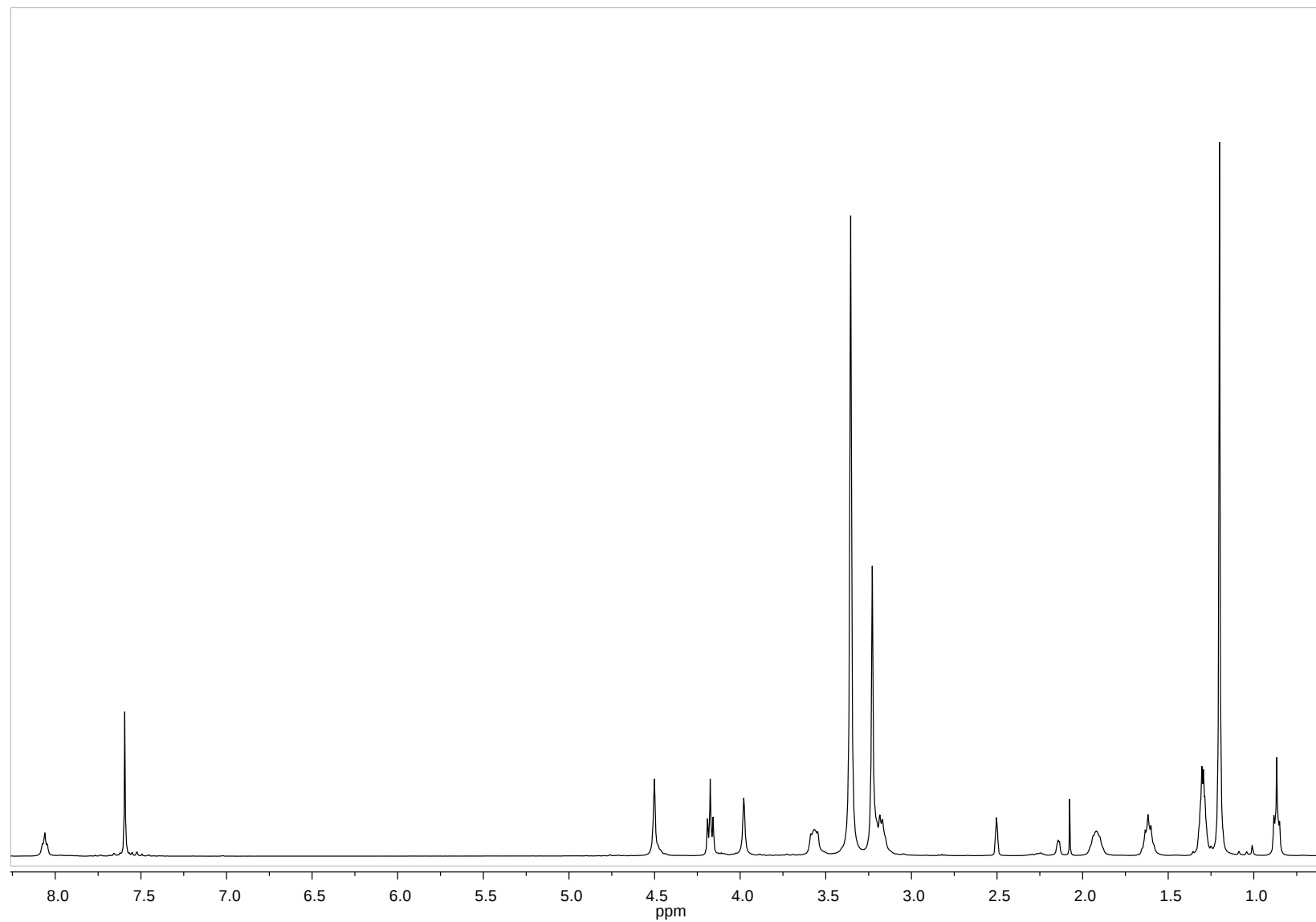


Fig. S11. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiocalix[4]arene tetrabromide (1,3-*alternate* 30), DMSO- d_6 , 298 K, 400 MHz

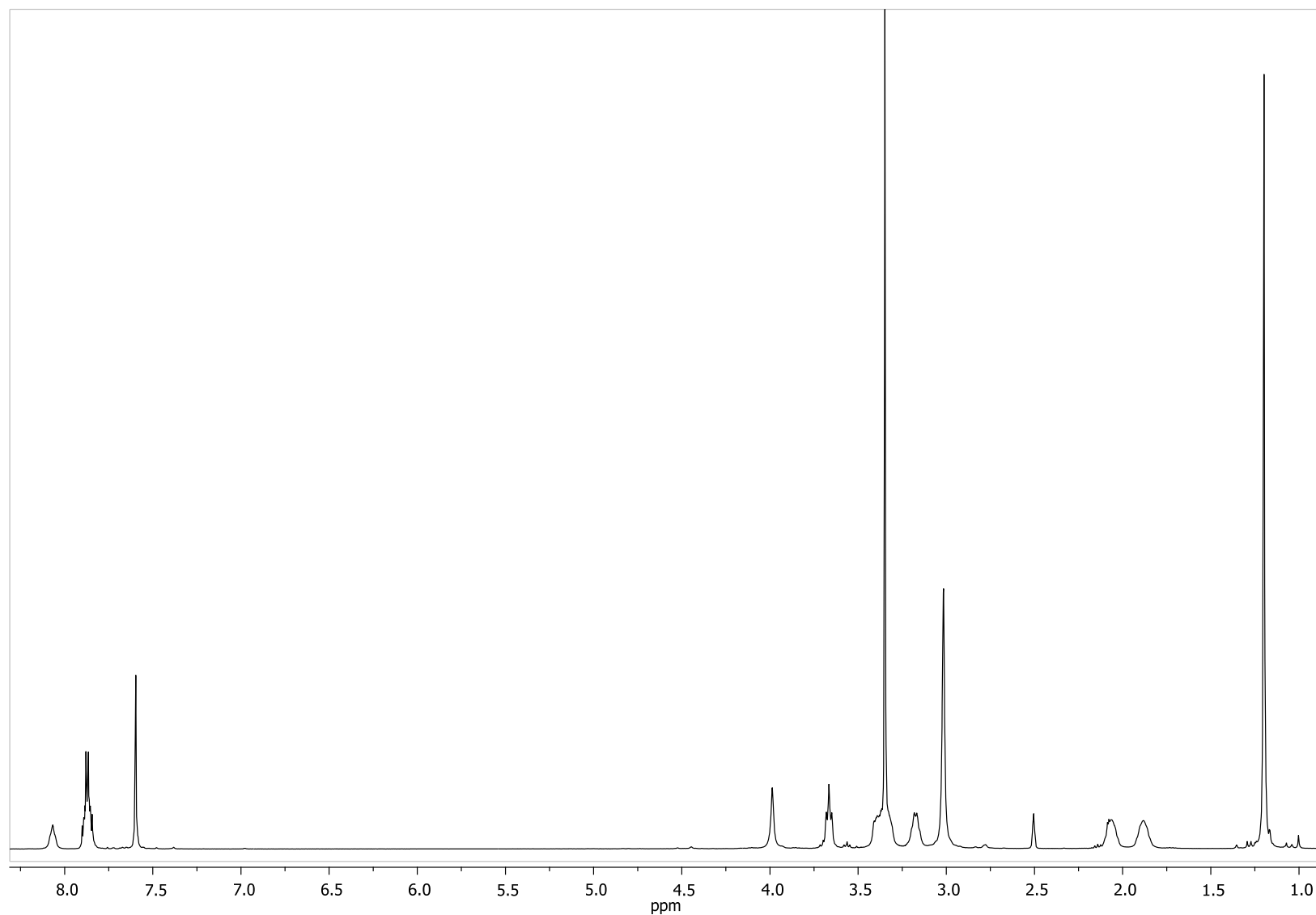


Fig. S12. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-alternate **31**), DMSO- d_6 , 298 K, 400 MHz

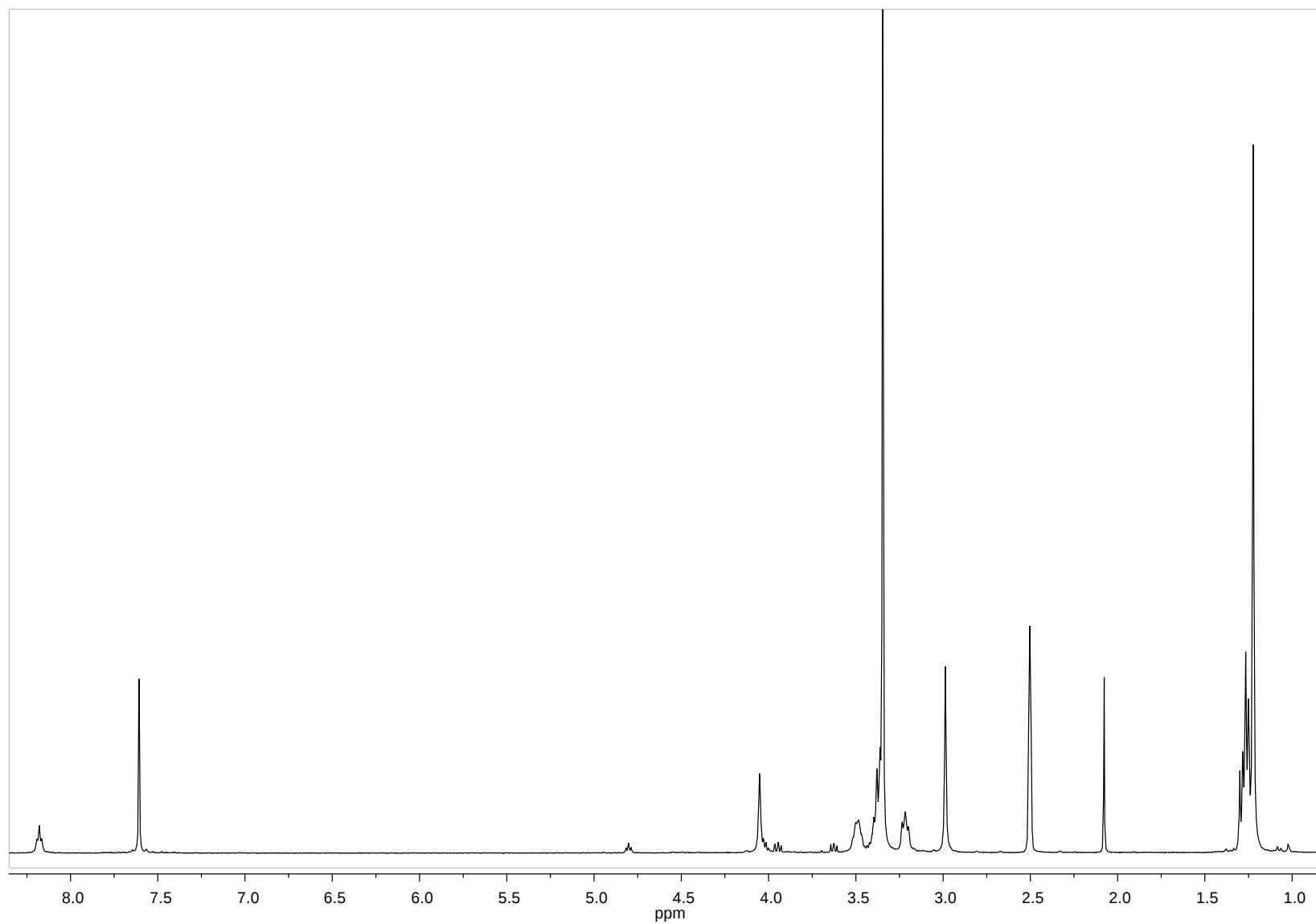


Fig. S13. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-*alternate* 32), DMSO- d_6 , 298 K, 400 MHz

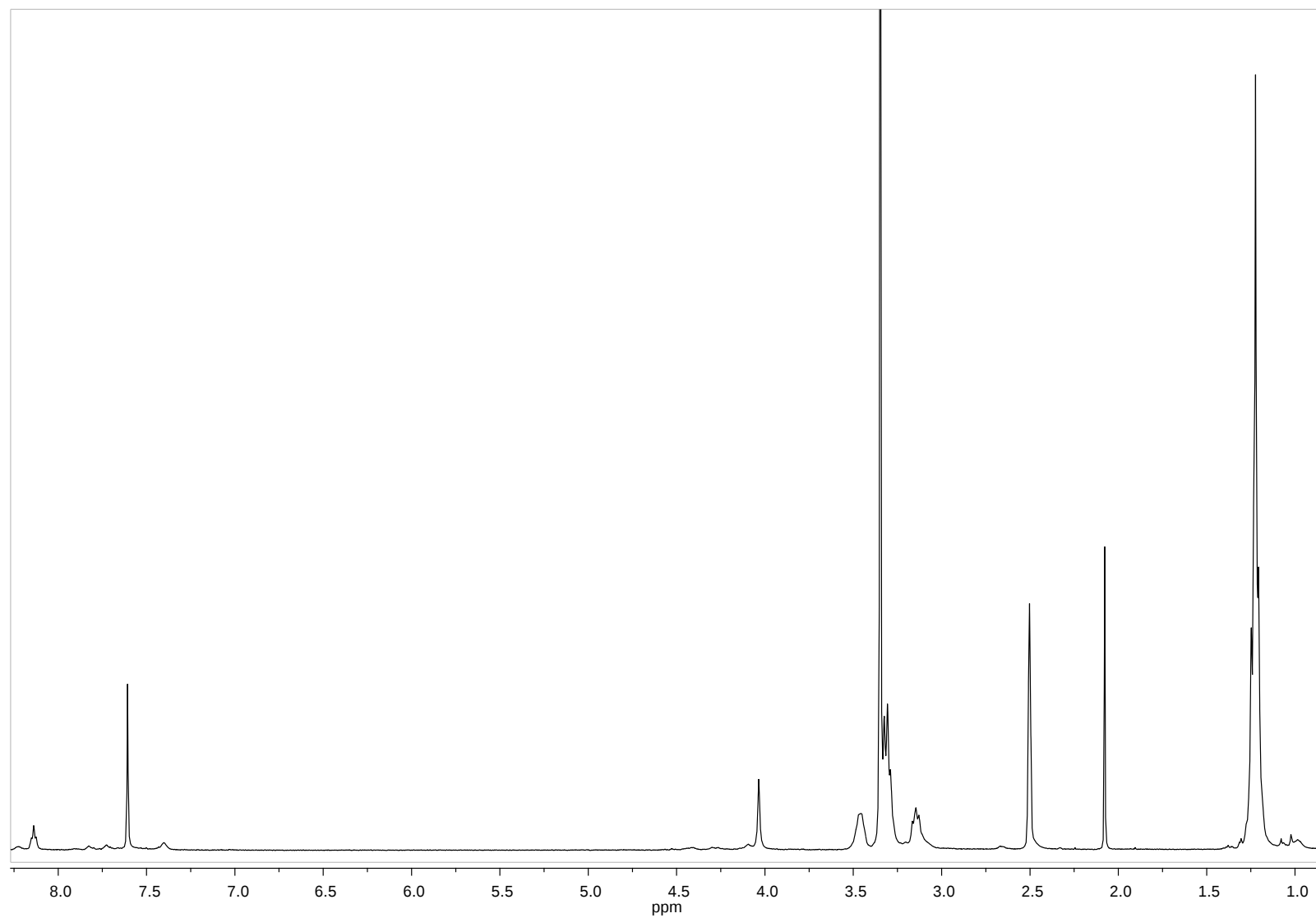


Fig. S14. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-alternate 33), DMSO- d_6 , 298 K, 400 MHz

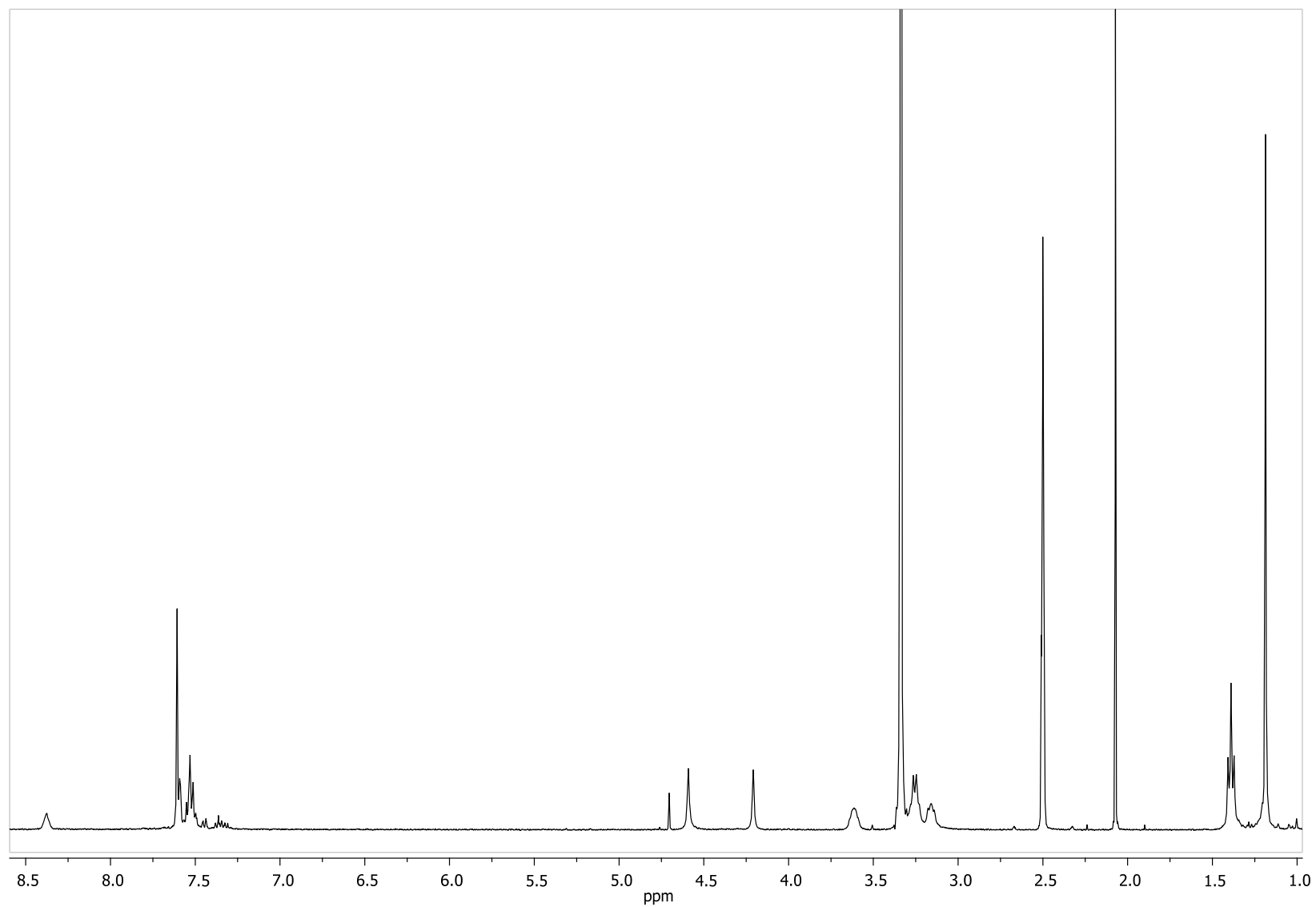


Fig. S15. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*1,3-alternate* 34), DMSO-d_6 , 298 K, 400 MHz

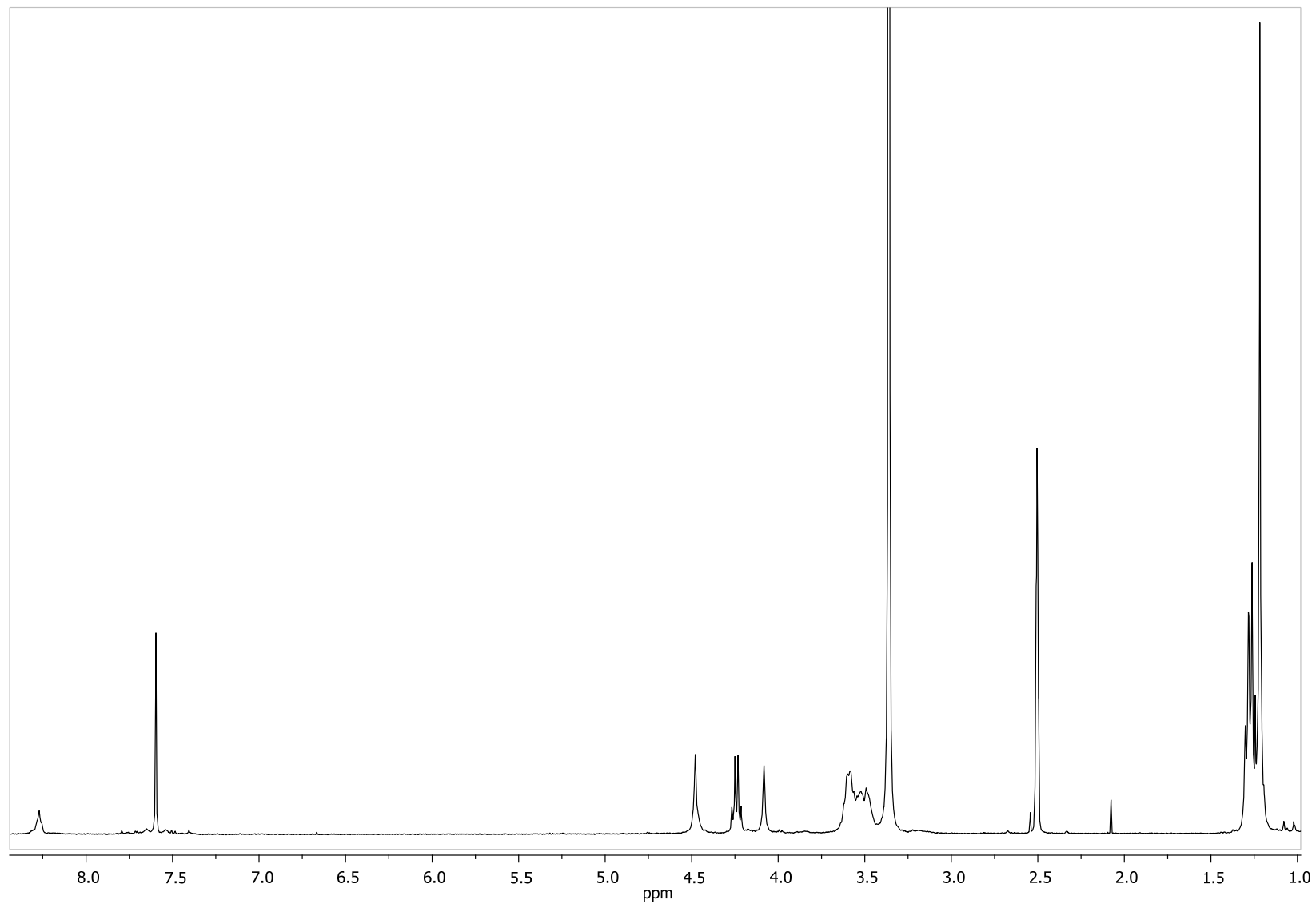


Fig. S16. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-alternate 35), DMSO- d_6 , 298 K, 400 MHz

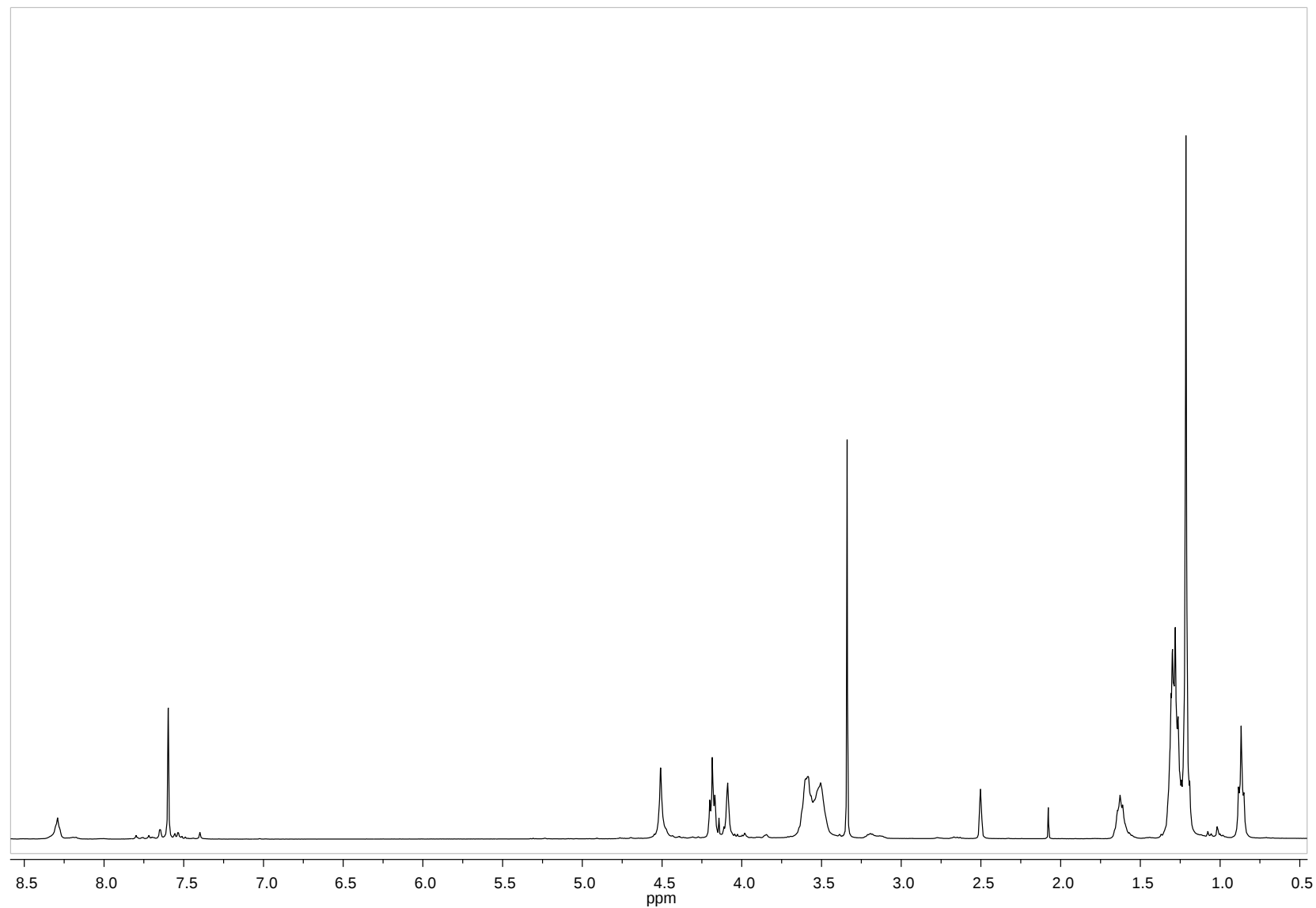


Fig. S17. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 39), $\text{DMSO-}d_6$, 298 K, 400 MHz

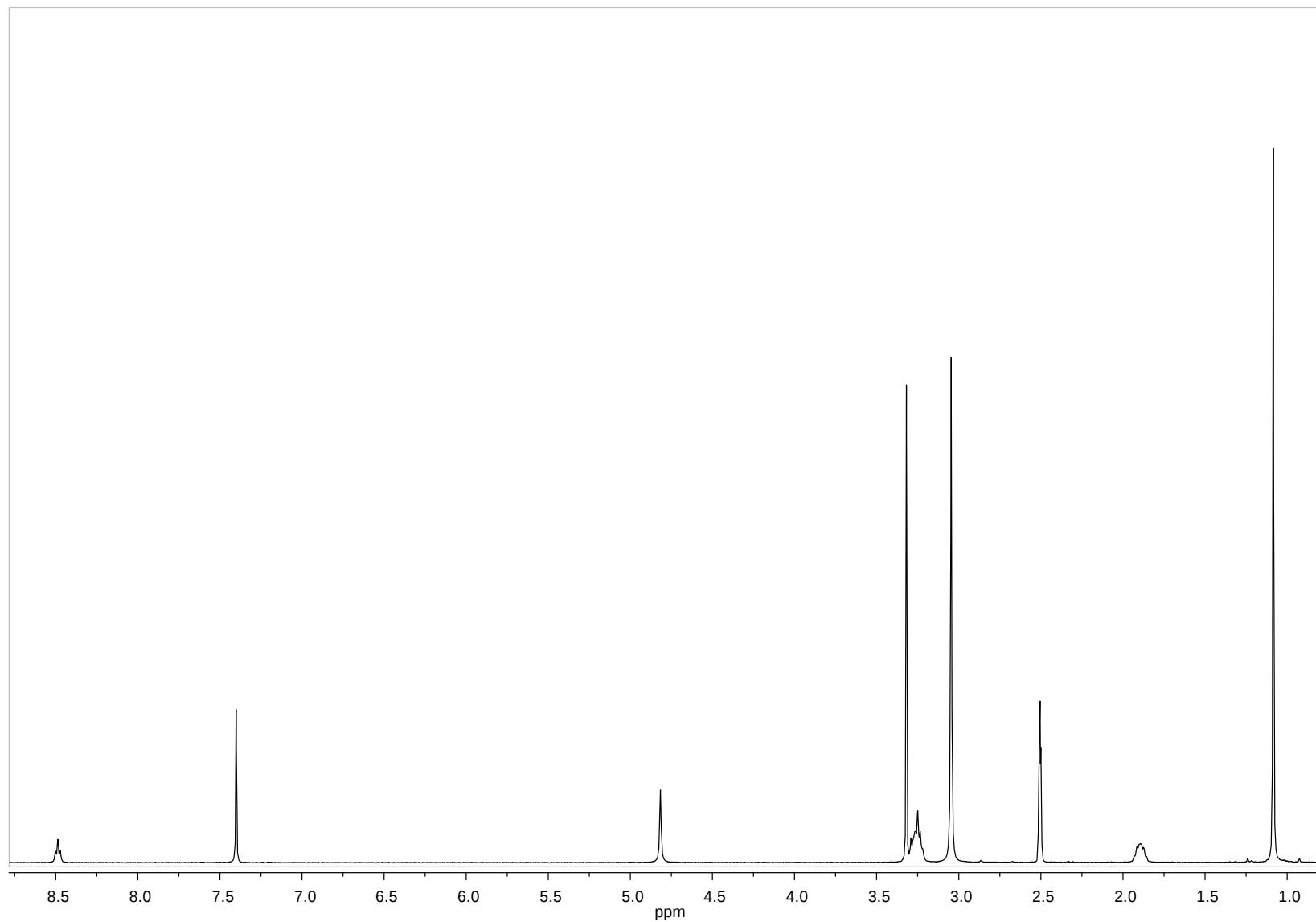


Fig. S18. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 40*), DMSO- d_6 , 298 K, 400 MHz

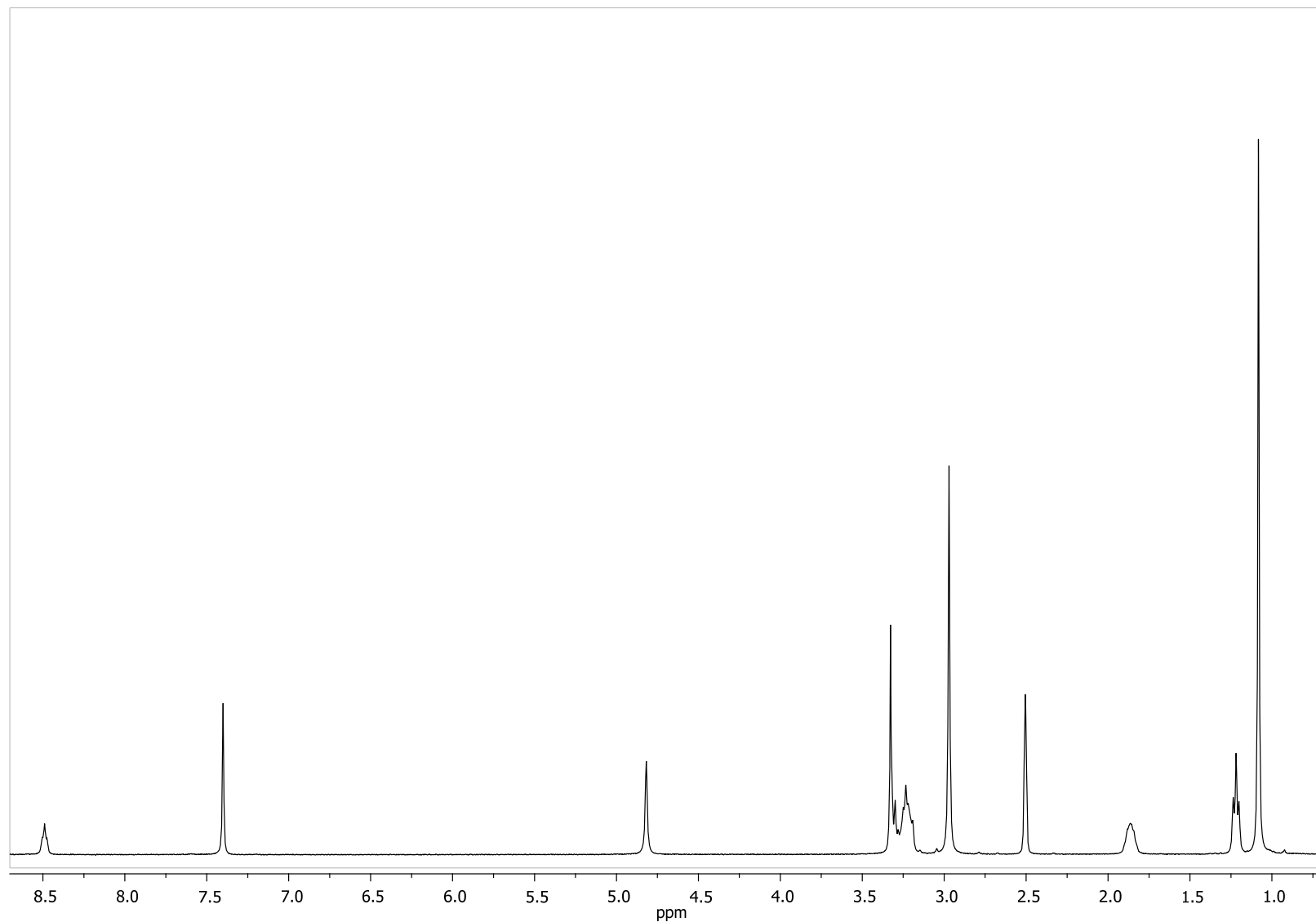


Fig. S19. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 41), CDCl_3 , 298 K, 400 MHz

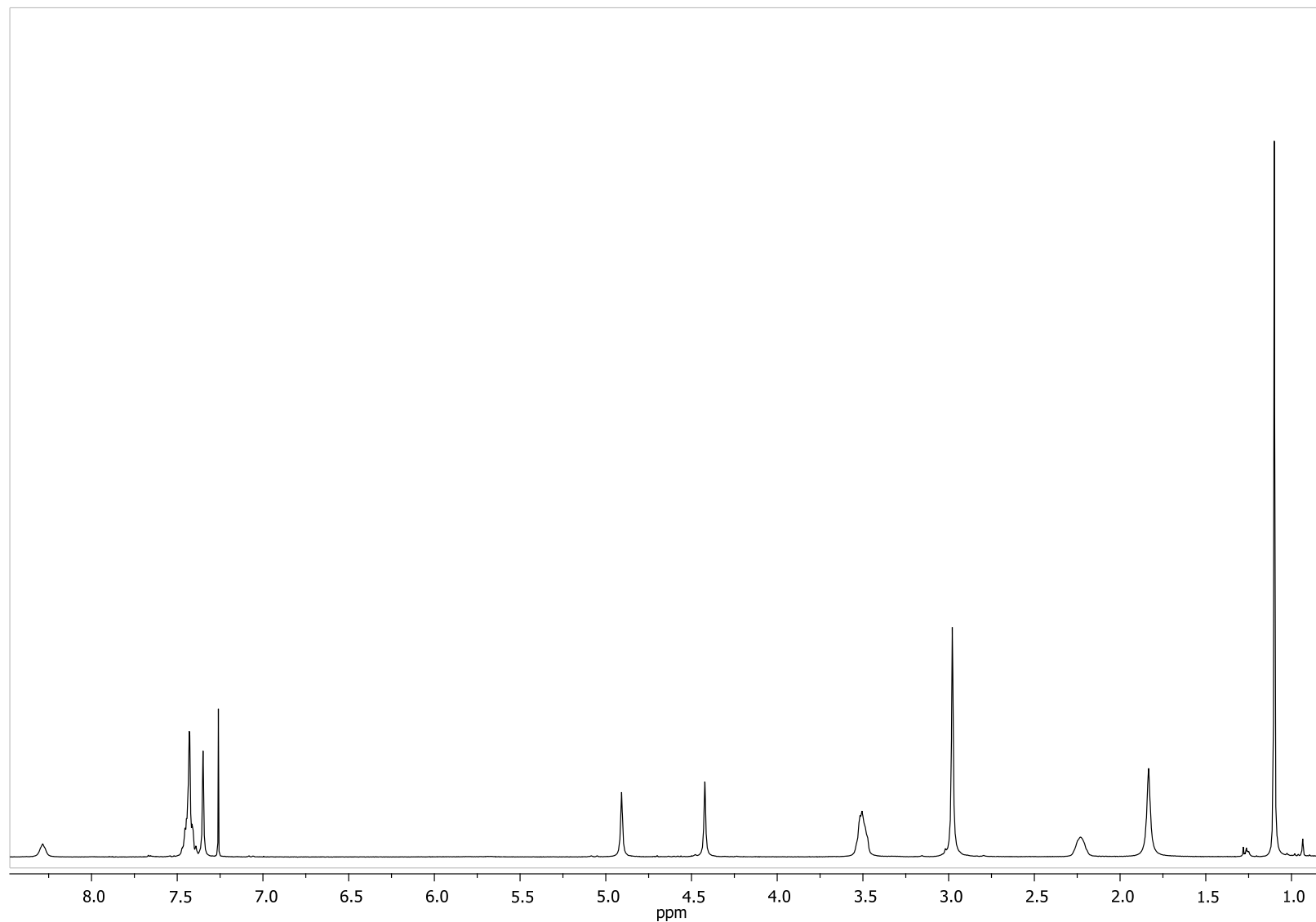


Fig. S20. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)ammoniumpropyl)carbomoylmethoxy)-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 42), CDCl_3 , 298 K, 400 MHz

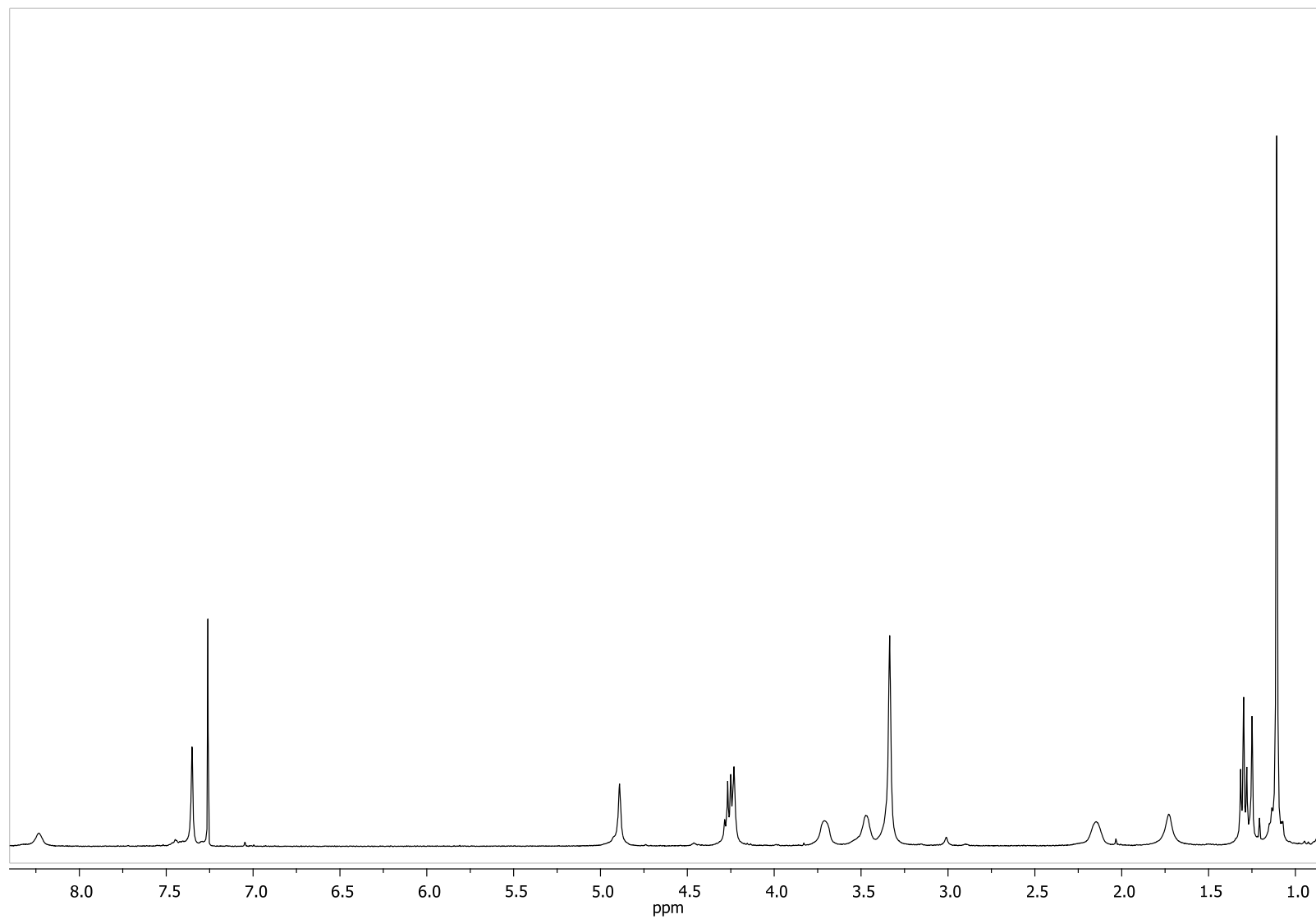


Fig. S21. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxy carbonylmethyl) ammoniumpropyl) carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 43), DMSO- d_6 , 298 K, 400 MHz

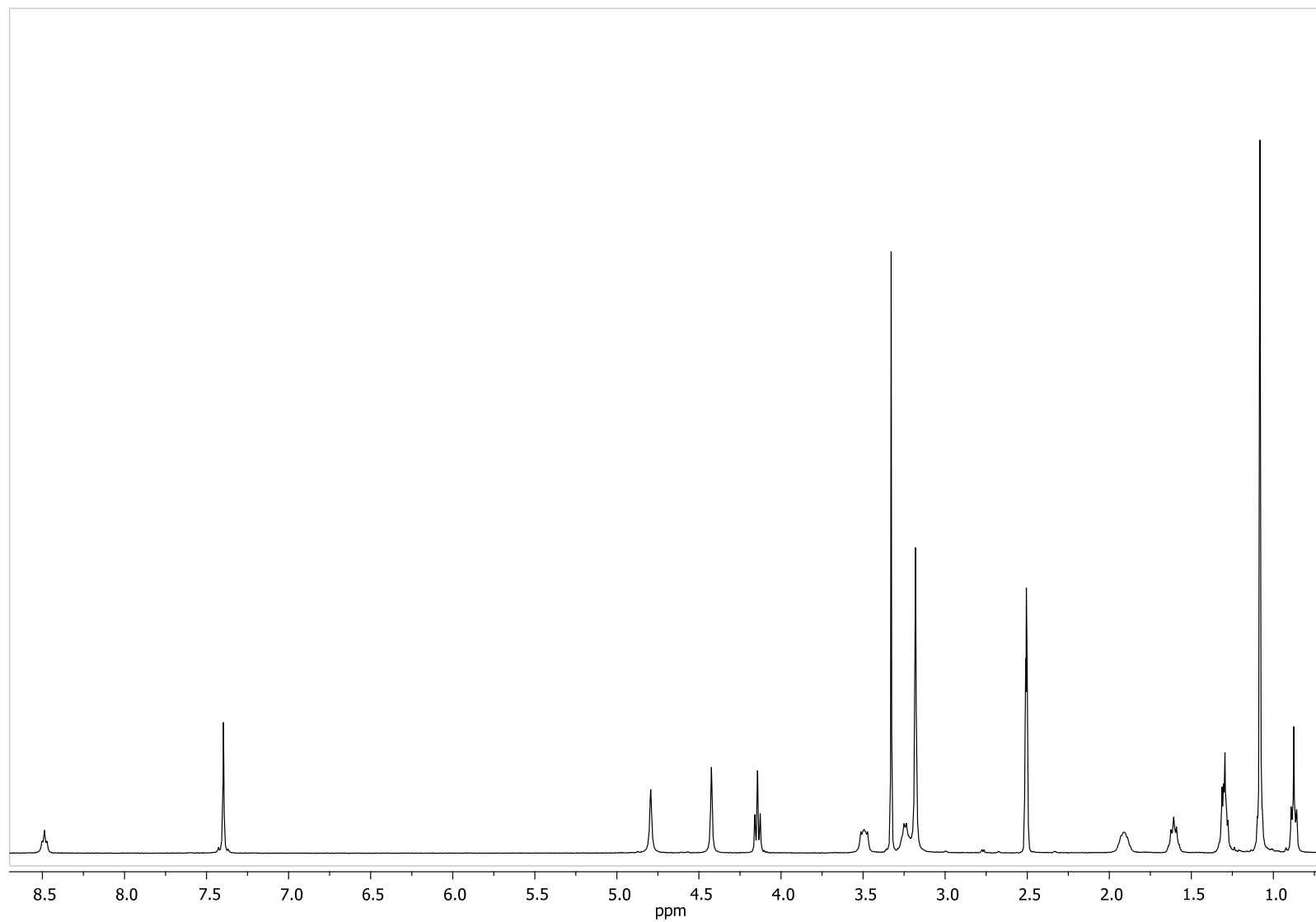


Fig. S22. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonylmethyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 44*), CDCl_3 , 298 K, 400 MHz

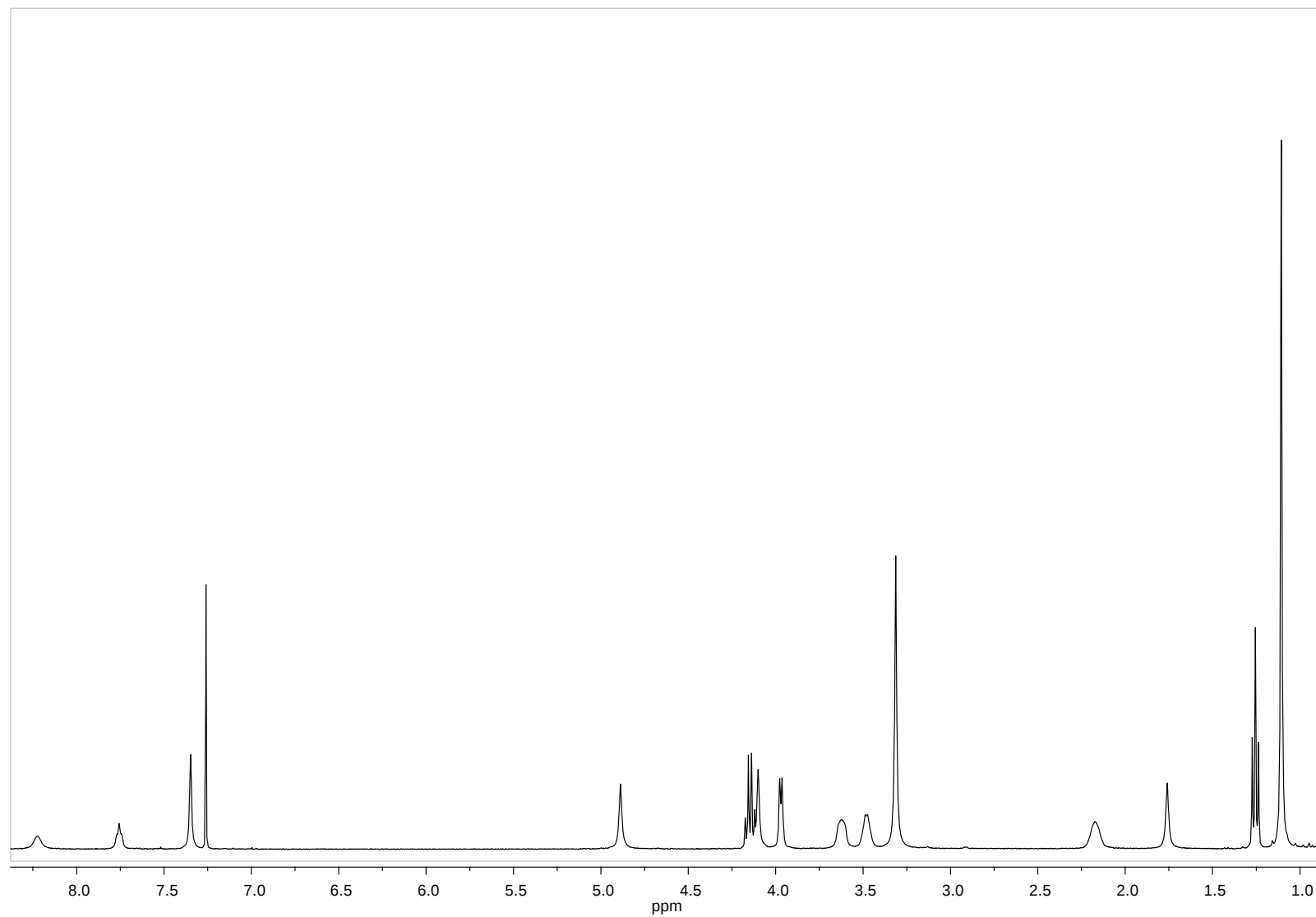


Fig. S23. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{[ethoxycarbonylmethyl]-amidocarbonylmethyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 45), DMSO- d_6 , 298 K, 400 MHz

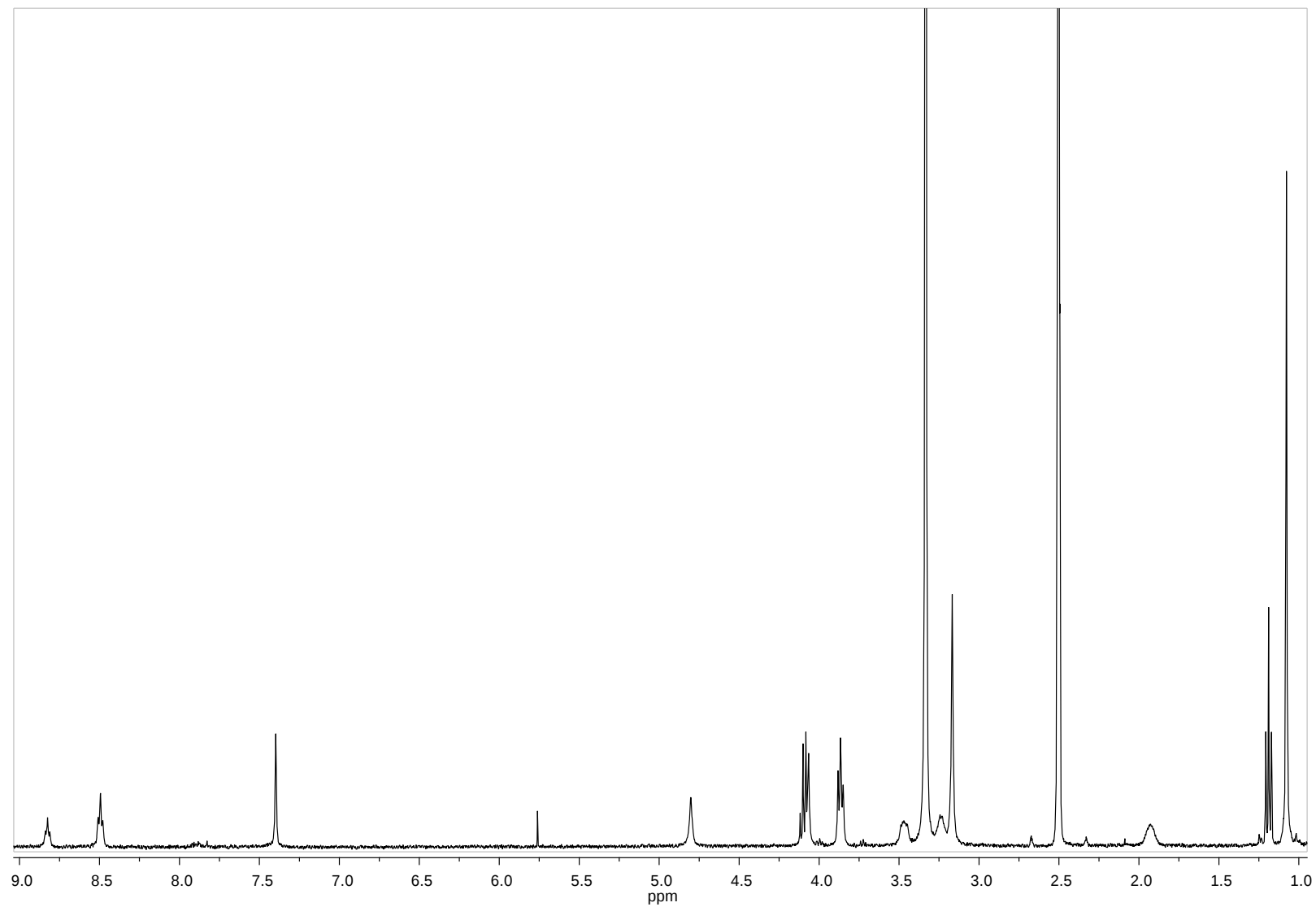


Fig. S24. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 46), DMSO- d_6 , 298 K, 400 MHz

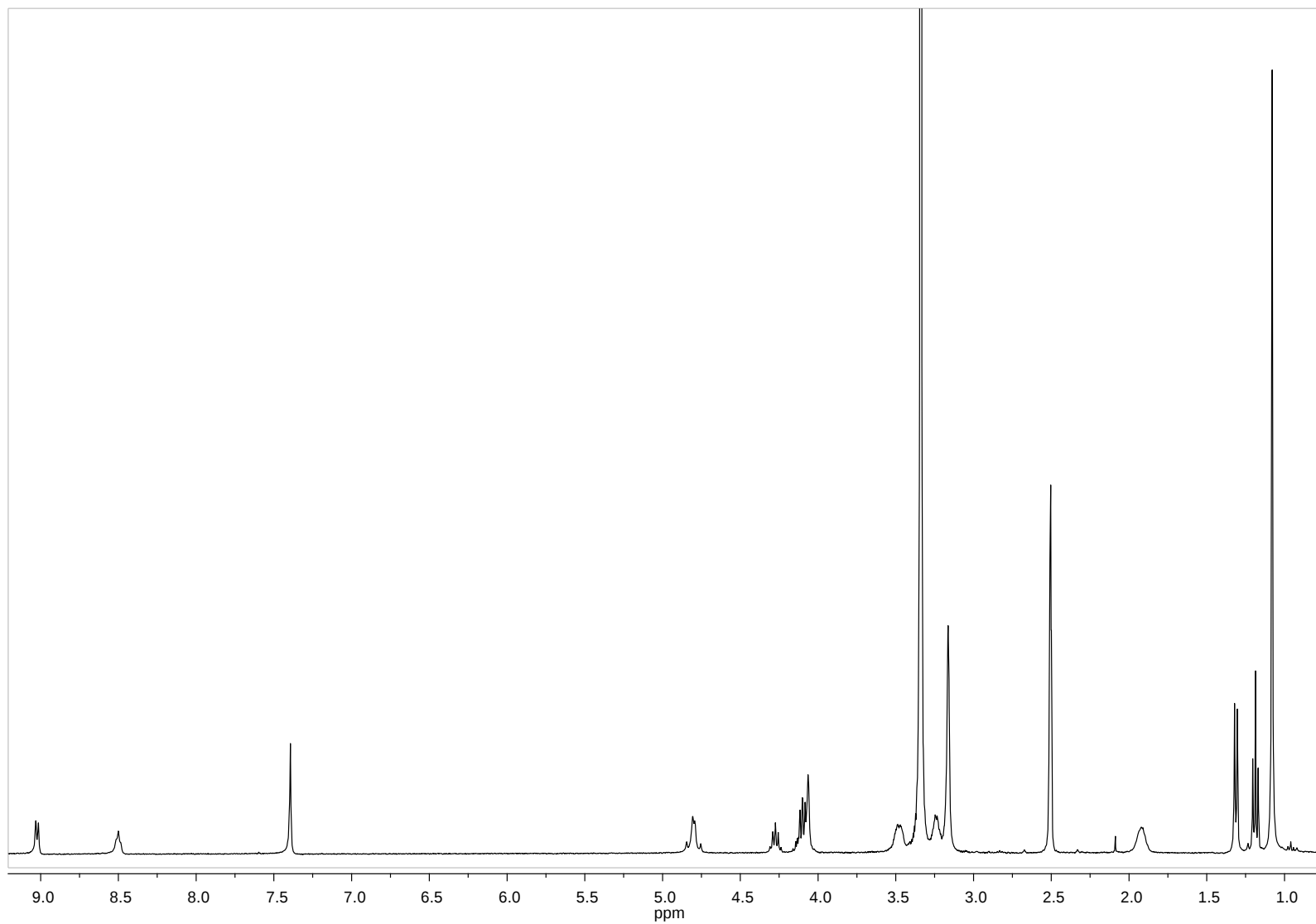


Fig. S25. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide}ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 47), DMSO- d_6 , 298 K, 400 MHz

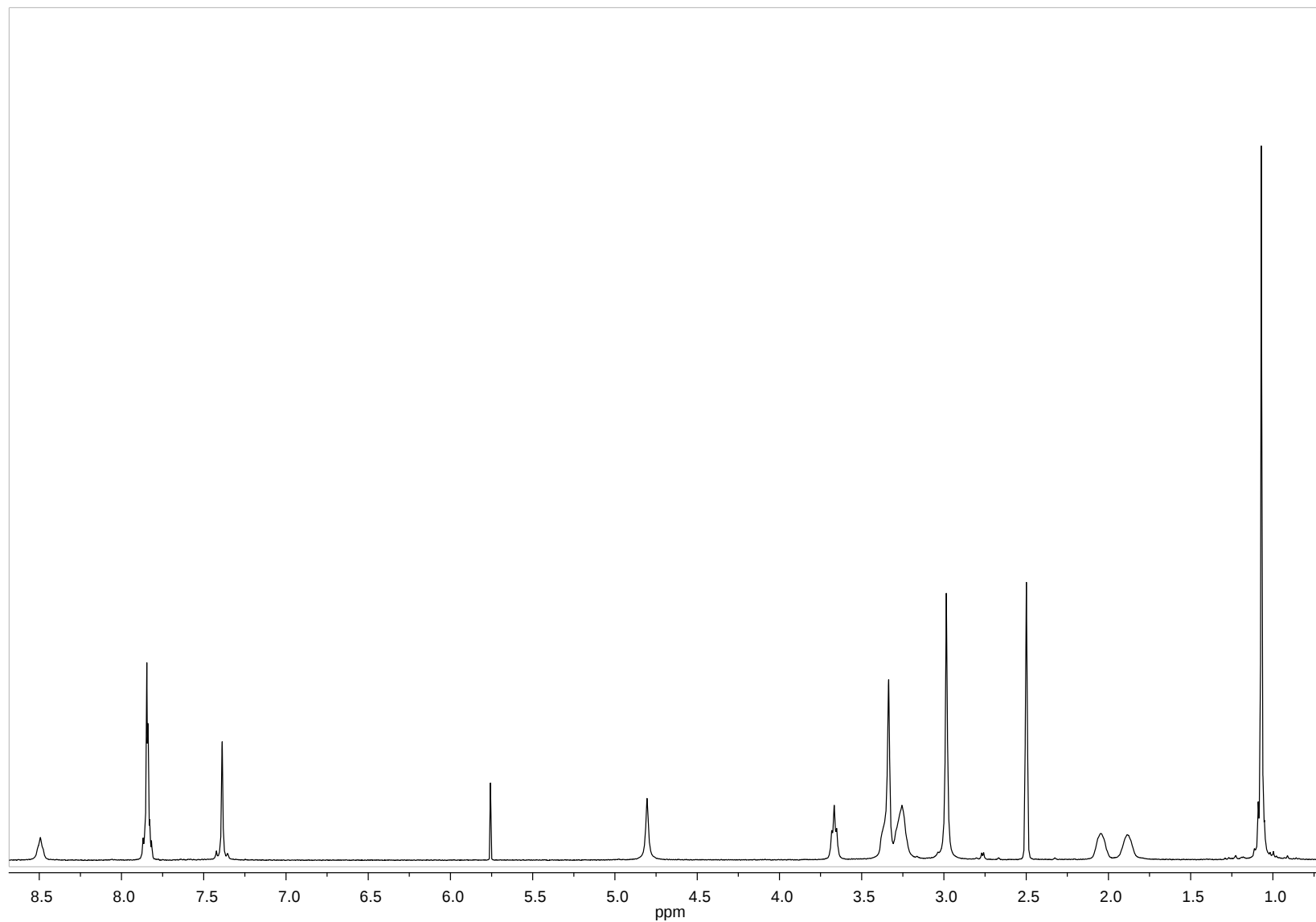


Fig. S26. ¹H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[N-(2'-methyl-2',2'-diethyl)ammoniummethyl]carbomoylmethoxy]-2,8,14,20-tetrathiocalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 48), CDCl₃, 298 K, 400 MHz

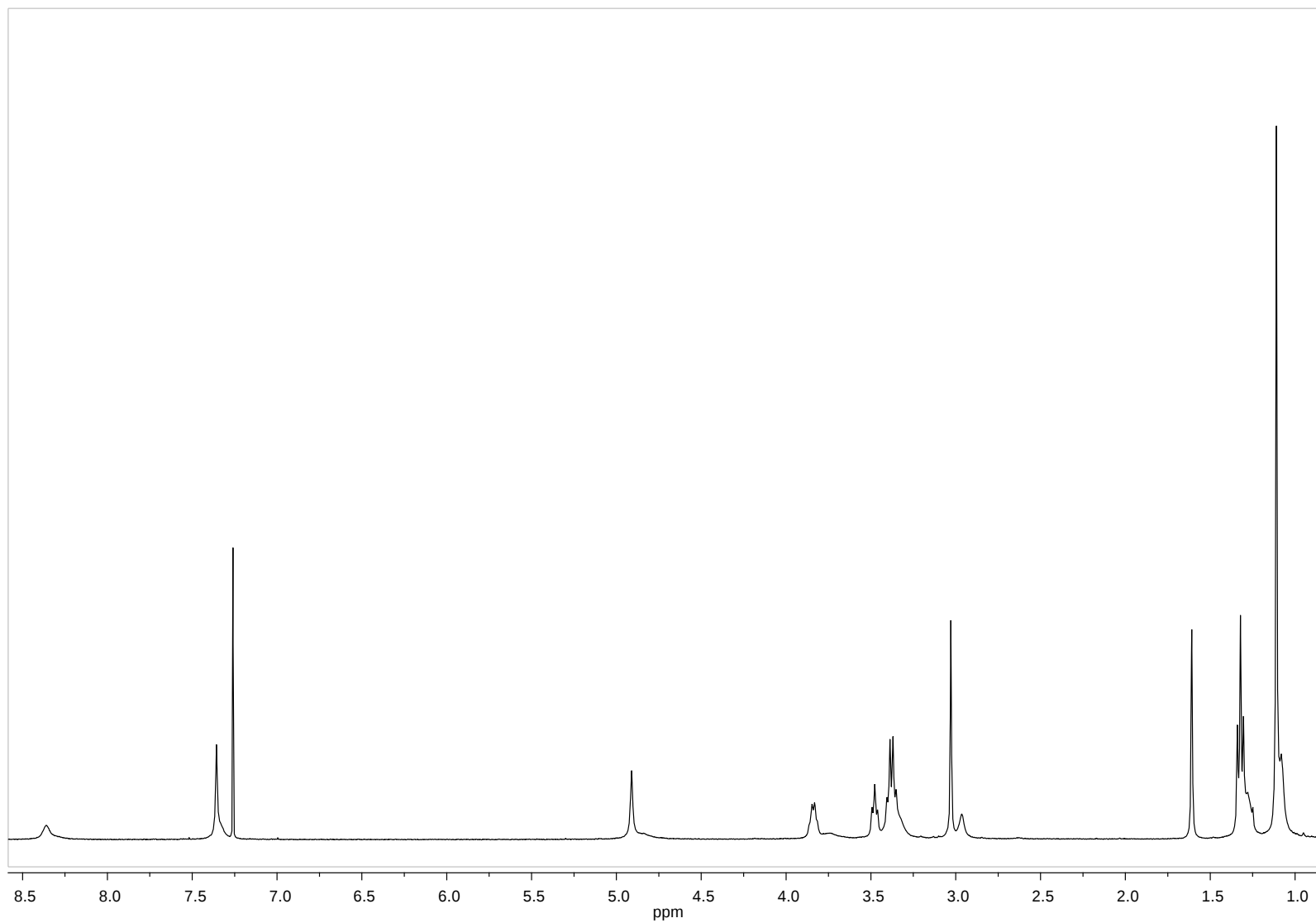


Fig. S27. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 49), CDCl_3 , 298 K, 400 MHz

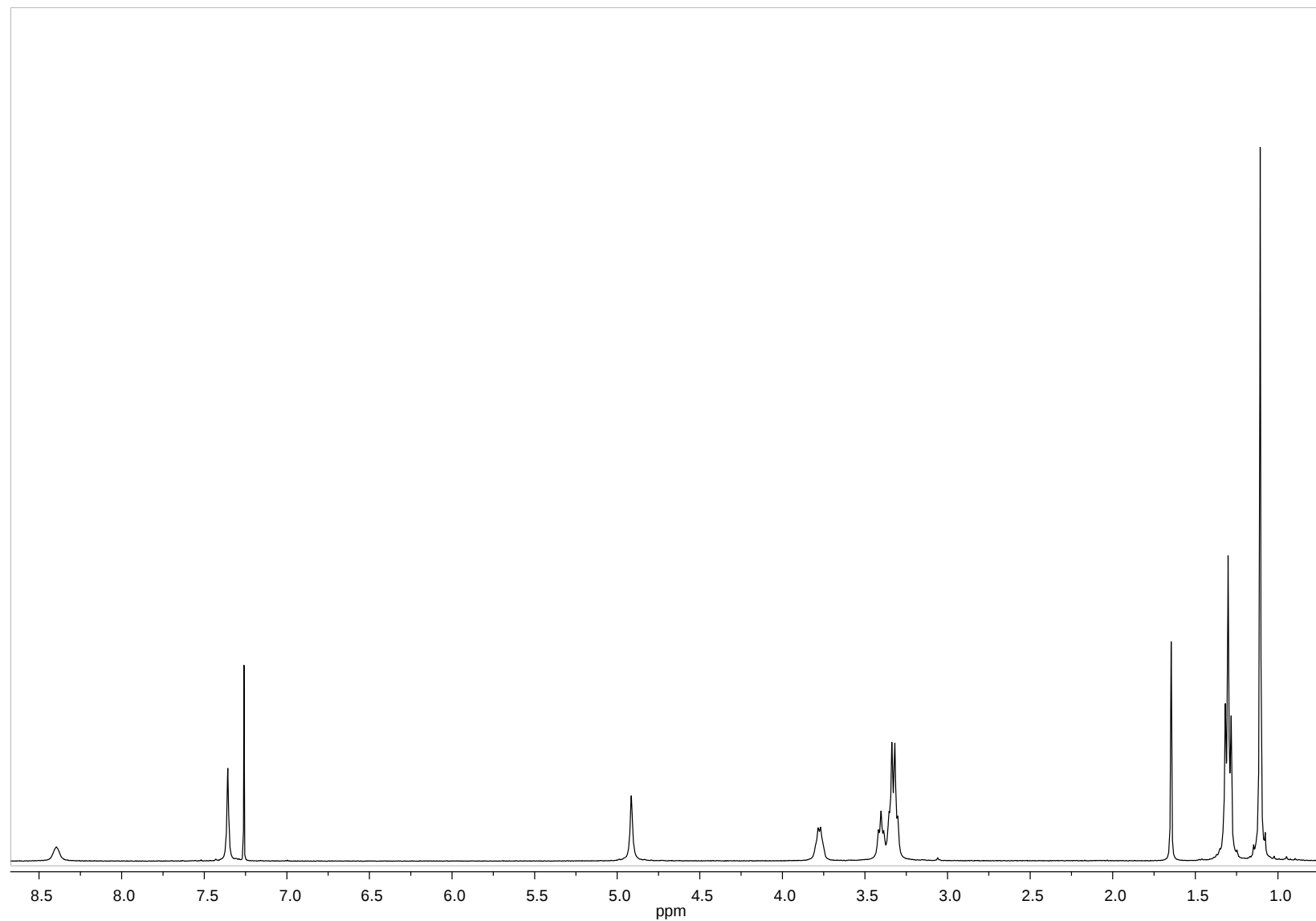


Fig. S28. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 50), CDCl_3 , 298 K, 400 MHz

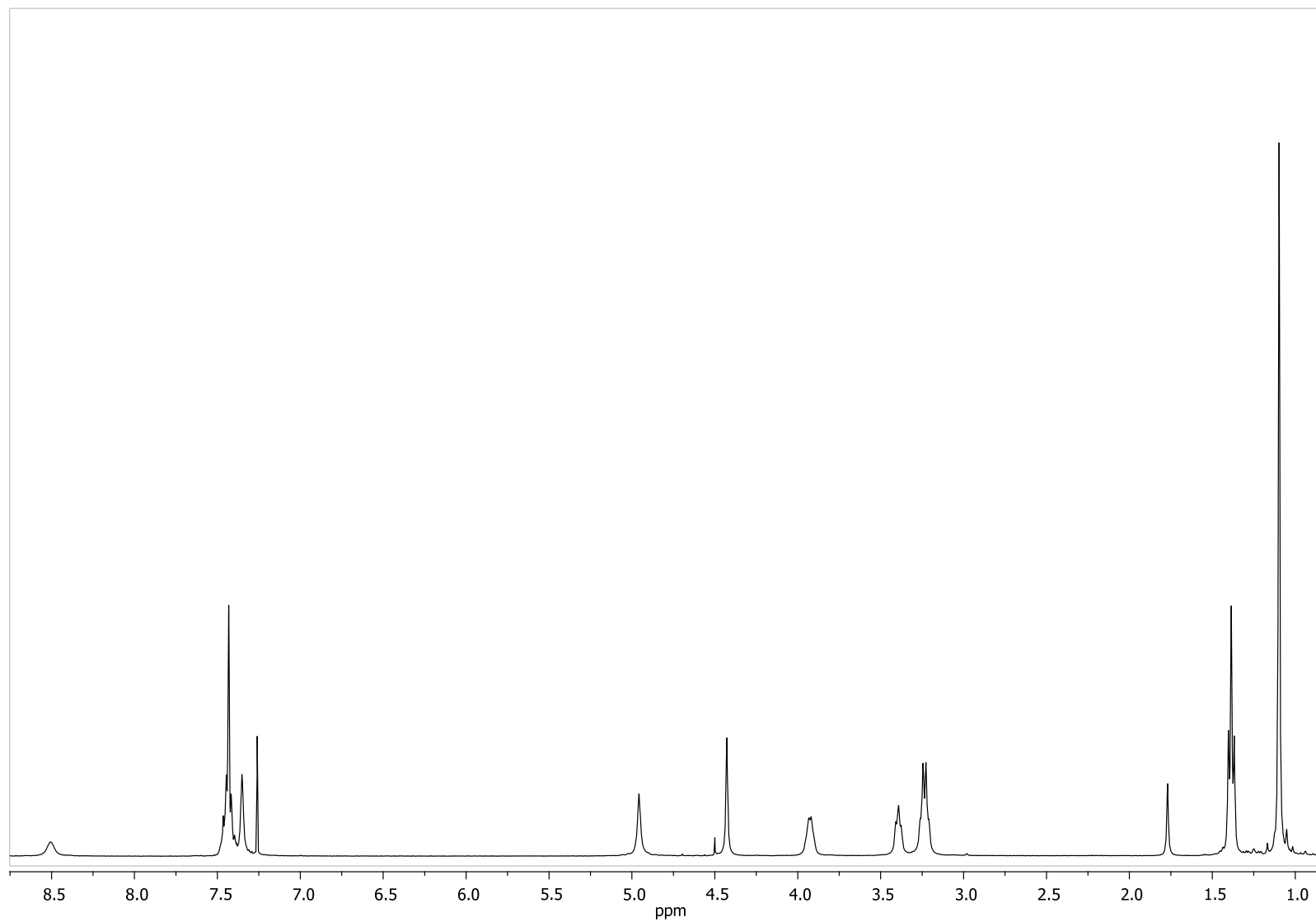


Fig. S29. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 51), CDCl_3 , 298 K, 400 MHz

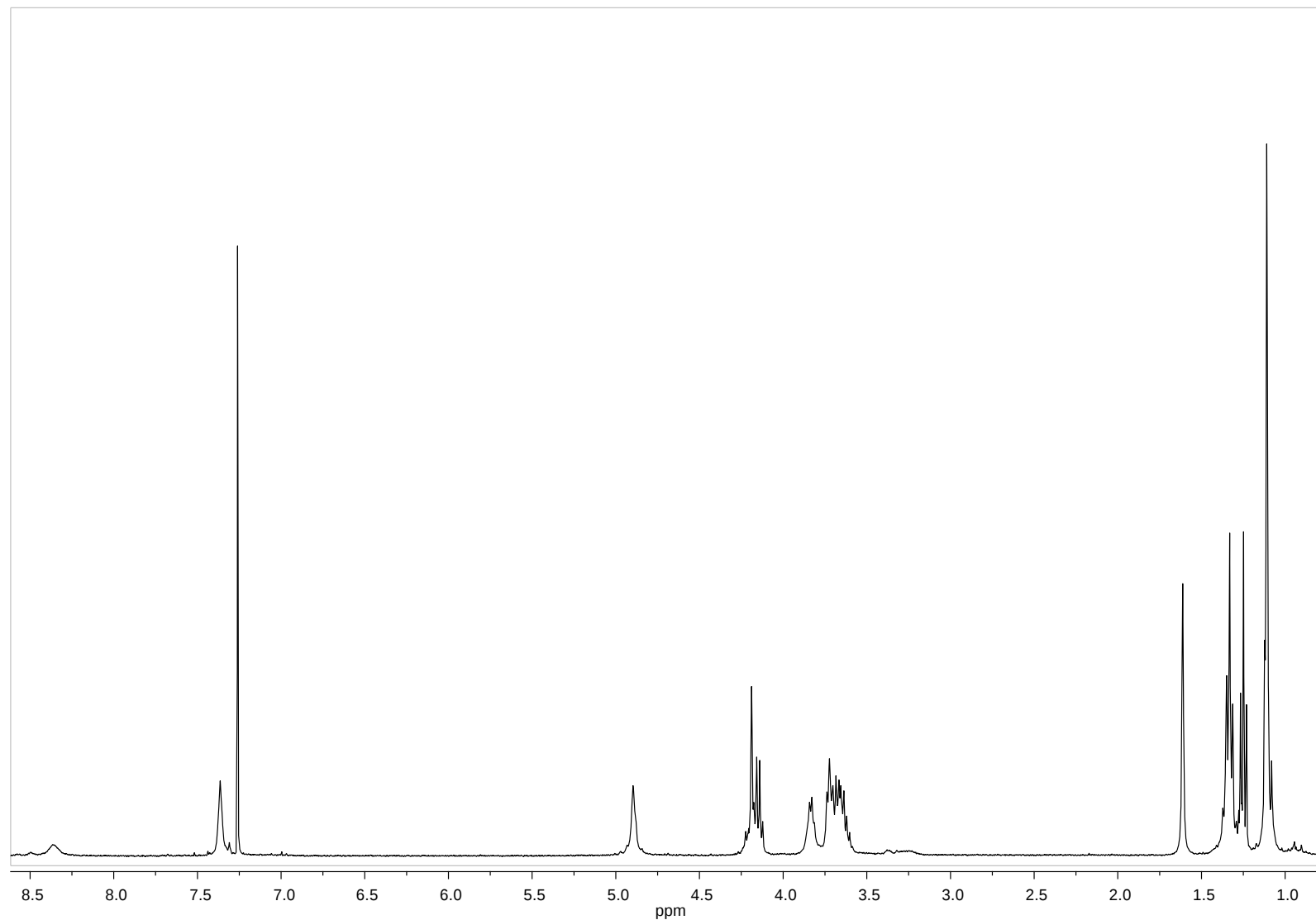


Fig. S30. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 52), DMSO- d_6 , 298 K, 400 MHz

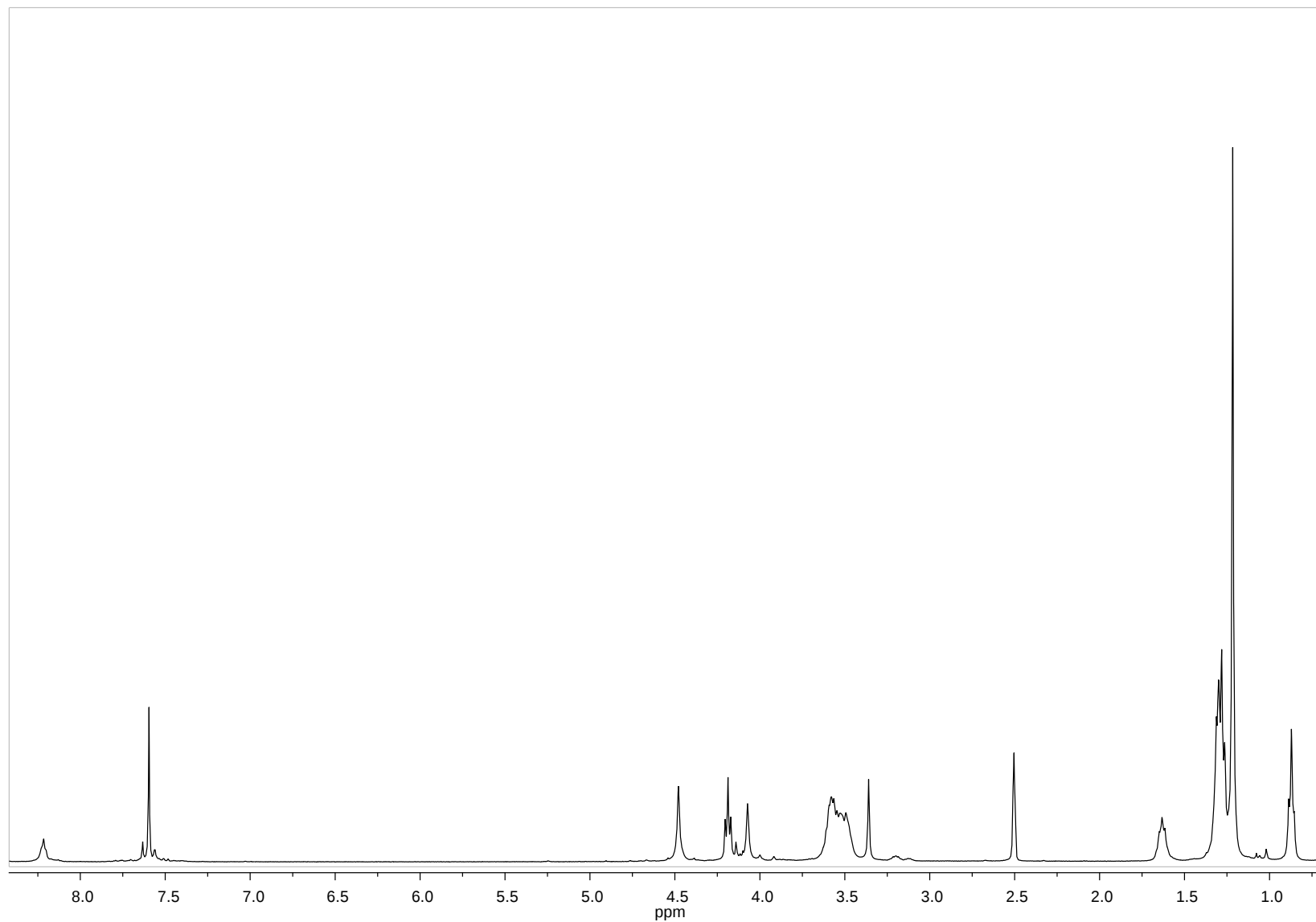


Fig. S31. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonylmethyl)amidocarbonylmethyl})ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 53), DMSO- d_6 , 298 K, 400 MHz

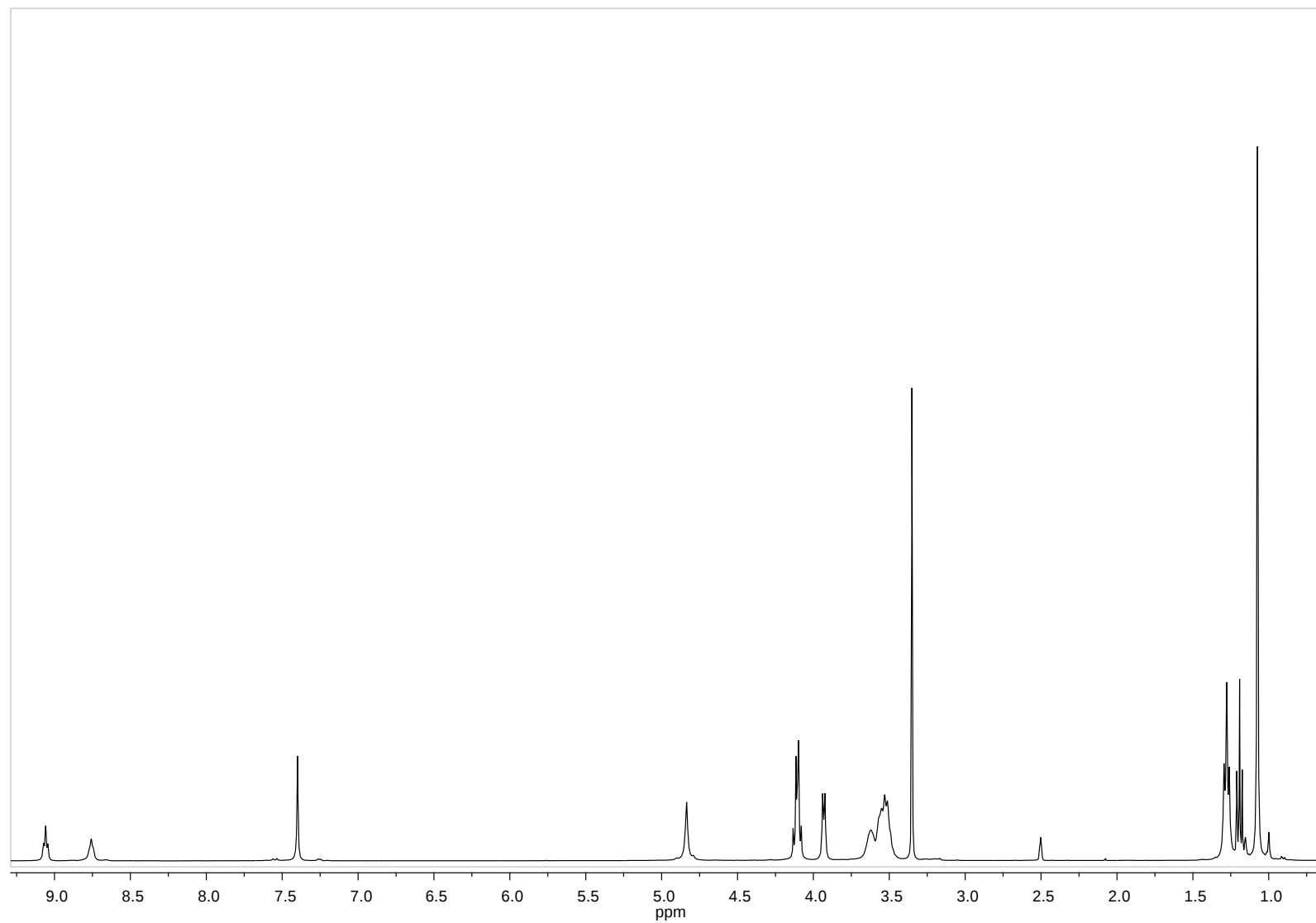


Fig. S32. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{([ethoxycarbonylmethyl]-amidocarbonylmethyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 54), DMSO- d_6 , 298 K, 400 MHz

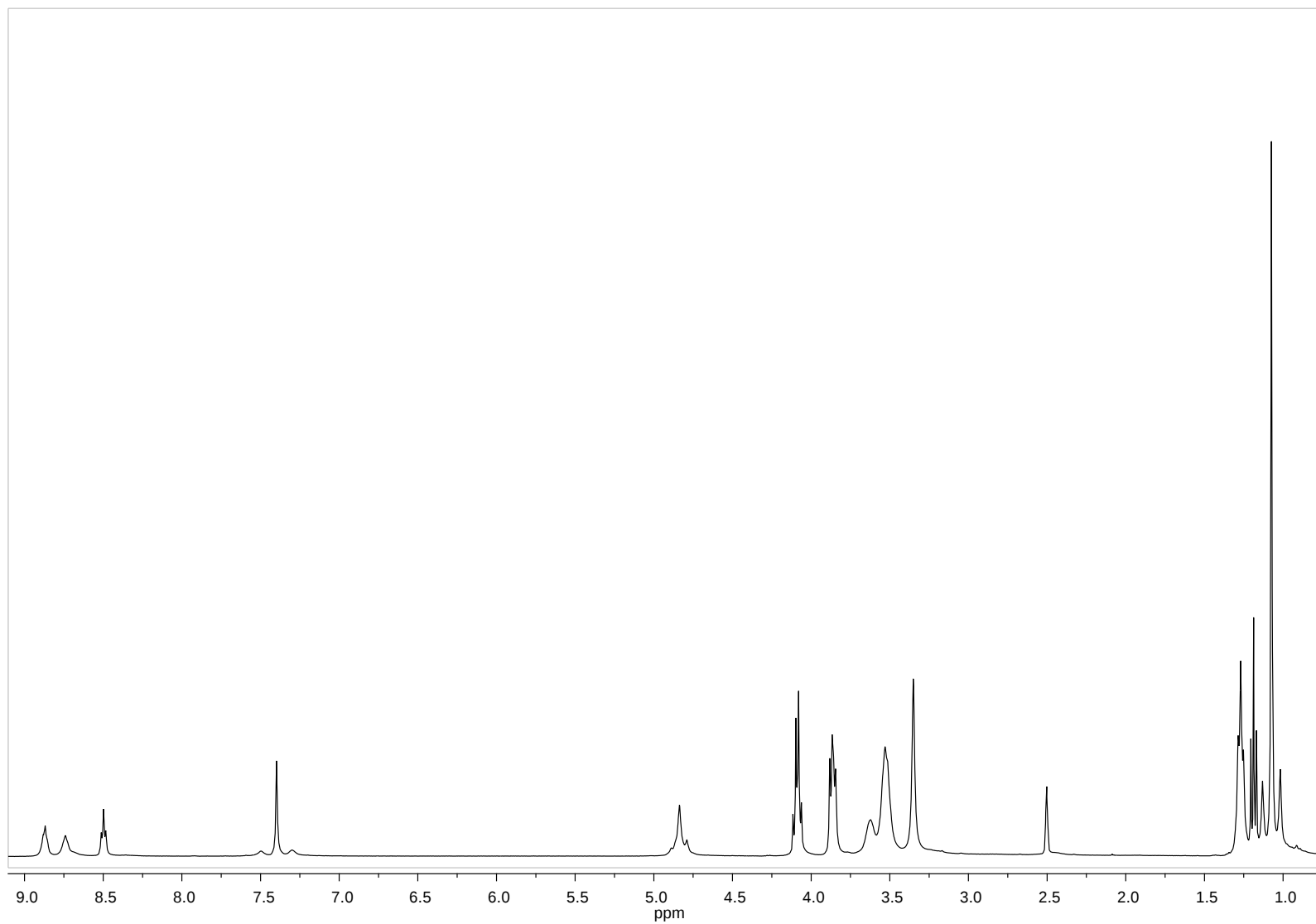


Fig. S33. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonyl}[S-methyl]methyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 55), DMSO- d_6 , 298 K, 400 MHz

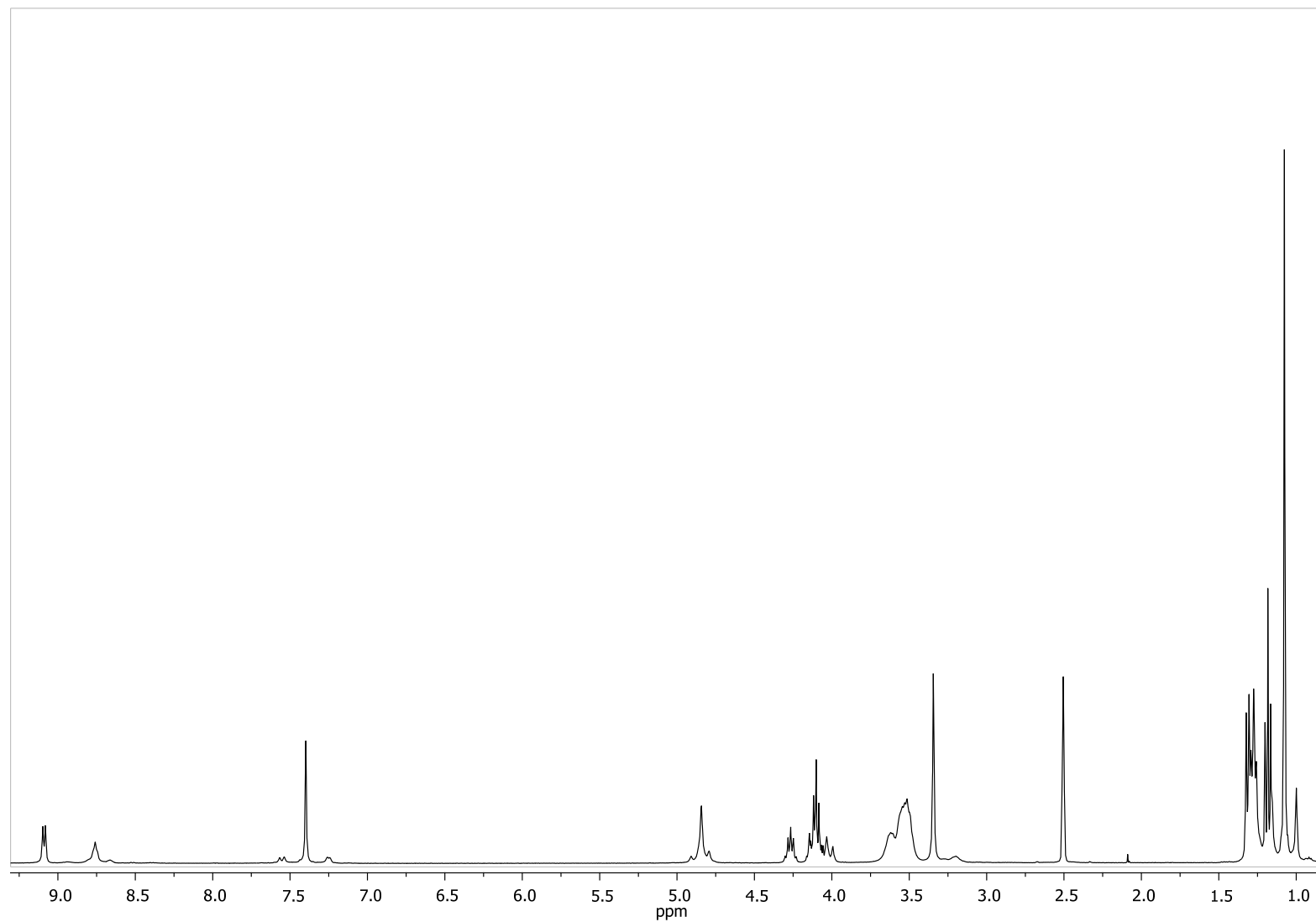


Fig. S34. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 56), DMSO-d_6 , 298 K, 400 MHz

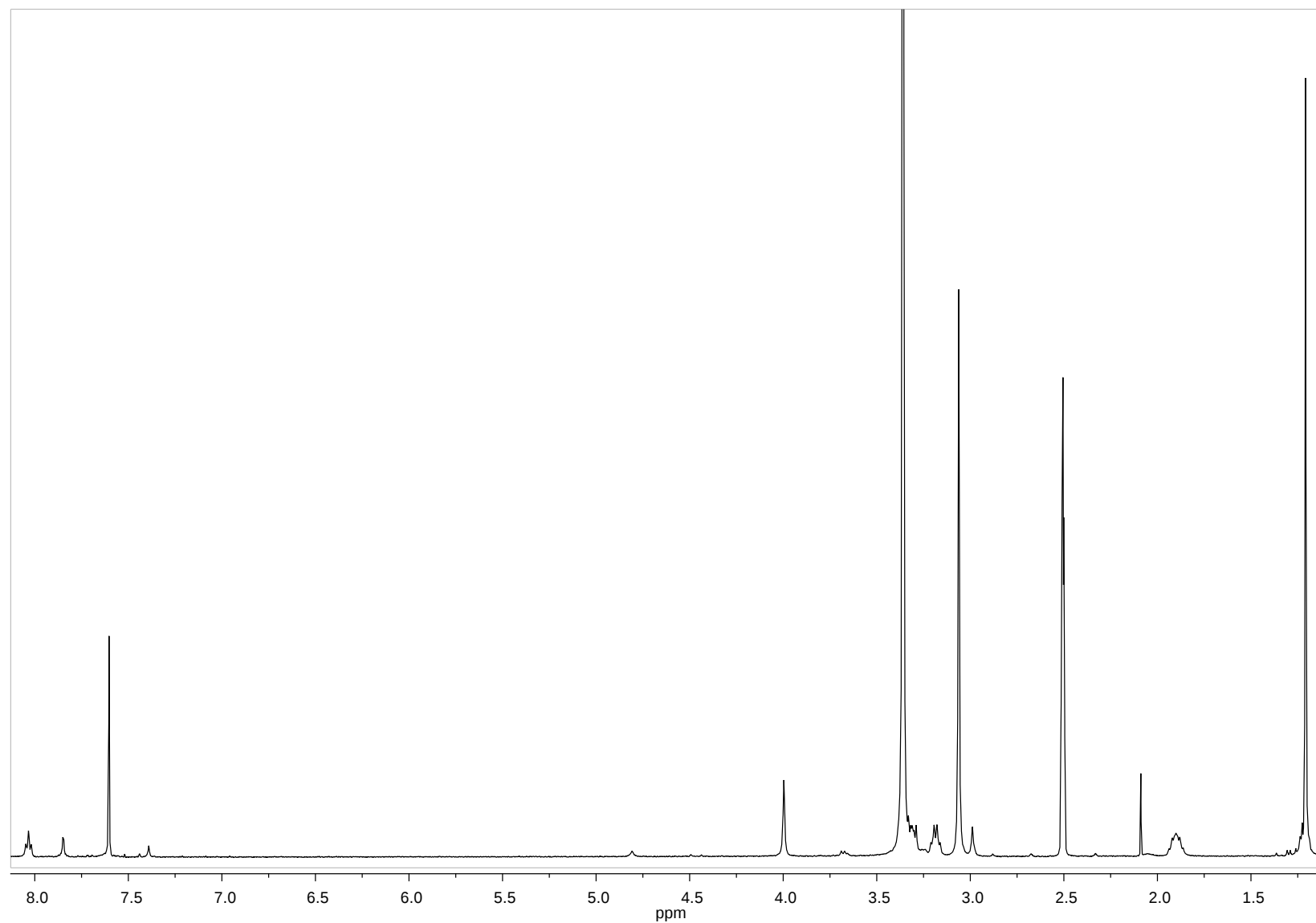


Fig. S35. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 57), DMSO-d_6 , 298 K, 400 MHz

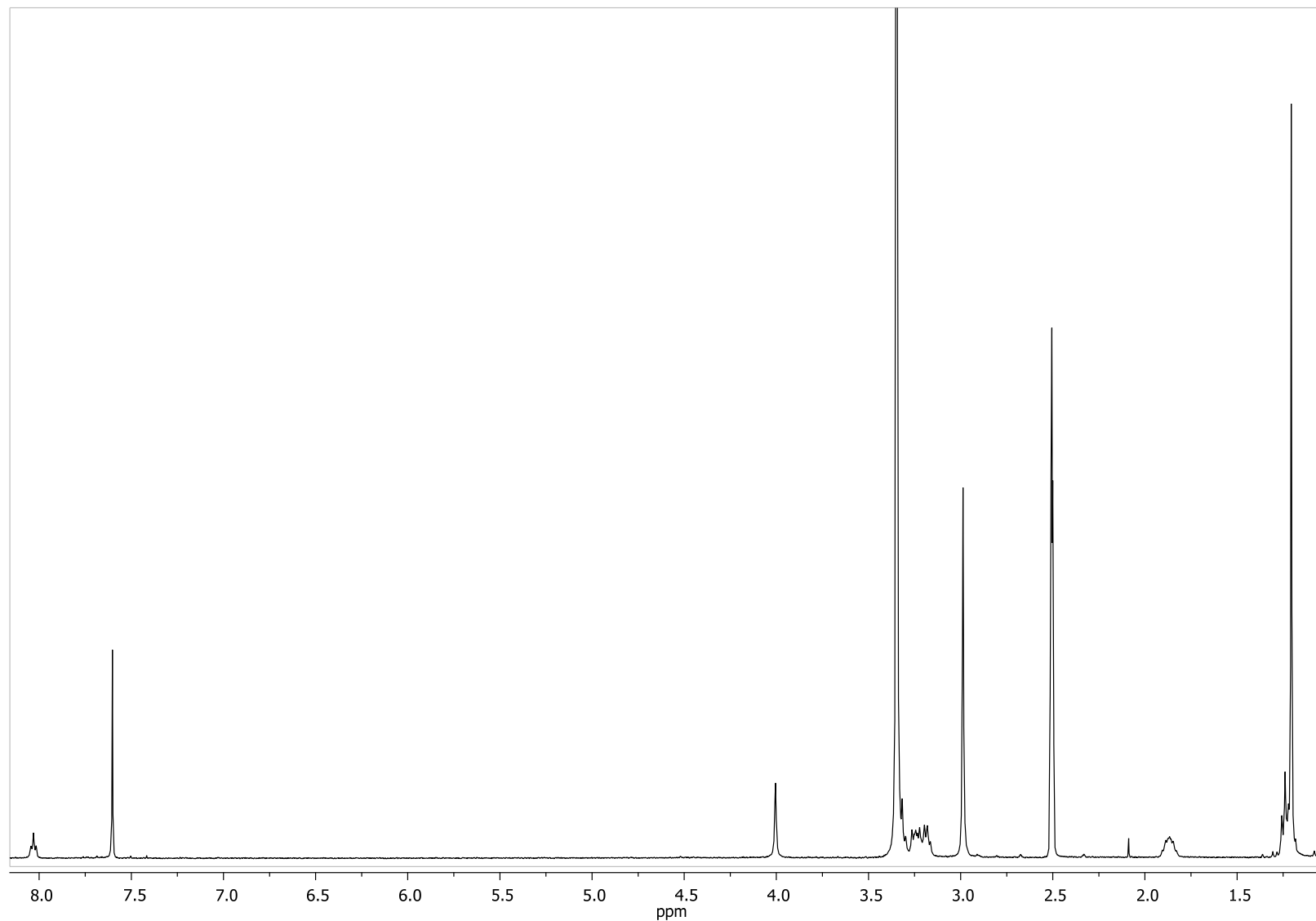


Fig. S36. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 58), DMSO-d_6 , 298 K, 400 MHz

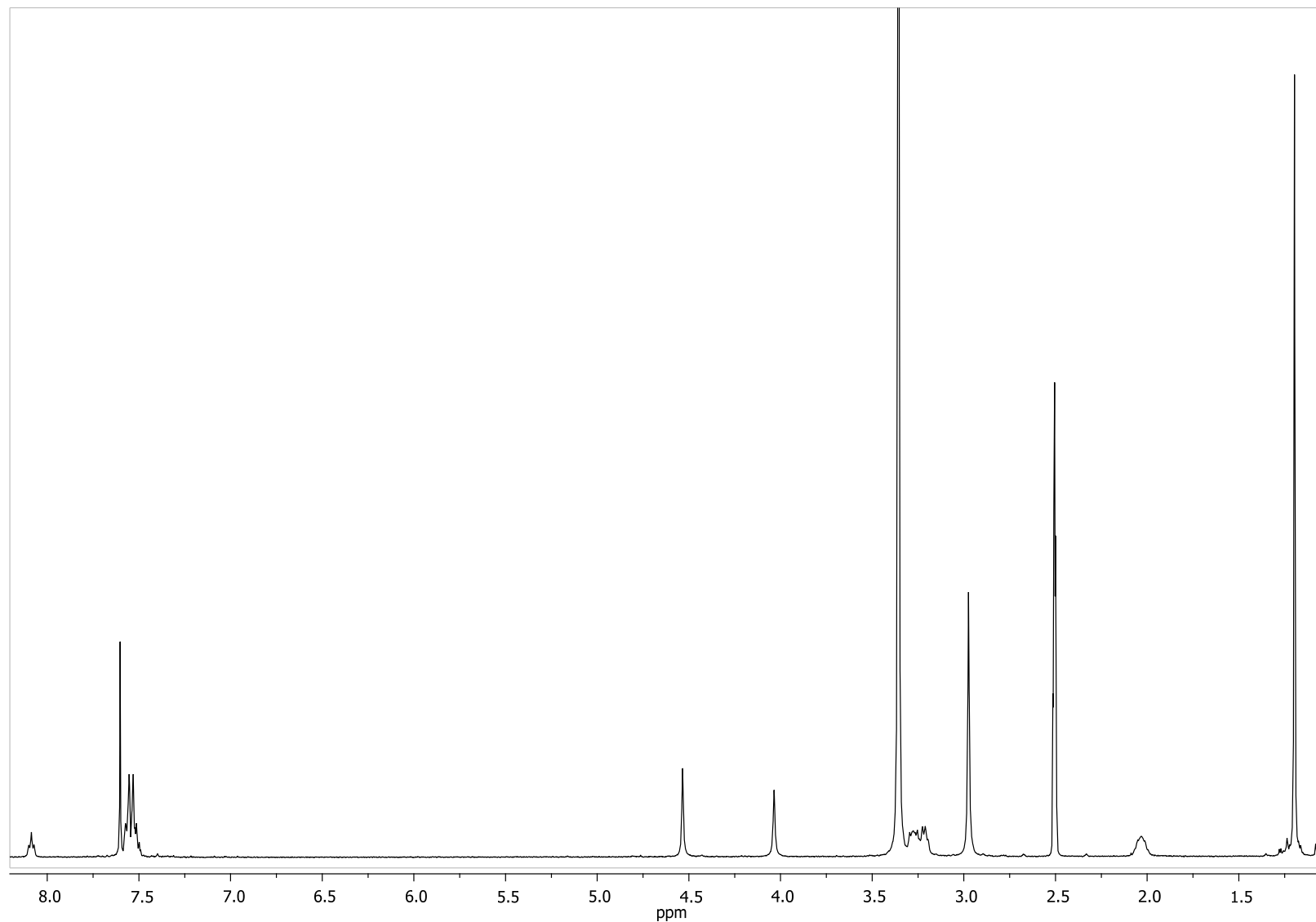


Fig. S37. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)ammoniumpropyl)carbomoylmethoxy)-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 59), DMSO- d_6 , 298 K, 400 MHz

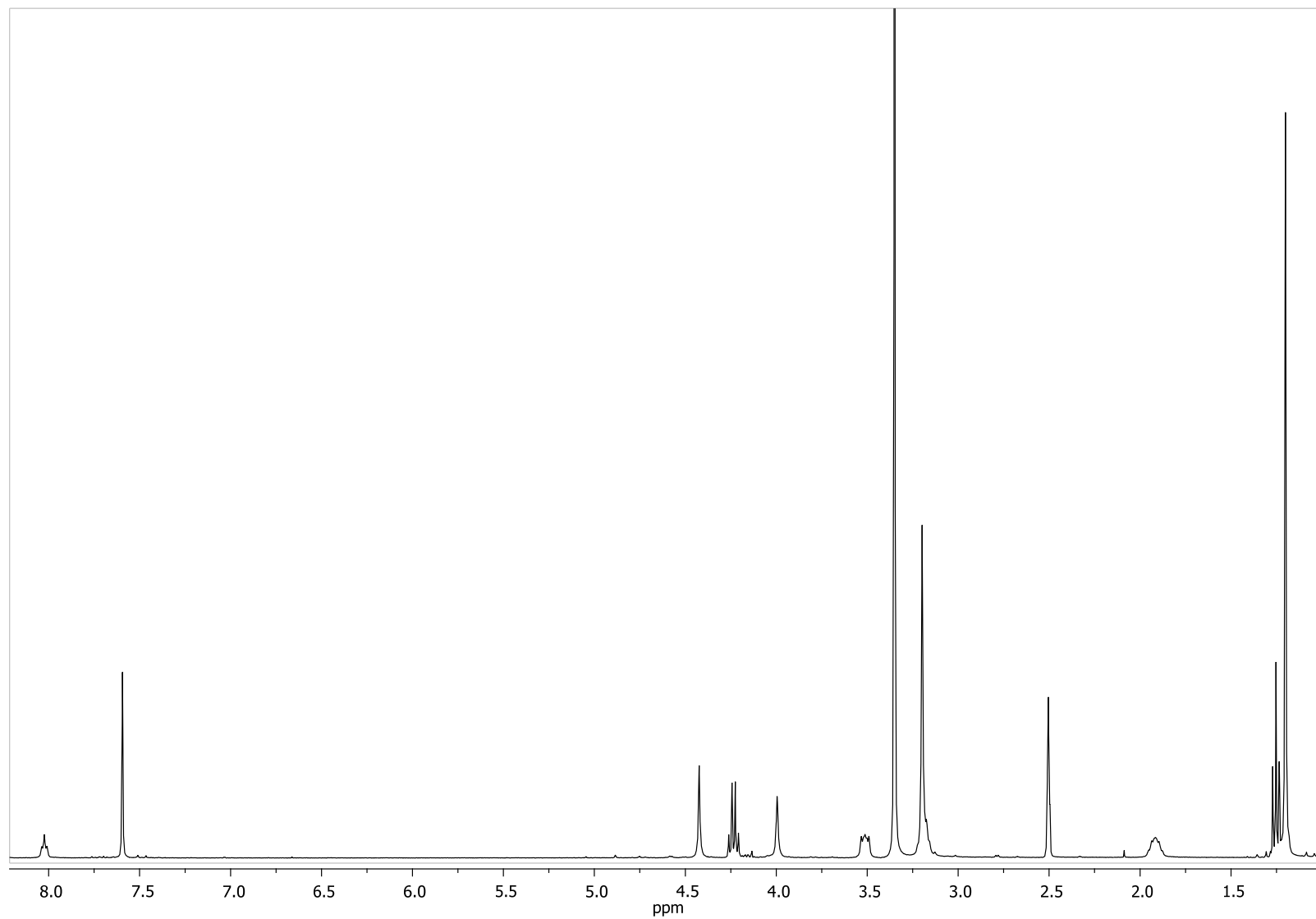


Fig. S38. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxy carbonylmethyl) ammoniumpropyl) carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate **60**), DMSO- d_6 , 298 K, 400 MHz

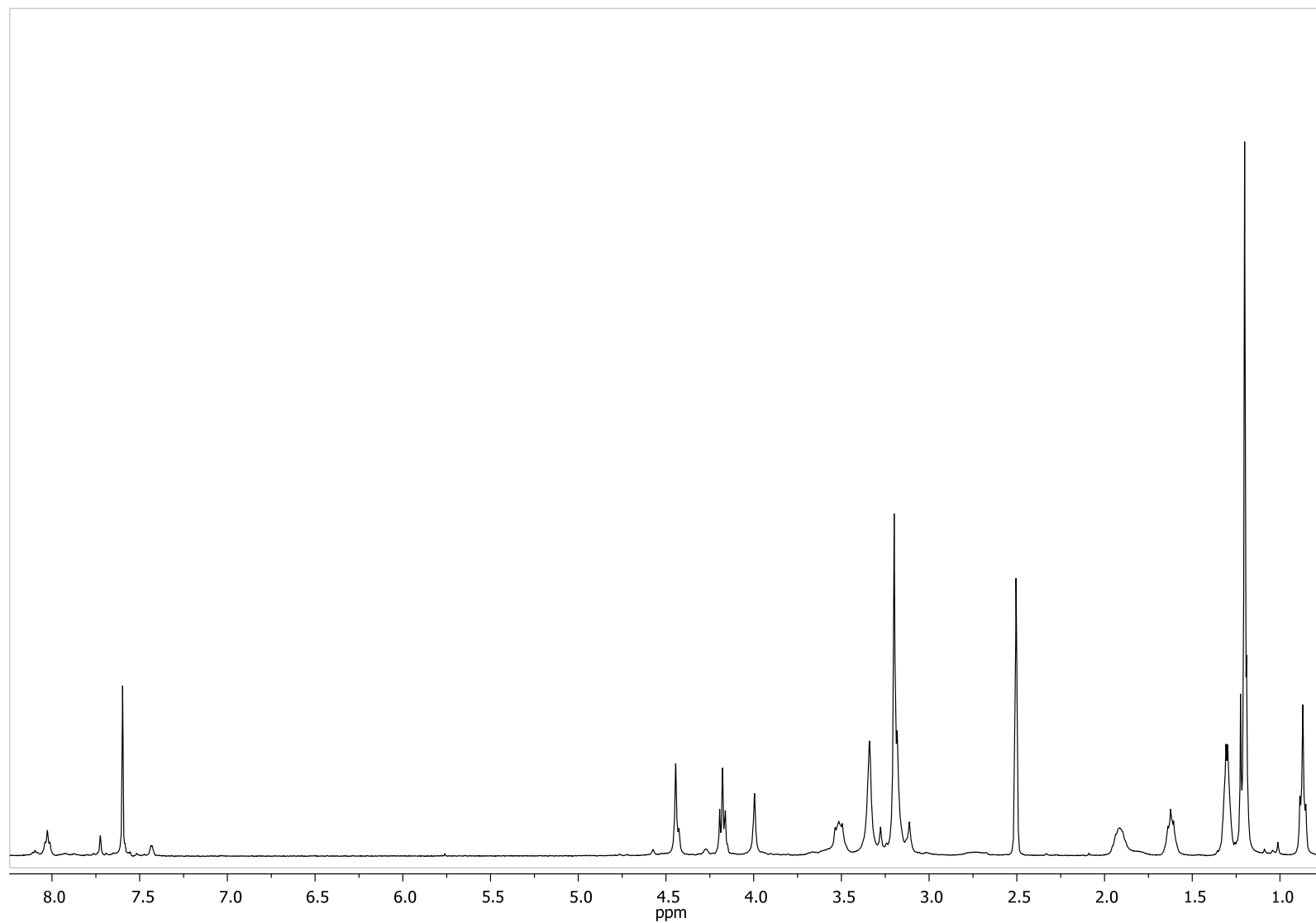


Fig. S39. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonylmethyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* **61**), DMSO- d_6 , 298 K, 400 MHz

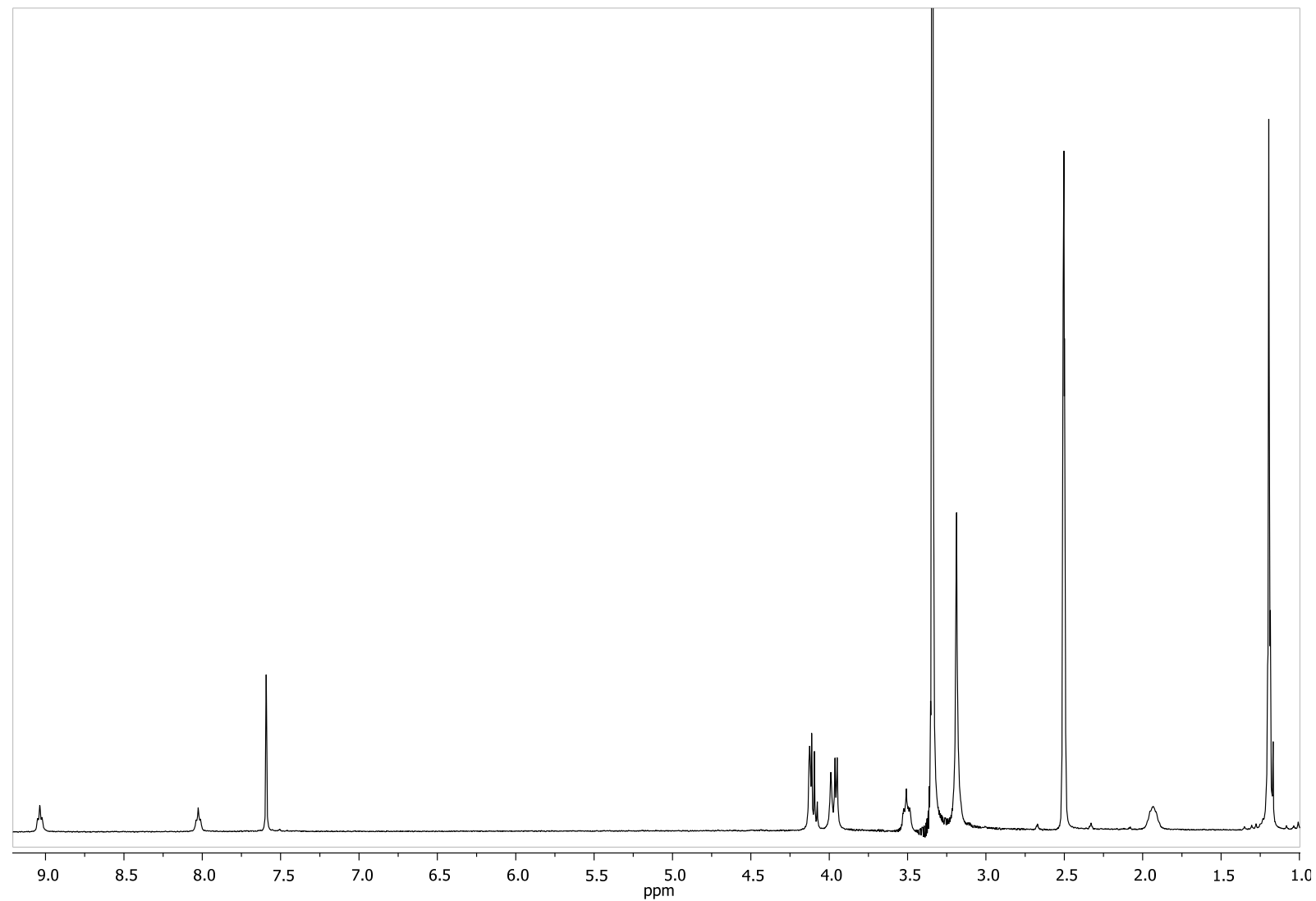


Fig. S40. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{([ethoxycarbonylmethyl]amidocarbonylmethyl)amidocarbonylmethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 62), DMSO- d_6 , 298 K, 400 MHz

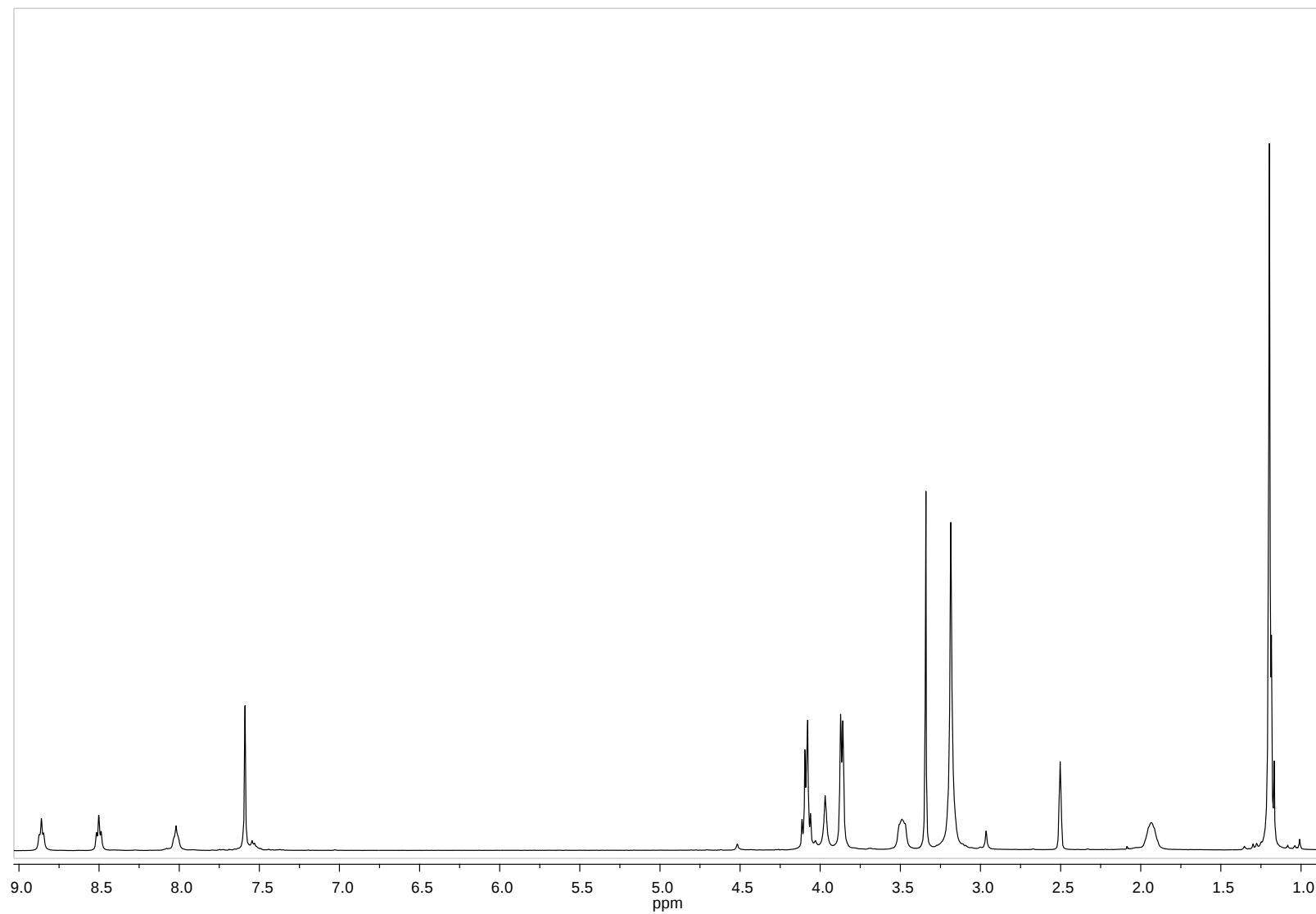


Fig. S41. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy)-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 63), DMSO- d_6 , 298 K, 400 MHz

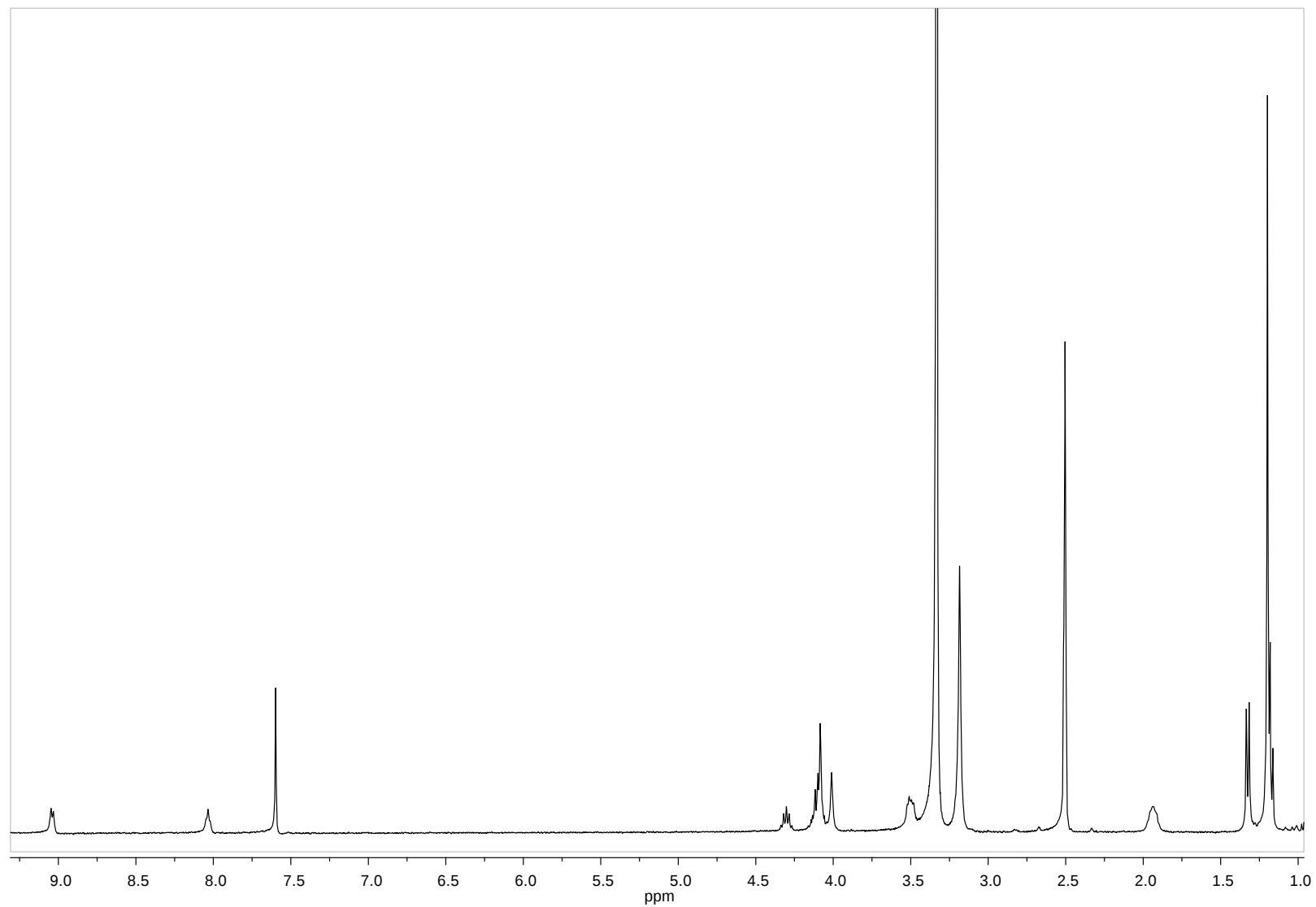


Fig. S42. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide}ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* **64**), $\text{DMSO-}d_6$, 298 K, 400 MHz

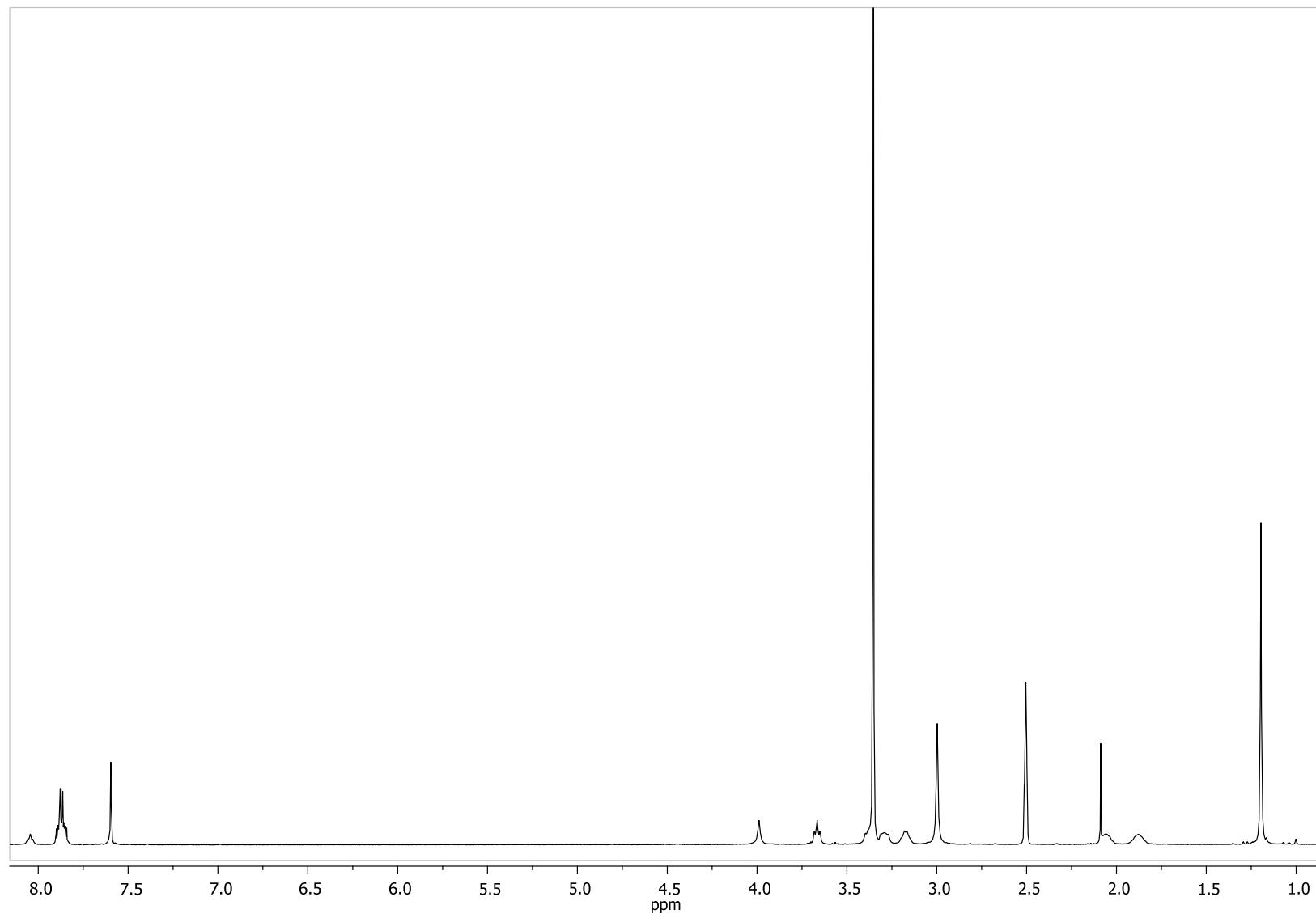


Fig. S43. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 65), DMSO-d_6 , 298 K, 400 MHz

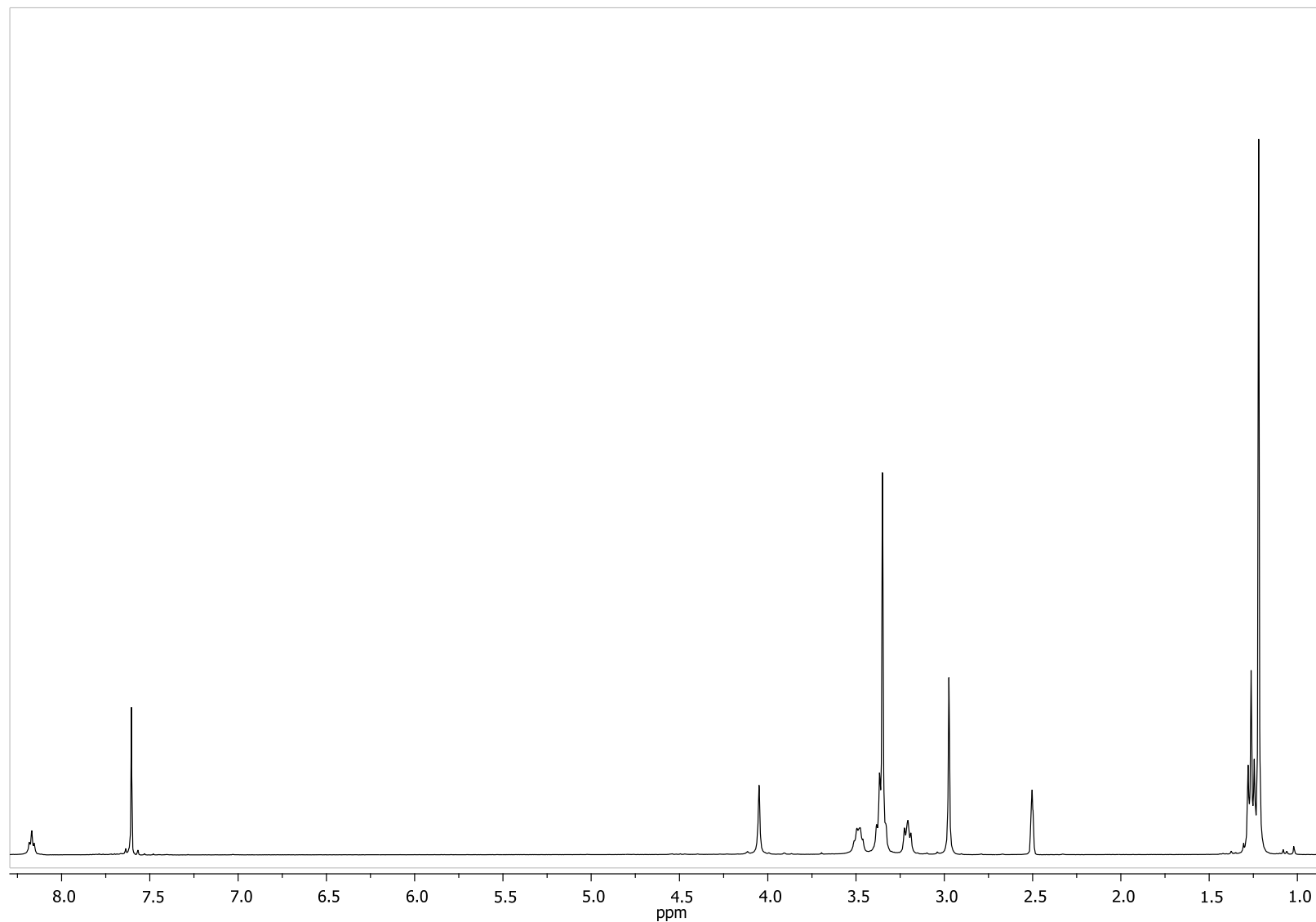


Fig. S44. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* **66**), DMSO-d_6 , 298 K, 400 MHz

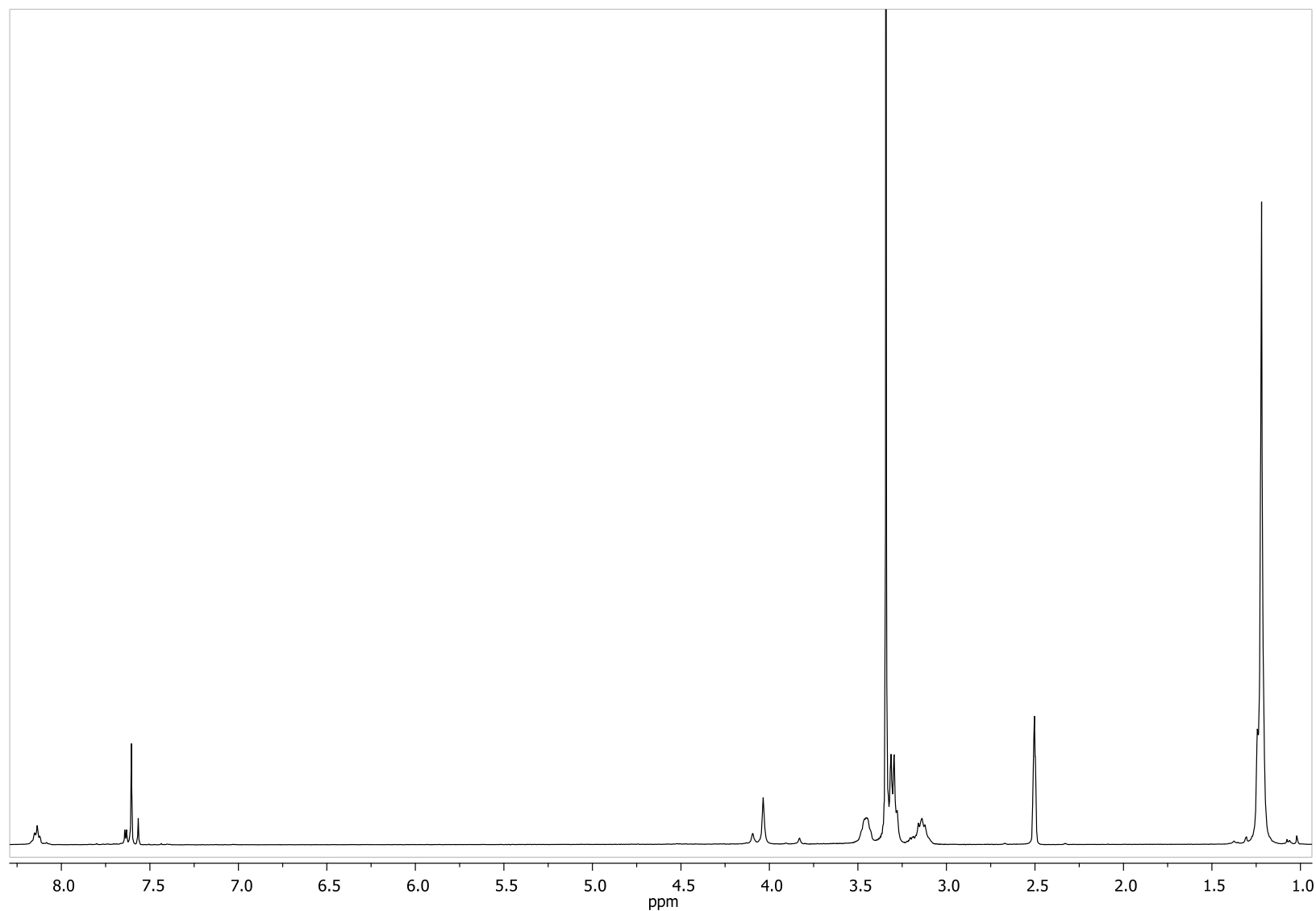


Fig. S45. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 67), DMSO- d_6 , 298 K, 400 MHz

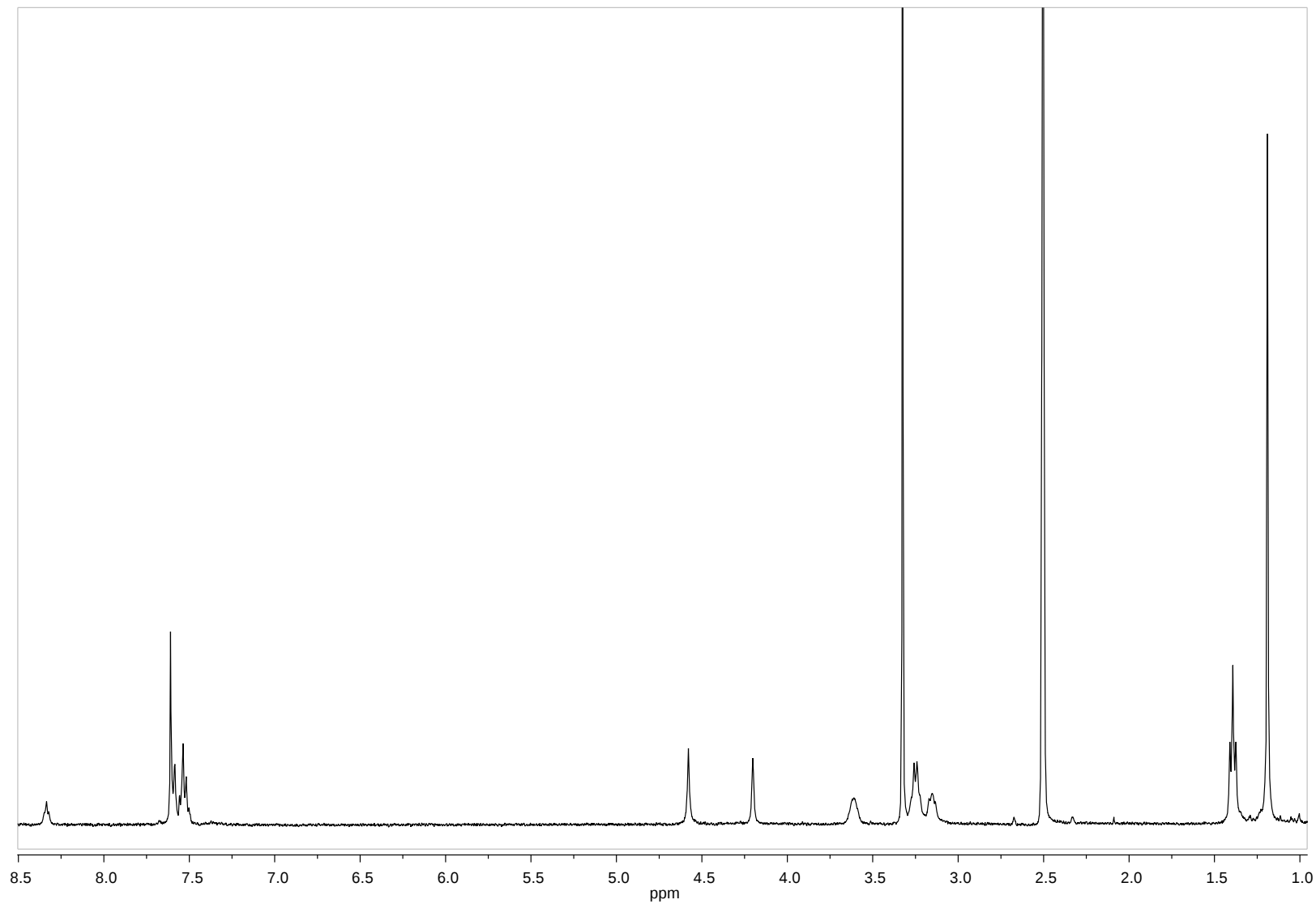


Fig. S46. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate **68**), DMSO-d_6 , 298 K, 400 MHz

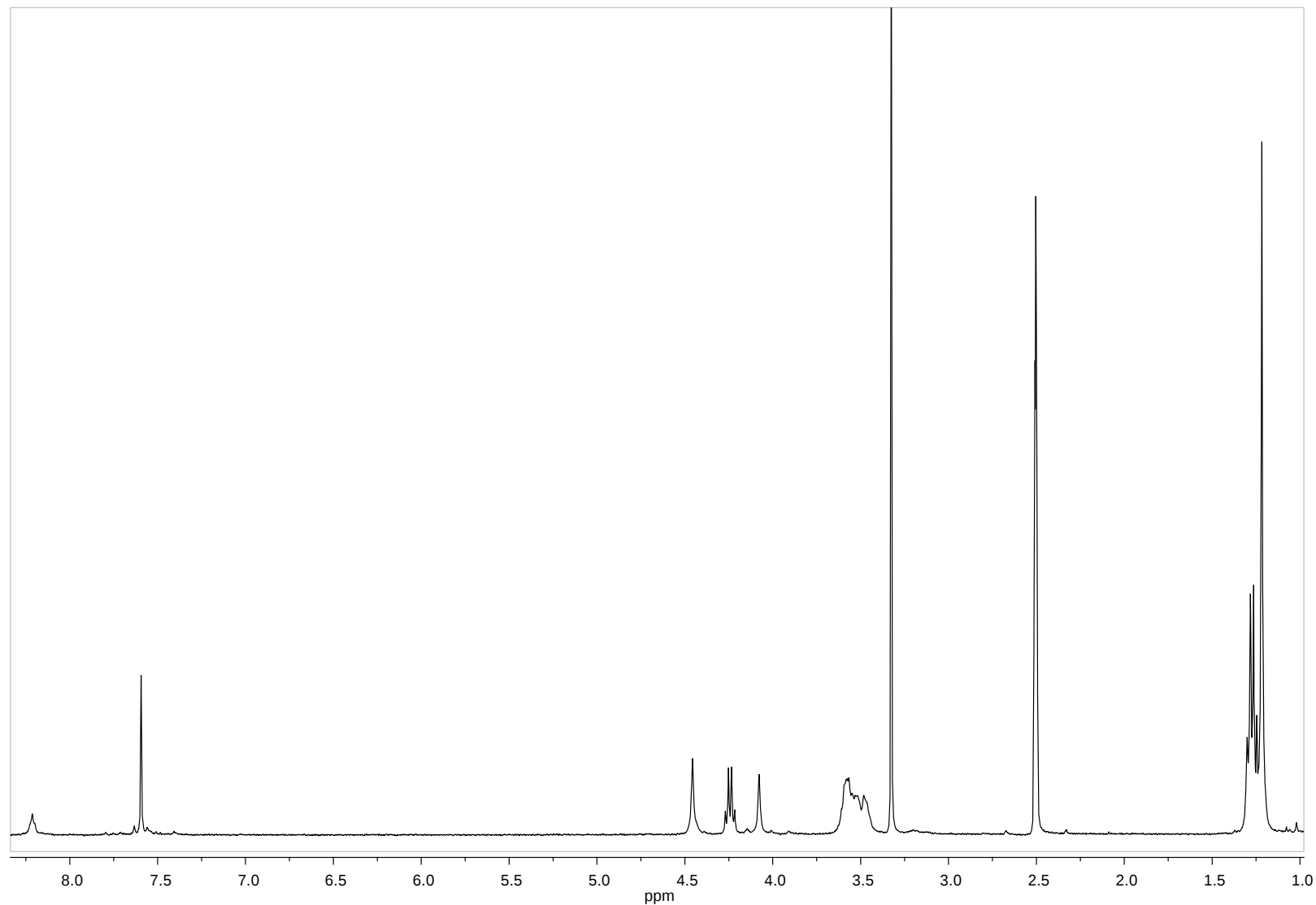


Fig. S47. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 69), $\text{DMSO}-d_6$, 298 K, 400 MHz

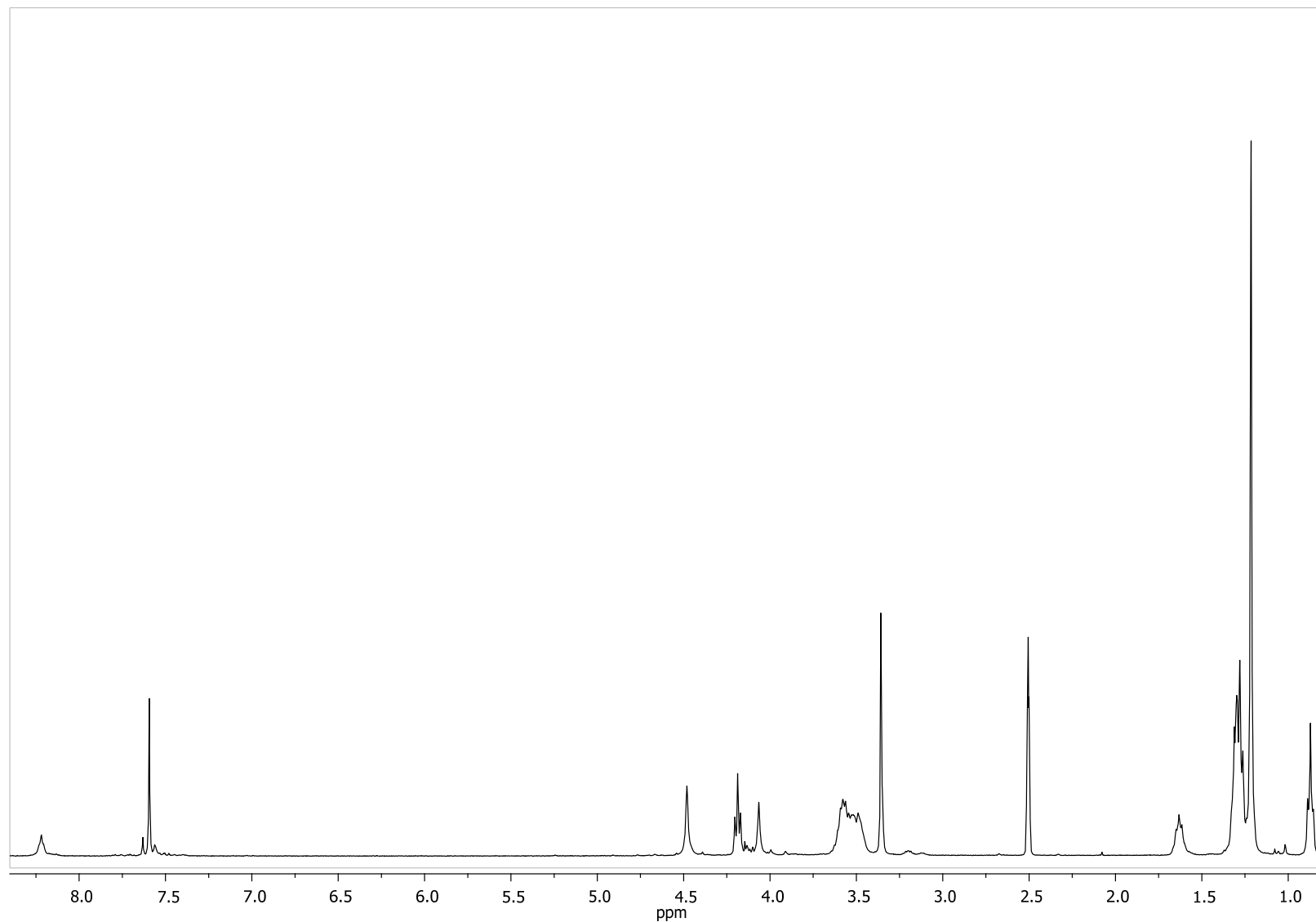


Fig. S48. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonylmethyl)amidocarbonylmethyl})ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate 70*), DMSO- d_6 , 298 K, 400 MHz

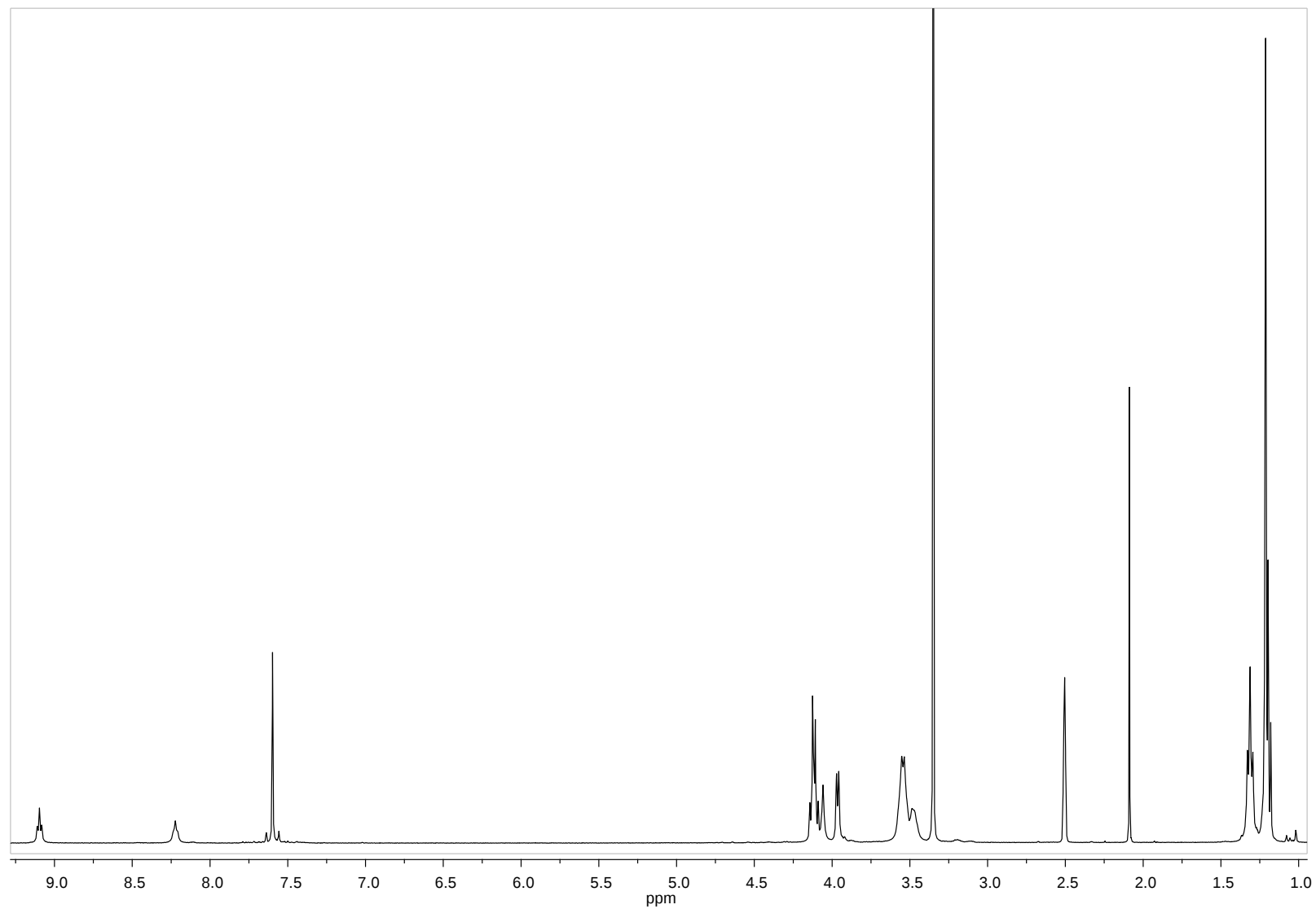


Fig. S49. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{([ethoxycarbonylmethyl]amidocarbonylmethyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate 71*), DMSO- d_6 , 298 K, 400 MHz

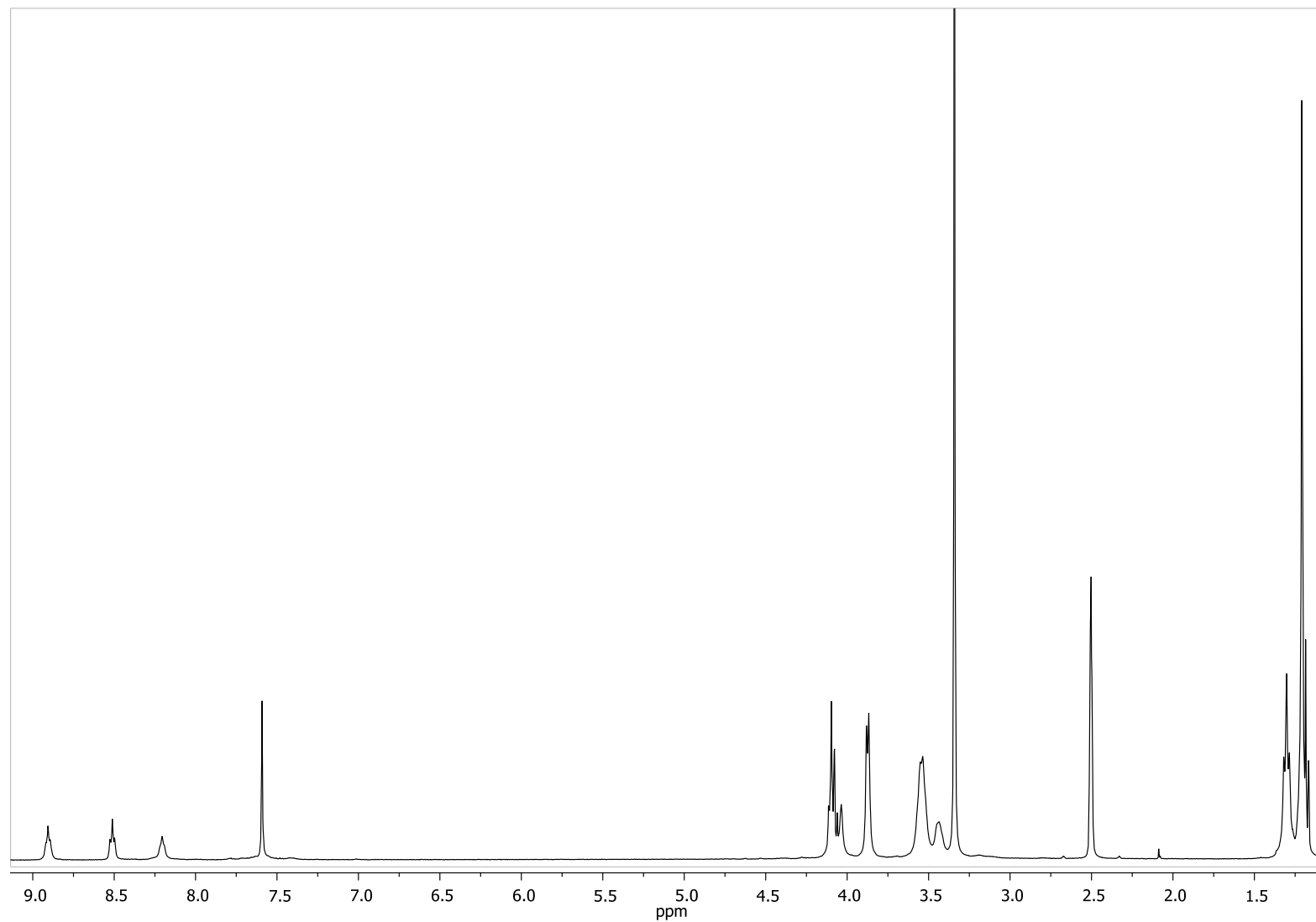


Fig. S50. ^1H NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonyl}[S-methyl]methyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate 72*), DMSO- d_6 , 298 K, 400 MHz

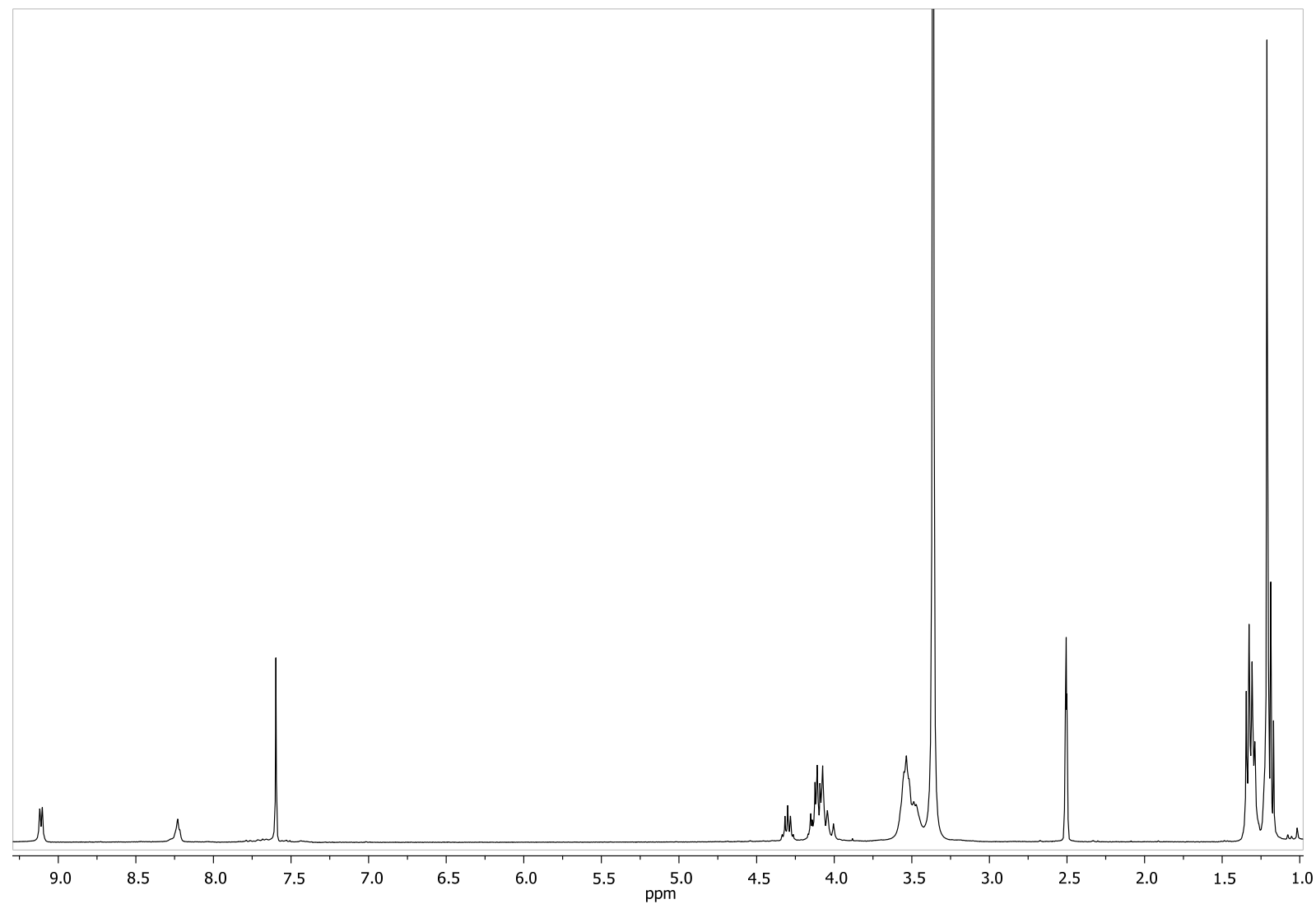


Fig. S51. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxy carbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone 9*), DMSO- d_6 , 298 K, 100 MHz

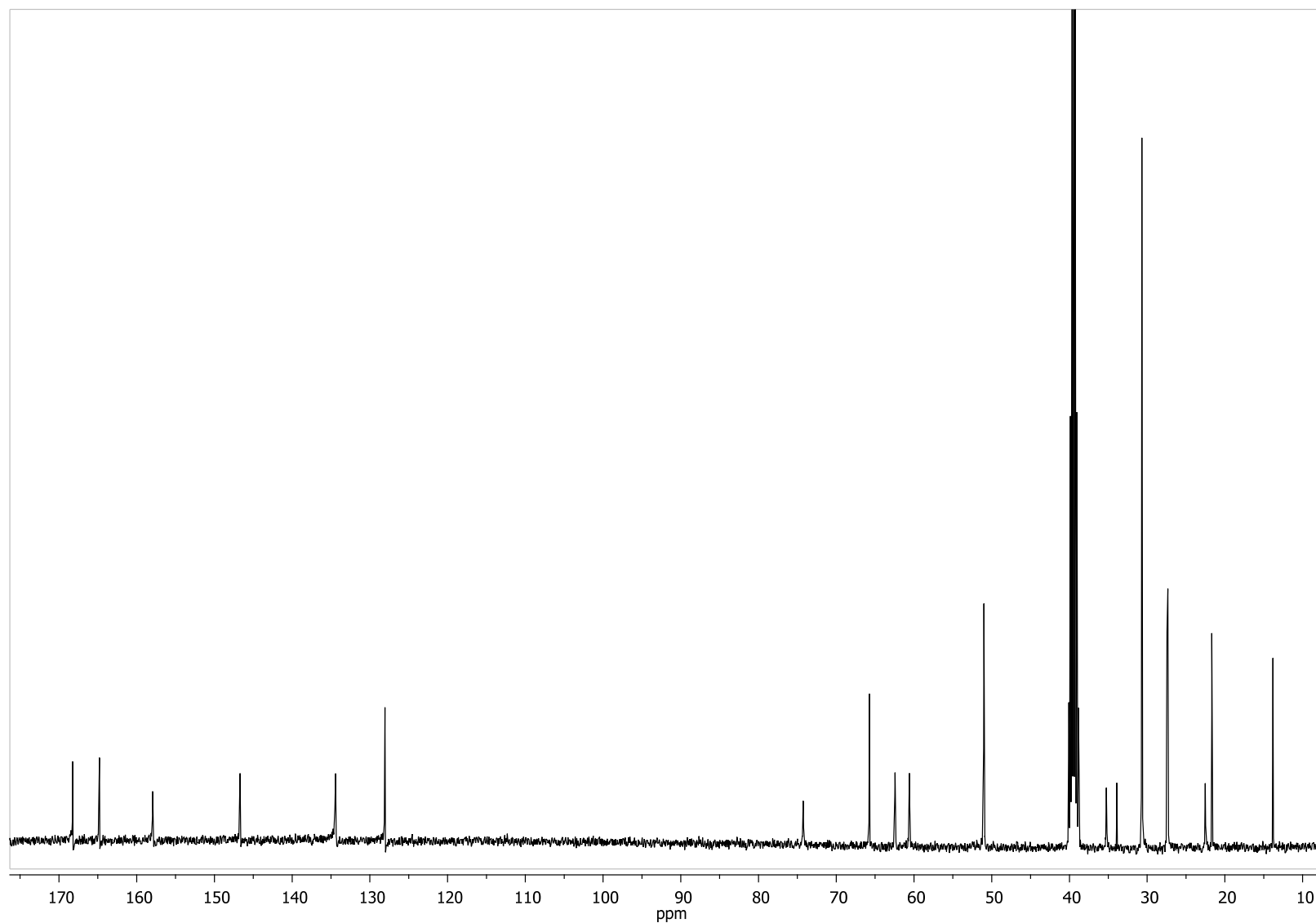


Fig. S52. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (*cone* 15), CDCl_3 , 298 K, 100 MHz

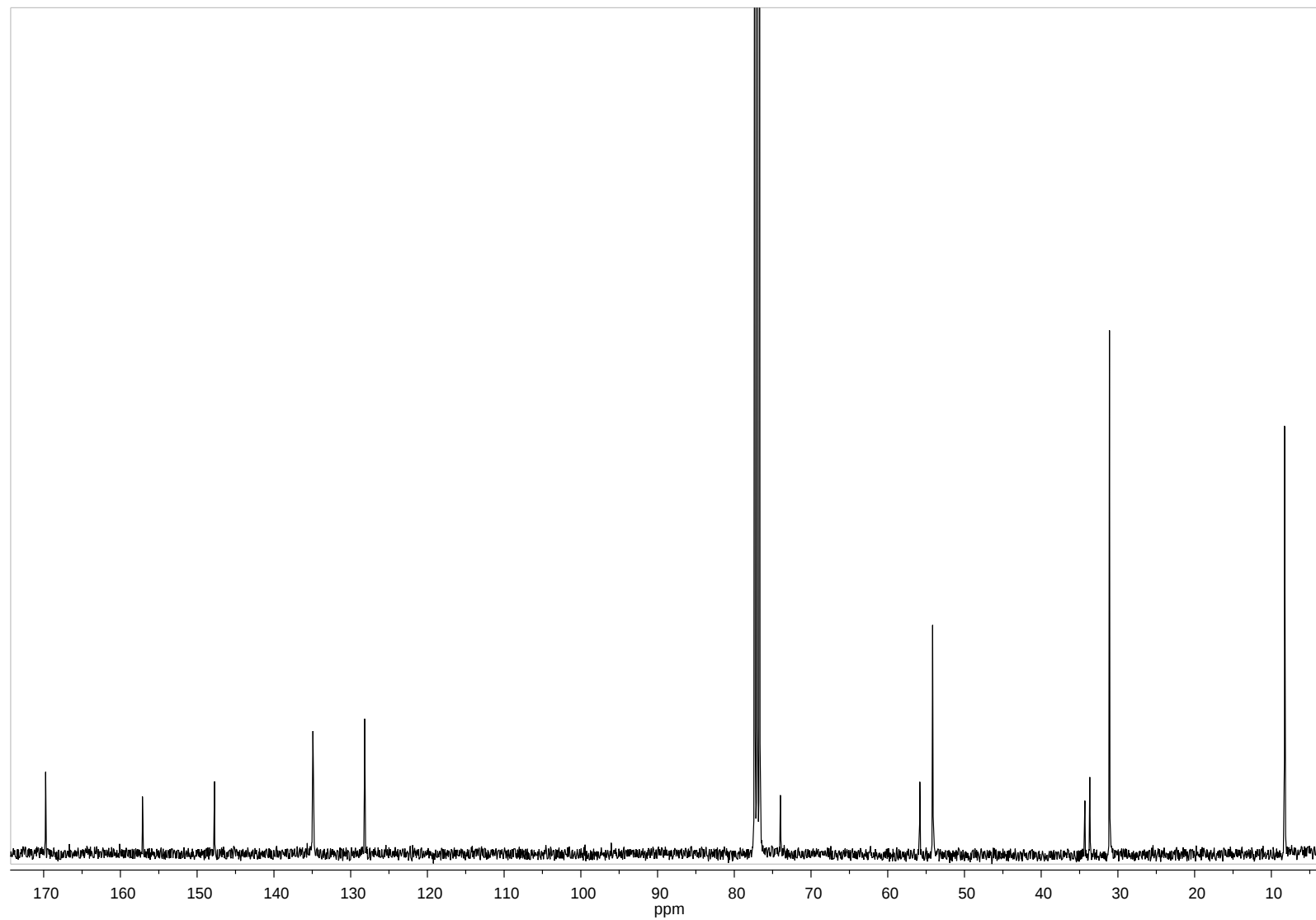


Fig. S53. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 16), CDCl_3 , 298 K, 100 MHz

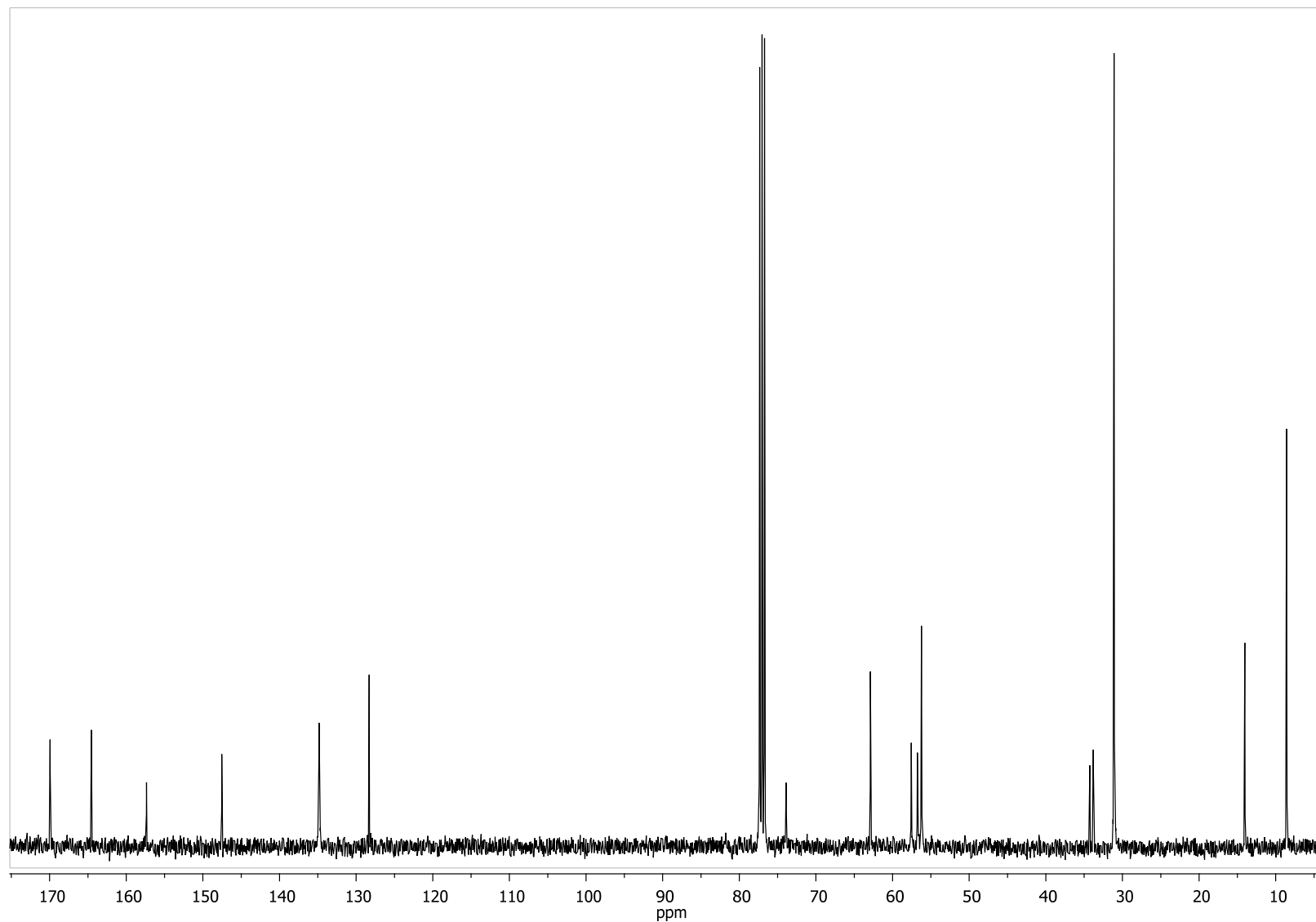


Fig. S54. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone 17*), CDCl_3 , 298 K, 100 MHz

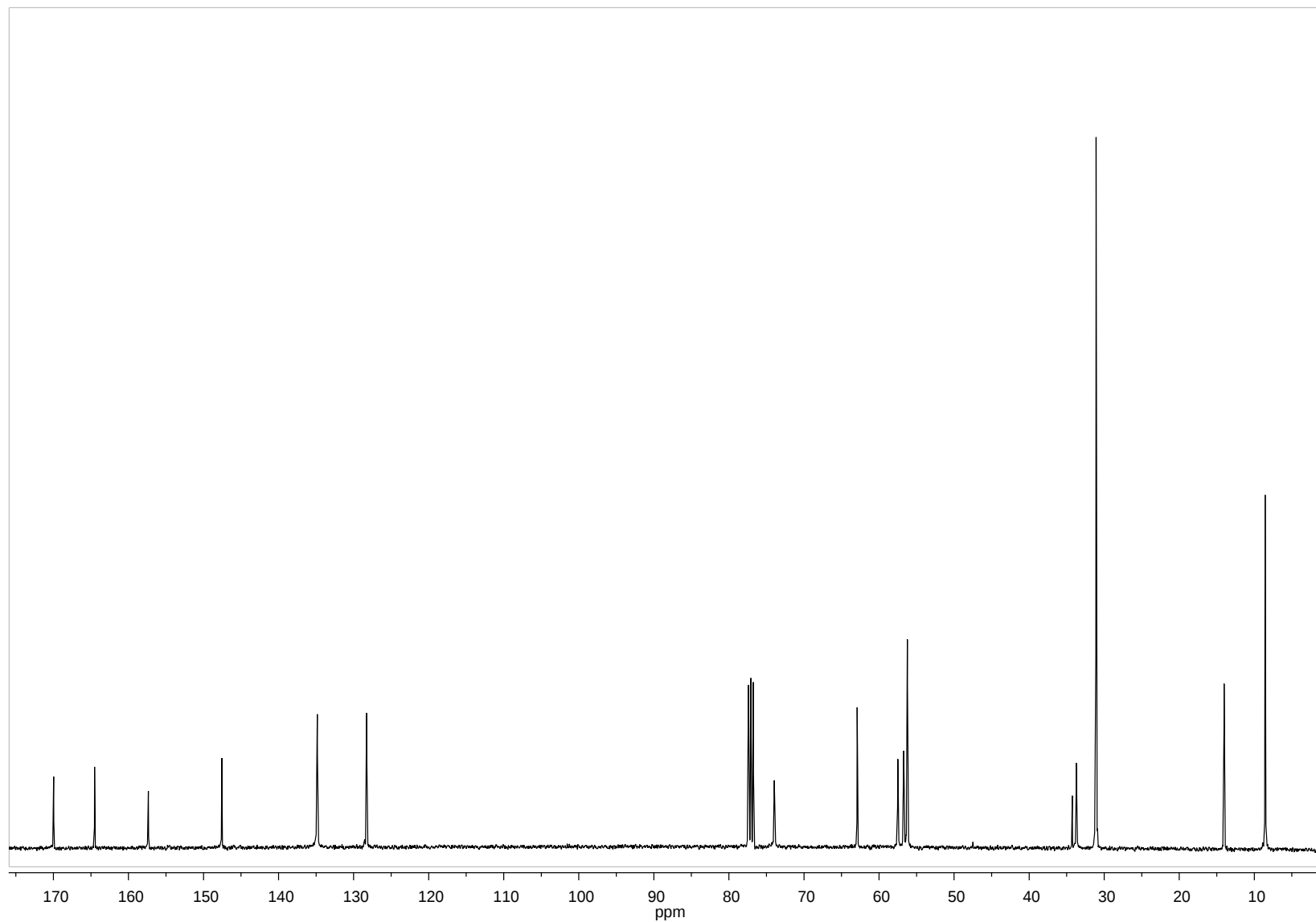


Fig. S55. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone 18*), CDCl_3 , 298 K, 100 MHz

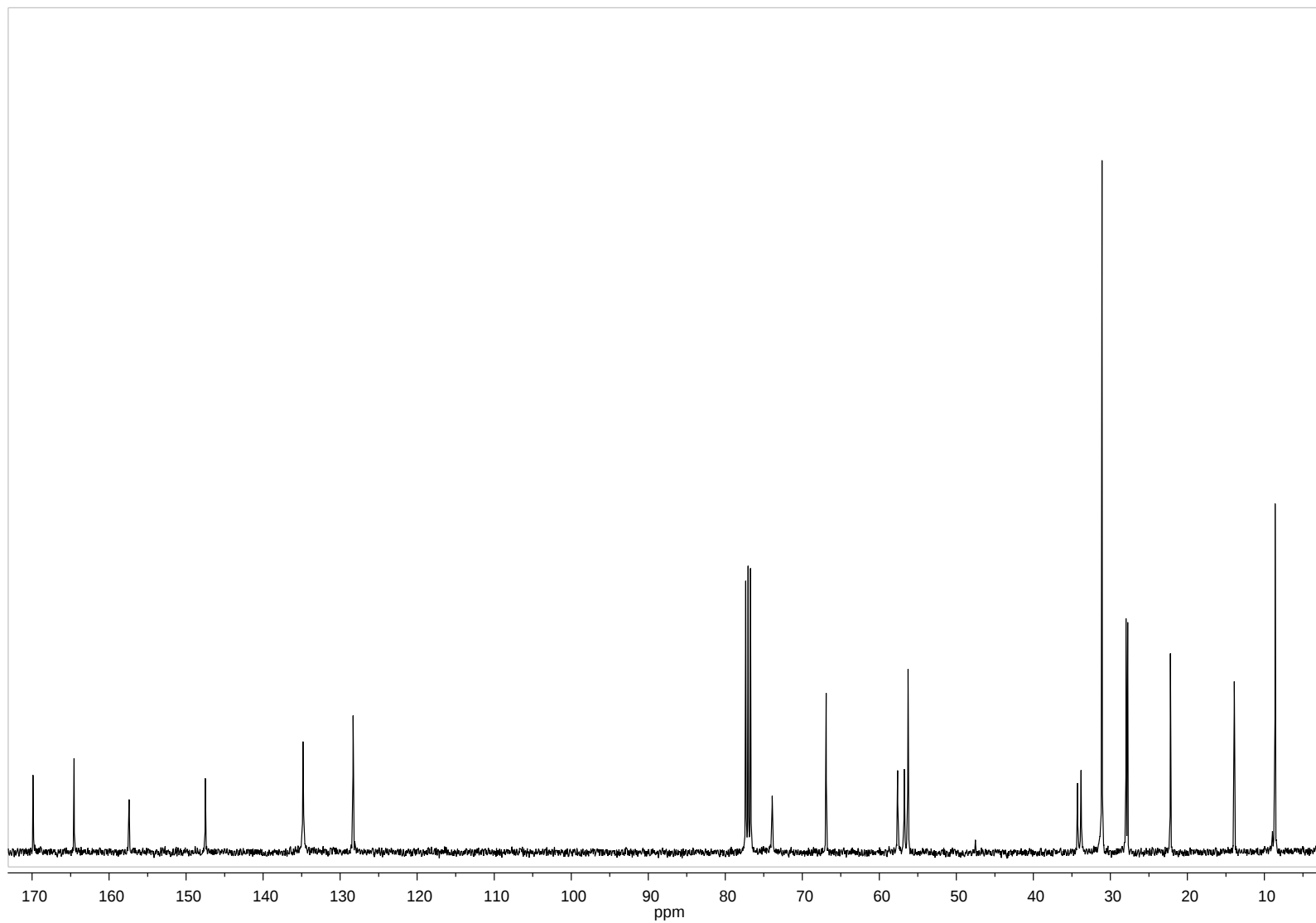


Fig. S56. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-*alternate* 22), DMSO- d_6 , 298 K, 100 MHz

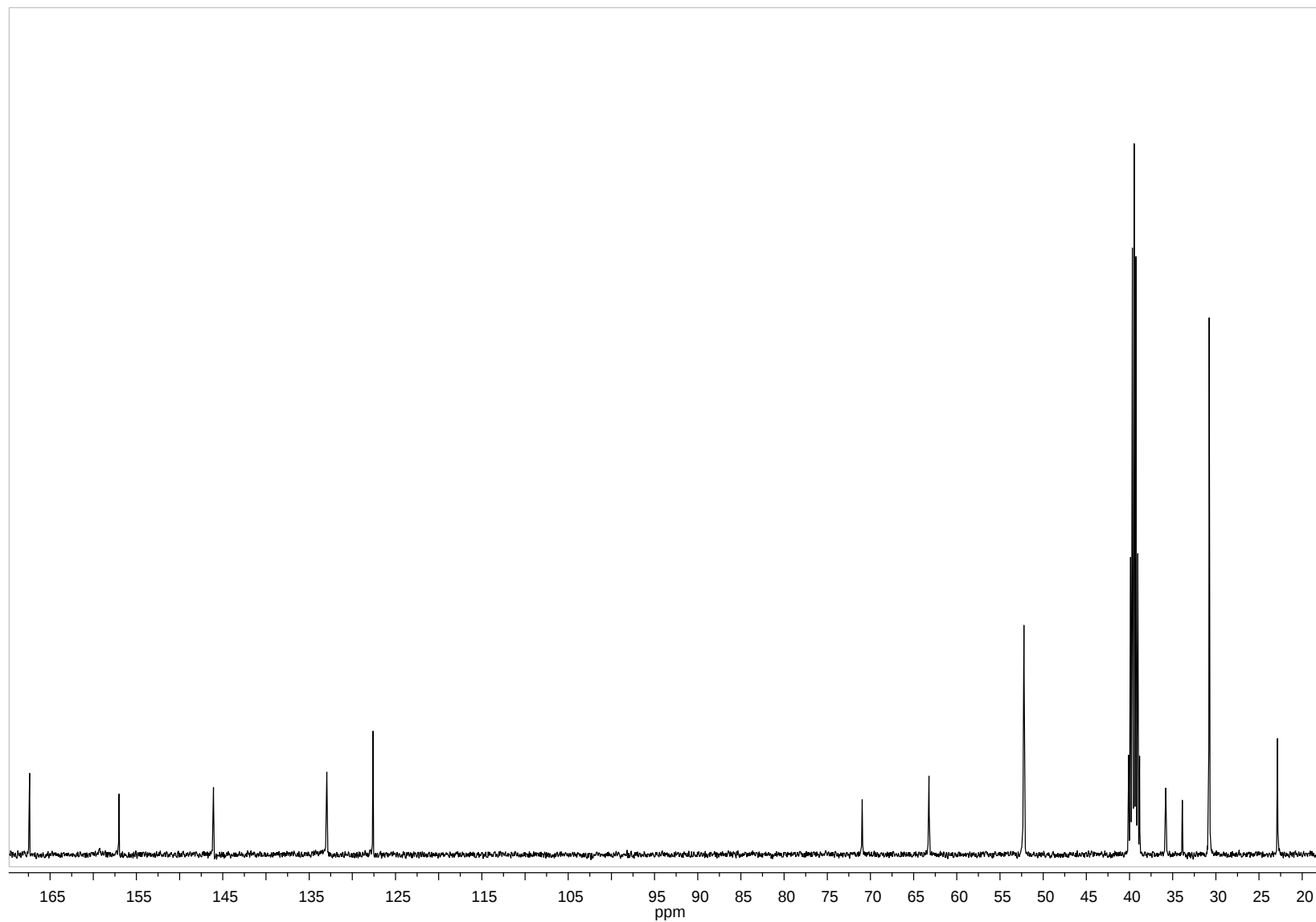


Fig. S57. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-alternate 23), DMSO- d_6 , 298 K, 100 MHz

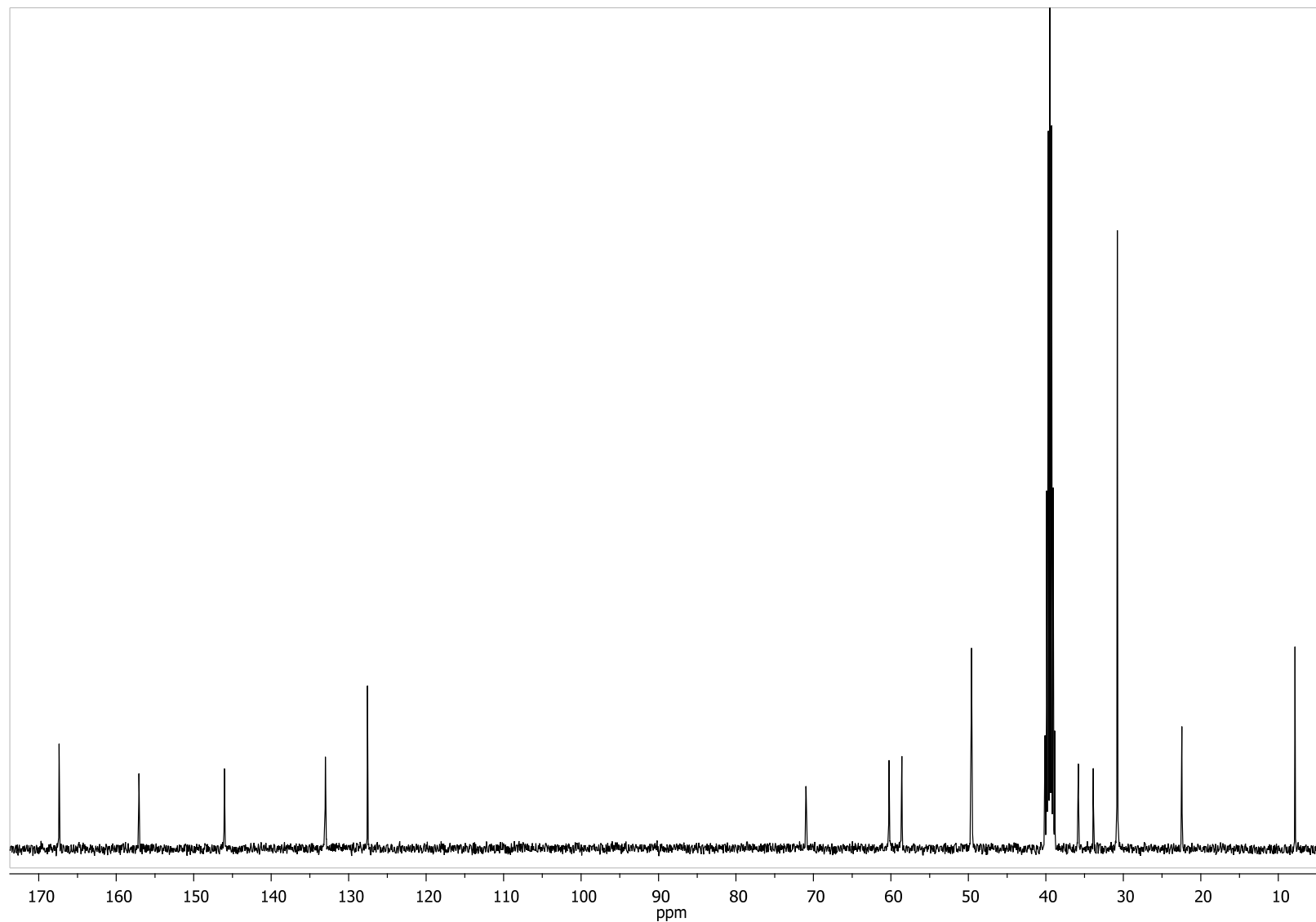


Fig. S58. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 24), DMSO- d_6 , 298 K, 100 MHz

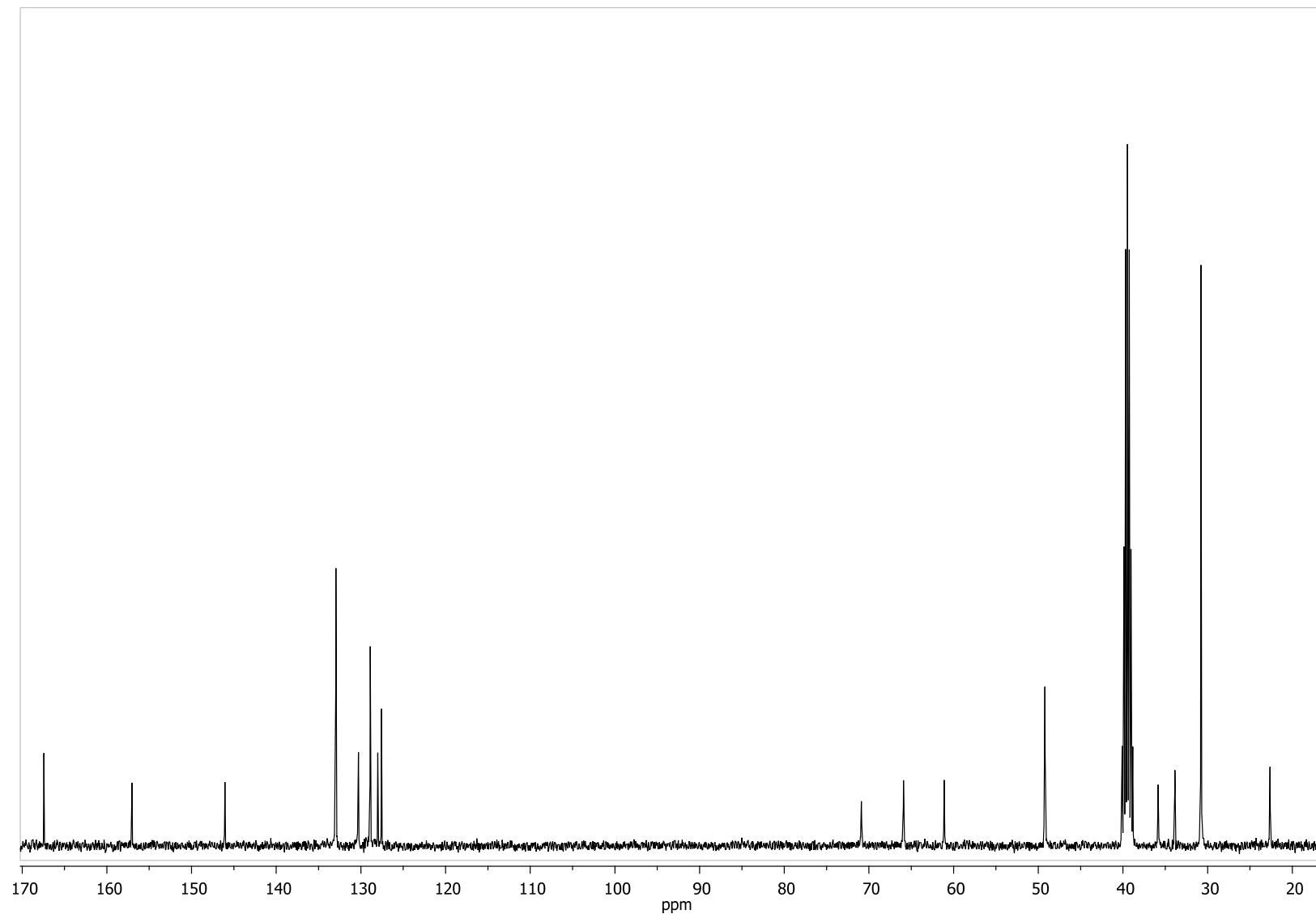


Fig. S59. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*1,3-alternate* 25), DMSO- d_6 , 298 K, 100 MHz

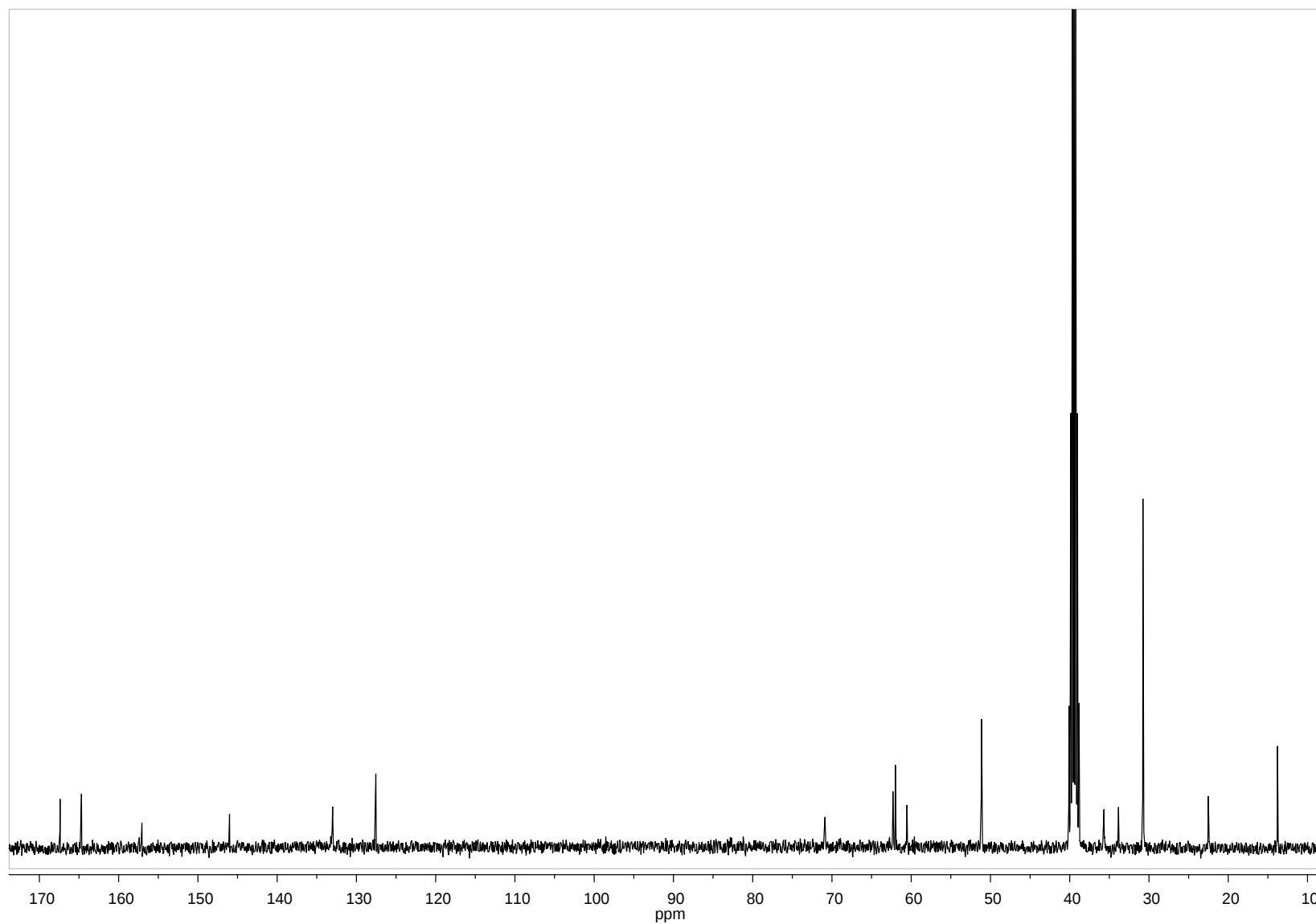


Fig. S60. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxy carbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*1,3-alternate* 26), DMSO- d_6 , 298 K, 100 MHz

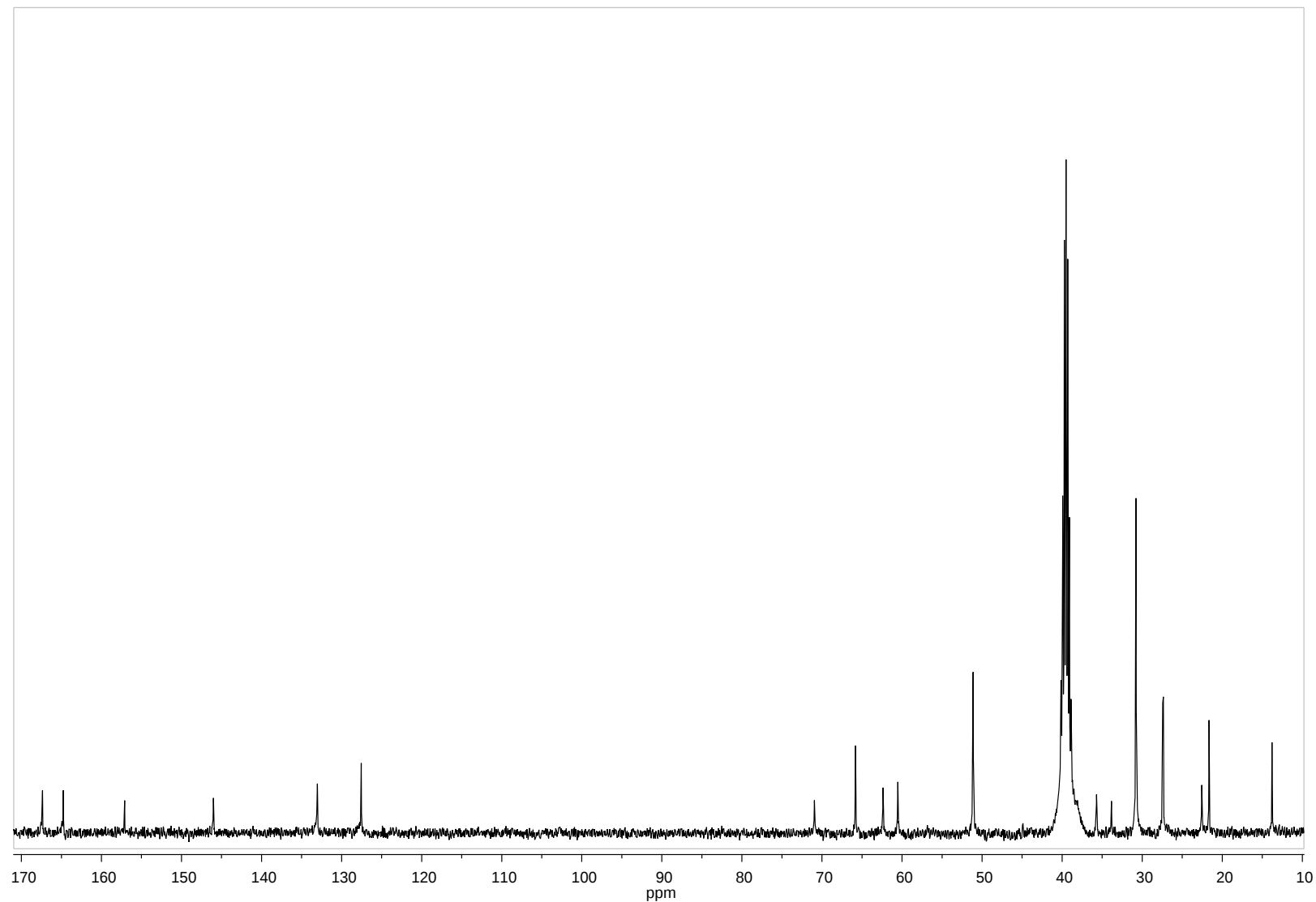


Fig. S61. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide}-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*1,3-alternate* **30**), DMSO- d_6 , 298 K, 100 MHz

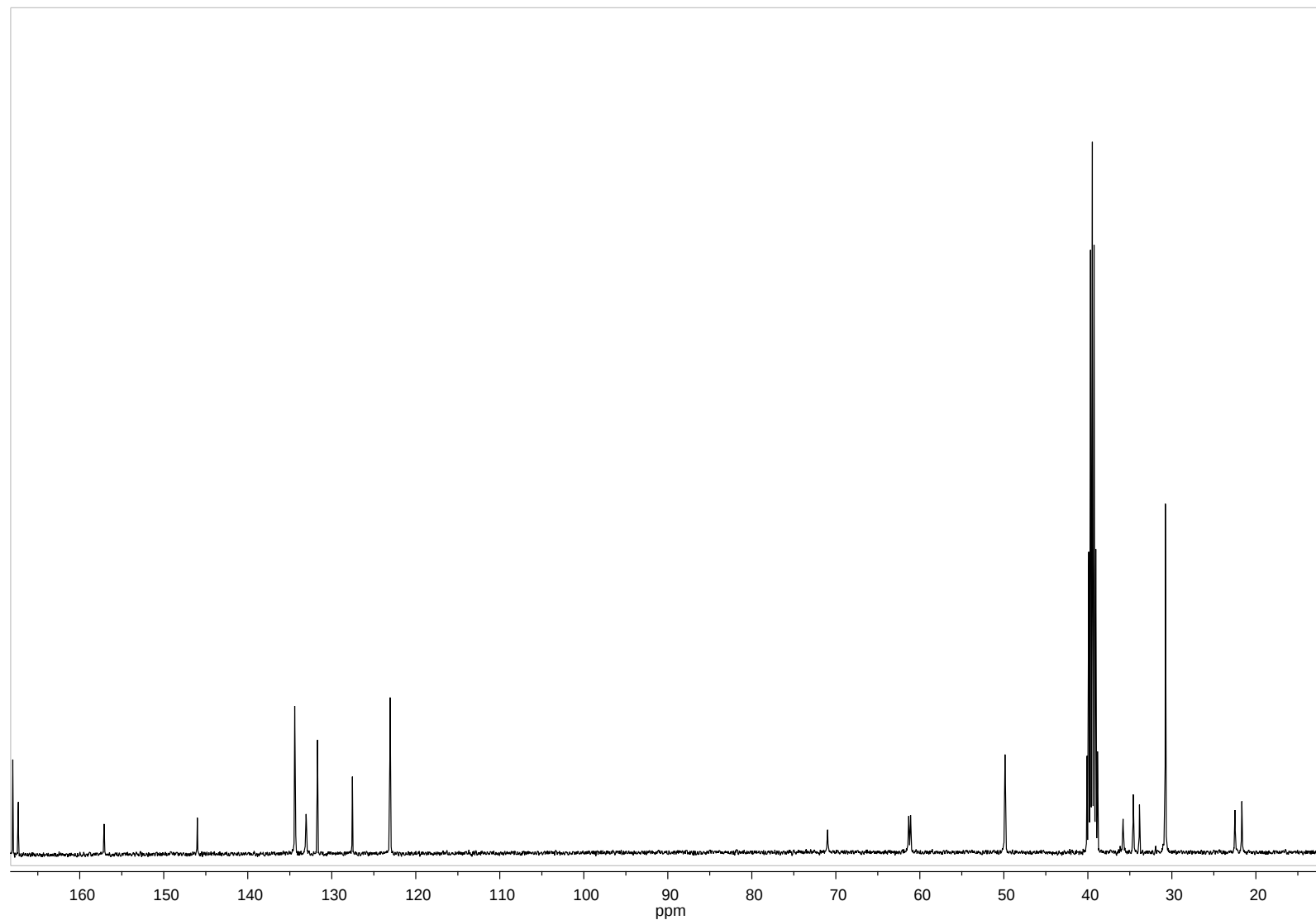


Fig. S62. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-alternate 31), DMSO- d_6 , 298 K, 100 MHz

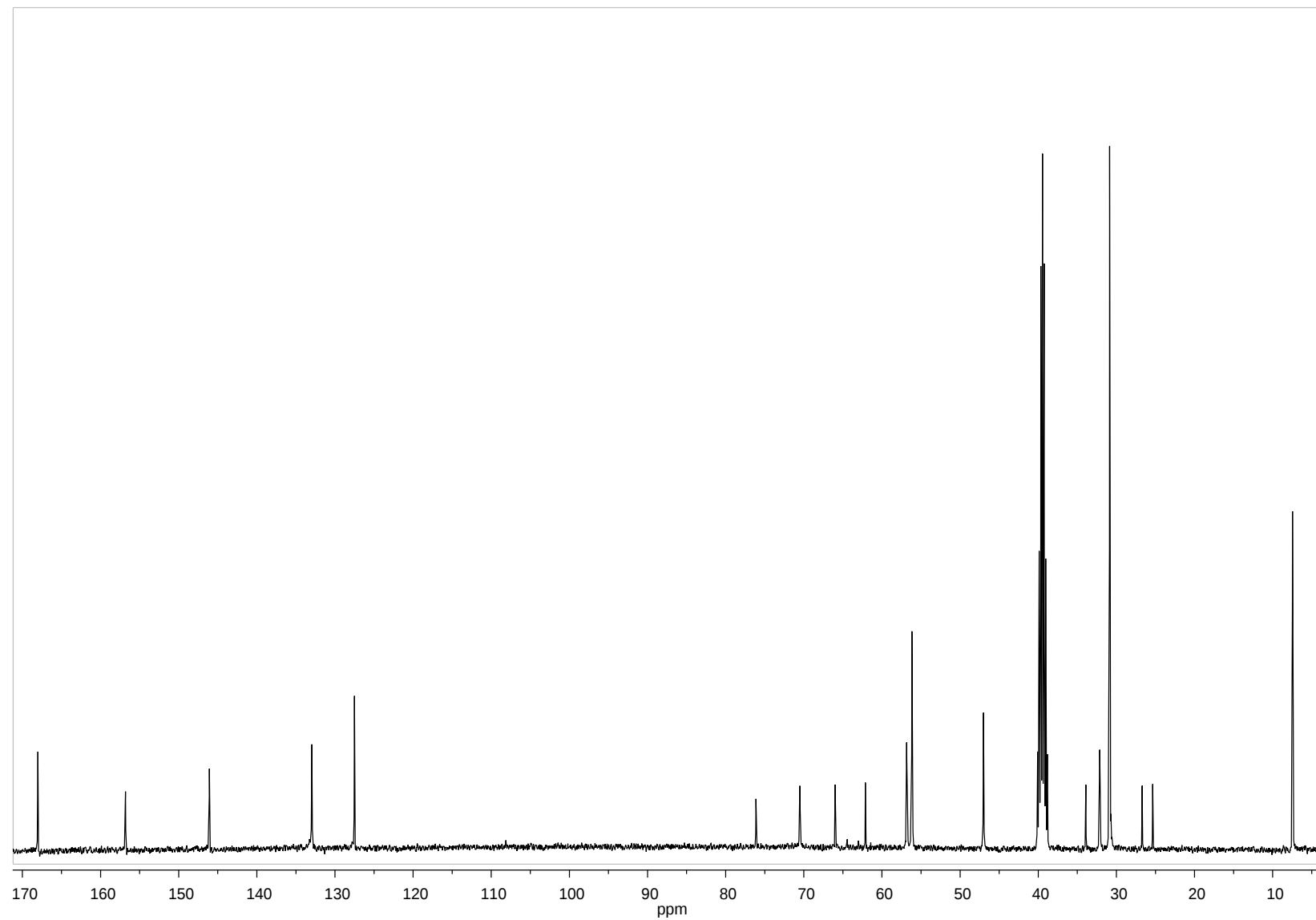


Fig. S63. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-alternate **32**), DMSO- d_6 , 298 K, 100 MHz

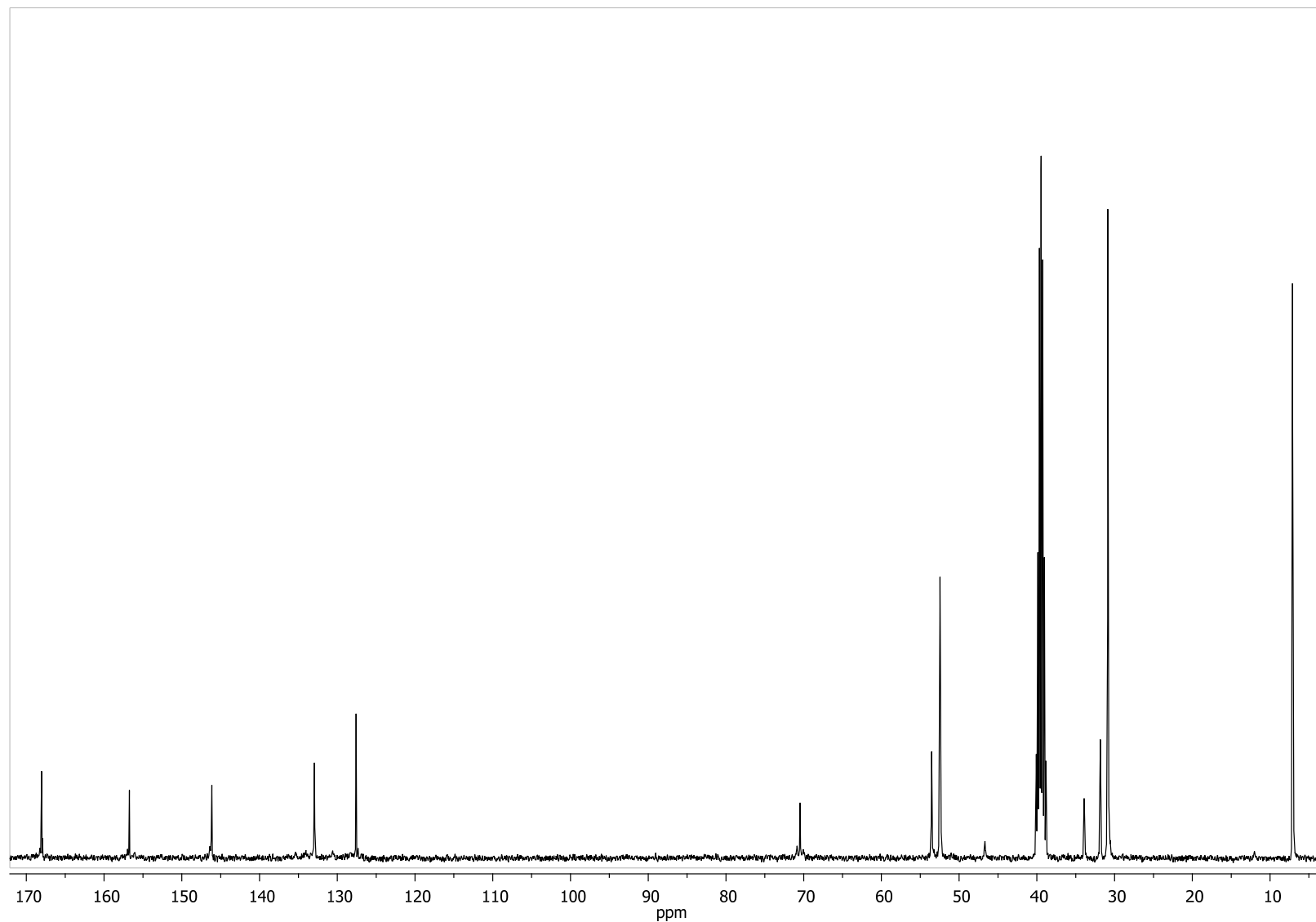


Fig. S64. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 33), DMSO- d_6 , 298 K, 100 MHz

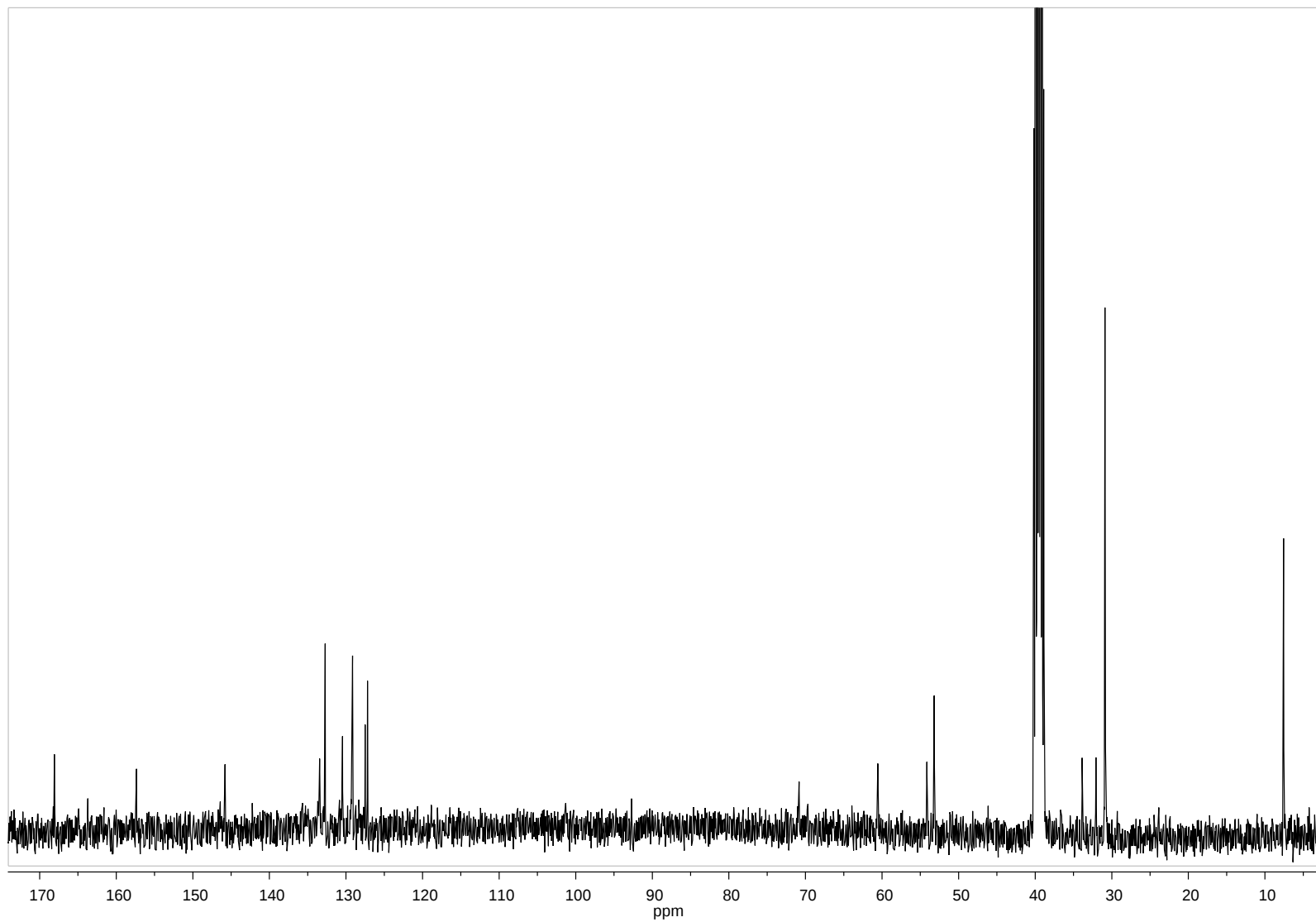


Fig. S65. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*1,3-alternate* 34), DMSO- d_6 , 298 K, 100 MHz

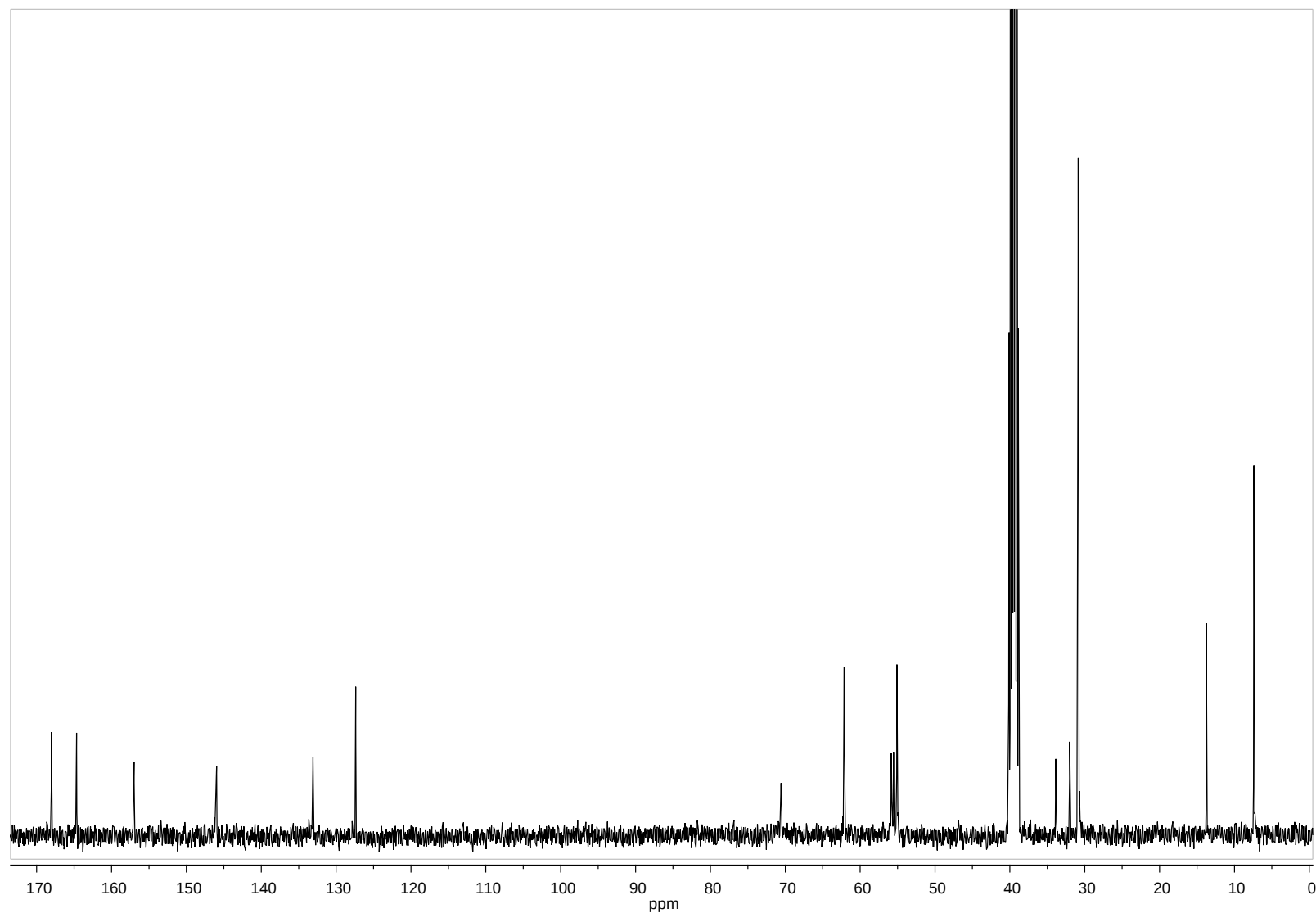


Fig. S66. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*1,3-alternate* 35), DMSO- d_6 , 298 K, 100 MHz

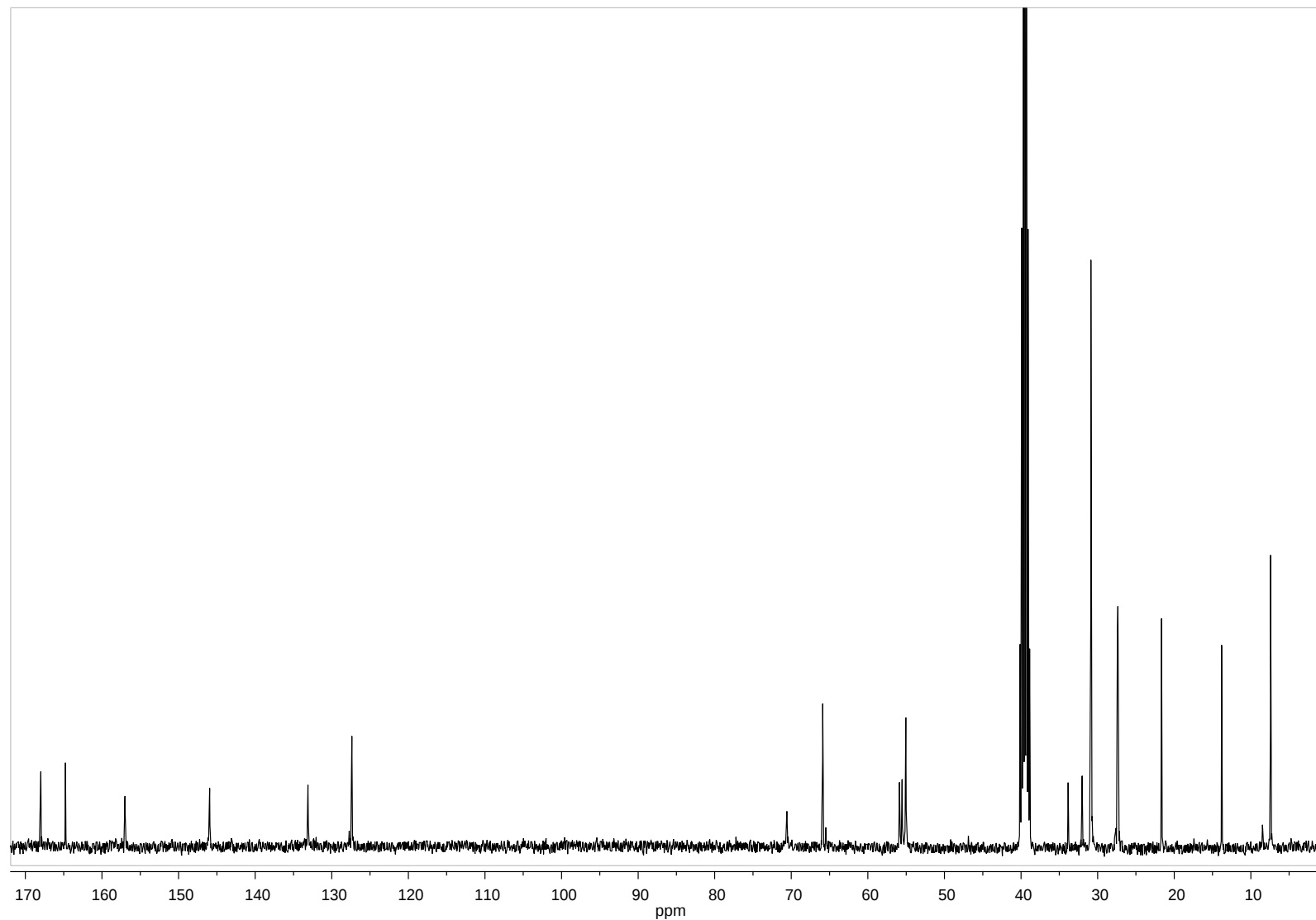


Fig. S67. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 39), DMSO- d_6 , 298 K, 100 MHz

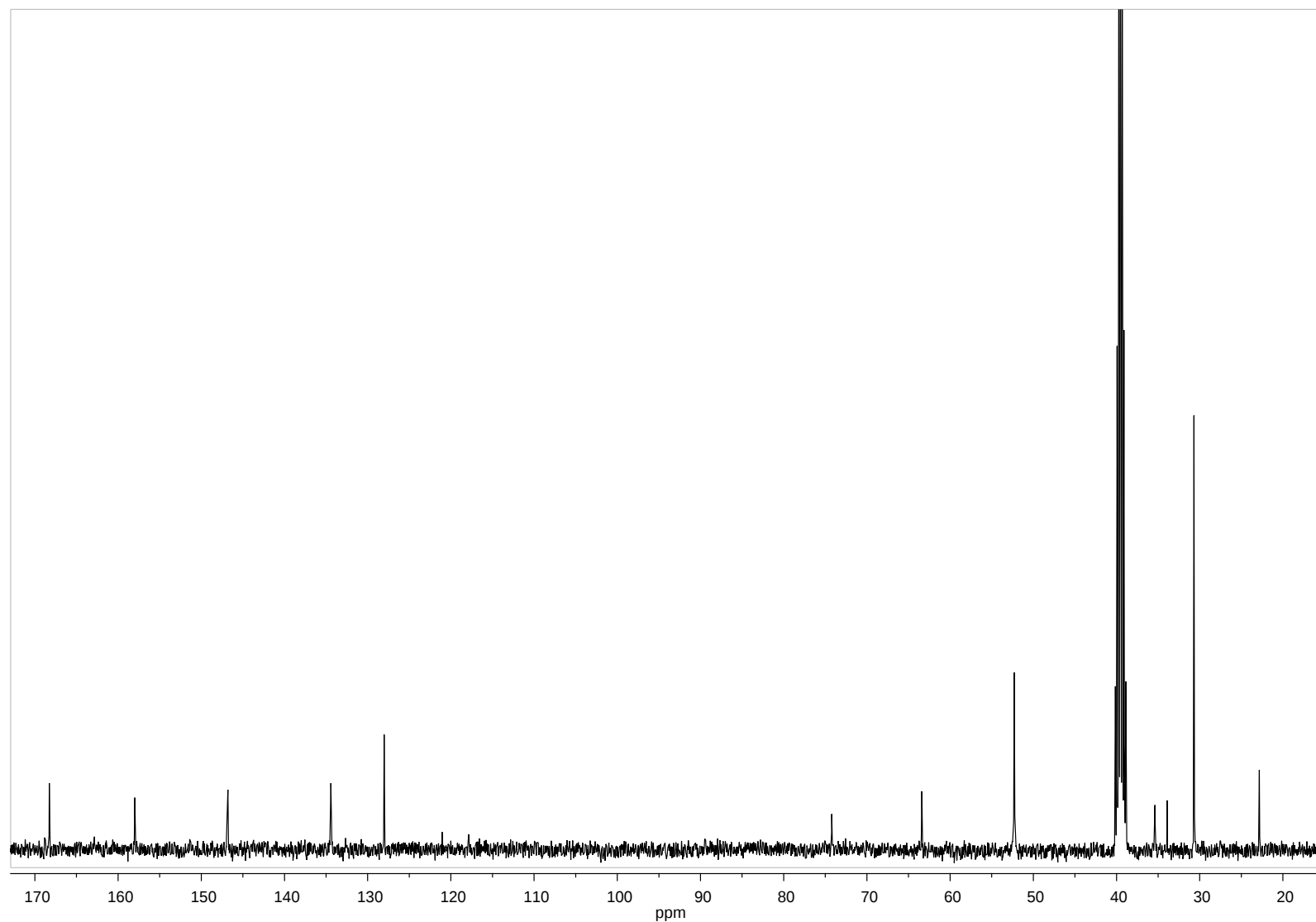


Fig. S68. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 40*), DMSO- d_6 , 298 K, 100 MHz

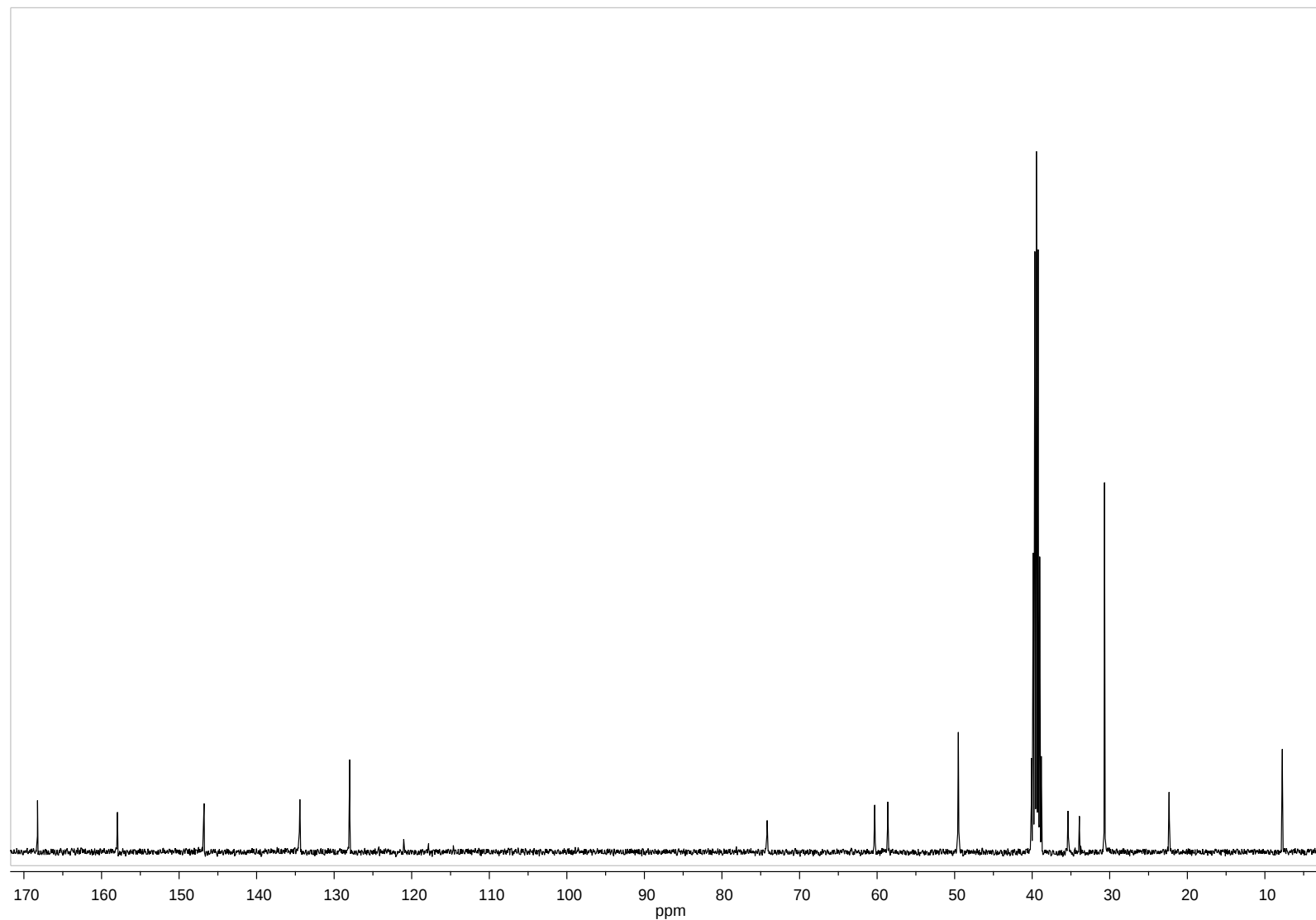


Fig. S69. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 41*), CDCl_3 , 298 K, 100 MHz

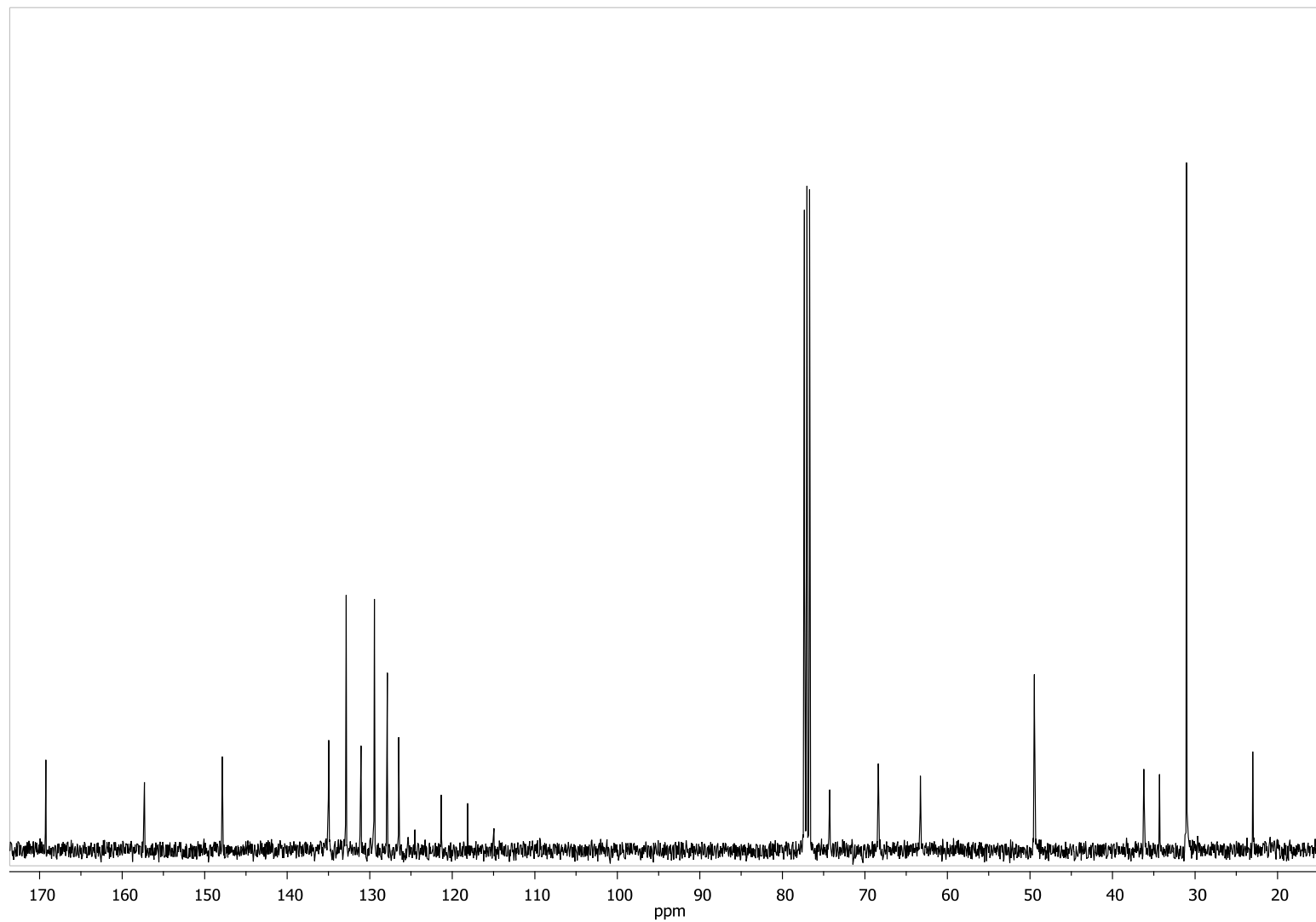


Fig. S70. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 42), CDCl_3 , 298 K, 100 MHz

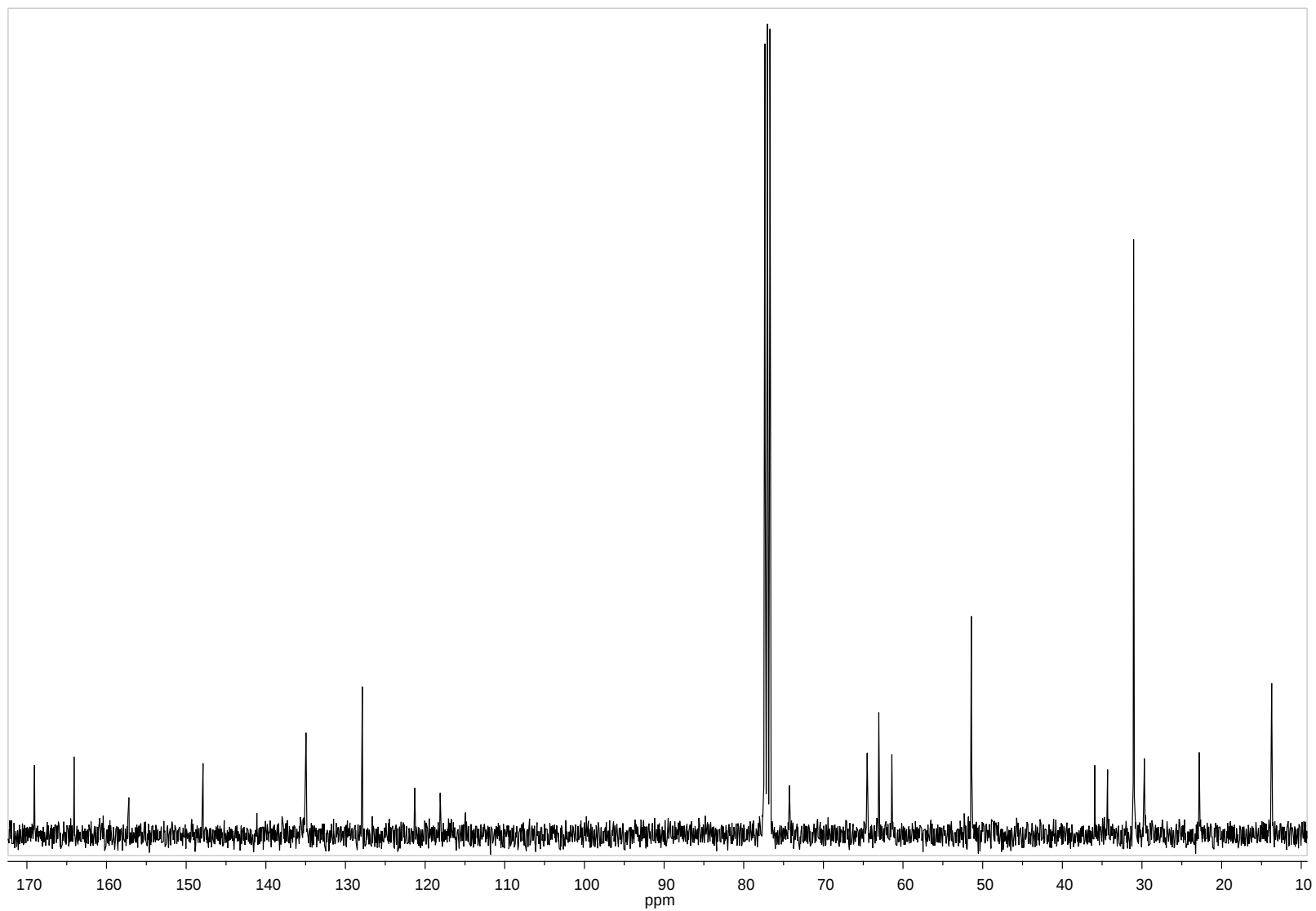


Fig. S71. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxy carbonylmethyl) ammoniumpropyl) carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 43), DMSO- d_6 , 298 K, 100 MHz

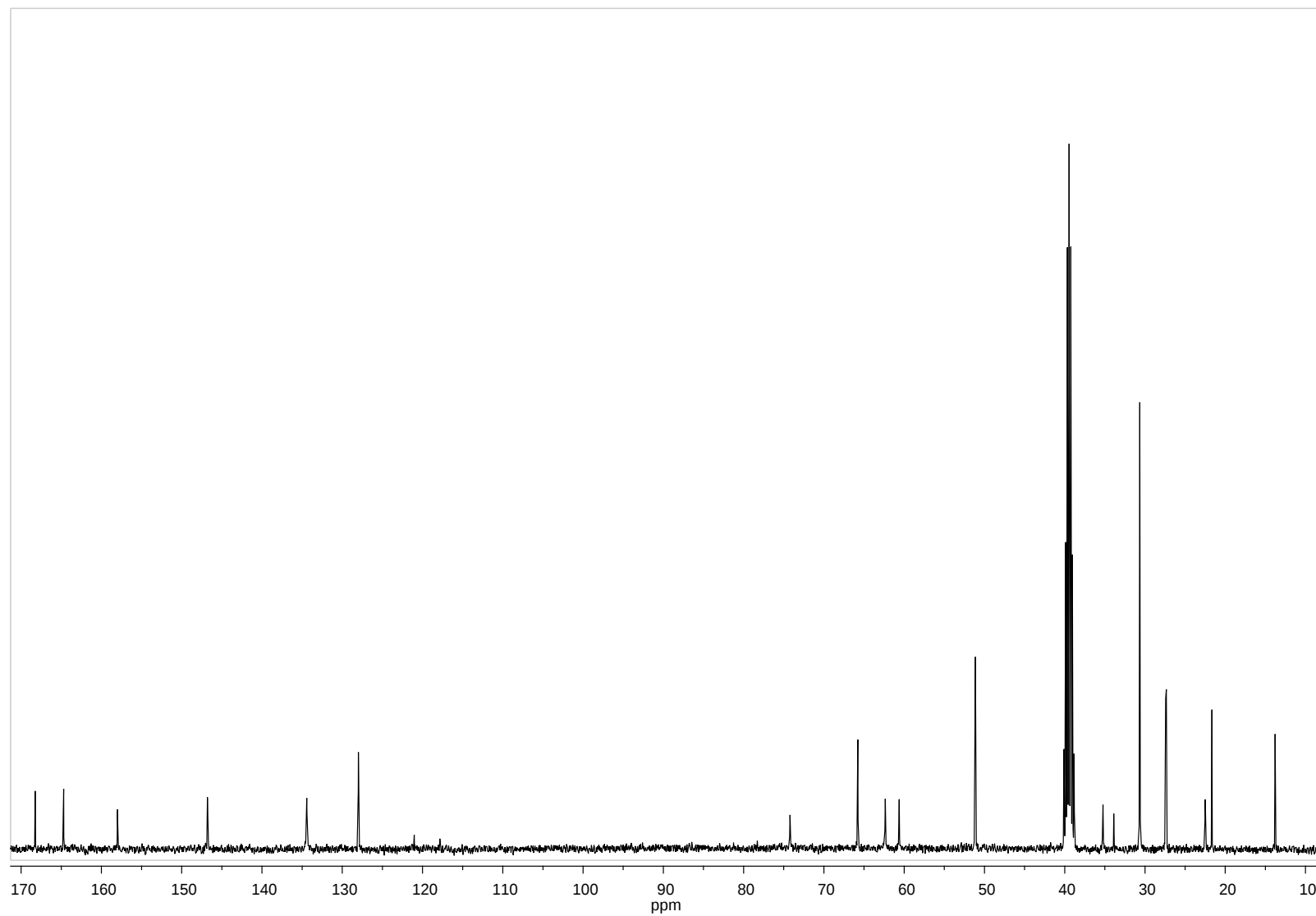


Fig. S72. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonylmethyl)amidocarbonylmethyl}-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 44*), CDCl_3 , 298 K, 100 MHz

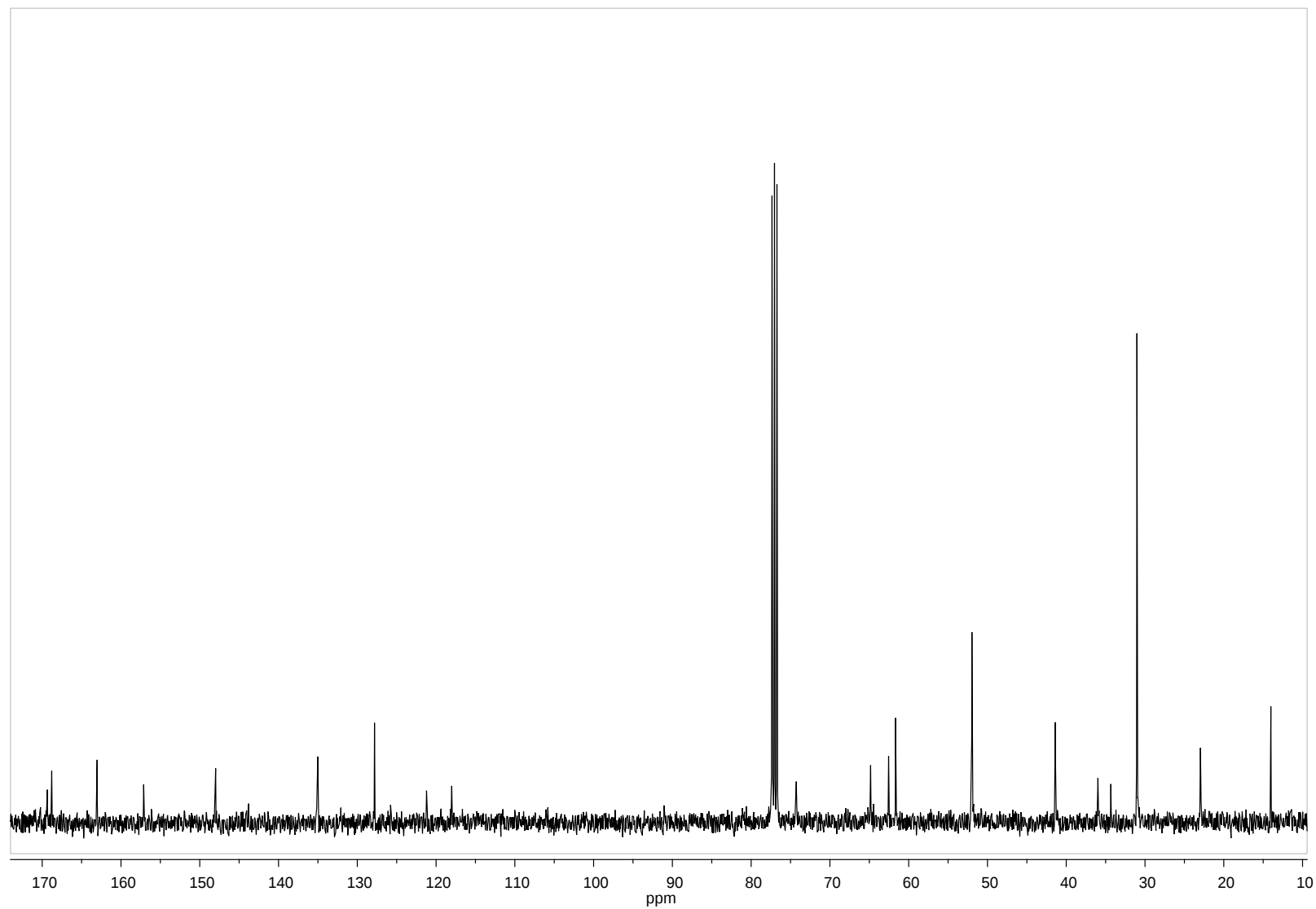


Fig. S73. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{[ethoxycarbonylmethyl]-amidocarbonylmethyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 45), DMSO- d_6 , 298 K, 100 MHz

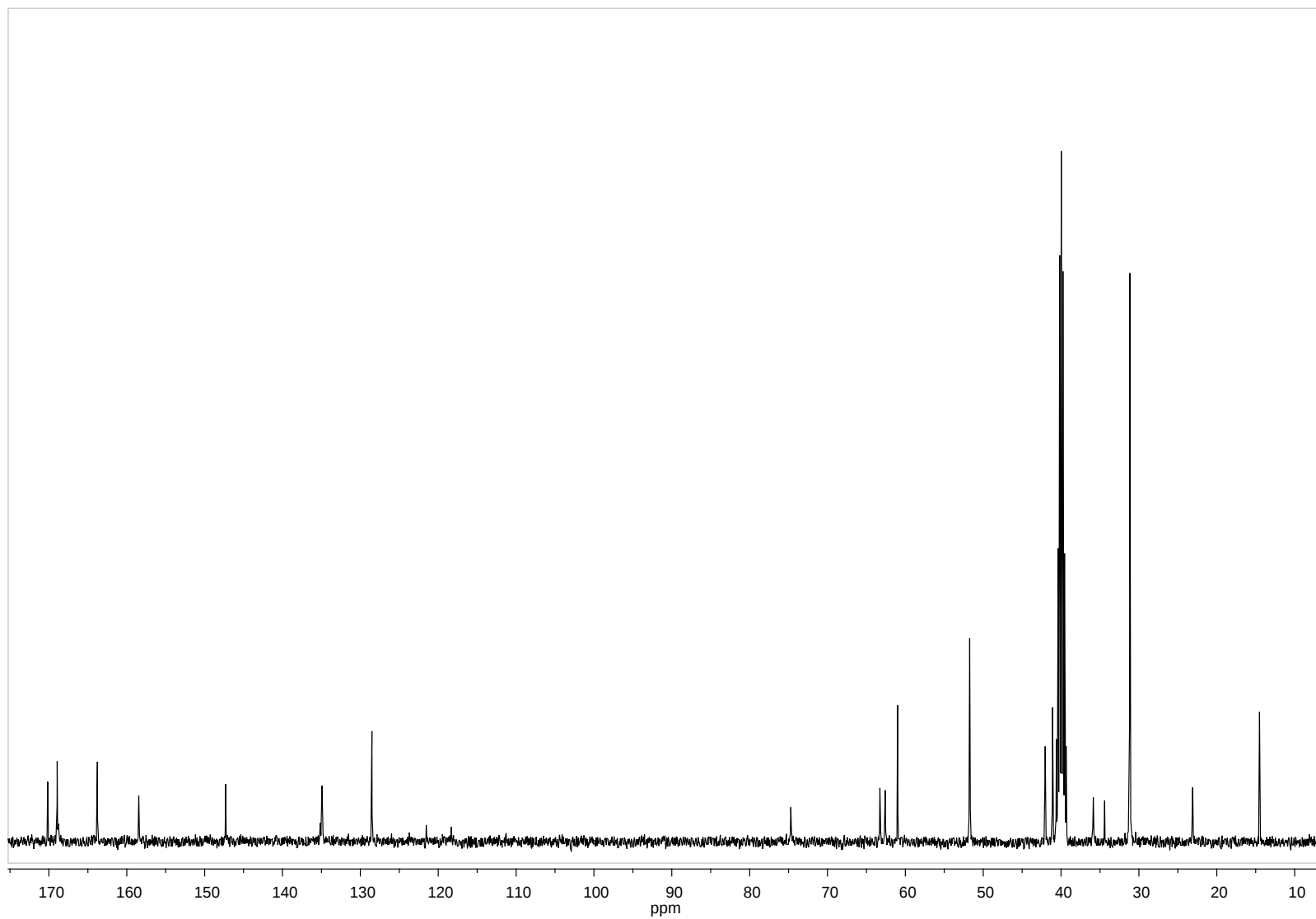


Fig. S74. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 46*), DMSO- d_6 , 298 K, 100 MHz

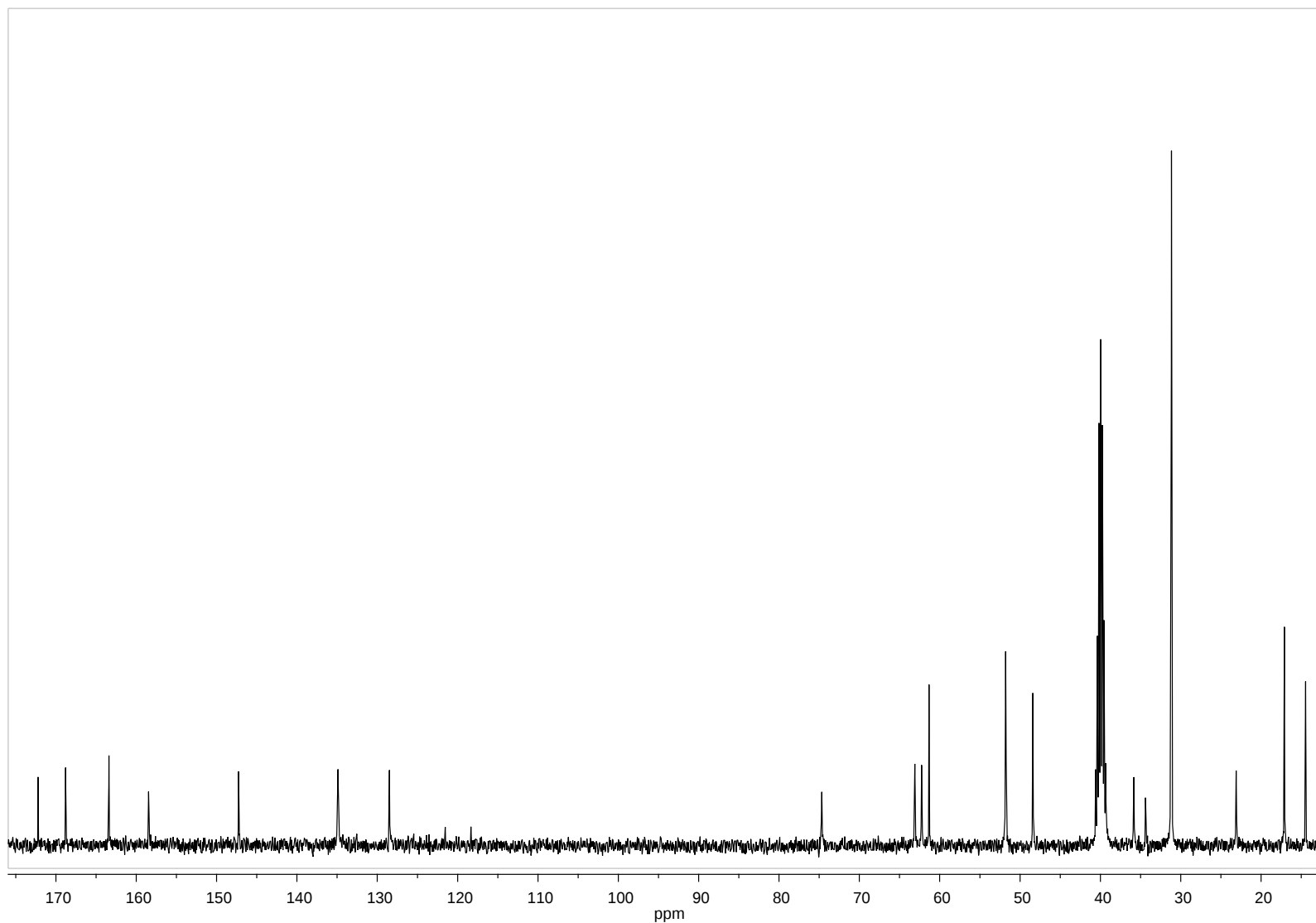


Fig. S75. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 47), DMSO- d_6 , 298 K, 100 MHz

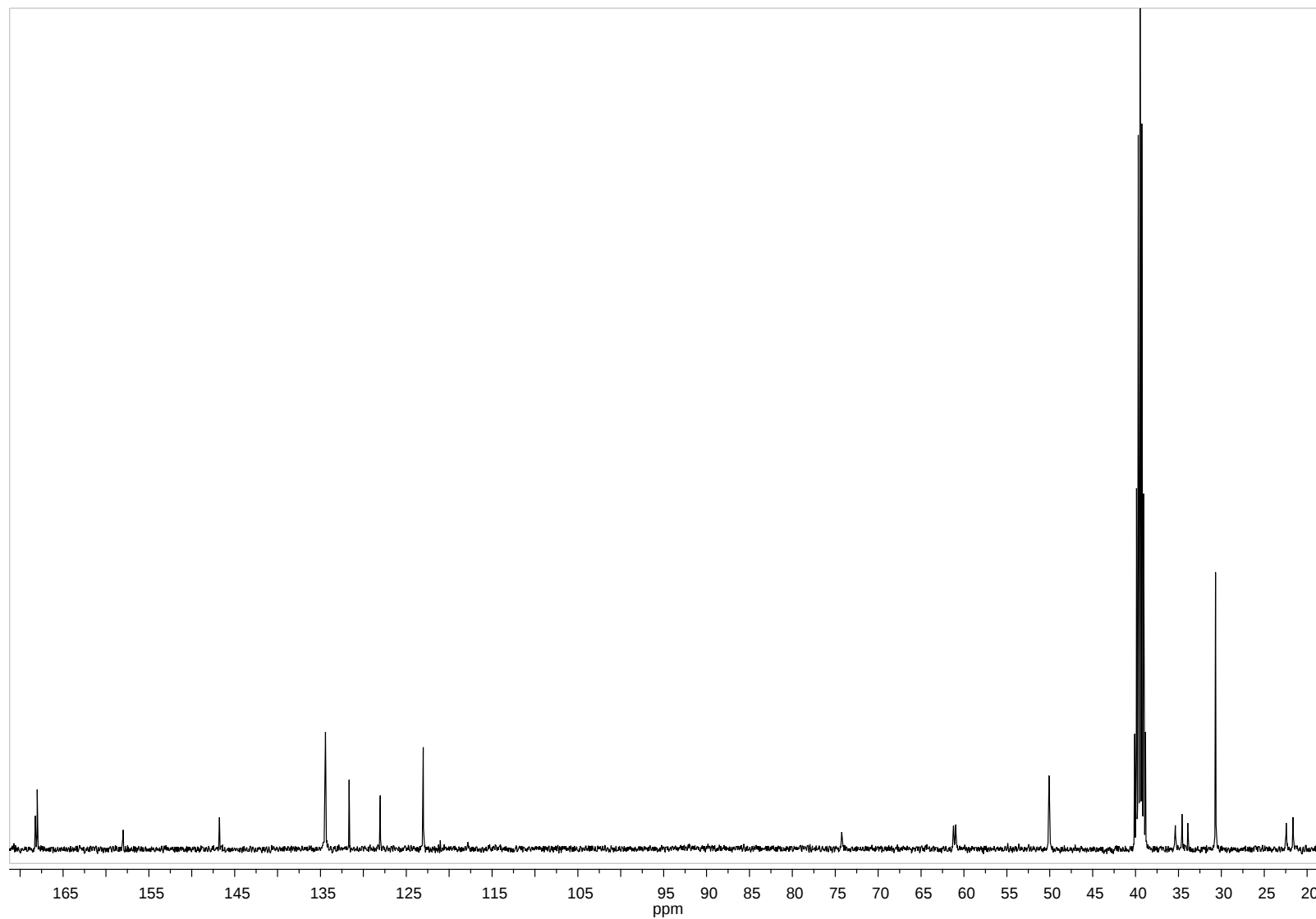


Fig. S76. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 48), CDCl_3 , 298 K, 100 MHz

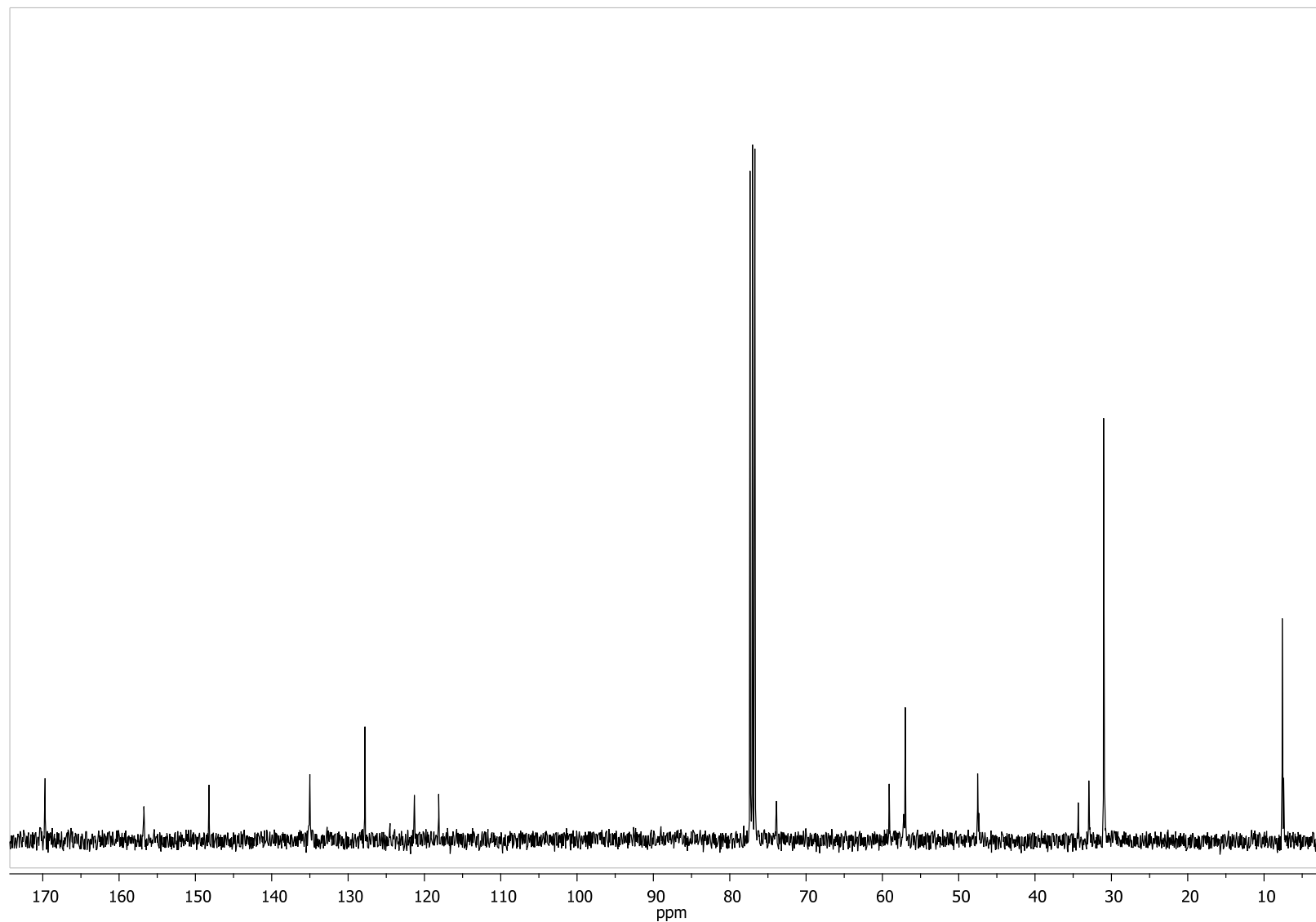


Fig. S77. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 49), CDCl_3 , 298 K, 100 MHz

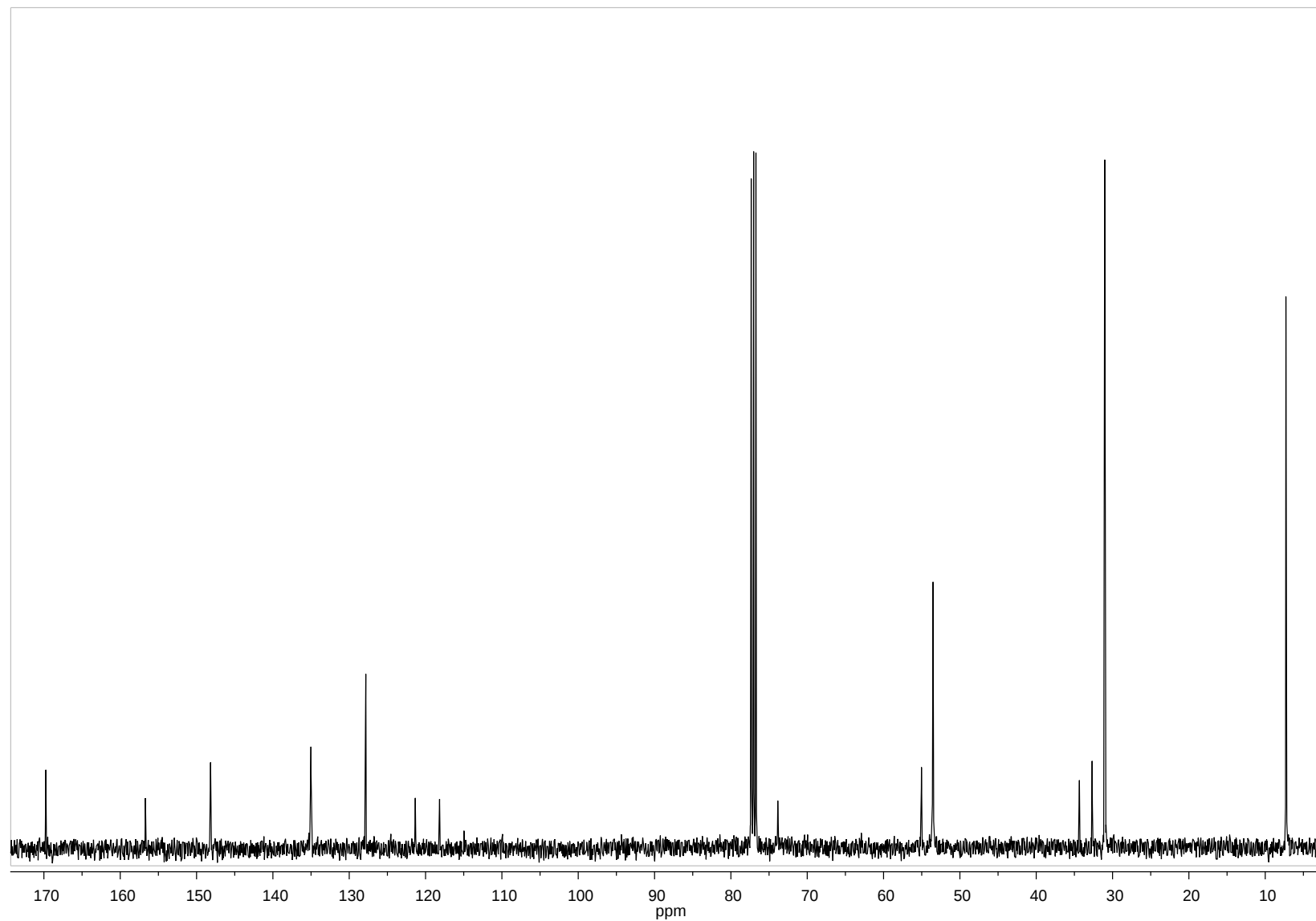


Fig. S78. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 50), CDCl_3 , 298 K, 100 MHz

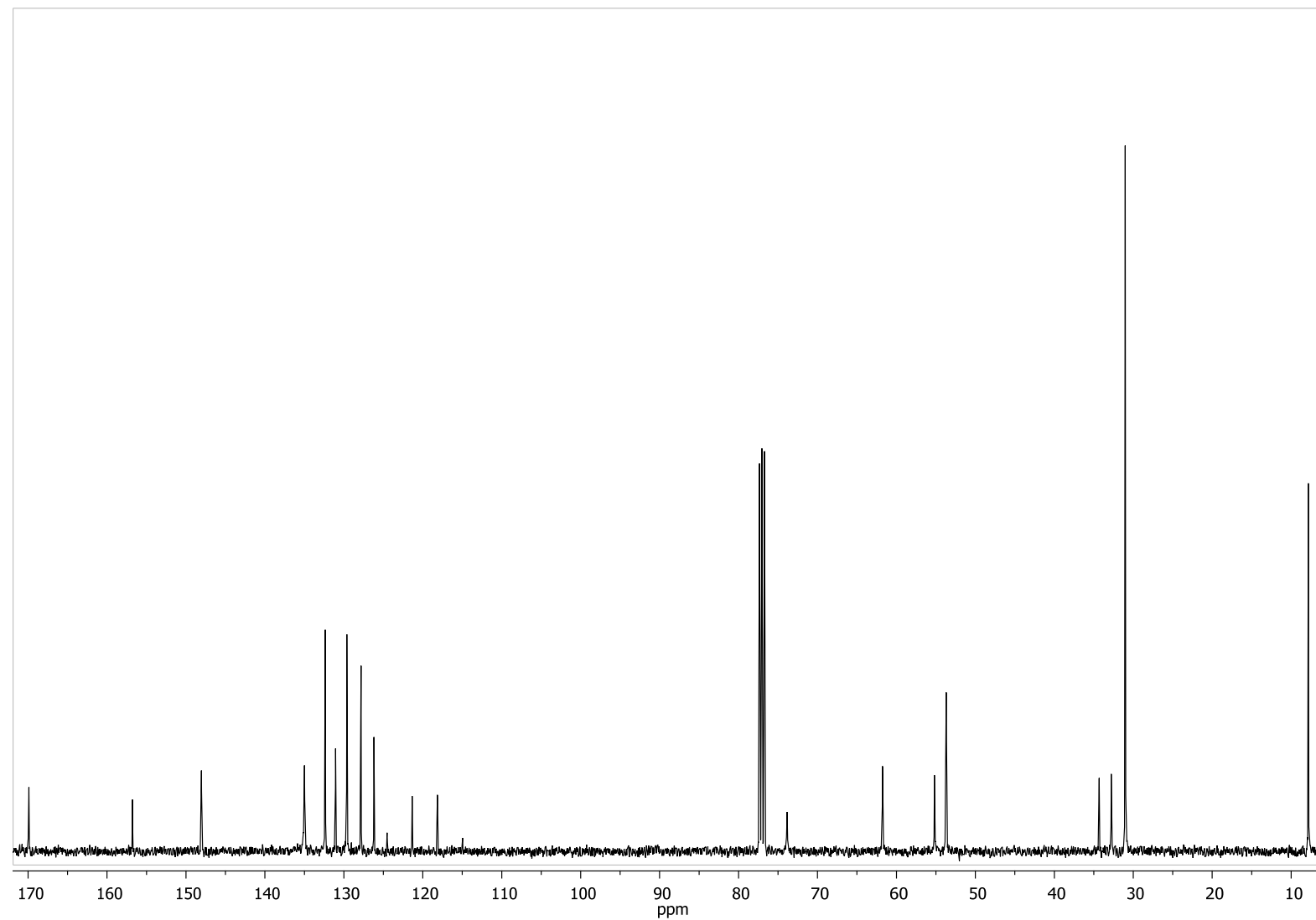


Fig. S79. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 51), CDCl_3 , 298 K, 100 MHz

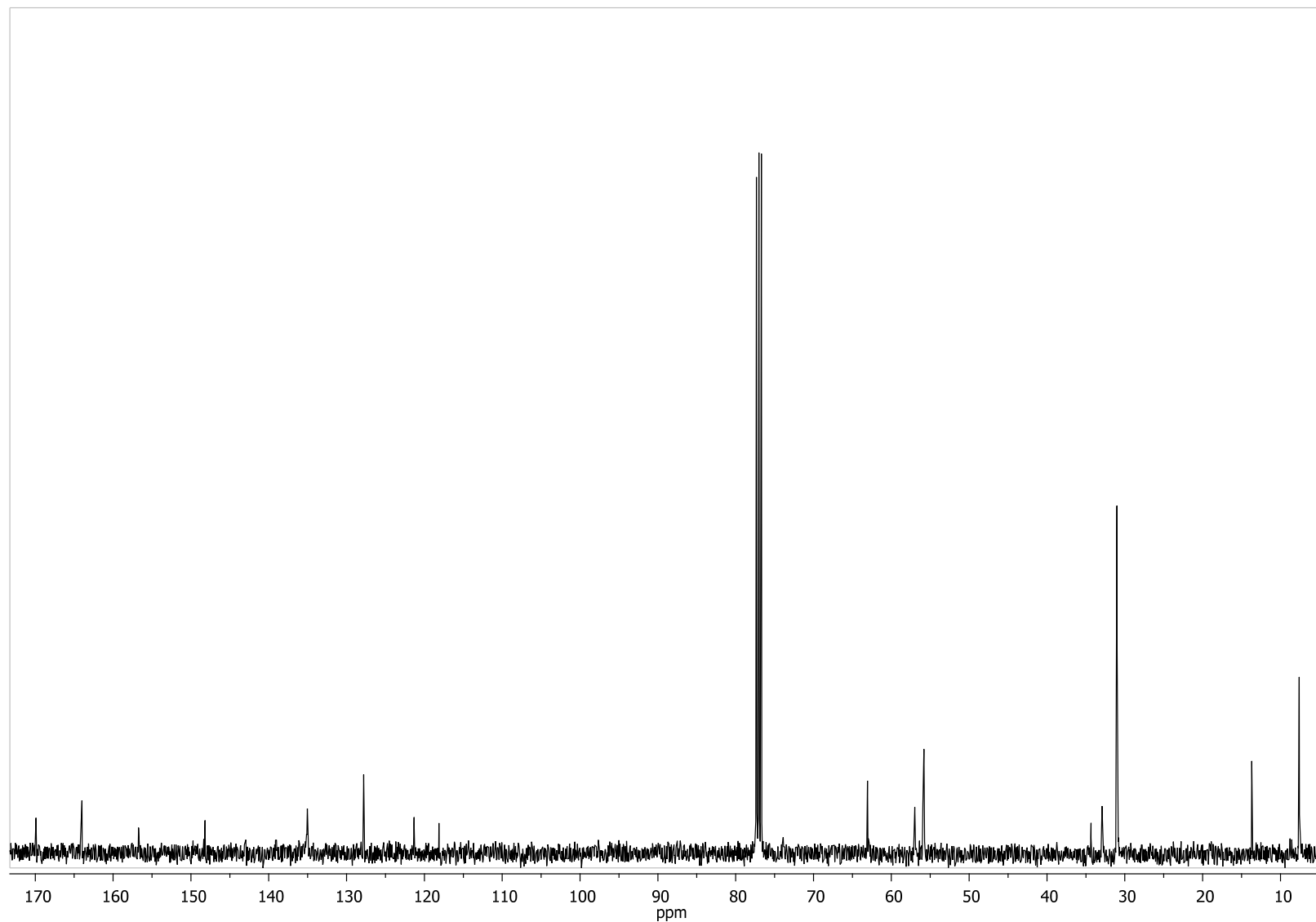


Fig. S80. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 52), DMSO- d_6 , 298 K, 100 MHz

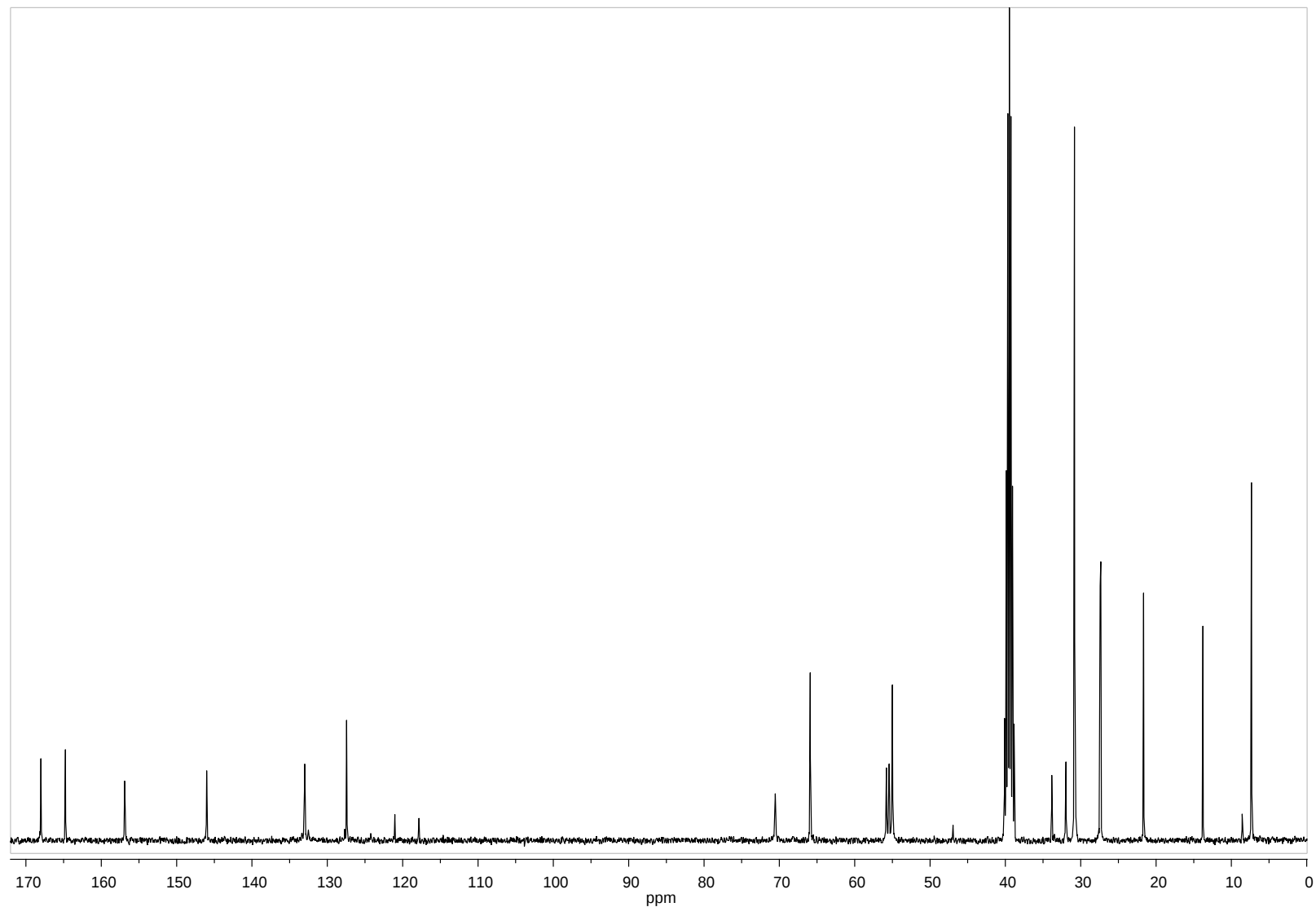


Fig. S81. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonylmethyl)amidocarbonylmethyl})-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 53), DMSO- d_6 , 298 K, 100 MHz

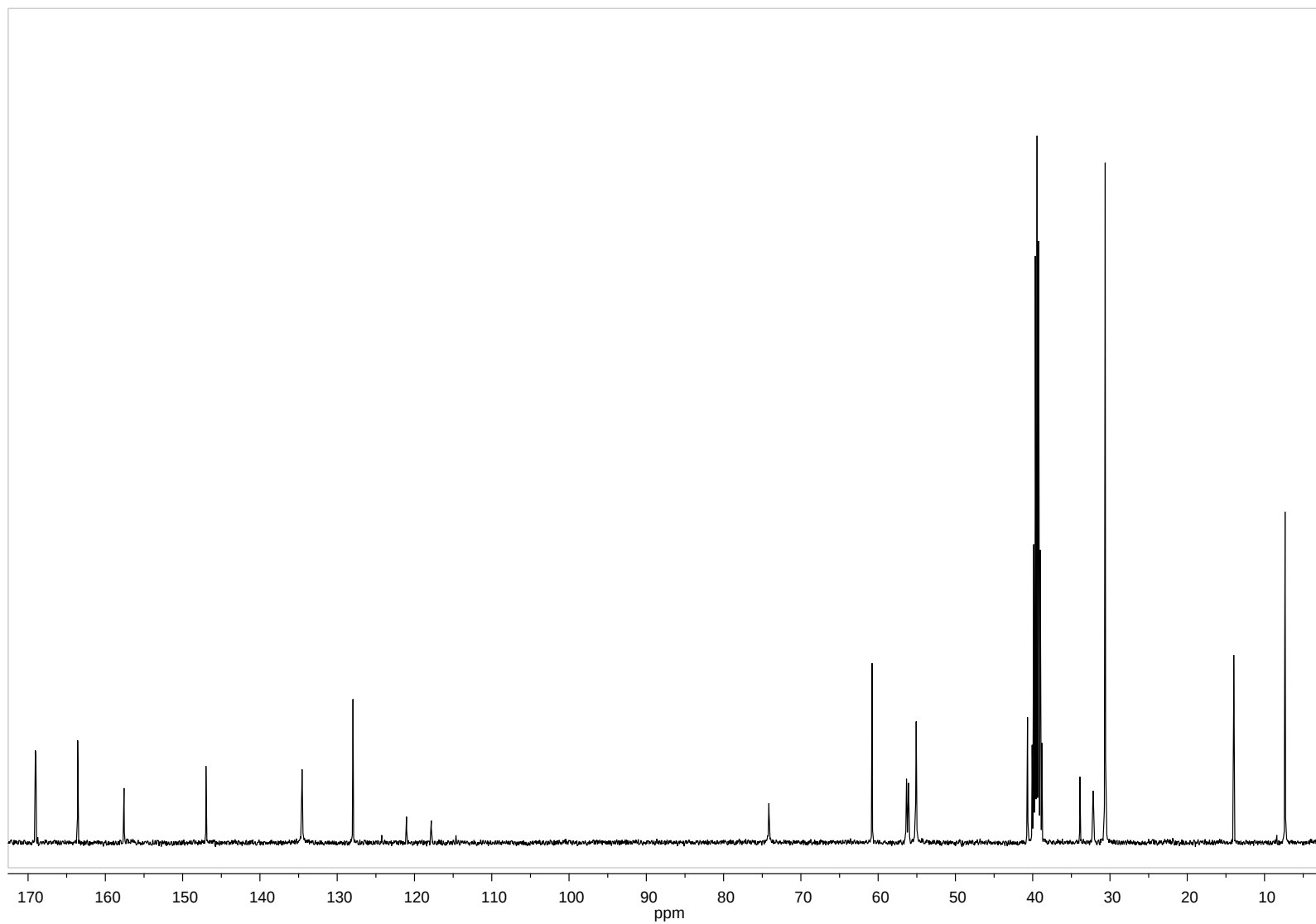


Fig. S82. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{[ethoxycarbonylmethyl]-amidocarbonylmethyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 54), DMSO- d_6 , 298 K, 100 MHz

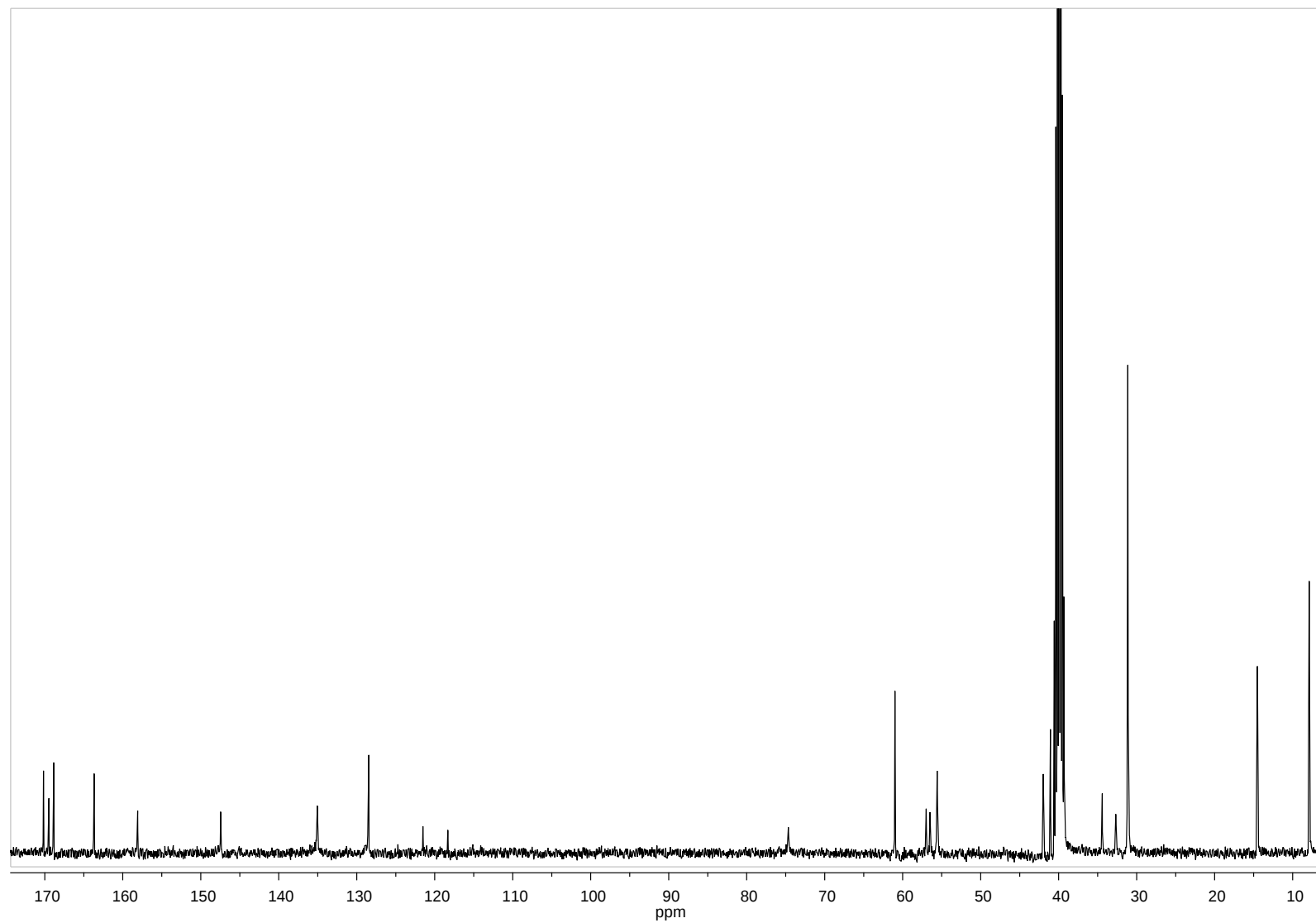


Fig. S83. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 55), DMSO- d_6 , 298 K, 100 MHz

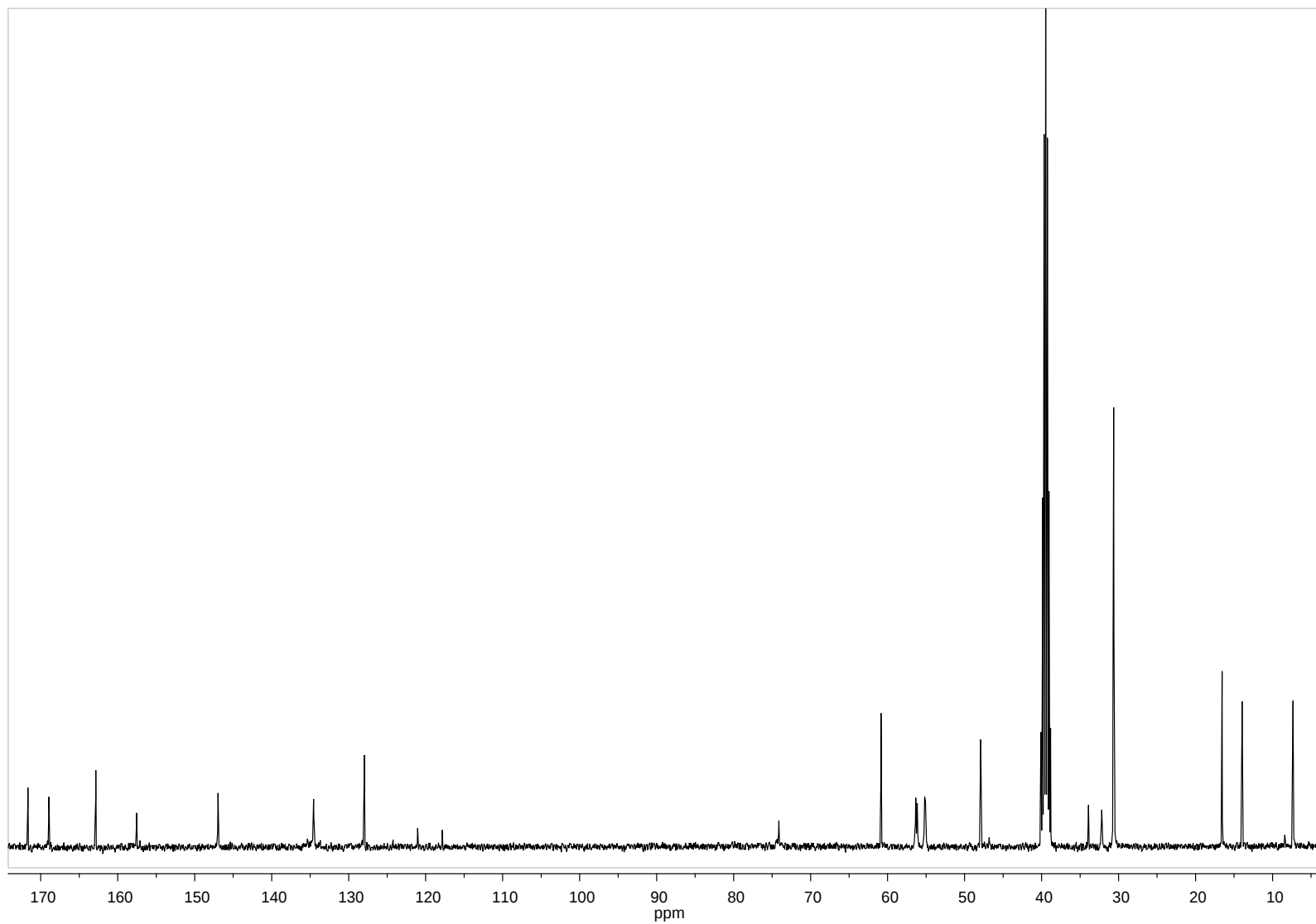


Fig. S84. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 56), DMSO- d_6 , 298 K, 100 MHz

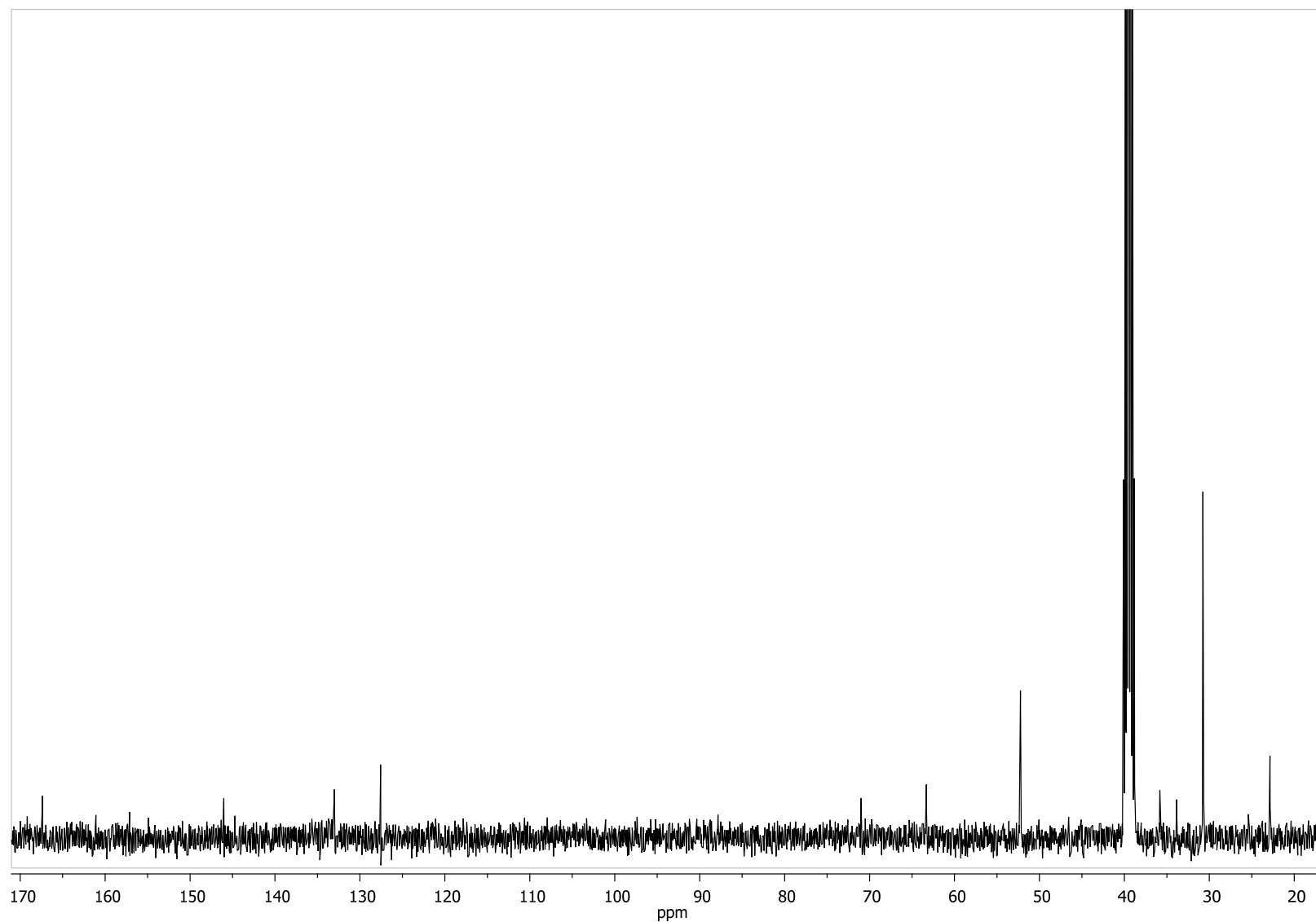


Fig. S85. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 57), DMSO- d_6 , 298 K, 100 MHz

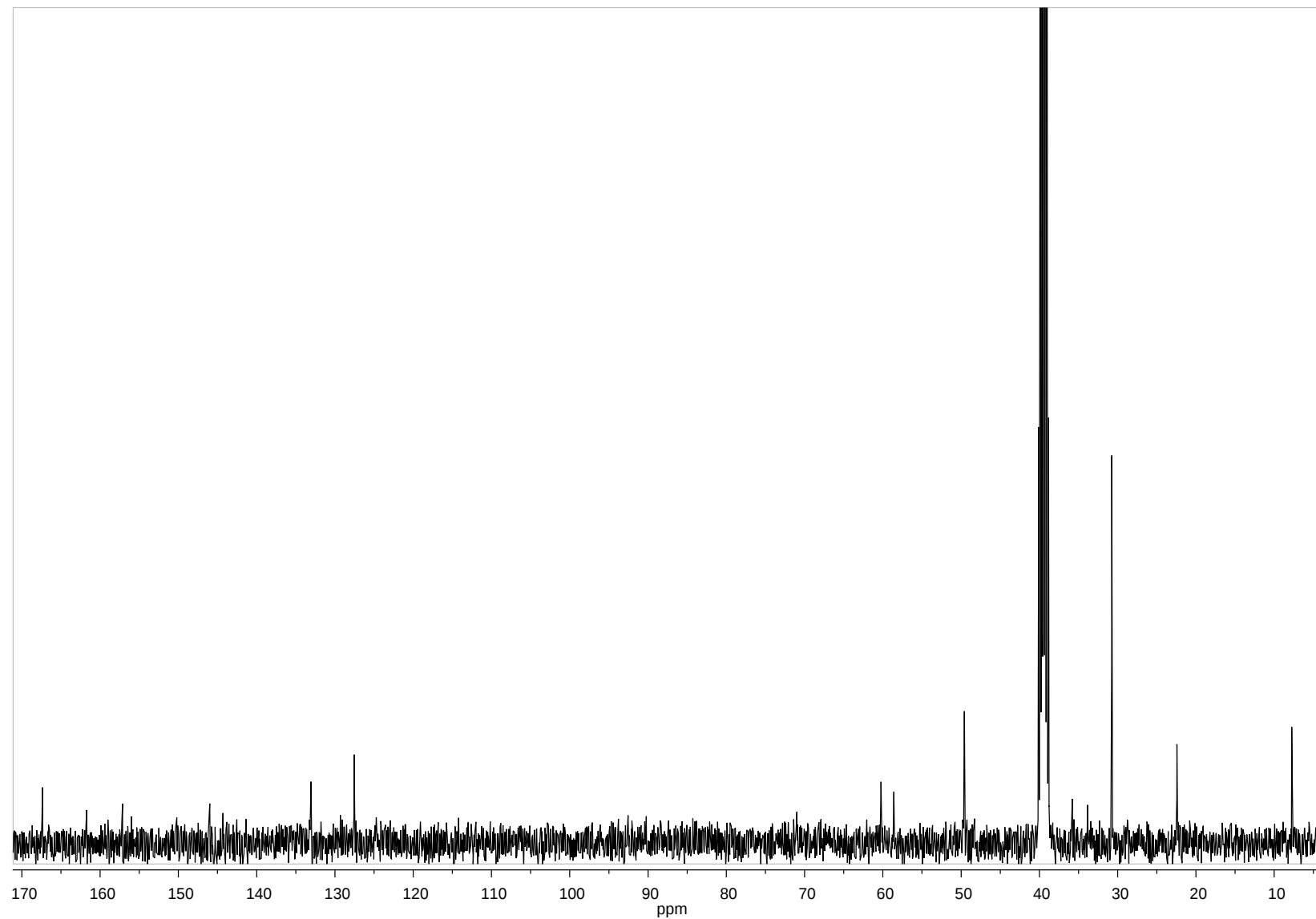


Fig. S86. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* **58**), DMSO- d_6 , 298 K, 100 MHz

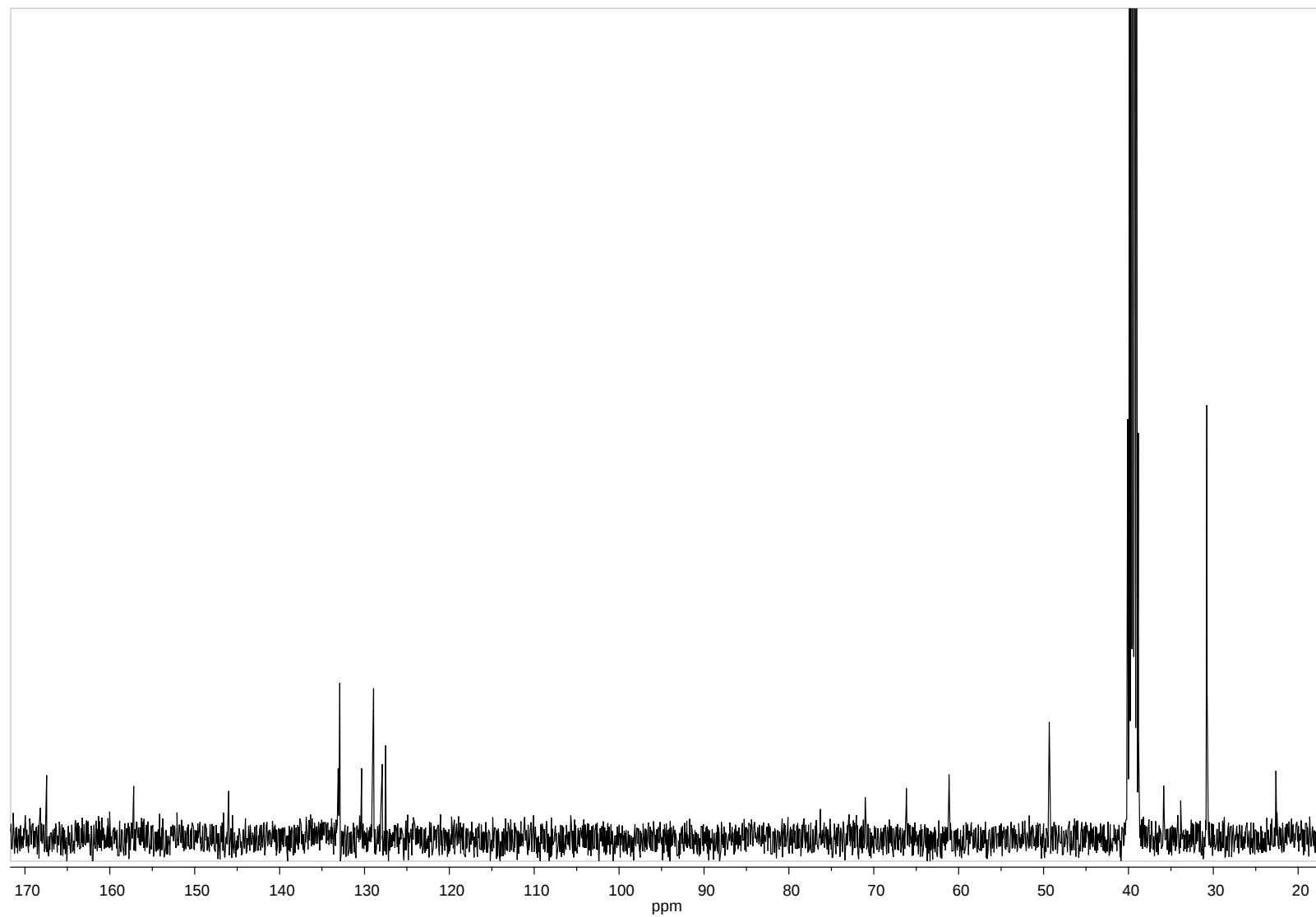


Fig. S87. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 59), DMSO- d_6 , 298 K, 100 MHz

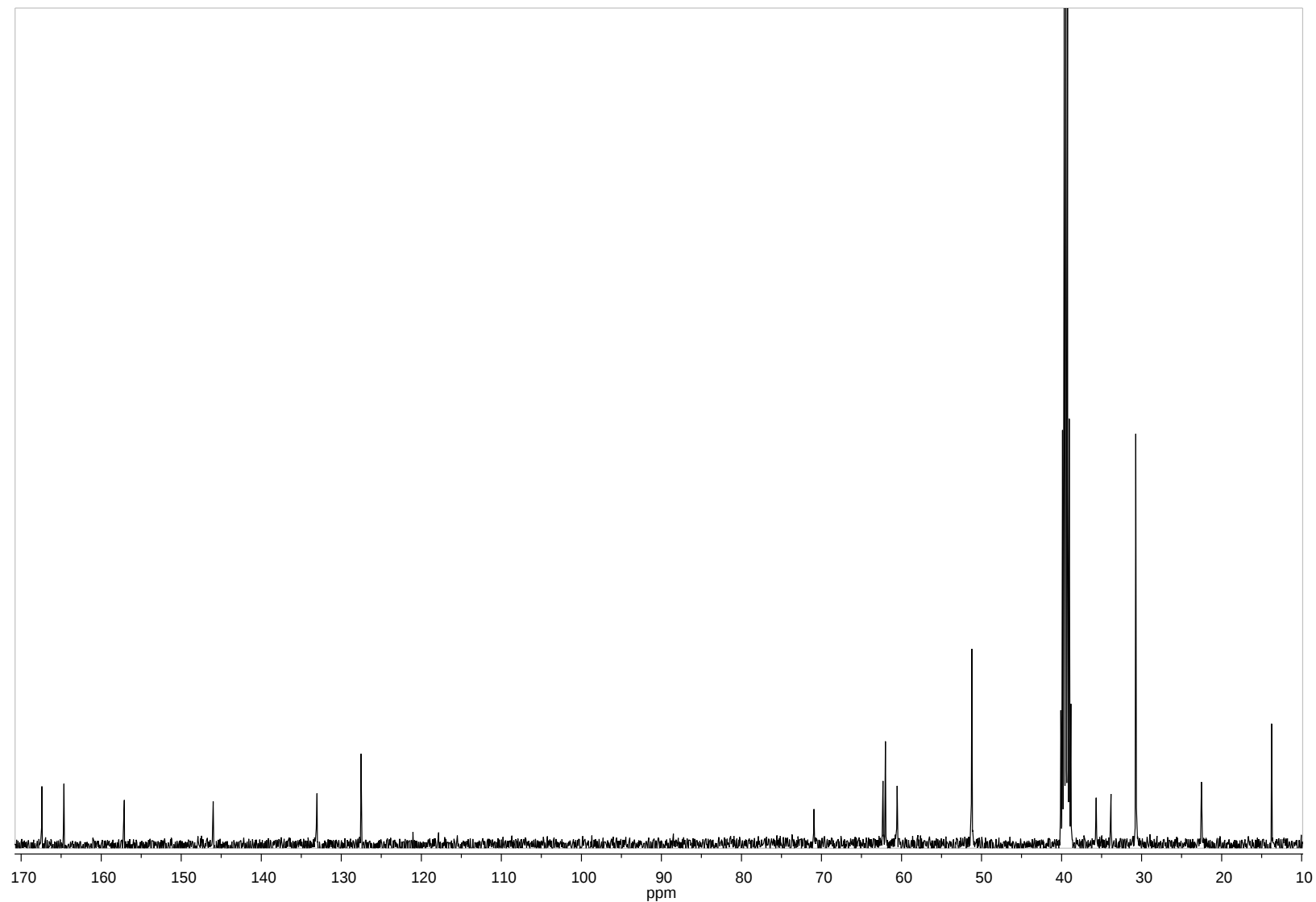


Fig. S88. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxy carbonylmethyl) ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* **60**), DMSO- d_6 , 298 K, 100 MHz

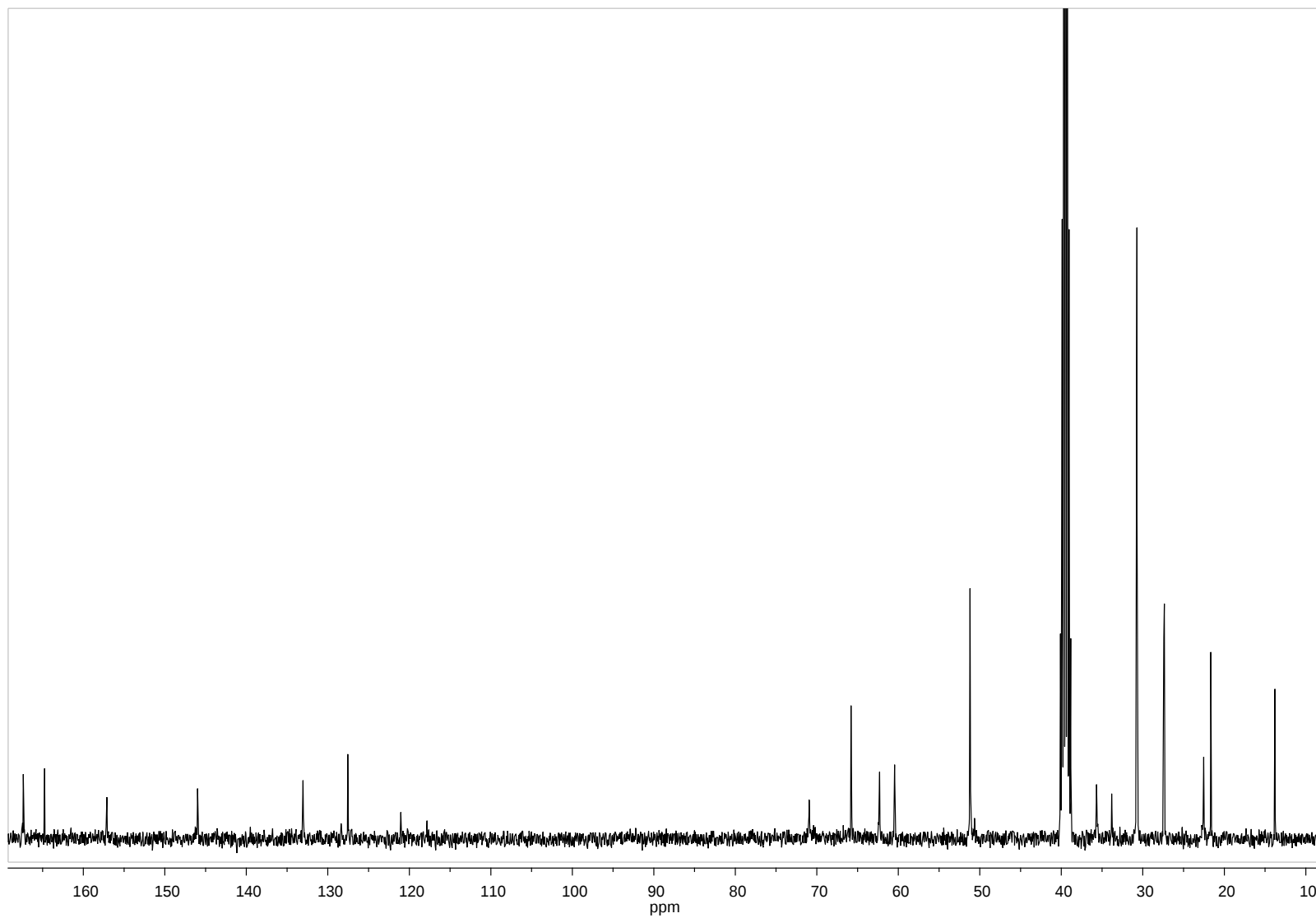


Fig. S89. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonylmethyl)amidocarbonylmethyl}-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate 61*), DMSO- d_6 , 298 K, 100 MHz

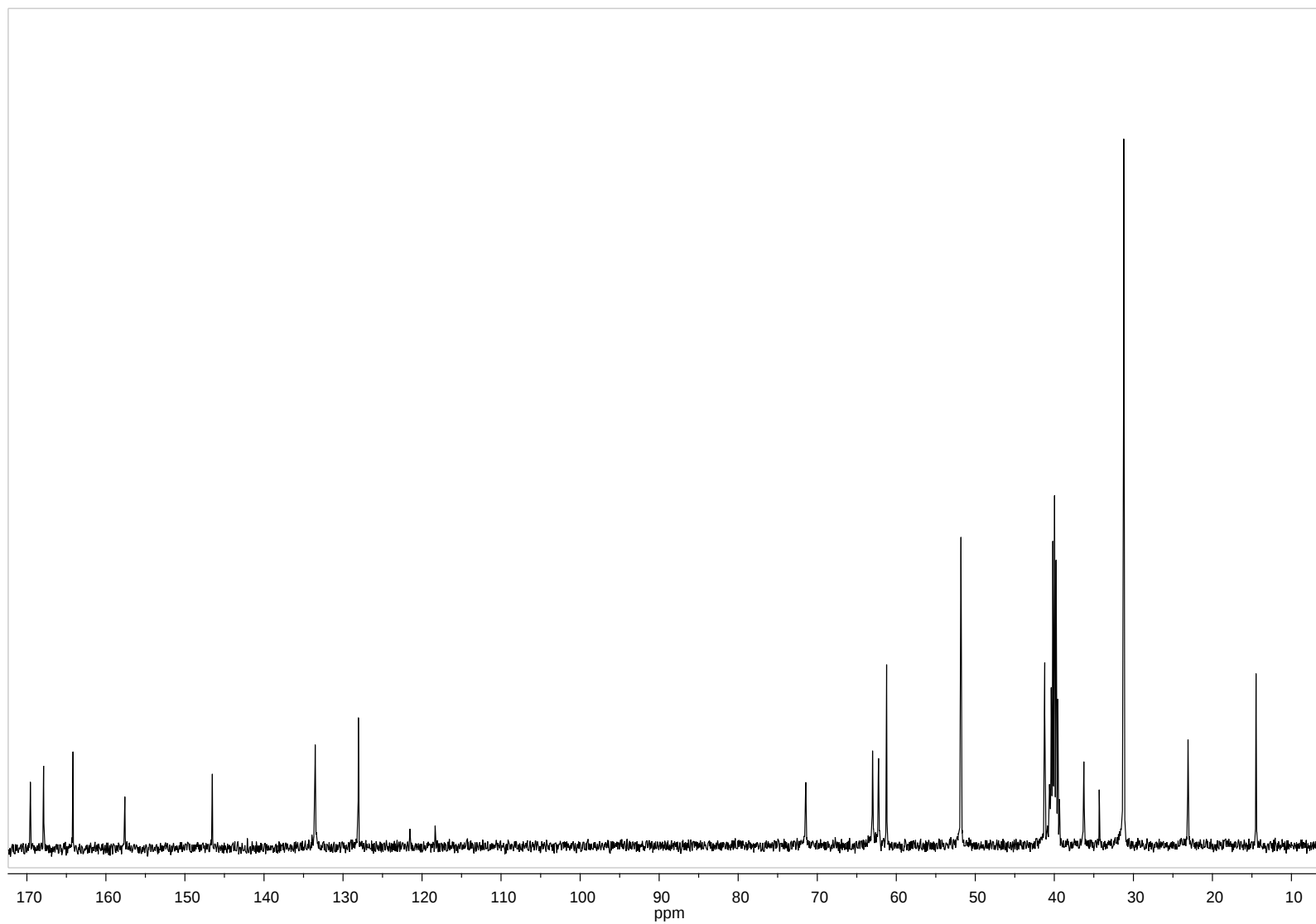


Fig. S90. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{([ethoxycarbonylmethyl]amidocarbonylmethyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 62), DMSO- d_6 , 298 K, 100 MHz

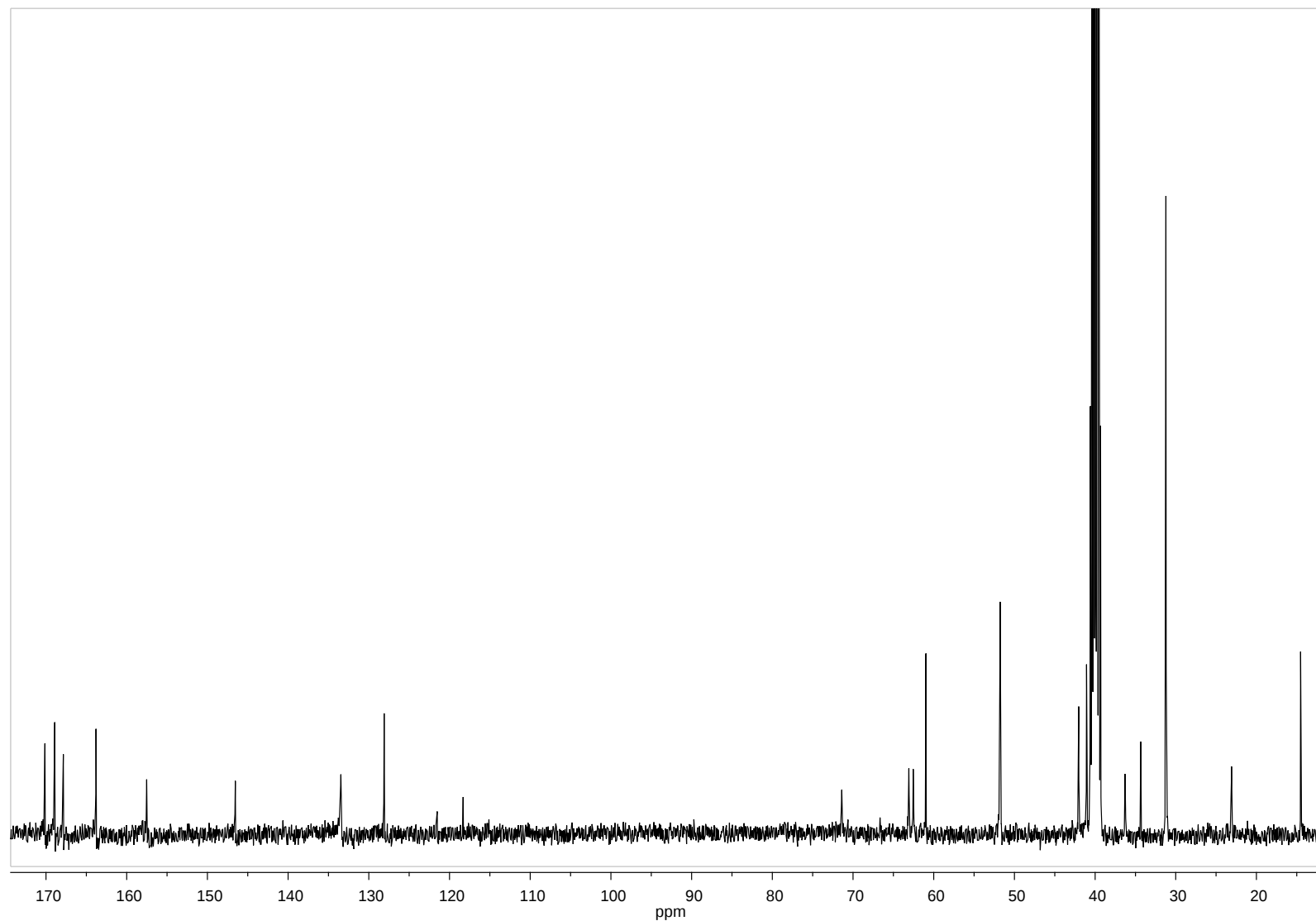


Fig. S91. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 63), DMSO- d_6 , 298 K, 100 MHz

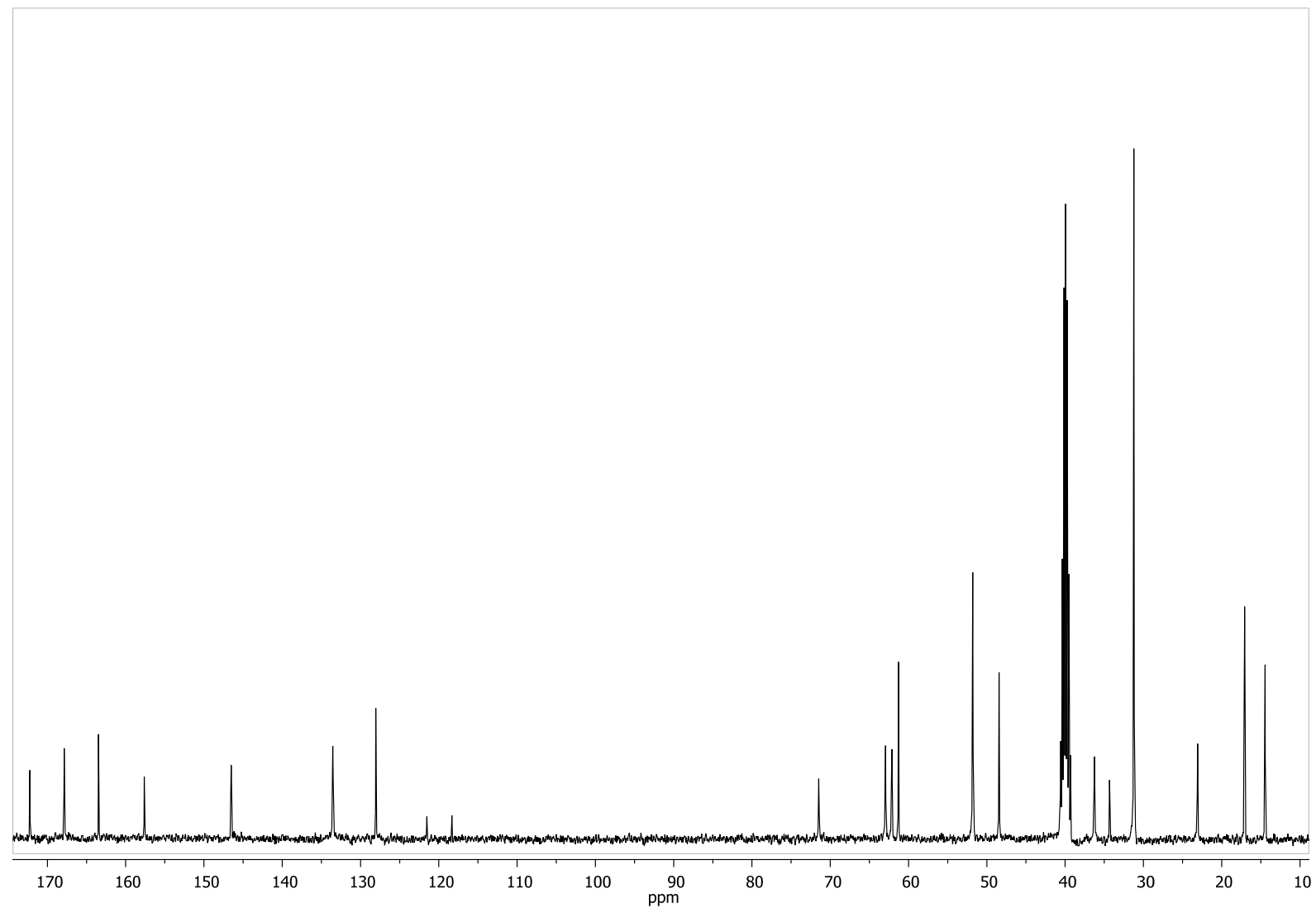


Fig. S92. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide}ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* **64**), DMSO- d_6 , 298 K, 100 MHz

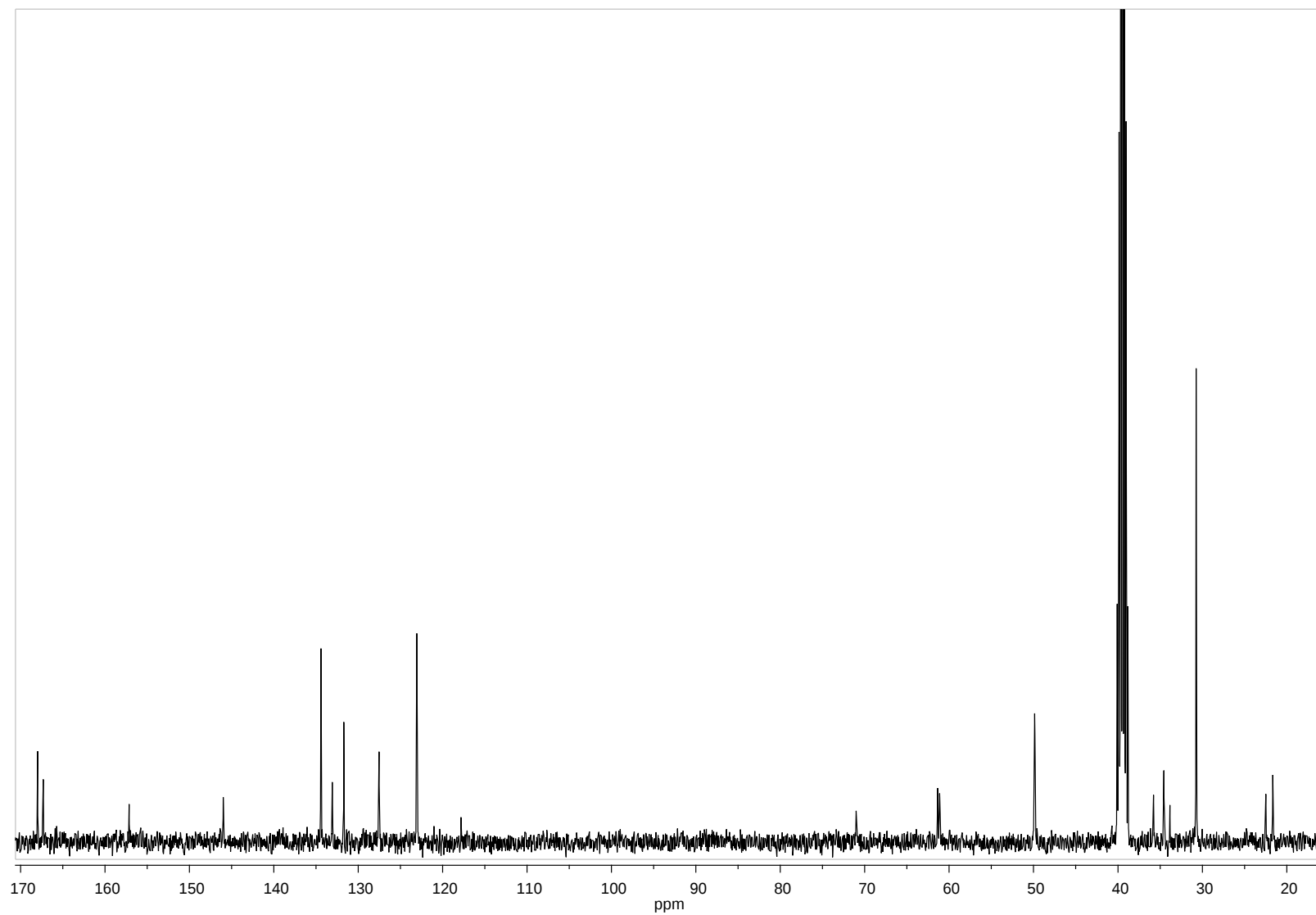


Fig. S93. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* **65**), DMSO- d_6 , 298 K, 100 MHz

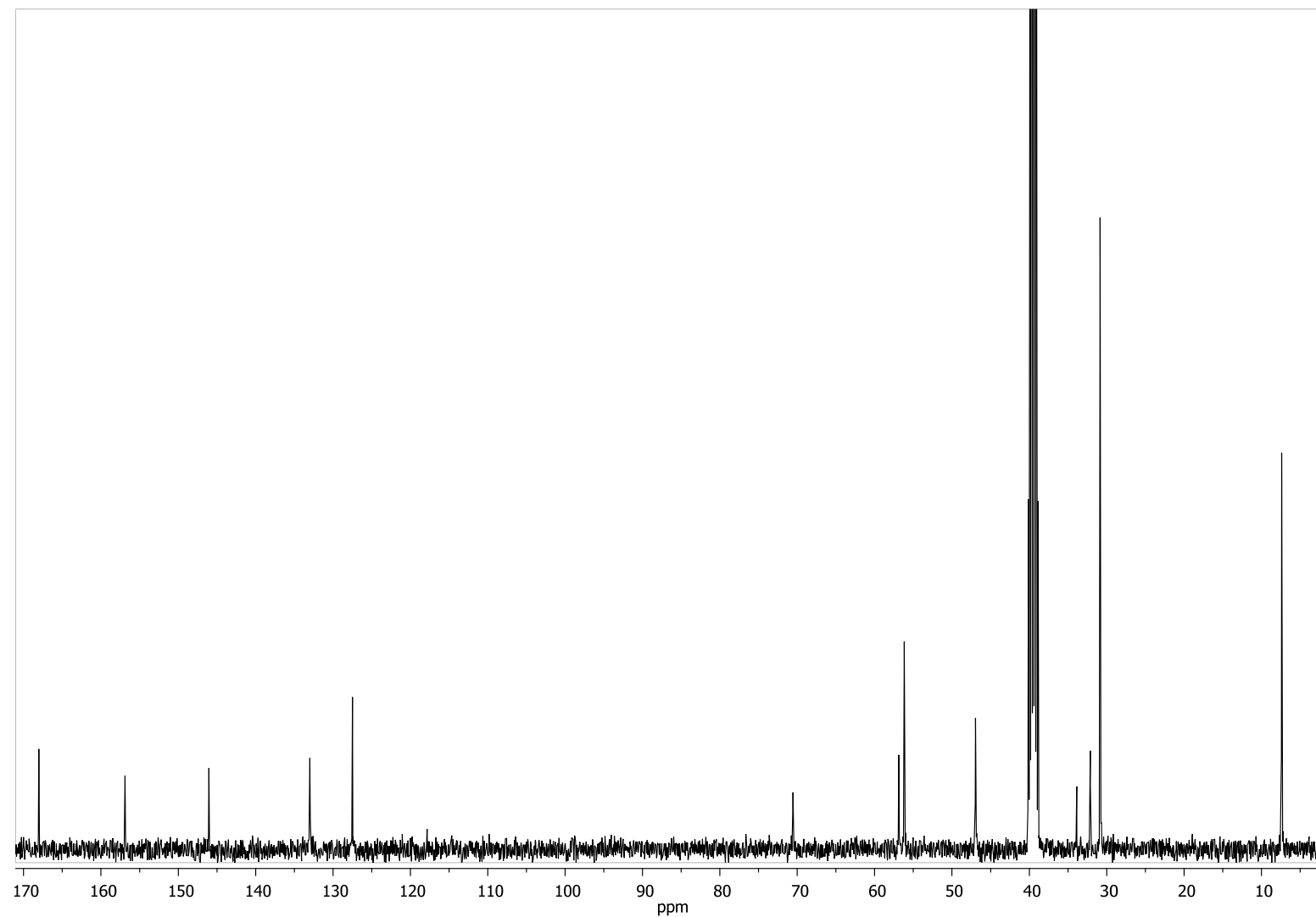


Fig. S94. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* **66**), DMSO- d_6 , 298 K, 100 MHz

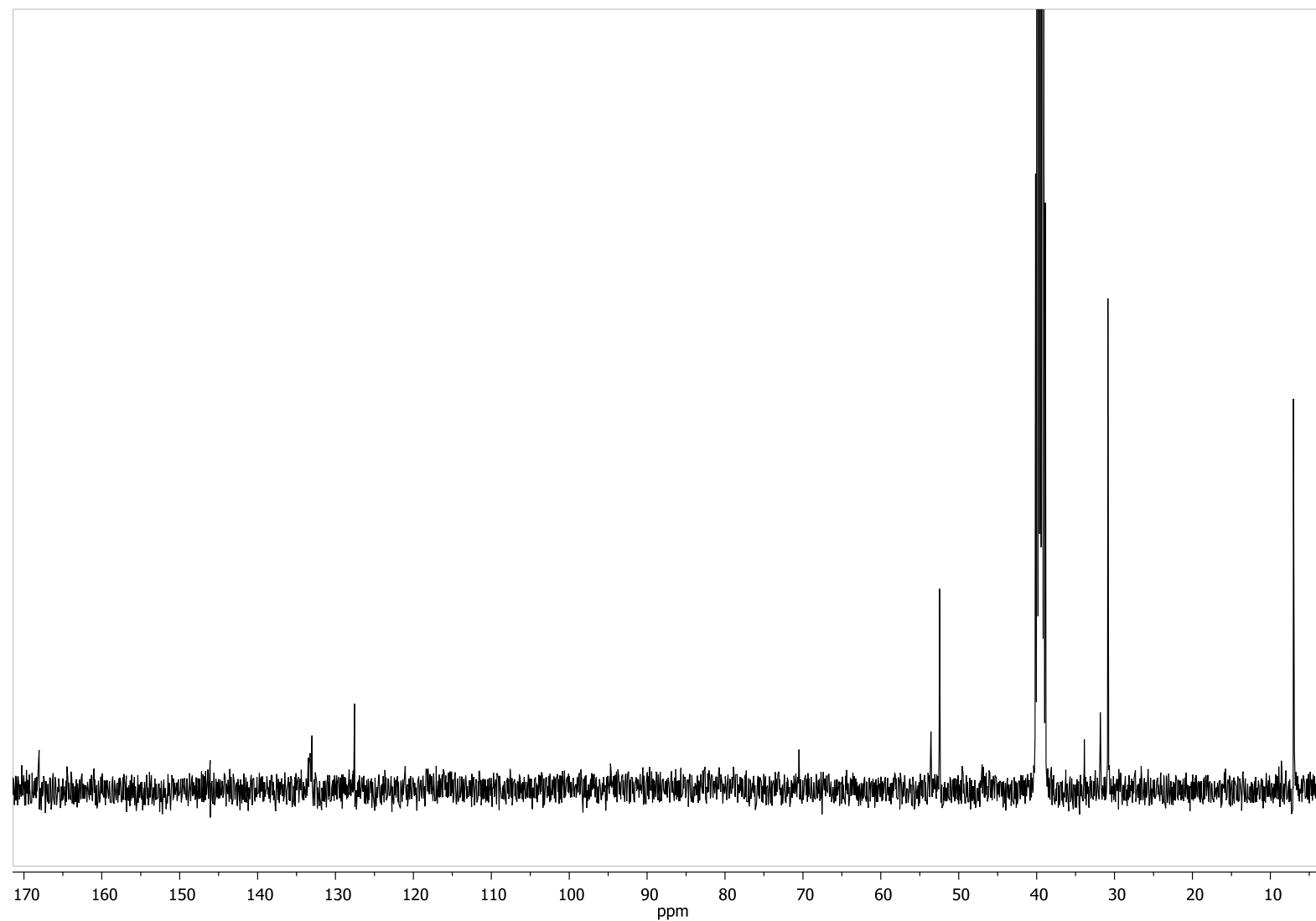


Fig. S95. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate **67**), DMSO- d_6 , 298 K, 100 MHz

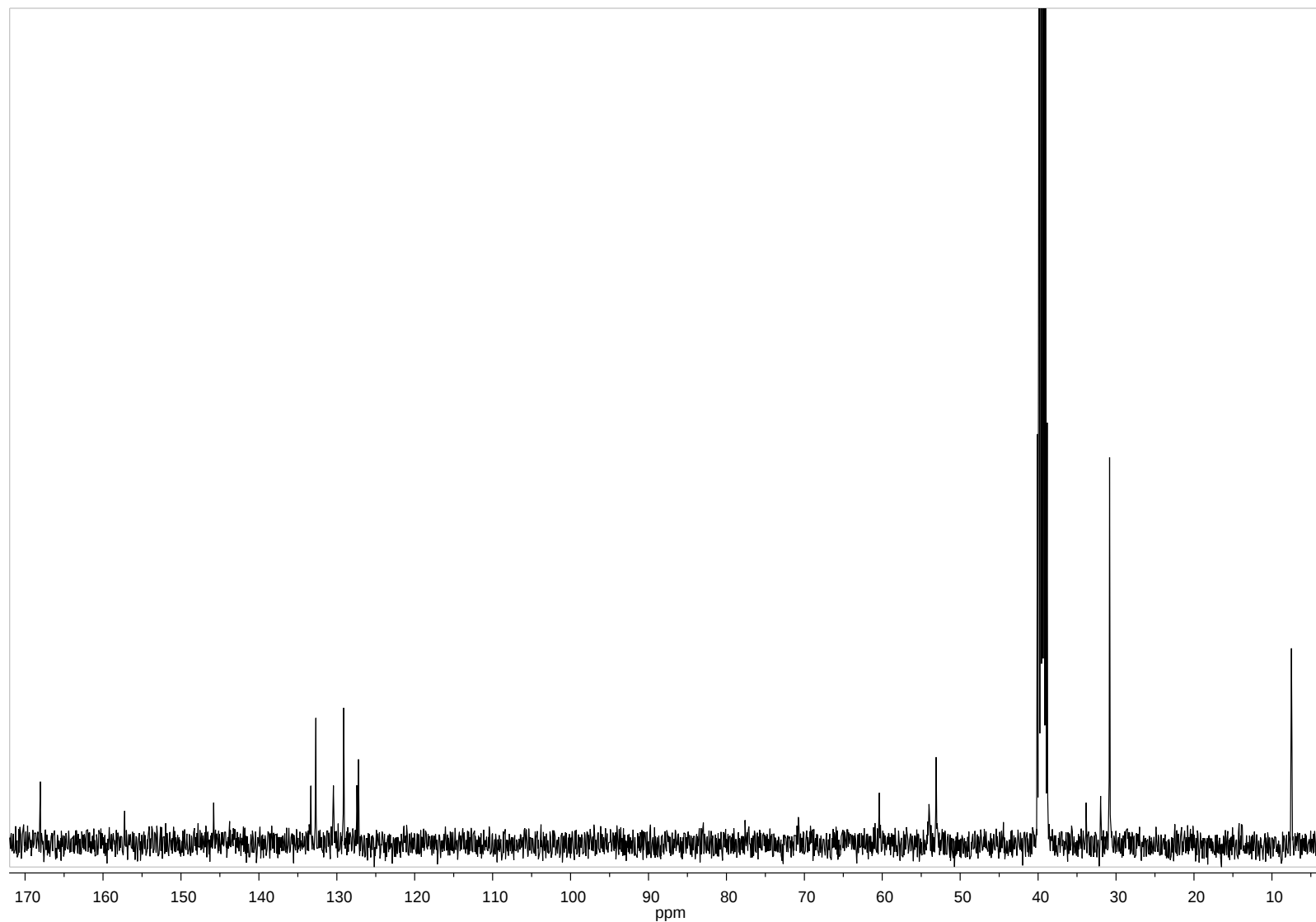


Fig. S96. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 68), DMSO- d_6 , 298 K, 100 MHz

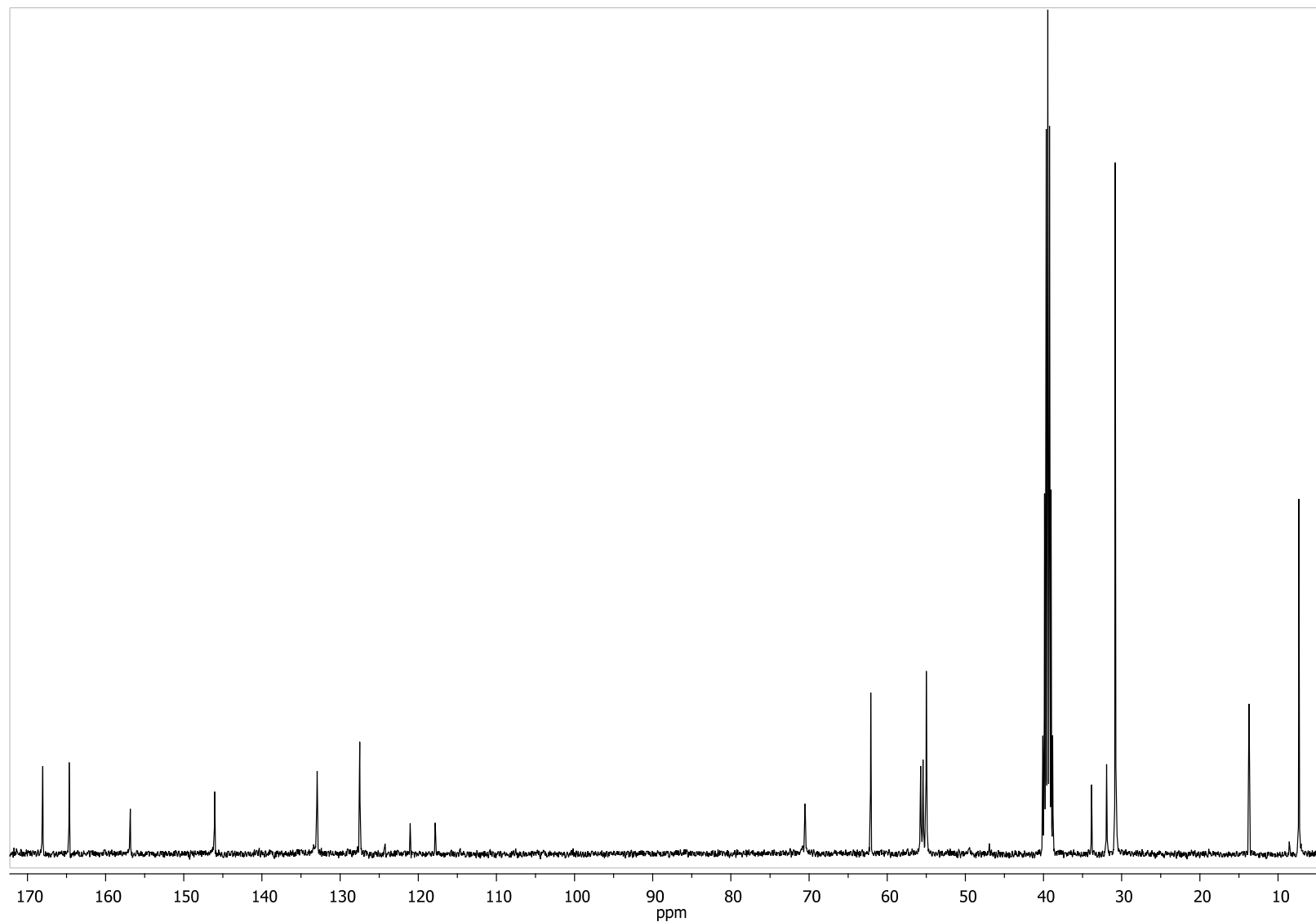


Fig. S97. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate* 69), DMSO- d_6 , 298 K, 100 MHz

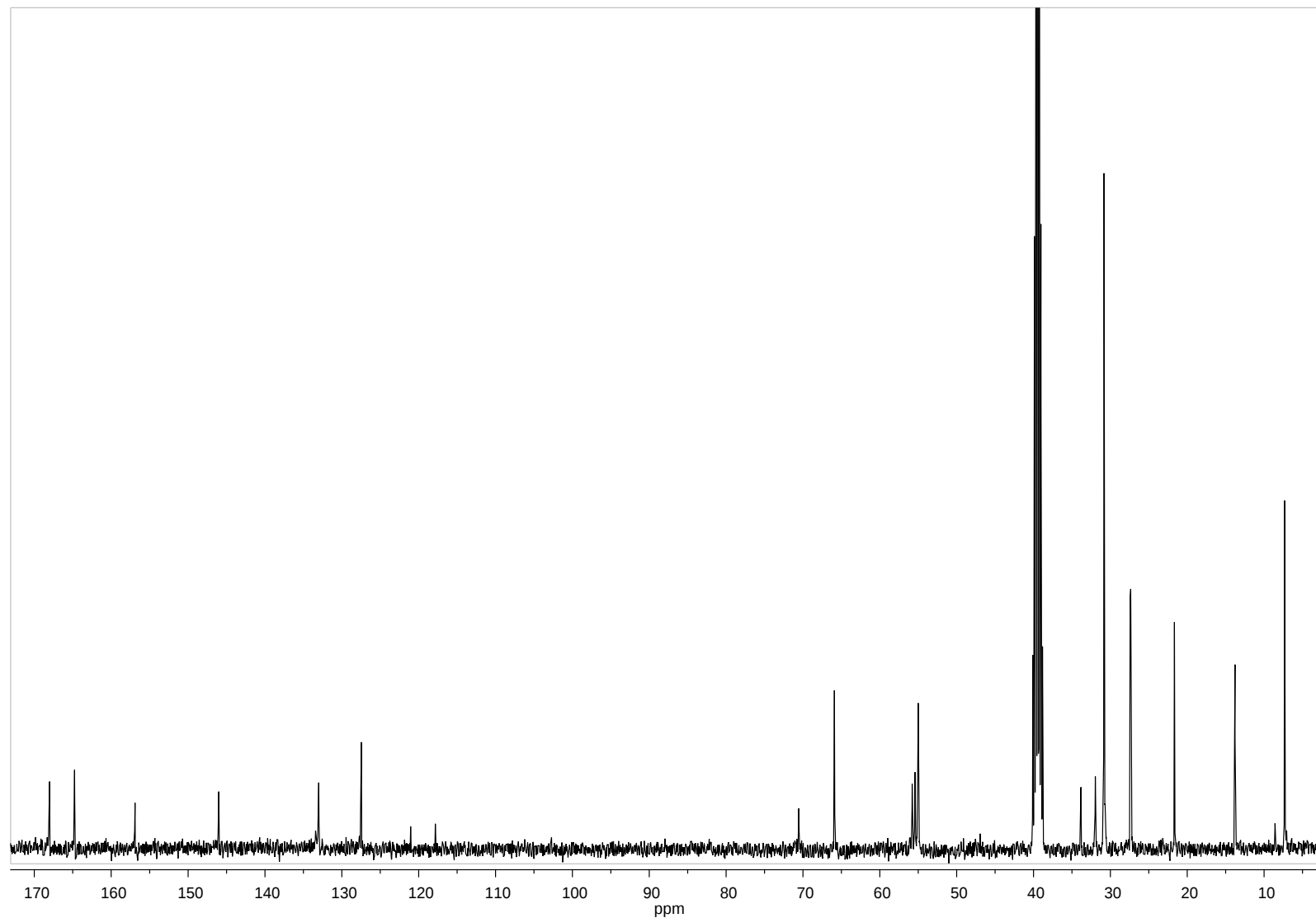


Fig. S98. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(2',2'-diethyl-2'-{(ethoxycarbonylmethyl)amidocarbonylmethyl})-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate 70*), DMSO- d_6 , 298 K, 100 MHz

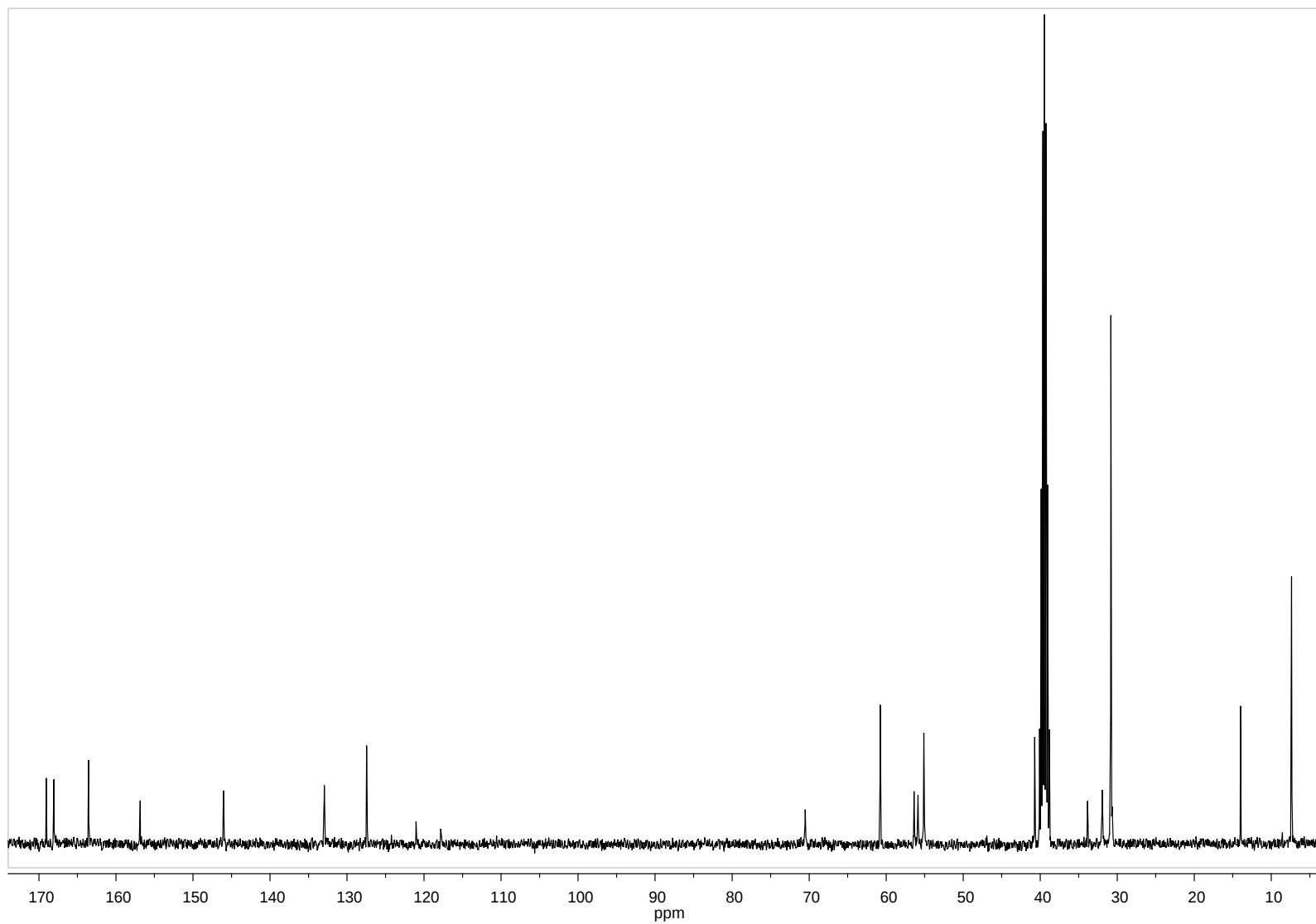


Fig. S99. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-
{([ethoxycarbonylmethyl]amidocarbonylmethyl)amidocarbonylmethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene
tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate 71*), DMSO- d_6 , 298 K, 100 MHz

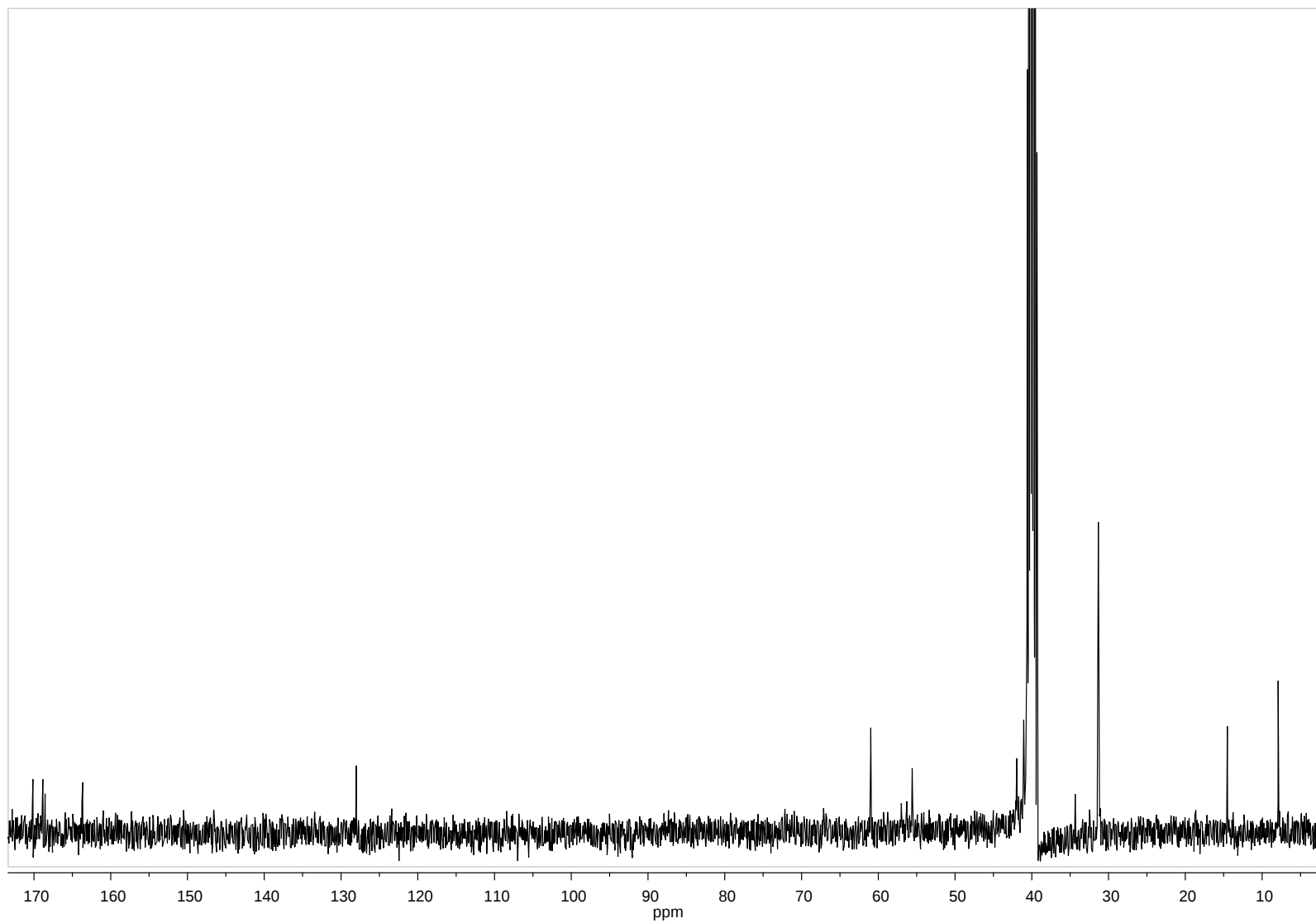


Fig. S100. ^{13}C NMR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 72), DMSO- d_6 , 298 K, 100 MHz

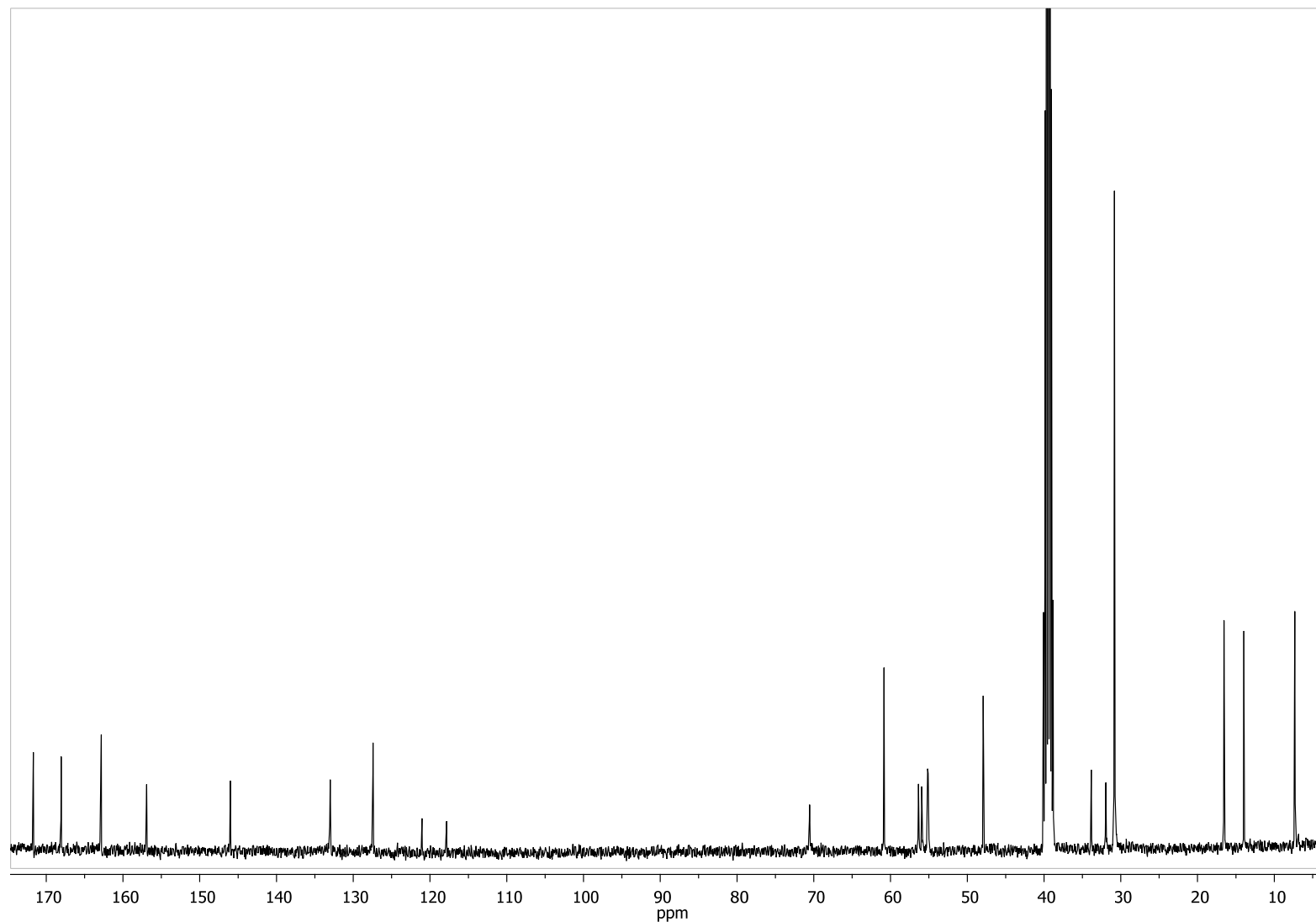


Fig. S101. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxycarbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 9)

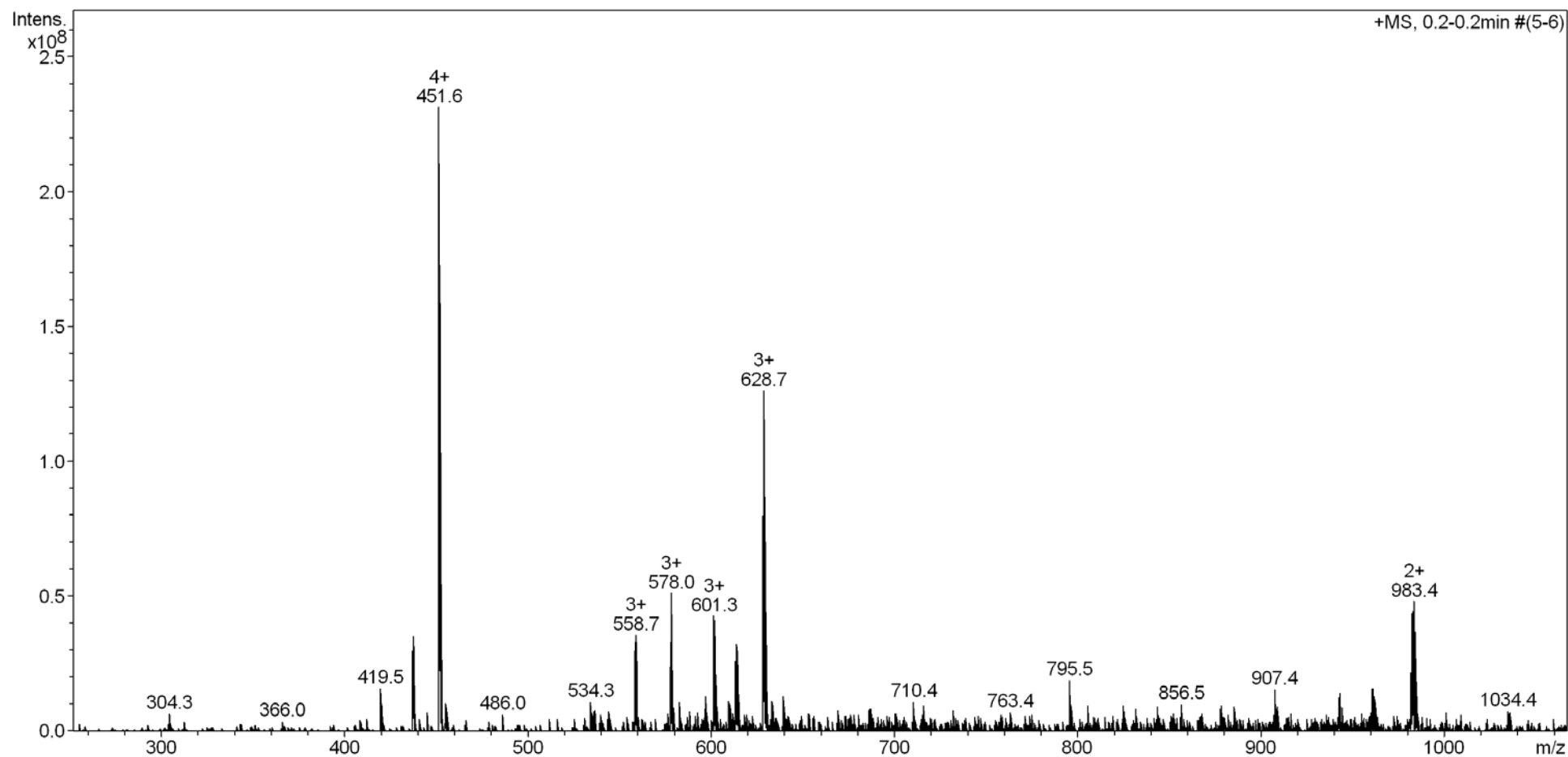


Fig. S102. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (cone 15)

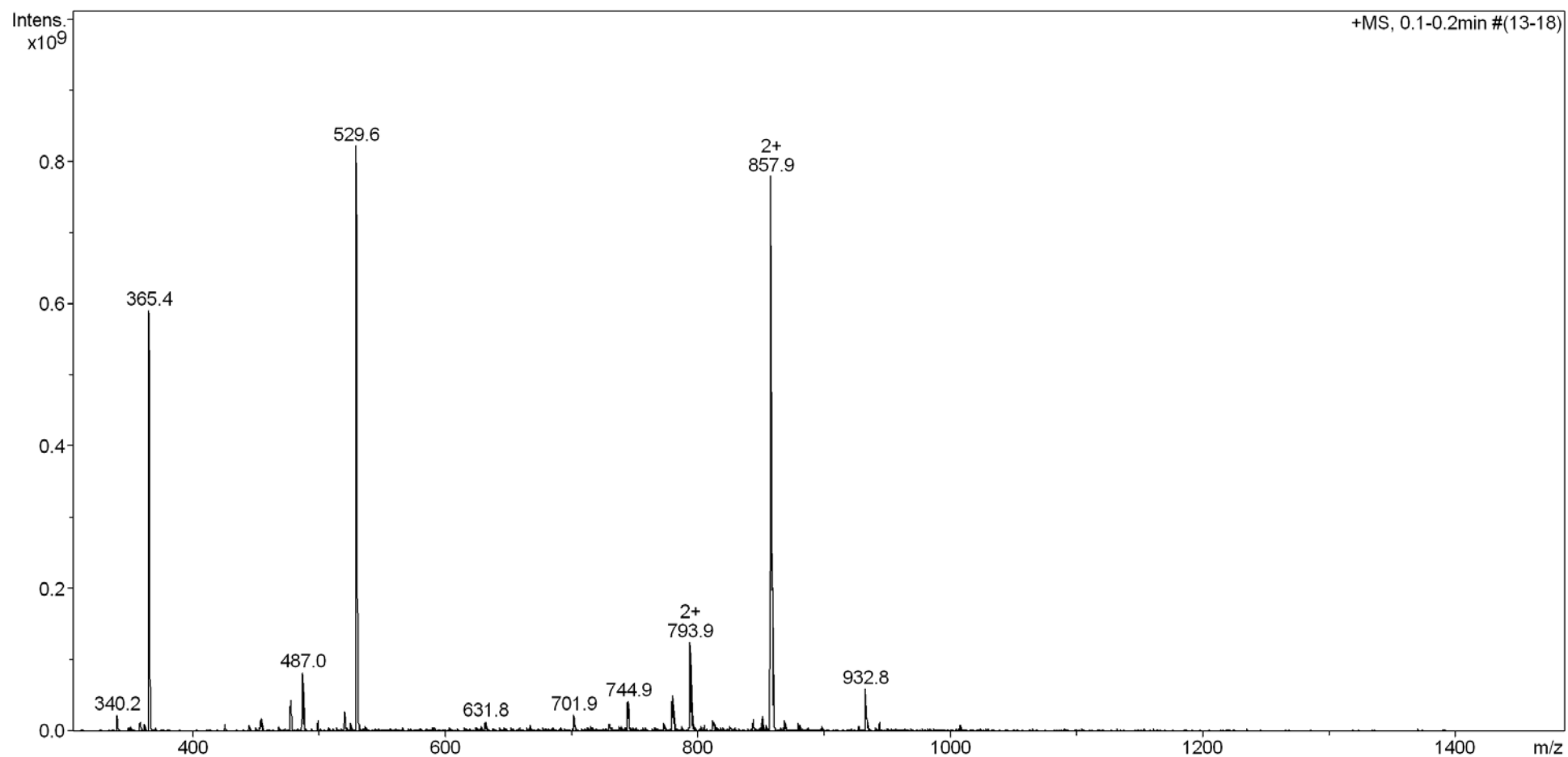


Fig. S103. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 16)

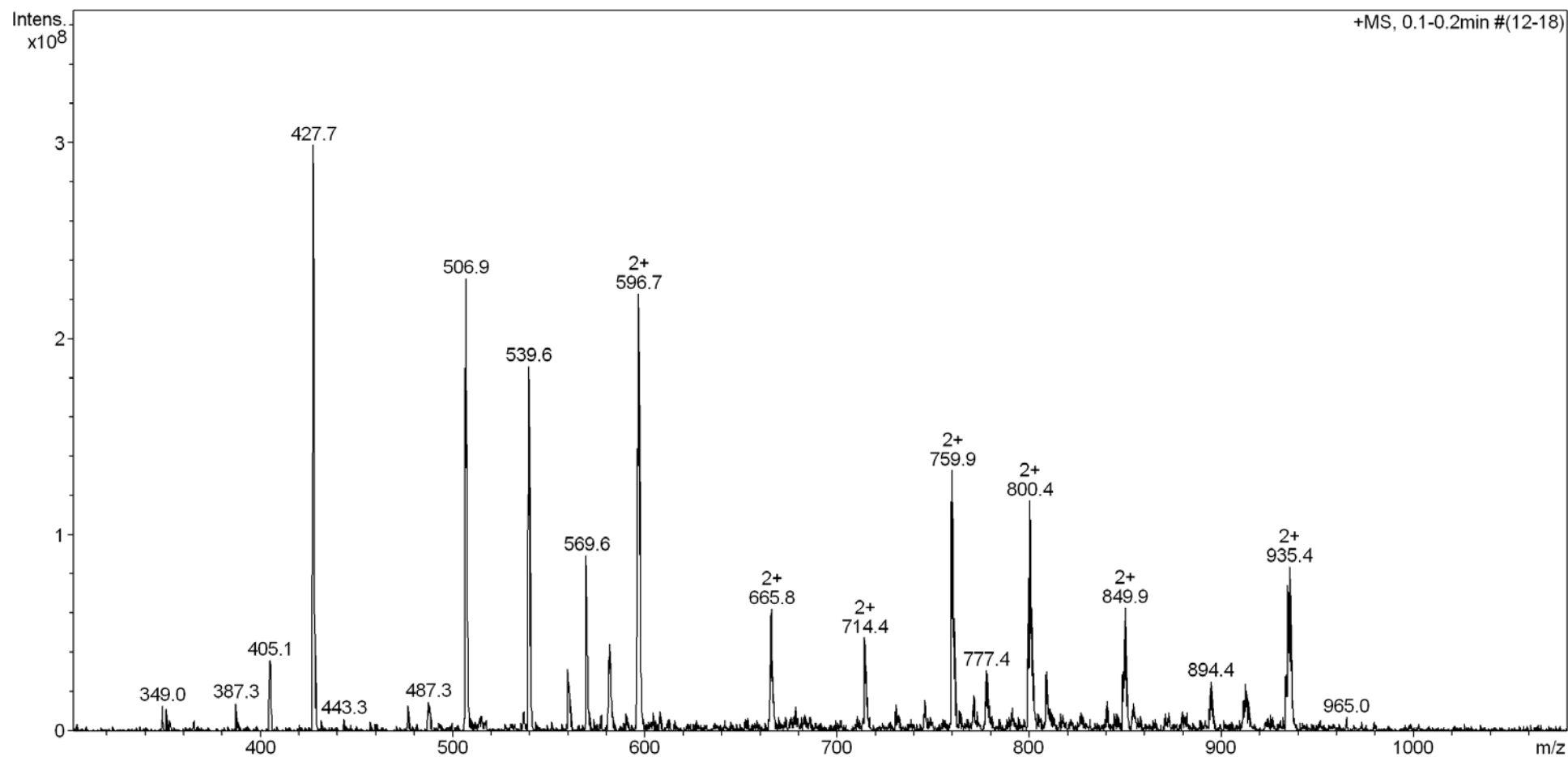


Fig. S104. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 17)

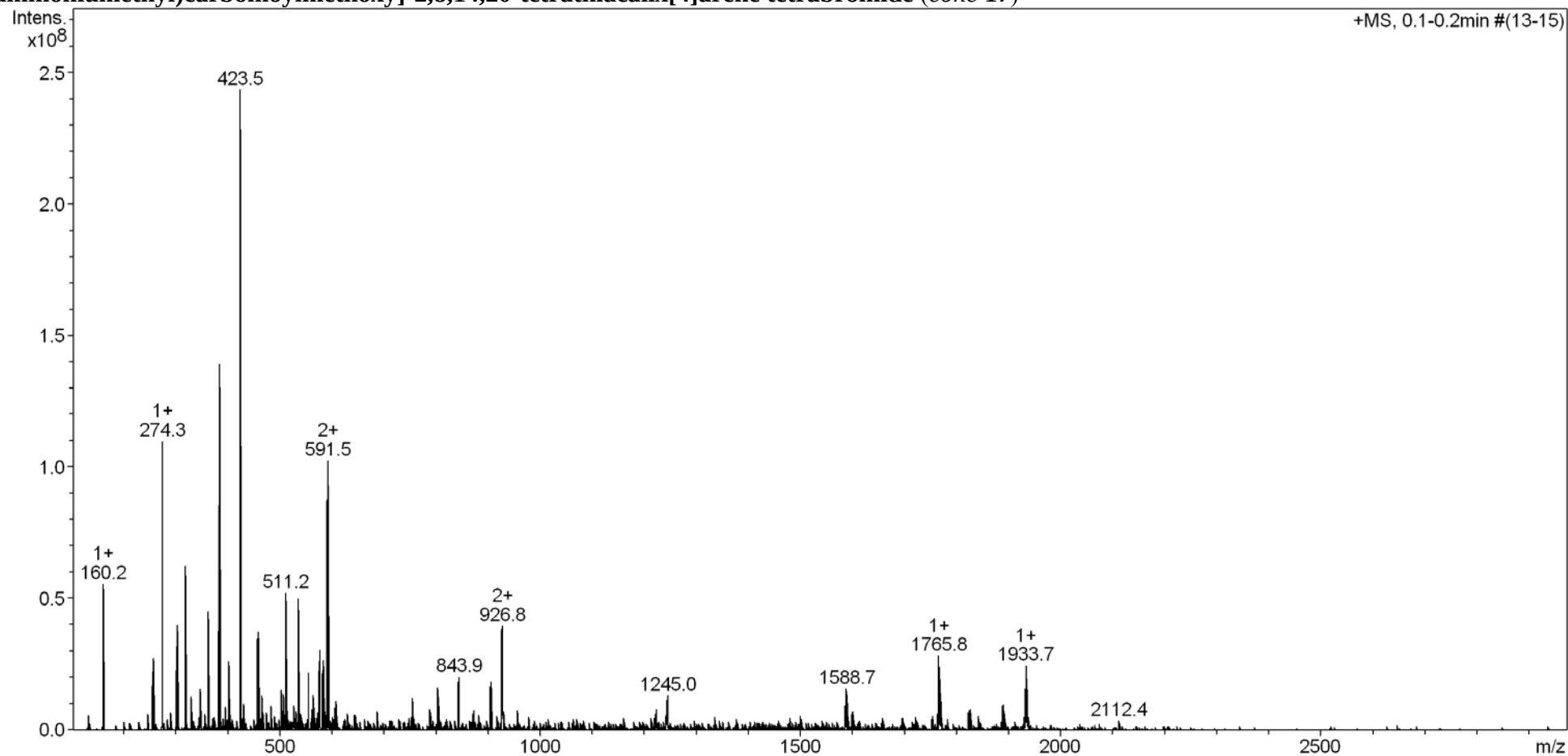


Fig. S105. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 18)

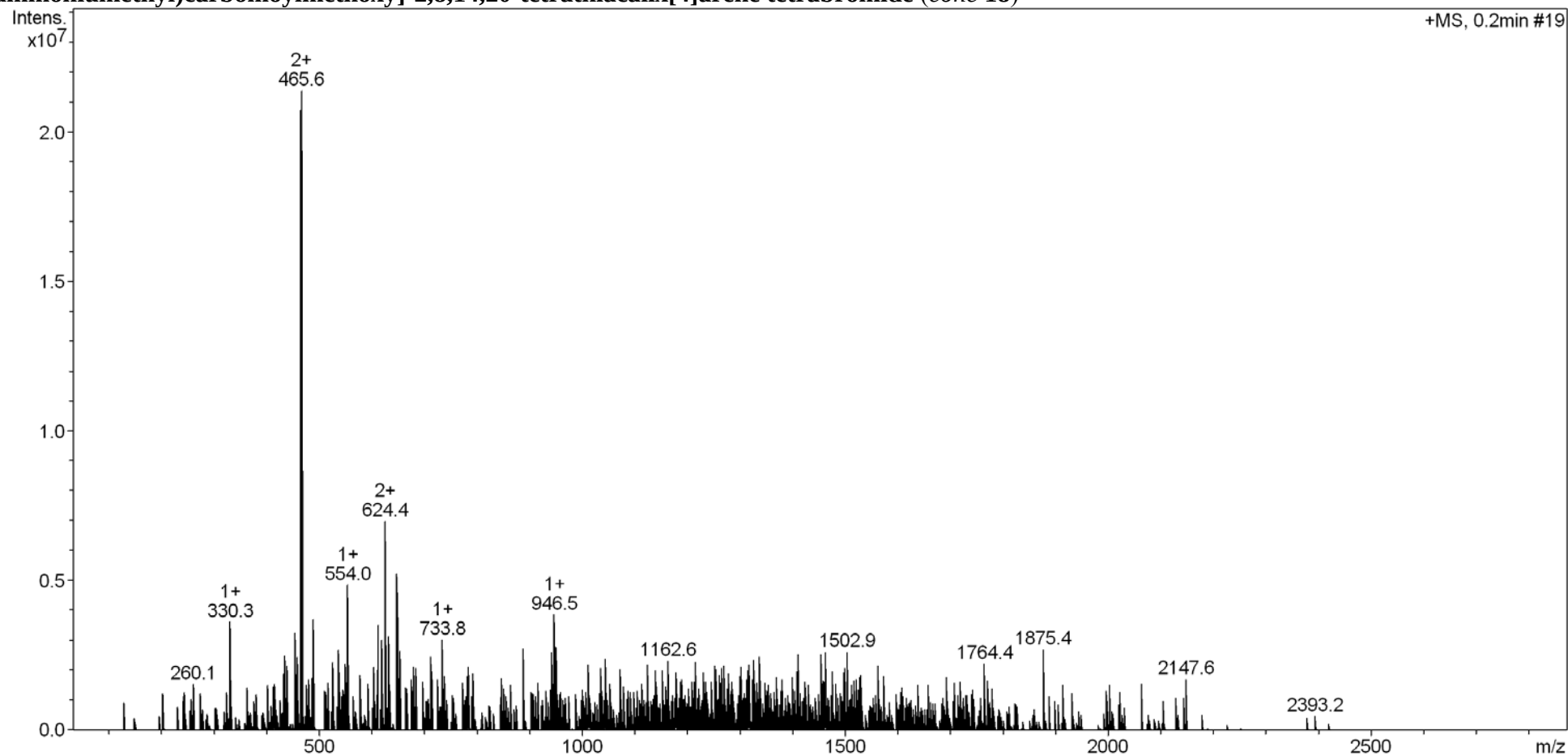


Fig. S106. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-alternate 22)

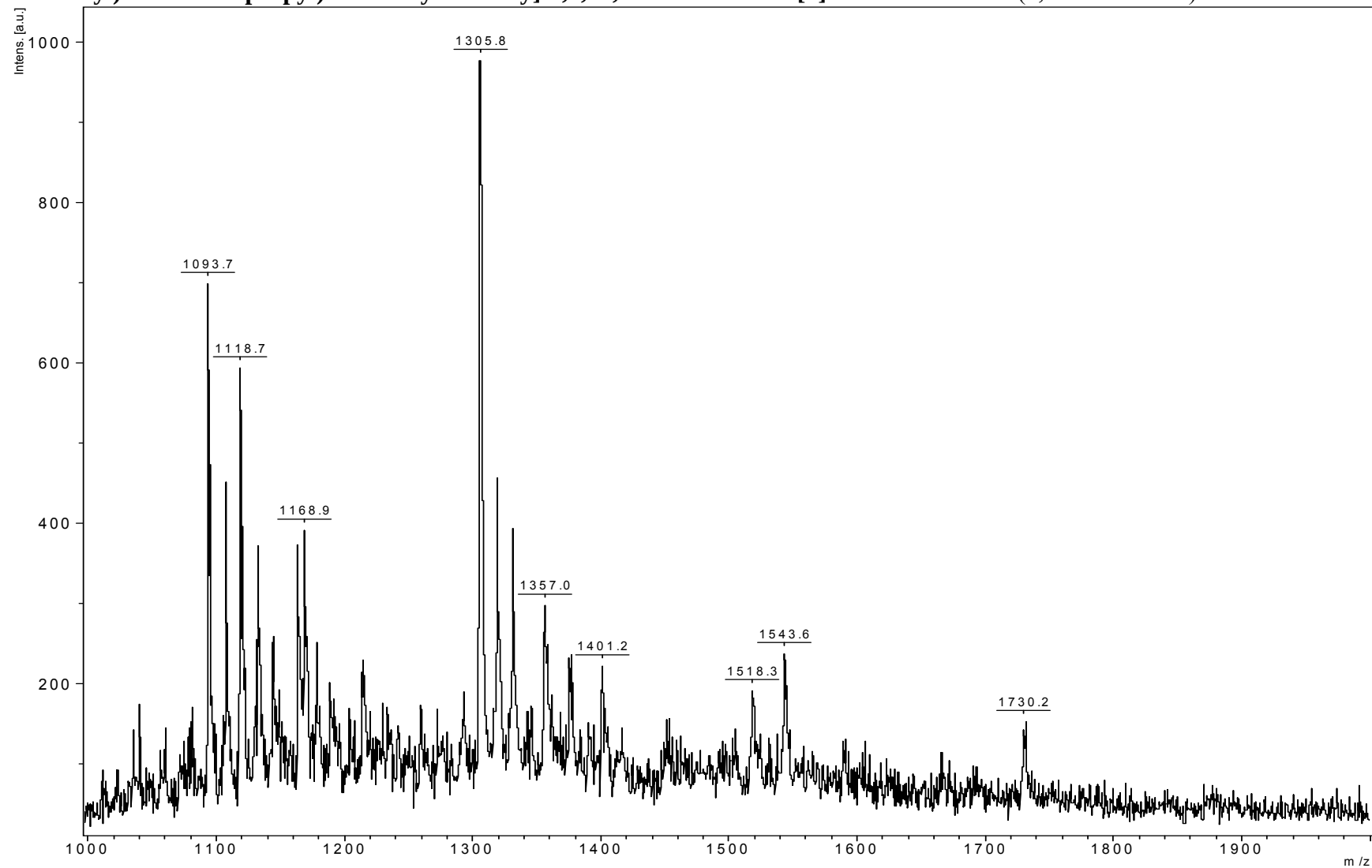


Fig. S107. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-*alternate* 23)

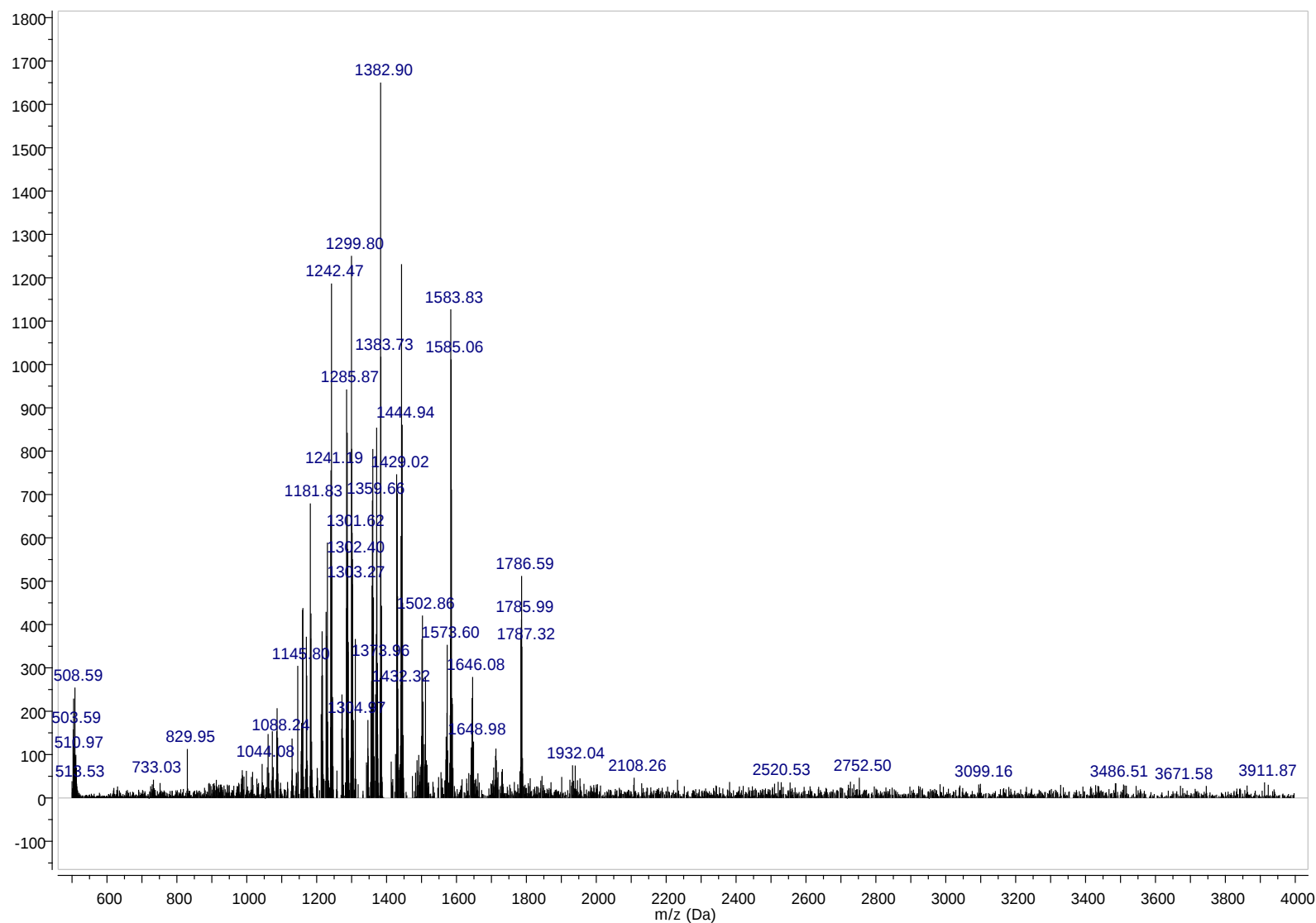


Fig. S108. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 24)

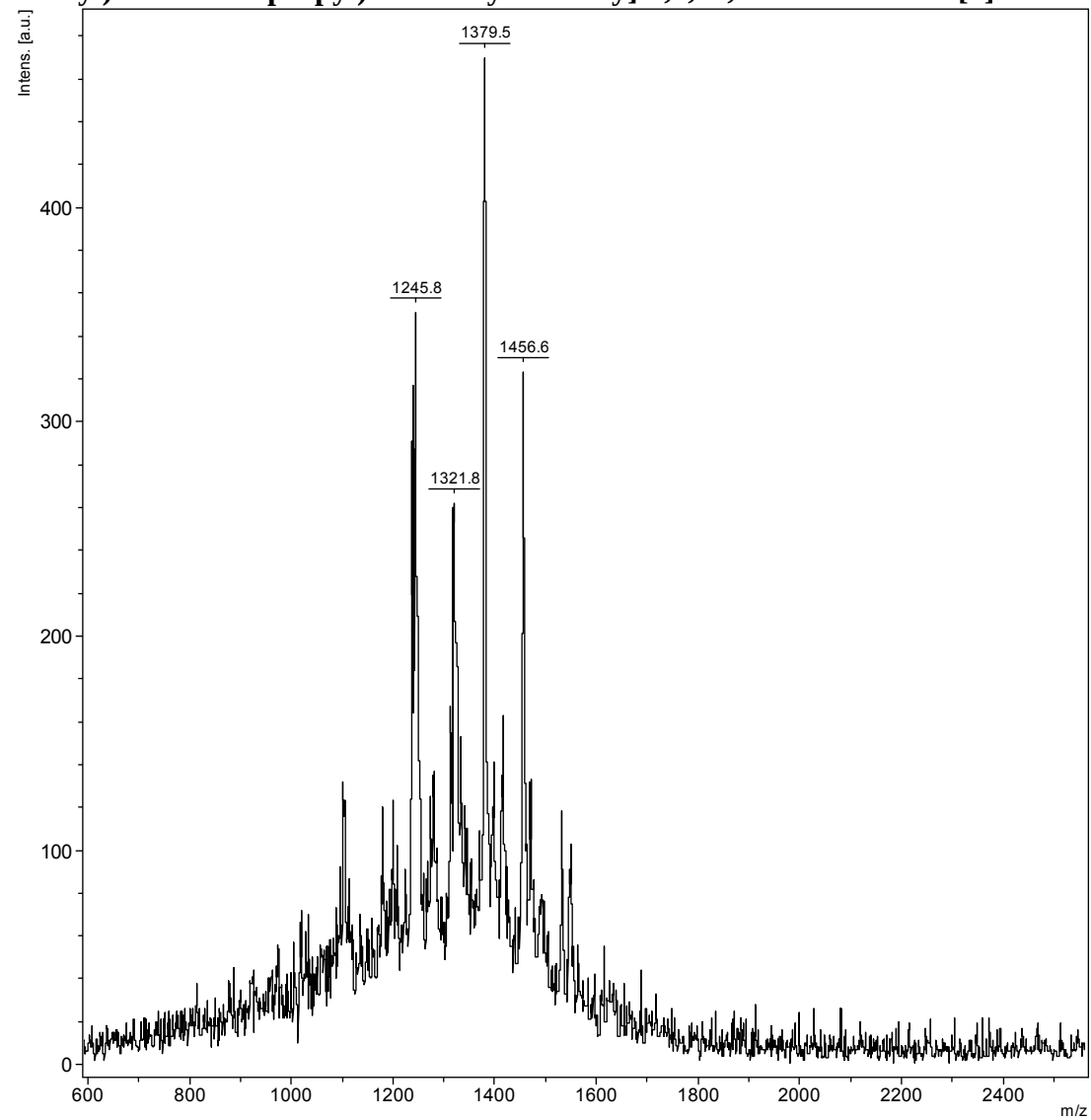


Fig. S109. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 25)

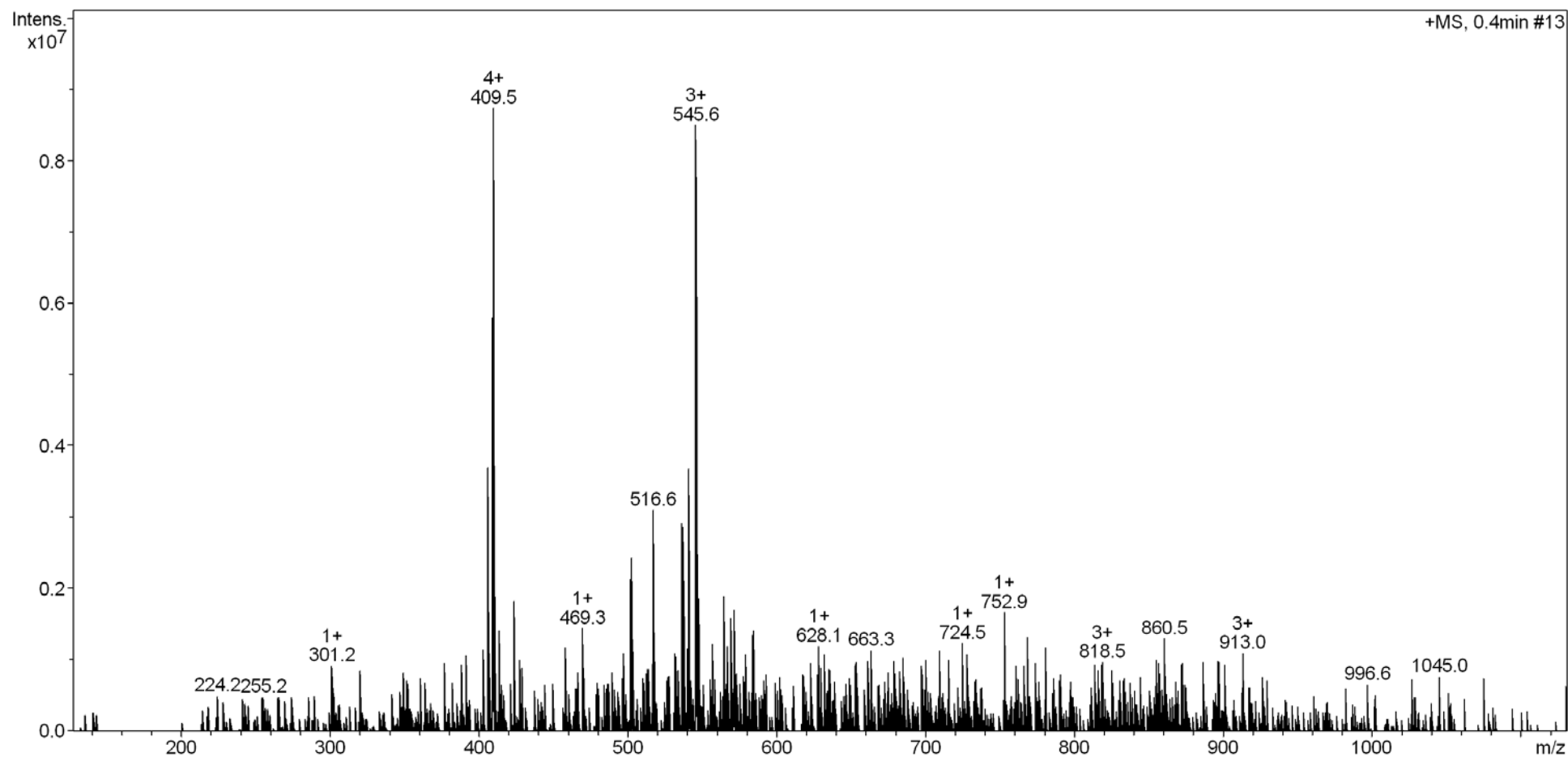


Fig. S110. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxycarbonylmethyl)-ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 26)

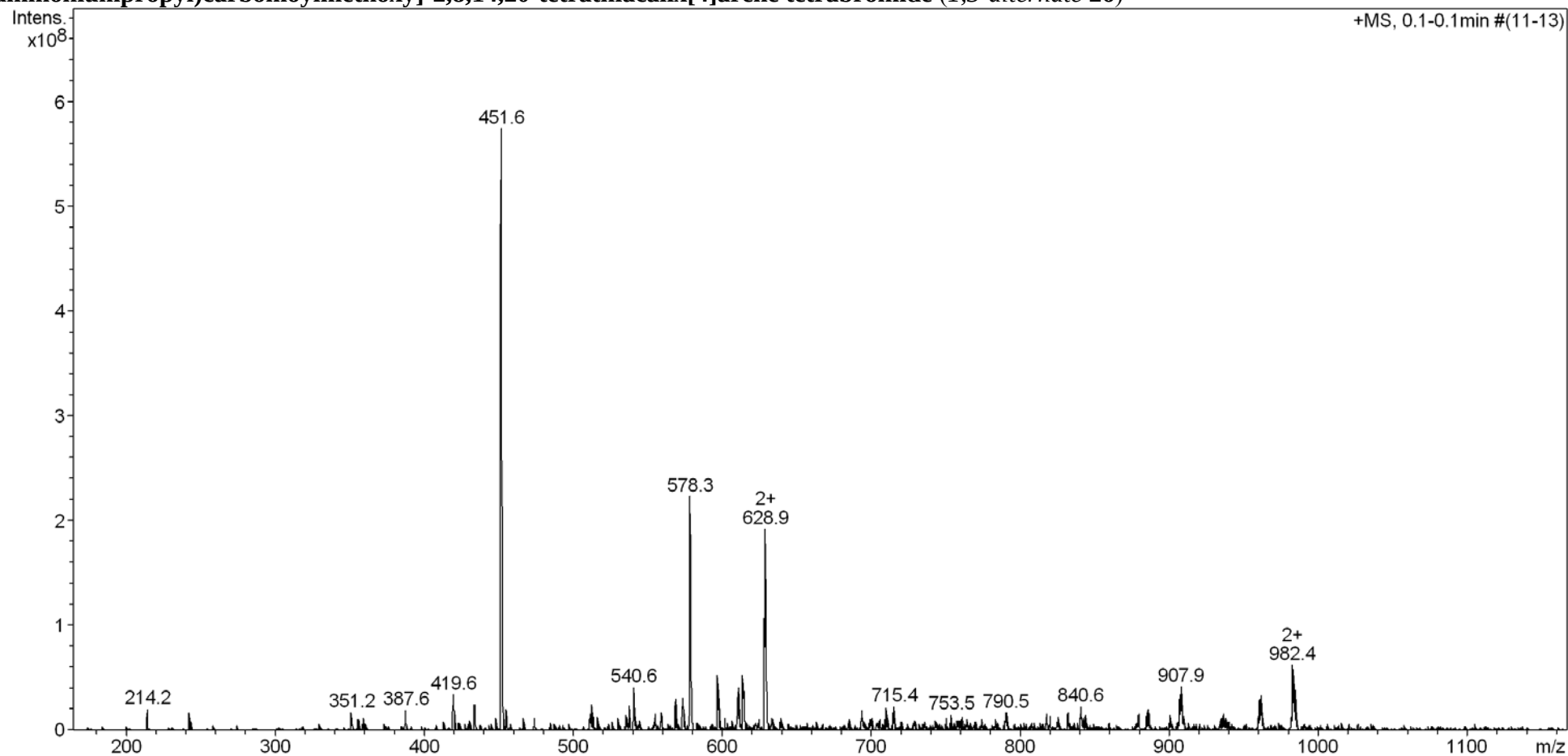


Fig. S111. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphtalimide}-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 30)

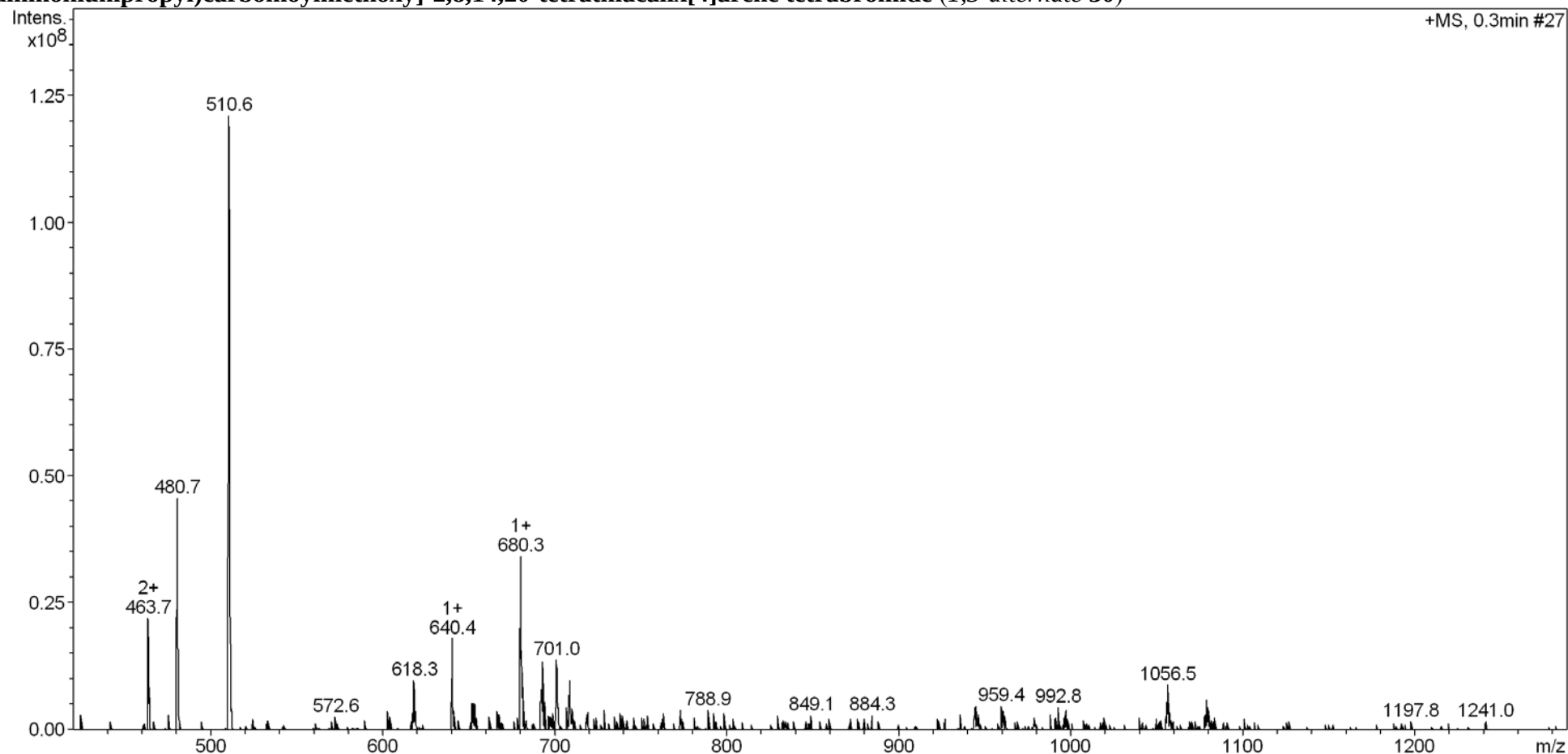


Fig. S112. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-alternate 31)

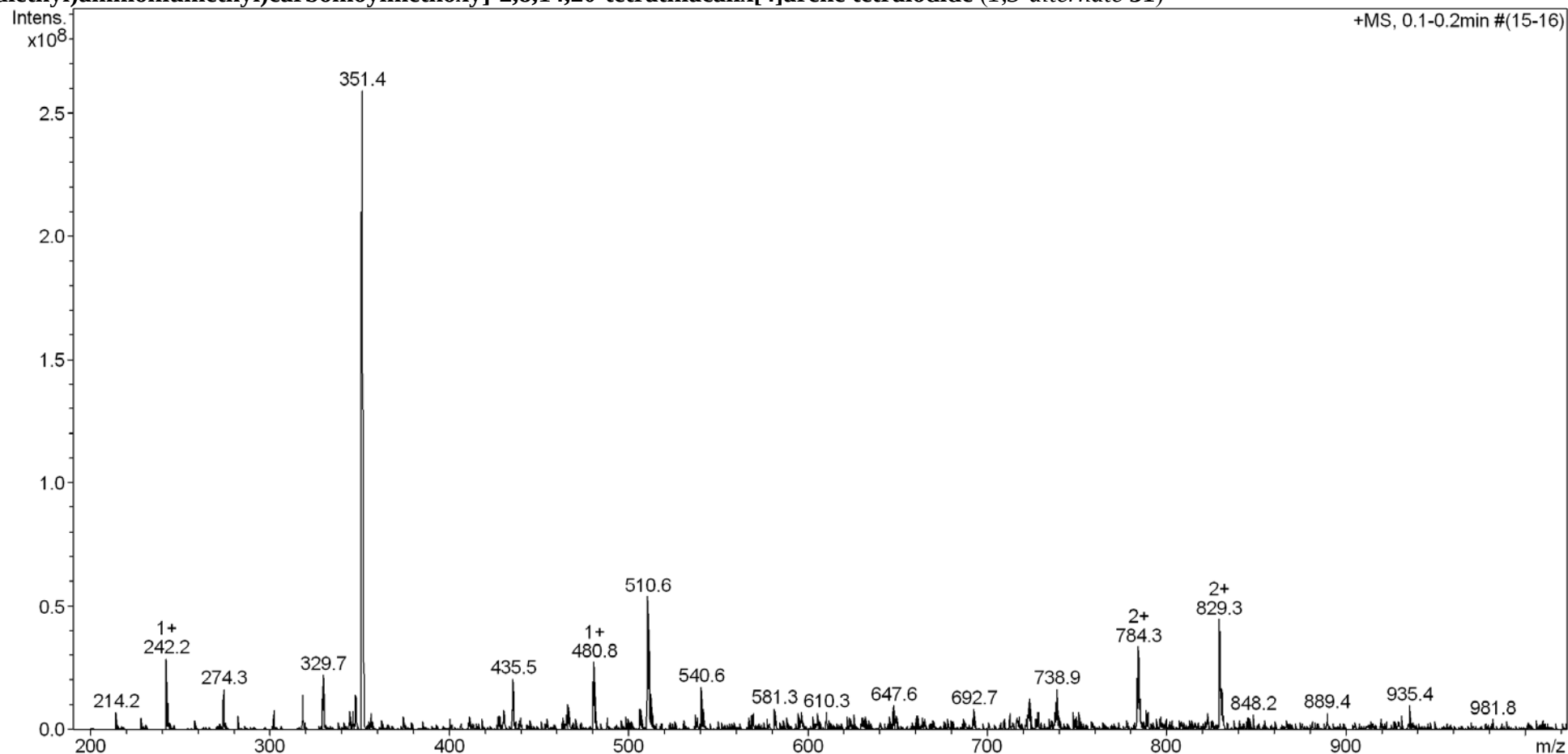


Fig. S113. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-alternate 32)

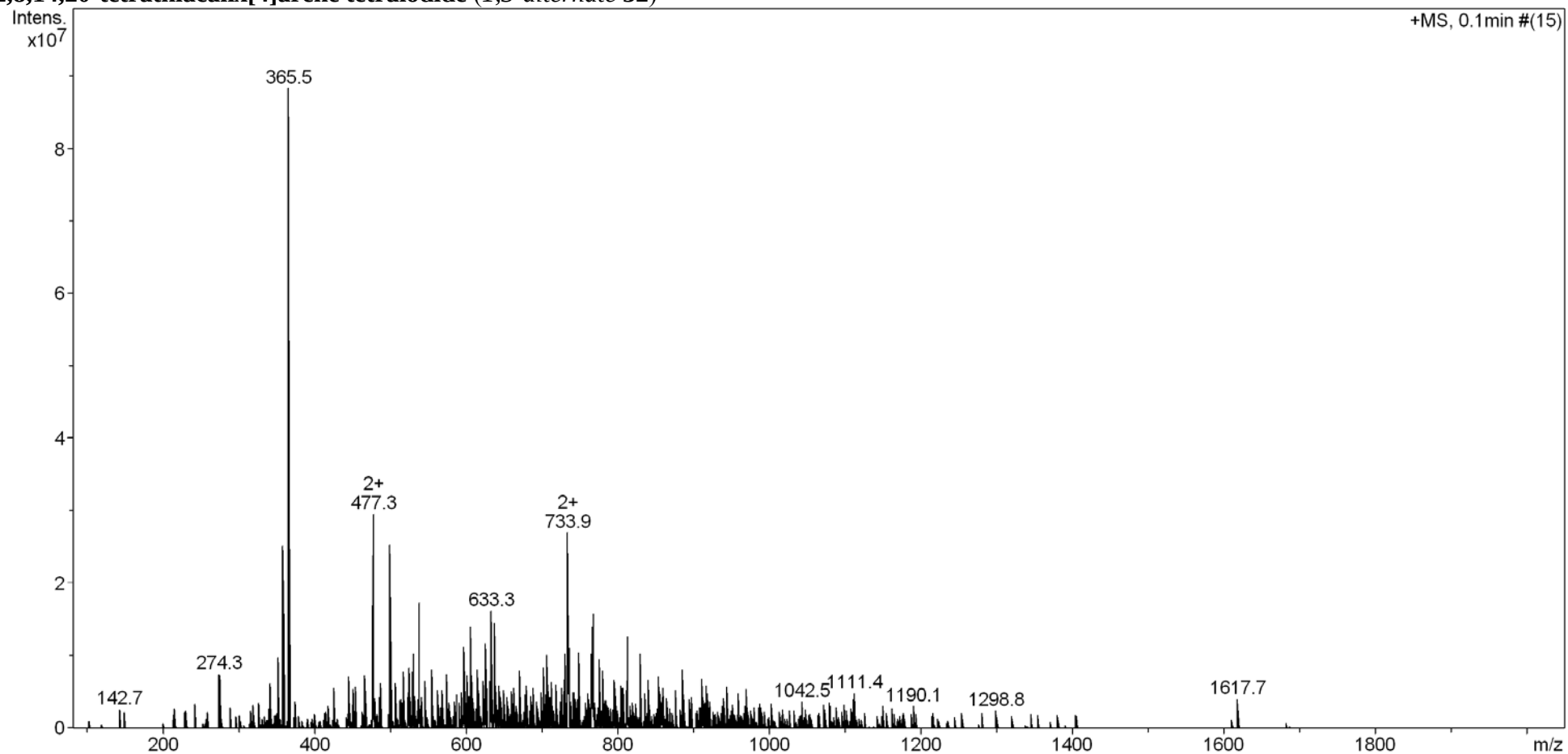


Fig. S114. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(benzyl)ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-alternate 33)

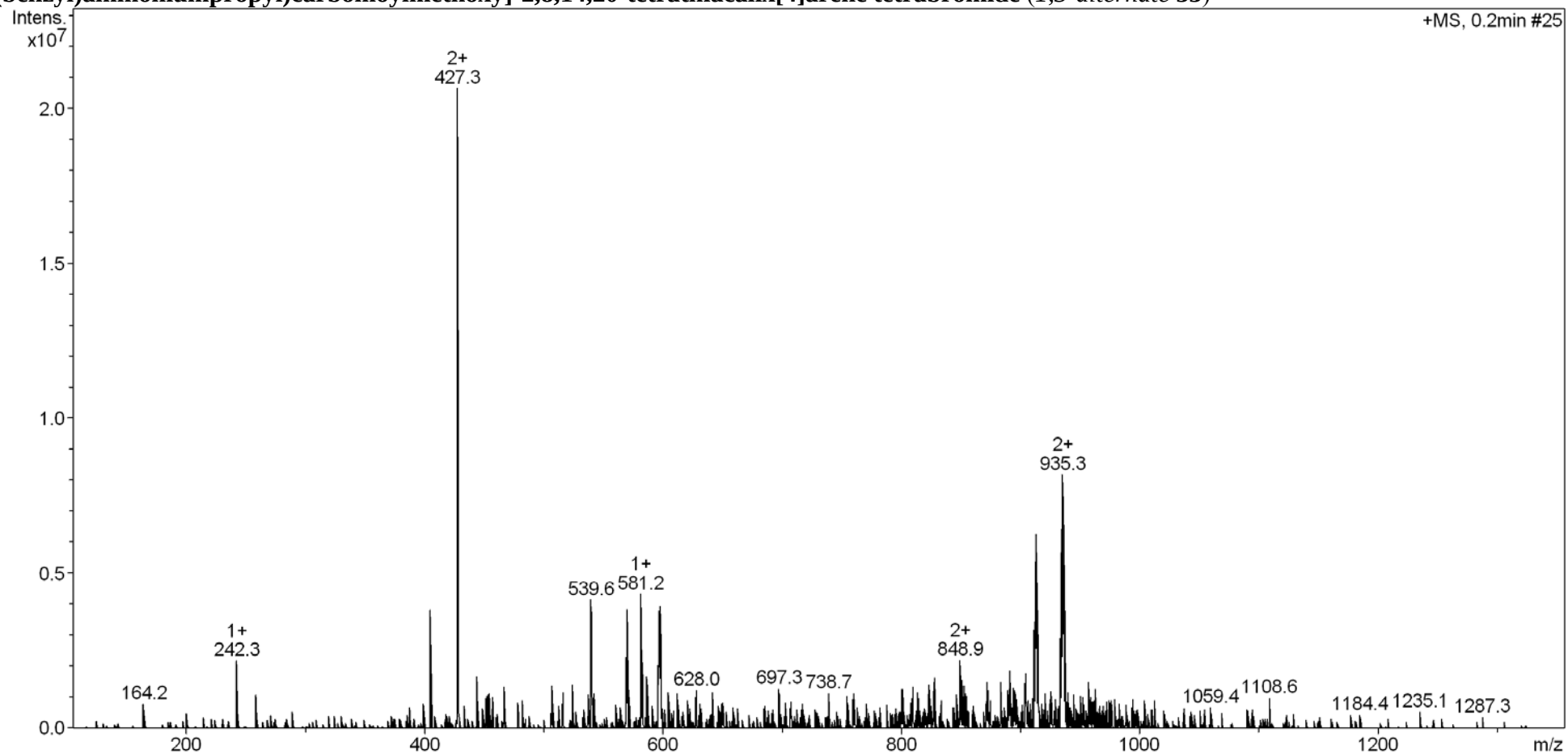


Fig. S115. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 34)

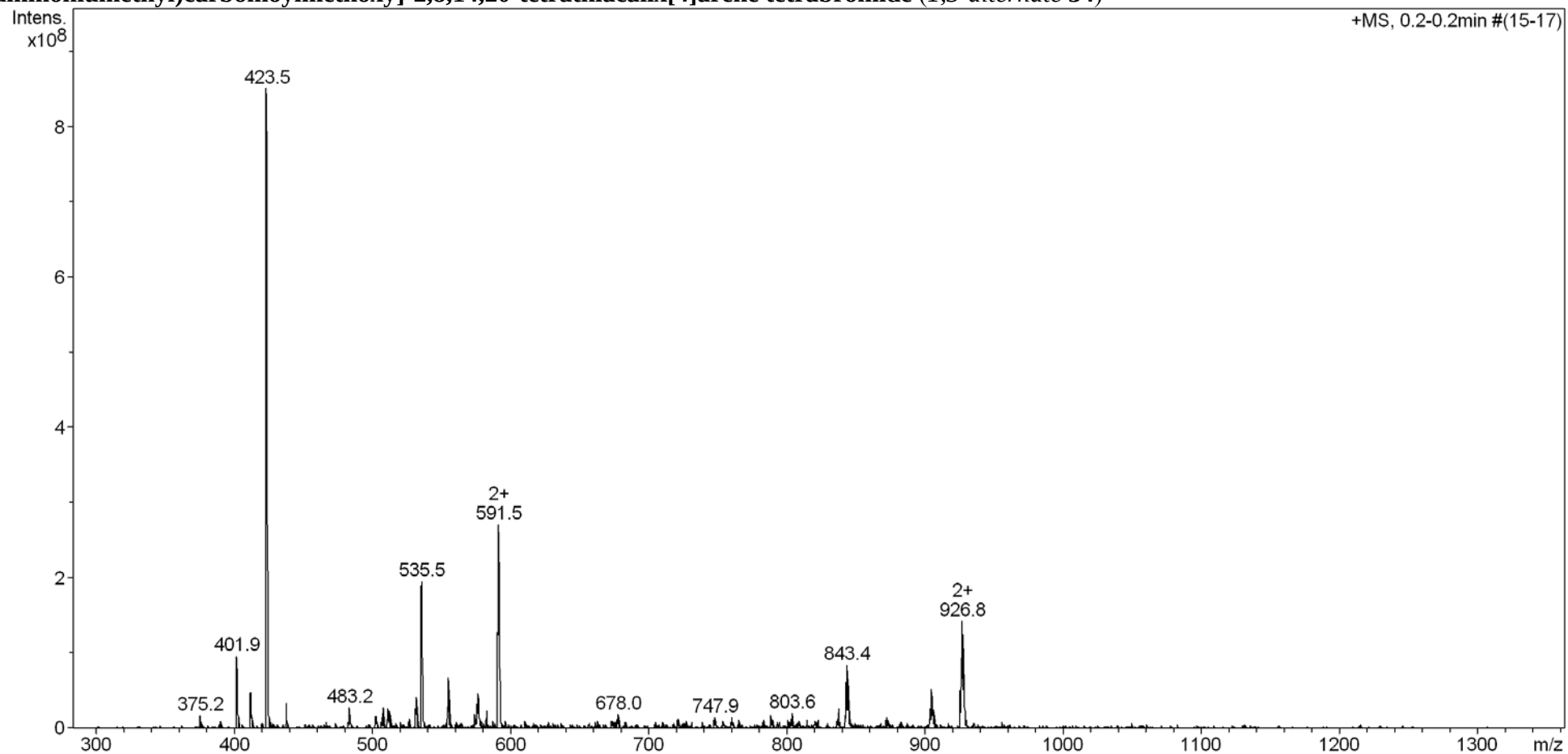


Fig. S116. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 35)

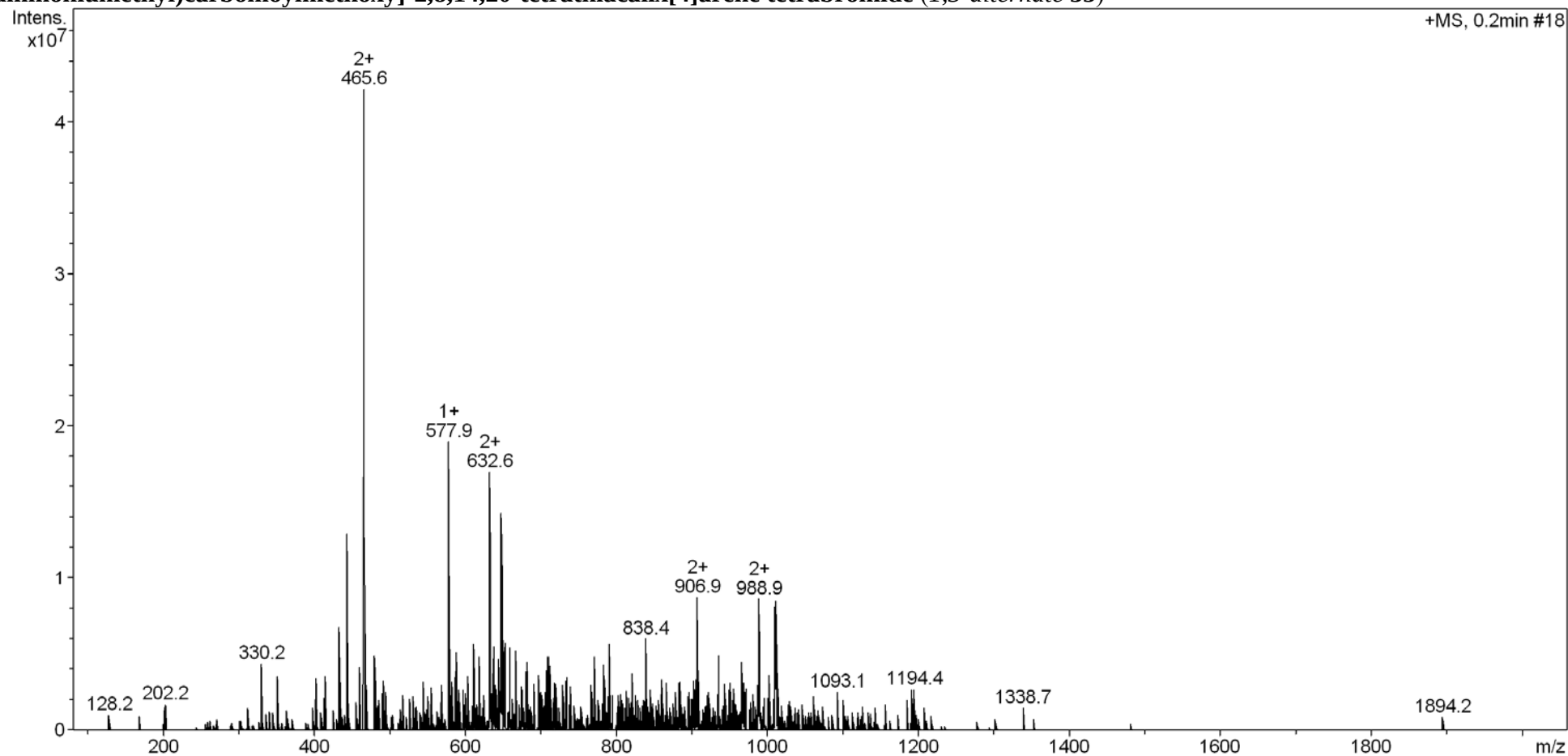


Fig. S117. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 39)

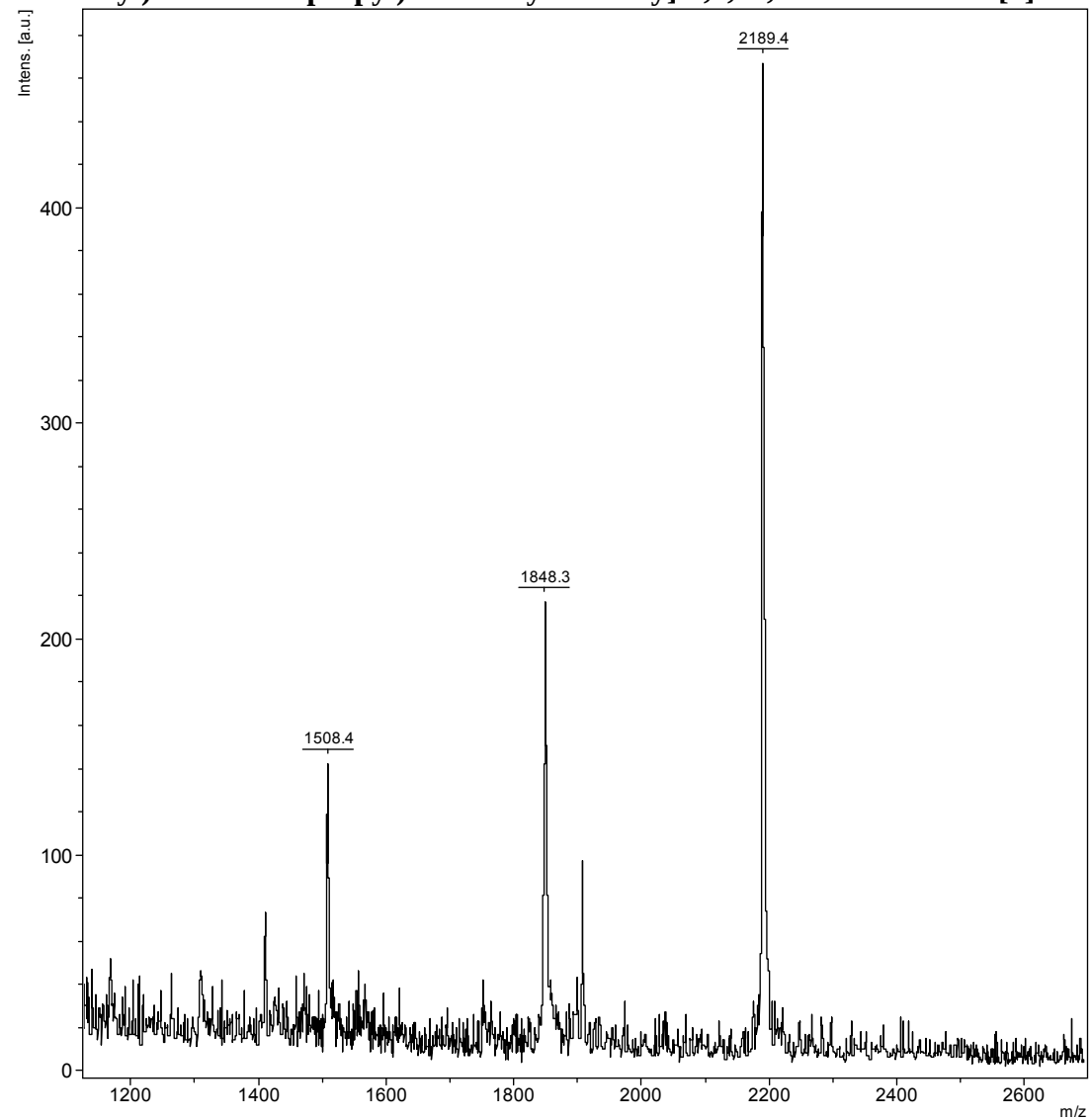


Fig. S118. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 40)

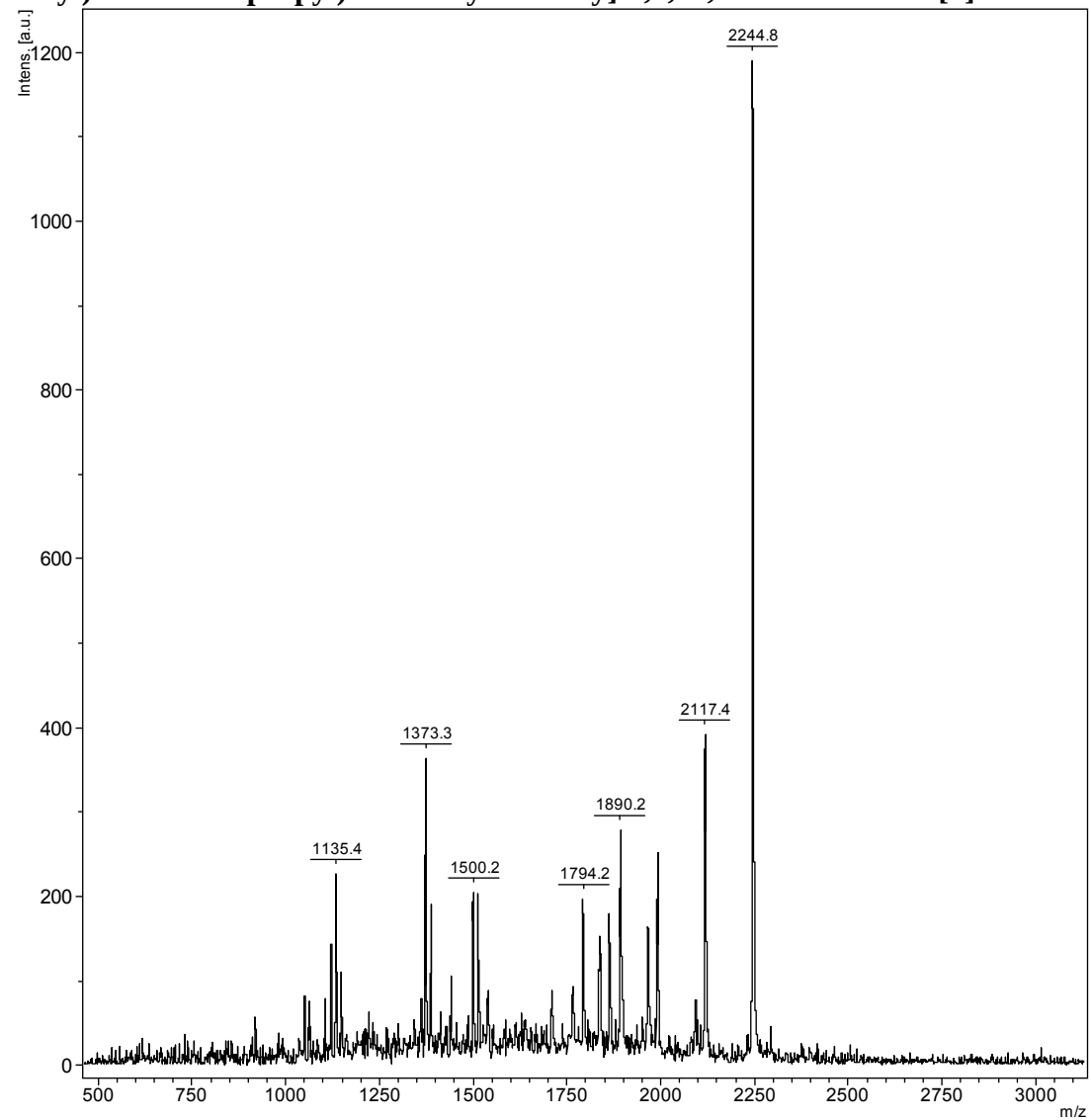


Fig. S119. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 41)

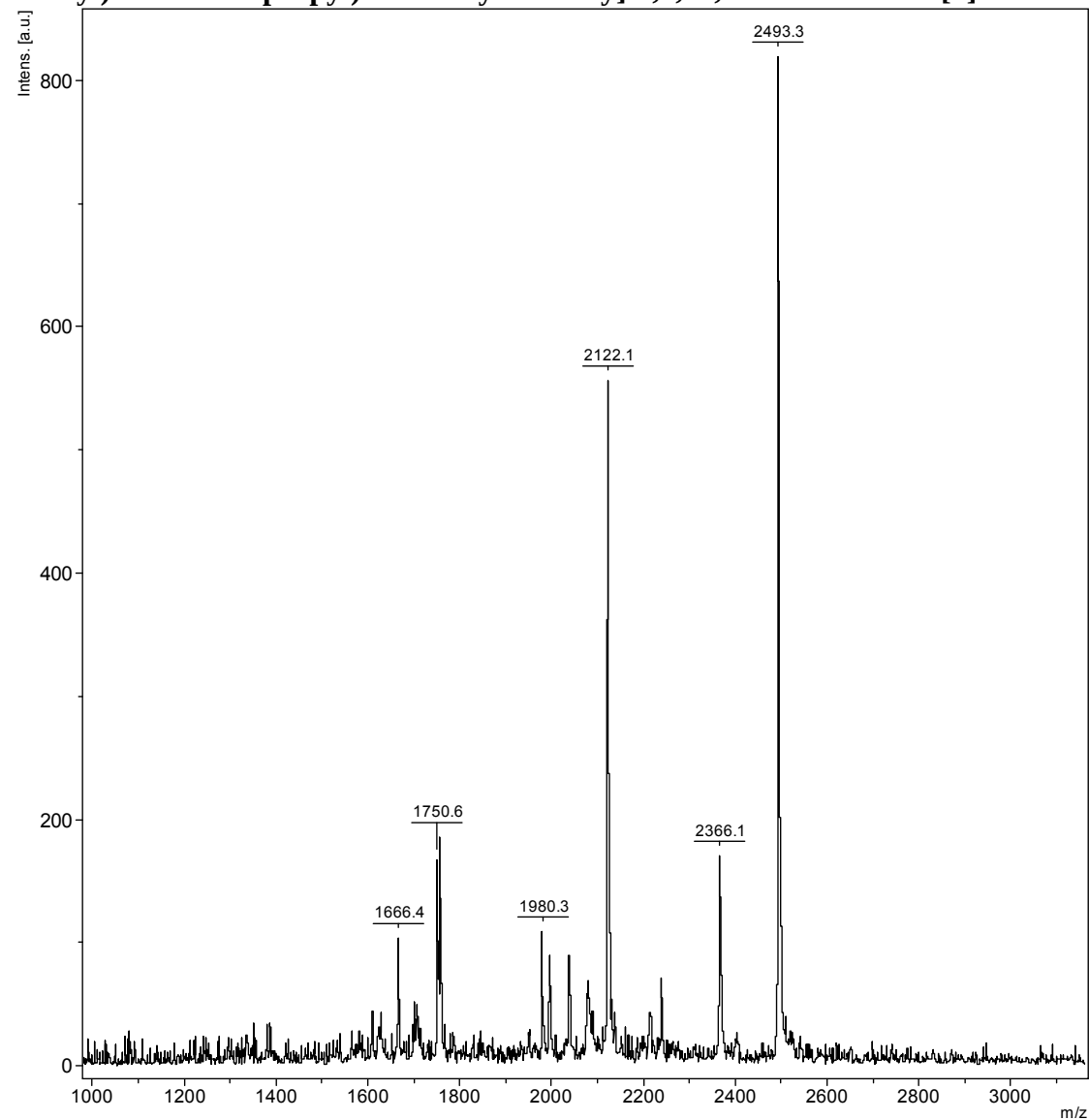


Fig. S120. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)ammoniumpropyl)-carbomoyl-methoxy]-2,8,14,20-tetrathiocalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 42)

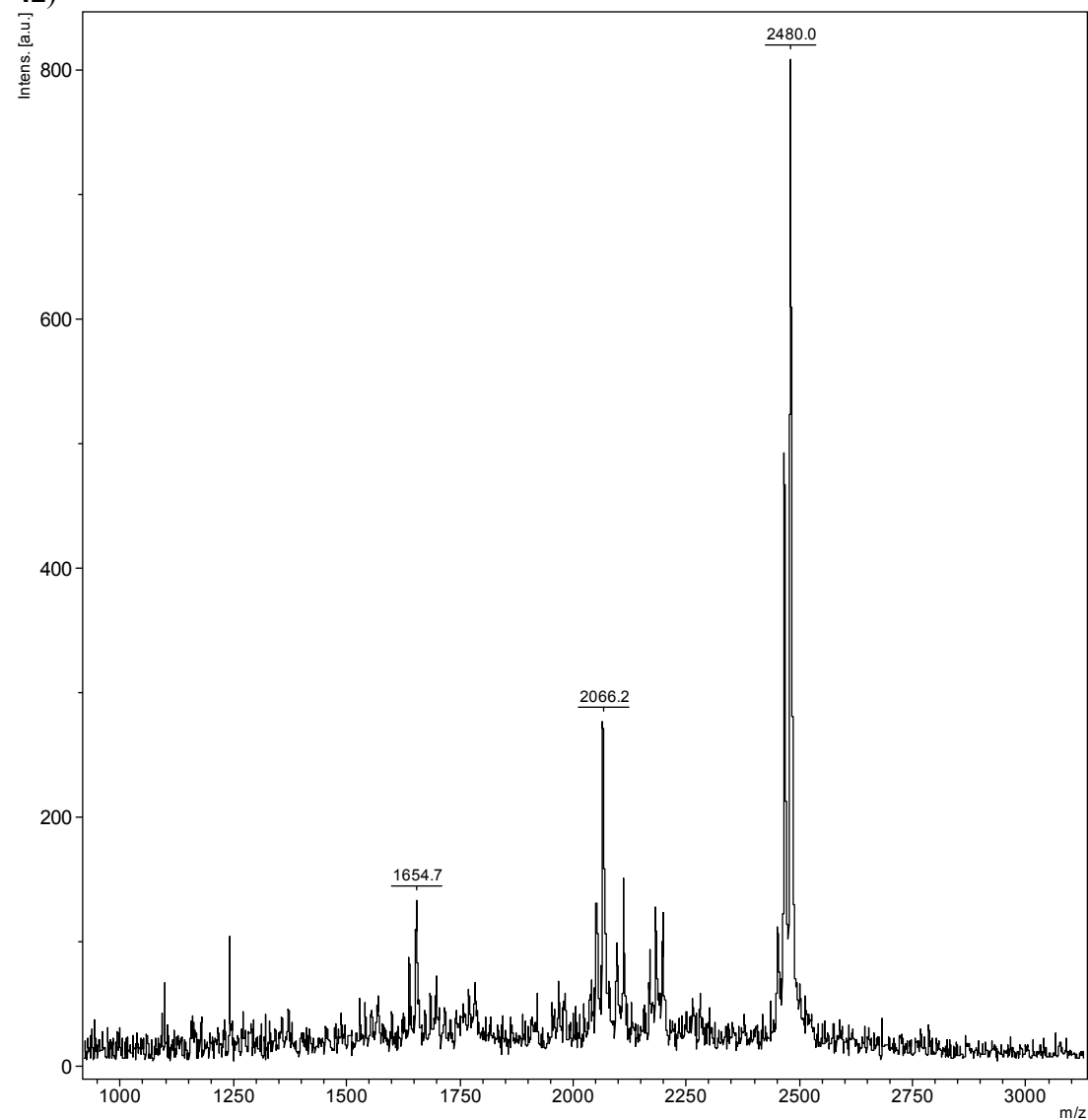
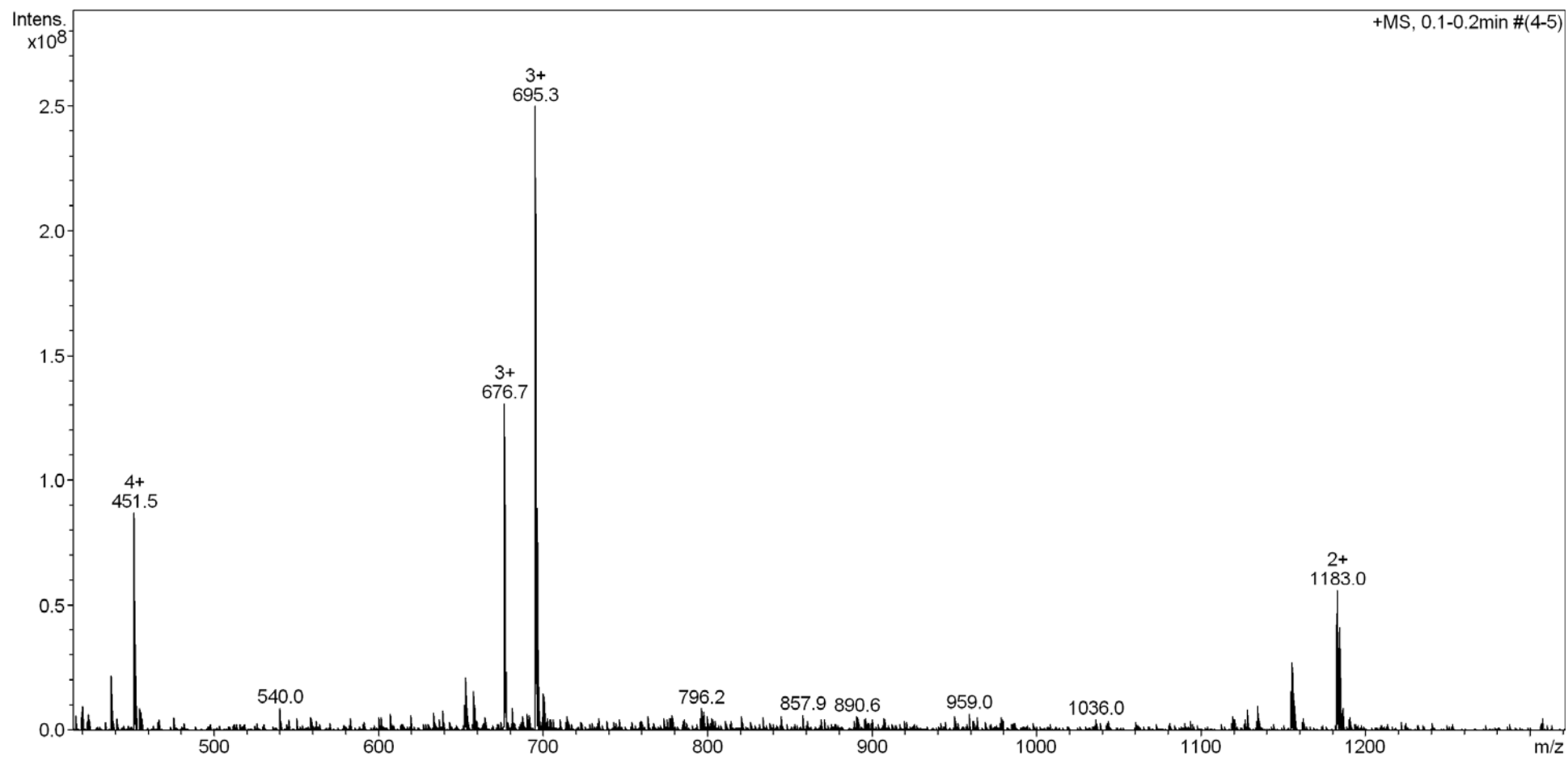


Fig. S121. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxycarbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 43)



**Fig. S122. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-
{(ethoxycarbonylmethyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene
tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 44)**

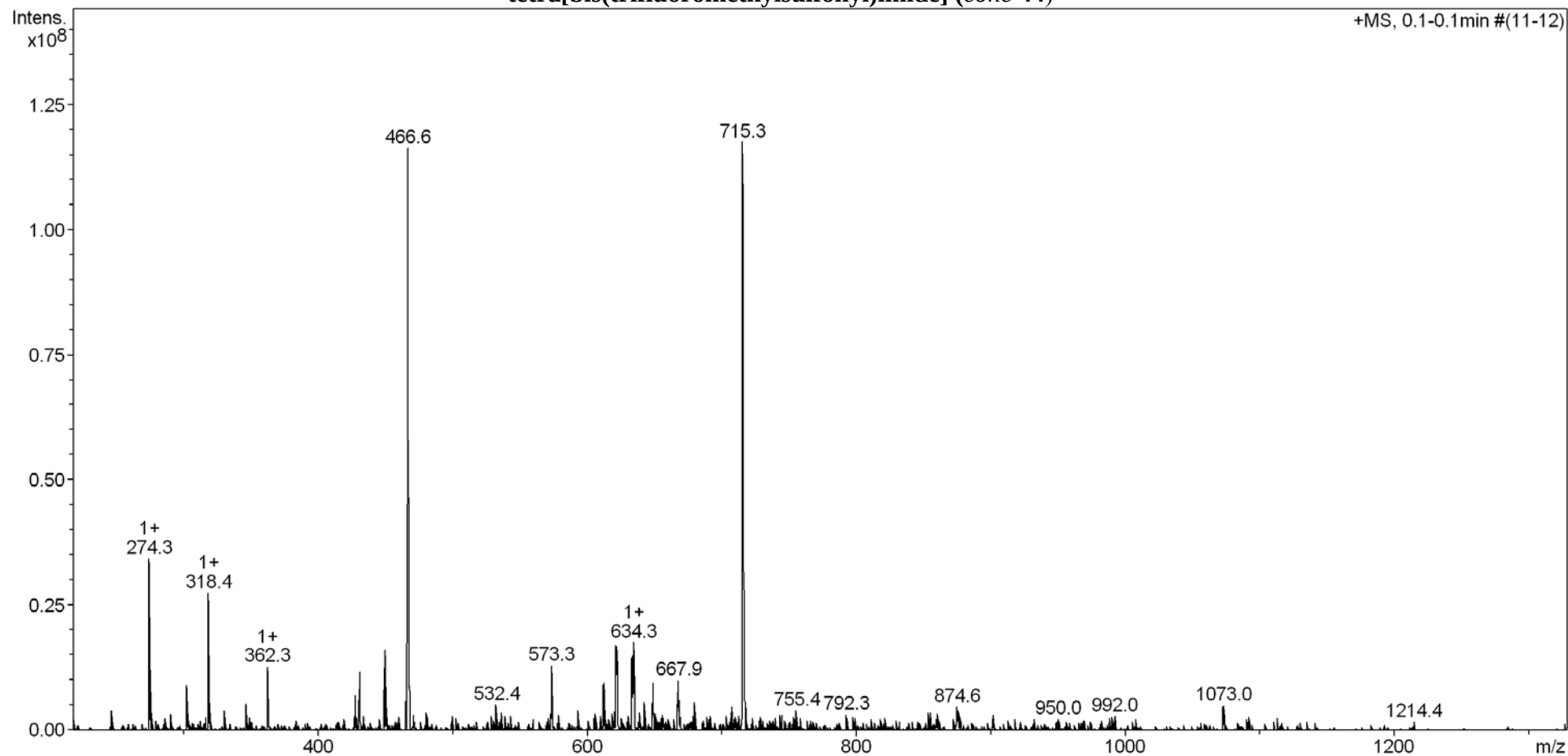


Fig. S123. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{[ethoxycarbonylmethyl]-amidocarbonylmethyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 45)

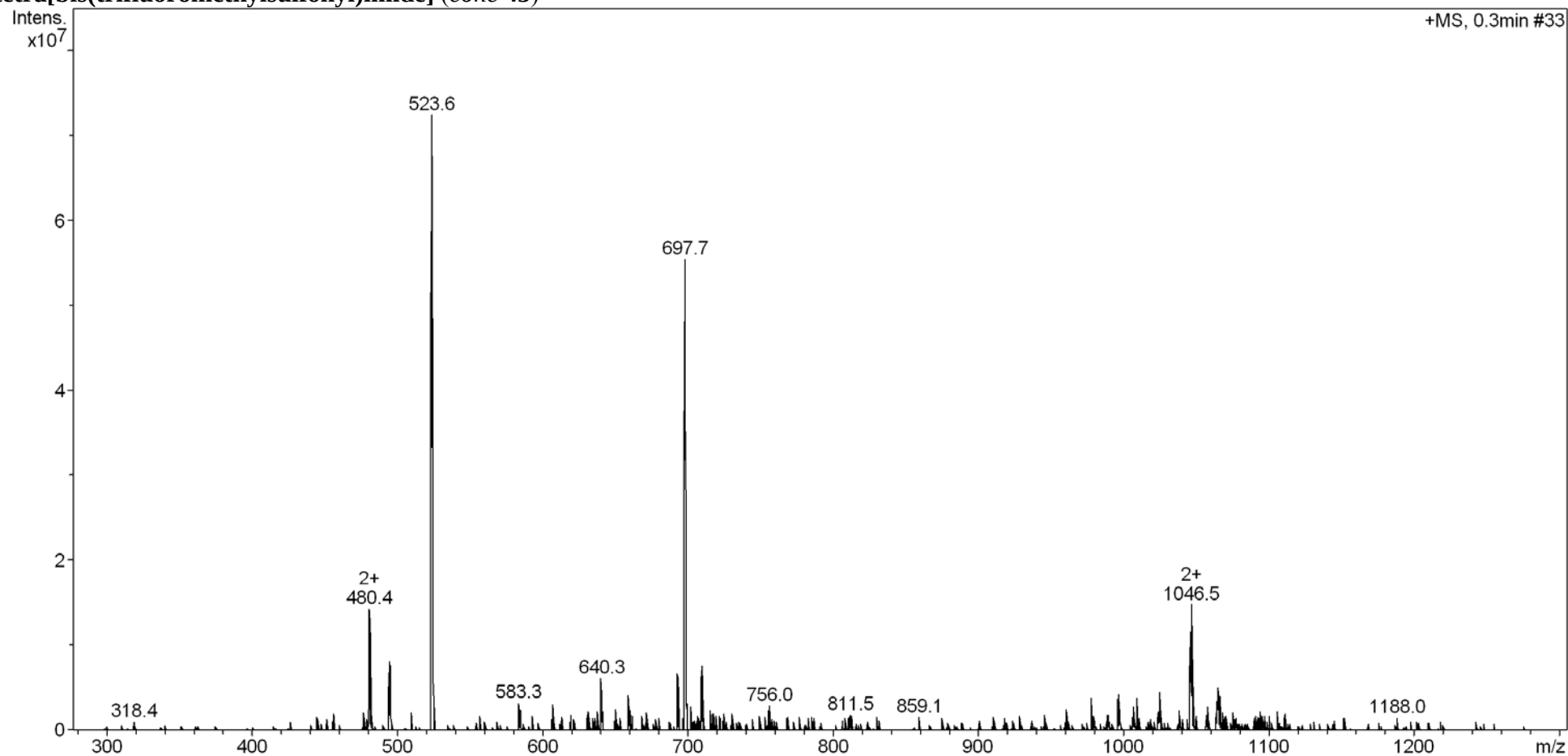


Fig. S124. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 46)

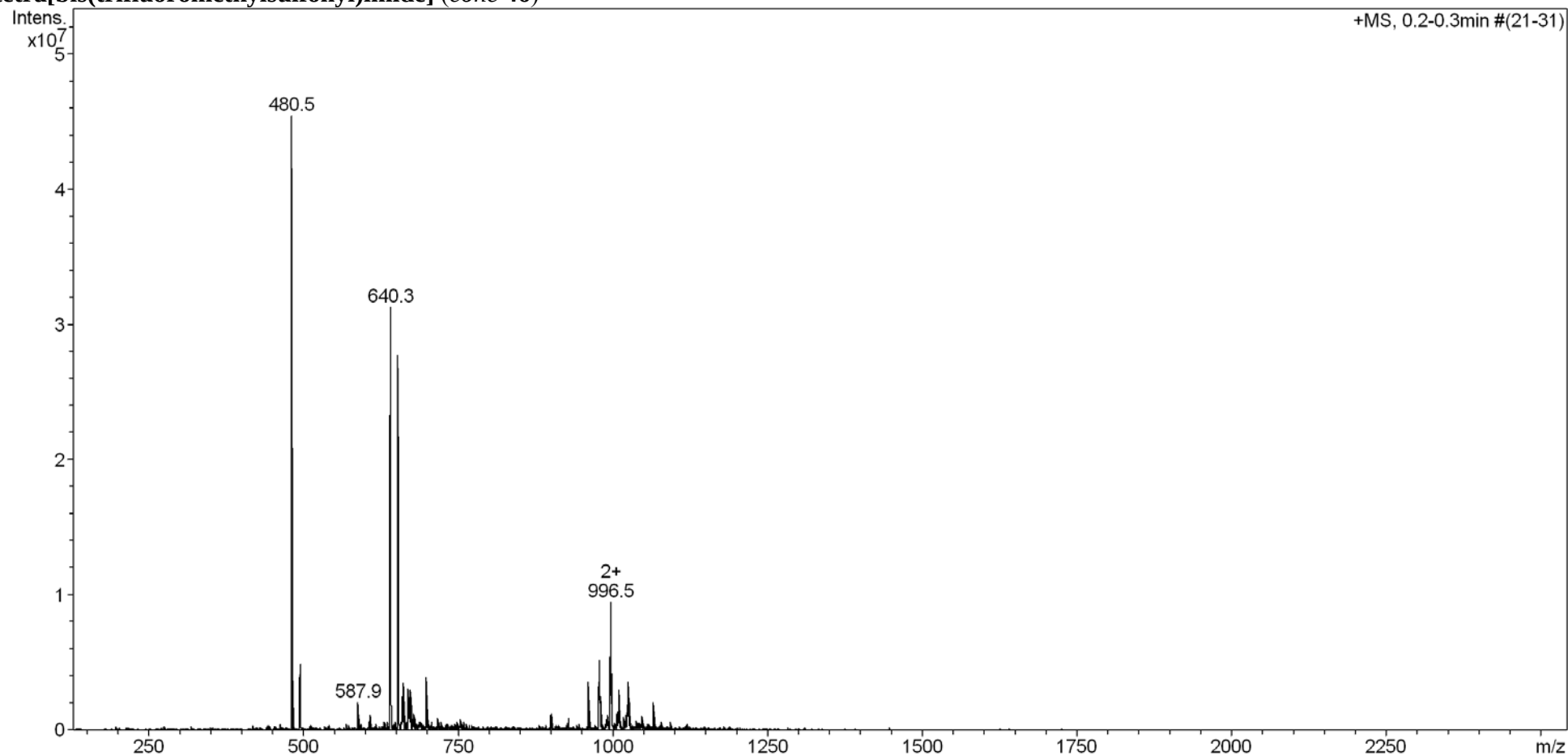


Fig. S125. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide}ammoniumpropyl)-carbomoxymethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 47*)

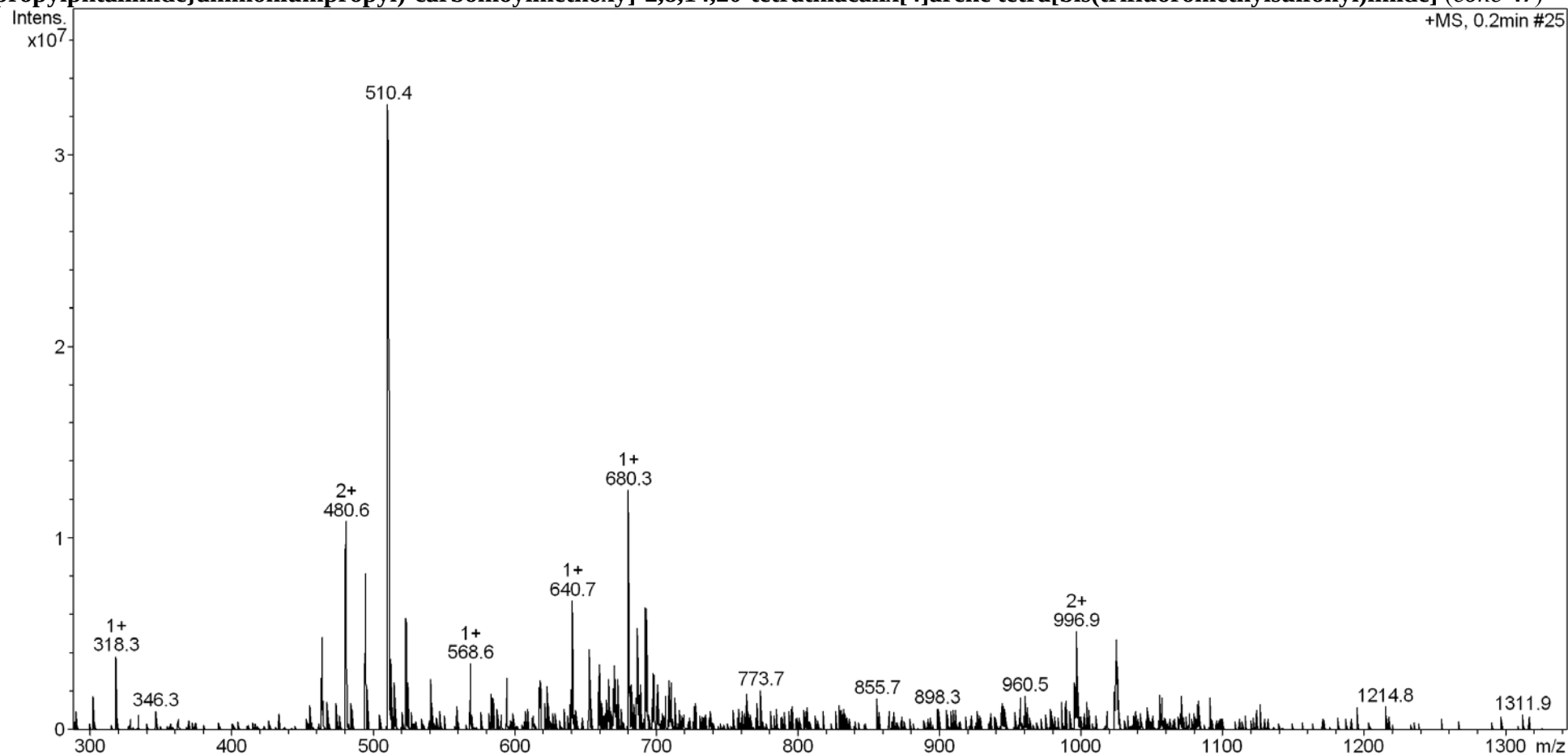


Fig. S126. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 48)

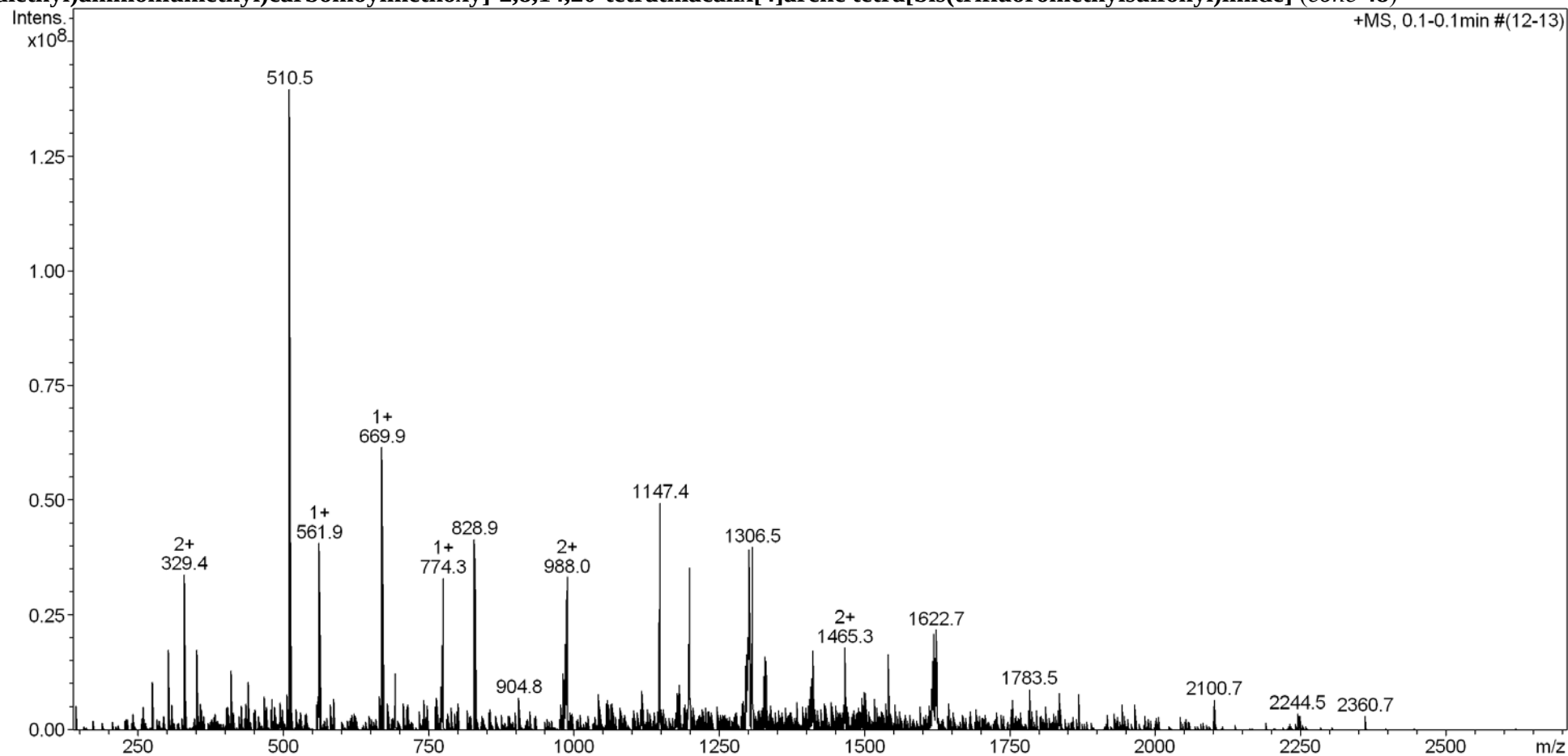


Fig. S127. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 49)

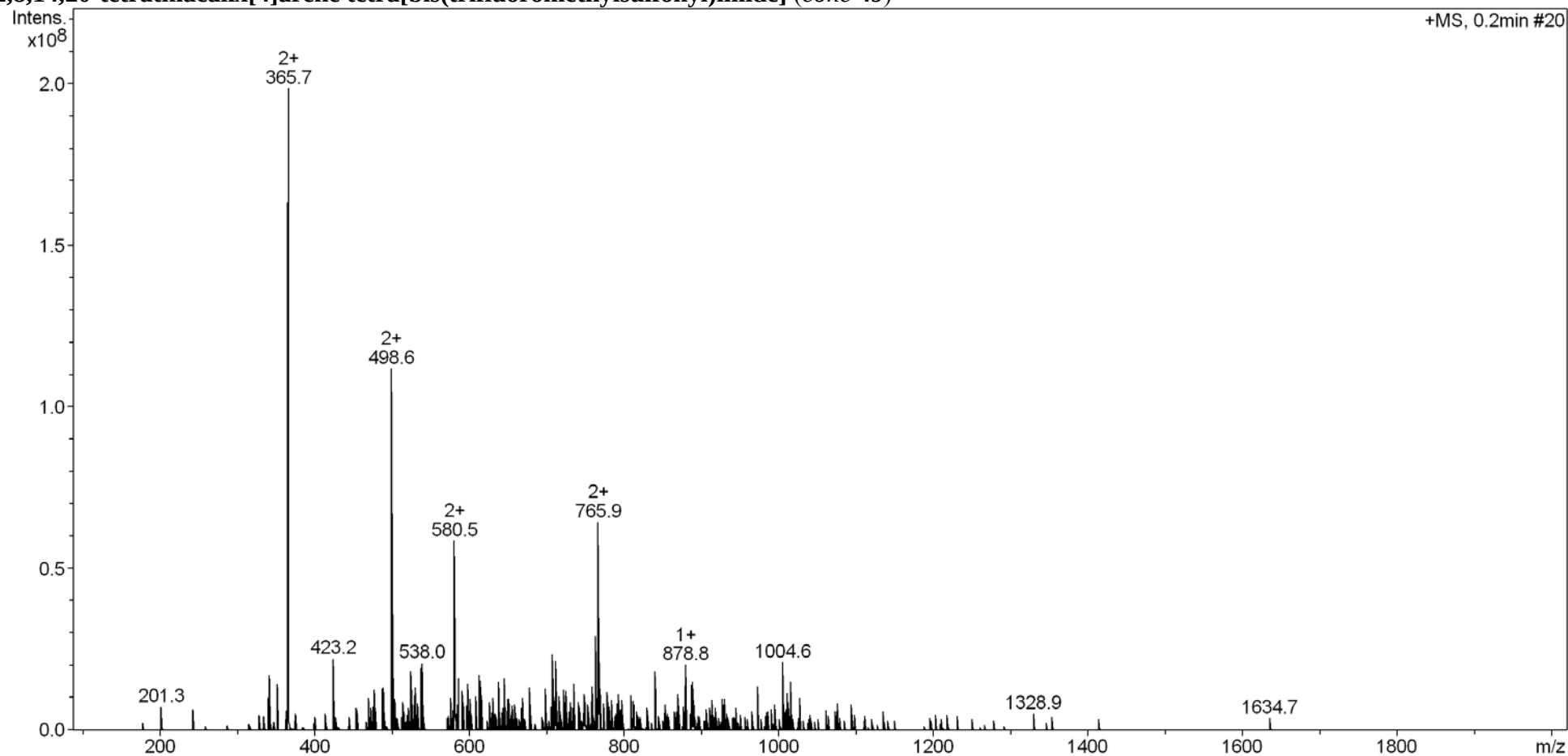


Fig. S128. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 50)

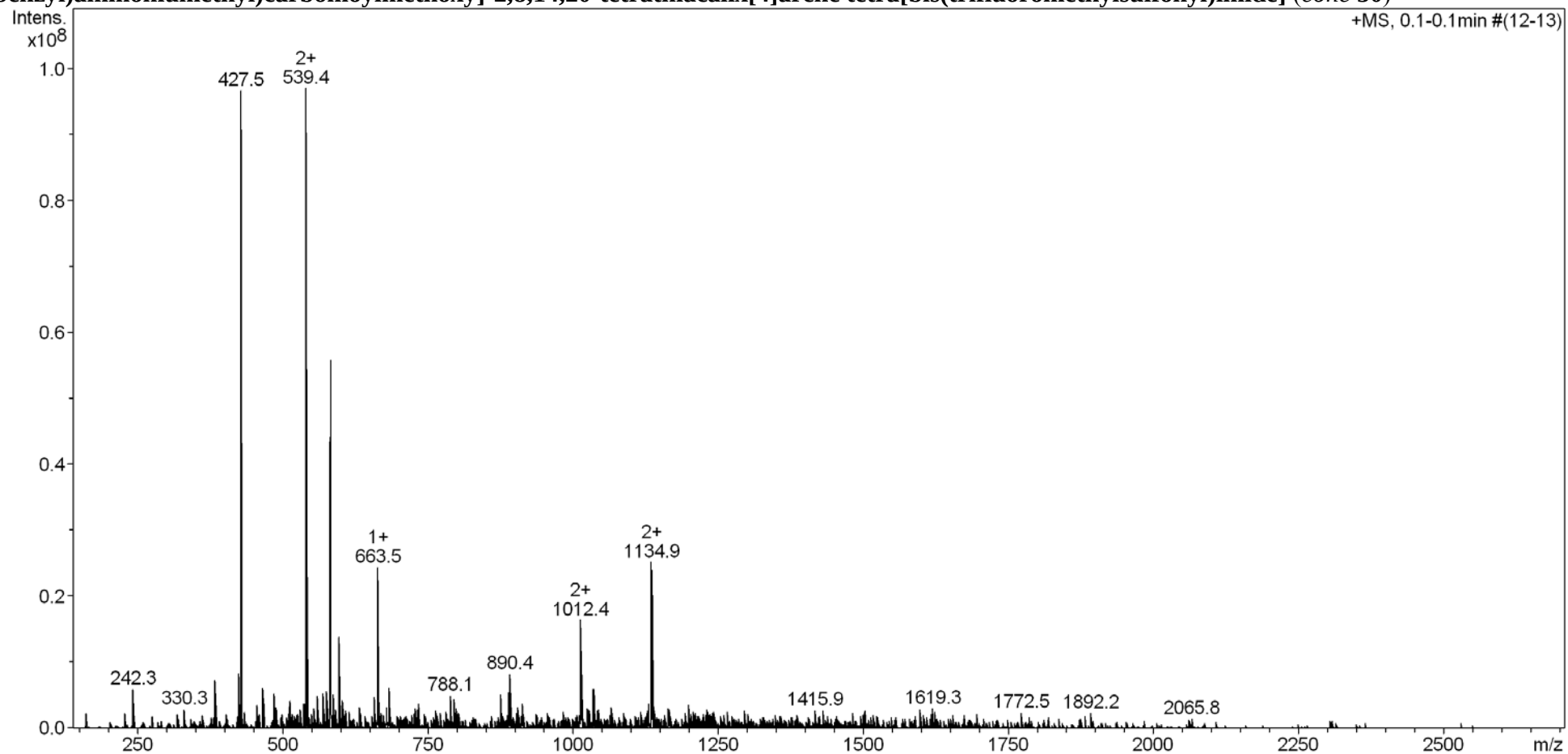


Fig. S129. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (cone 51)

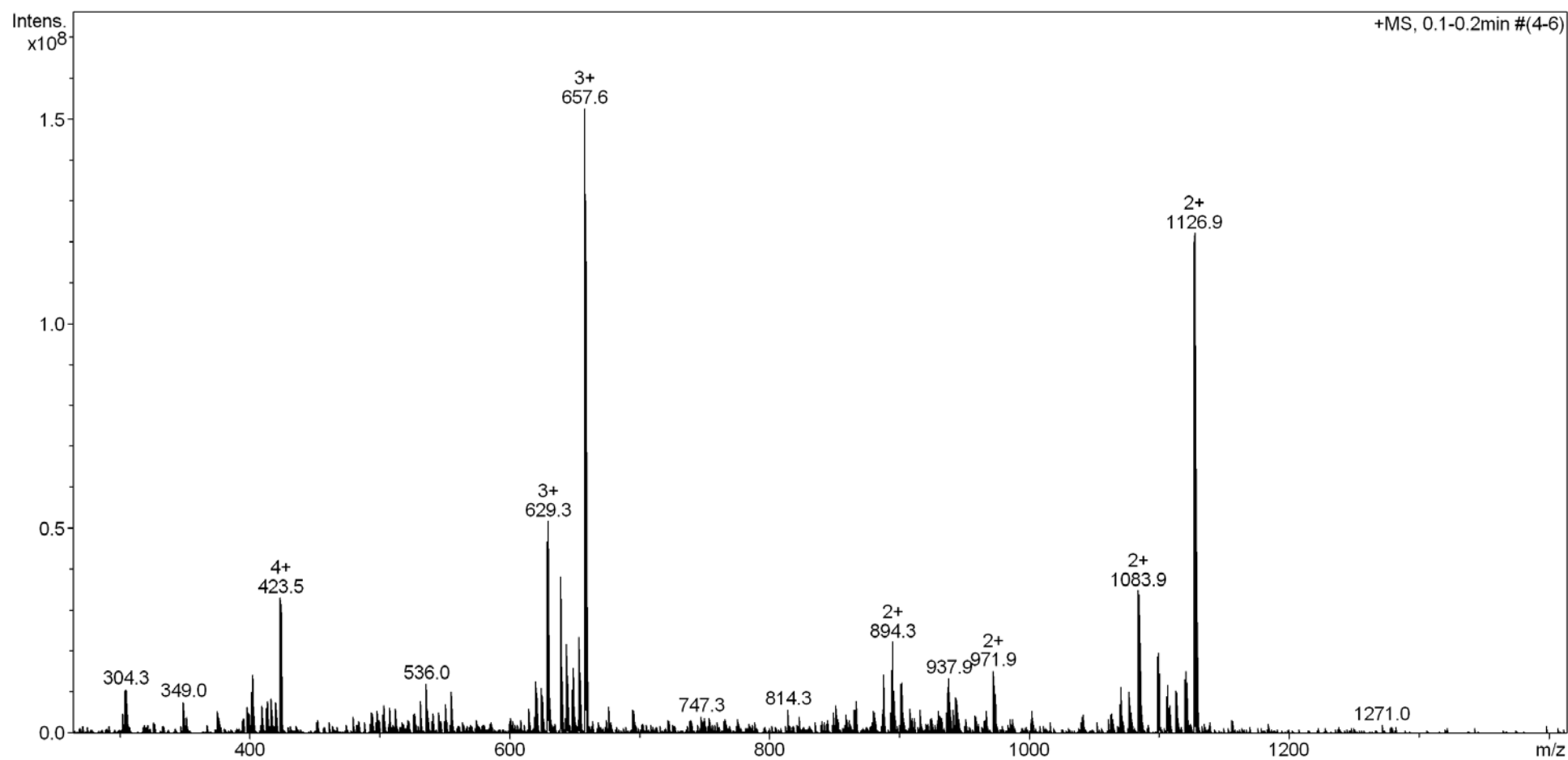


Fig. S130. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)-ammoniummethyl) carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 52)

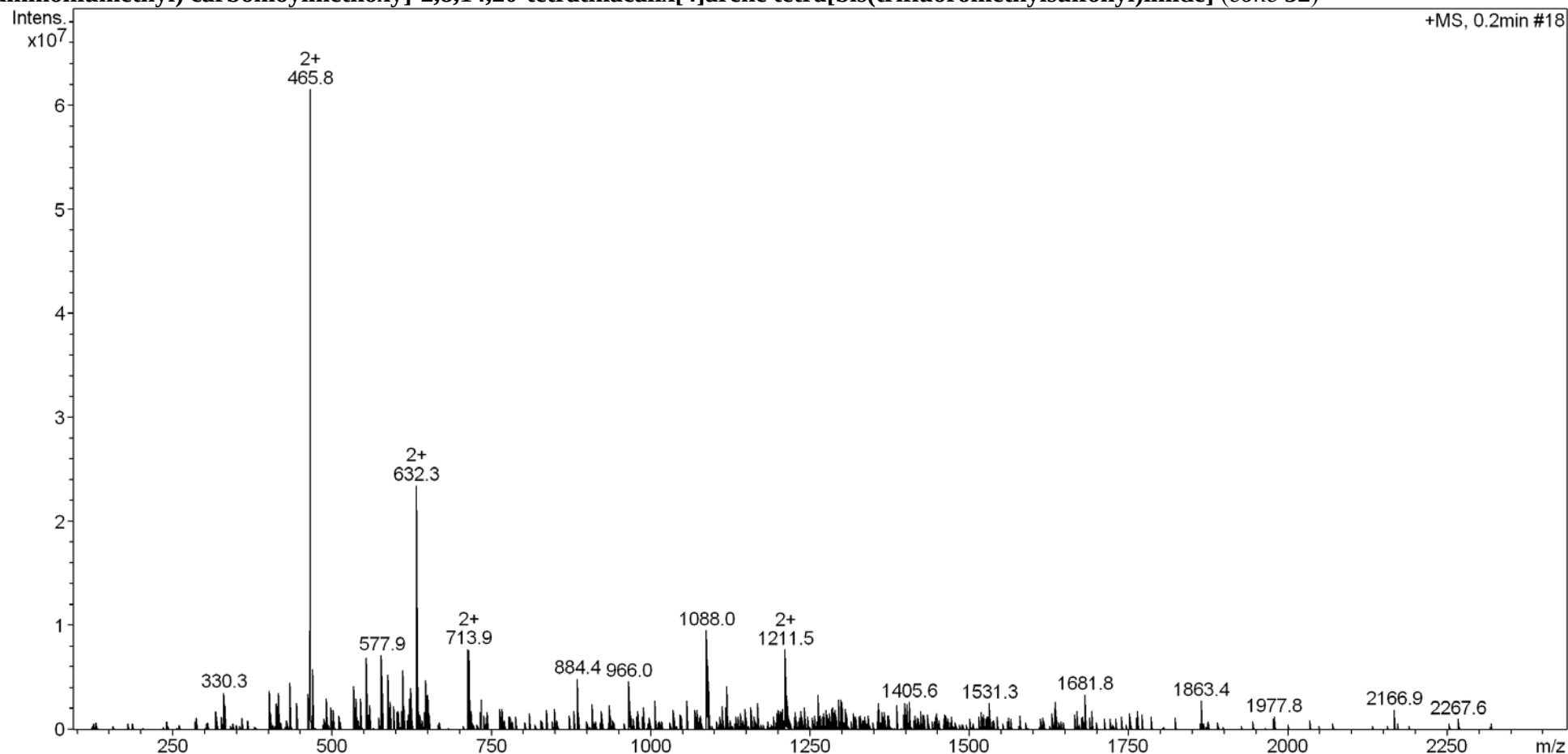


Fig. S131. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonylmethyl)amidocarbonylmethyl})-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 53)

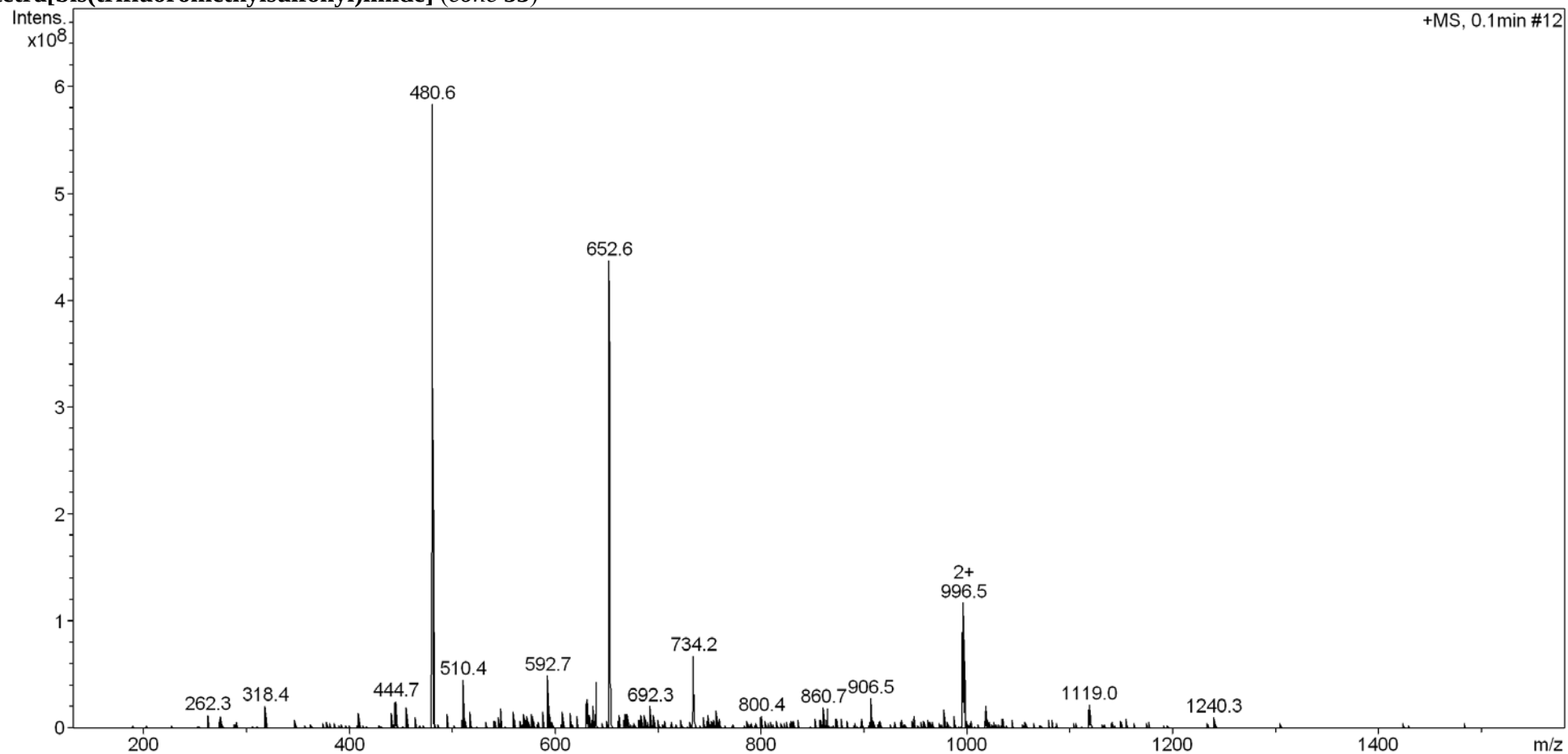


Fig. S132. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{([ethoxycarbonylmethyl]-amidocarbonylmethyl)amidocarbonylmethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 54)

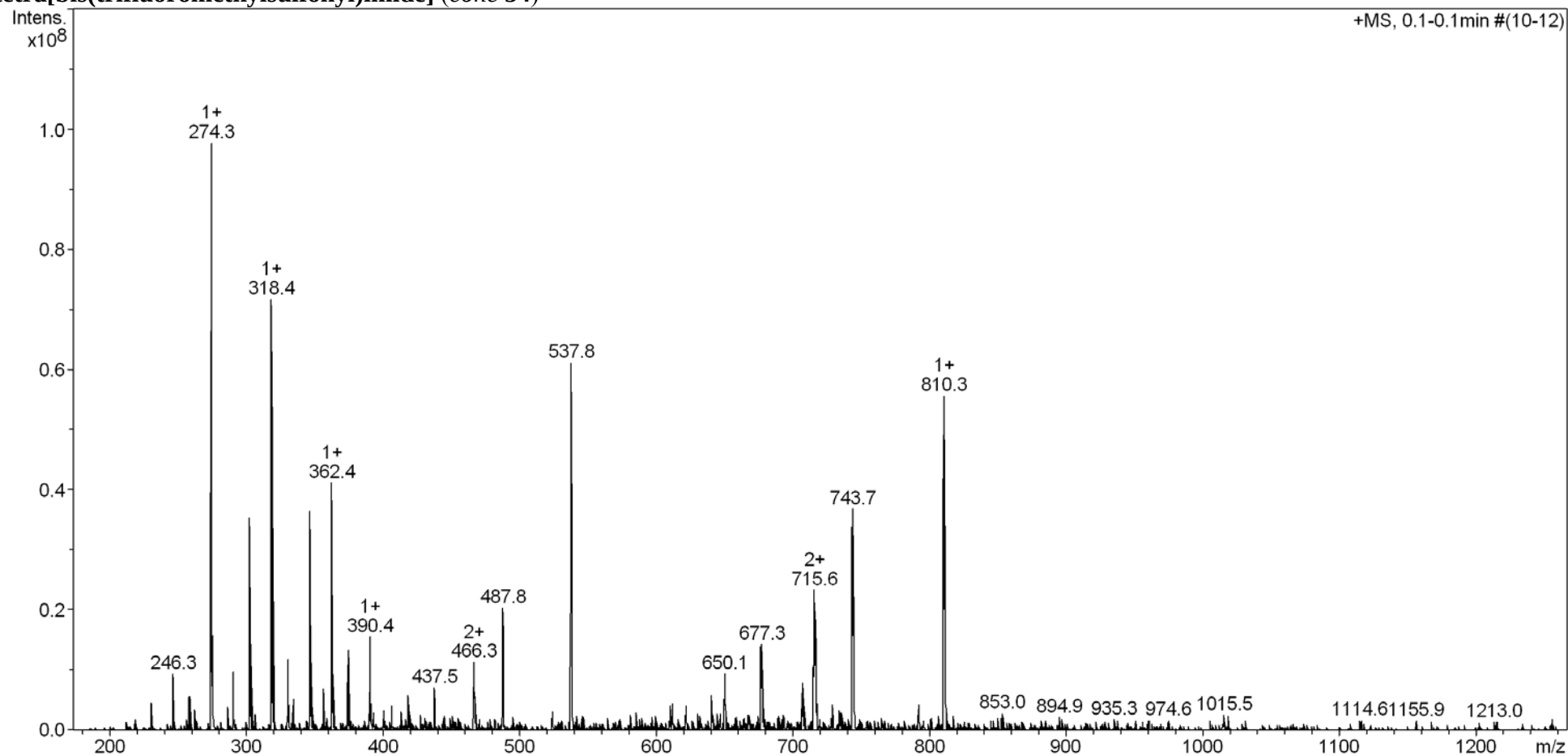


Fig. S133. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 55)

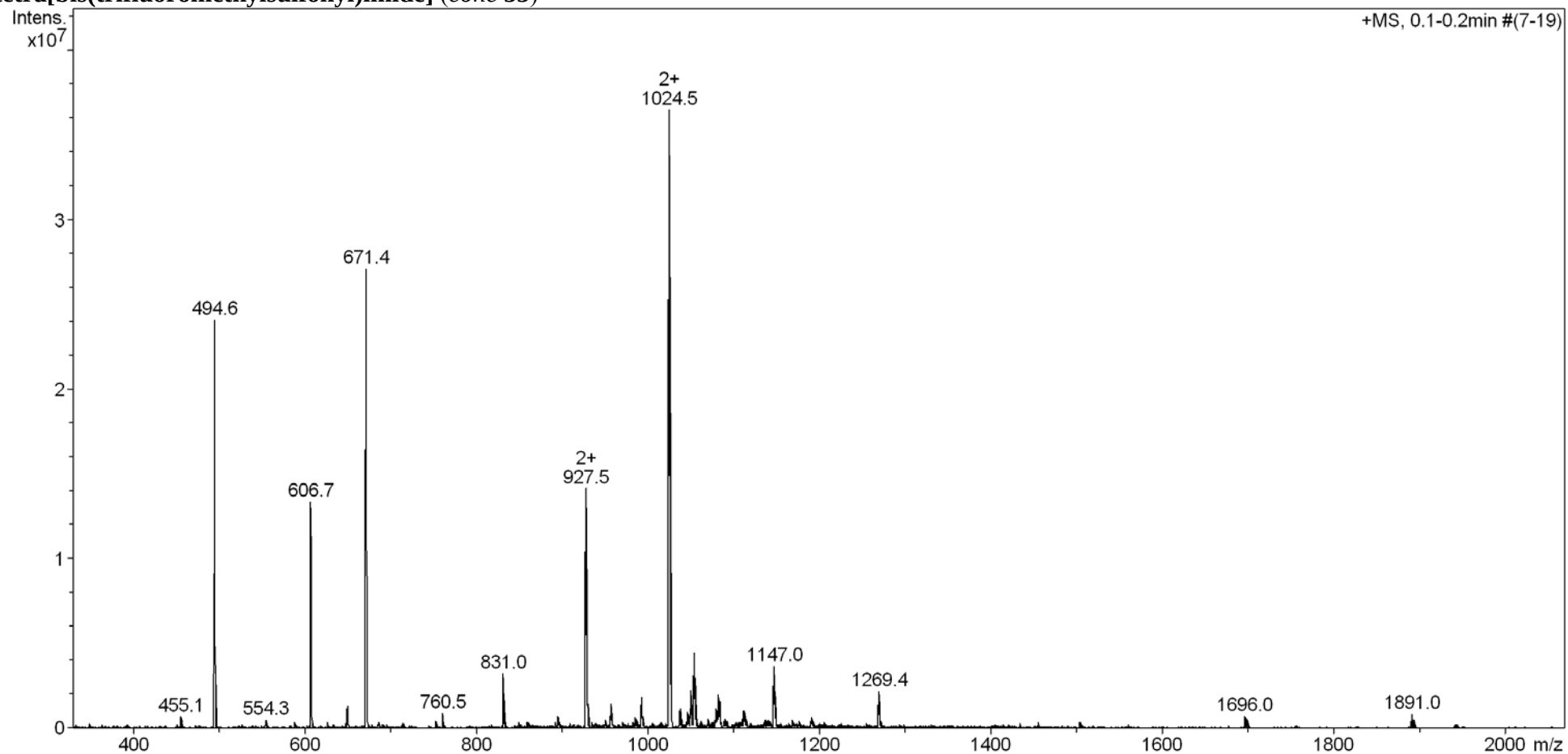


Fig. S134. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 56)

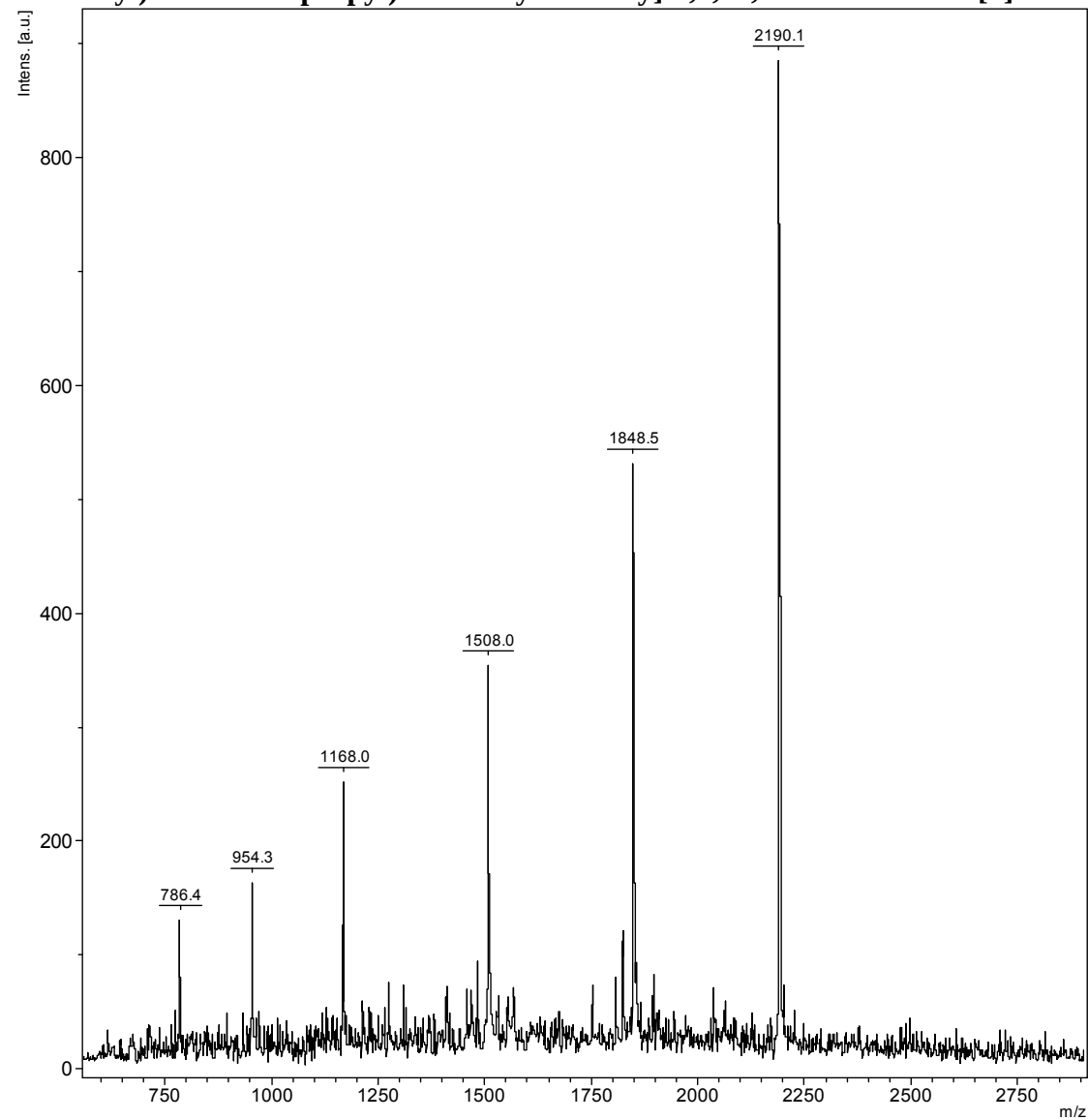


Fig. S135. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)carboxymethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 57)

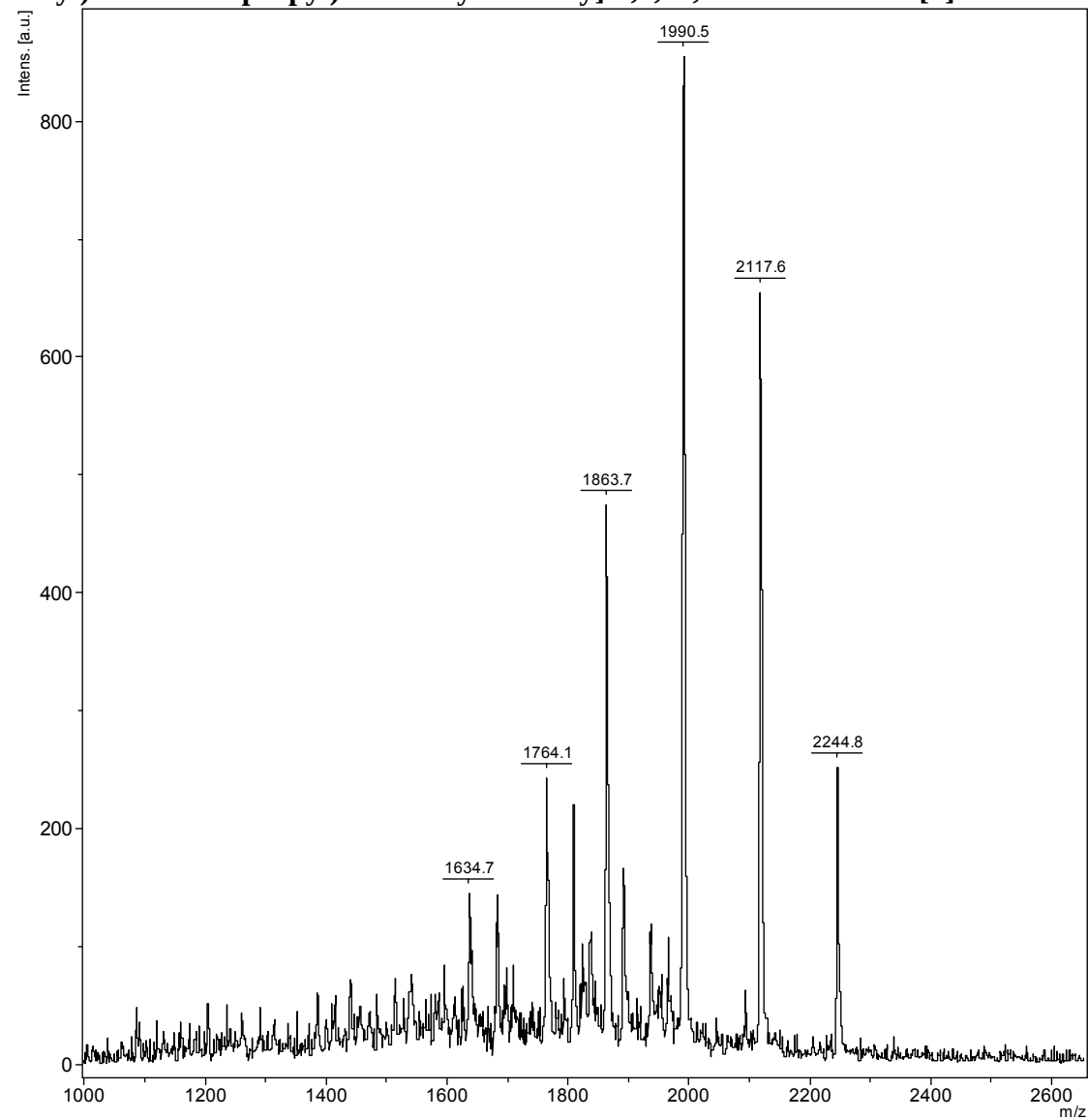


Fig. S136. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 58)

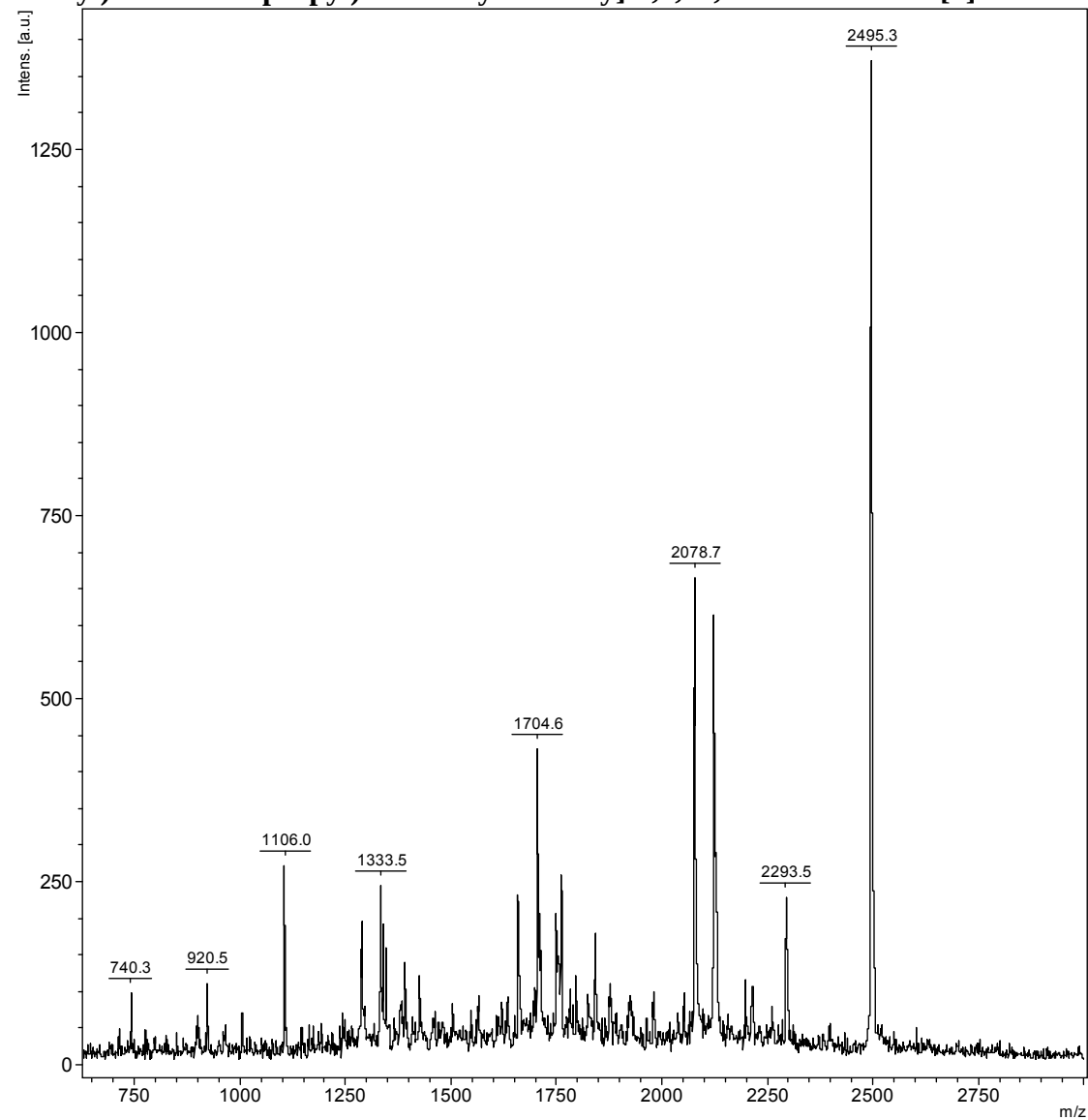


Fig. S137. Mass spectrum (MALDI-TOF, 4-nitroaniline matrix) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 59)

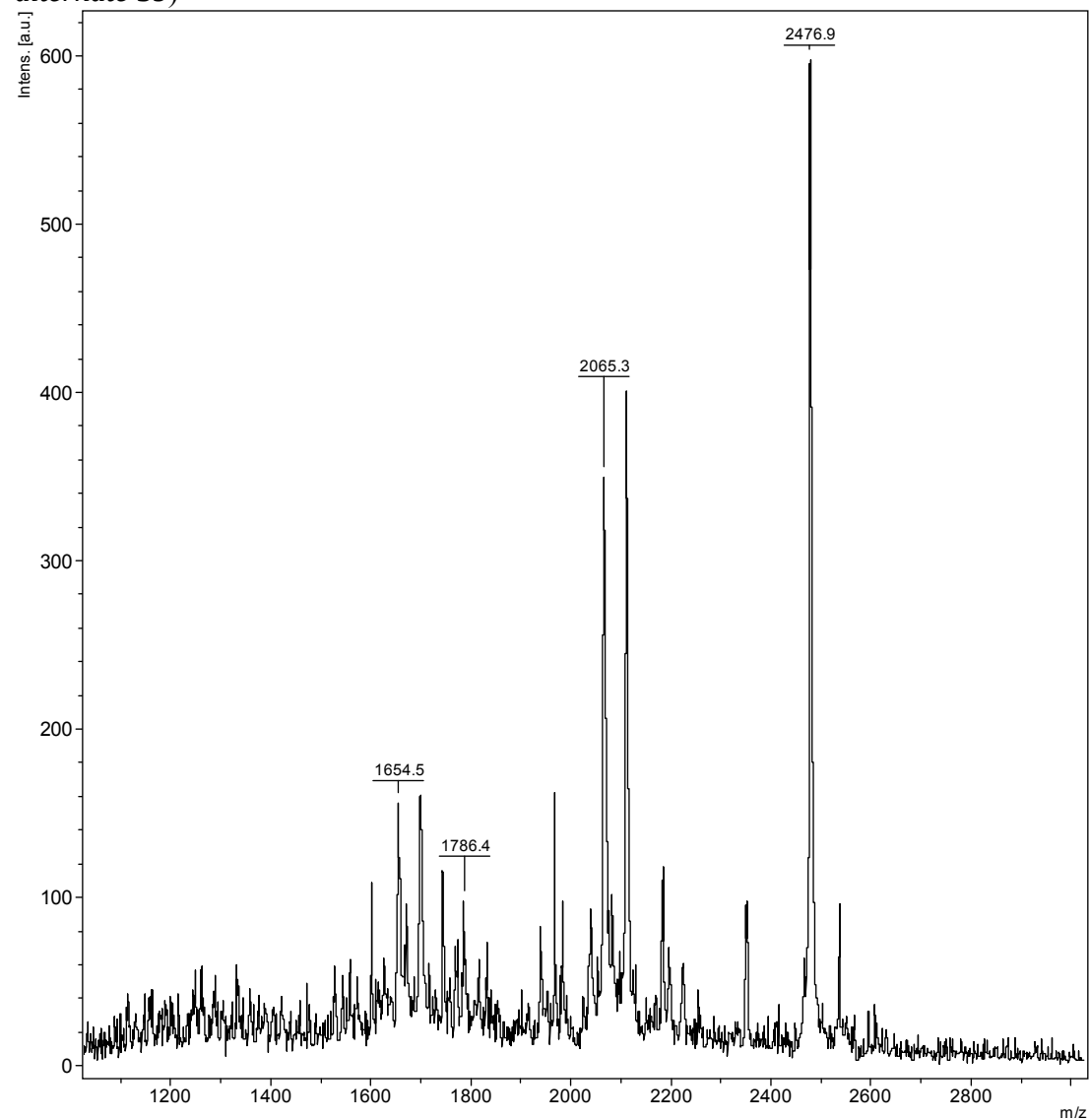


Fig. S138. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(pentoxycarbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate 60*)

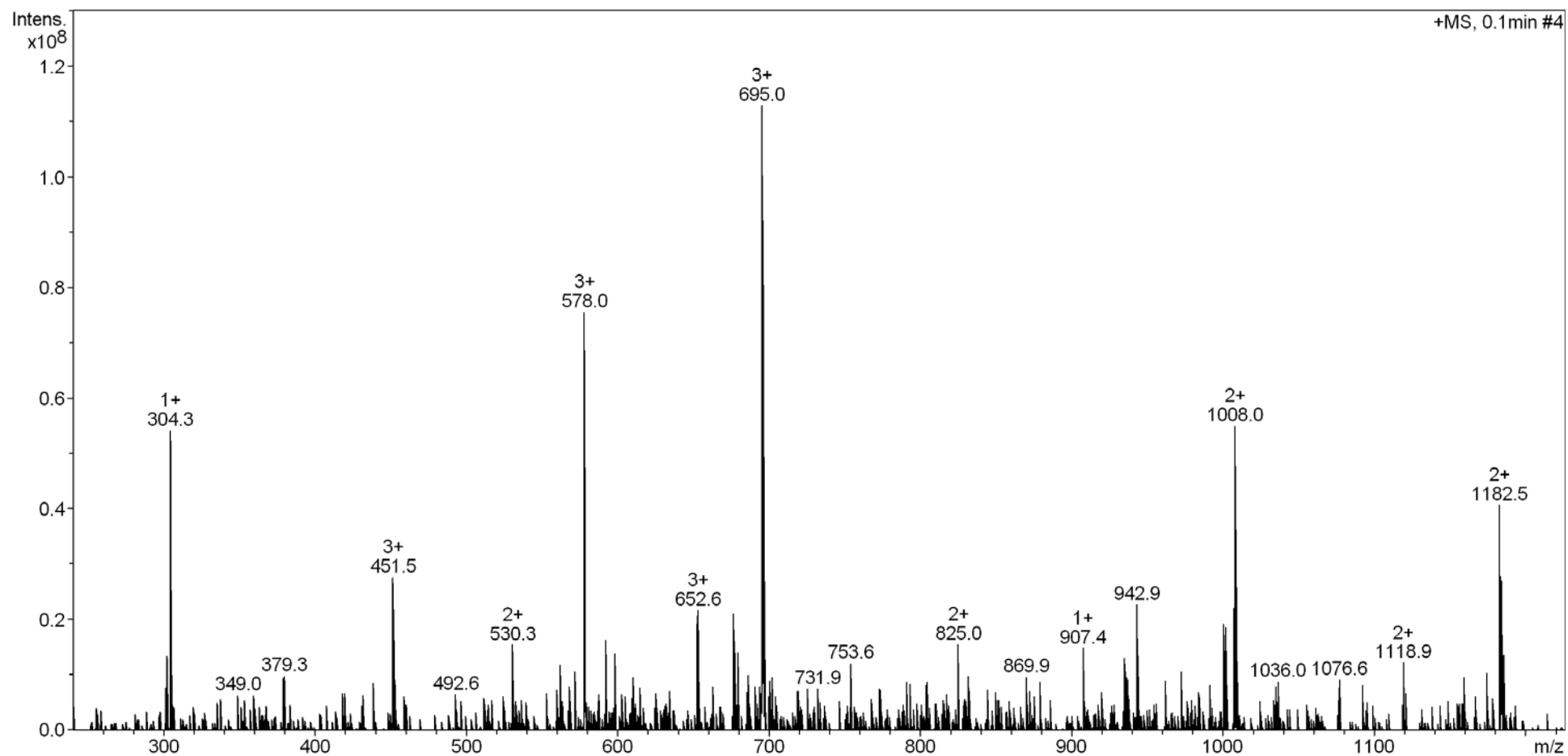


Fig. S139. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{ethoxycarbonylmethyl}amidocarbonylmethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 61)

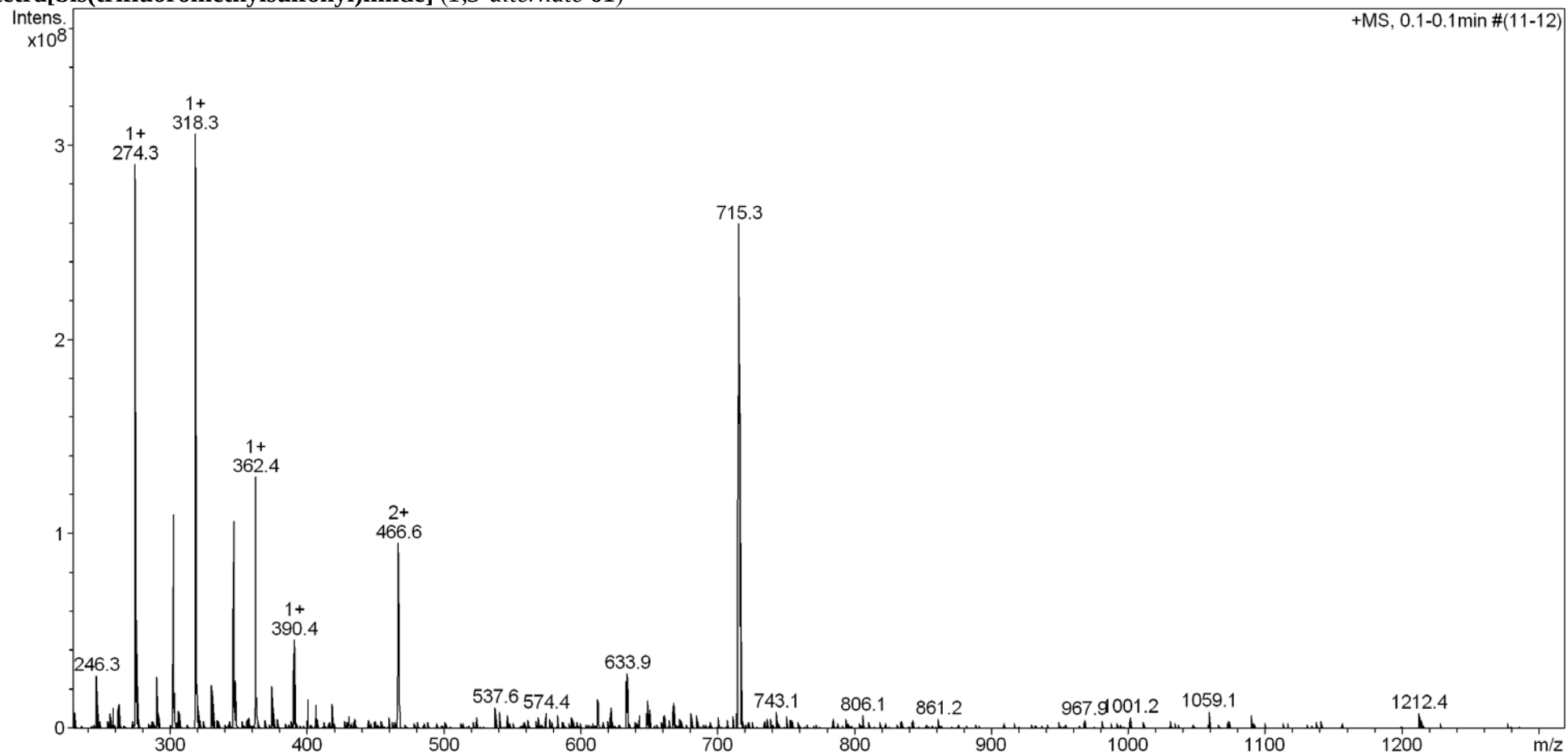


Fig. S140. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{([ethoxycarbonylmethyl]amidocarbonylmethyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiocalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 62)

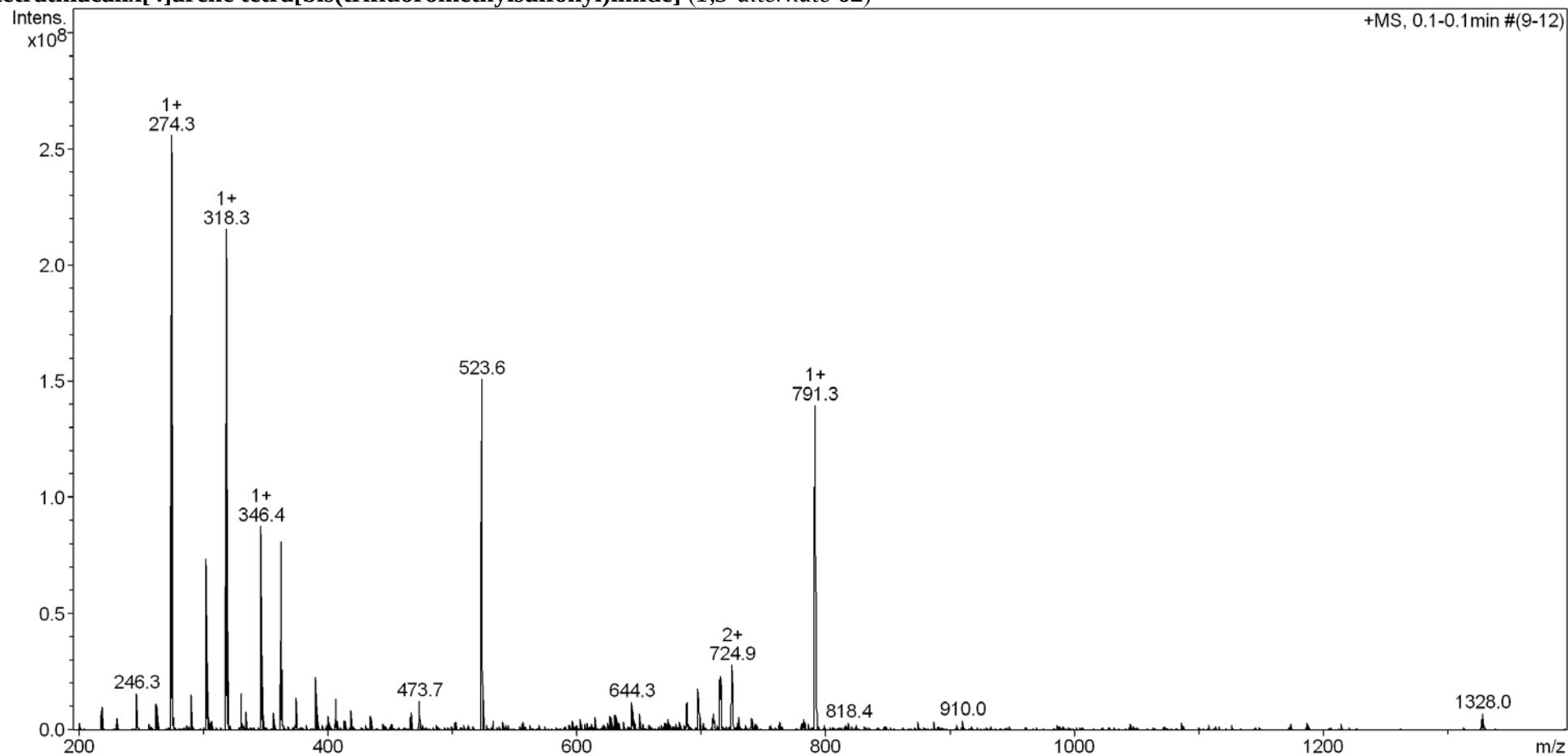


Fig. S141. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 63)

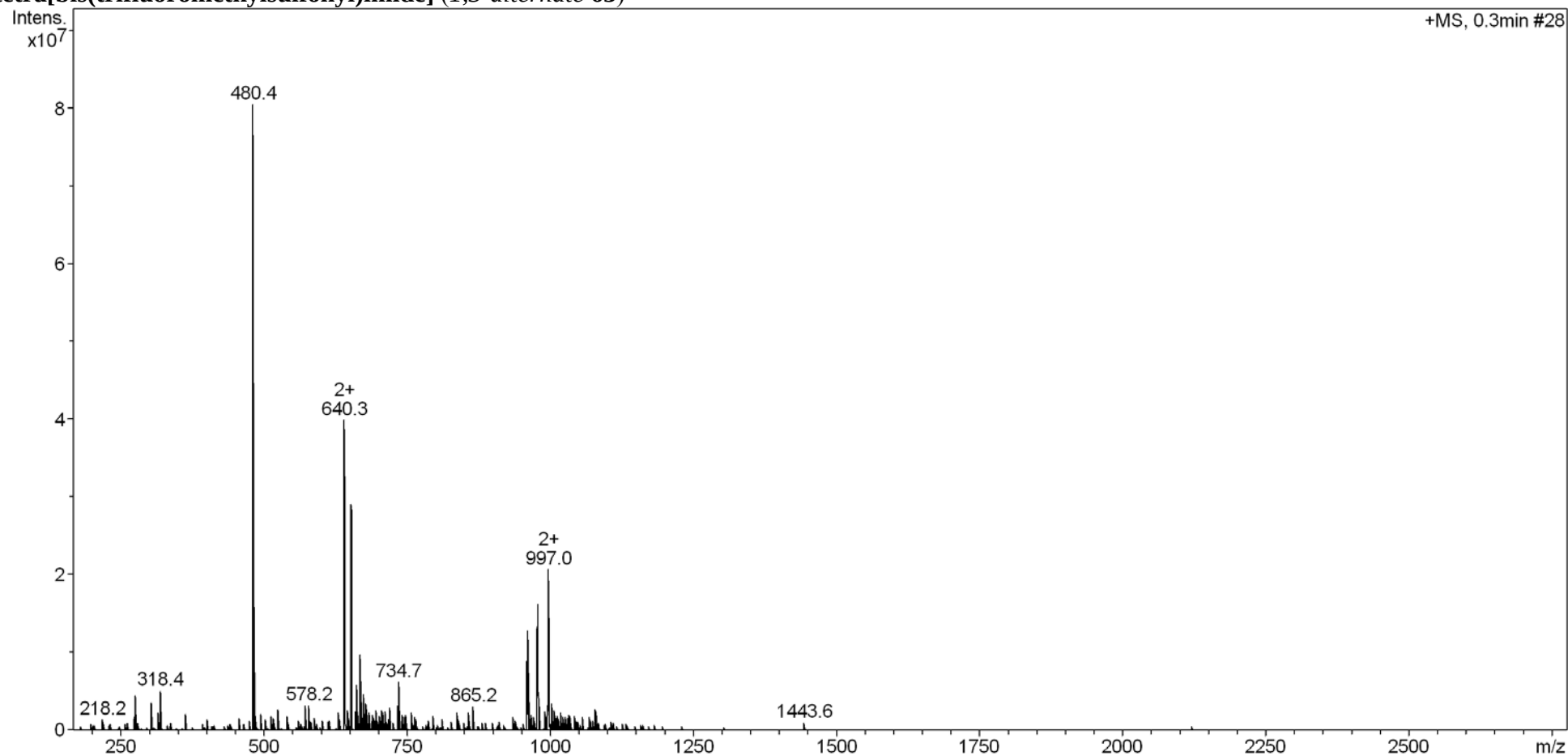


Fig. S142. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide}ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 64)

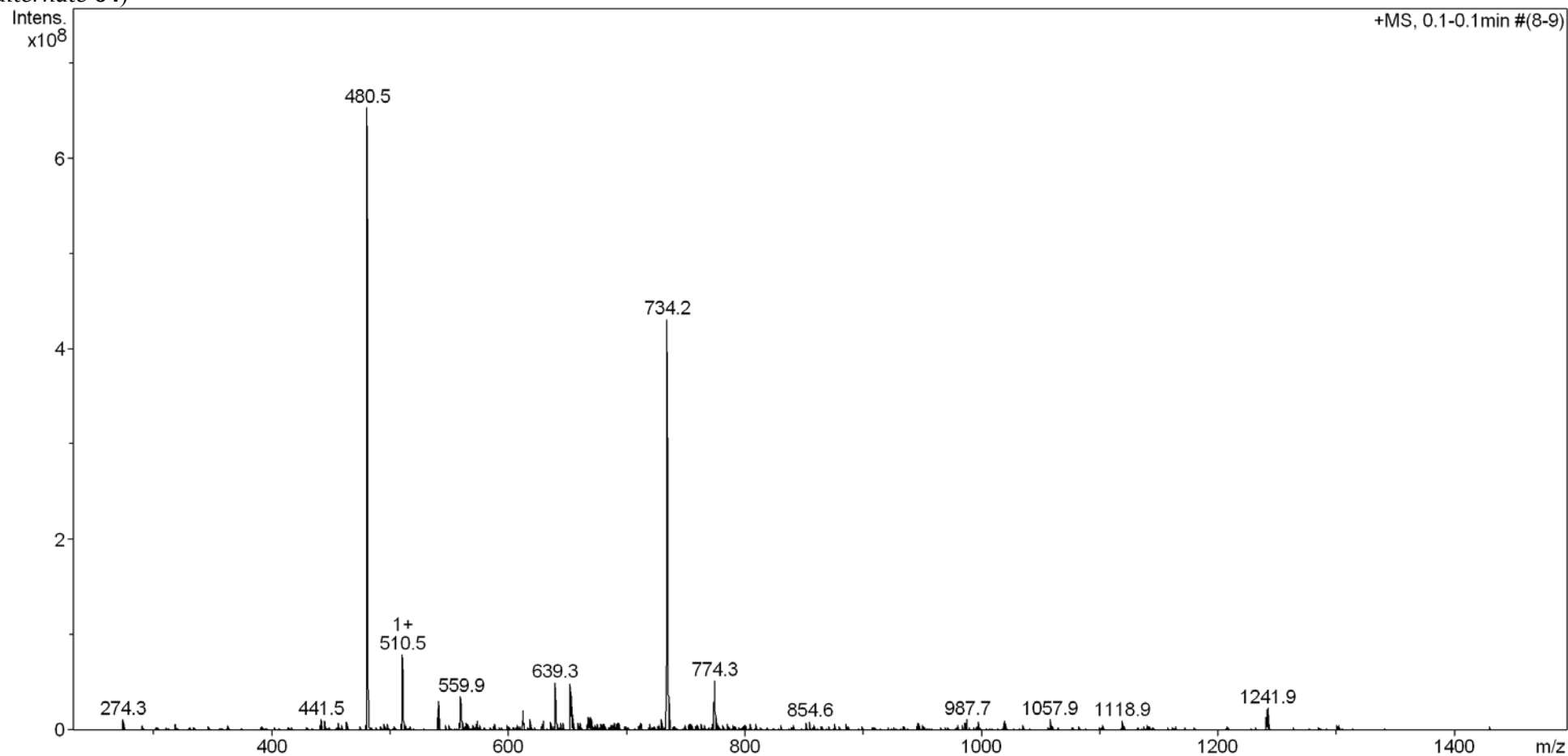


Fig. S143. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 65)

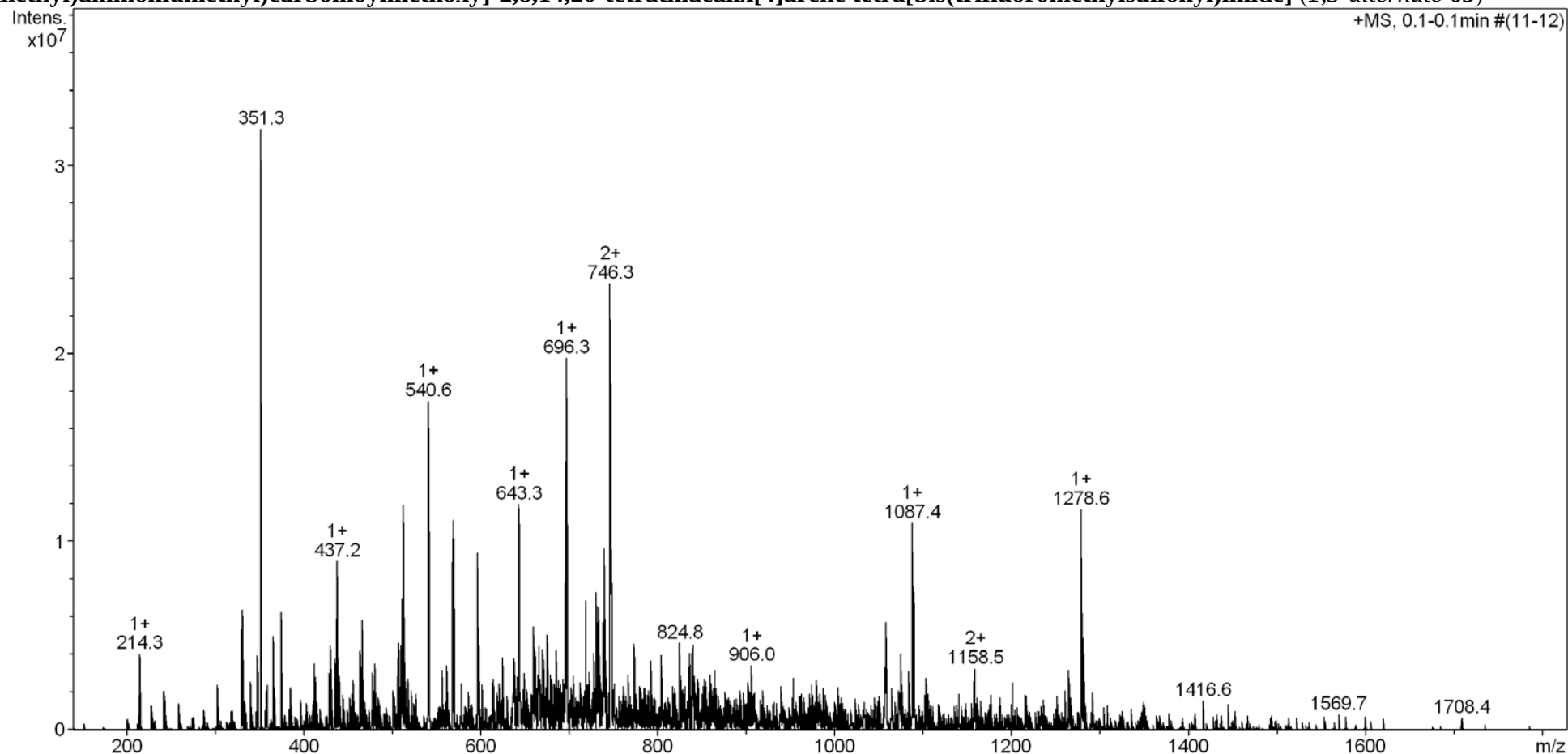


Fig. S144. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 66)

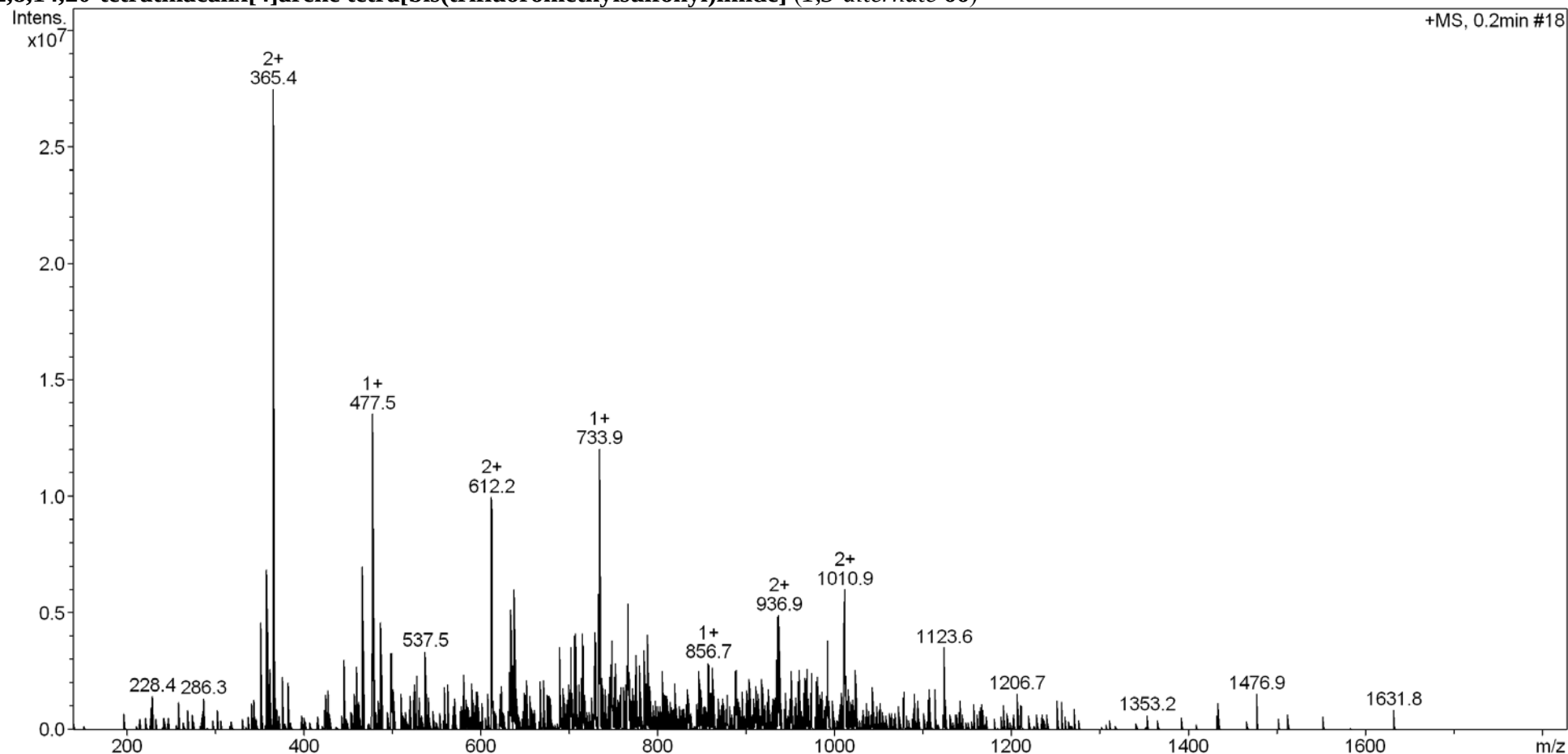


Fig. S145. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 67)

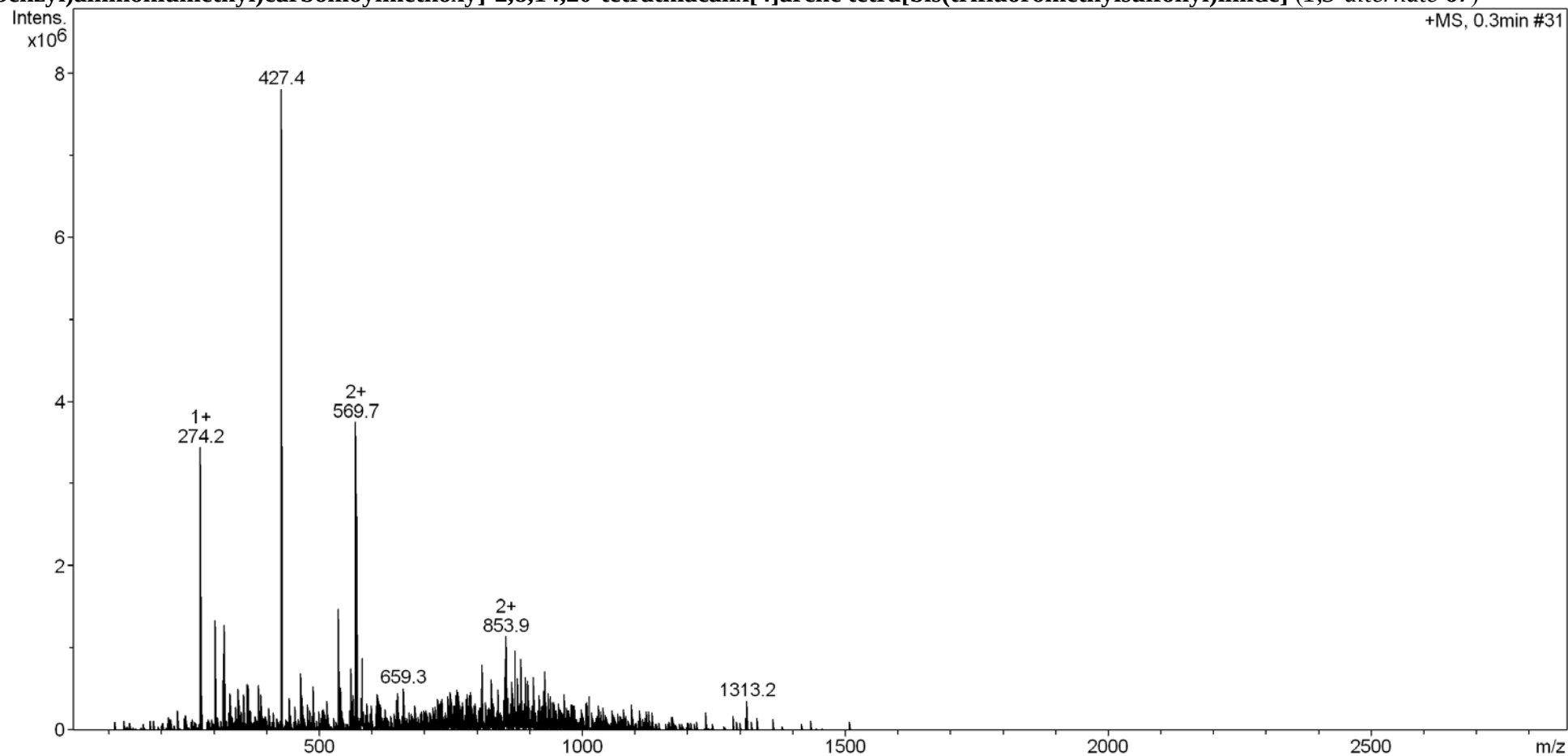


Fig. S146. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiocalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate **68)**

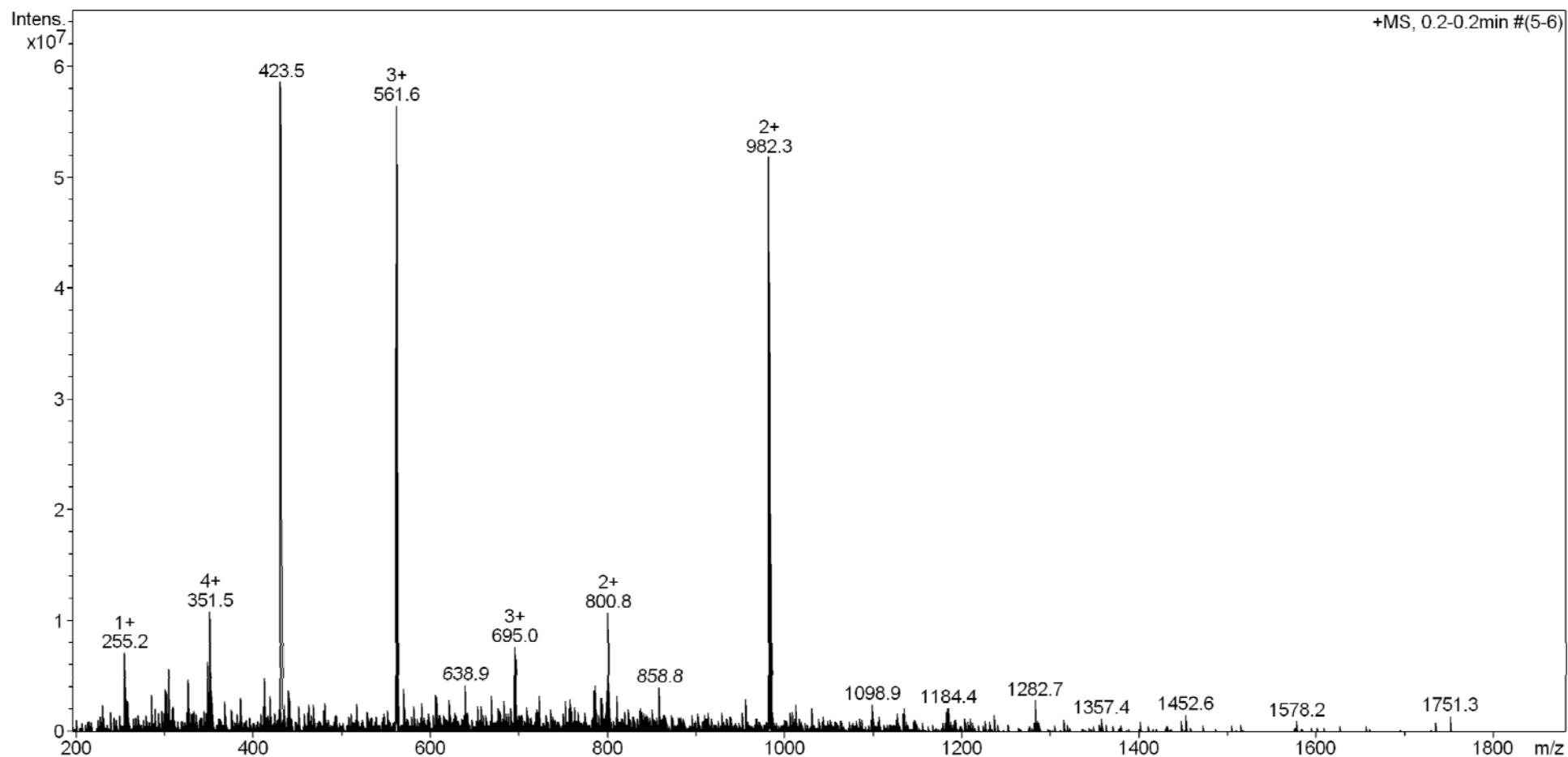


Fig. S147. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 69)

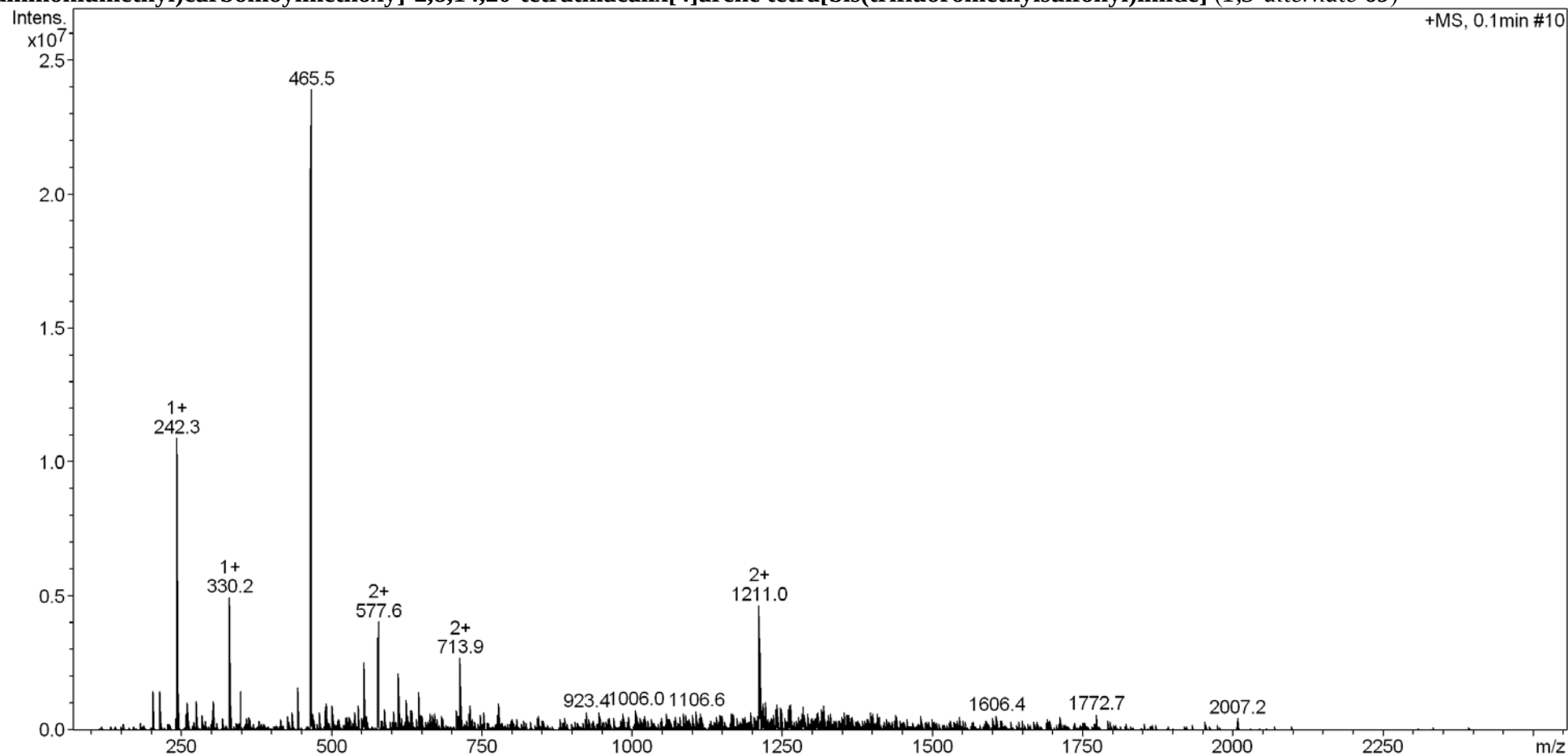


Fig. S148. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{ethoxycarbonylmethyl}amidocarbonylmethyl))-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 70)

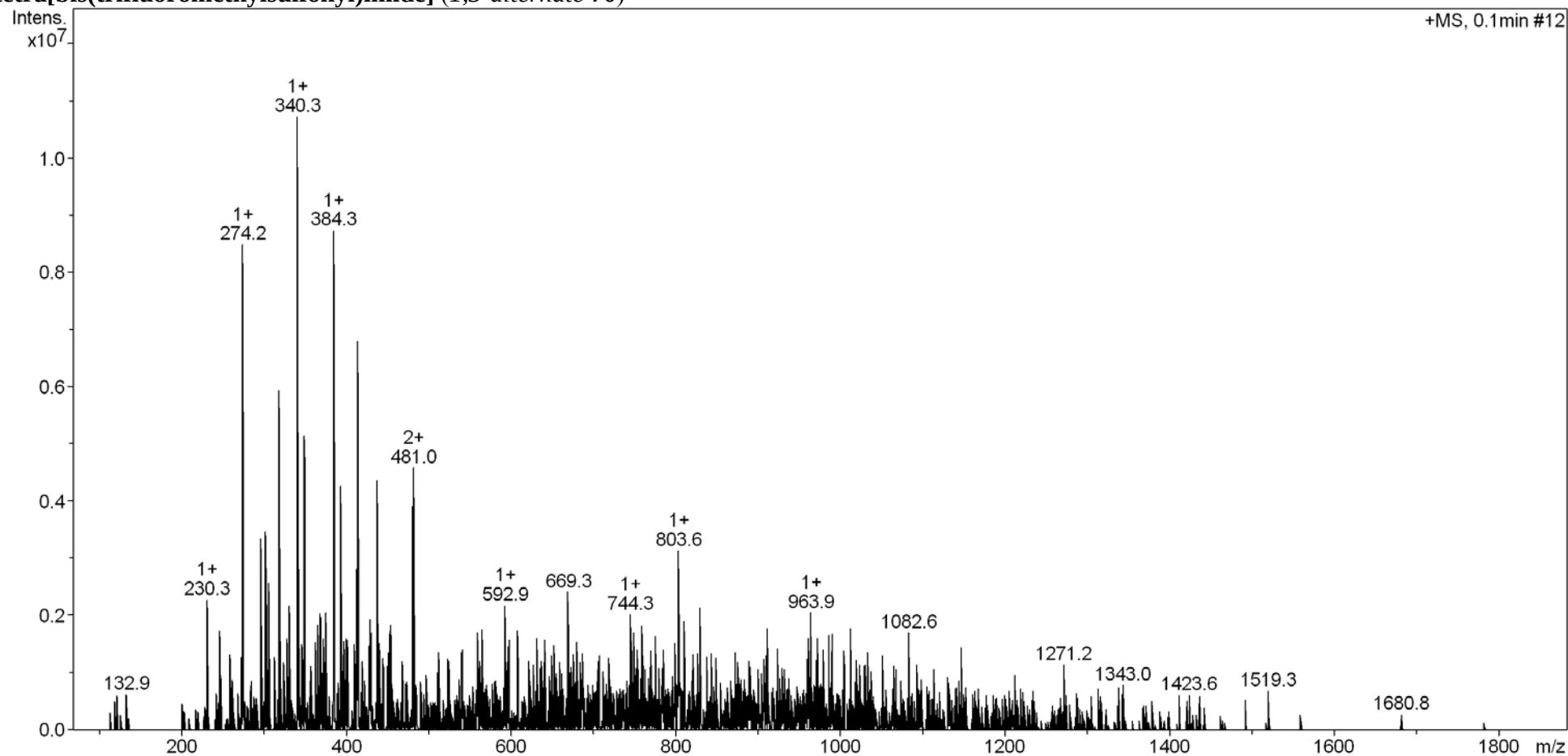


Fig. S149. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{[(ethoxycarbonylmethyl]amidocarbonylmethyl)-amidocarbonylmethyl]ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 71)

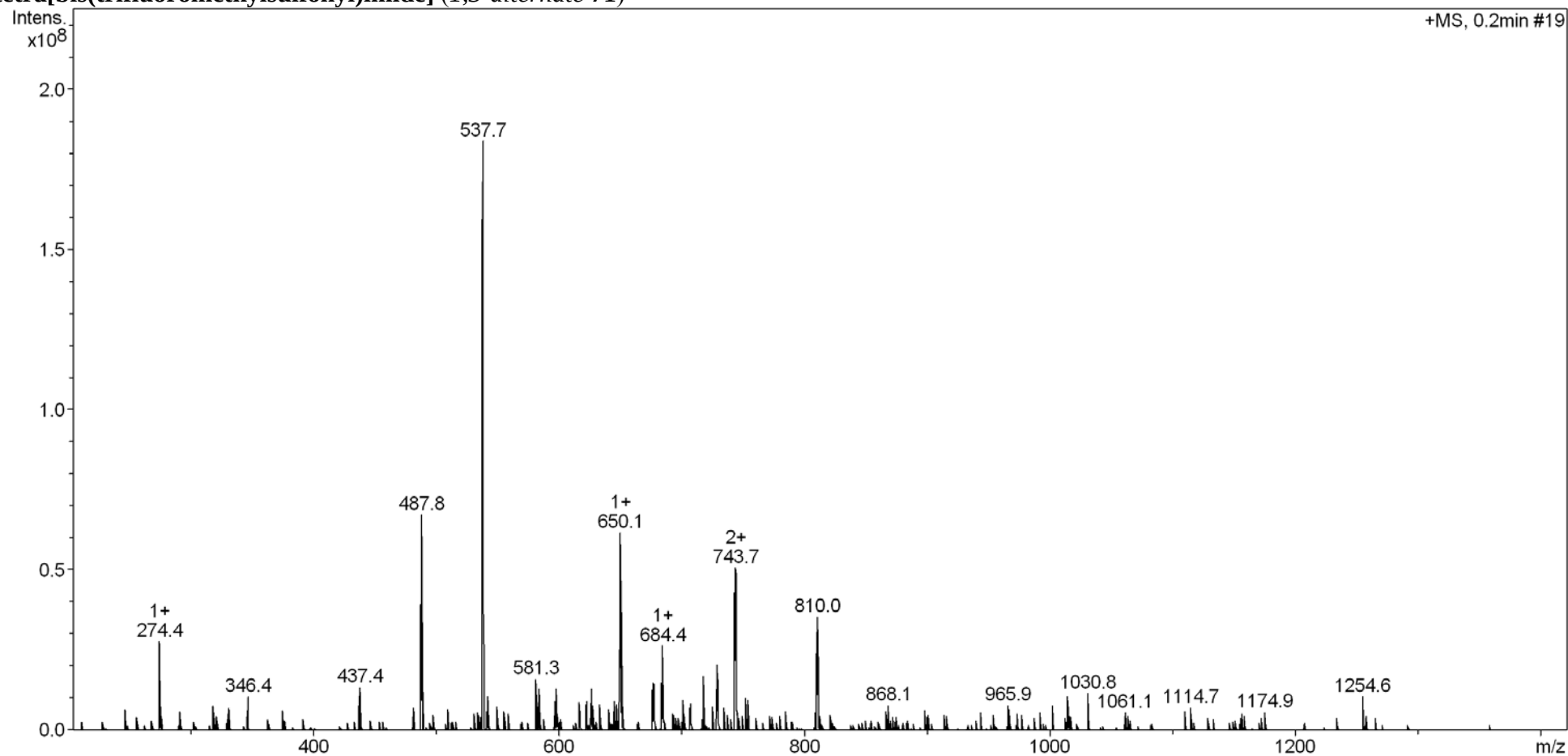


Fig. S150. Mass spectrum (ESI) of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 72)

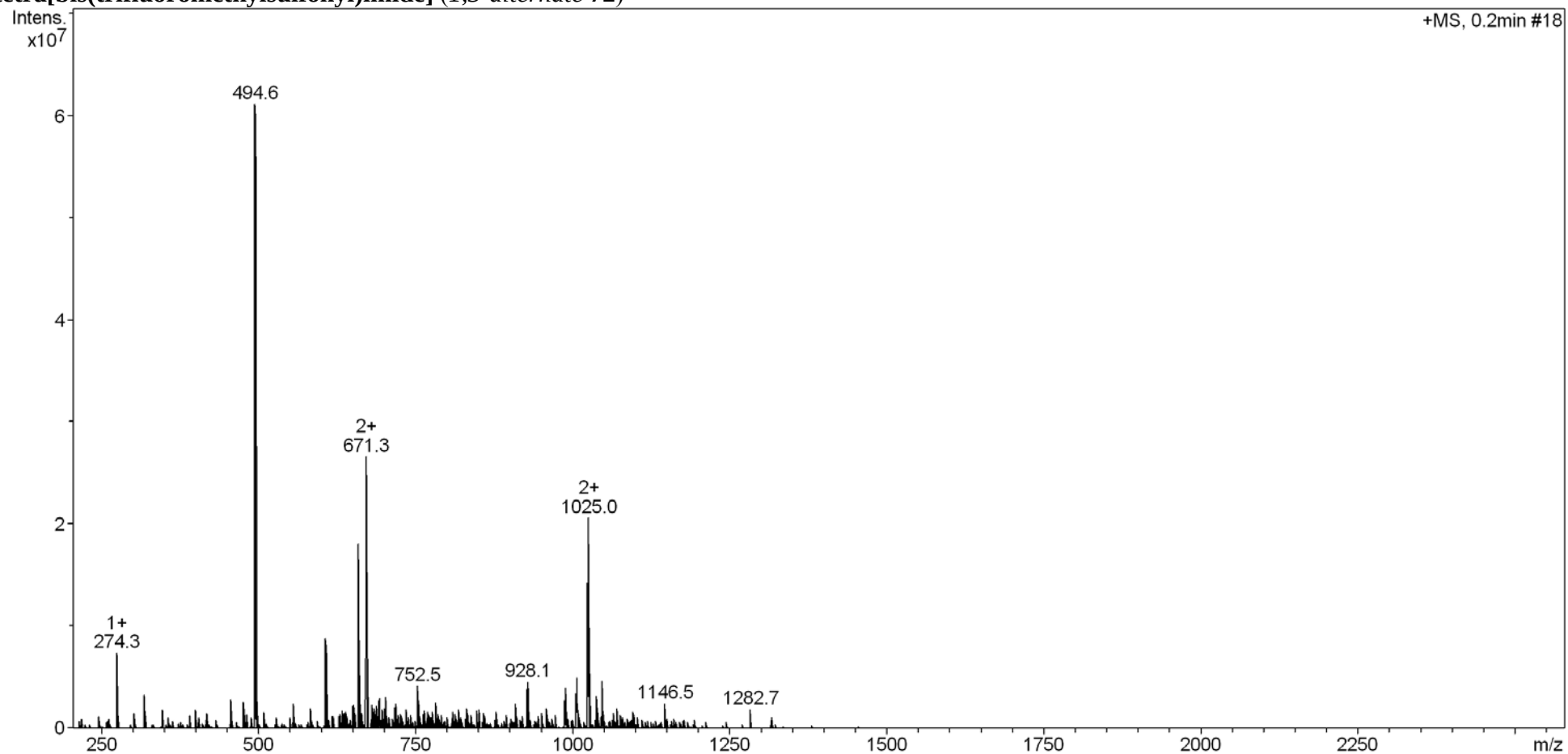


Fig. S151. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-(pentoxycarbonylmethyl)-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 9)



Fig. S152. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (*cone* 15)

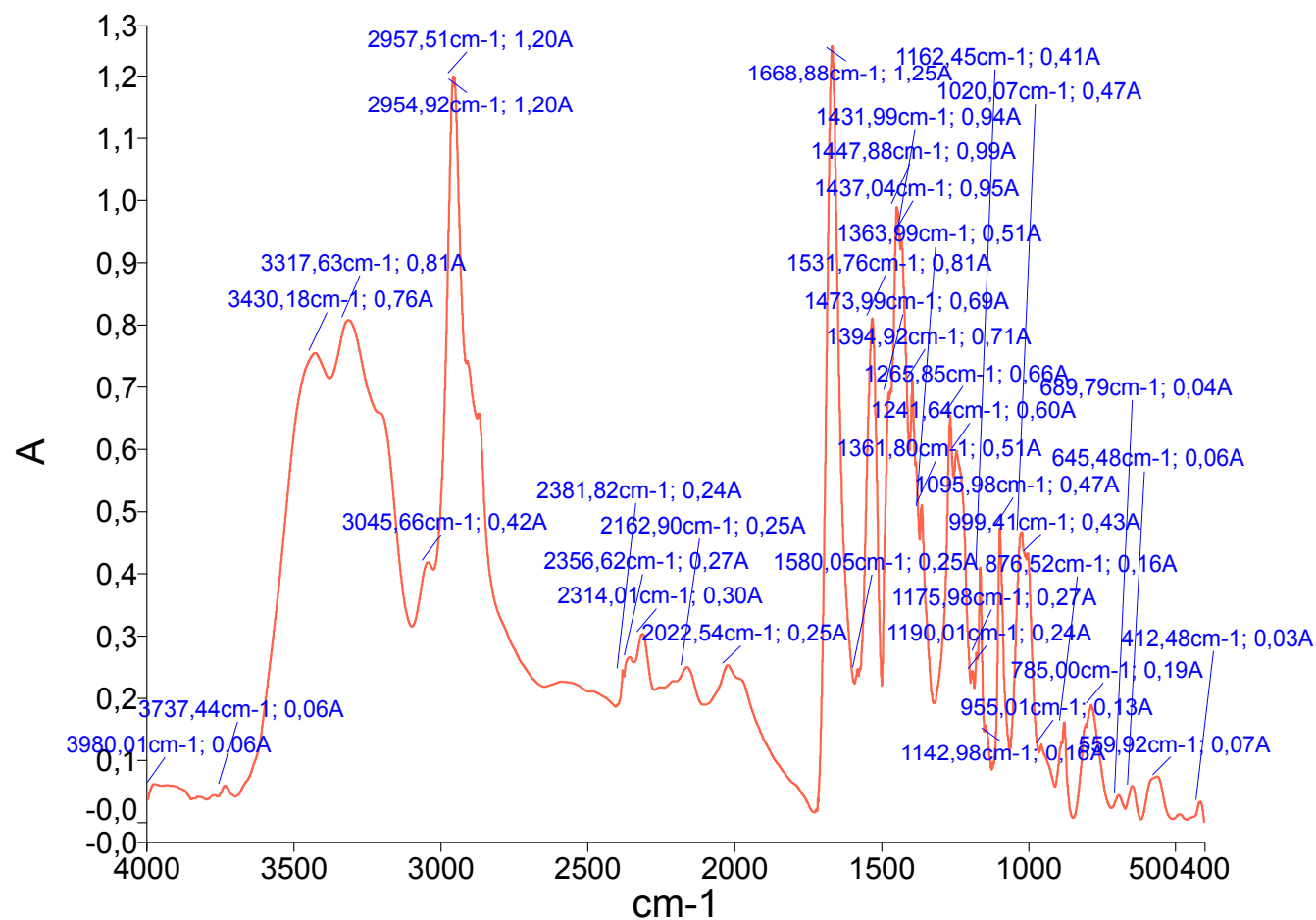


Fig. S153. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 16)

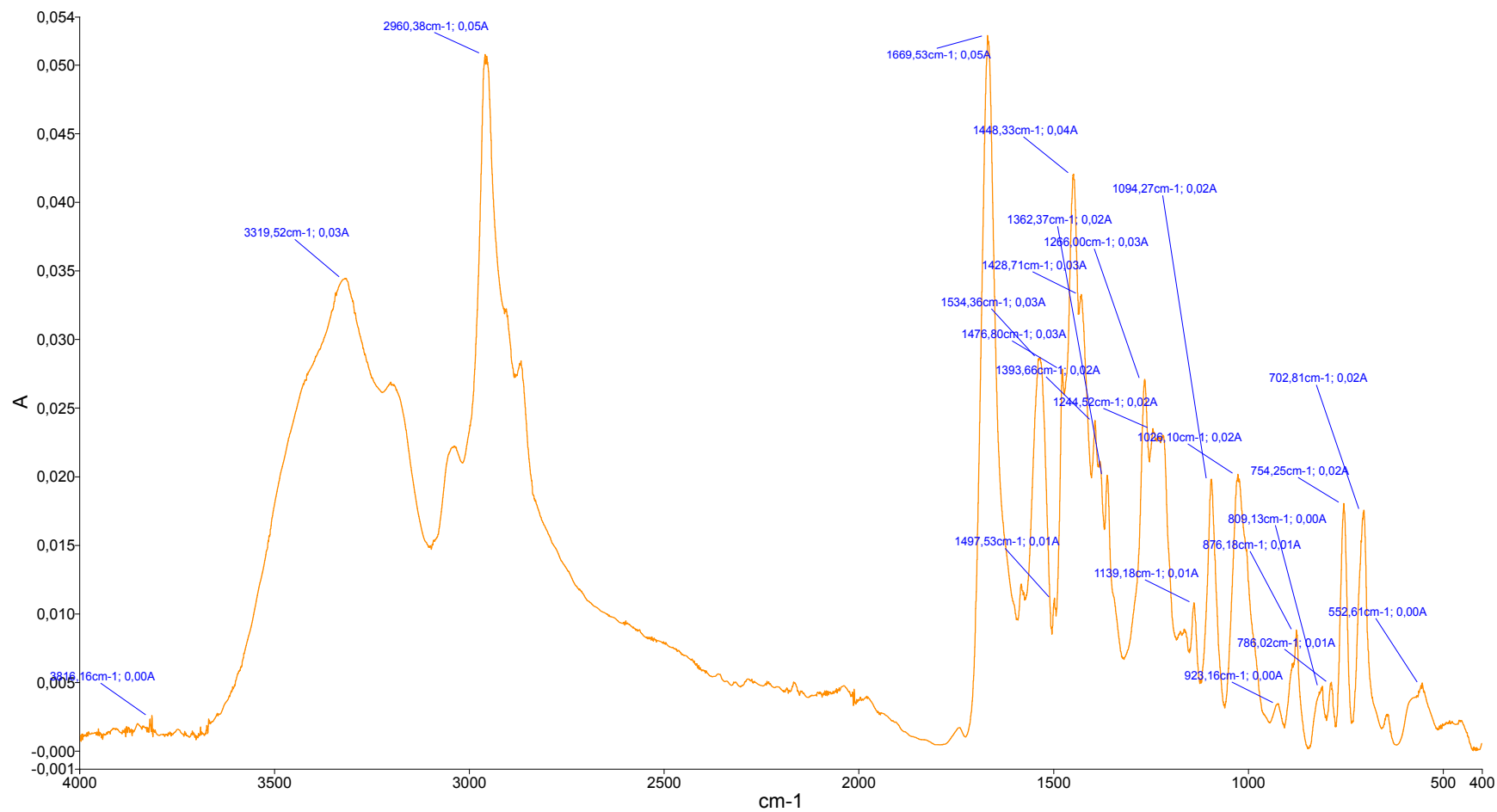


Fig. S154. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 17)

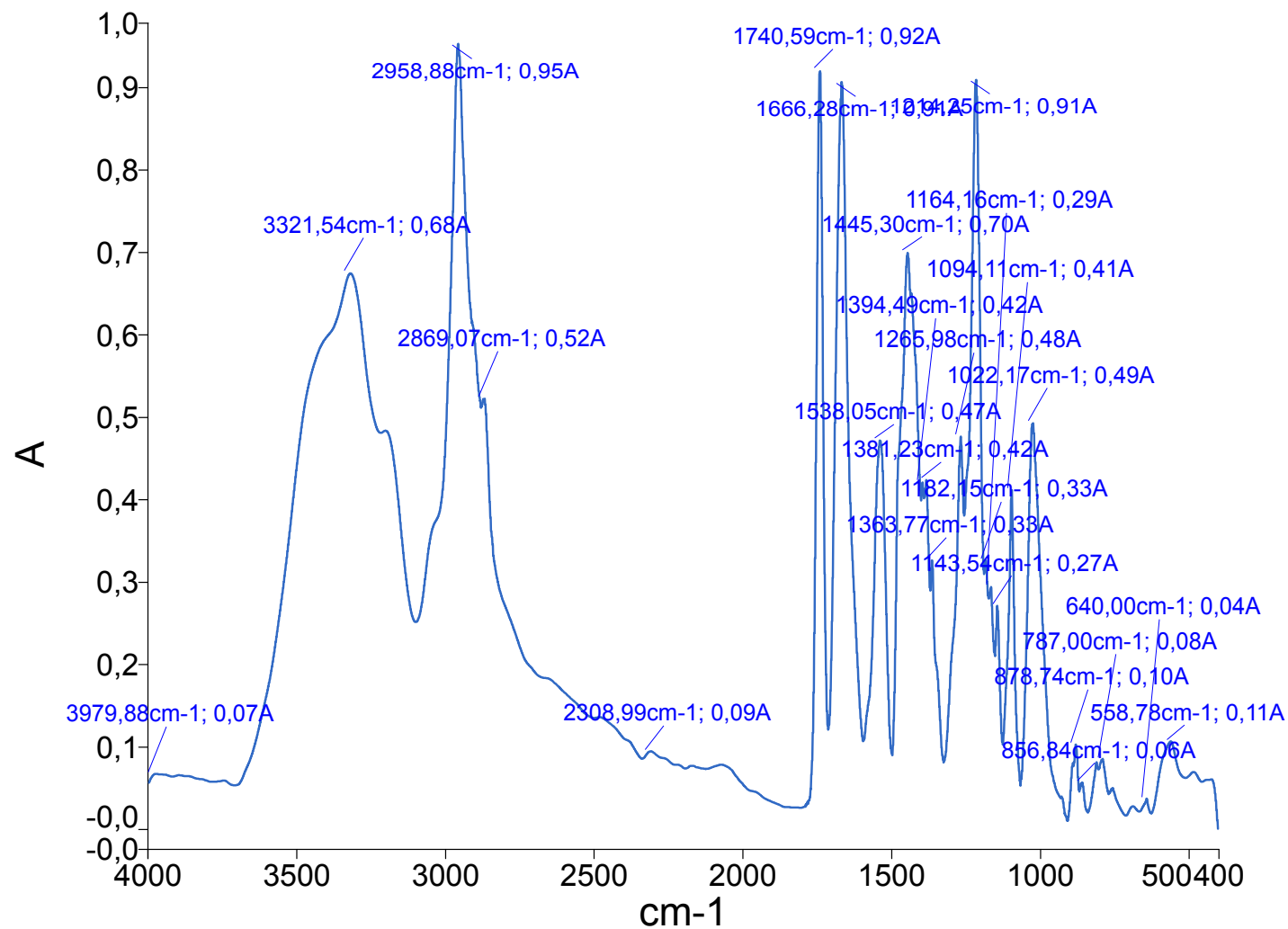


Fig. S155. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(2',2'-diethyl-2'-(pentoxy carbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (*cone* 18)

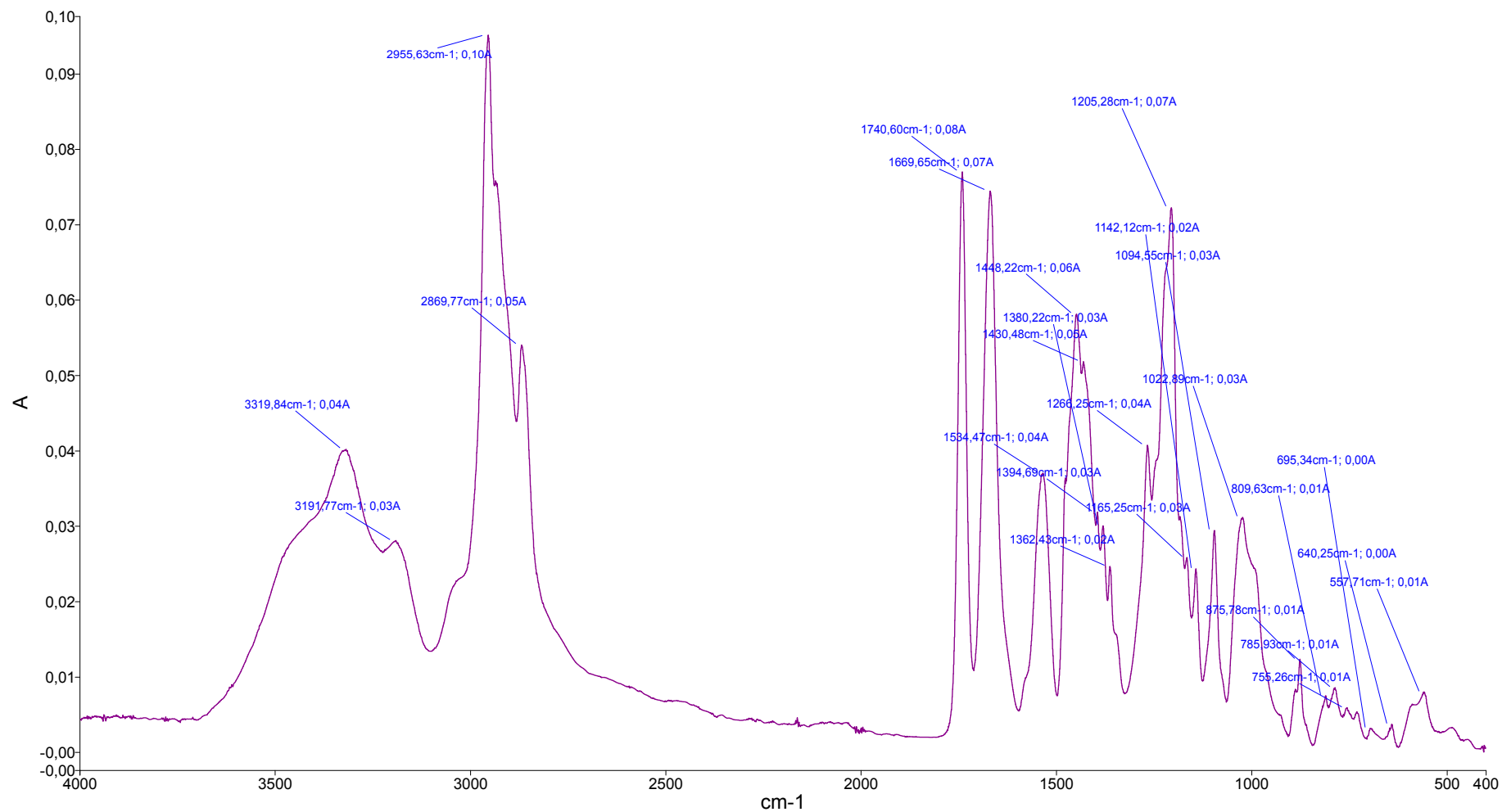


Fig. S156. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbomoxymethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-*alternate* 22)

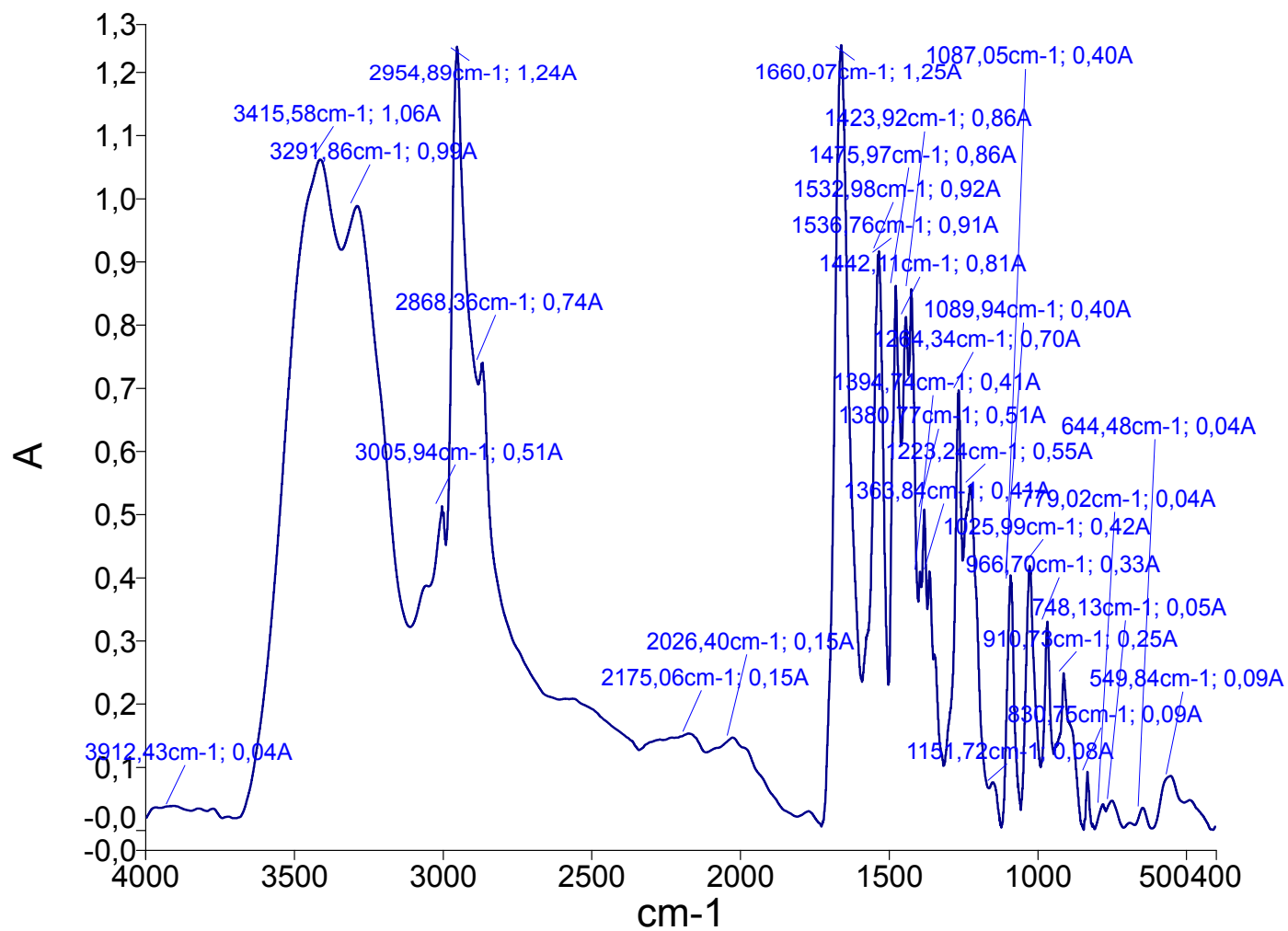


Fig. S157. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-*alternate* 23)

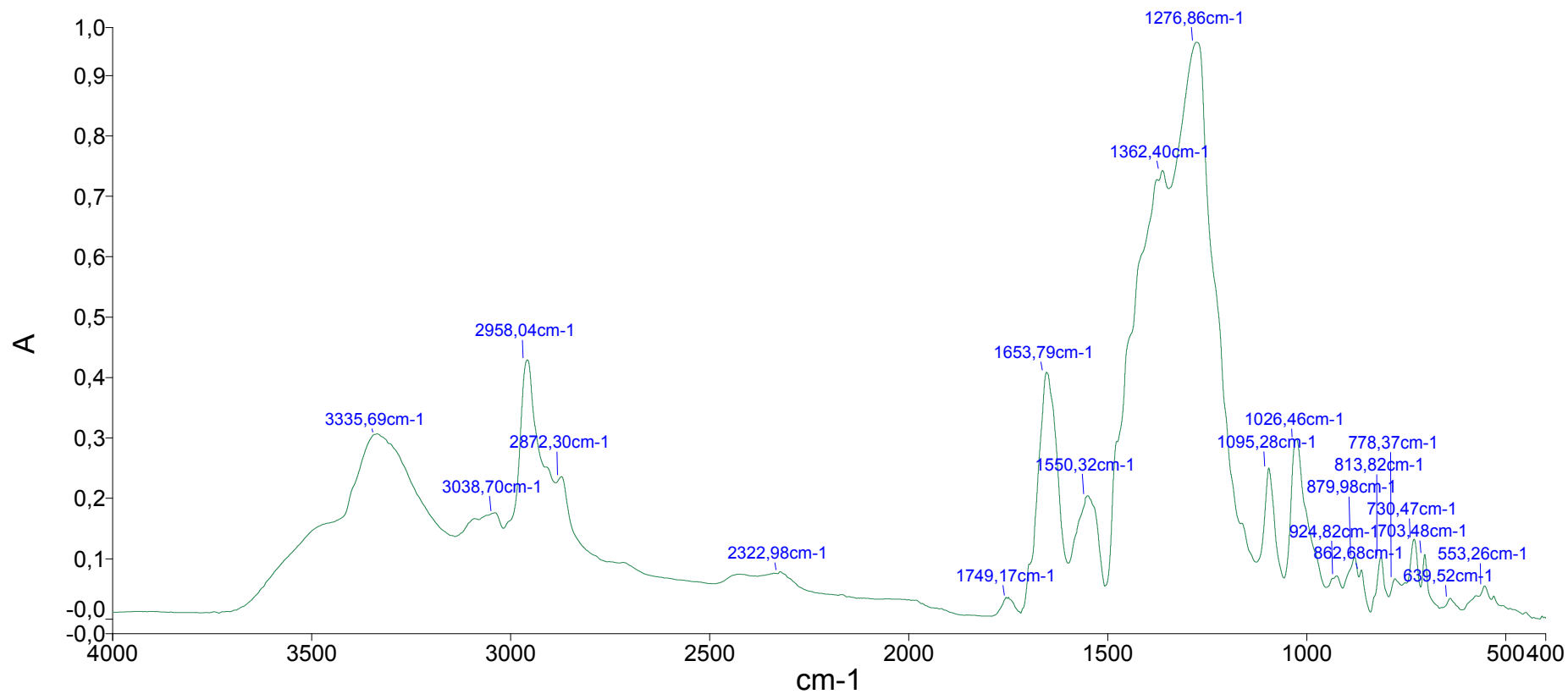


Fig. S158. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)carbomoxymethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 24)

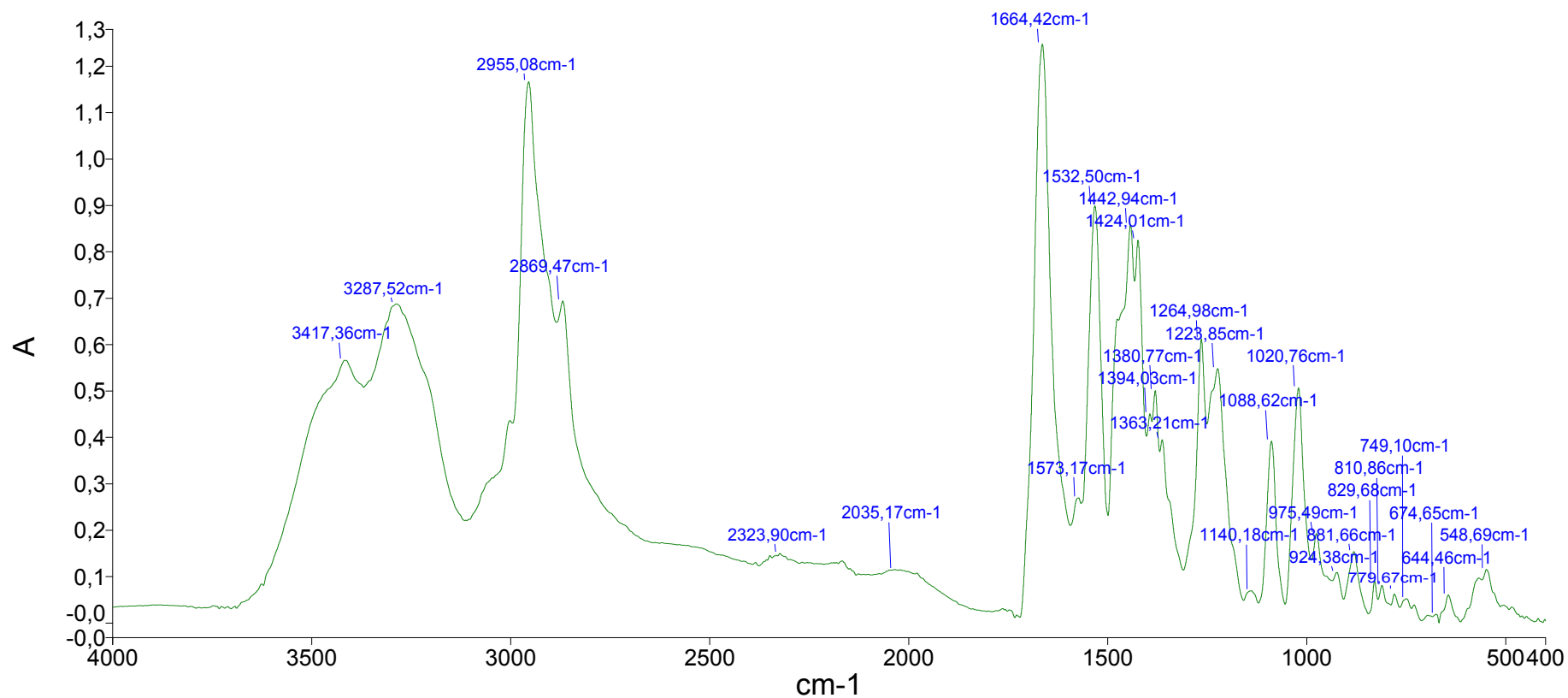


Fig. S159. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)-ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-alternate 25)

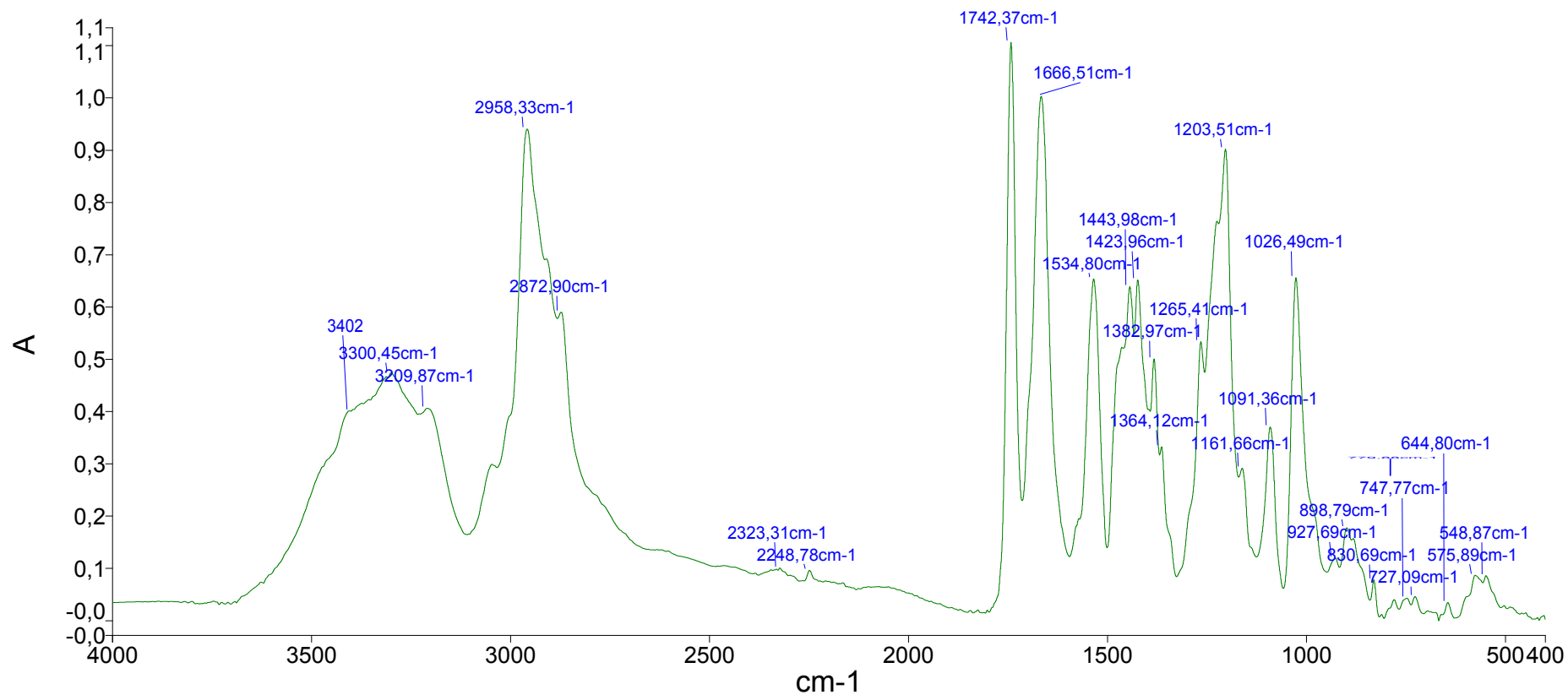


Fig. S160. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-(pentoxycarbonylmethyl)-ammoniumpropyl)carbomoylethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 26)

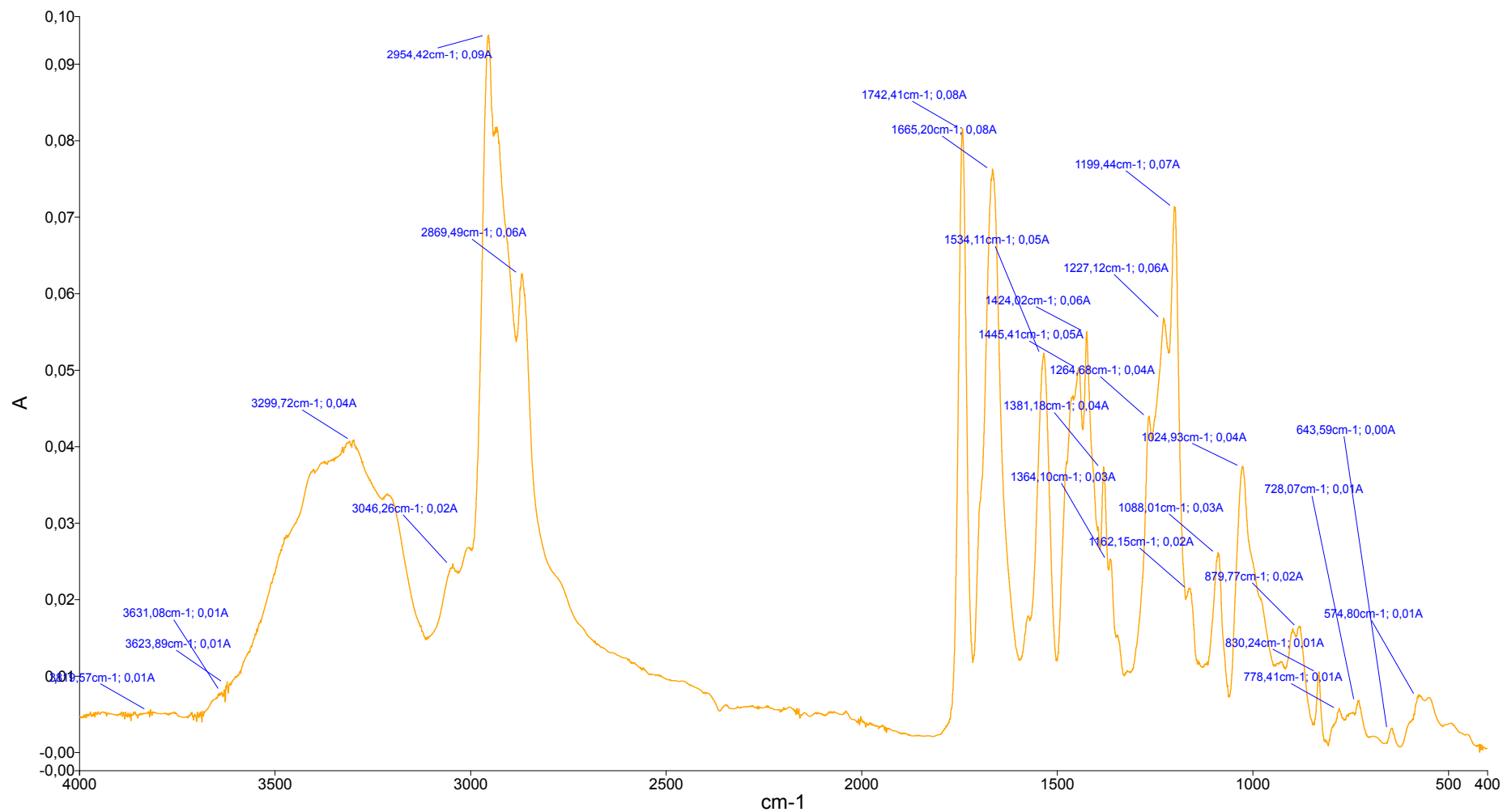


Fig. S161. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-{3''-propylphthalimide)-ammoniumpropyl)carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-alternate **30**)

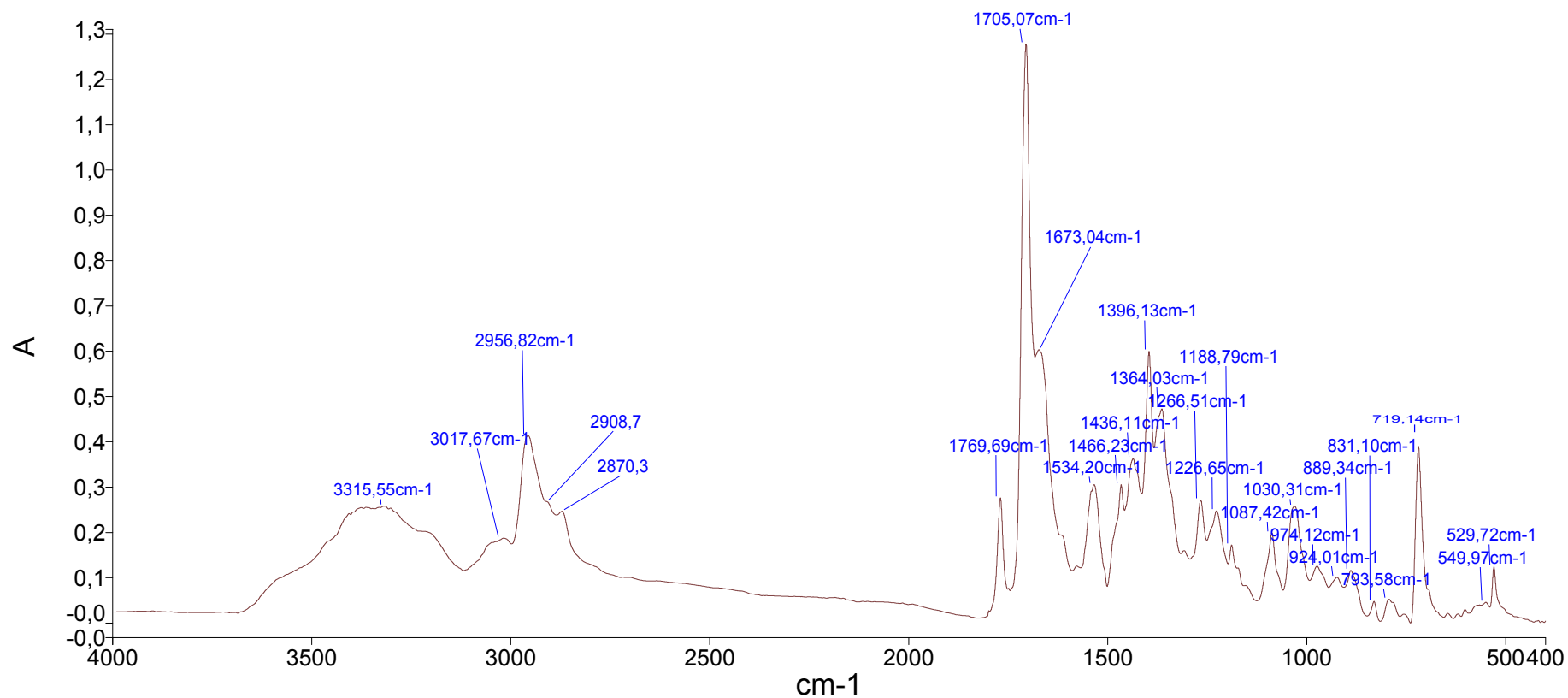


Fig. S162. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-*alternate* 31)

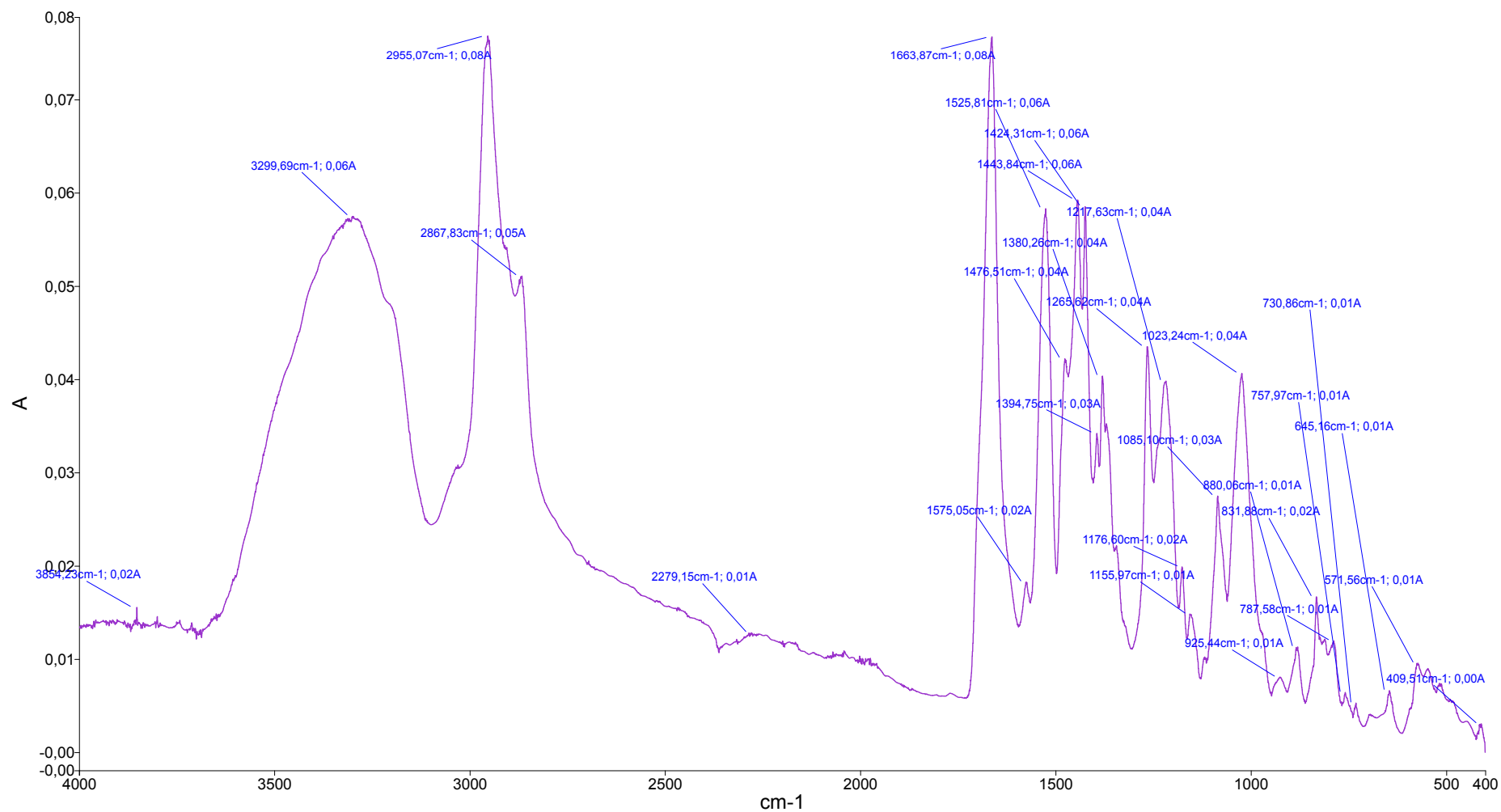


Fig. S163. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetraiodide (1,3-alternate **32**)

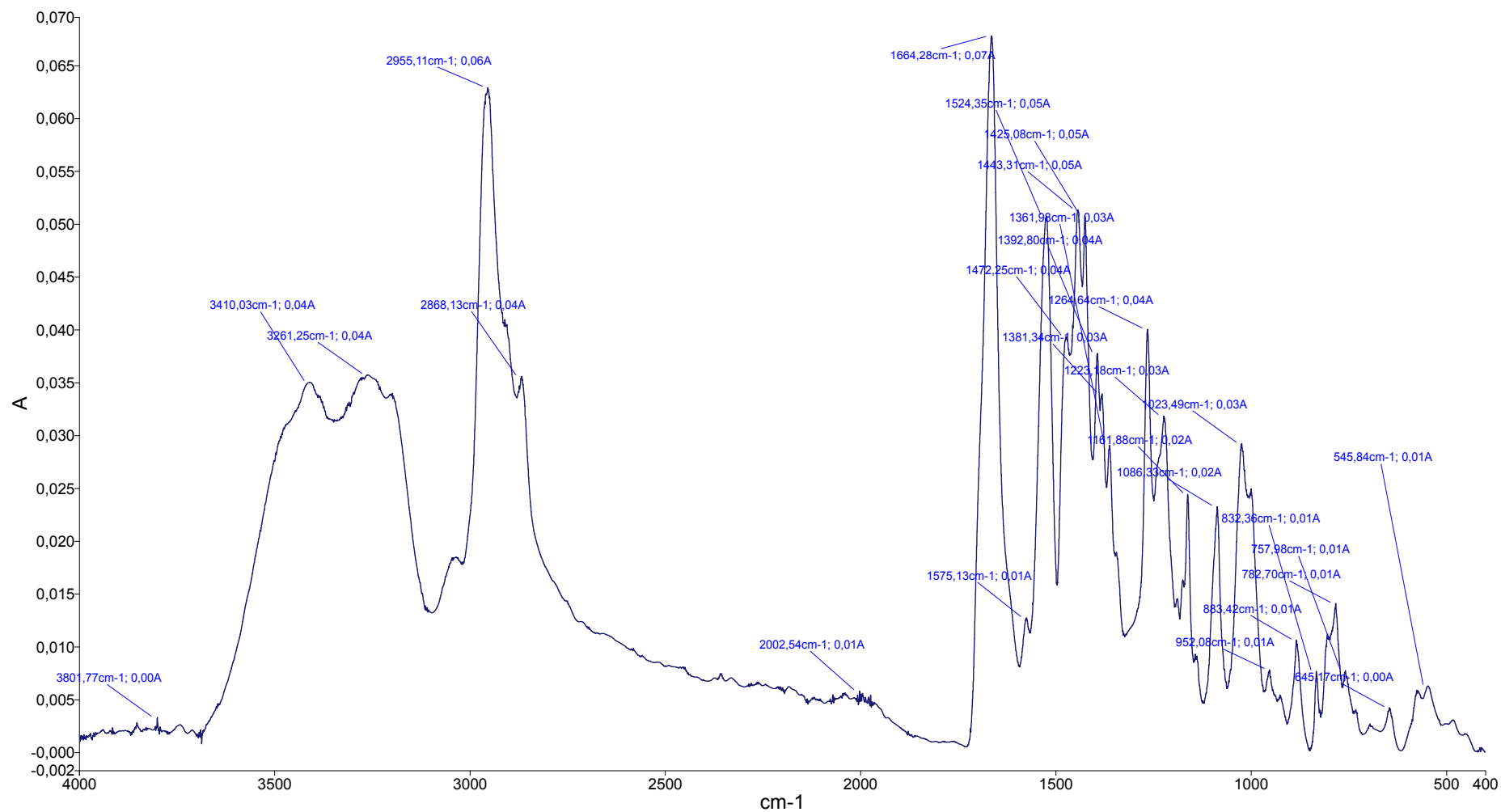


Fig. S164. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(2',2'-diethyl-2'-(benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 33)

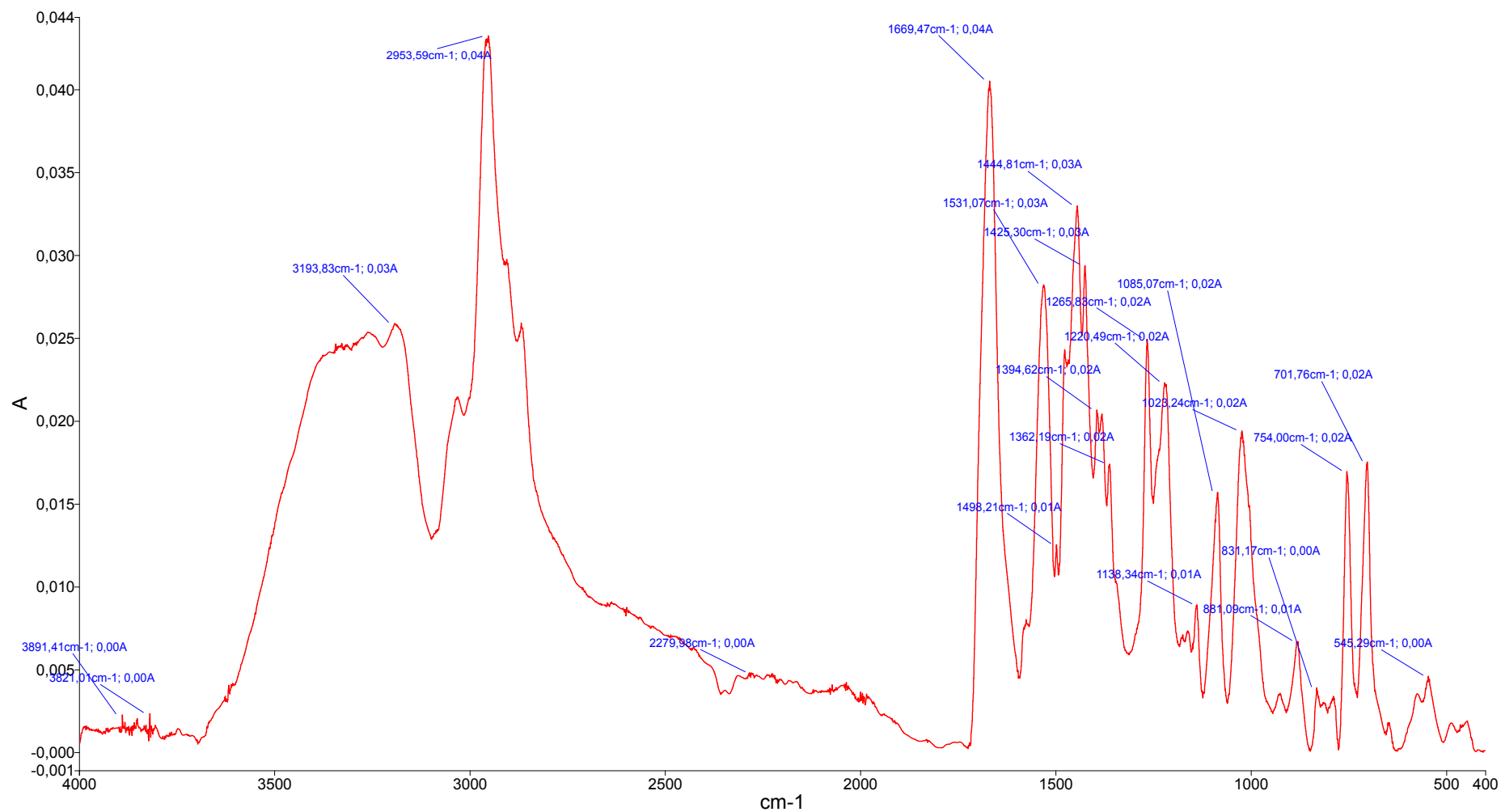


Fig. S165. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 34)

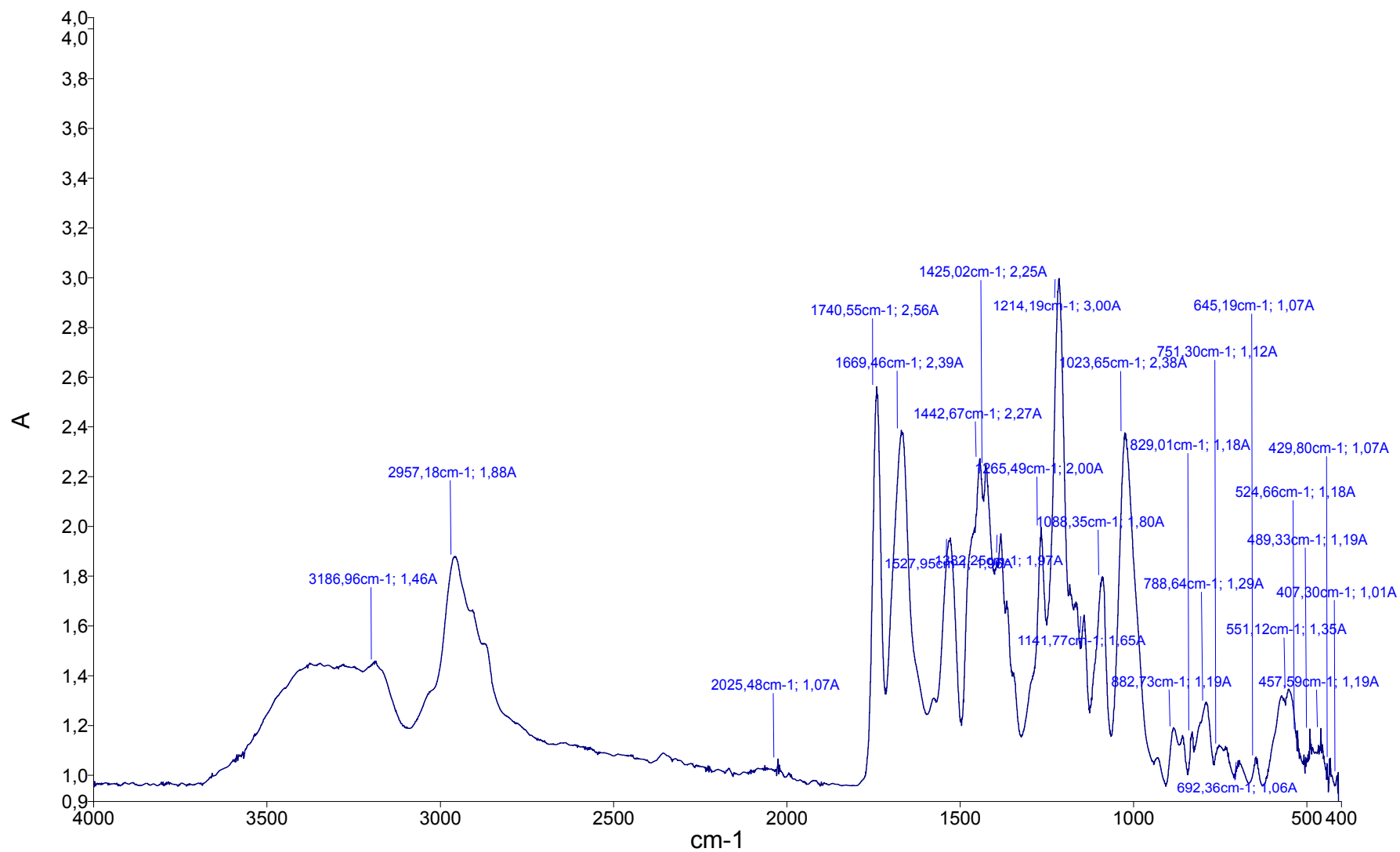


Fig. S166. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetrabromide (1,3-*alternate* 35)

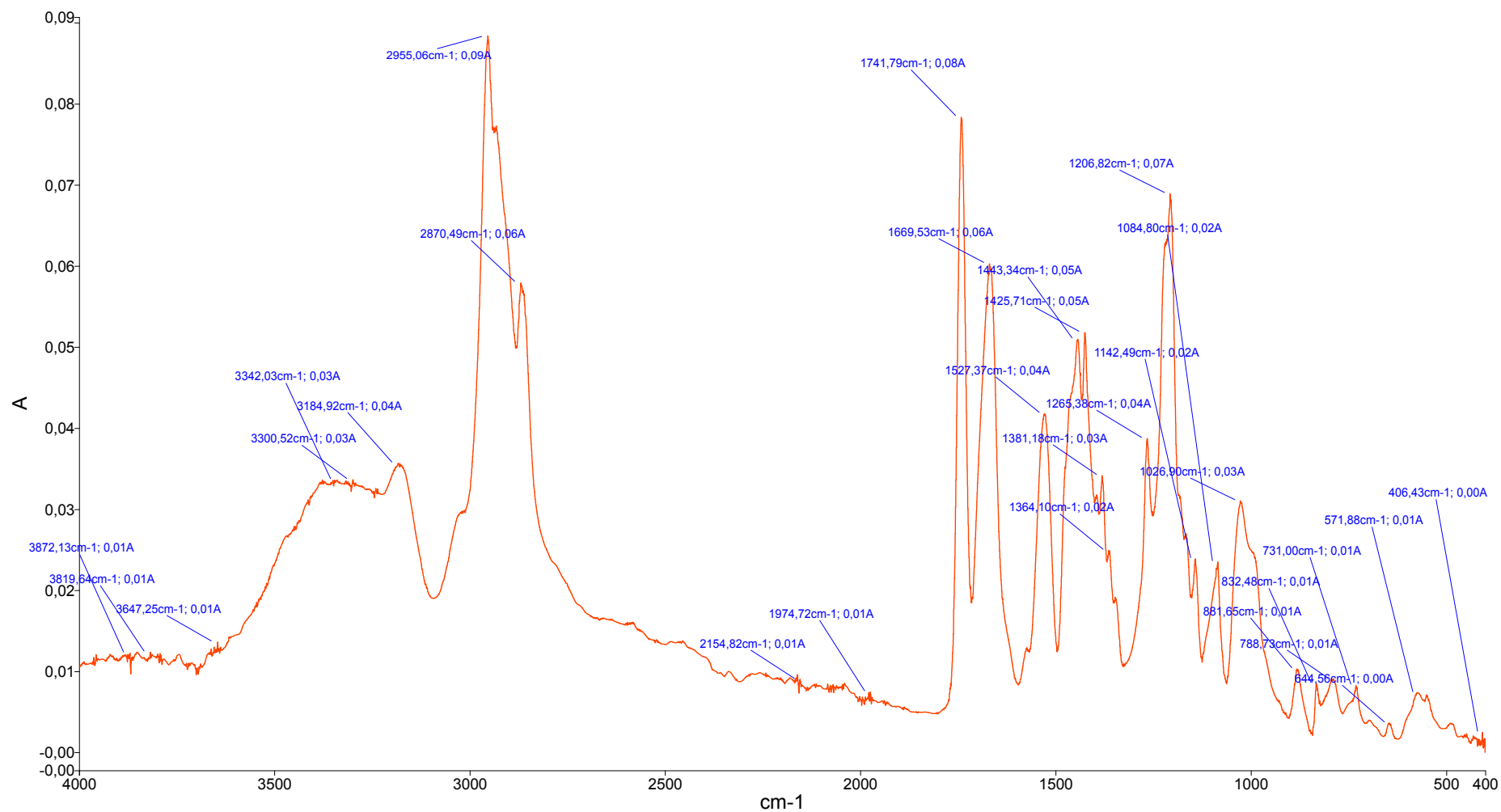


Fig. S167. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 39)

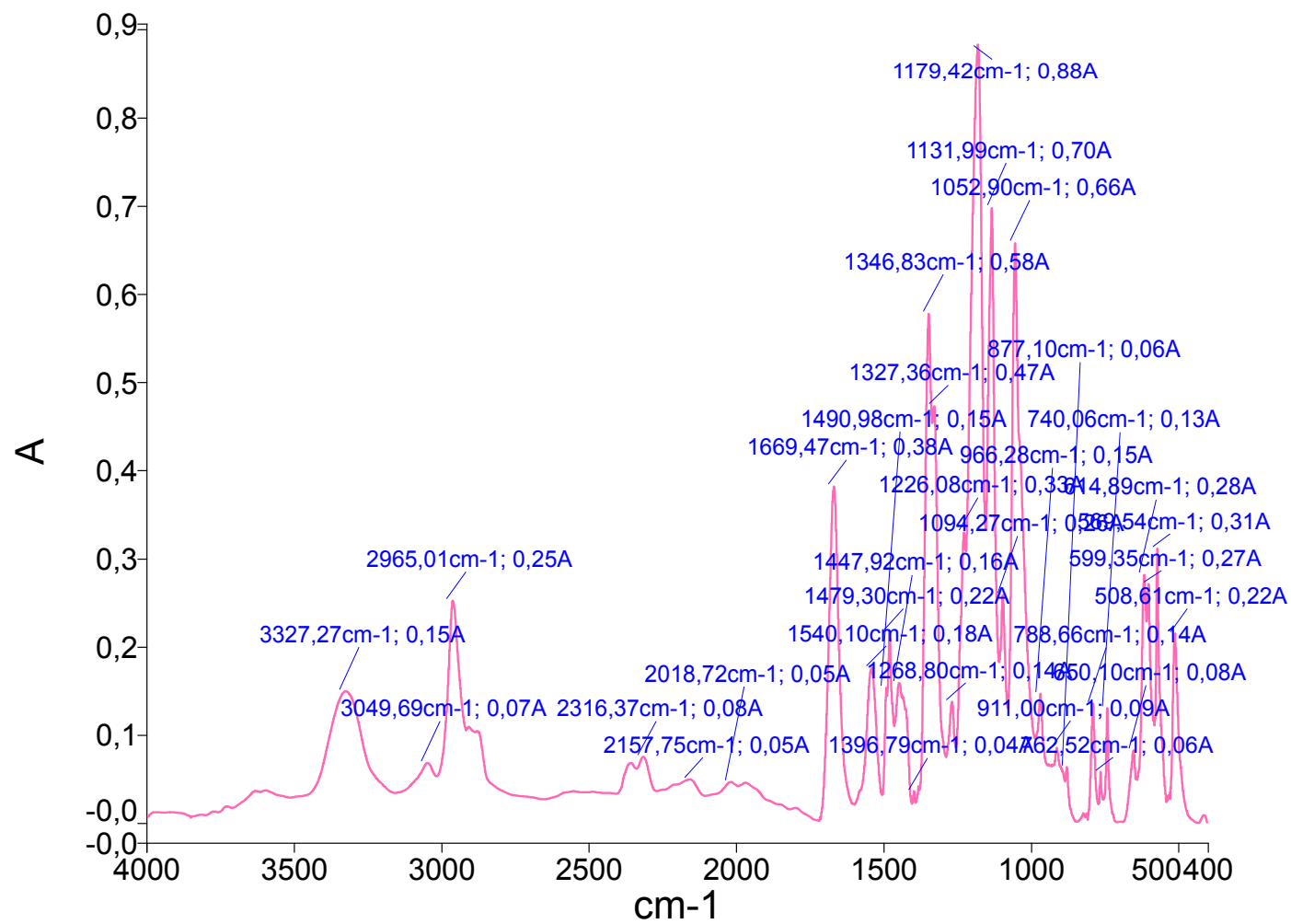


Fig. S168. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl]carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 40)

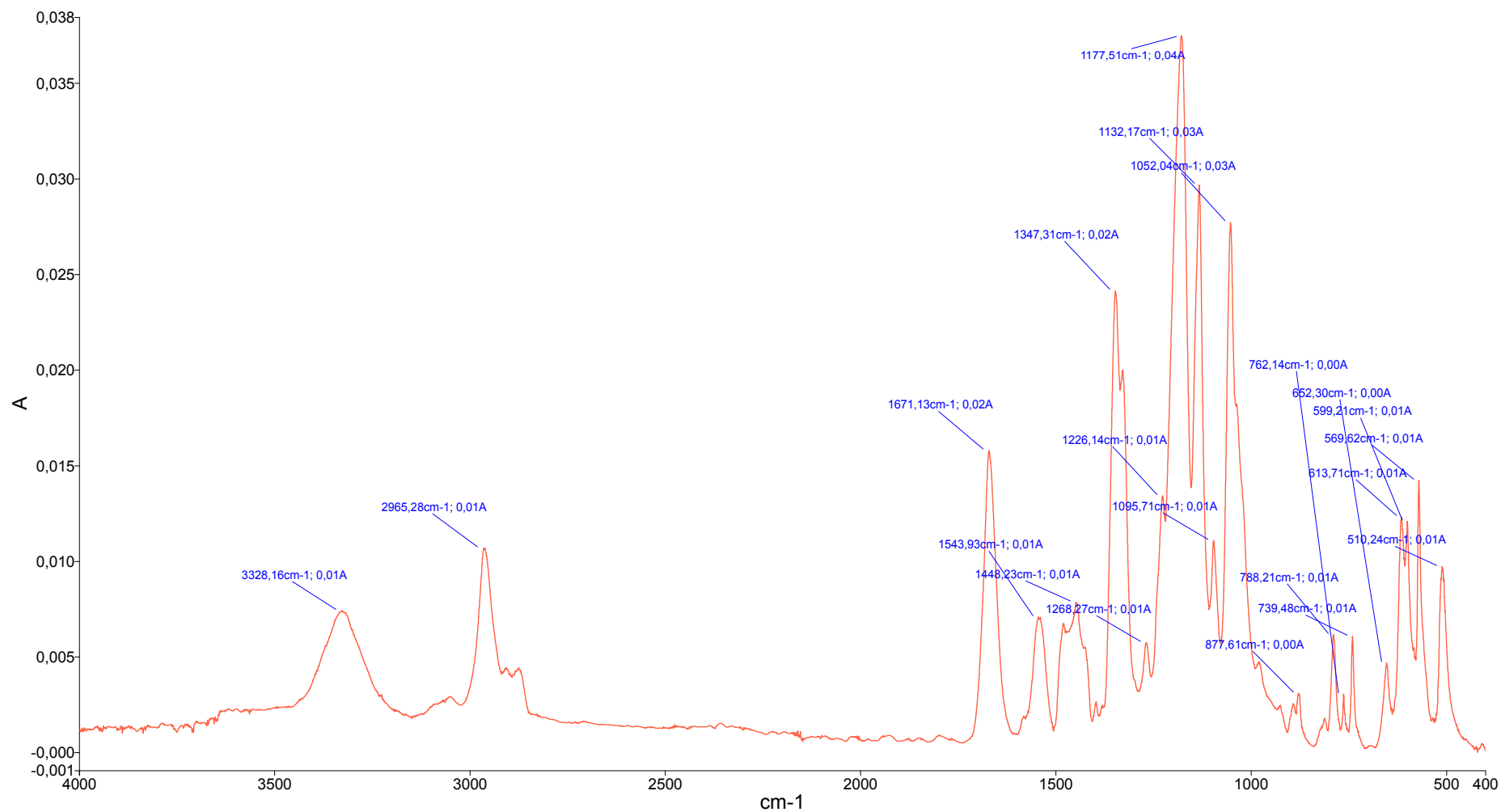


Fig. S169. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 41)

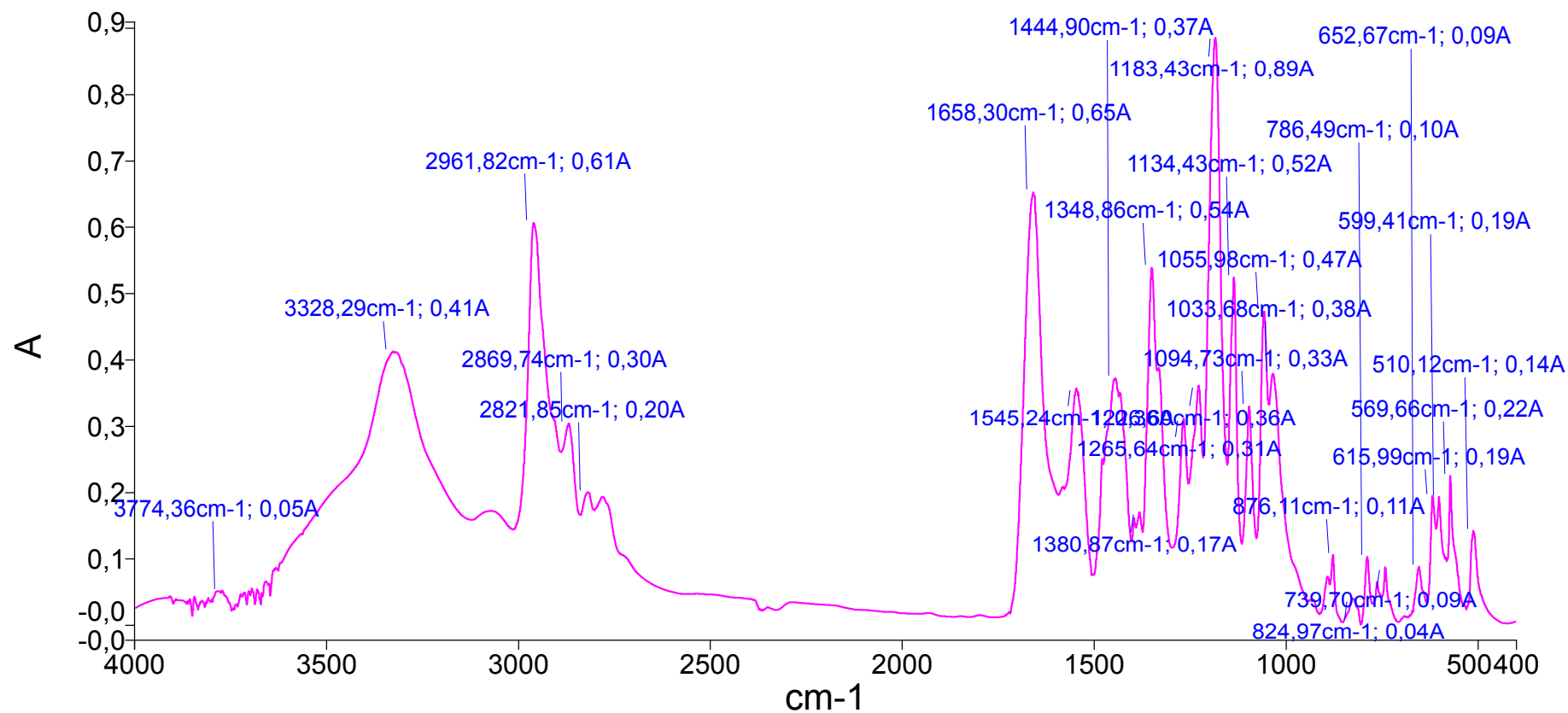


Fig. S170. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)ammoniumpropyl)-carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 42*)

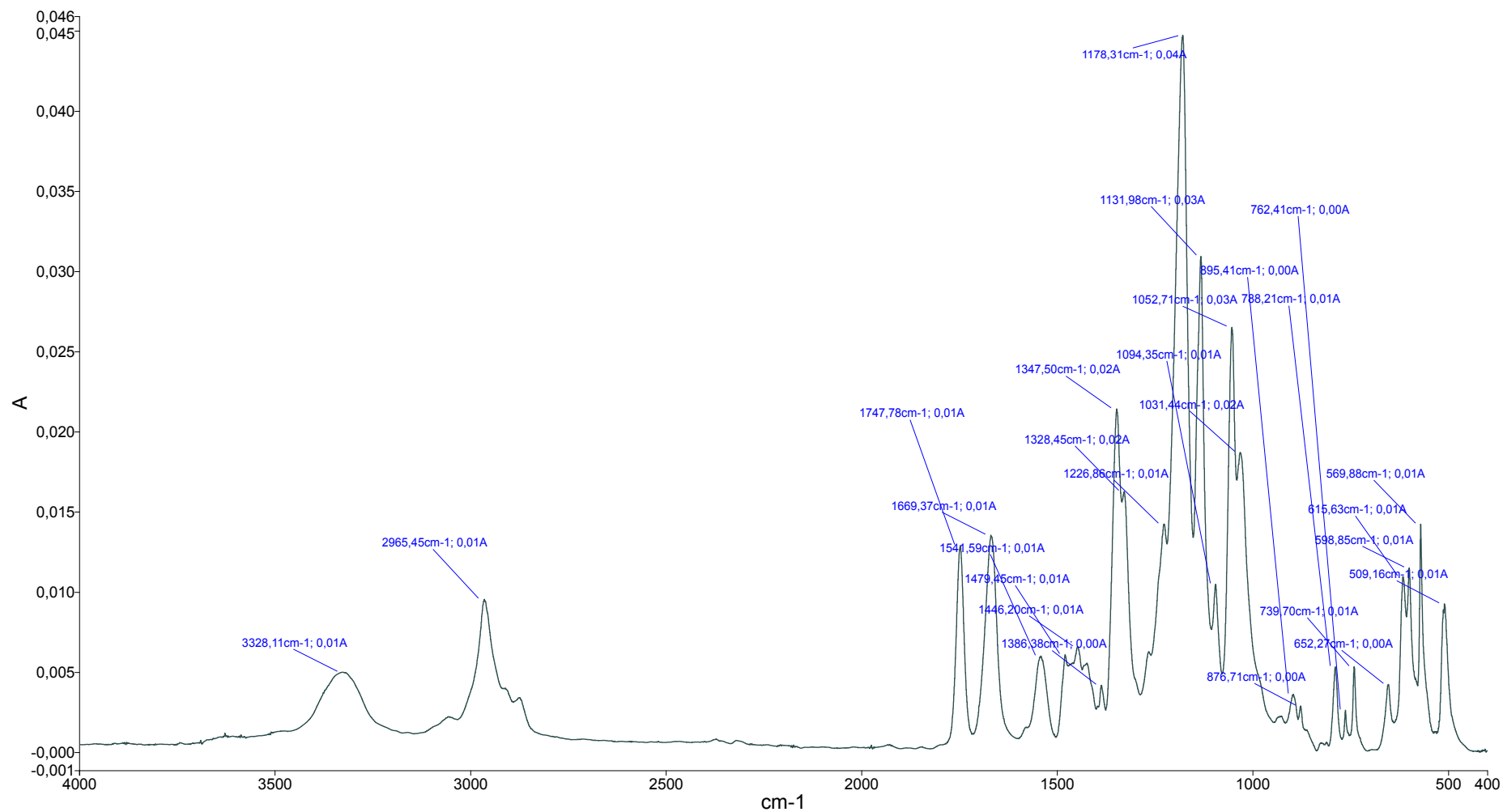


Fig. S171. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-(pentoxycarbonylmethyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 43)

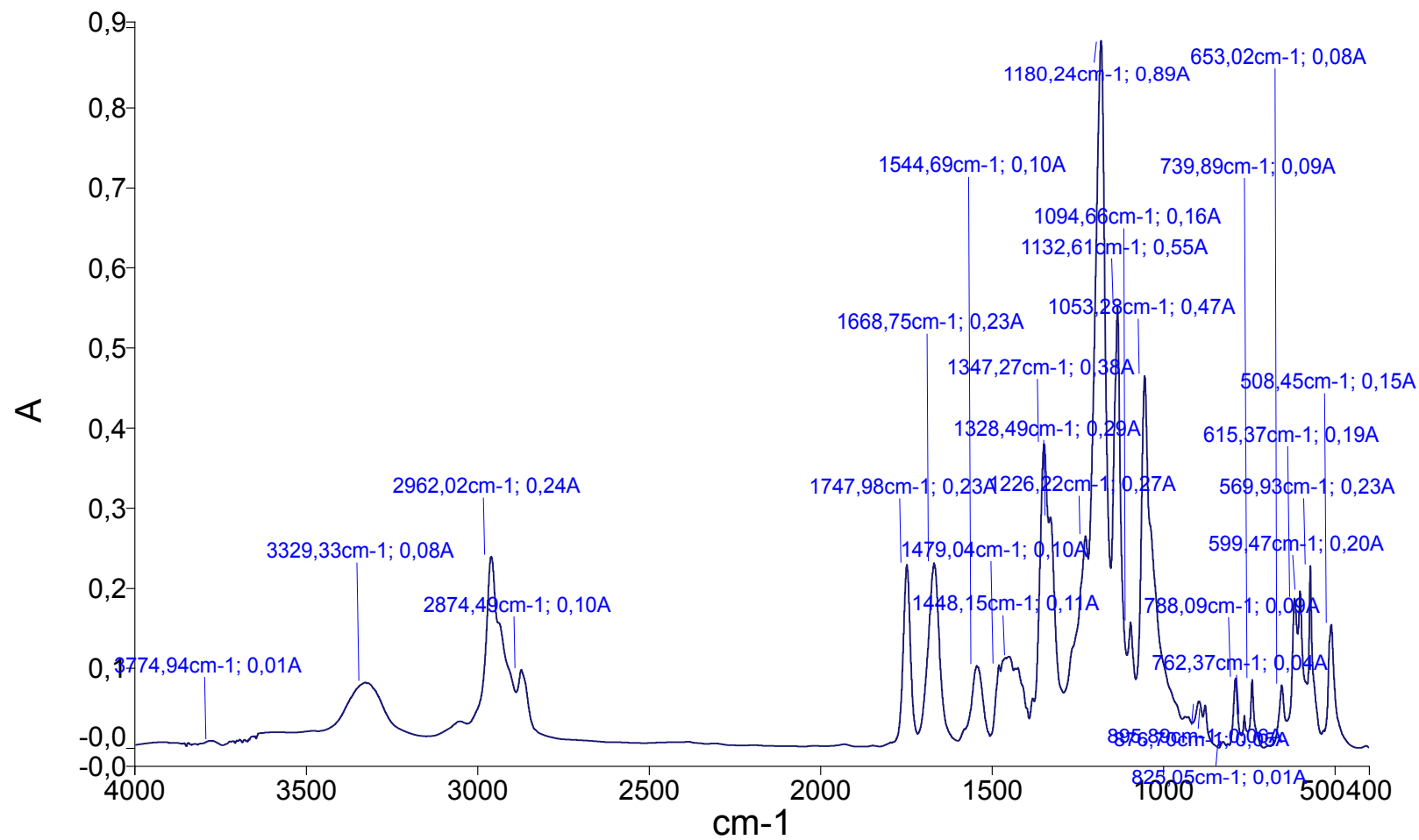


Fig. S172. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-{(ethoxycarbonylmethyl)amidocarbonylmethyl}-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 44*)

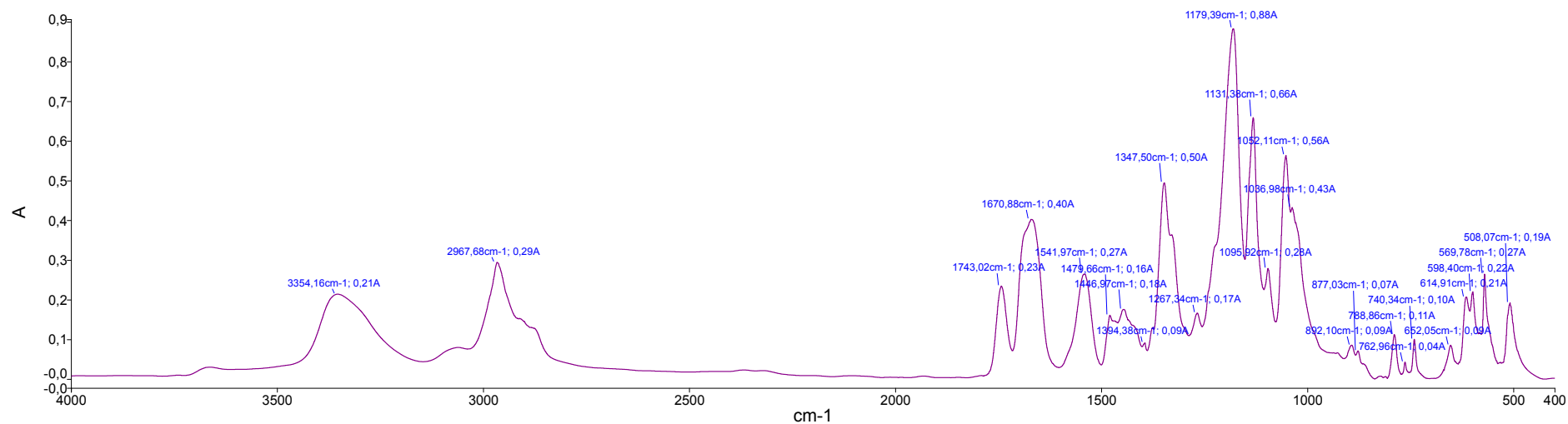


Fig. S173. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{[(ethoxycarbonylmethyl)-amidocarbonylmethyl]amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 45*)

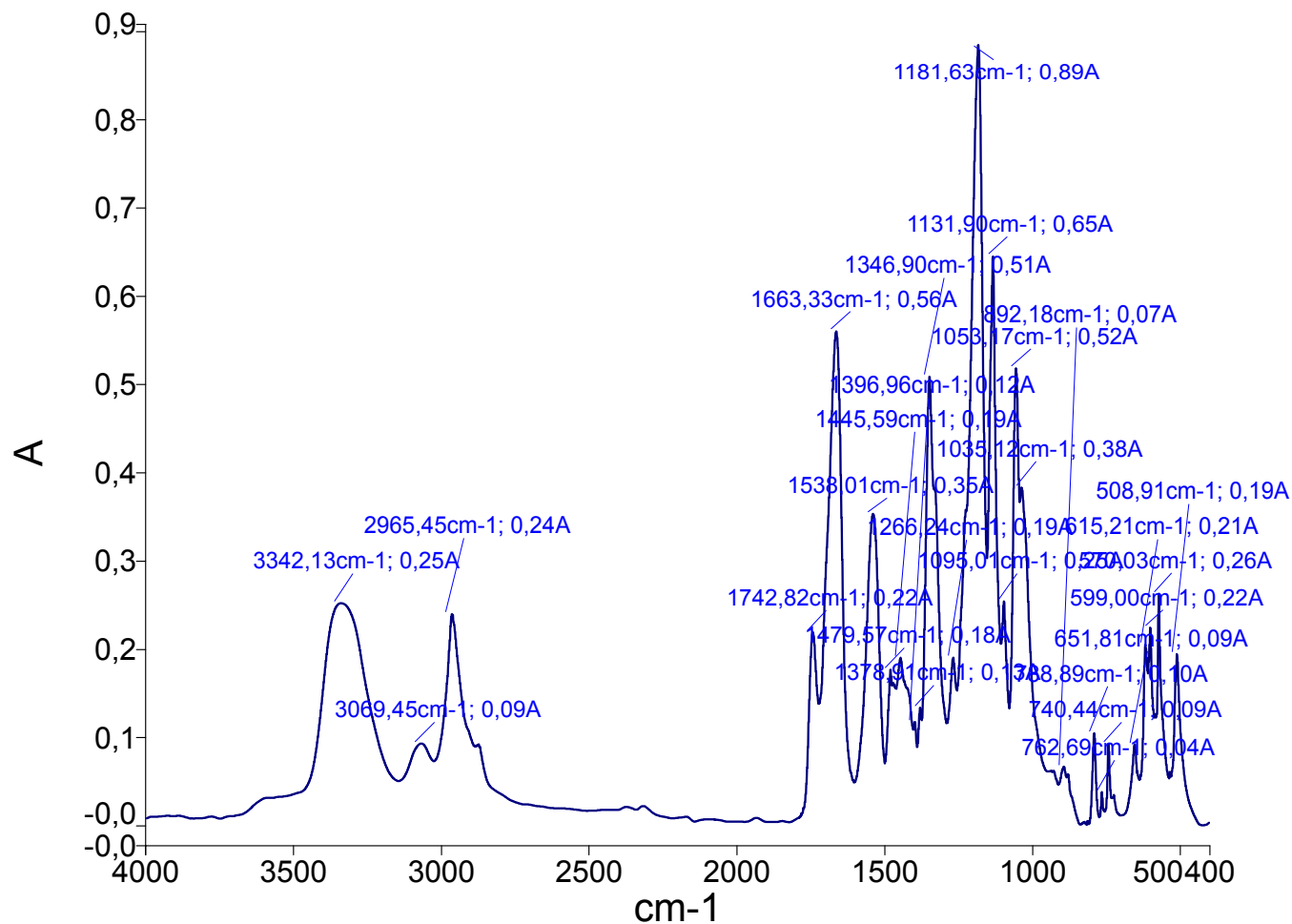


Fig. S174. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-{(ethoxycarbonyl[*S*-methyl]methyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiocalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone 46*)

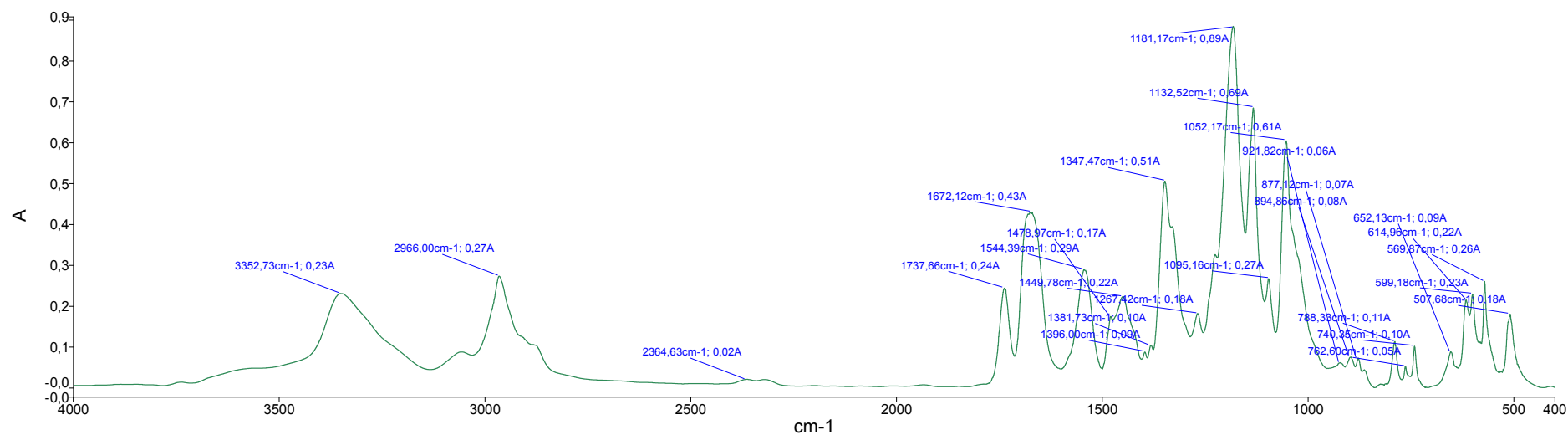


Fig. S175. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 47)

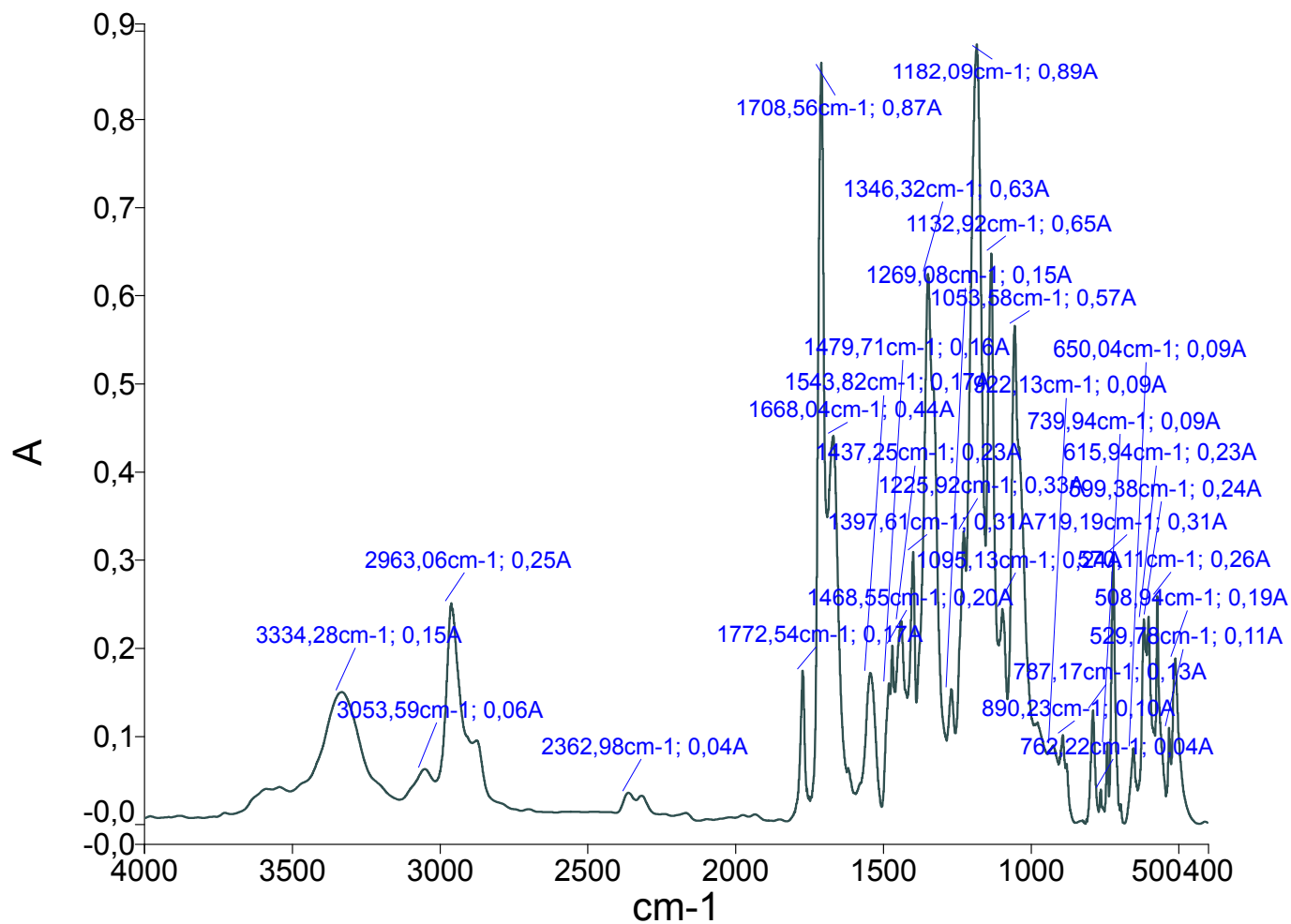


Fig. S176. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2'-methyl-2',2'-diethyl)ammoniummethyl)carbomoxymethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 48)

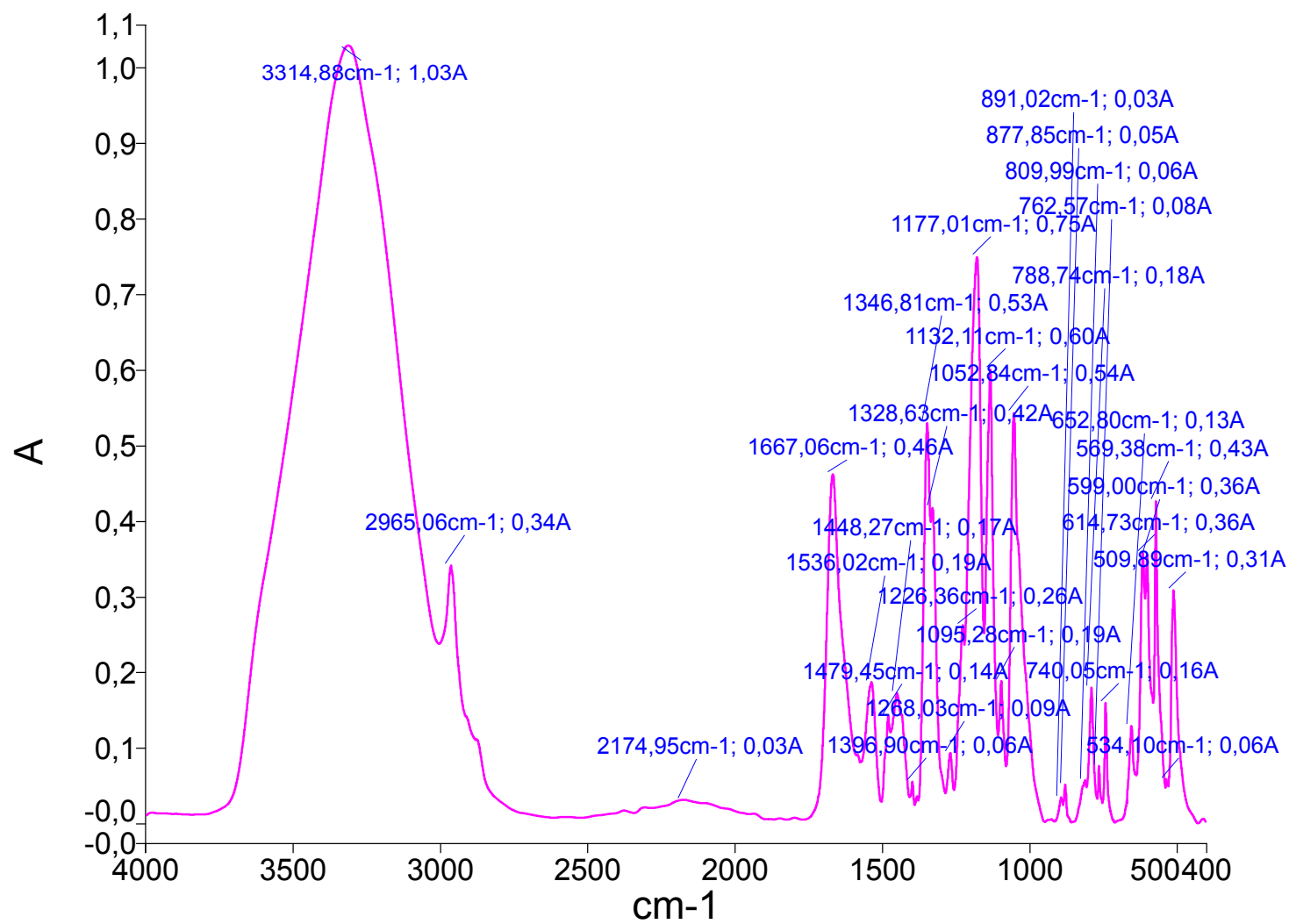


Fig. S177. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 49)

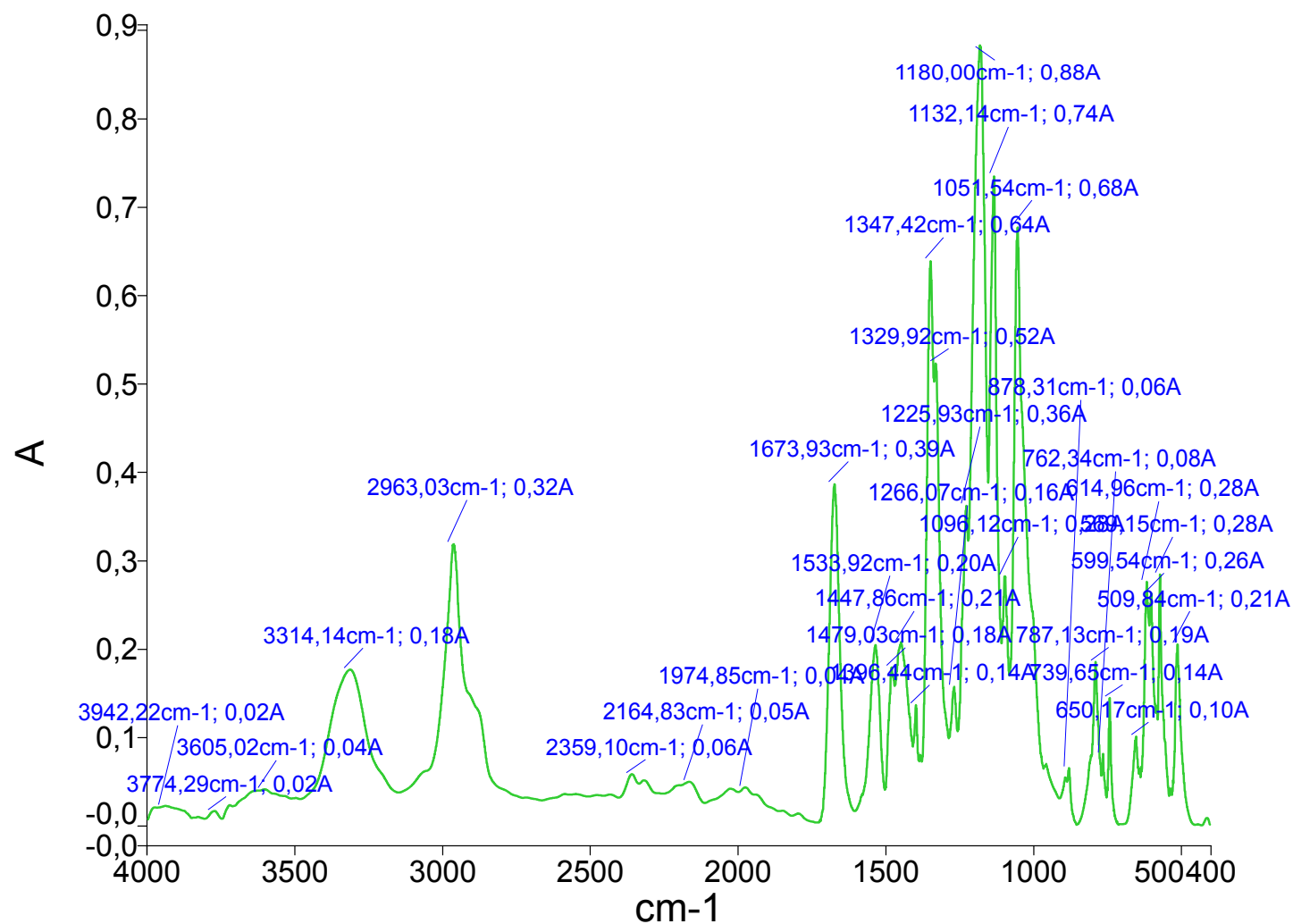


Fig. S178. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoxymethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 50)

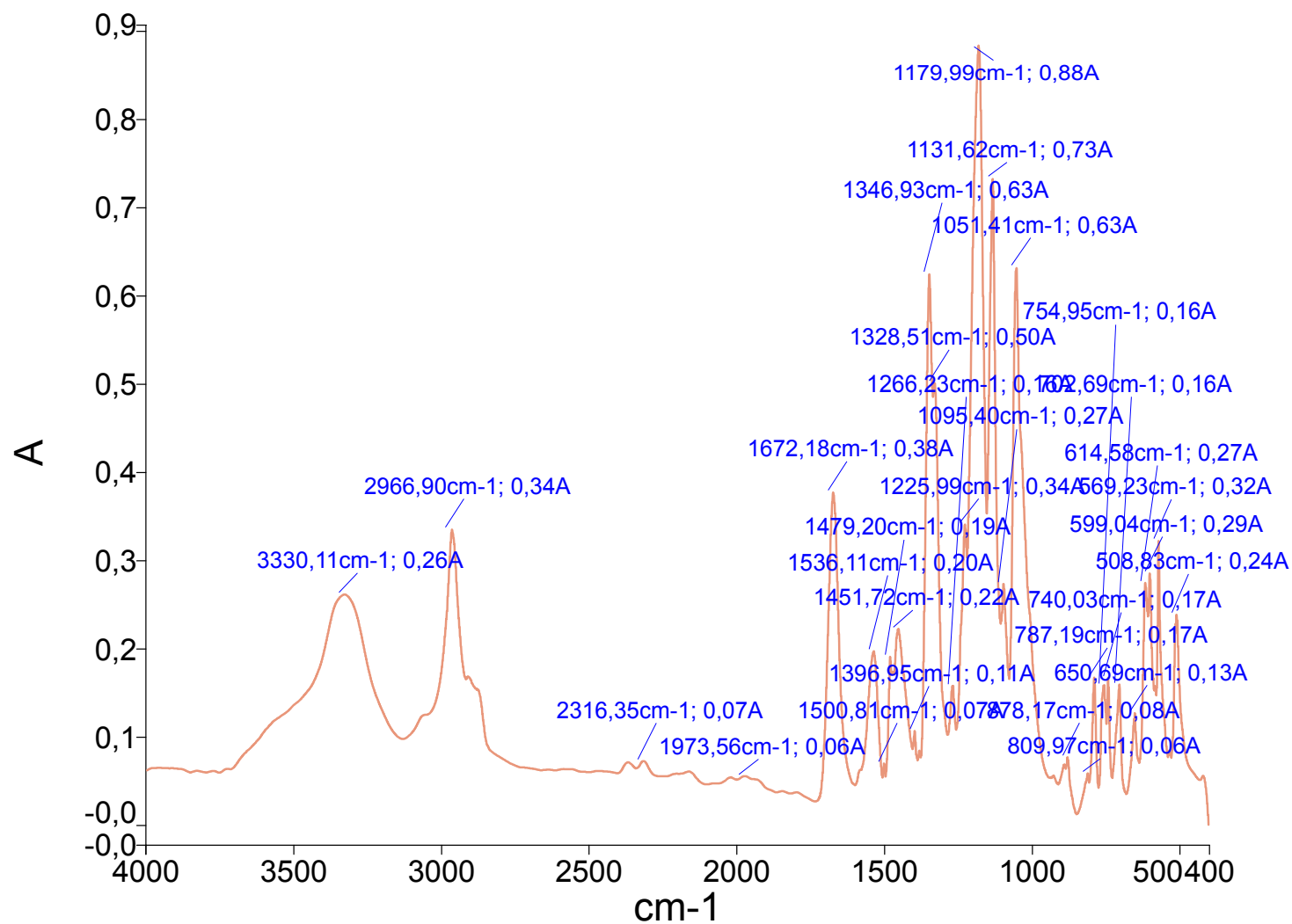


Fig. S179. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 51)

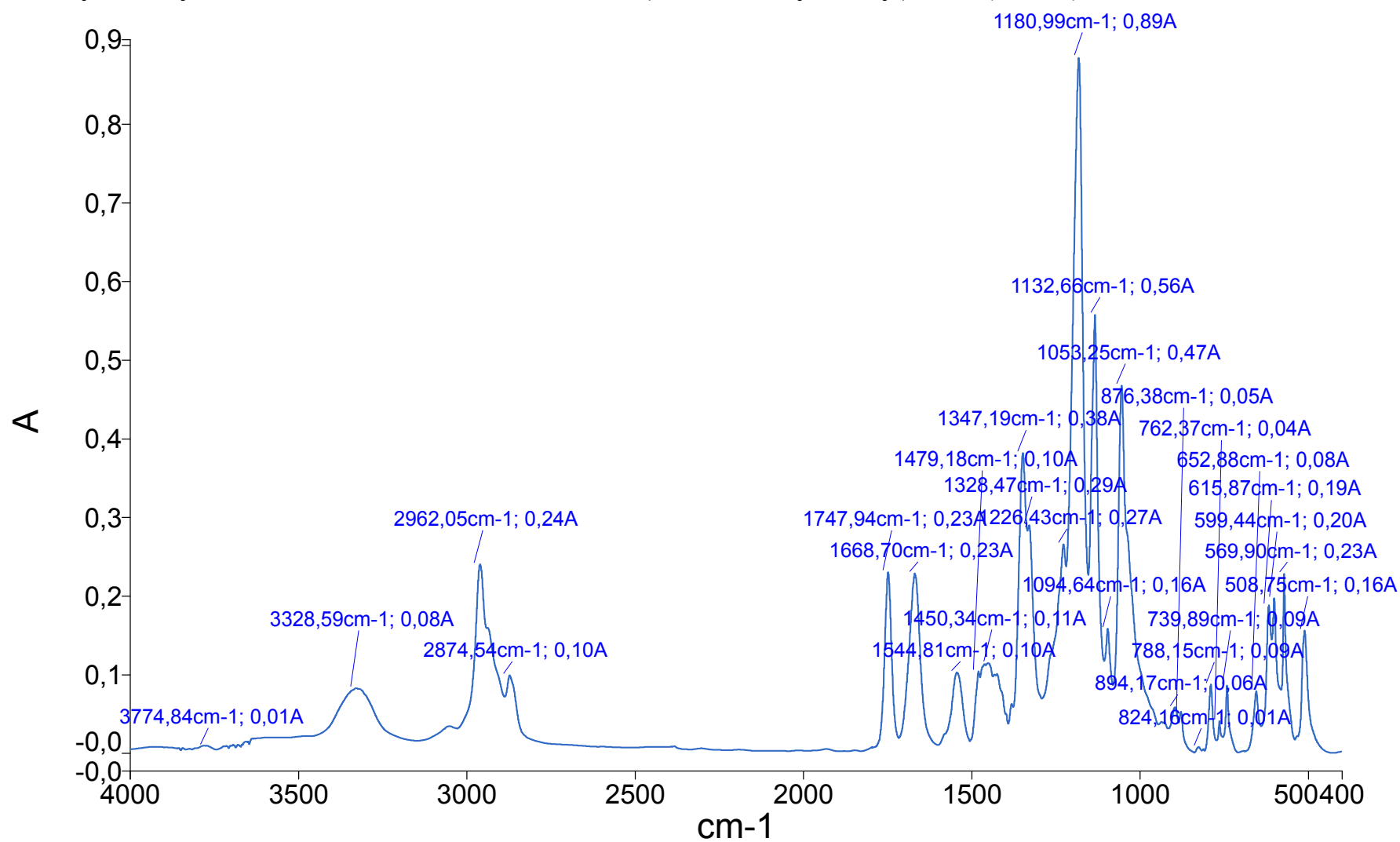


Fig. S180. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 52)

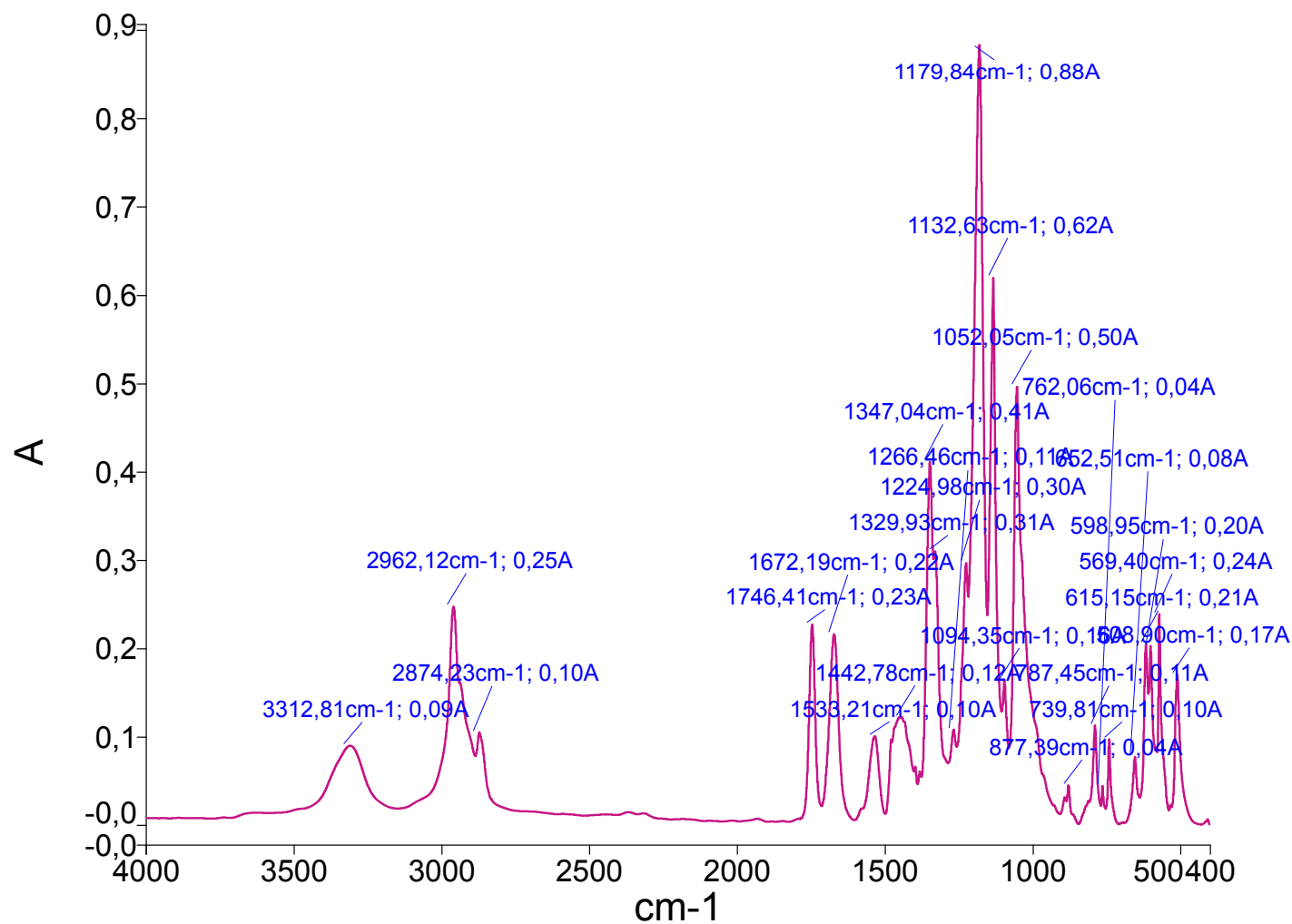


Fig. S181. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonylmethyl)amidocarbonylmethyl})-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 53)

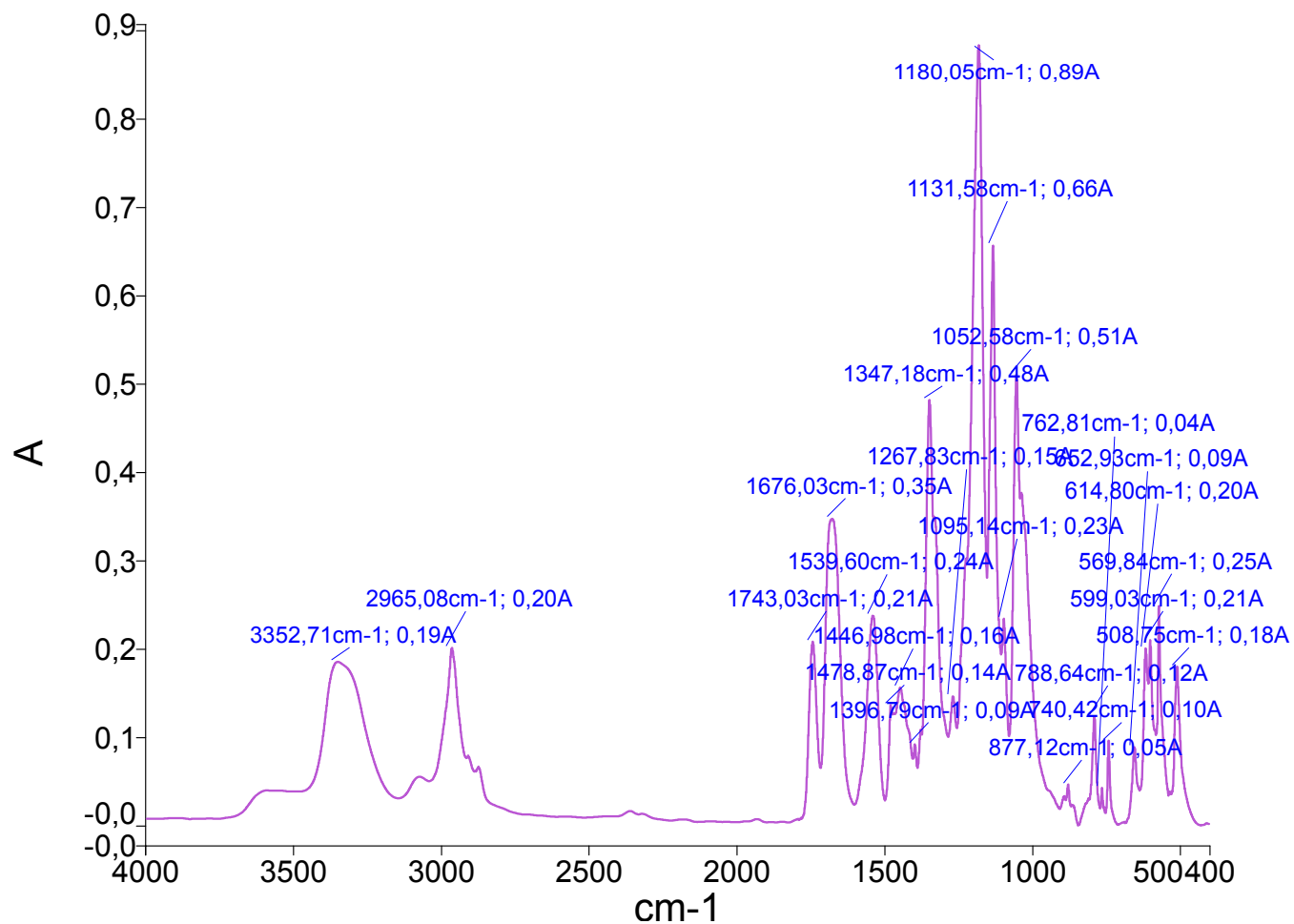


Fig. S182. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{[(ethoxycarbonylmethyl)-amidocarbonylmethyl]amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 54)

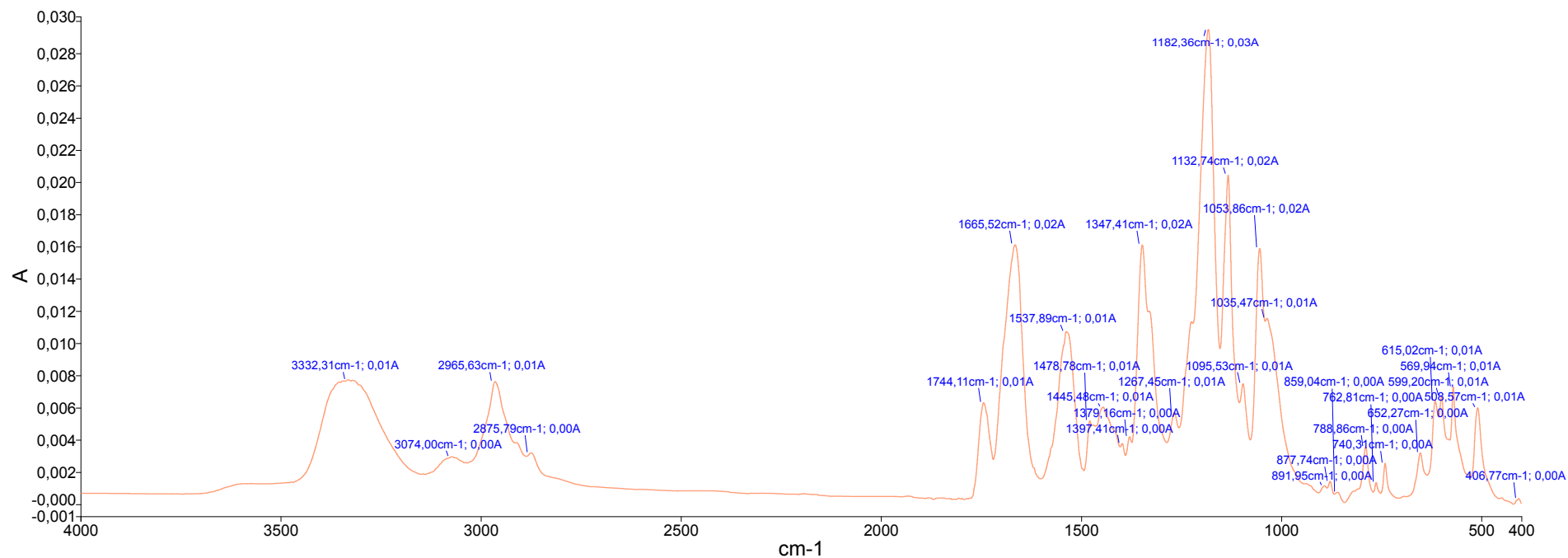


Fig. S183. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(2',2'-diethyl-2'-{(ethoxycarbonyl[*S*-methyl]methyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiocalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*cone* 55)

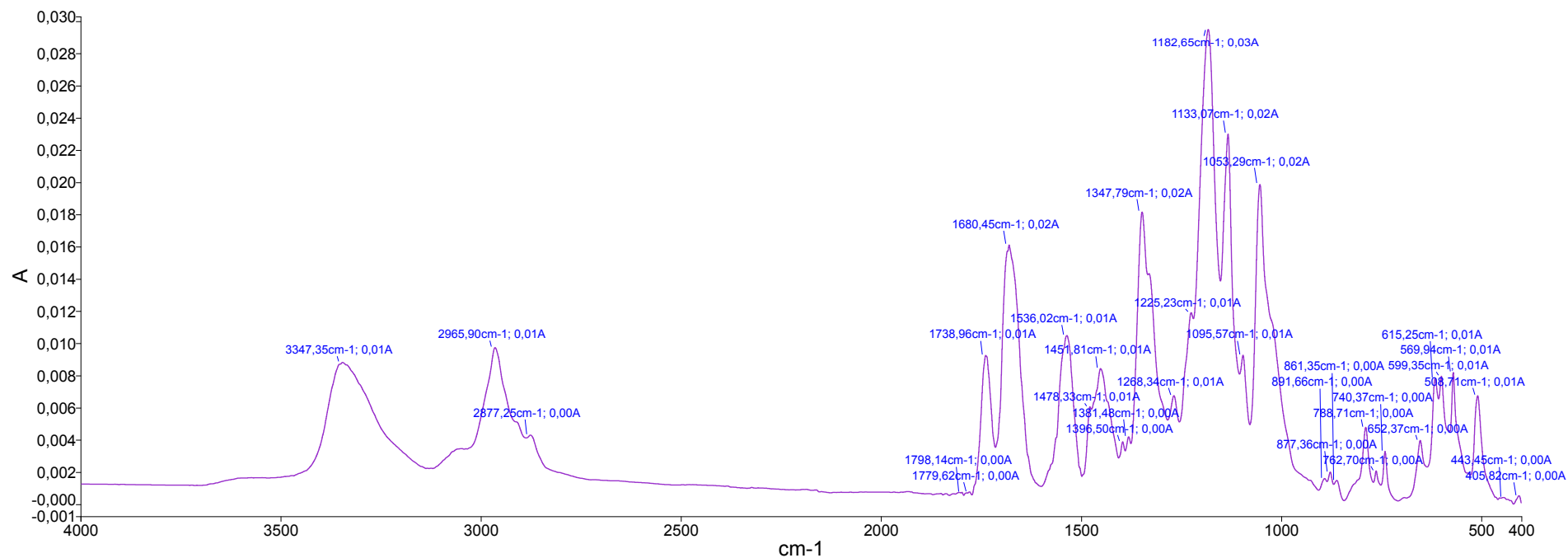


Fig. S184. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3',3'-trimethyl)ammoniumpropyl)carbomoxymethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 56)

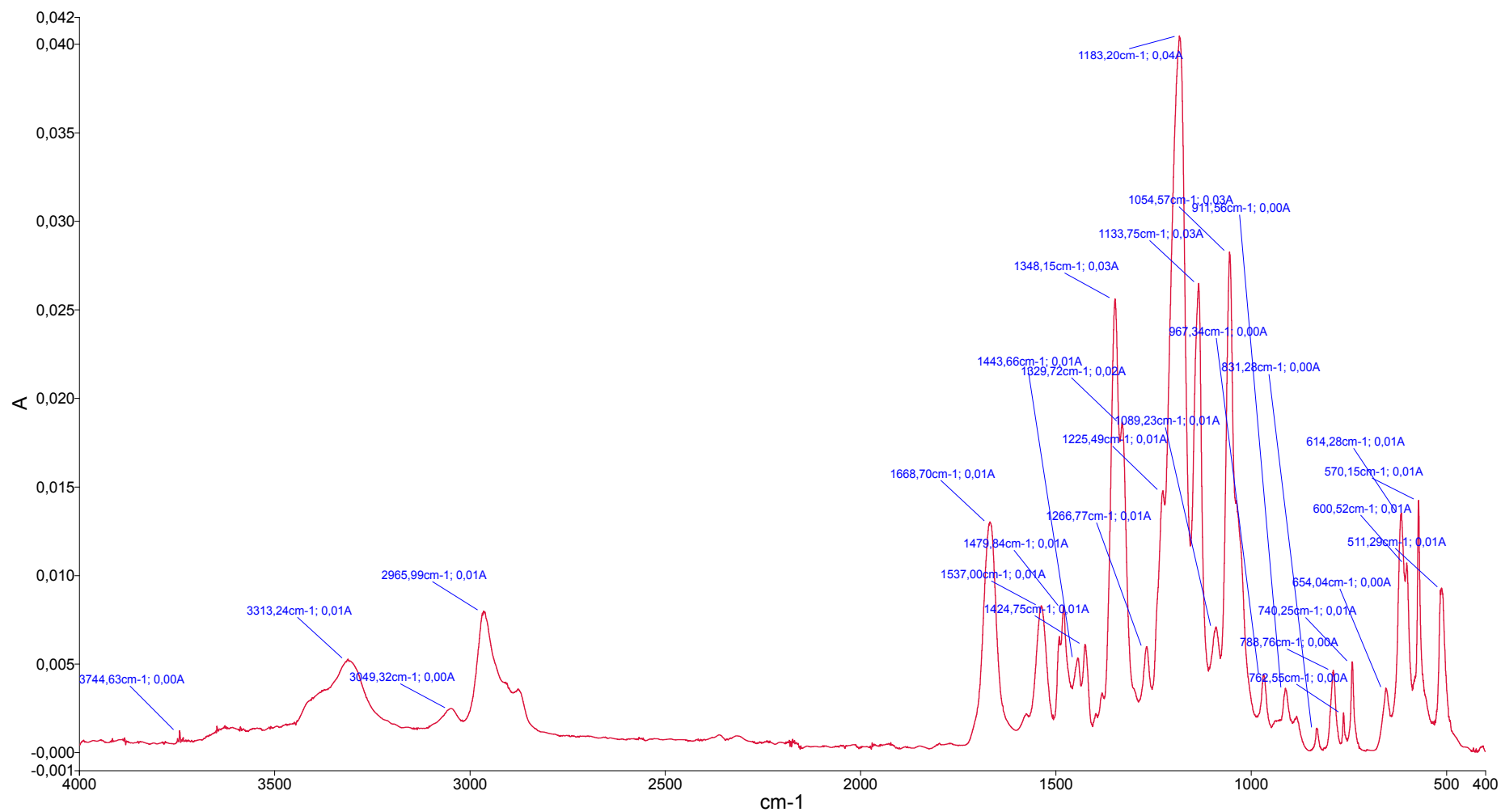


Fig. S185. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-ethyl)ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 57)

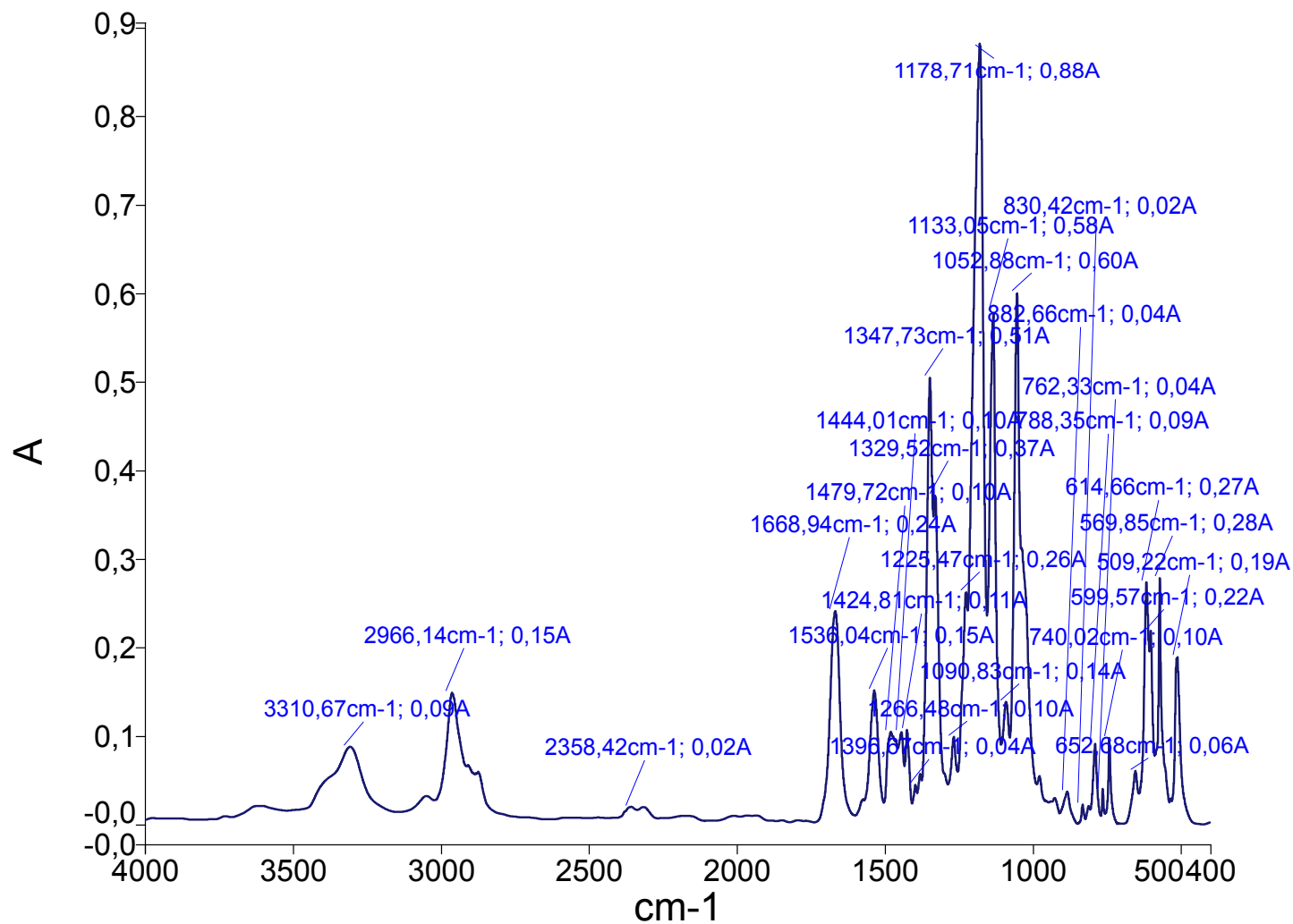


Fig. S186. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-benzyl)ammoniumpropyl)carbomoxymethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 58)

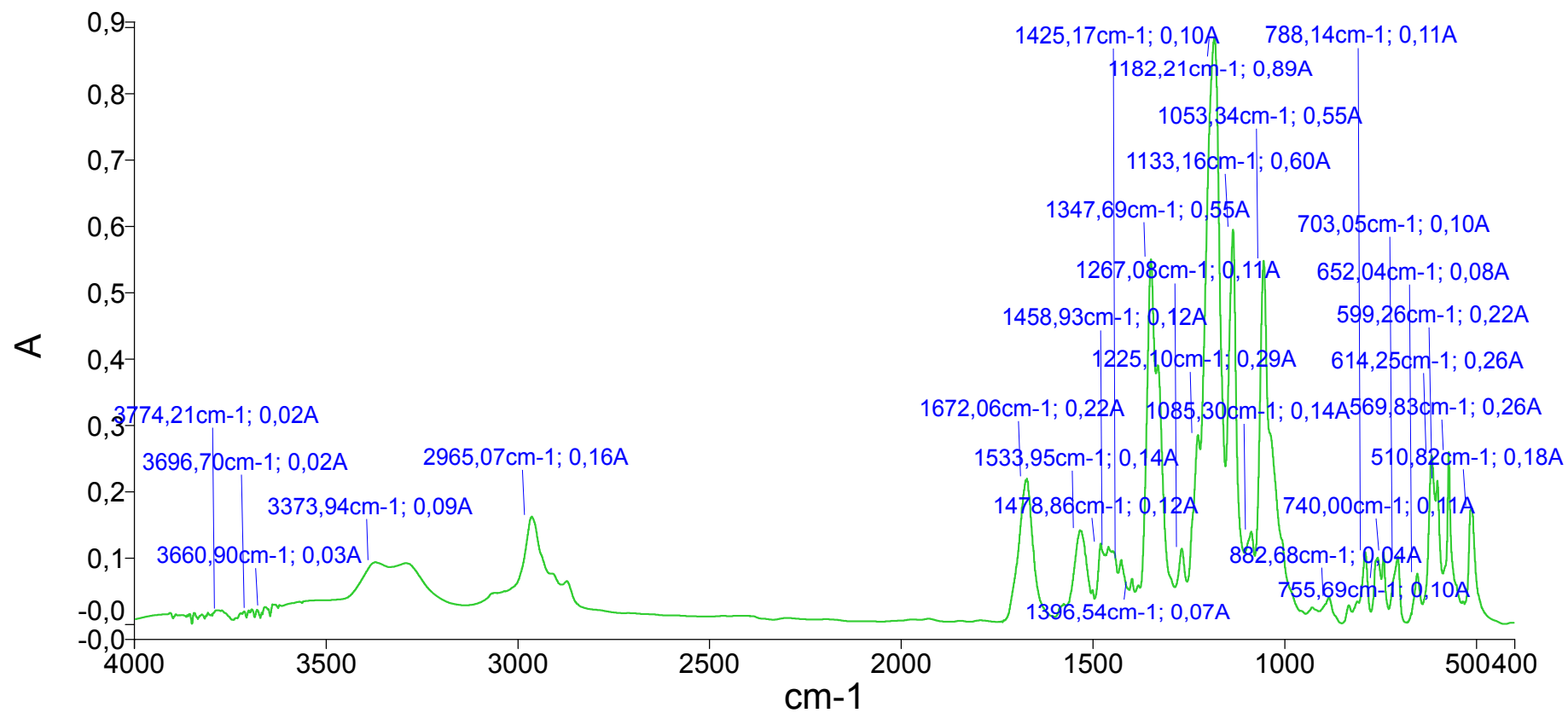


Fig. S187. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-(ethoxycarbonylmethyl)ammoniumpropyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate 59*)

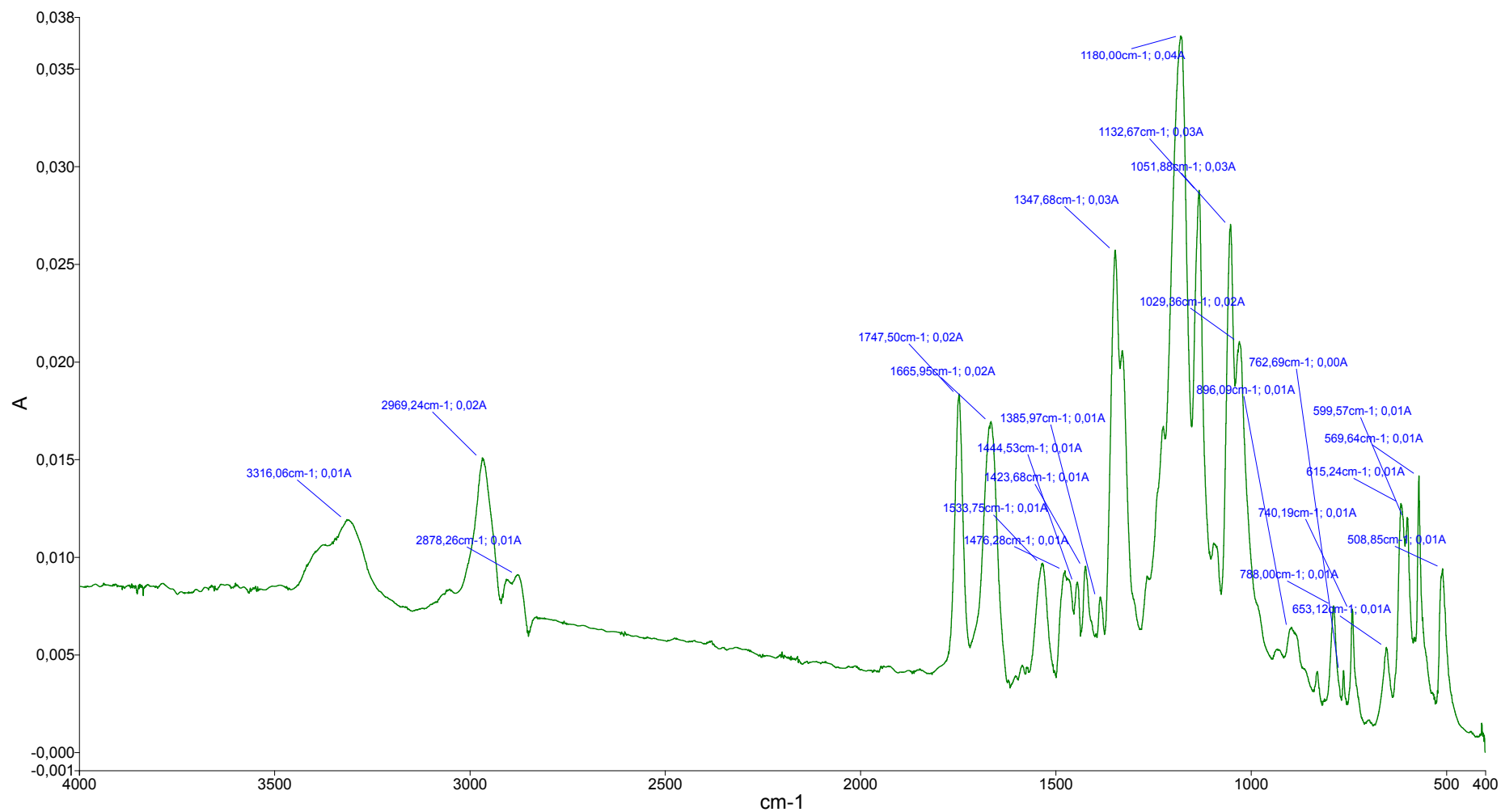


Fig. S188. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-(pentoxycarbonylmethyl)ammoniumpropyl)-carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 60)

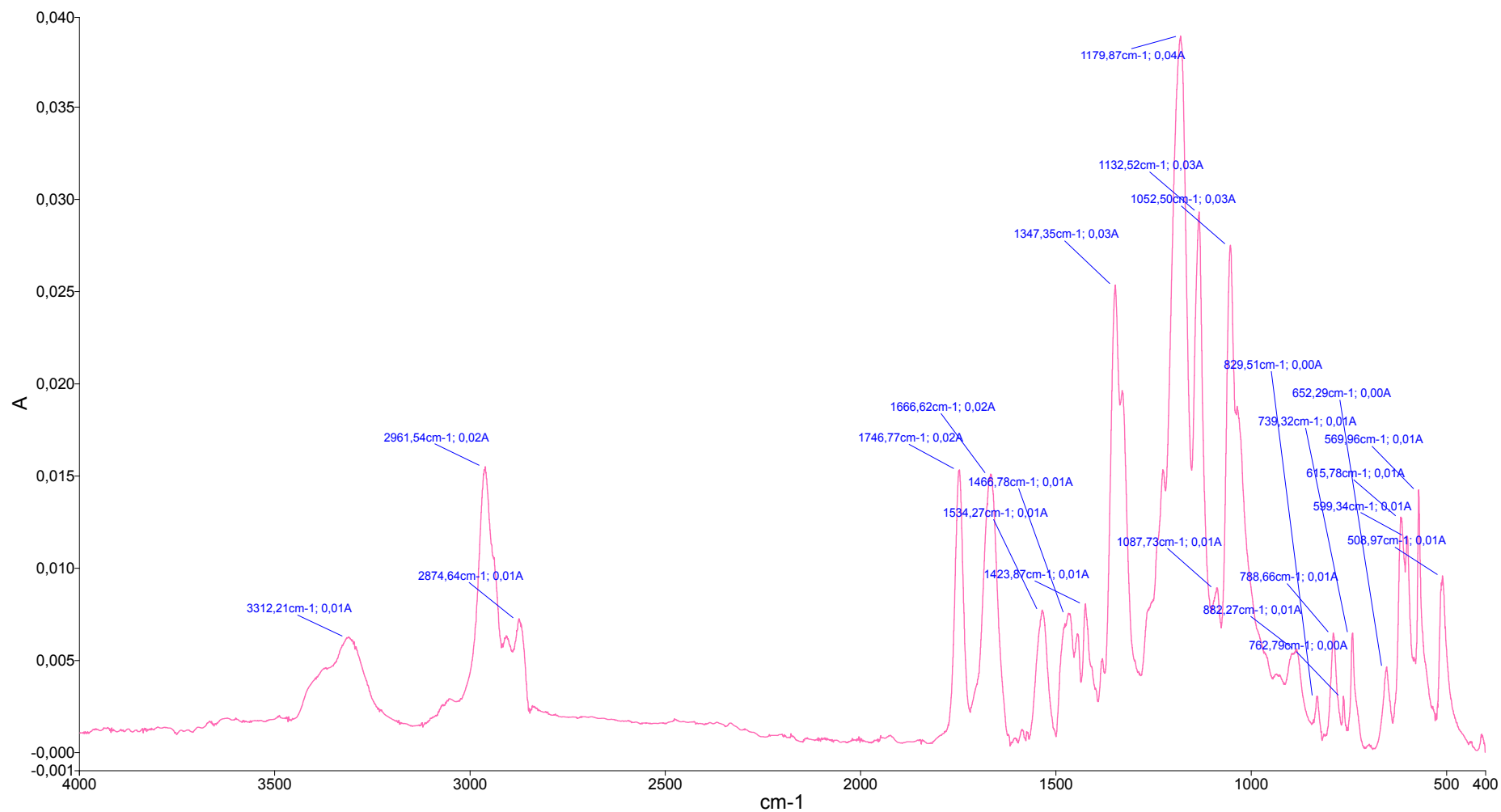


Fig. S189. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-{(ethoxycarbonylmethyl)amidocarbonylmethyl}-ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate **61**)

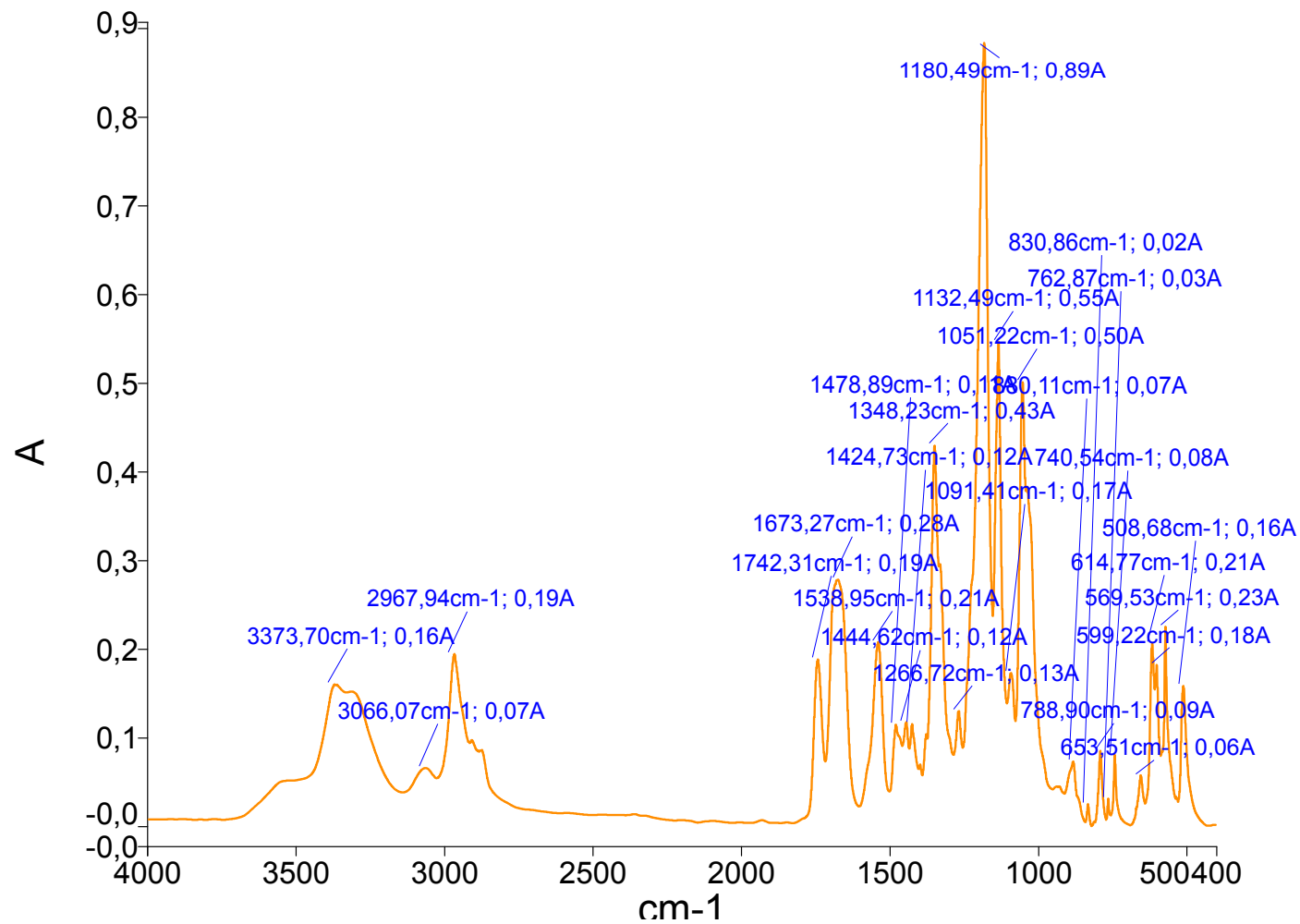


Fig. S190. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-{([ethoxycarbonylmethyl]amidocarbonylmethyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (*1,3-alternate 62*)

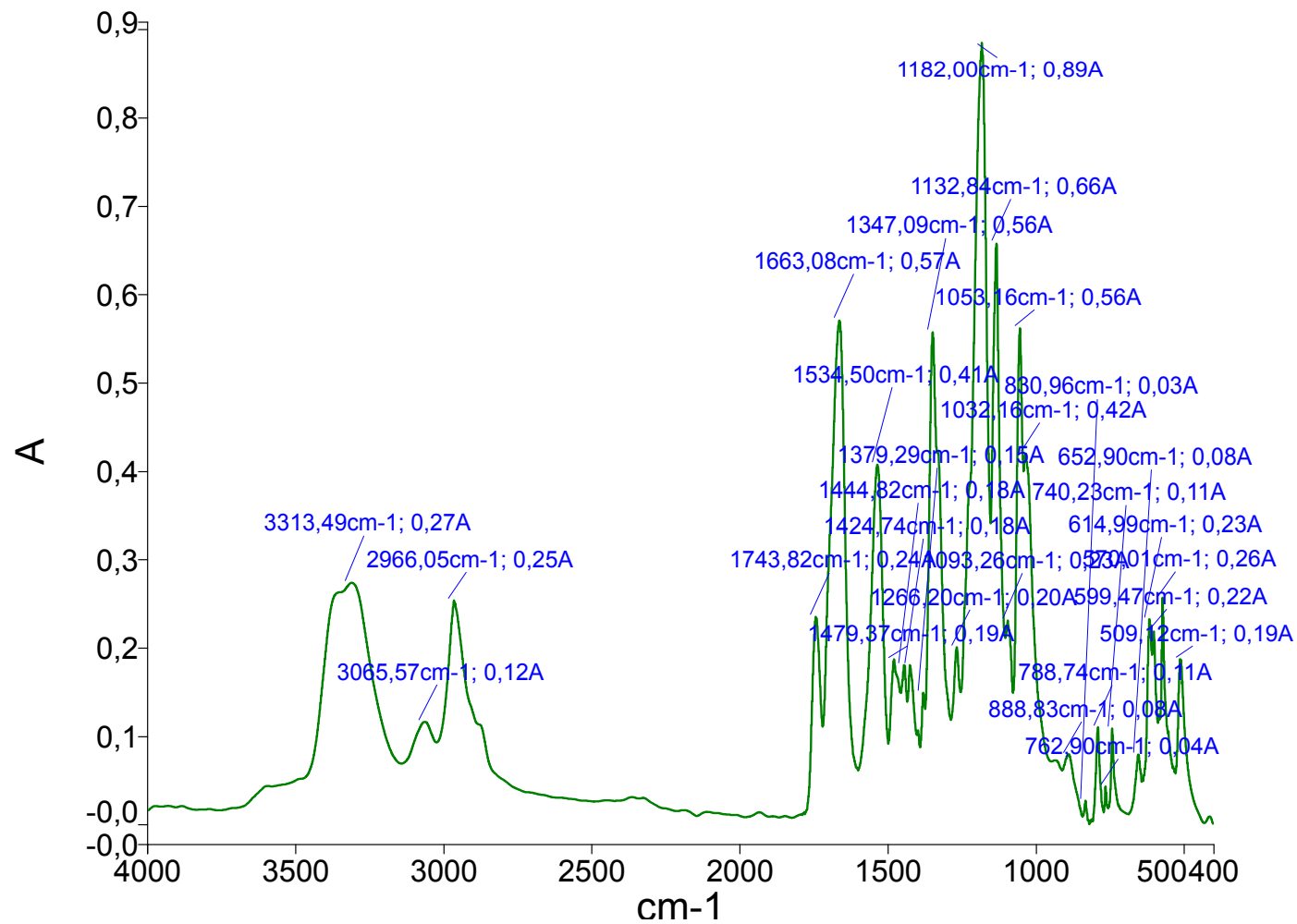


Fig. S191. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(3',3'-dimethyl-3'-{(ethoxycarbonyl[*S*-methyl]methyl)amidocarbonylmethyl}ammoniumpropyl)carbomoylmethoxy]-2,8,14,20-tetrathiocalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 63)

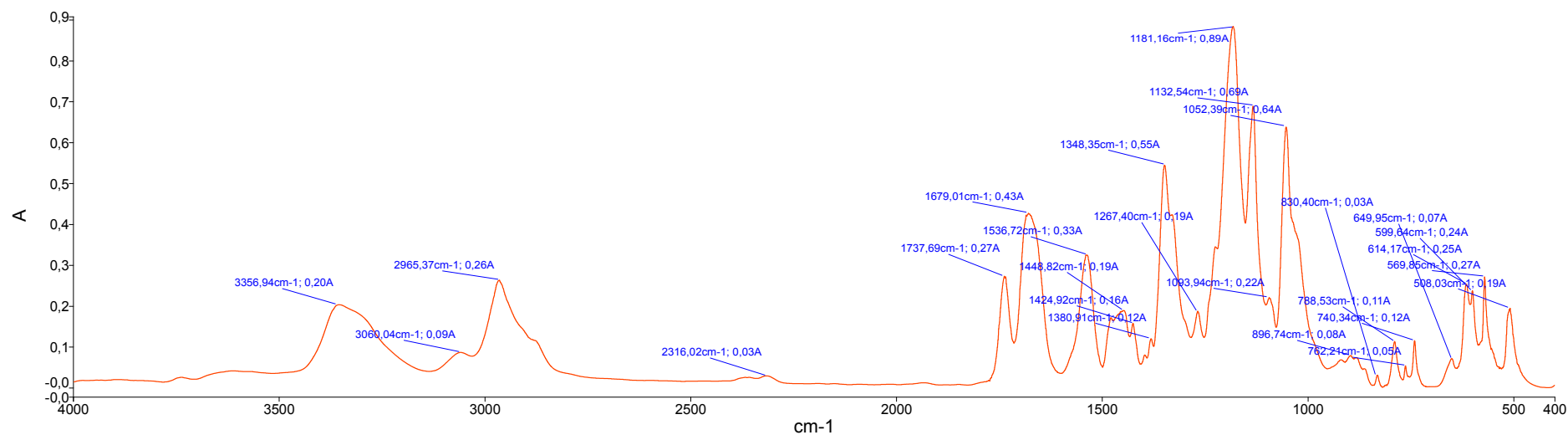


Fig. S192. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(3',3'-dimethyl-3'-{3''-propylphthalimide}ammoniumpropyl)-carbamoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate **64**)

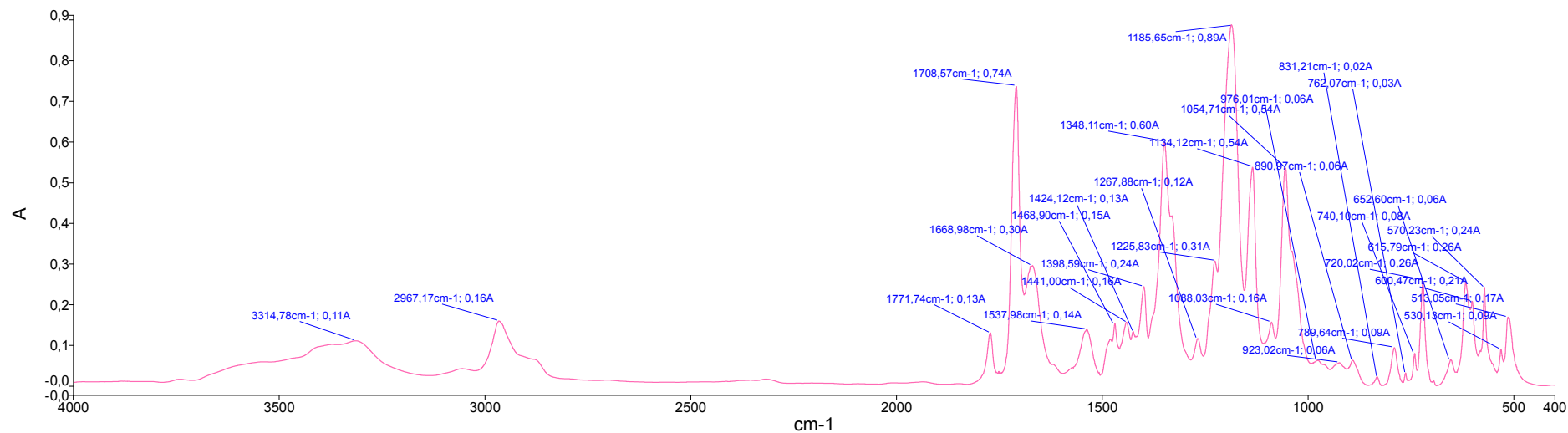


Fig. S193. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[N-(2'-methyl-2',2'-diethyl)ammoniummethyl]carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 65)

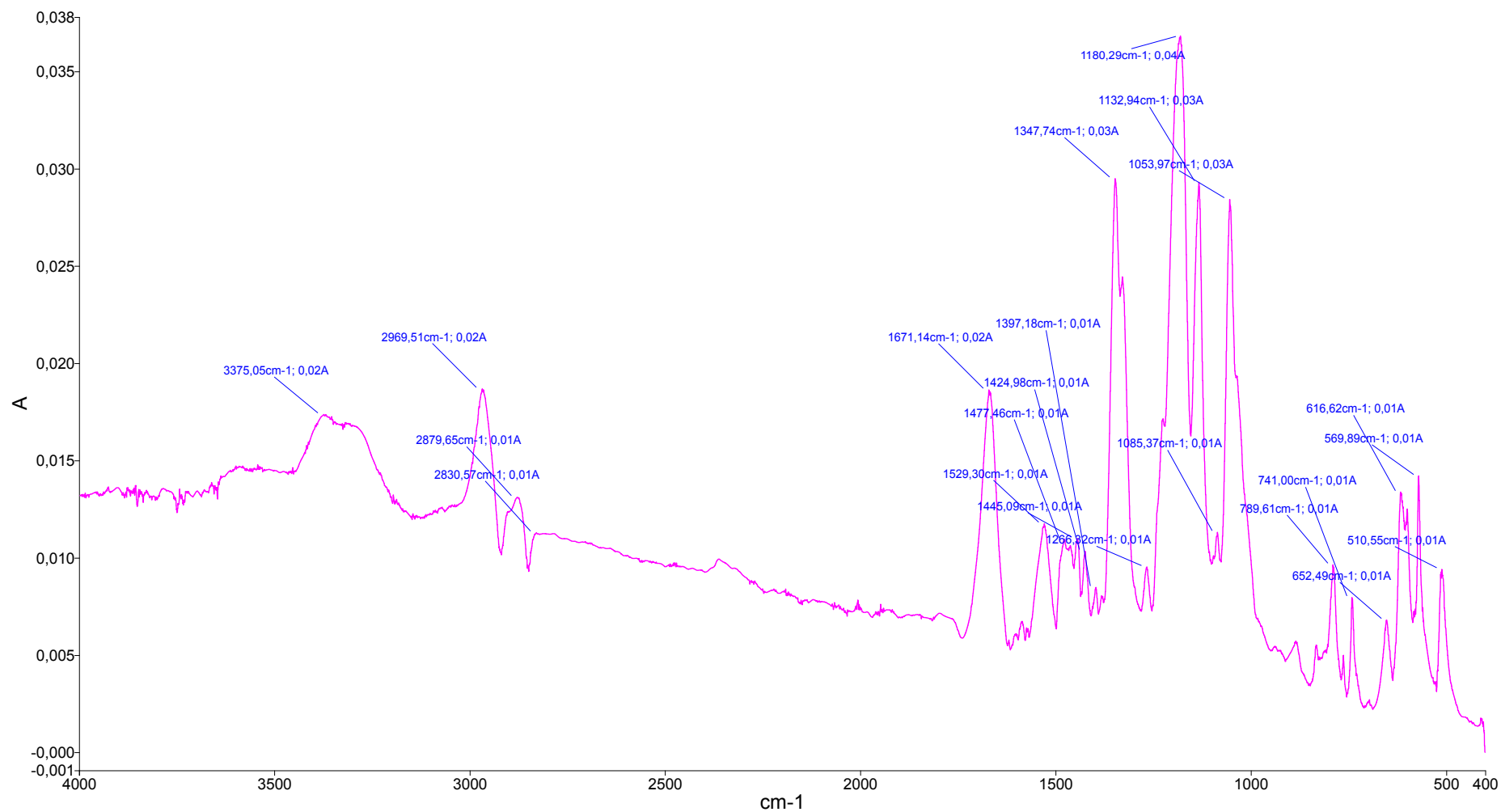


Fig. S194. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2',2'-triethyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 66)

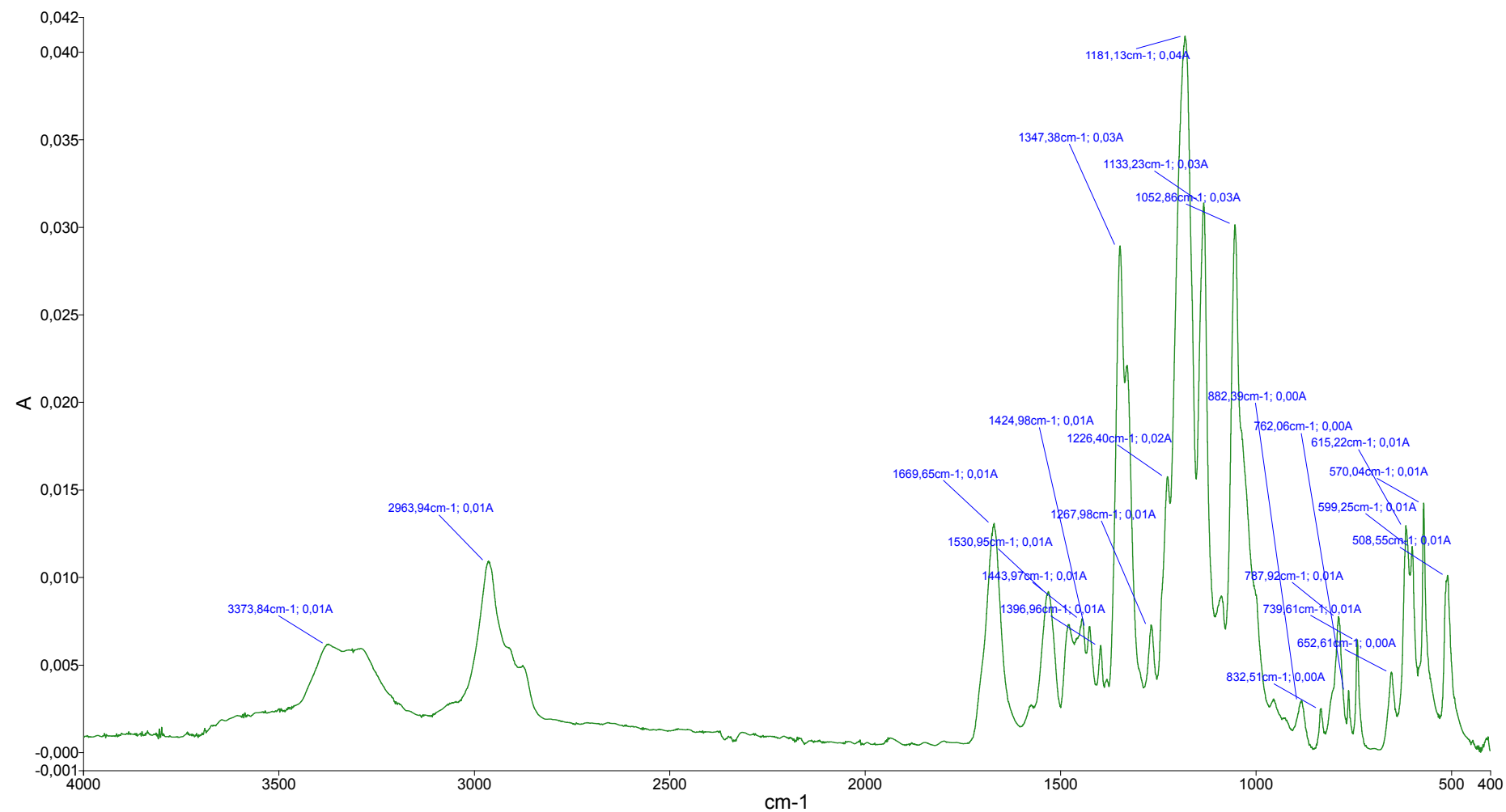


Fig. S195. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-benzyl)ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate **67**)

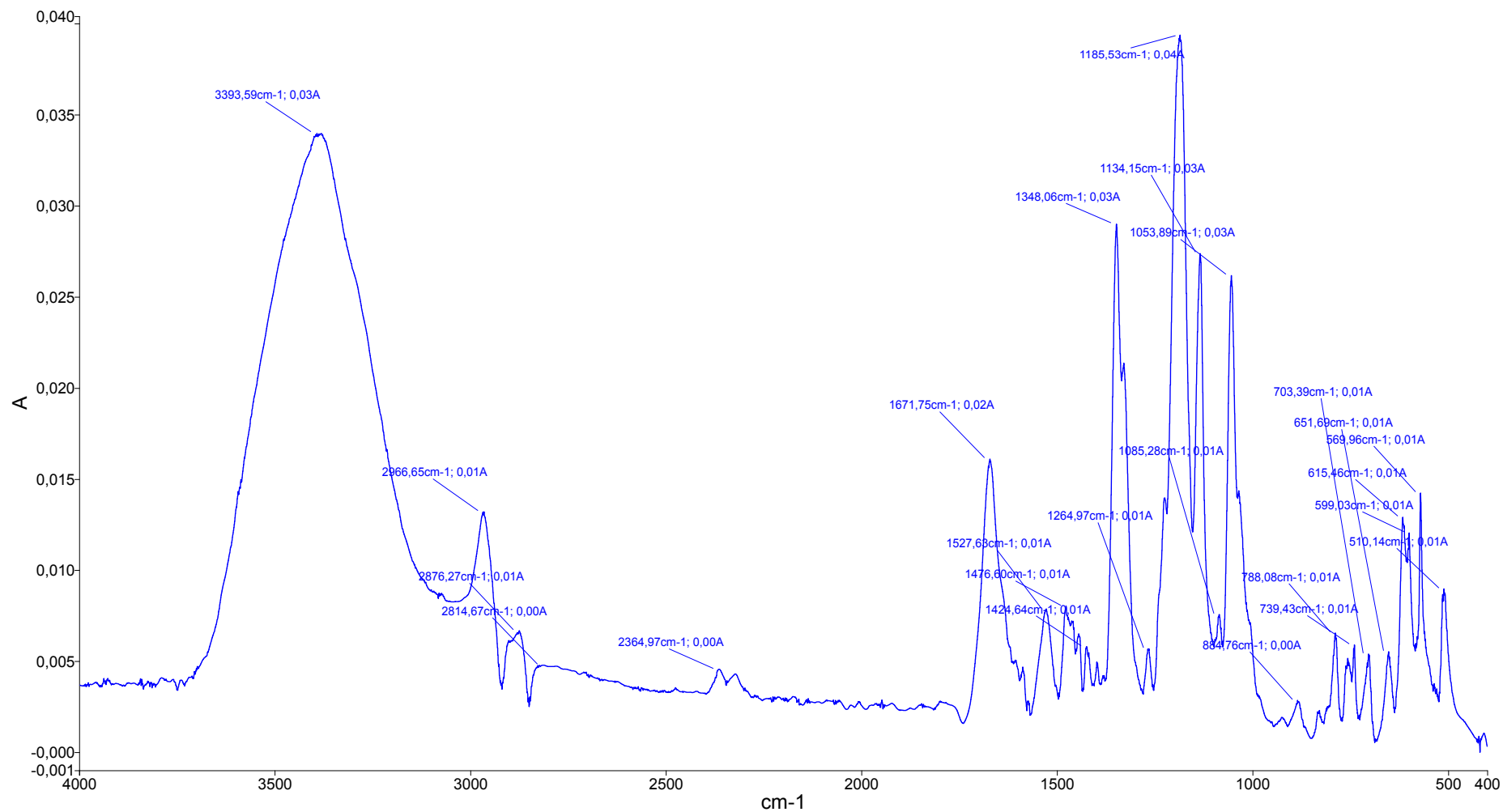


Fig. S196. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(ethoxycarbonylmethyl)ammoniummethyl)-carboxymethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate **68**)

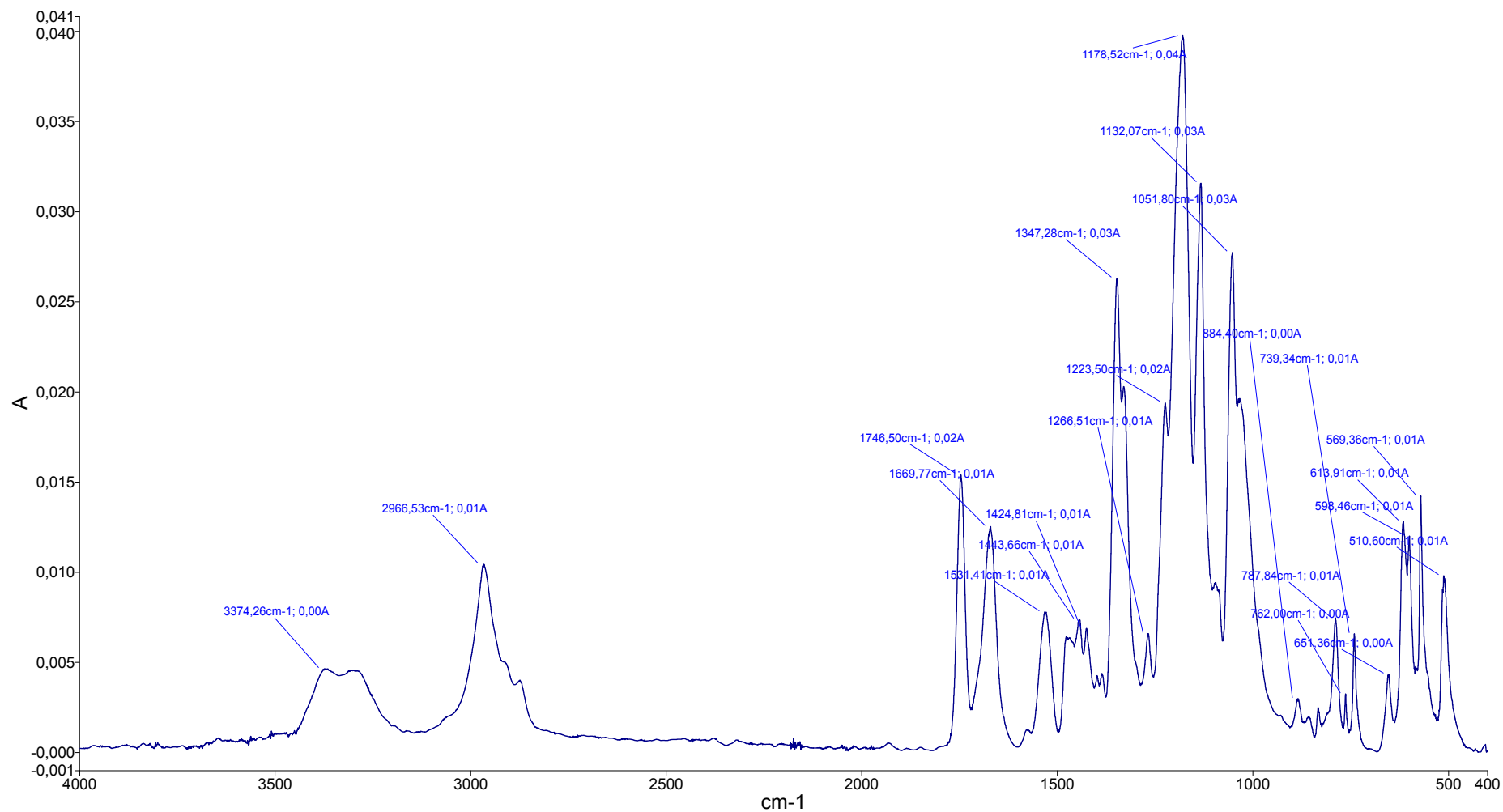


Fig. S197. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-(pentoxycarbonylmethyl)ammoniummethyl)-carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate **69**)

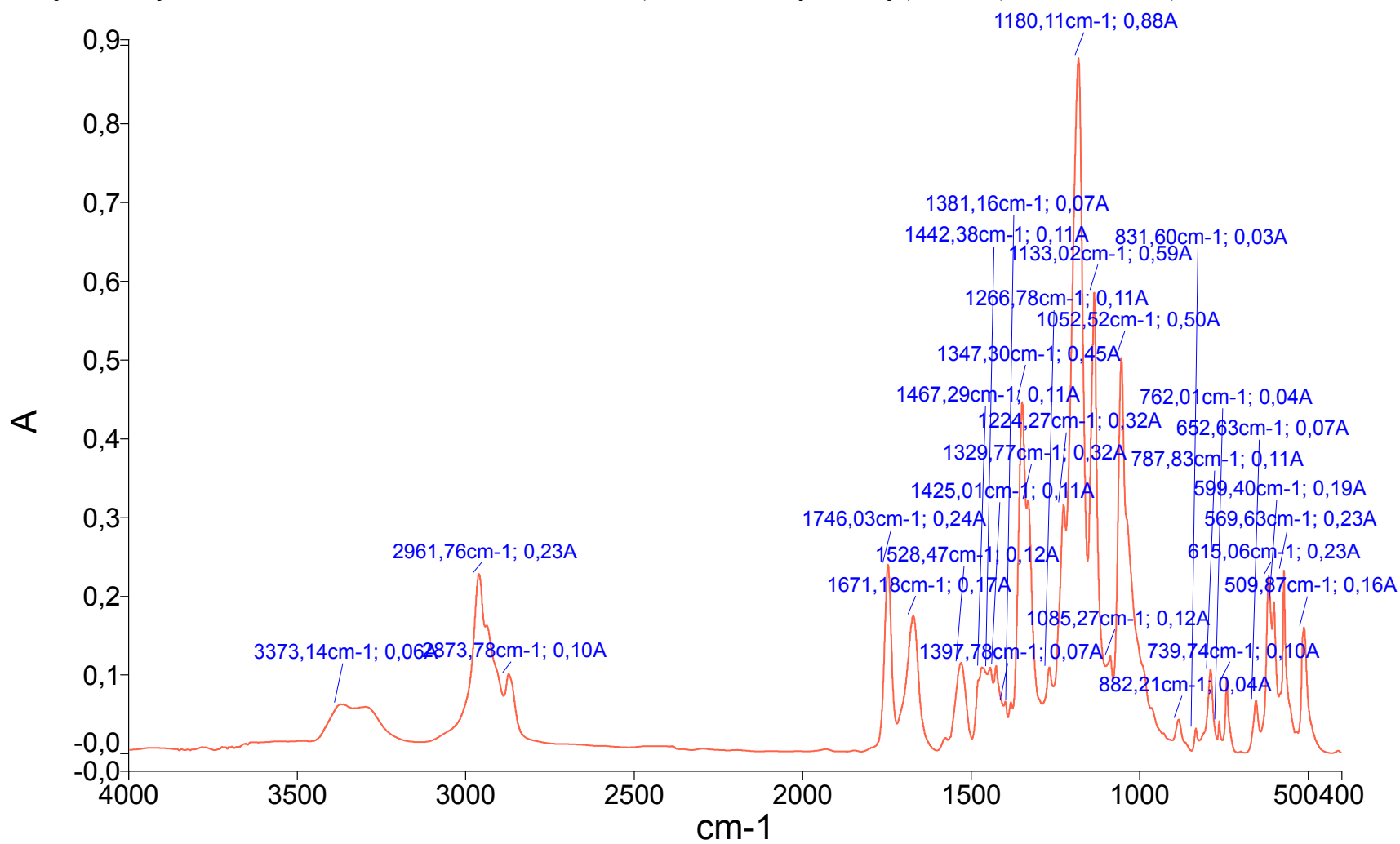


Fig. S198. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[*N*-(2',2'-diethyl-2'-{(ethoxycarbonylmethyl)amidocarbonylmethyl})-ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-*alternate* 70)

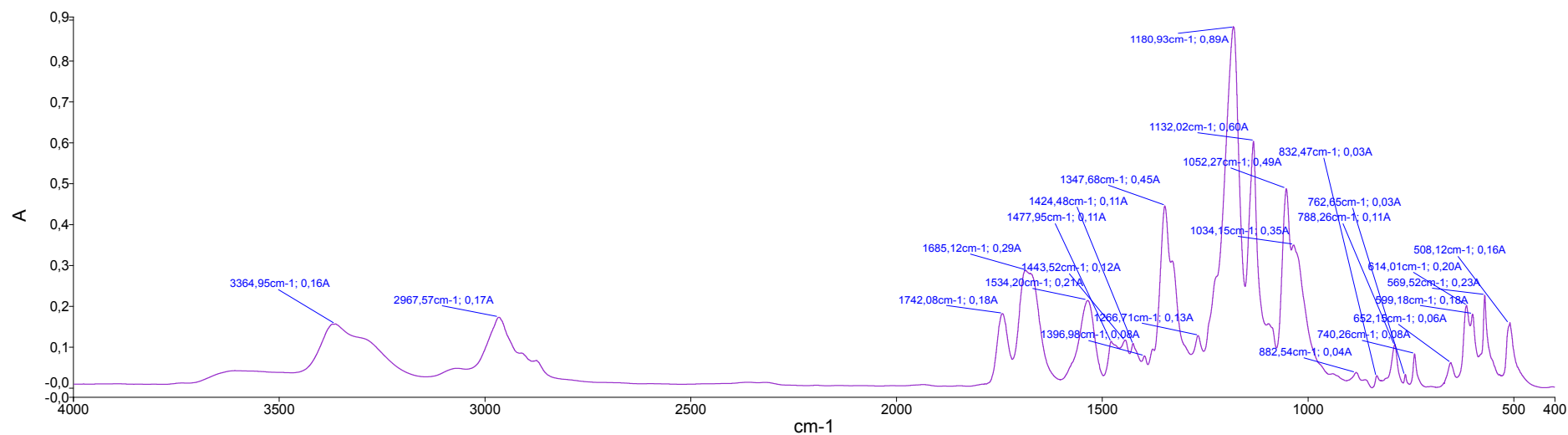


Fig. S199. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{[(ethoxycarbonylmethyl)amidocarbonylmethyl]-amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 71)

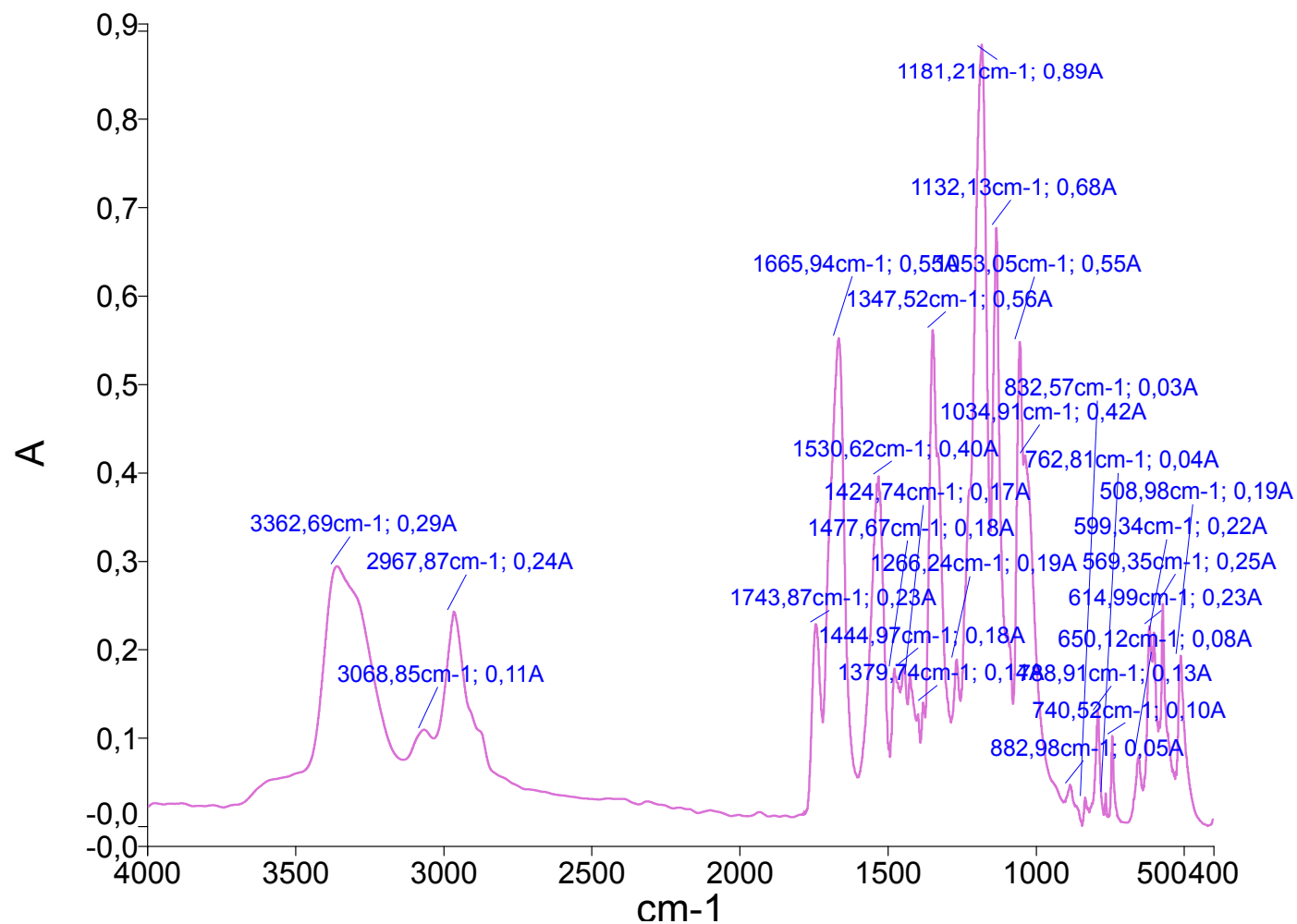
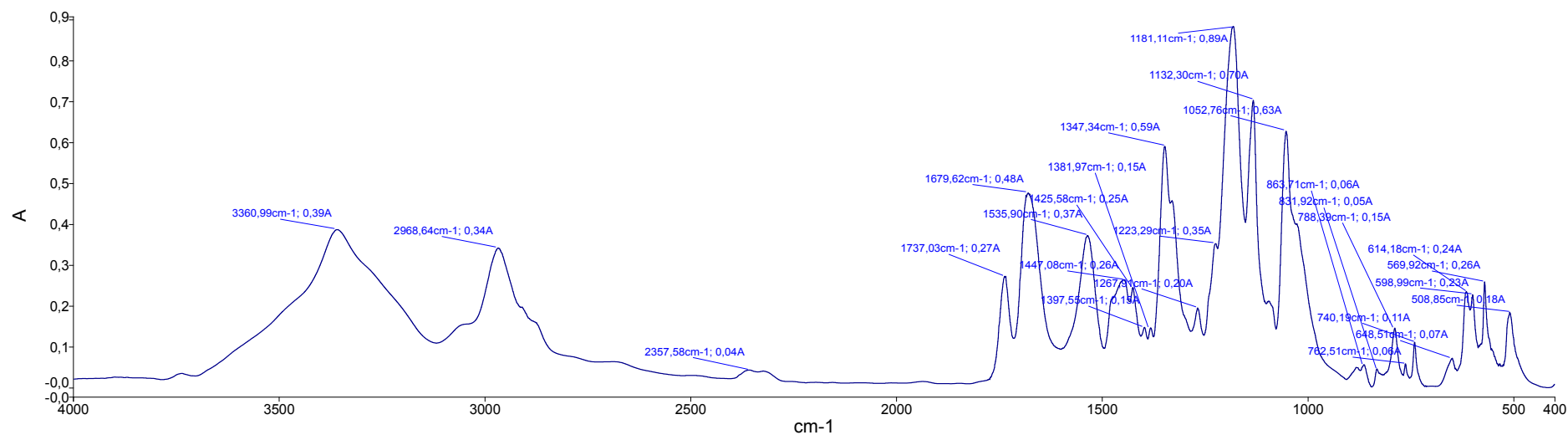


Fig. S200. IR spectrum of 5,11,17,23-tetra-*tert*-butyl-25,26,27,28-tetrakis[(N-(2',2'-diethyl-2'-{(ethoxycarbonyl[S-methyl]methyl)amidocarbonylmethyl}ammoniummethyl)carbomoylmethoxy]-2,8,14,20-tetrathiacalix[4]arene tetra[bis(trifluoromethylsulfonyl)imide] (1,3-alternate 72)



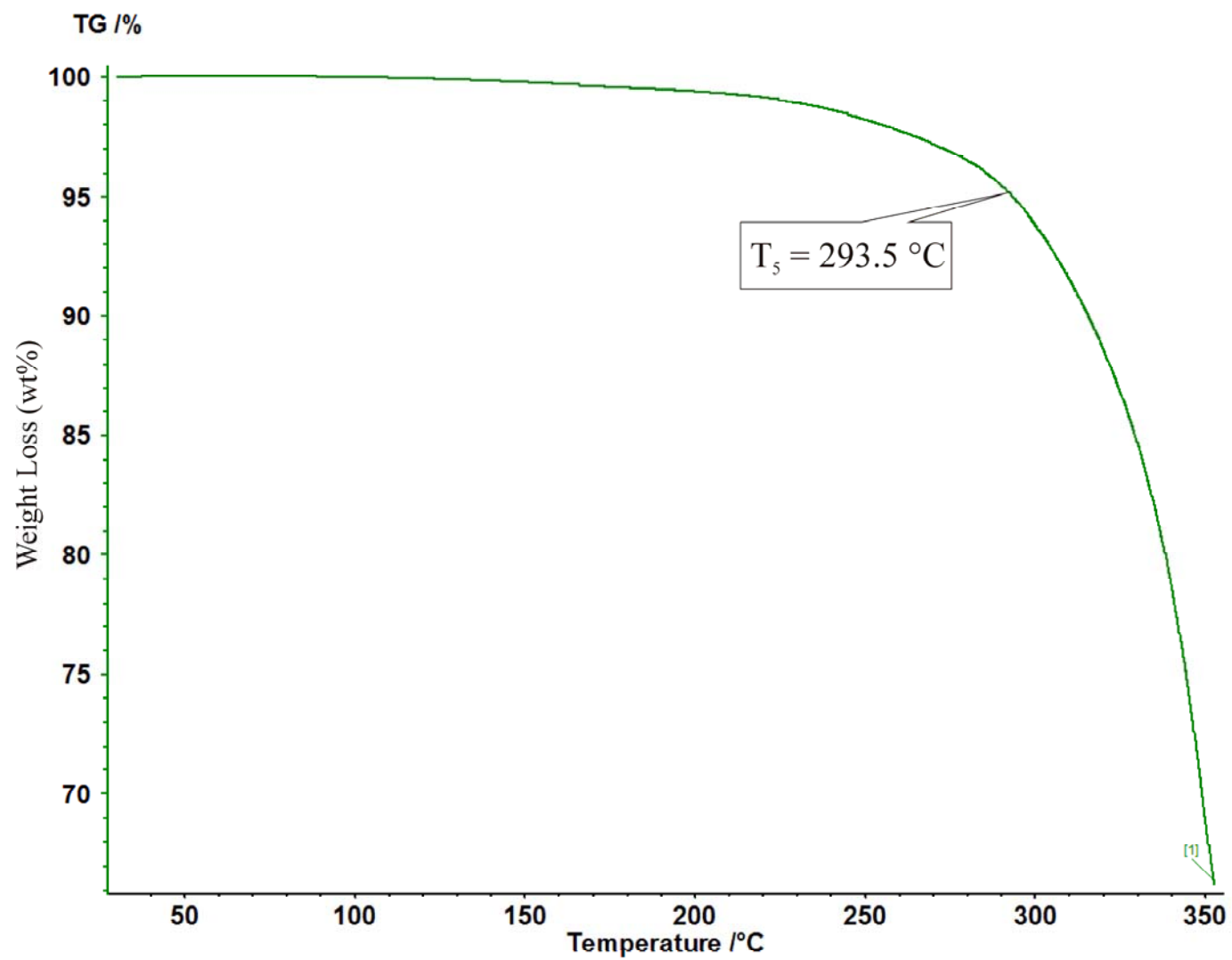


Fig. S201. Thermogravimetric analysis of the compound cone-43.