Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Synthesis of diphenylamine macrocycles and their anti-inflammatory effect

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Table 1. Data of 12-*O*-tetradecanoylphorbol acetate induced mouse ear edema. Screening at 1 μM

Compound	Control (mg)	Edema (mg)	Inhibition (%)
Indomethacin		2.88±0.73	78.76±
5a	14.17±1.00	9.73±0.67*	31.29±4.76*
5b	14.07±1.09	1.33±0.61**	90.52±3.72**
5c	14.07±1.09	1.43±0.03**	89.81±0.24**
5d	14.17±1.00	4.20±0.91**	70.35±6.41**
5e	14.07±1.09	**0.33±0.29****	**97.63±0.07****
5f	14.07±1.09	1.63±0.52**	88.39±3.72**
5g	14.17±1.00	1.90±0.45**	86.59±3.18**
5h	14.07±1.09	5.47±0.98**	66.32±6.06**
5i	15.58±0.33	3.63±1.32**	76.68±8.49**
5j	14.07±1.09	4.37±1.28**	68.95±9.11**
5k	14.07±1.09	4.67±1.49**	66.82±10.59**
5l	14.07±1.09	2.27±0.33*	83.89±2.37**
5m	14.07±1.09	5.50±0.61**	60.92±4.33**
5n	14.07±1.09	**1.10±0.17****	**92.18±1.23****

Table 2. Data of the determination of ID_{50} of compounds **5e** and **5n.**

Compound	Dose (µmol/ear)	Edema (mg)	Inhibition (%)	DI_{50} (µmol/ear)
TPA		14.75±1.13		
	0.031	12.78±1.21	13.36	0.236
	0.1	10.74±1.13*	27.19*	R^2=0.966
Indomethacin	0.31	5.62±0.89**	61.90**	R=0.983
	1	2.88±0.73**	78.76**	

Compound	Dose (µmol/ear)	Edema (mg)	Inhibition (%)	DI_{50} (µmol/ear)
TPA		15.58±0.33		
	0.031	11.76±2.15	24.52±13.8	0.18
	0.1	8.80±2.04*	43.52±13.12*	R^2=0.78
5e	0.31	7.60±1.23**	51.22±7.89**	R=0.88
	1	2.42±0.68**	84.47±2.10**	

Compound	Dose (µmol/ear)	Edema (mg)	Inhibition (%)	DI_{50} (µmol/ear)
TPA		13.98±0.72		
	0.031	12.00±0.42	14.46±2.99	0.29
	0.1	9.00±1.01**	35.62±7.23**	R^2=0.79
5n	0.31	7.92±1.51**	43.35±10.79**	R=0.89
	1	3.38±1.13**	75.82±8.06**	

Table 3. Data of the myeloperoxidase assay

Sample	Dosis (µmol/ear)	MPO (OD_{450nm}/biopsy)	MPO (% of inhibition)
Basal	-	0.013±0.004	-
TPA	-	0.202±0.055	-
TPA + Indomethacin	0.031	0.104±0.017	48.5
	0.1	0.087±0.034	56.93
	0.31	0.039±0.012	80.69
	1	0.018±0.006	91.08

Sample	Dosis (µmol/ear)	MPO (OD_{450nm}/biopsy)	MPO (% of inhibition)
Basal	-	0.128±0.028	-
TPA	-	0.619±0.157	-
TPA + 5e	0.031	0.369±0.102	40.39±16.54
	0.1	0.147±0.026	76.19±4.25
	0.31	0.228±0.008	63.10±1.34
	1	0.157±0.019	74.63±3.05

Sample	Dosis (µmol/ear)	MPO (OD_{450nm}/biopsy)	MPO (% of inhibition)
Basal	-	0.099±0.006	-
TPA	-	0.447±0.131	-
TPA + 5n	0.031	0.342±0.068	23.53±15.16
	0.1	0.161±0.024	63.94±5.29
	0.31	0.151±0.035	66.22±7.87
	1	0.145±0.028	67.61±6.25

Table 4. Data of the Nitrite assay and cell viability

Compound	Concentration (µM)	Nitrite (µM)	Inhibition Production (%)	Viability
LPS (1µg/ml)		6.53±0.2		
Aminoguanidine	100	0.54±0.1	91.8	82.9±5.4
	31	2.0±0.4	69.4	84.7±5.6
	10	3.53±0.2	45.9	88.7±3.7
	3.1	4.47±0.4	31.6	88.1±3.9
5b	31	4.16±0.2	36.3	71.0±6.2
	18	5.54±0.7	15.1	71.8±7.9
	10	6.0±0.2	8.1	70.2±4.1
	3.1	6.4±0.2	2. 7	68.1±4.5
5e	31	5.18±0.2	20.7	73.3±5.3
	18	5.23±0.3	19.9	71.3±5.7
	10	5.52±0.2	15.4	76.3±4.5
	3.1	5.49±0.3	16.0	77.4±3.7
5n	31	4.74±0.5	27.5	83.0±4.0
	18	4.13±0.15	36.8	84.1±6.1
	10	5.41±0.4	17.2	82.1±3.8
	3.1	5.38±0.4	17.7	86.9±6.1

Table 5. Percentage of cytotoxicity screening of macrocycles **5a-n**

Comp.	U251 (50µM)	PC-3 (50µM)	K562 (50µM)	HCT-15 (50µM)	MCF-7 (50µM)	SKLU-1 (50µM)	COS-7 (25µM)	FGH (25µM)
5a	3.8	18.3	28.7	1.6	13.2	29.5	12.3	NC
5b	NC	96.5	NC	100	65.9	44.2	52.6	11.1
5c	70.5	100	100	50.6	44.8	20.6	NC	37.9
5d	39.9	86.7	100	56.3	91.7	68.9	52.3	17.1
5e	32.0	73.8	100	66.2	58.6	5.6	51	30.4
5f	97.9	100	100	59.8	77.6	78.3	62.6	23.5
5g	39.0	70.7	20.8	35.0	21.8	10.7	46.3	NC
5h	27.2	61.1	100	28.5	85.0	50.1	50.4	NC
5i	16.6	33.8	92.8	22.4	77.6	46.9	NC	NC
5j	54.4	81.2	100	56.9	44.3	7.5	51.8	24.3
5k	NC	34.4	100	27.5	14.7	NC	50.4	19.8
5l	NC	12.78	23.0	12.29	13.4	16.43	NC	NC
5m	NC	NC	9.66	2.31	13.48	21.74	NC	38.5
5n	NC	NC	30.0	6.23	10.39	34.46	NC	24.6

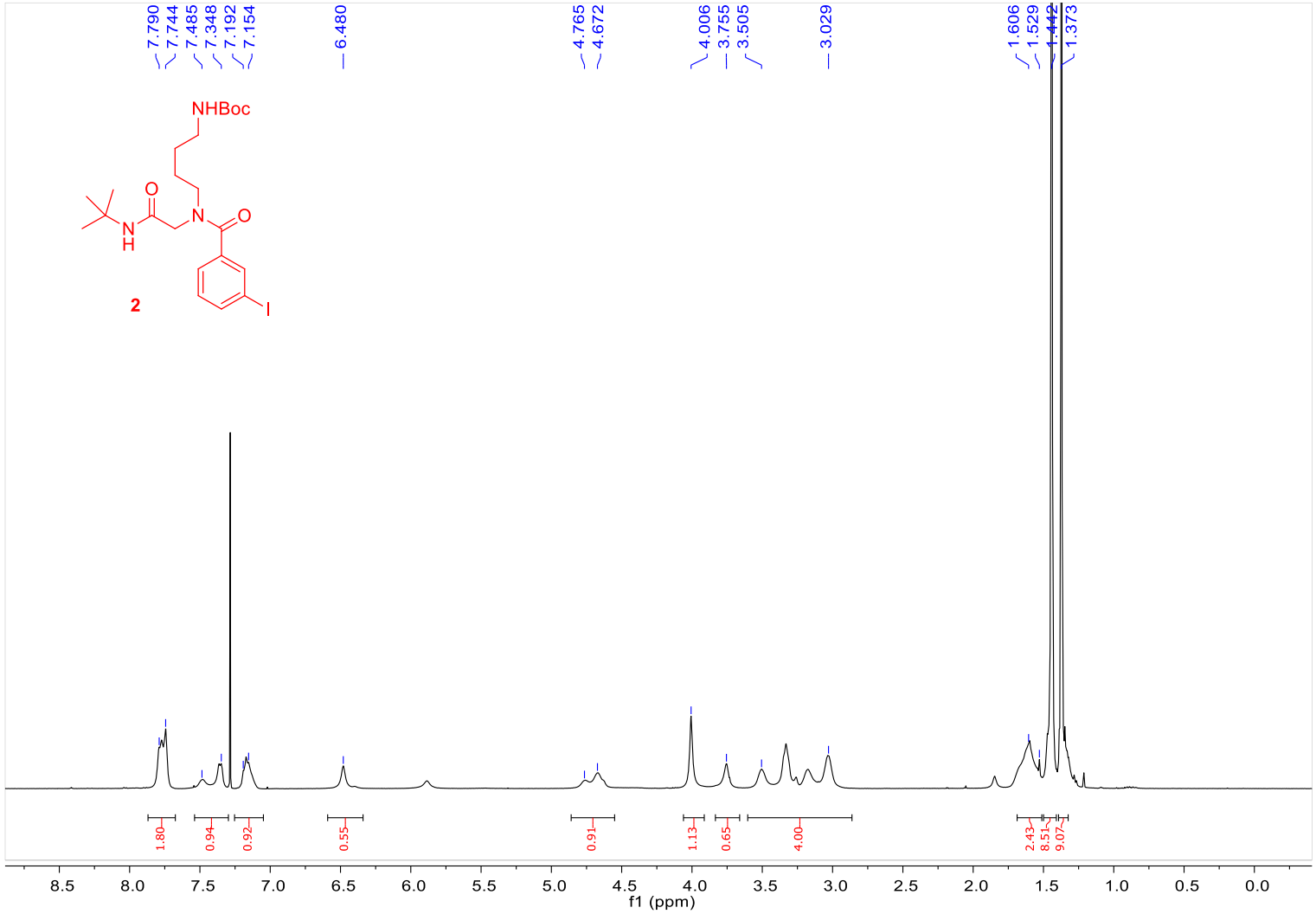


Figure 1. 400 MHz ^{1}H NMR spectra in $CDCl_3$ of *tert*-butyl (4-(N-(2-(tert-butylamino)-2-oxoethyl)-3-iodobenzamido)butyl)carbamate (**2**)

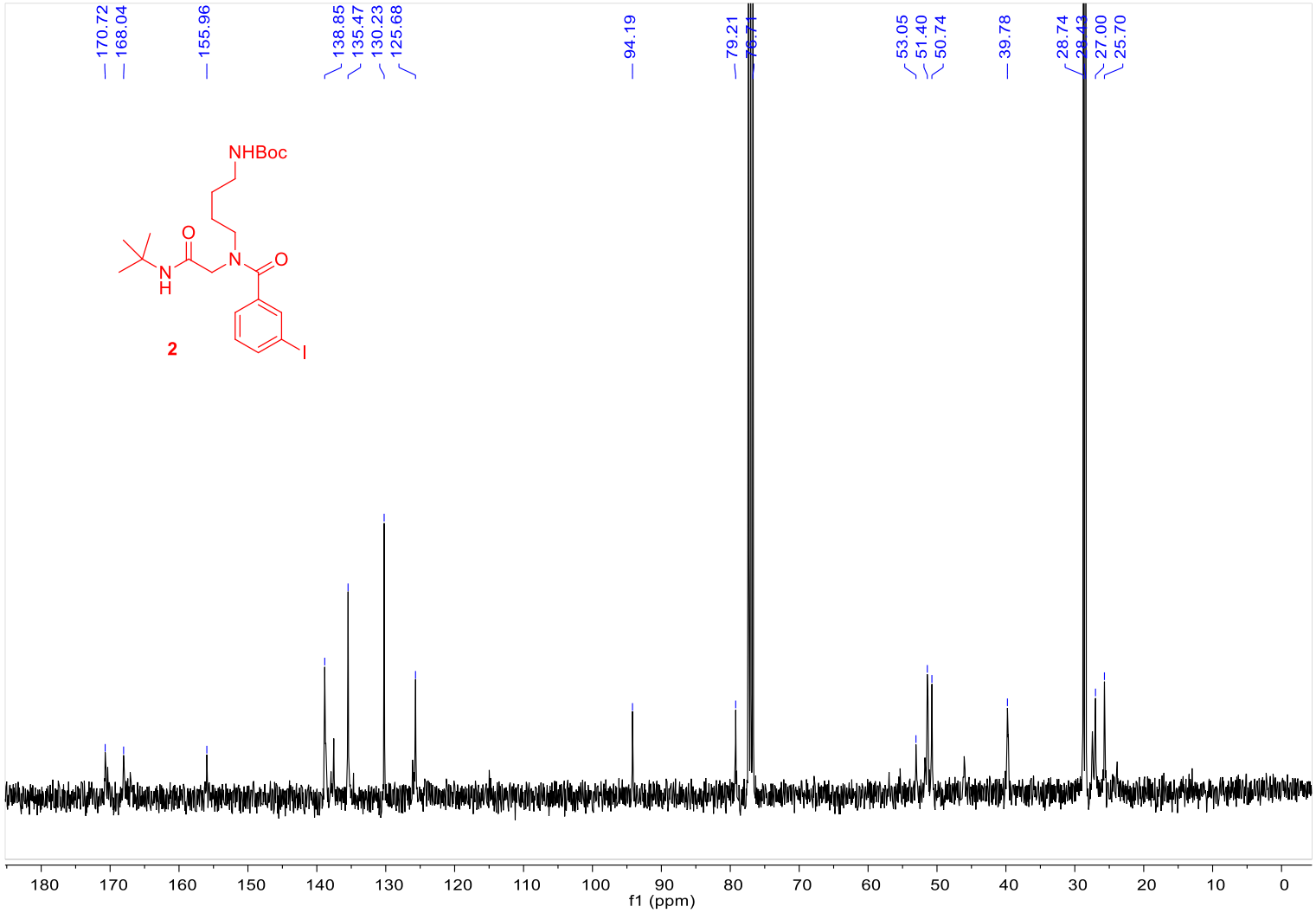


Figure 2. 100 MHz ^{13}C NMR spectra in $CDCl_3$ of *tert*-butyl (4-(N-(2-(tert-butylamino)-2-oxoethyl)-3-iodobenzamido)butyl)carbamate (**2**)

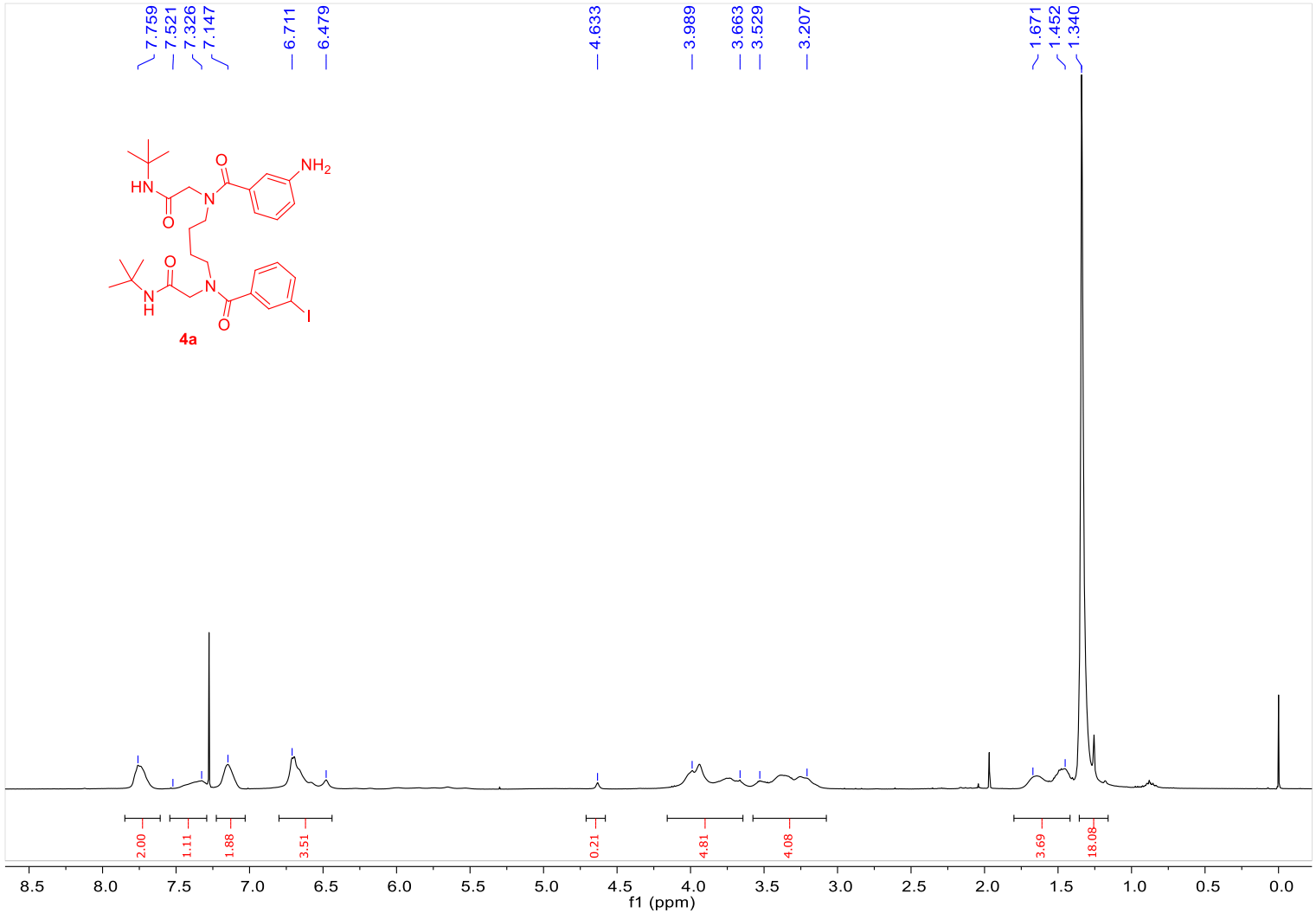


Figure 3. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(tert-butylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4a**).

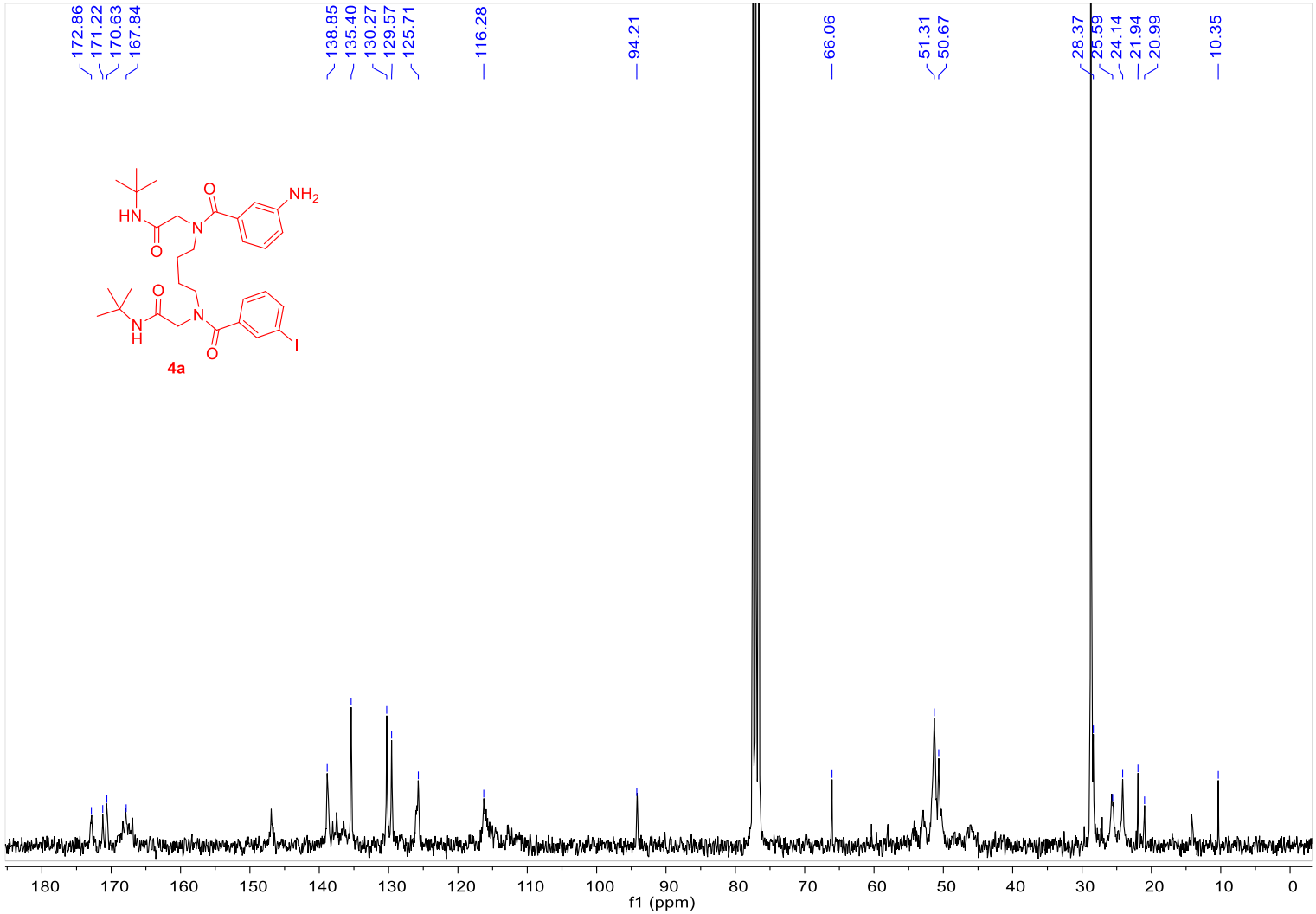


Figure 4. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(tert-butylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4a**).

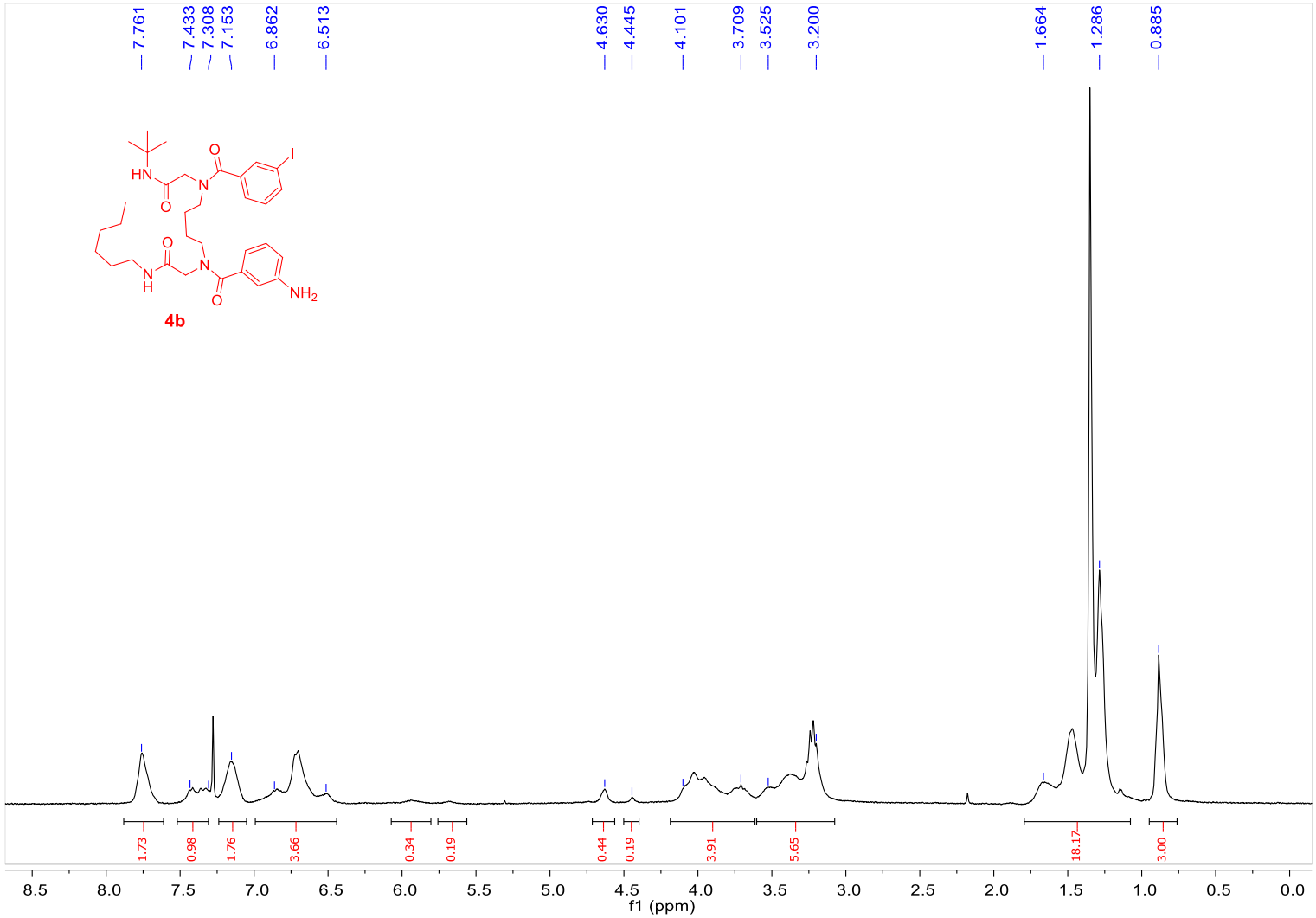


Figure 5. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(hexylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4b**)

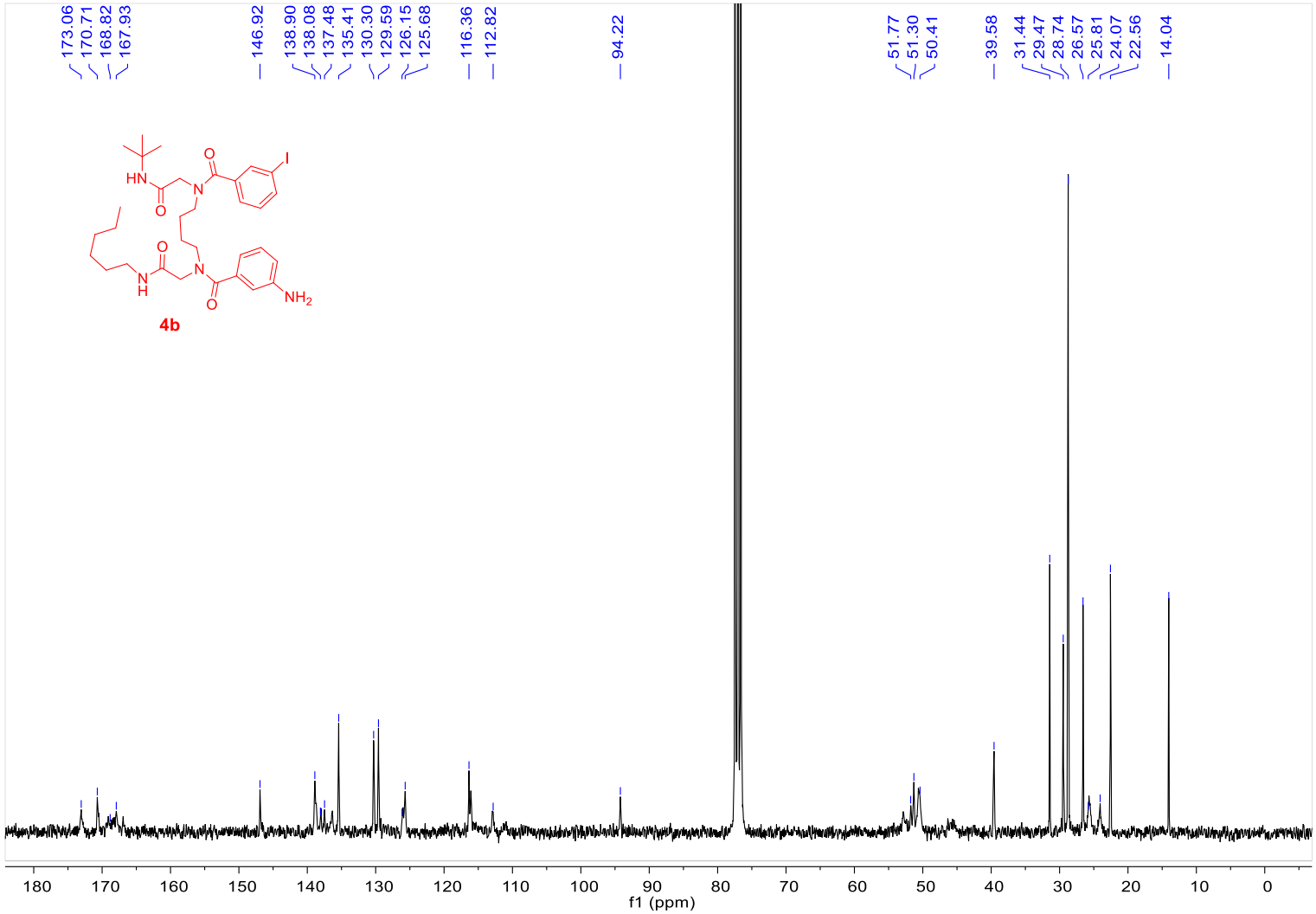


Figure 6. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(hexylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4b**)

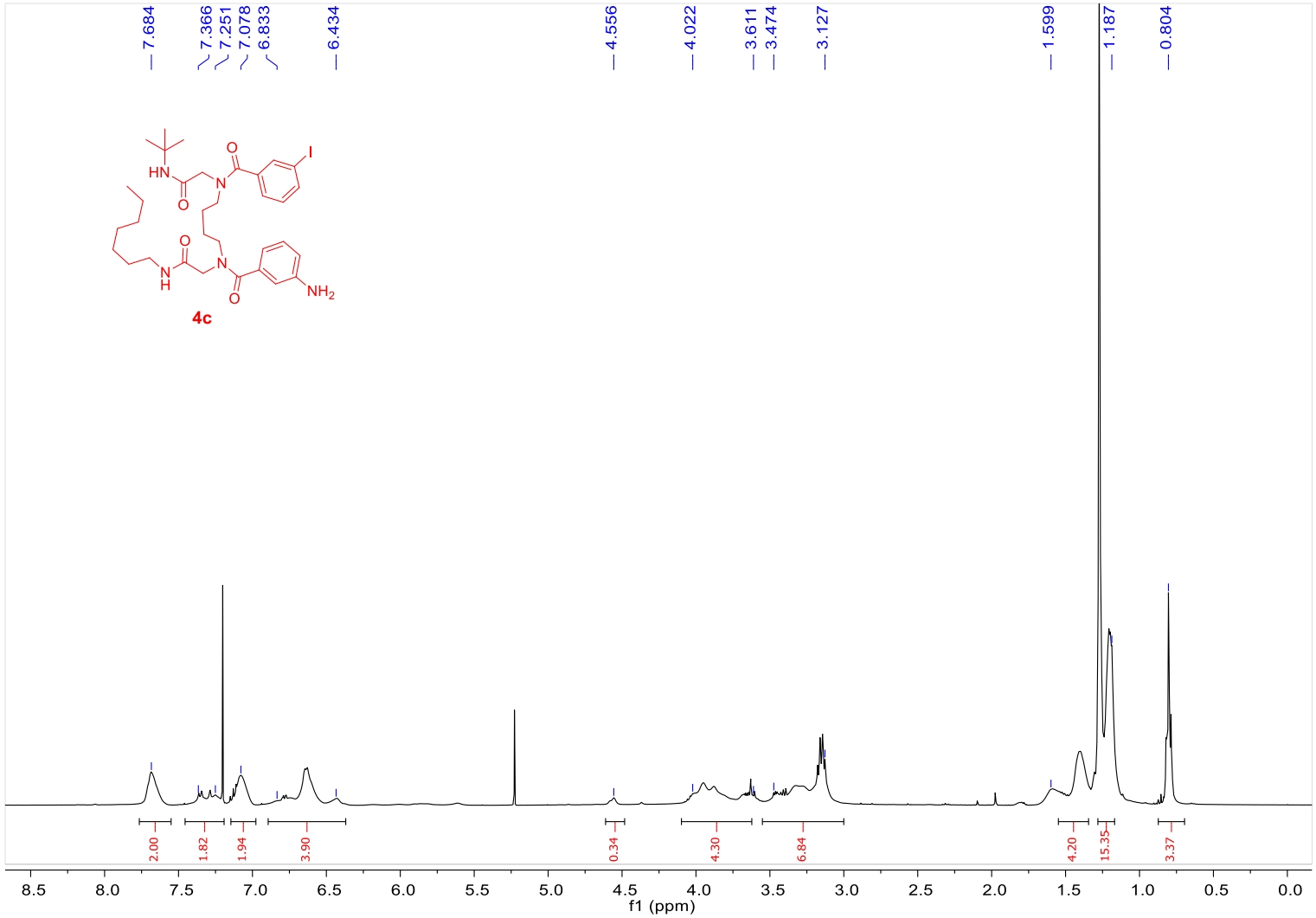


Figure 7. 400 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(heptylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4c**).

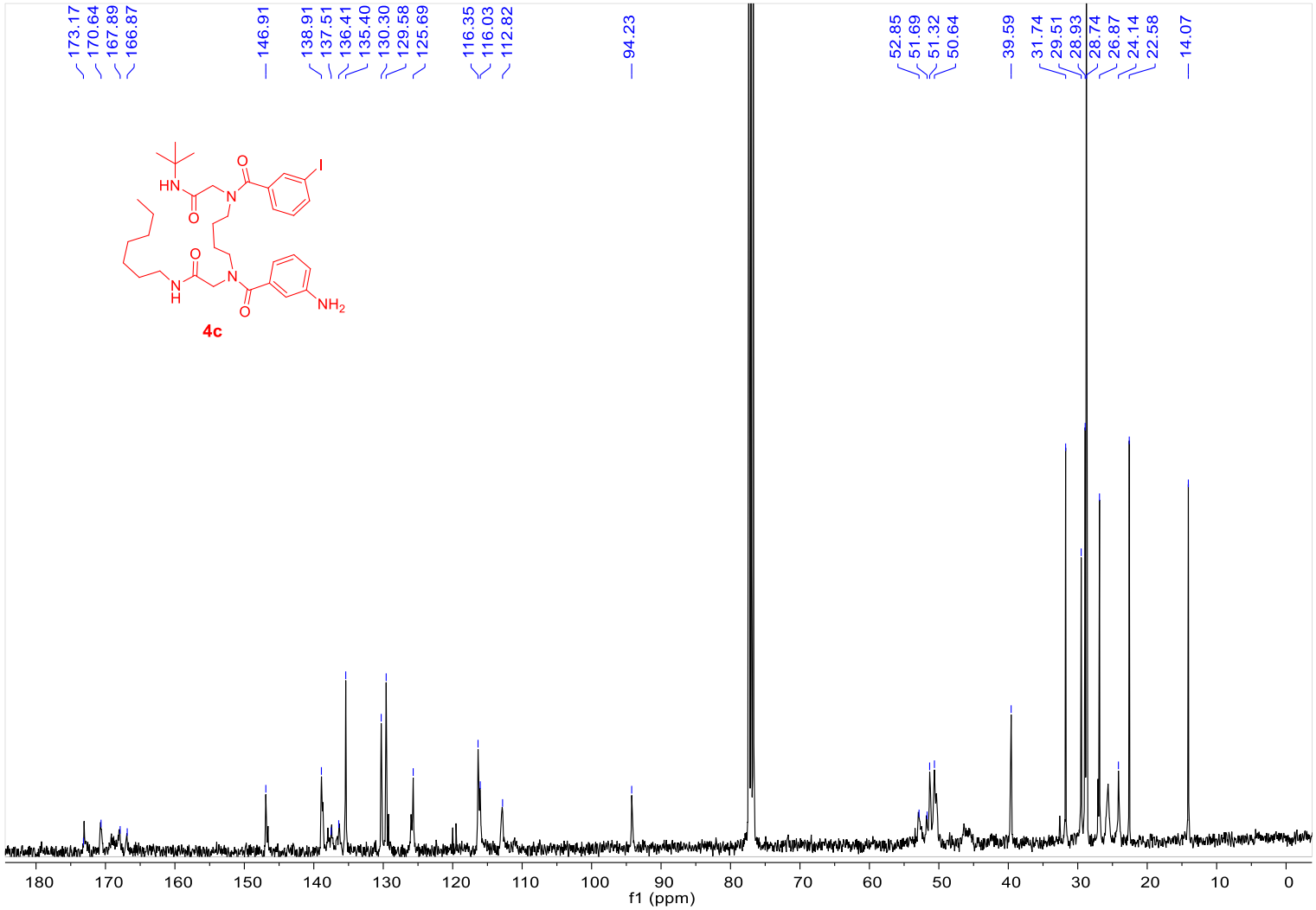


Figure 8. 100 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(heptylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4c**).

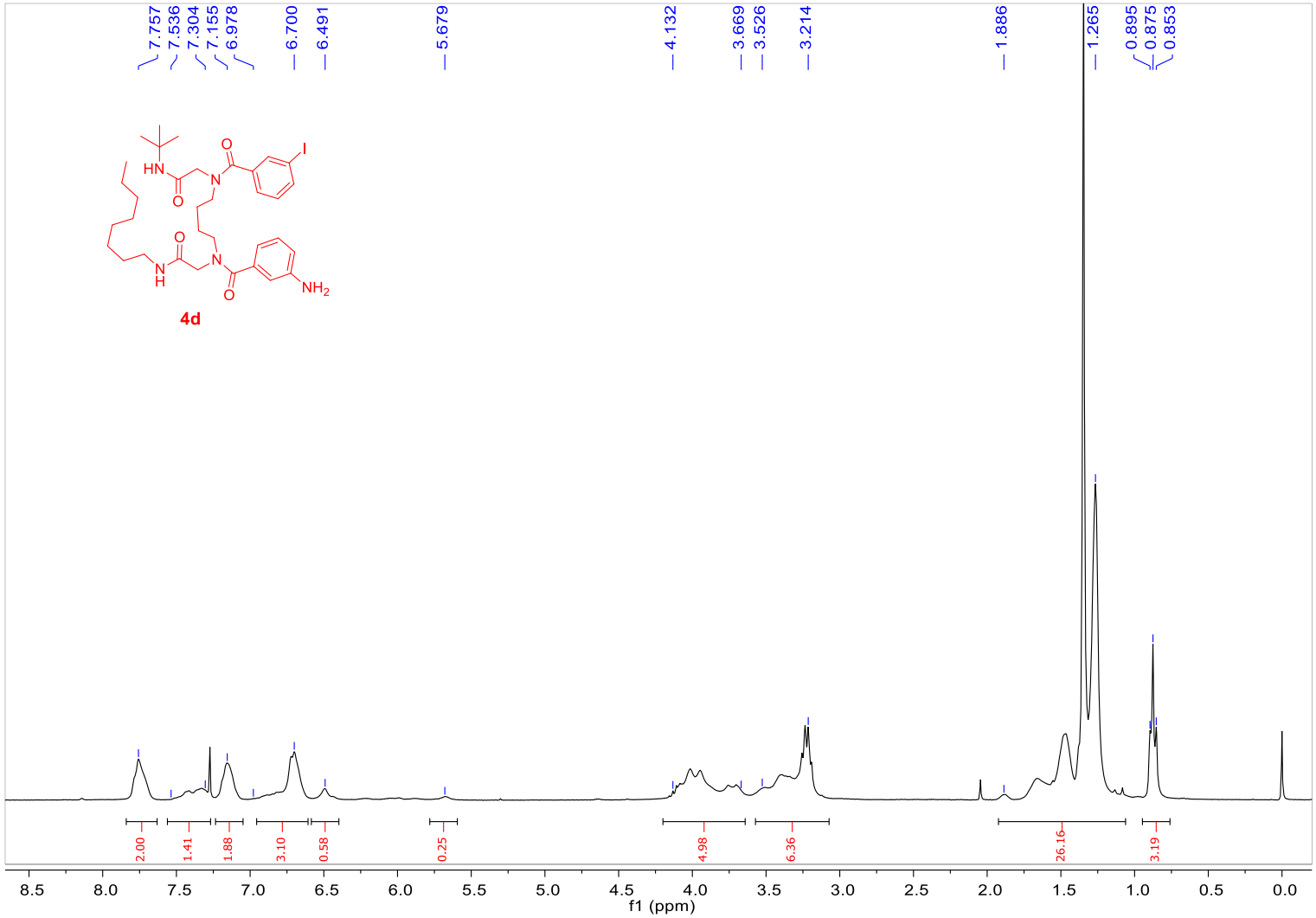


Figure 9. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(3-iodo-N-(2-(octylamino)-2-oxoethyl)benzamido)butyl)benzamide(**4d**).

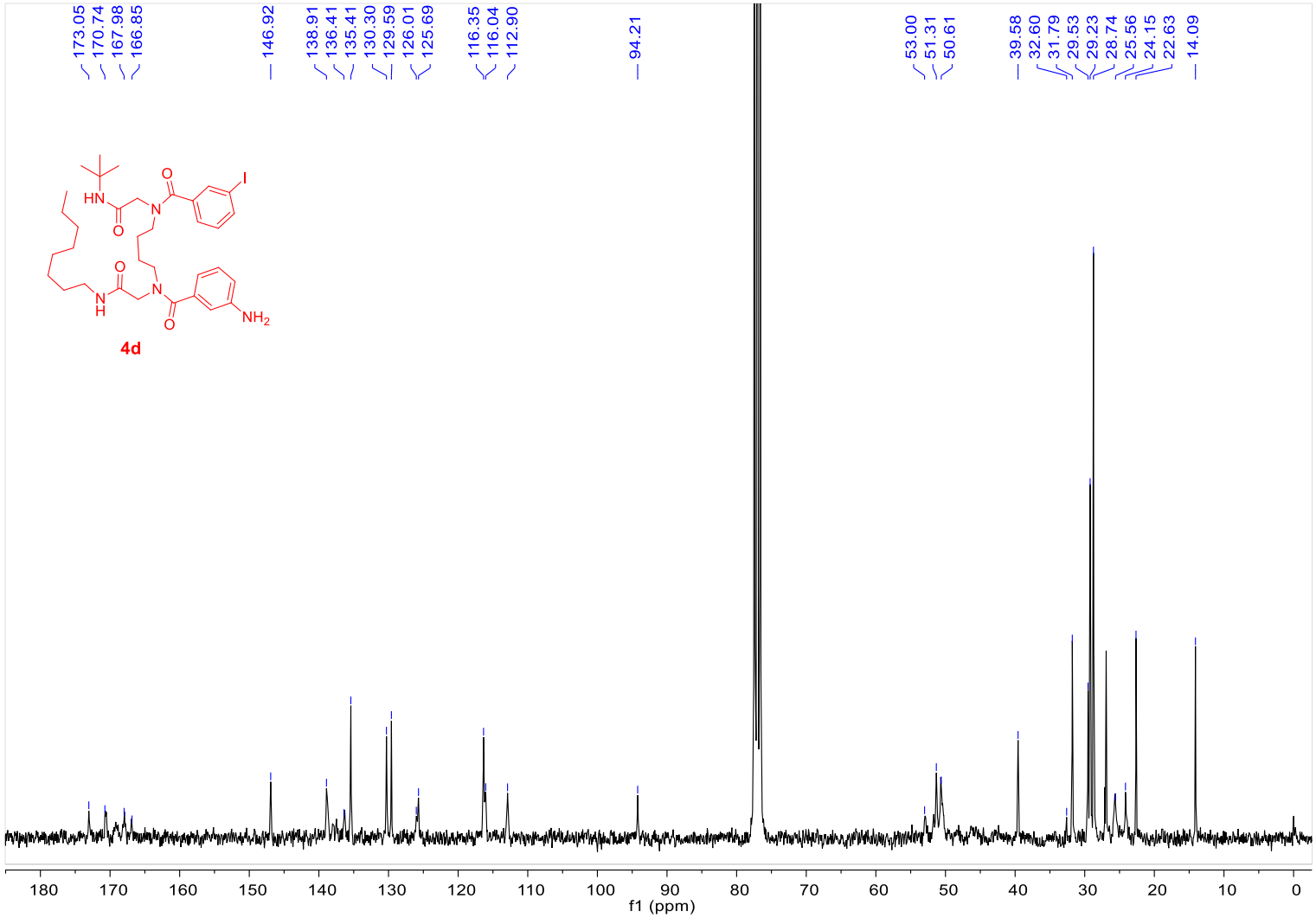


Figure 10. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(3-iodo-N-(2-(octylamino)-2-oxoethyl)benzamido)butyl)benzamide(**4d**).

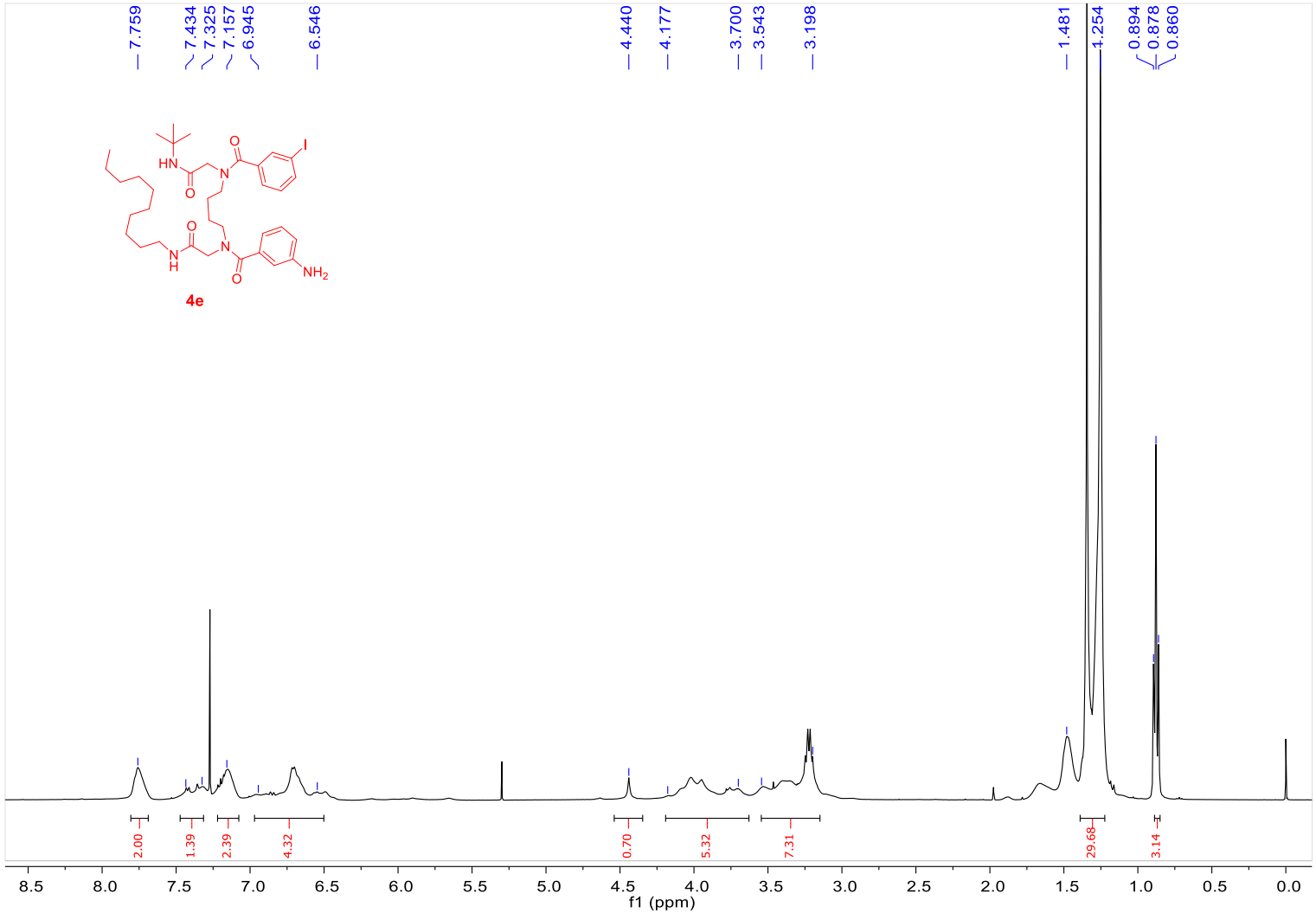


Figure 11. 400 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(decylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide(**4e**).

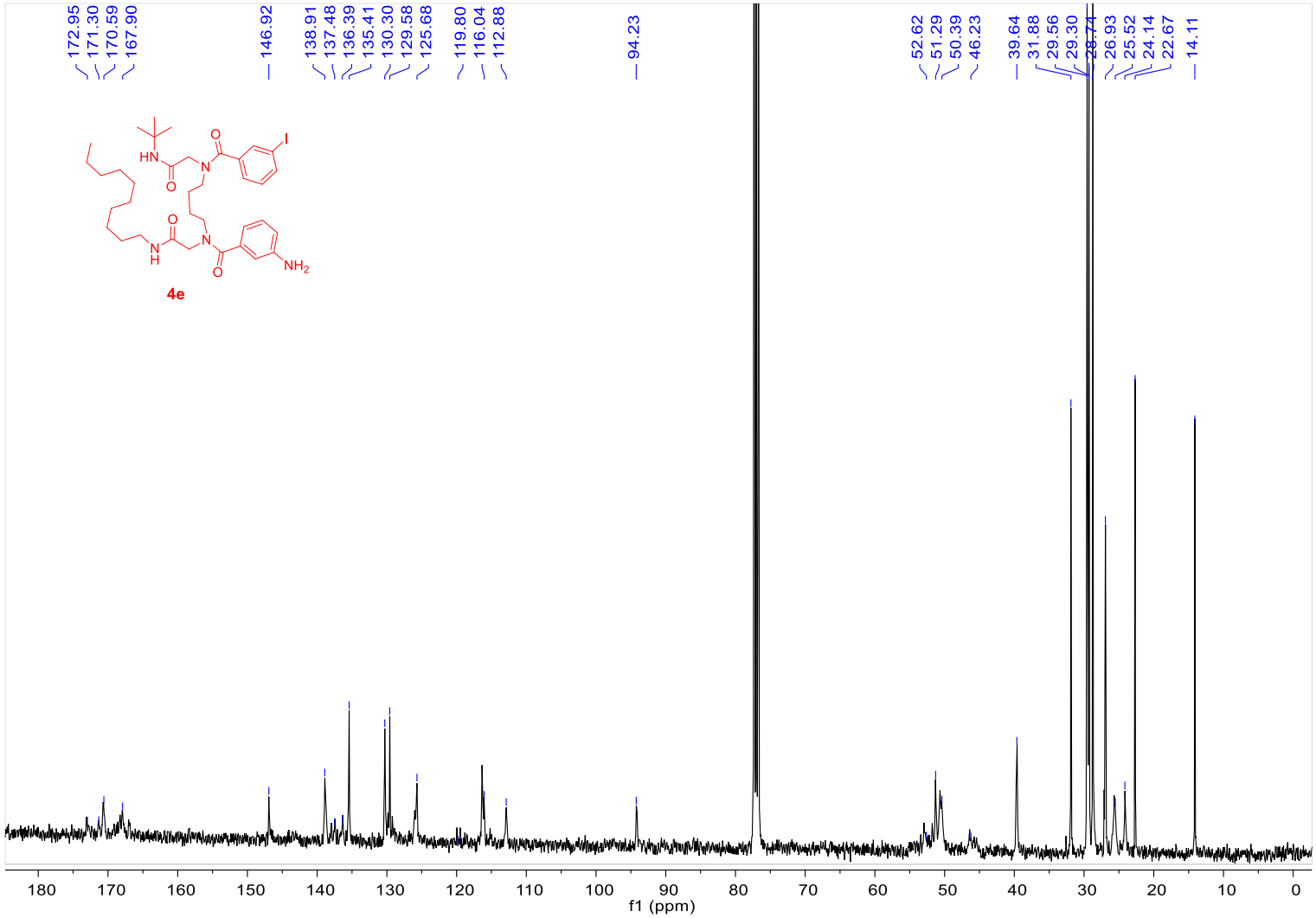


Figure 12. 100 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(decylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide(**4e**).

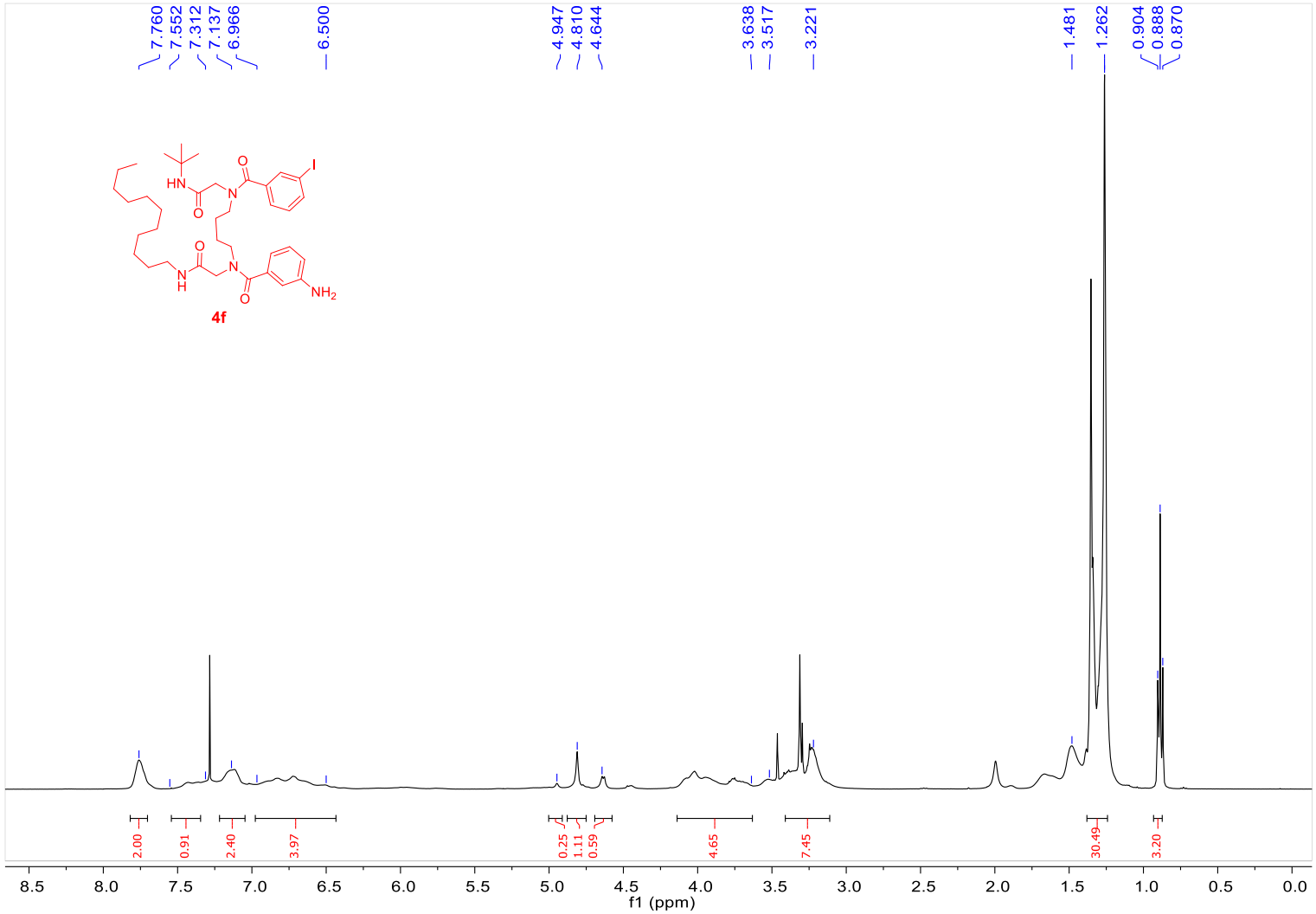


Figure 13. 400 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(3-iodo-N-(2-oxo-2-(undecylamino)ethyl)benzamido)butyl)benzamide (**4f**).

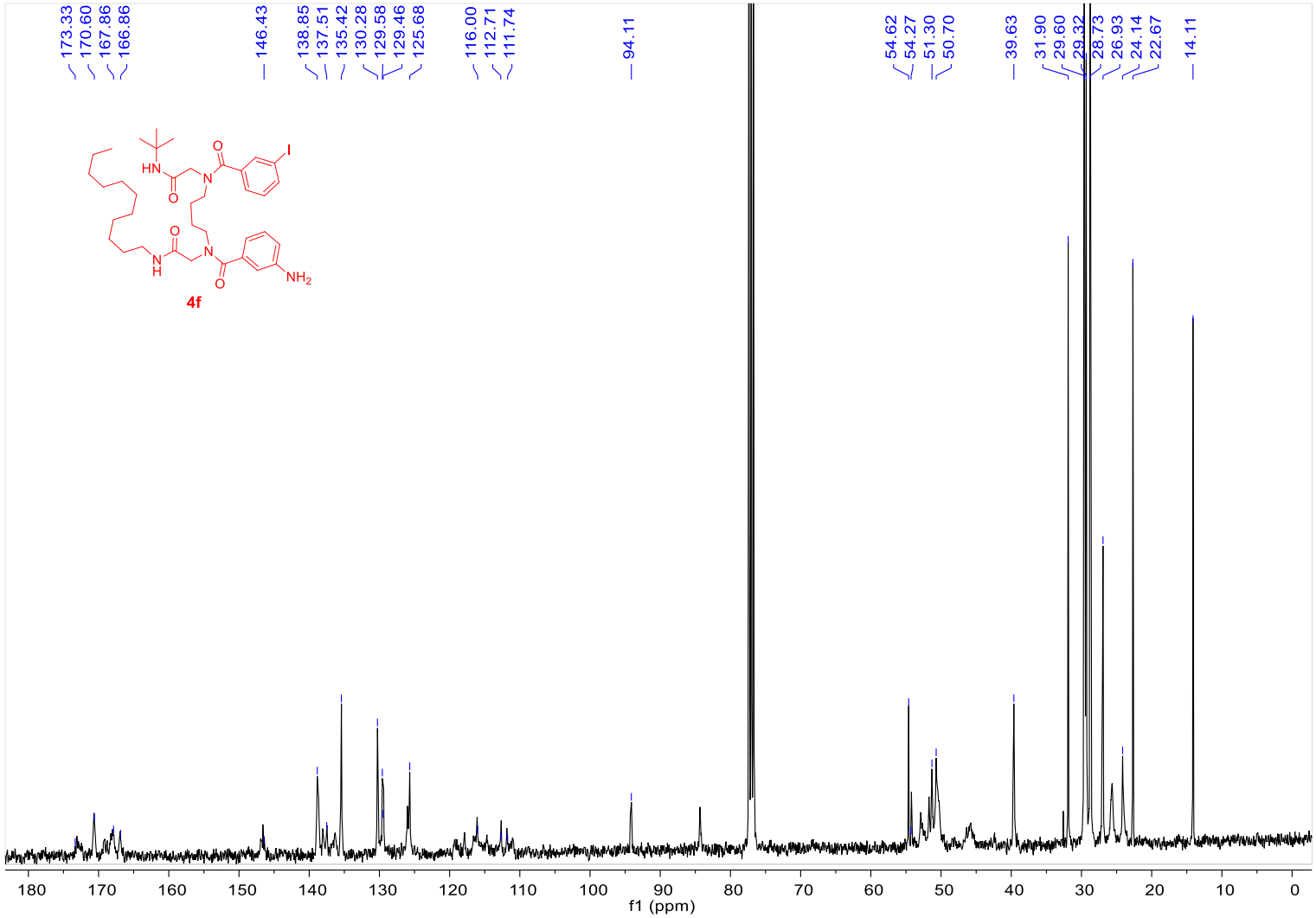


Figure 14. 100 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(3-iodo-N-(2-oxo-2-(undecylamino)ethyl)benzamido)butyl)benzamide (**4f**).

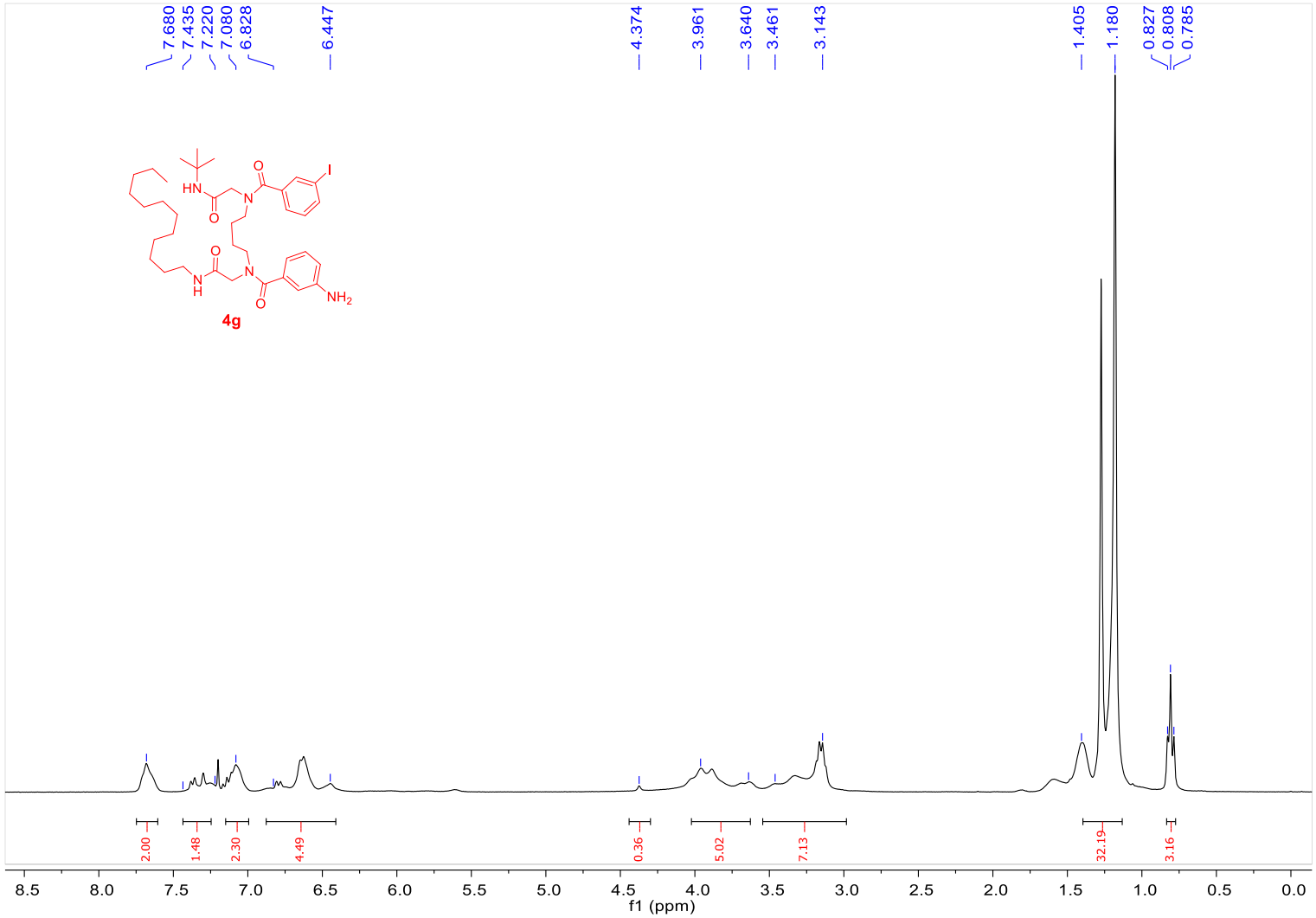


Figure 15. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(dodecylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4g**).

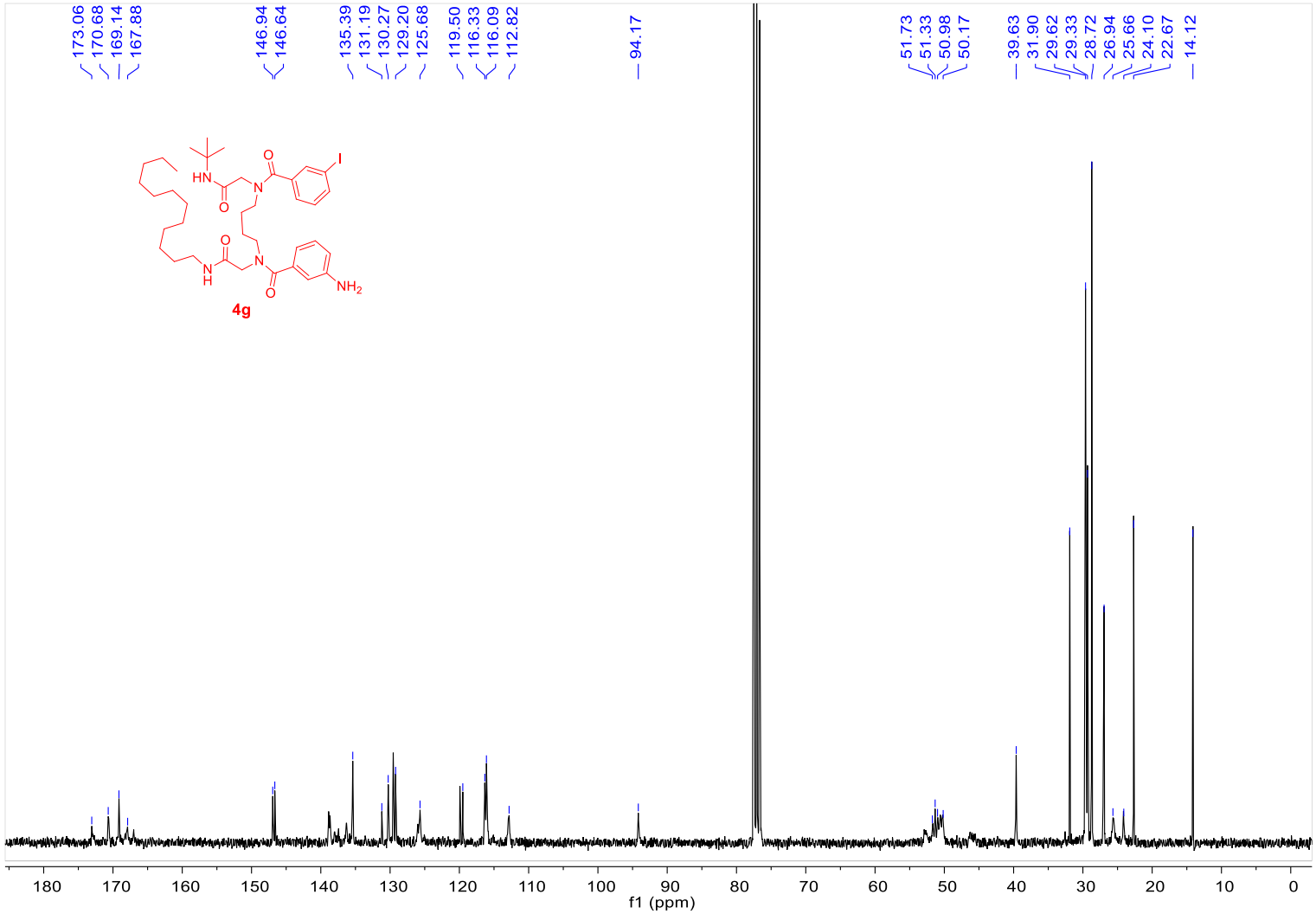


Figure 16. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(dodecylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4g**).

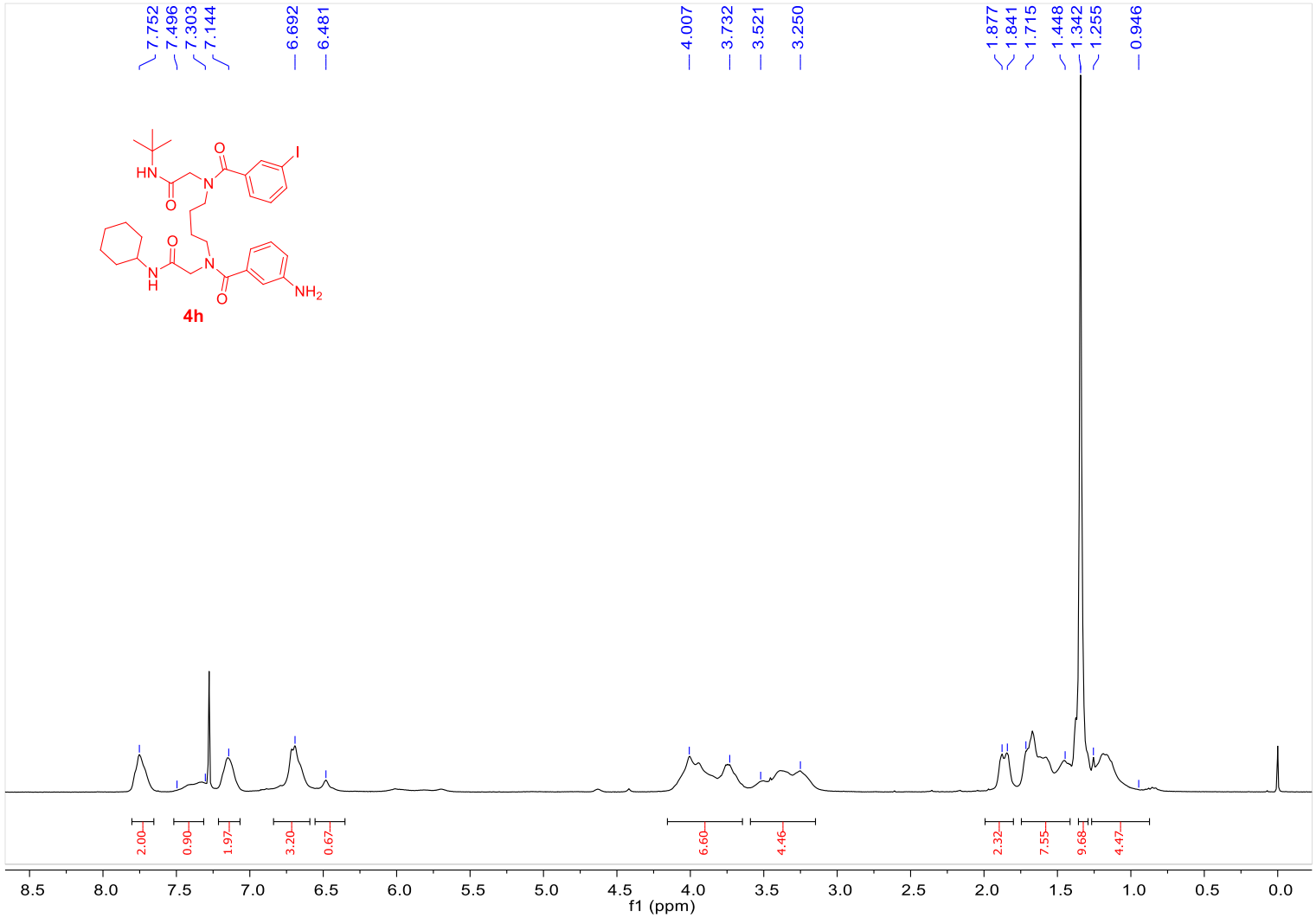


Figure 17. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(cyclohexylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4h**).

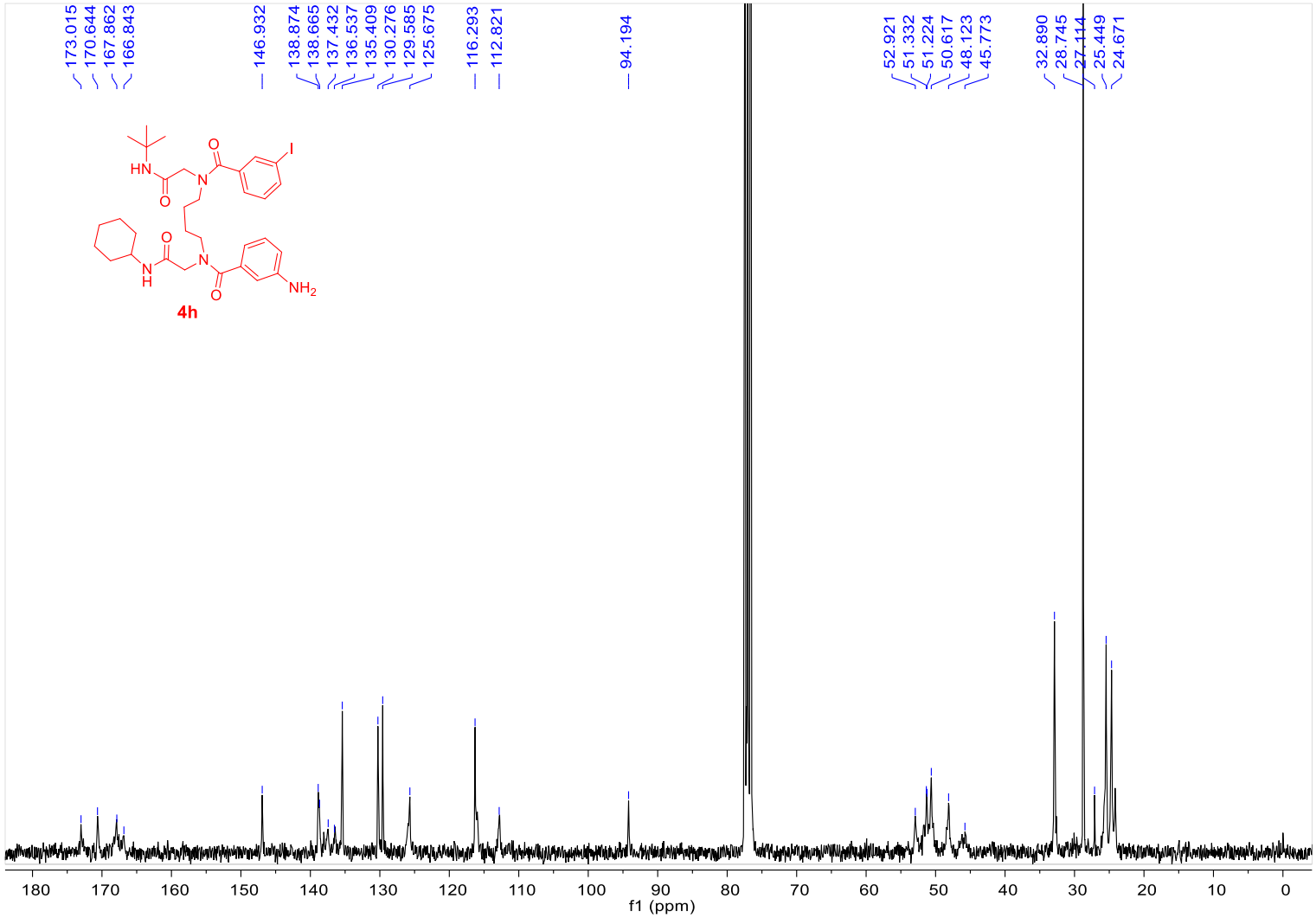


Figure 18. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(cyclohexylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4h**).

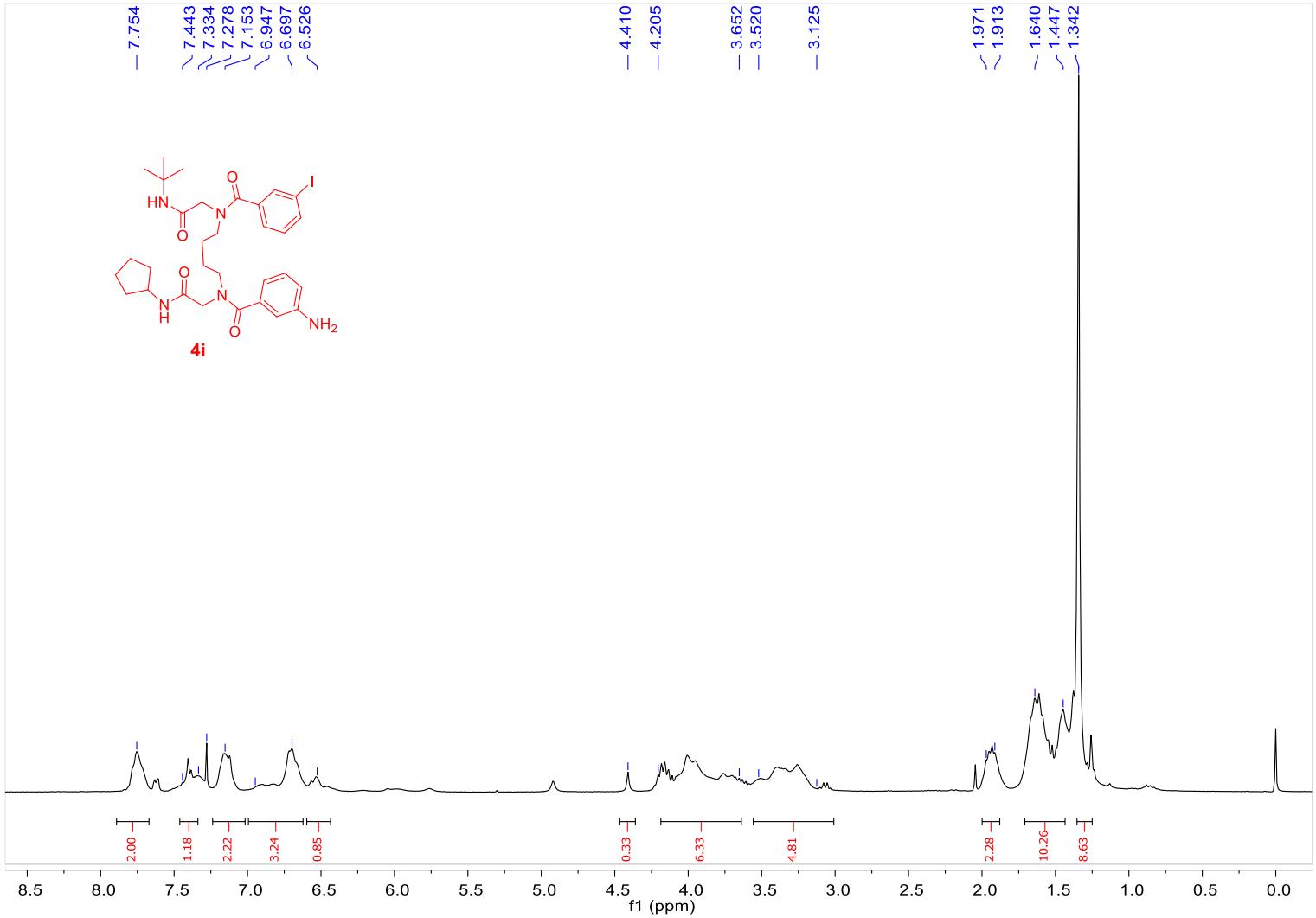


Figure 19. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(cyclopentylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4i**).

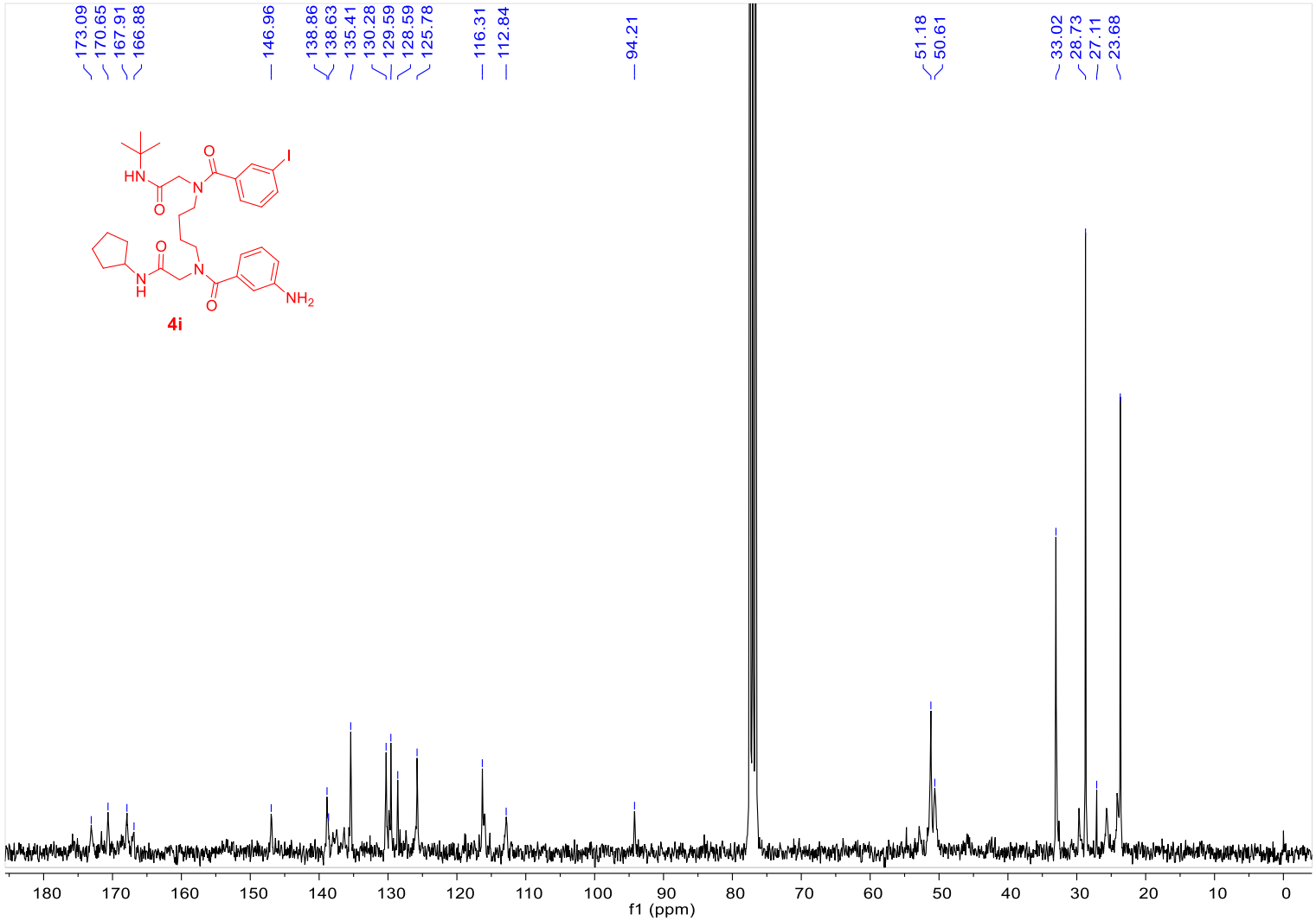


Figure 20. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(N-(2-(cyclopentylamino)-2-oxoethyl)-3-iodobenzamido)butyl)benzamide (**4i**).

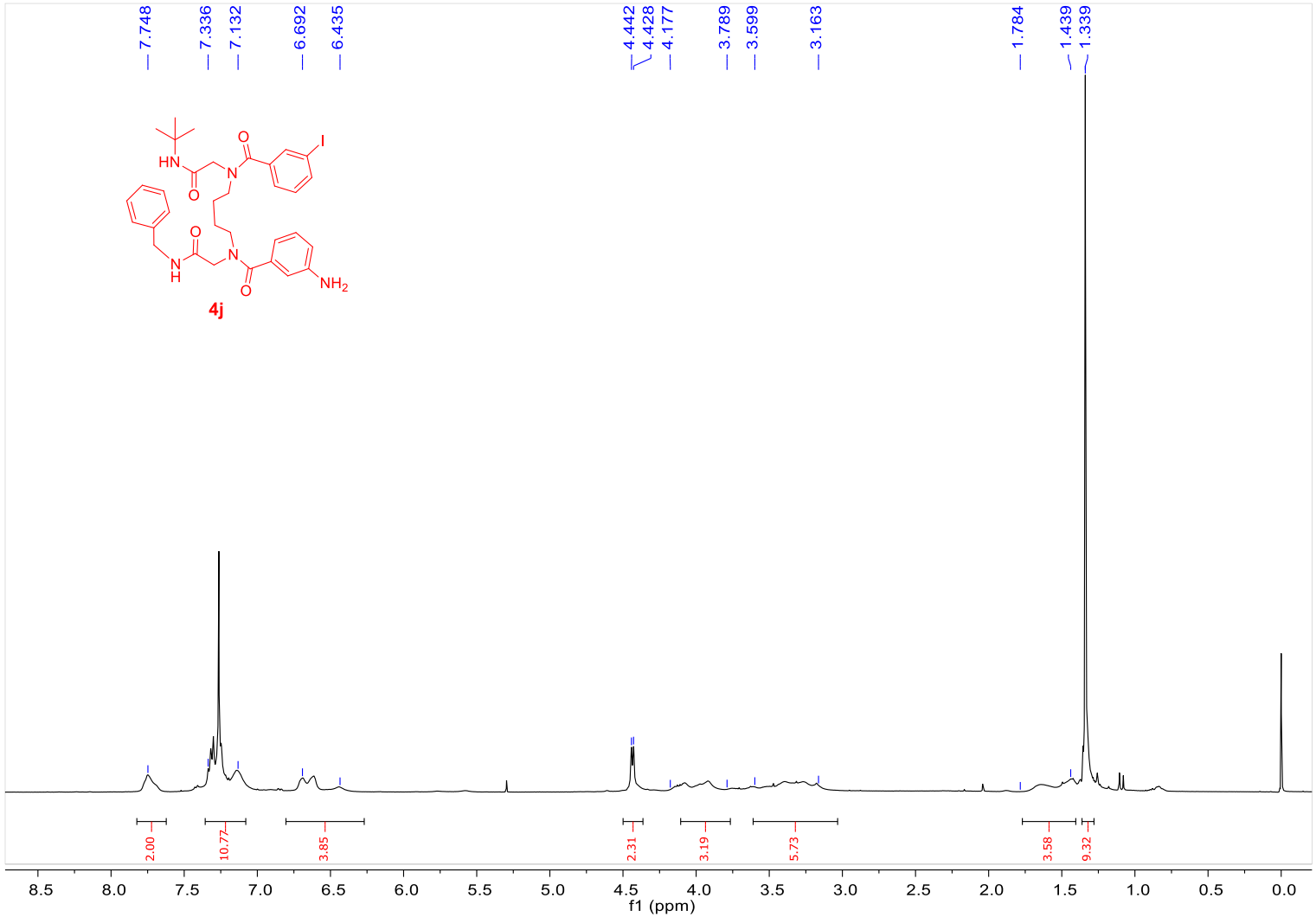


Figure 21. 400 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(4-(N-(2-(benzylamino)-2-oxoethyl)-3-iodobenzamido)butyl)-N-(2-(tert-butylamino)-2-oxoethyl)benzamide (**4j**).

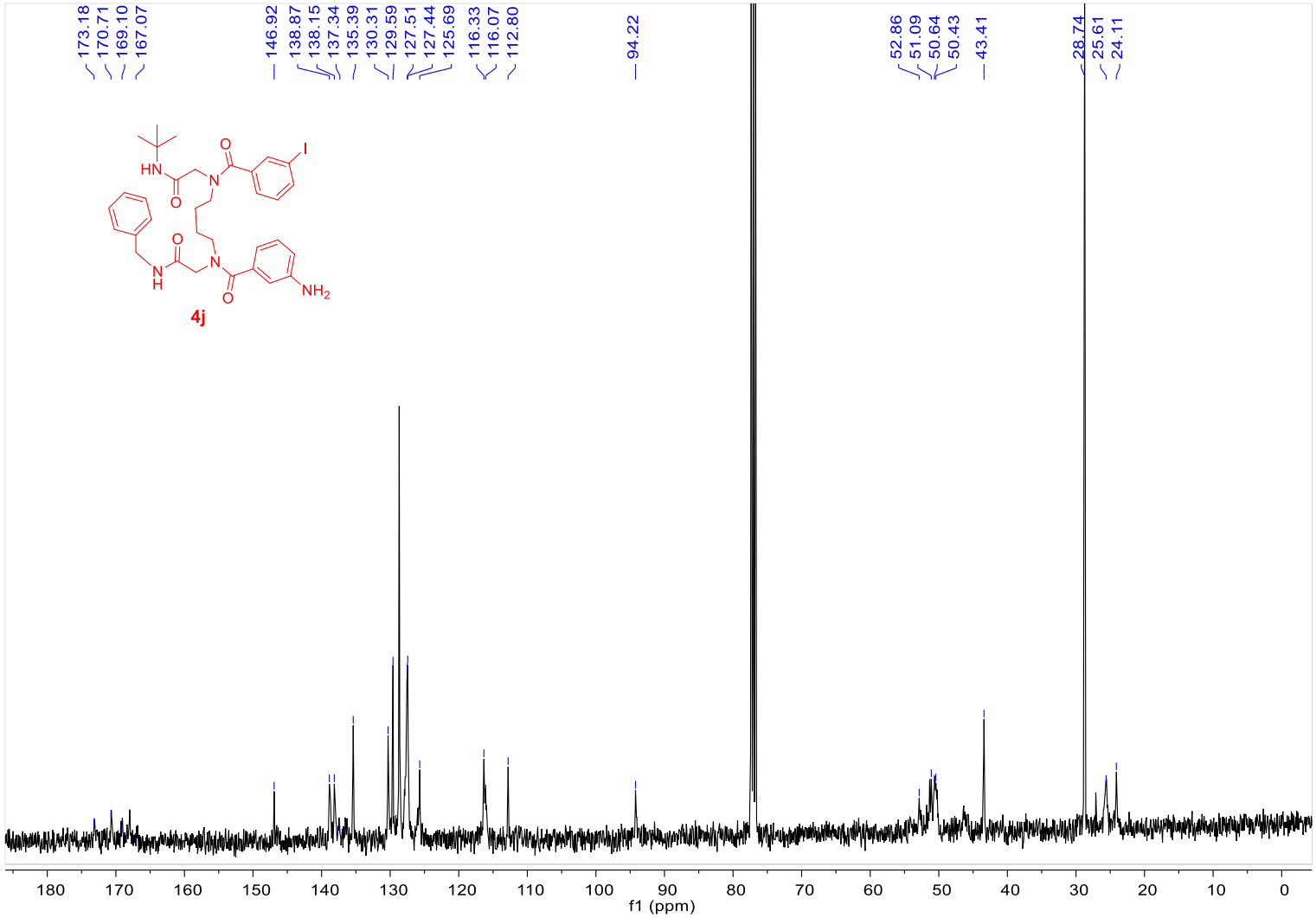


Figure 22. 100 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(4-(N-(2-(benzylamino)-2-oxoethyl)-3-iodobenzamido)butyl)-N-(2-(tert-butylamino)-2-oxoethyl)benzamide (**4j**).

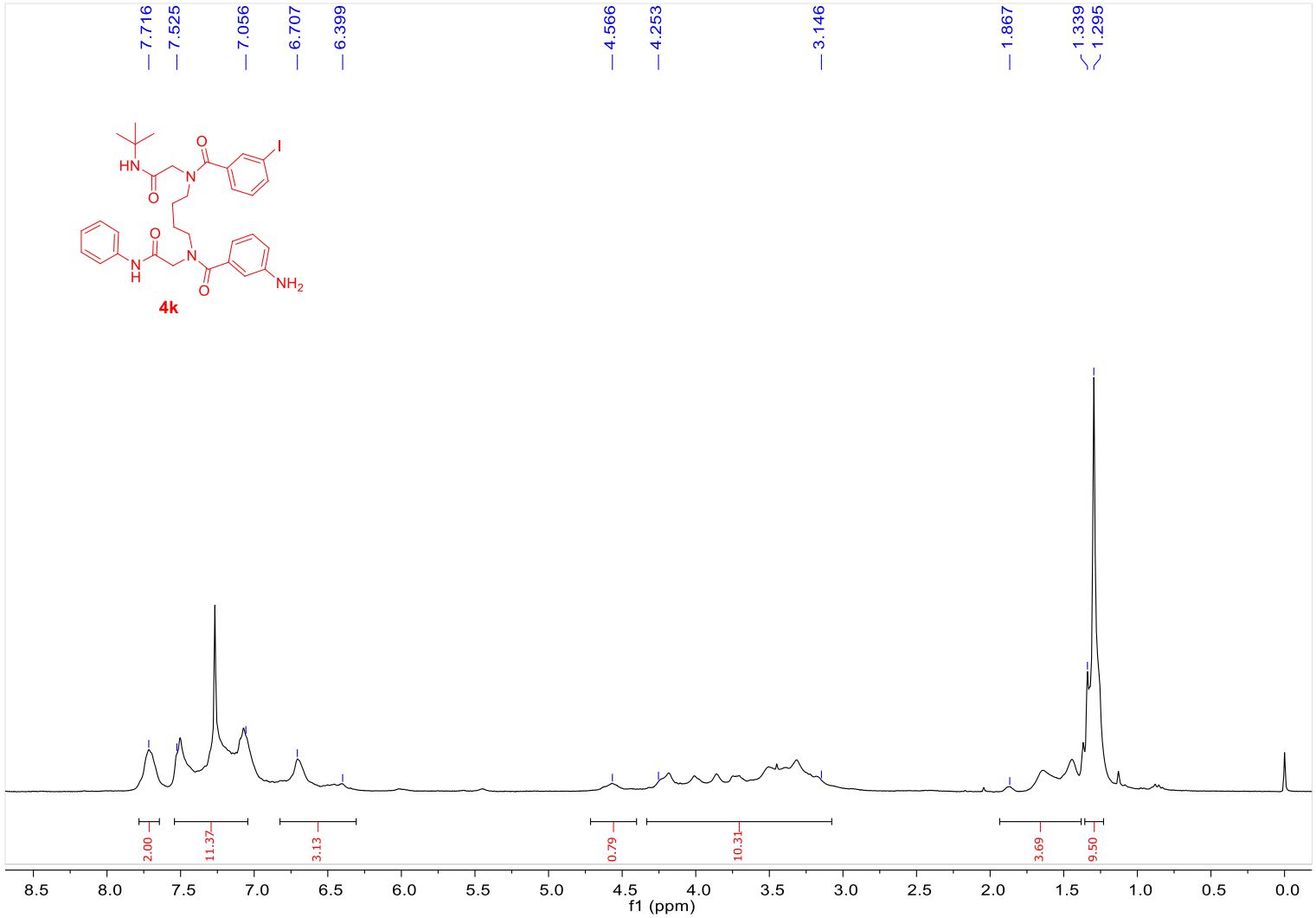


Figure 23. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(3-iodo-N-(2-oxo-2-(phenylamino)ethyl)benzamido)butyl)benzamide(**4k**).

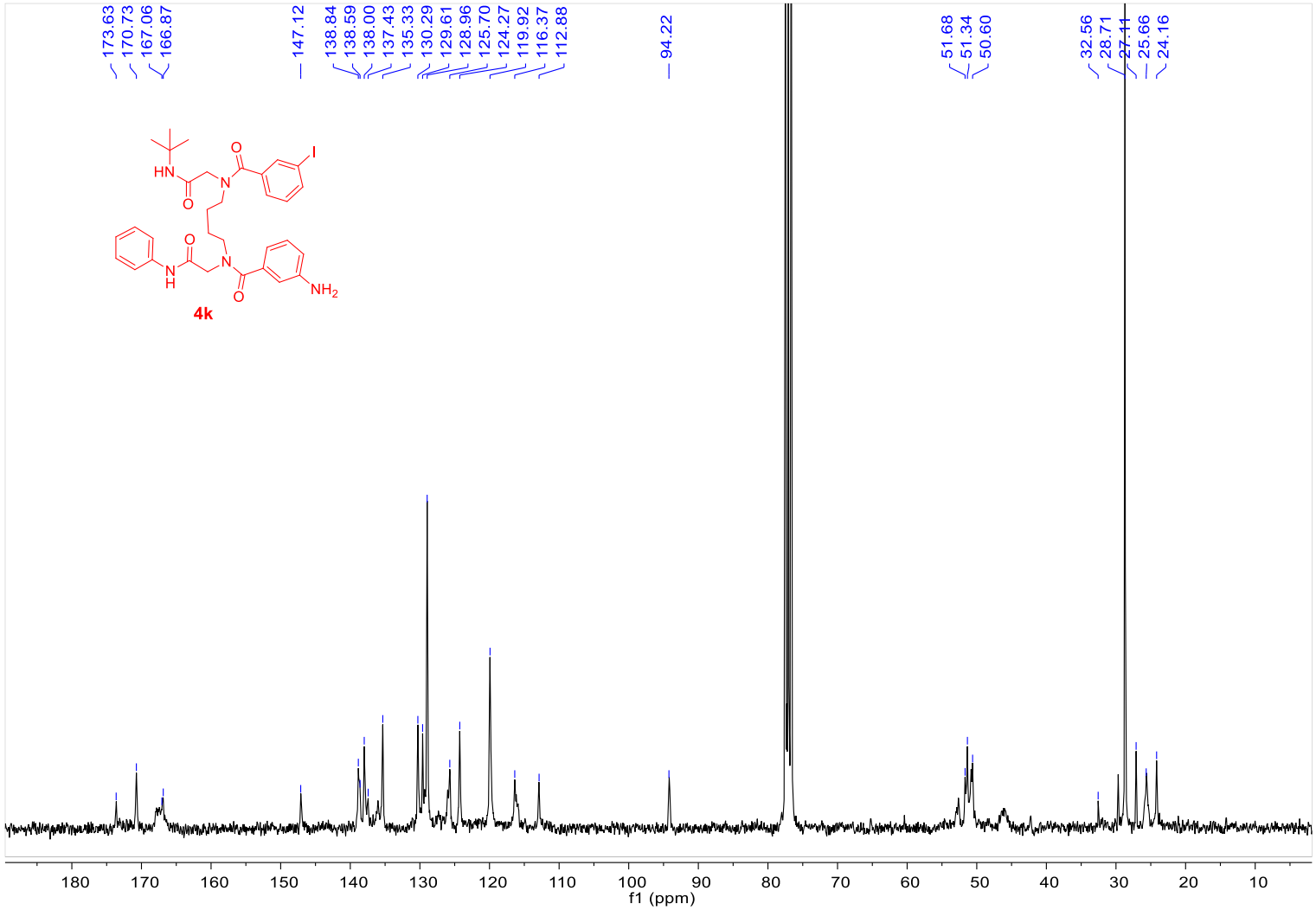


Figure 24. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(2-(tert-butylamino)-2-oxoethyl)-N-(4-(3-iodo-N-(2-oxo-2-(phenylamino)ethyl)benzamido)butyl)benzamide(**4k**).

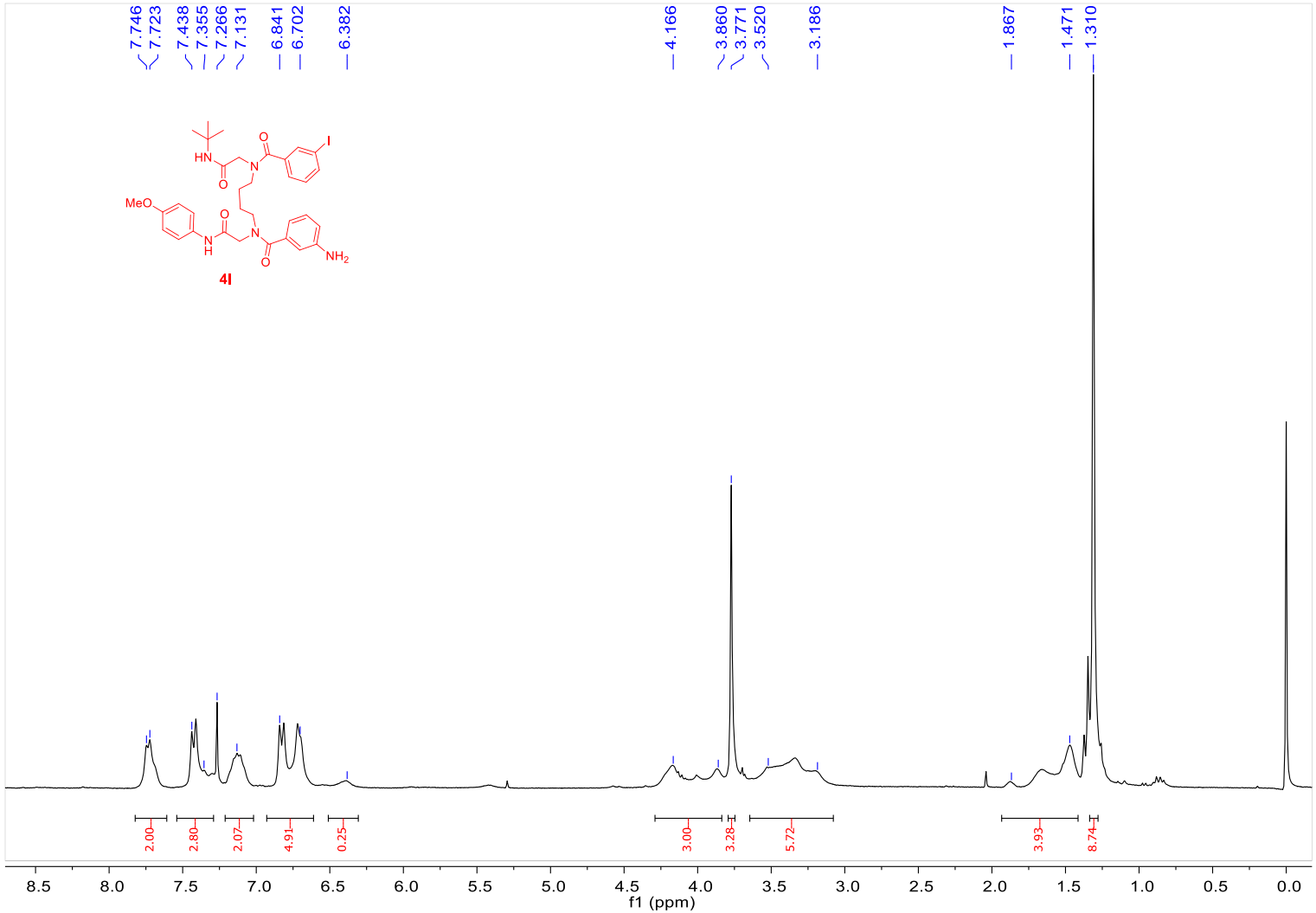


Figure 25. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(4-(N-(2-(tert-butylamino)-2-oxoethyl)-3-iodobenzamido)butyl)-N-(2-((4-methoxyphenyl)amino)-2-oxoethyl)benzamide (**4l**)

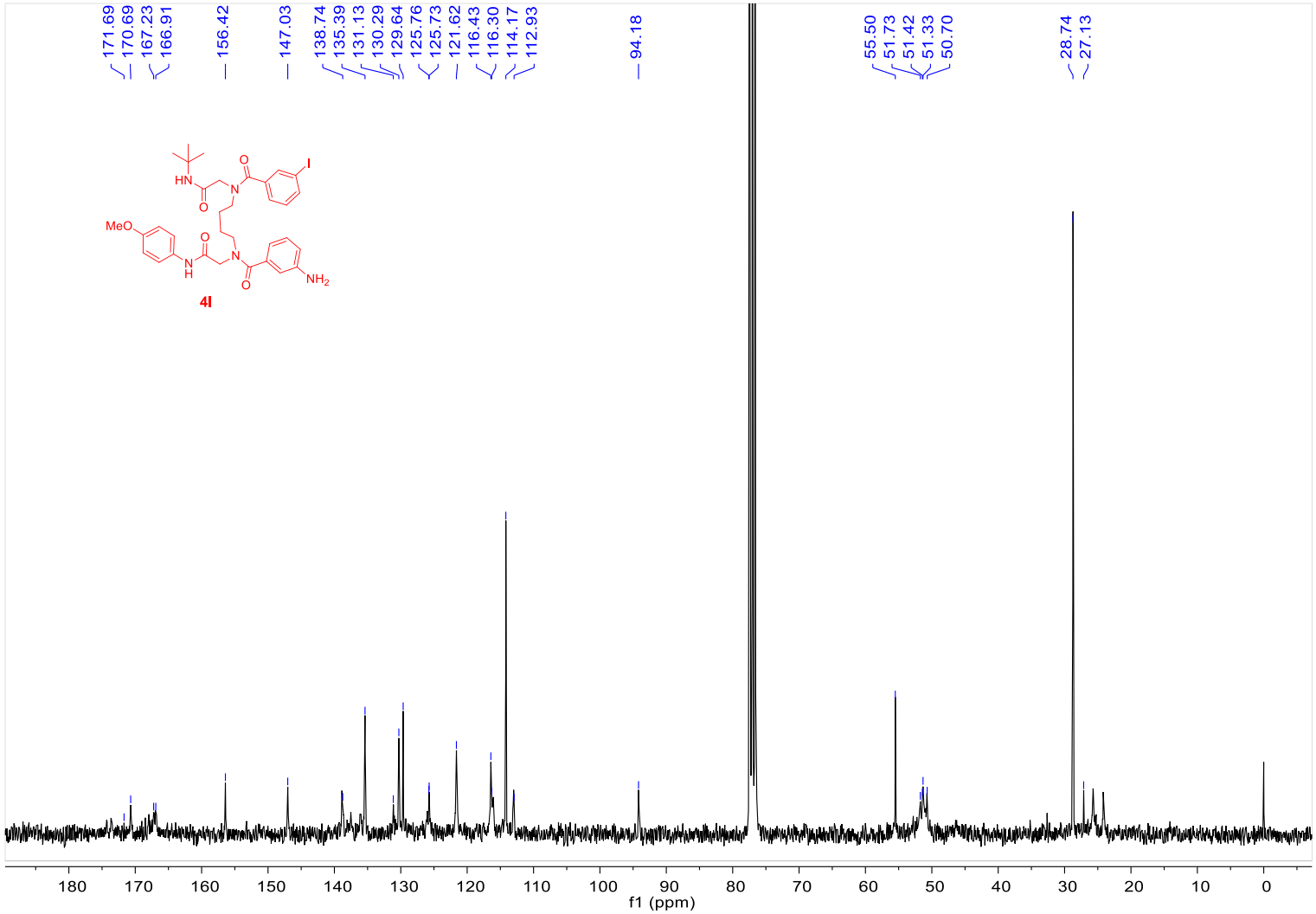


Figure 26. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(4-(N-(2-(tert-butylamino)-2-oxoethyl)-3-iodobenzamido)butyl)-N-(2-((4-methoxyphenyl)amino)-2-oxoethyl)benzamide (**4l**)

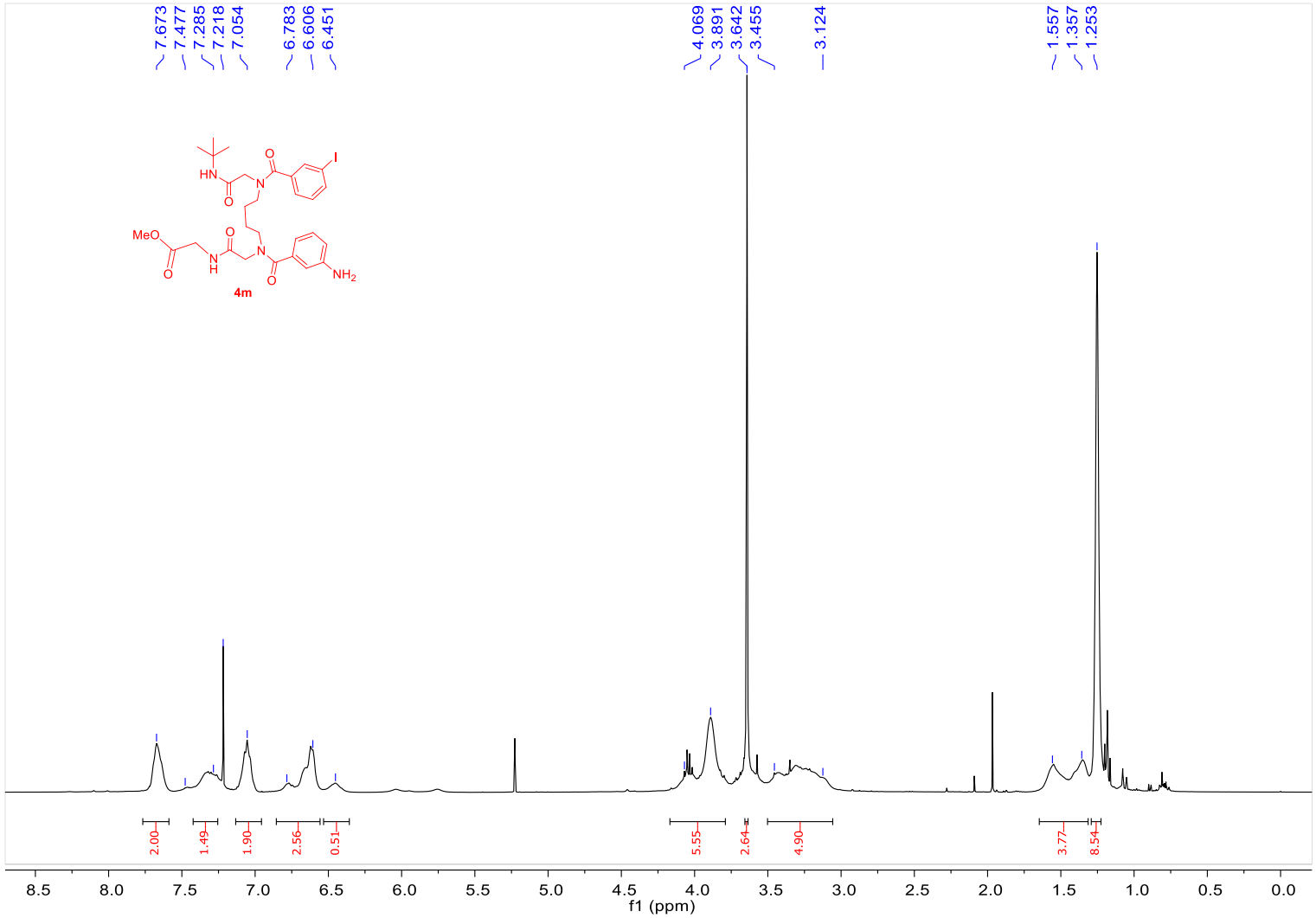


Figure 27. 400 MHz ^{1}H NMR spectra in $CDCl_3$ of methyl6-(3-aminobenzoyl)-11-(3-iodobenzoyl)-15,15-dimethyl-4,13-dioxo-3,6,11,14-tetraazahexadecan-1-oate (**4m**)

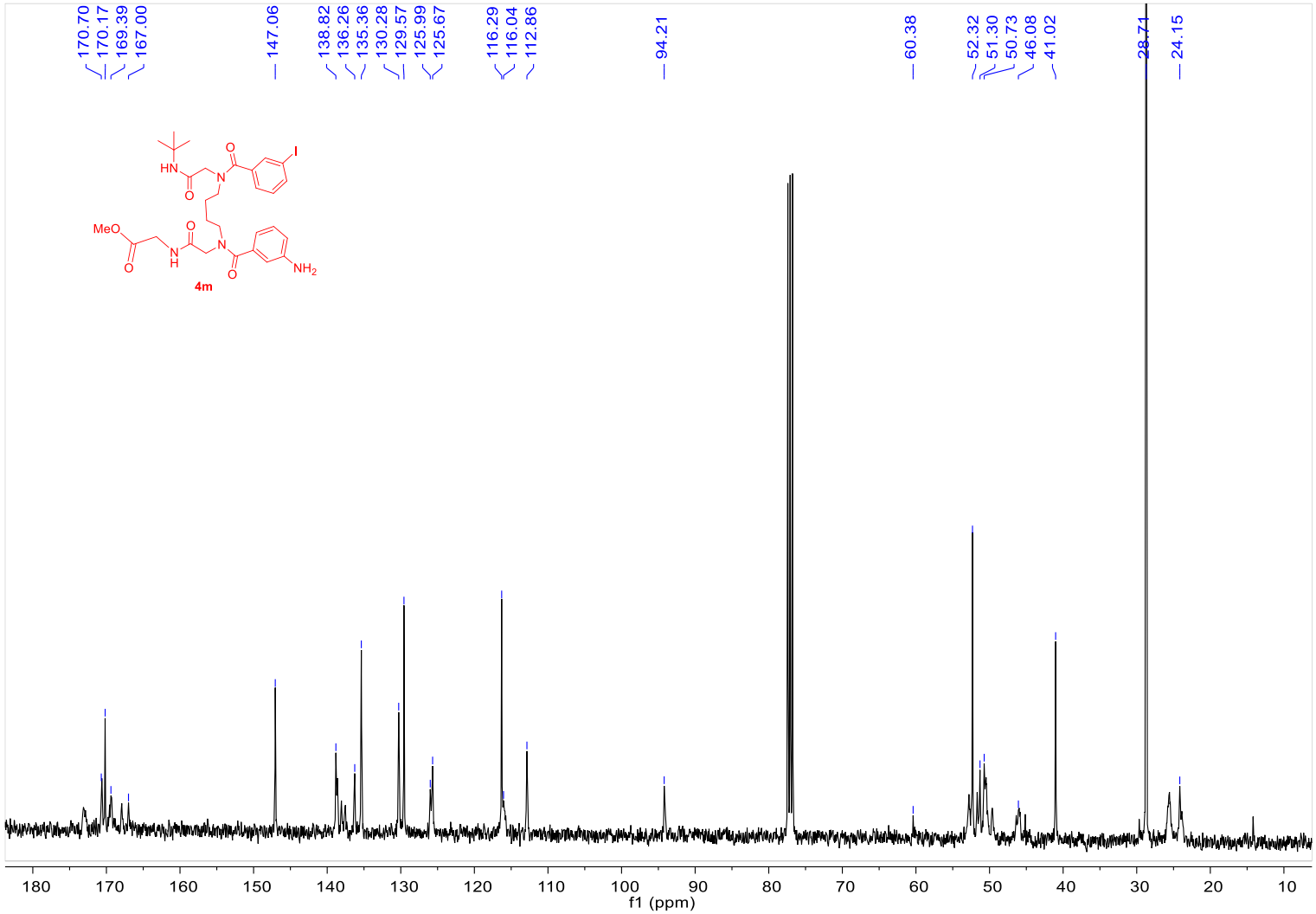


Figure 28. 100 MHz ^{13}C NMR spectra in $CDCl_3$ of methyl6-(3-aminobenzoyl)-11-(3-iodobenzoyl)-15,15-dimethyl-4,13-dioxo-3,6,11,14-tetraazahexadecan-1-oate (**4m**)

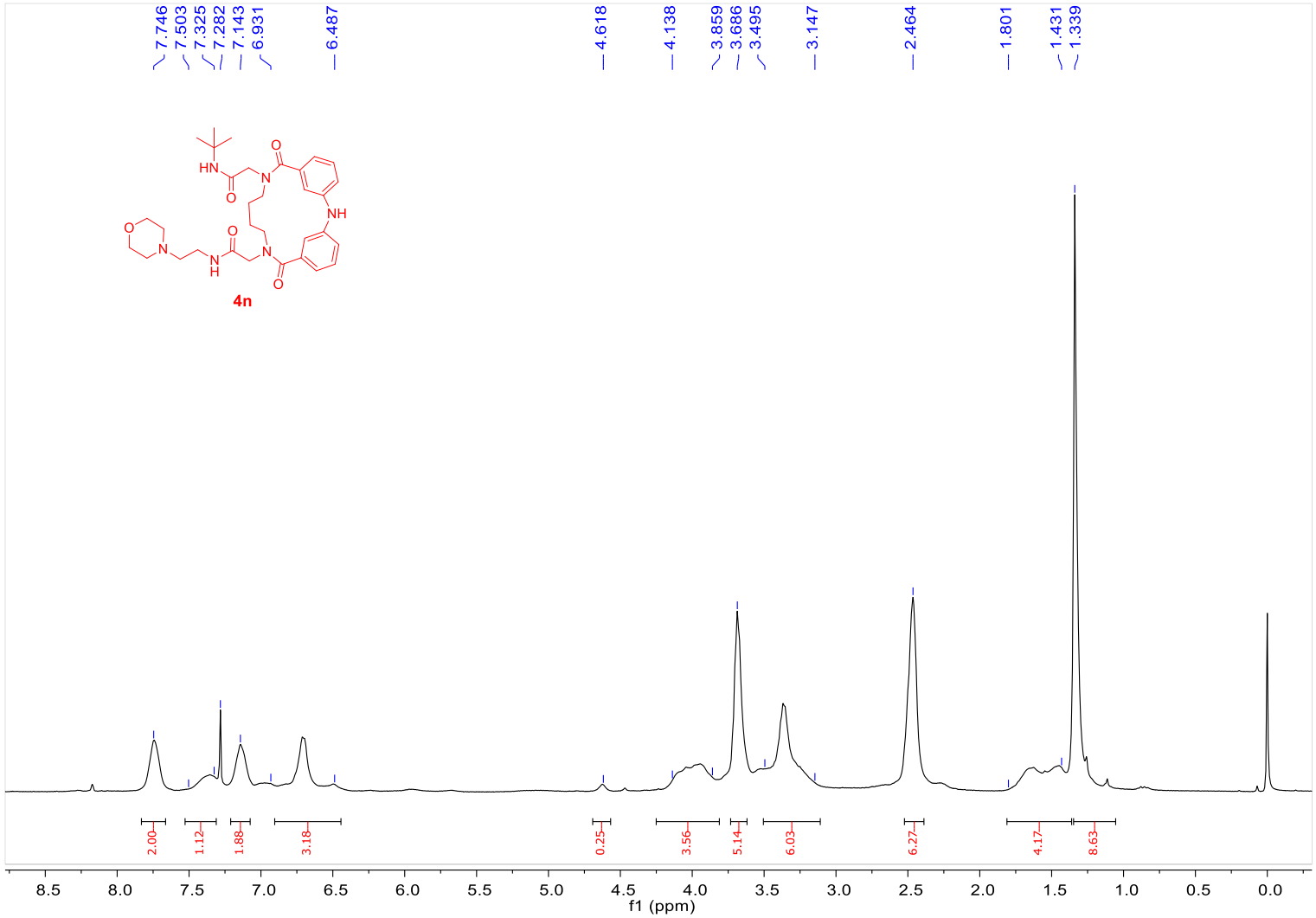


Figure 29. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 3-amino-N-(4-(N-(2-(tert-butylamino)-2-oxoethyl)-3-iodobenzamido)butyl)-N-(2-(morpholinoamino)-2-oxoethyl)benzamide (**4n**)

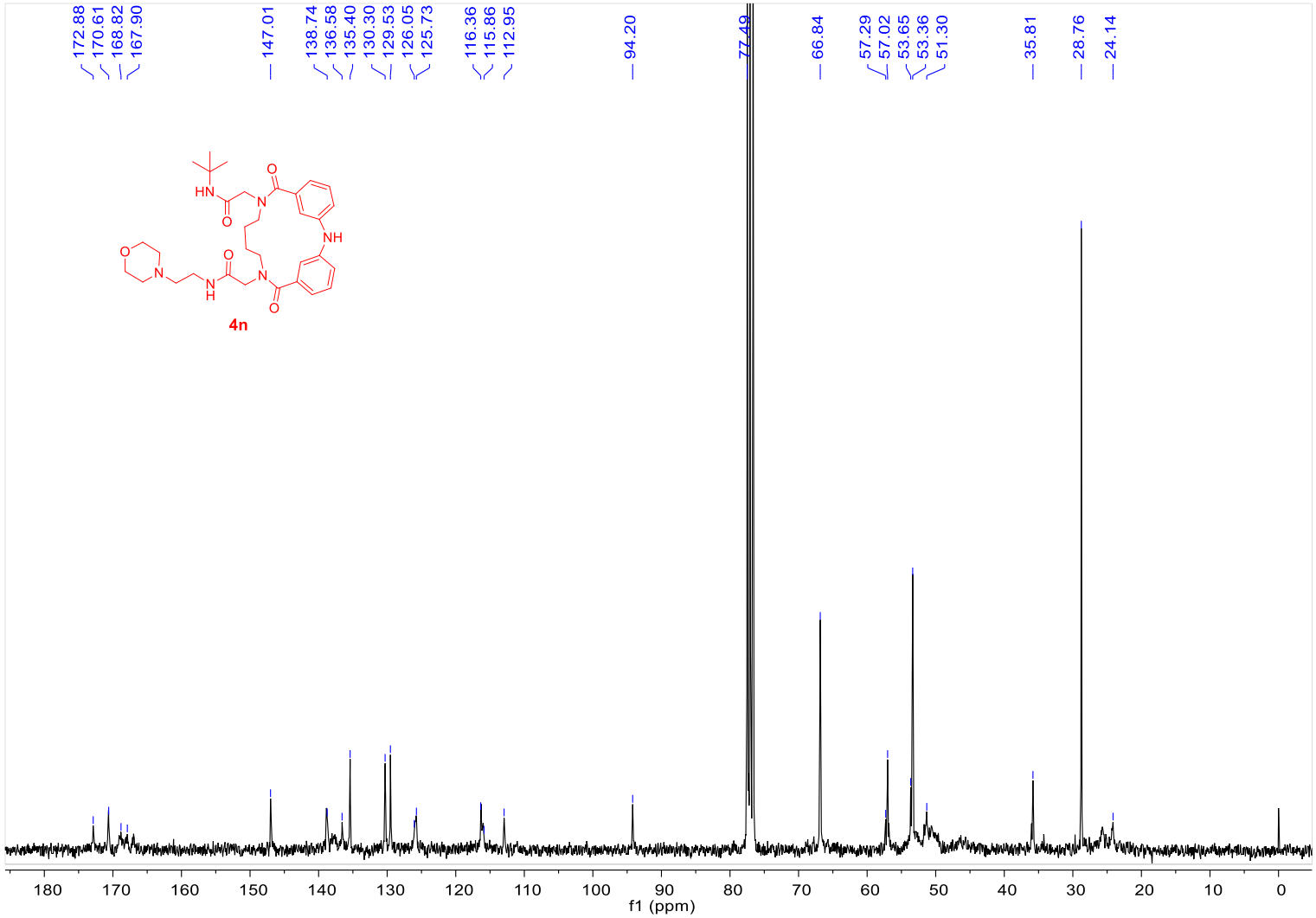


Figure 30. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 3-amino-N-(4-(N-(2-(tert-butylamino)-2-oxoethyl)-3-iodobenzamido)butyl)-N-(2-(morpholinoamino)-2-oxoethyl)benzamide (**4n**)

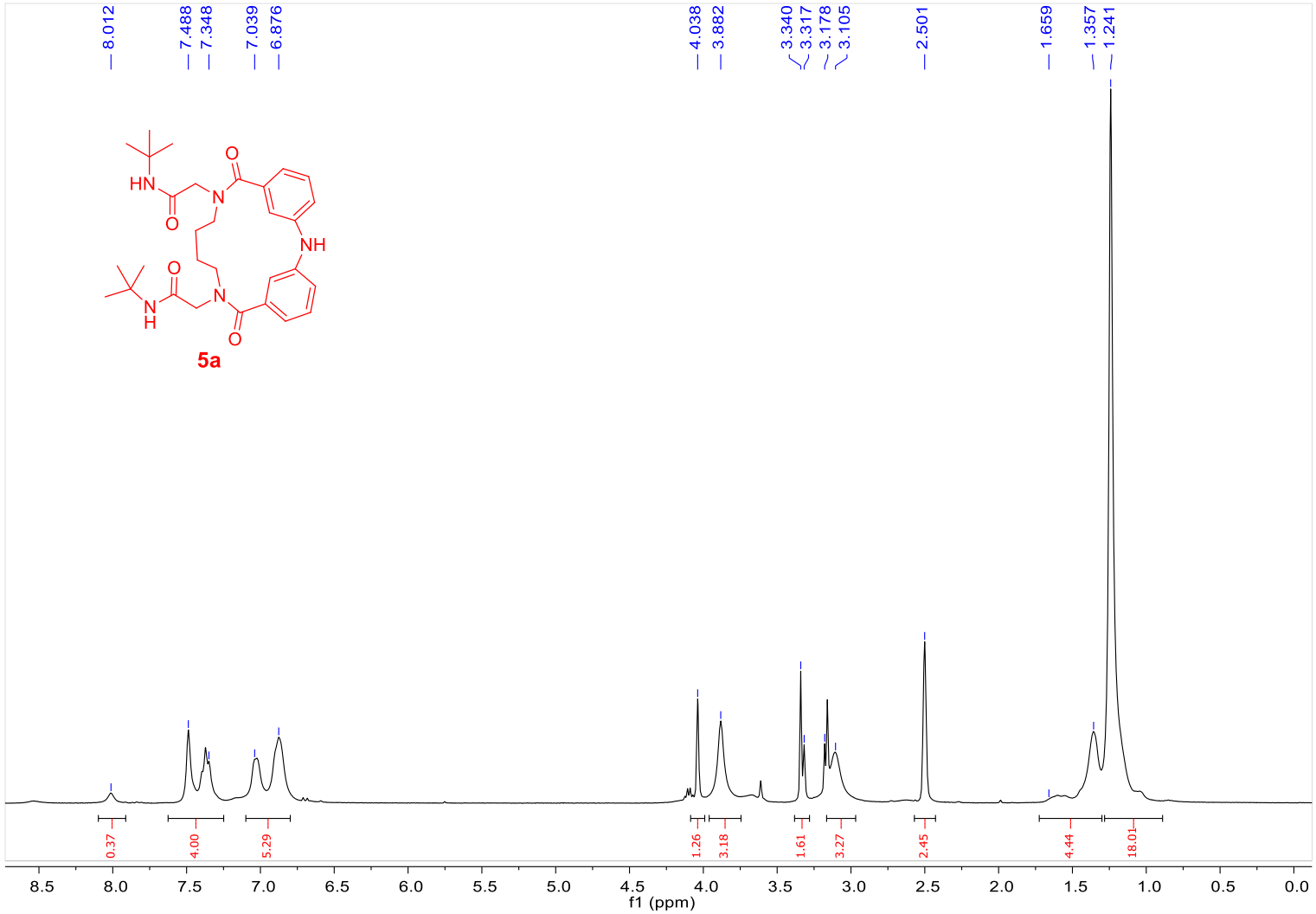


Figure 31. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of 2,2'-(4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5,10-diyl)bis(N-(tert-butyl)acetamide) (**5a**)

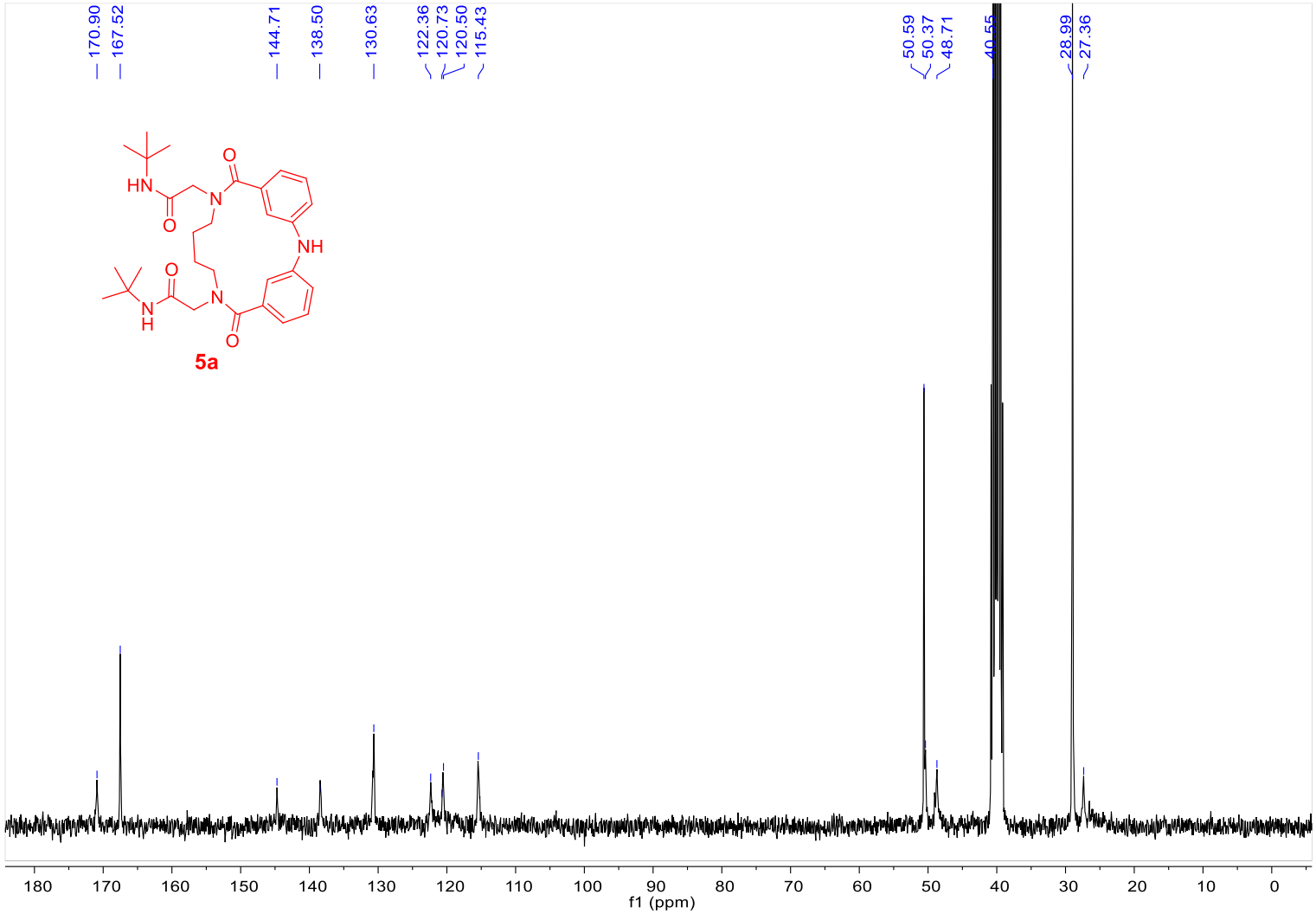


Figure 32. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of 2,2'-(4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5,10-diyl)bis(N-(tert-butyl)acetamide) (**5a**)

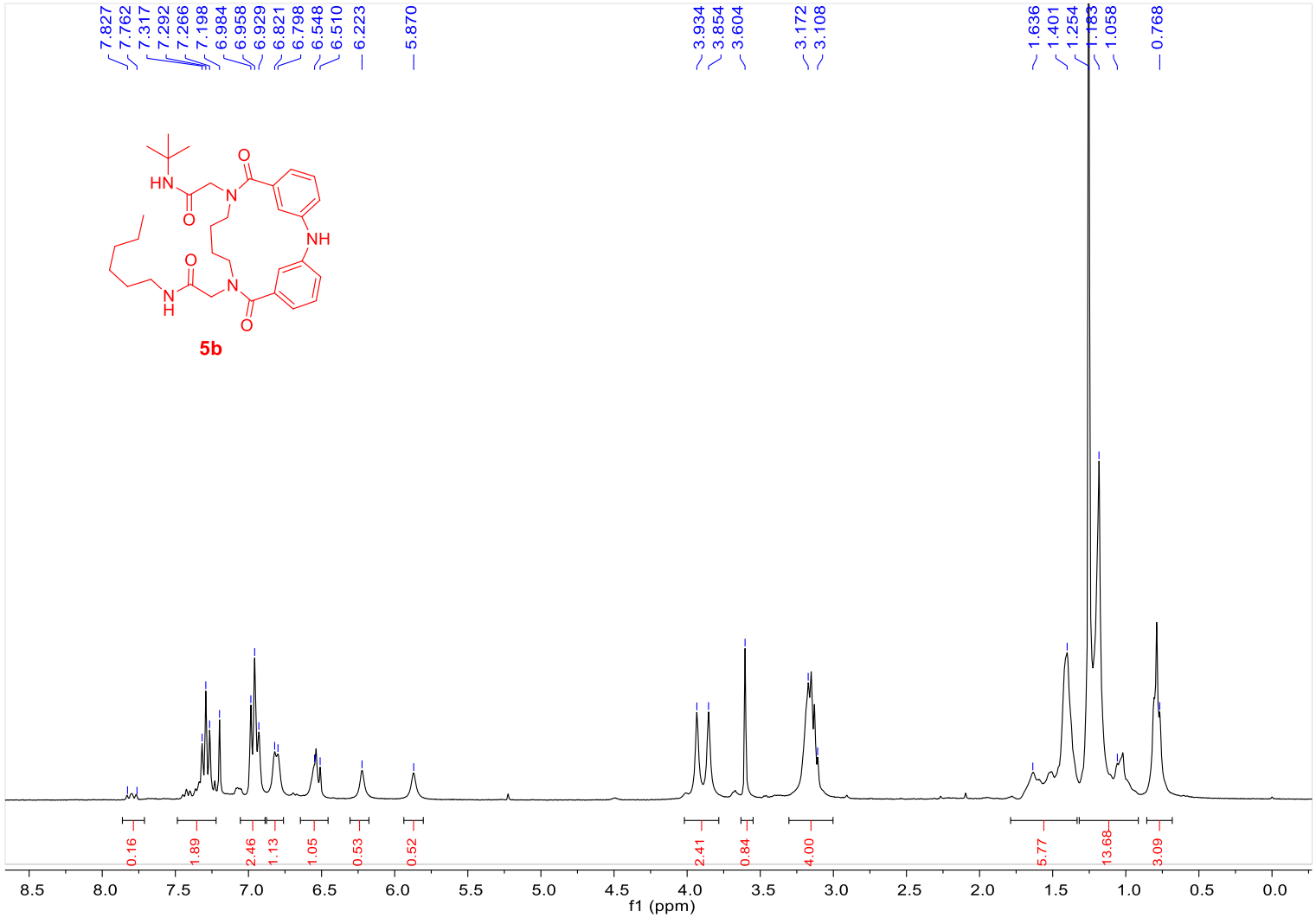


Figure 33. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(hexylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5b**)

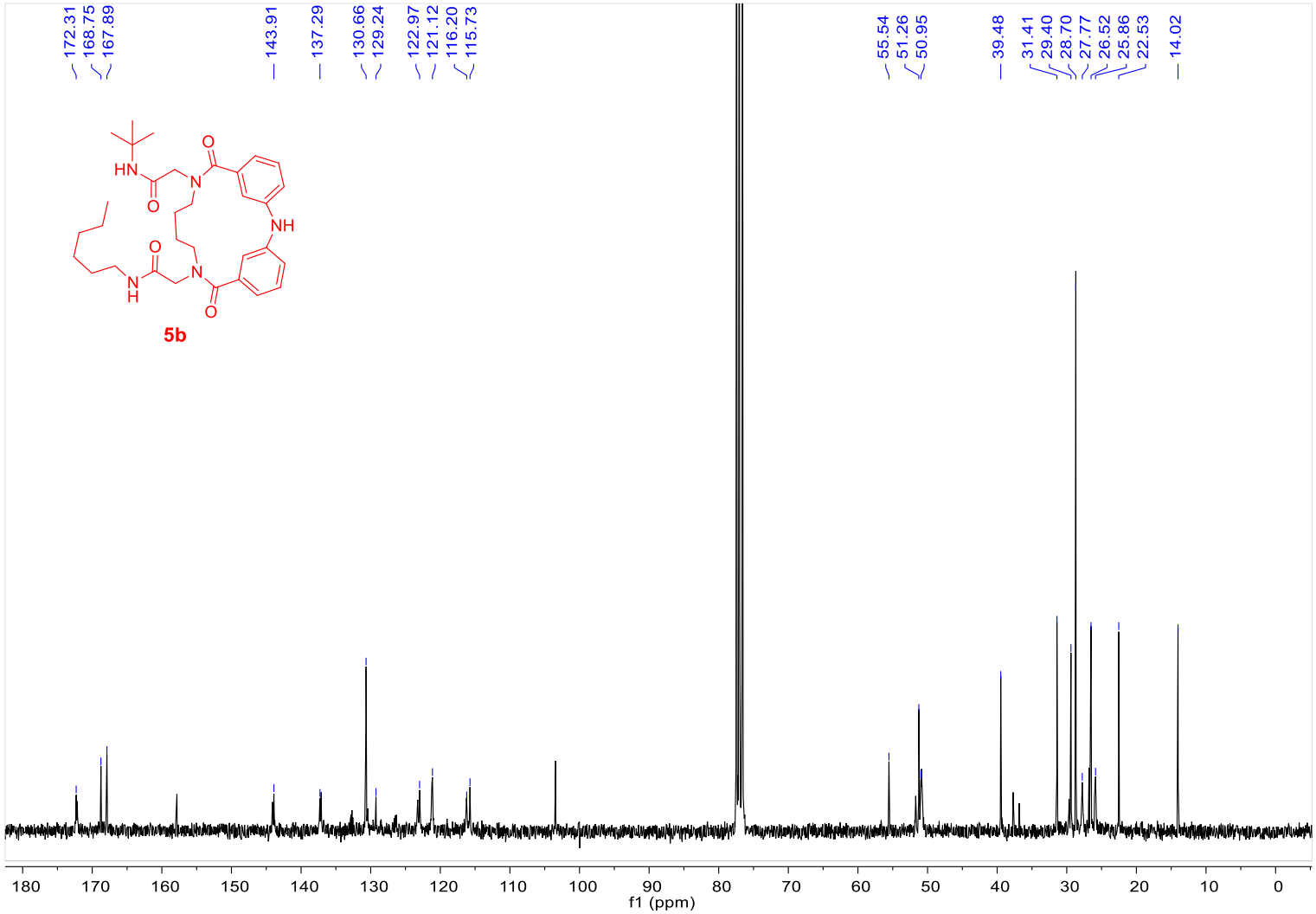


Figure 34. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(hexylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5b**)

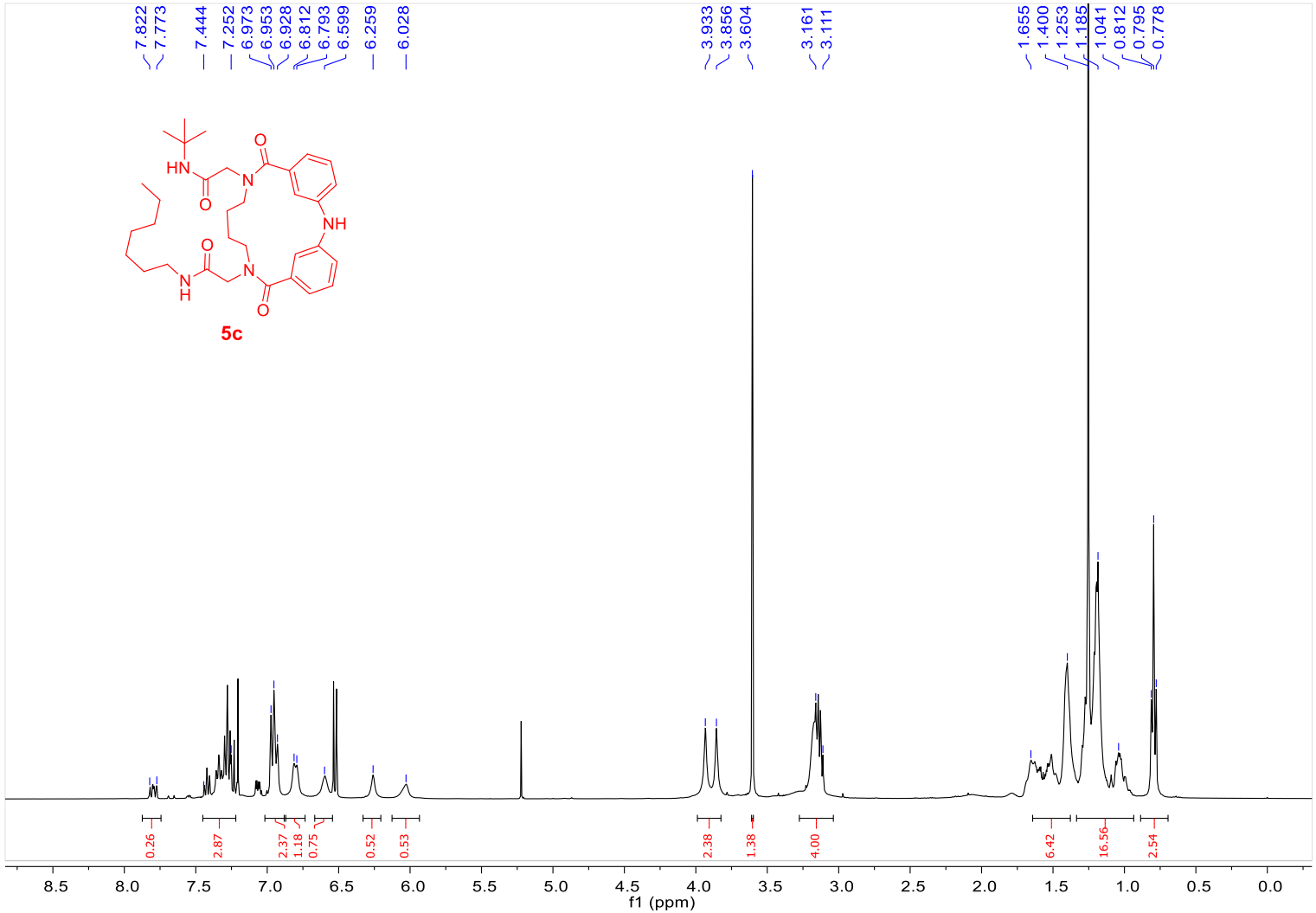


Figure 35. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(heptylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5c**)

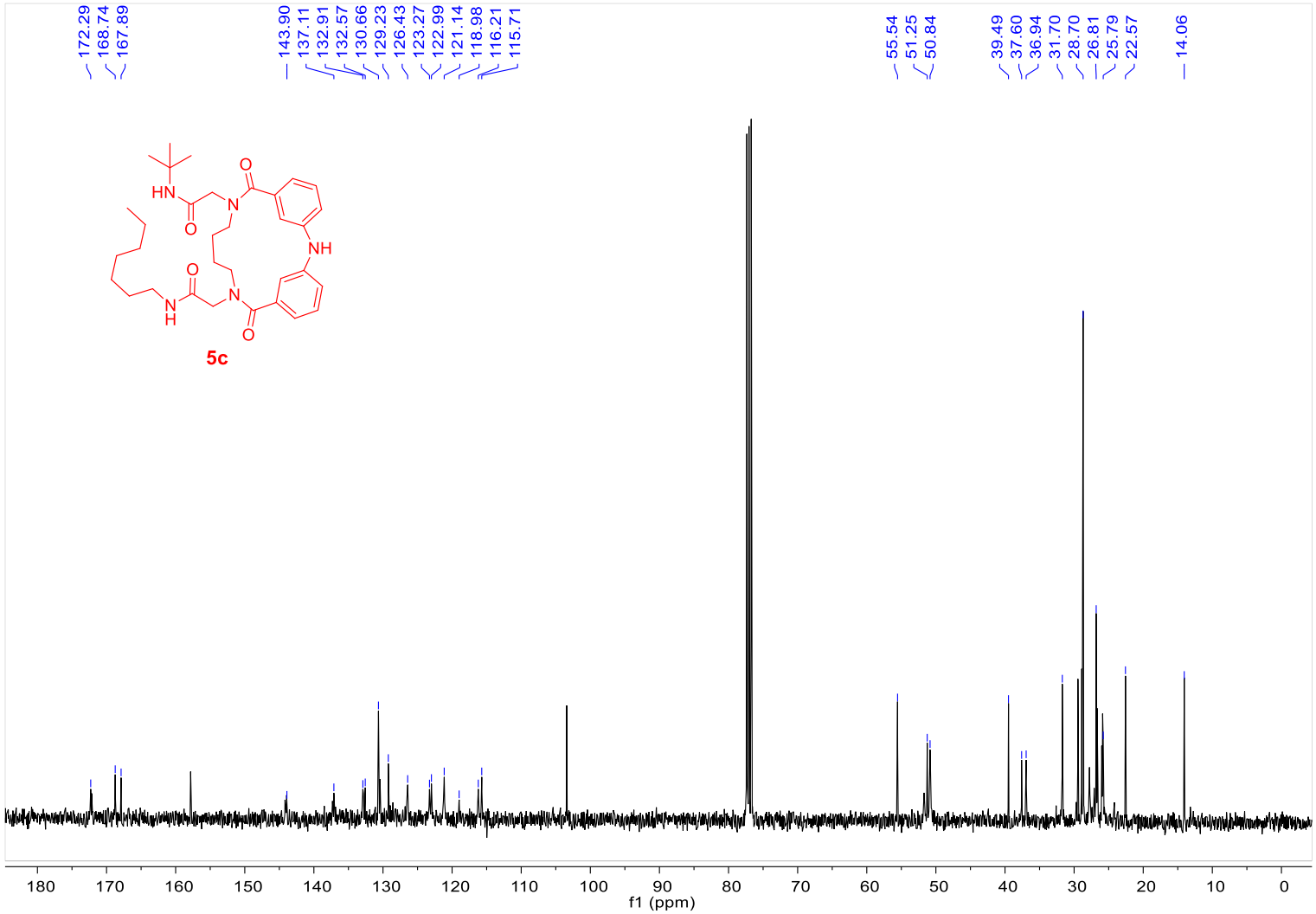


Figure 36. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(heptylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5c**)

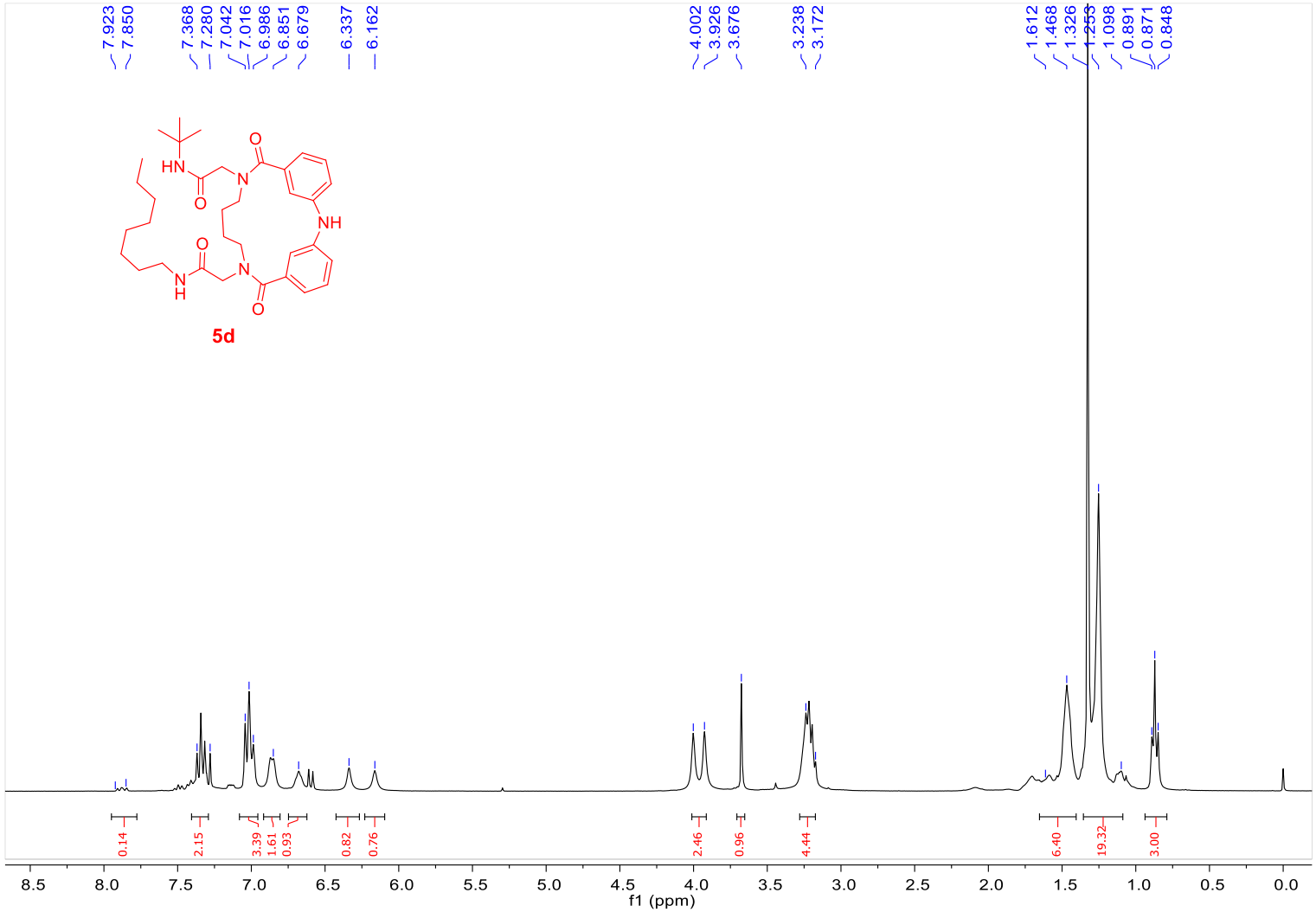


Figure 37. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(octylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5d**)

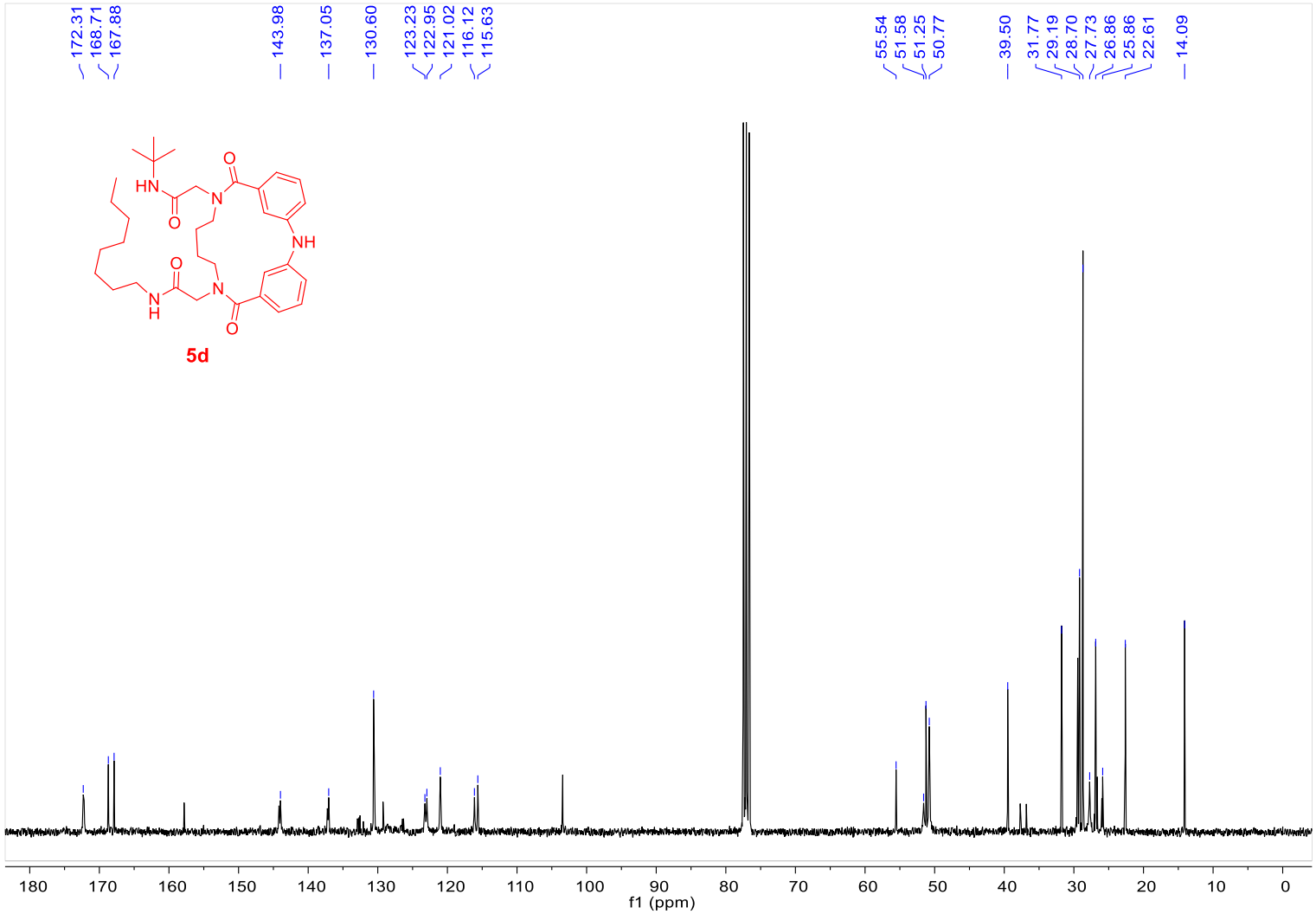


Figure 38. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(octylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5d**)

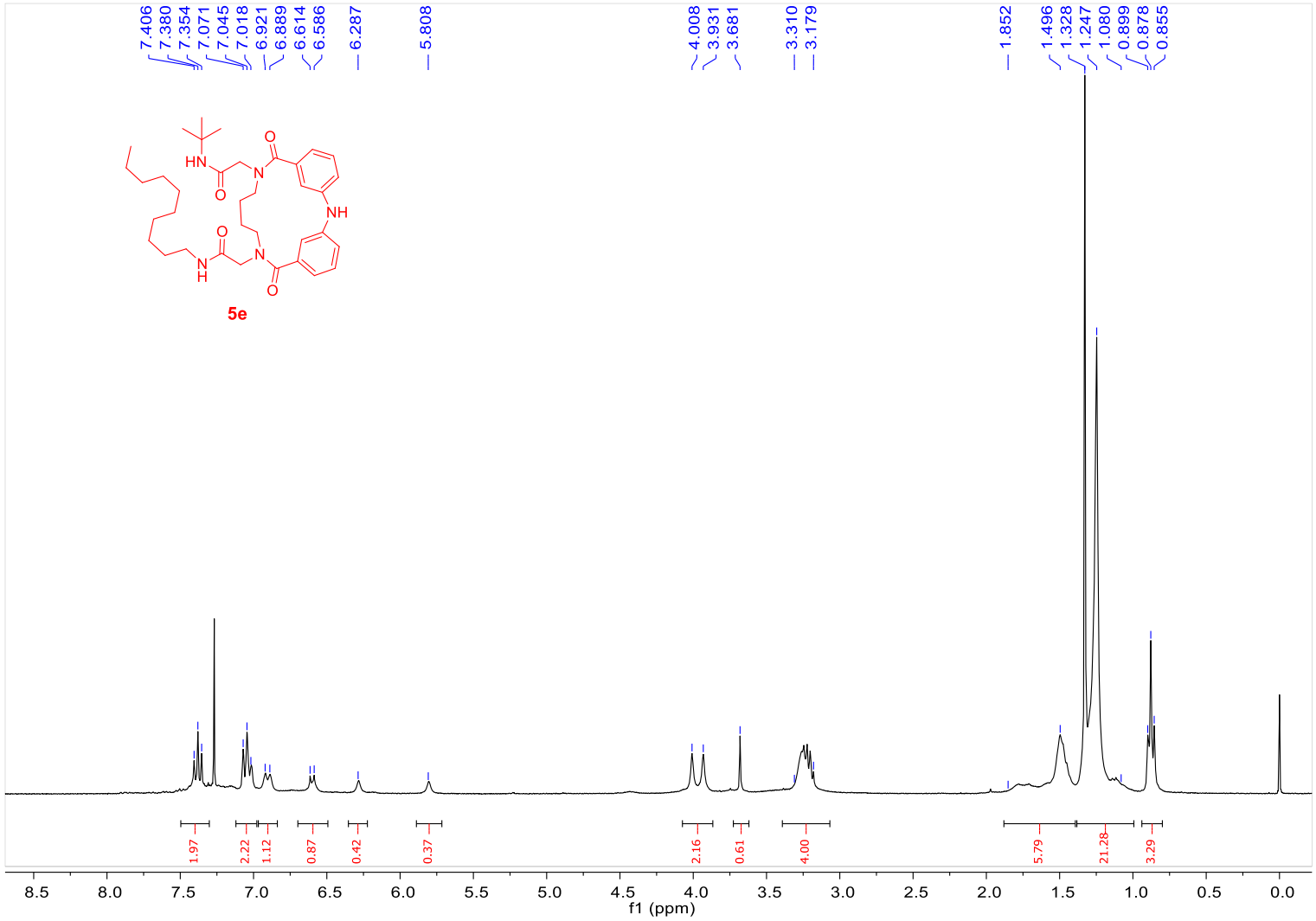


Figure 39. 400 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(decylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5e**)

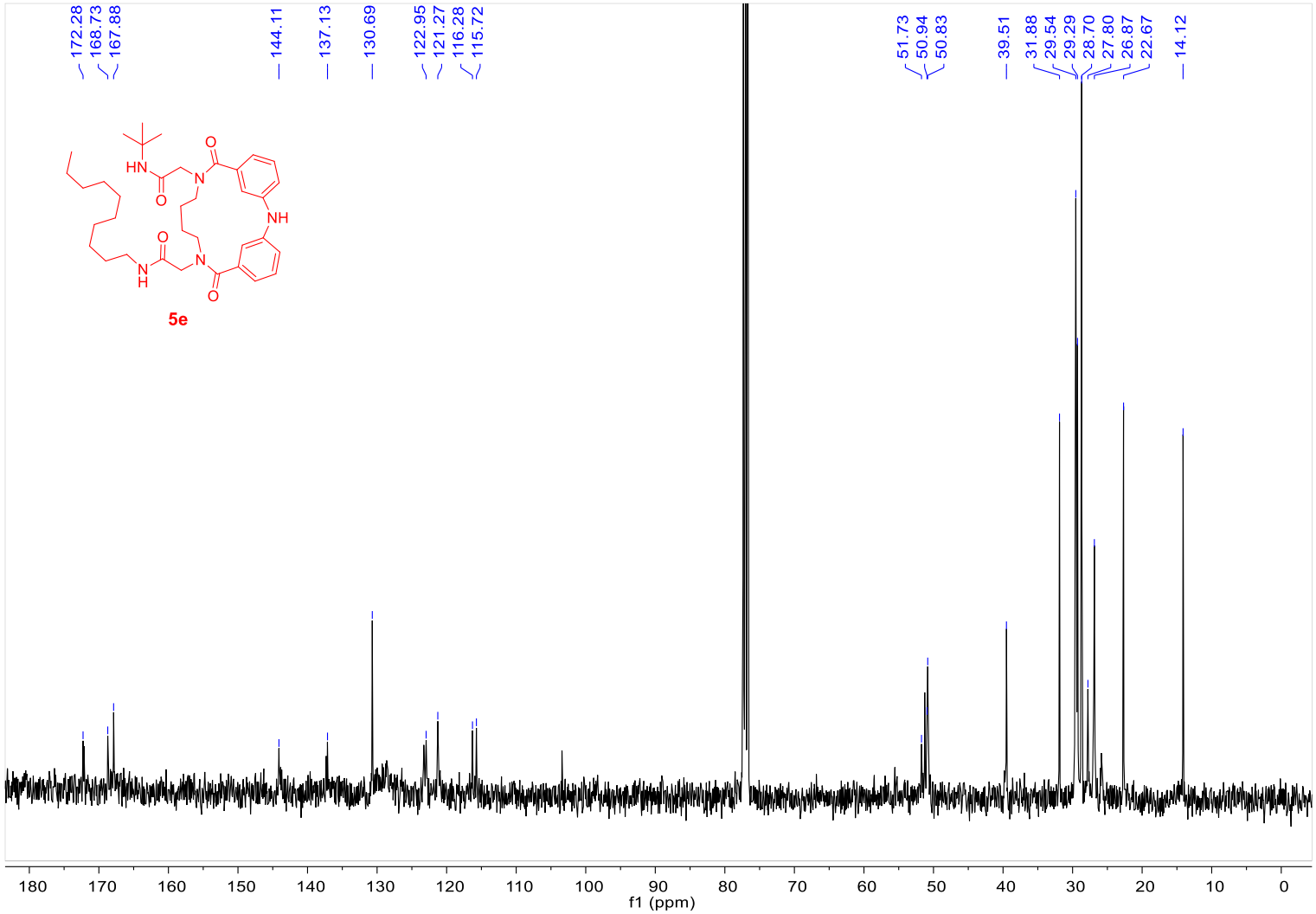


Figure 40. 100 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(decylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5e**)

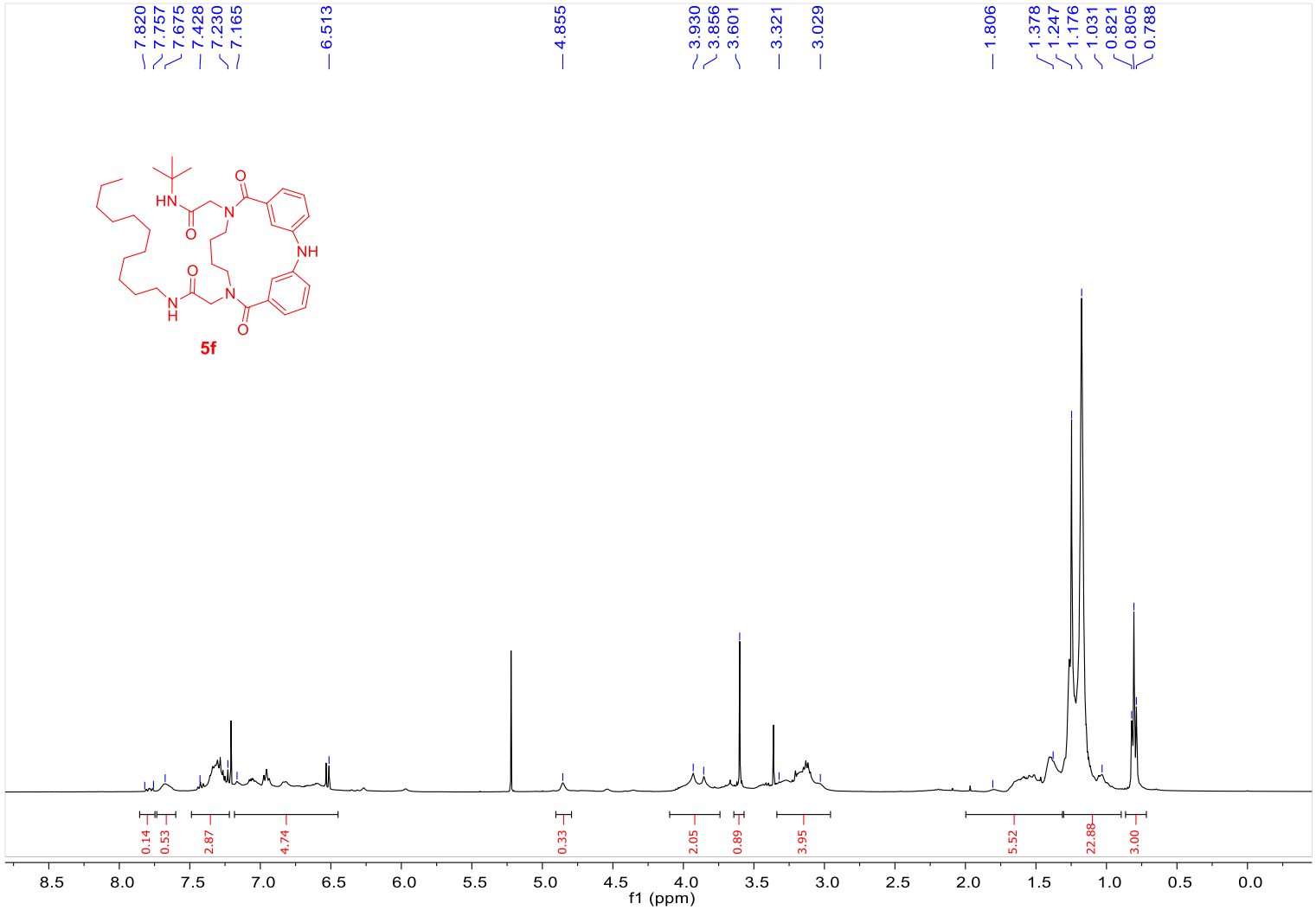


Figure 41. 400 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(4,11-dioxo-10-(2-oxo-2-(undecylamino)ethyl)-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5f**)

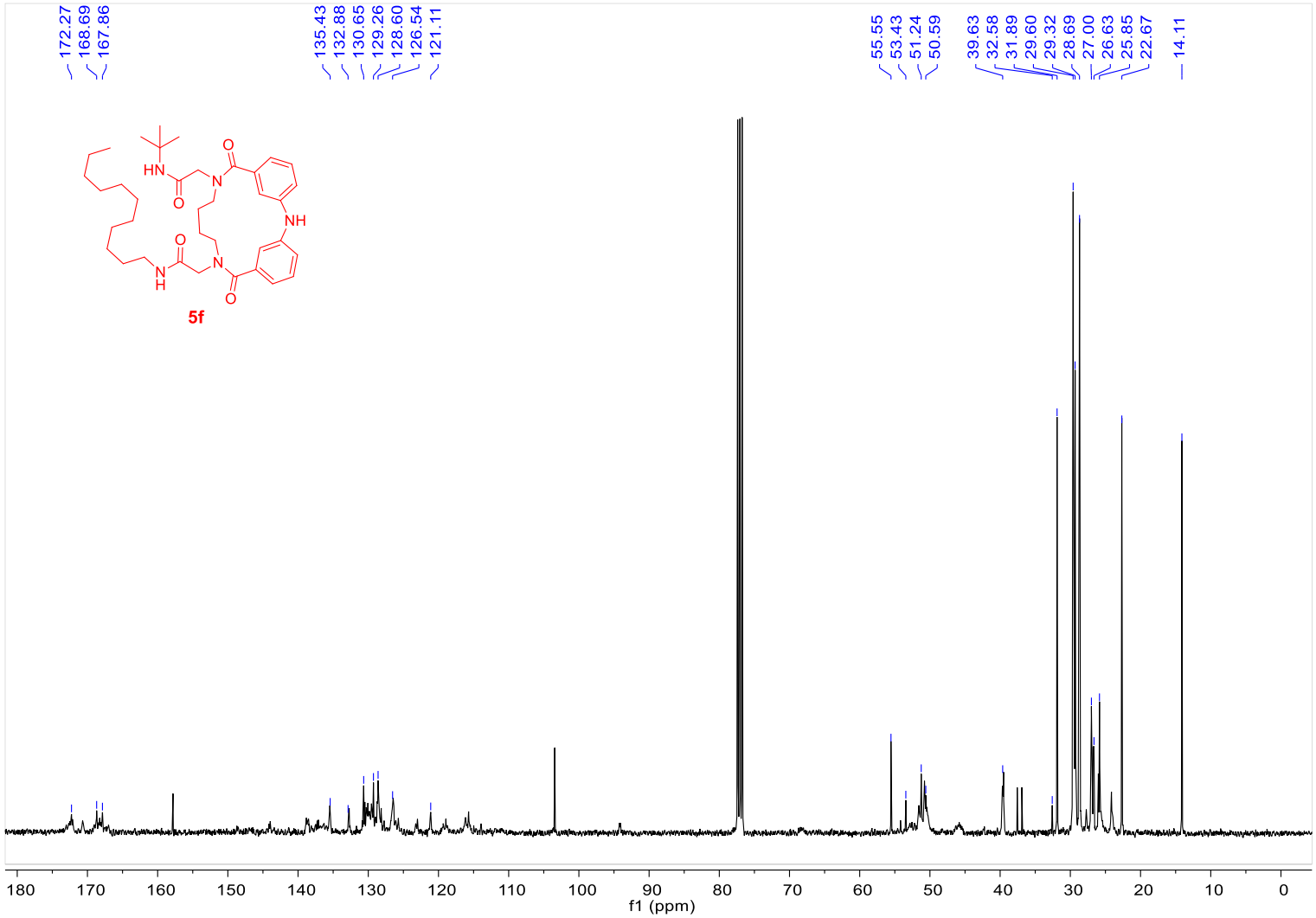


Figure 42. 100 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(4,11-dioxo-10-(2-oxo-2-(undecylamino)ethyl)-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5f**)

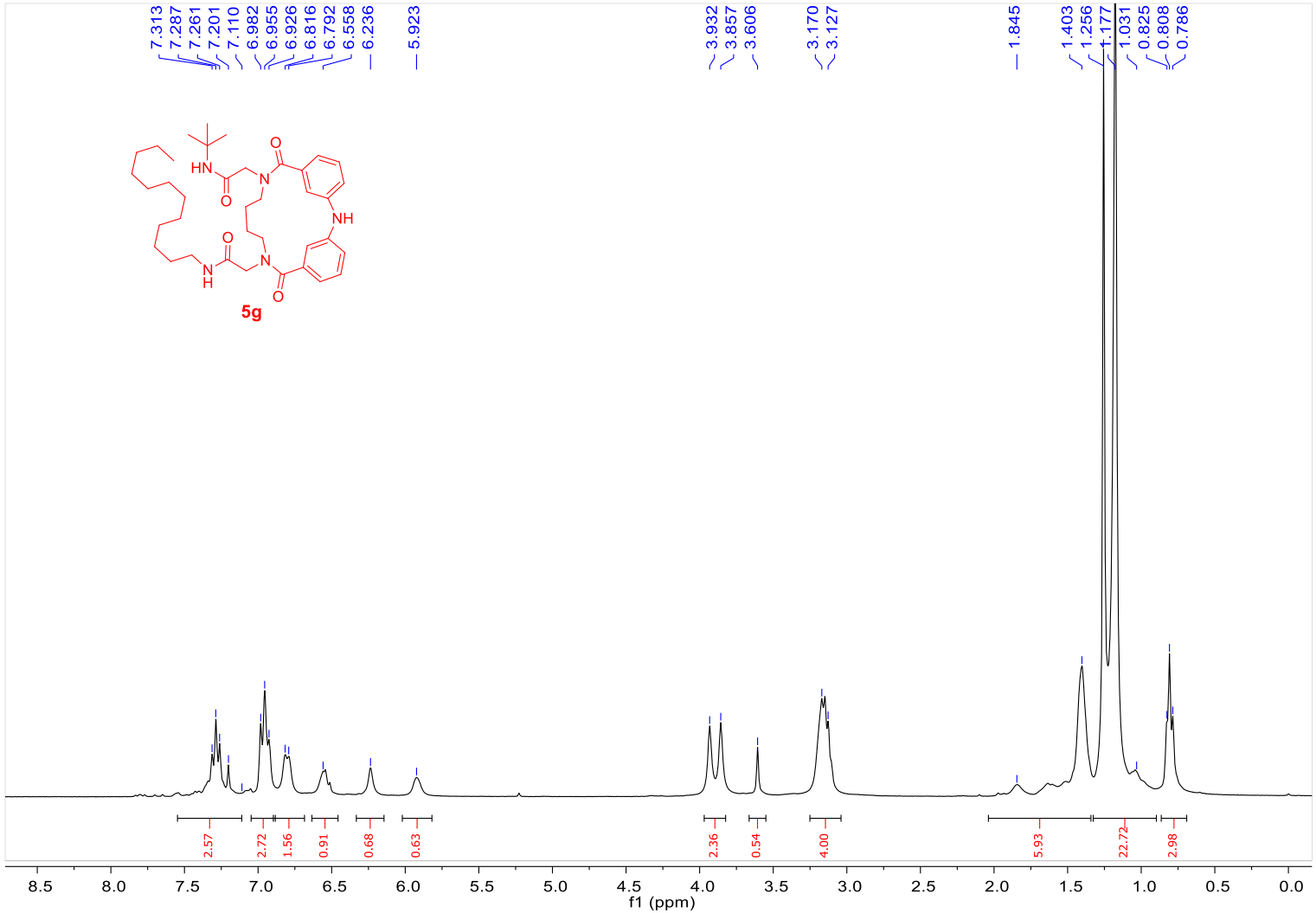


Figure 43. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(dodecylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5g**)

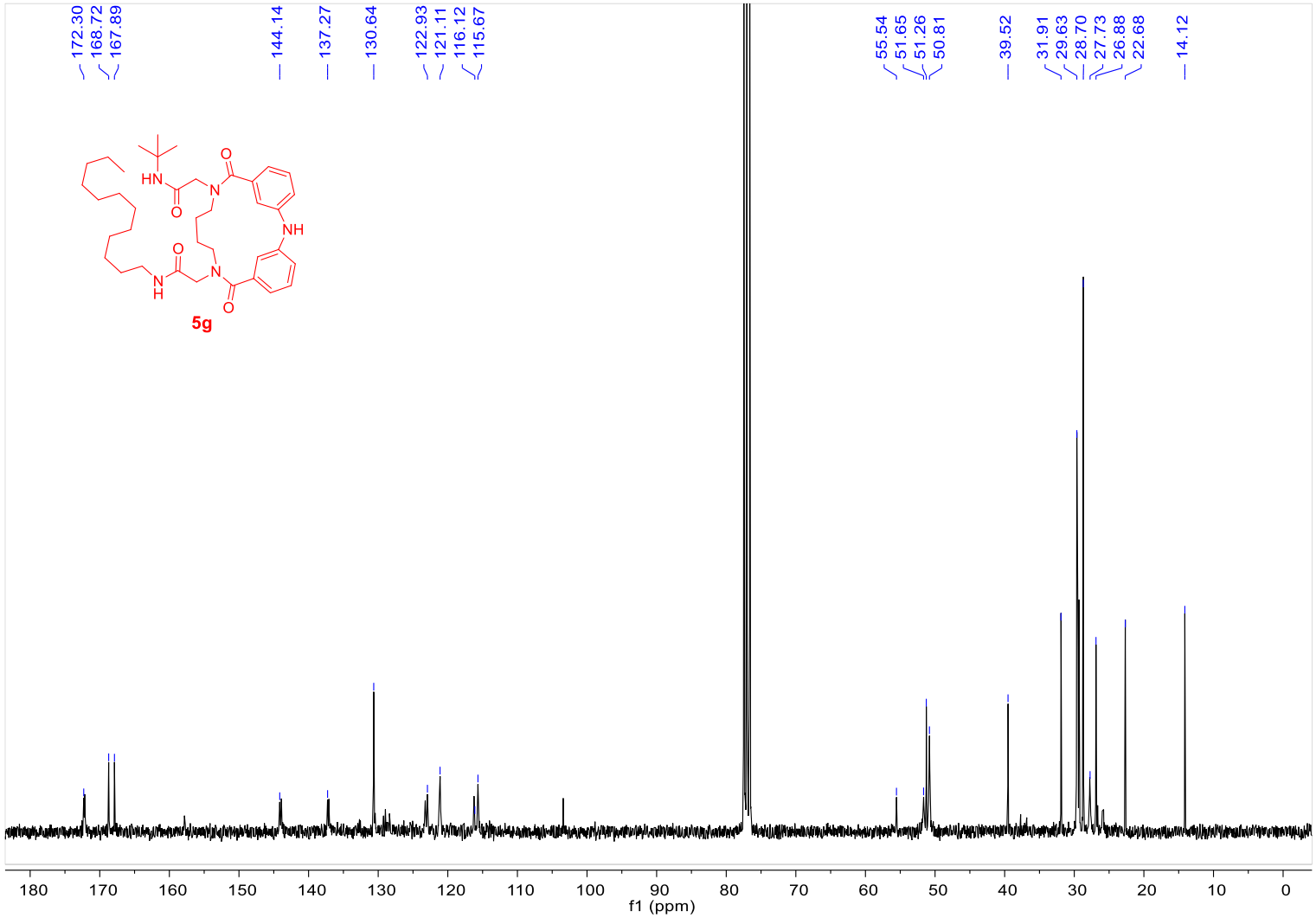


Figure 44. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(dodecylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5g**)

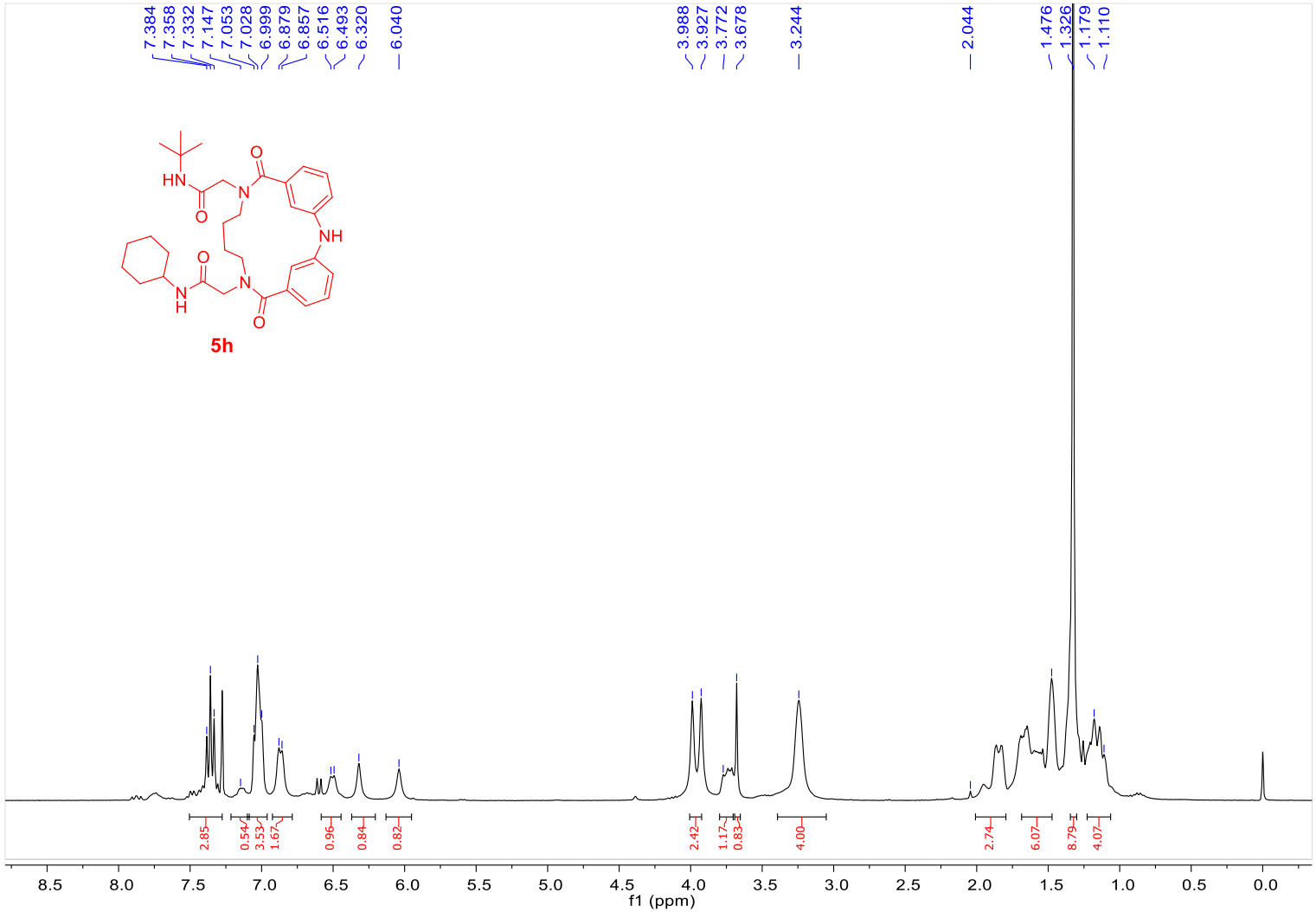


Figure 45. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(cyclohexylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5h**)

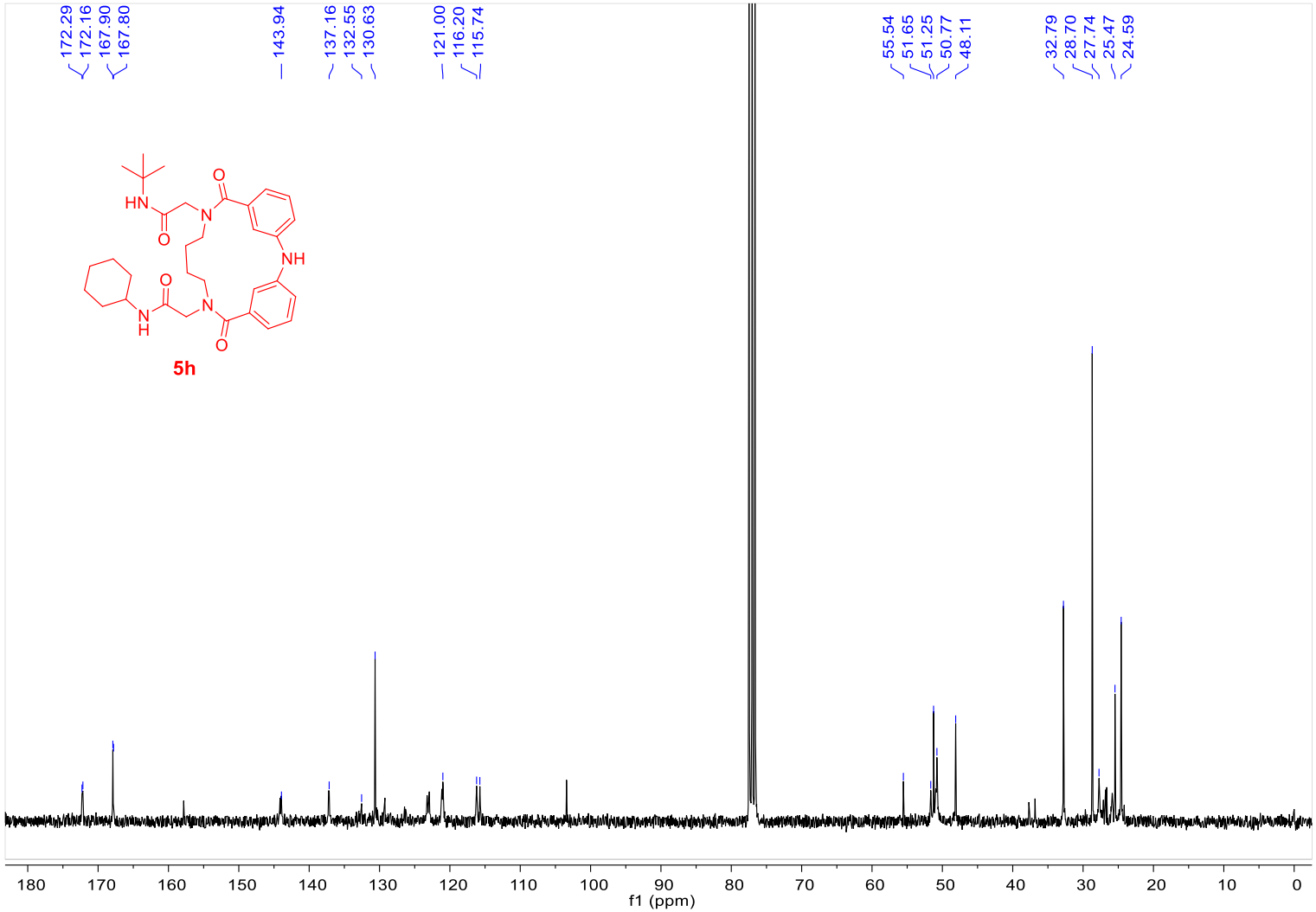


Figure 46. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(cyclohexylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5h**)

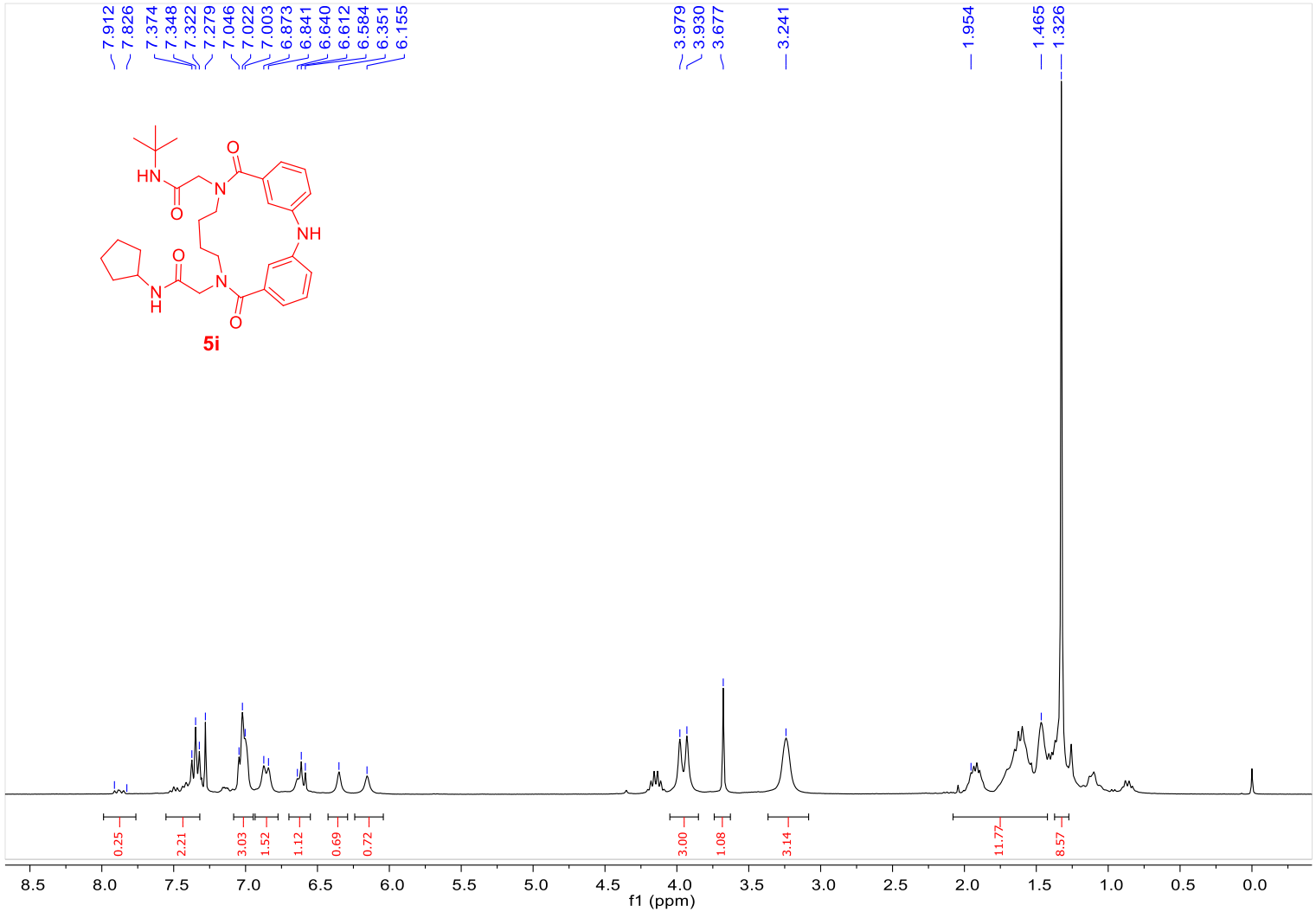


Figure 47. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(cyclopentylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5i**)

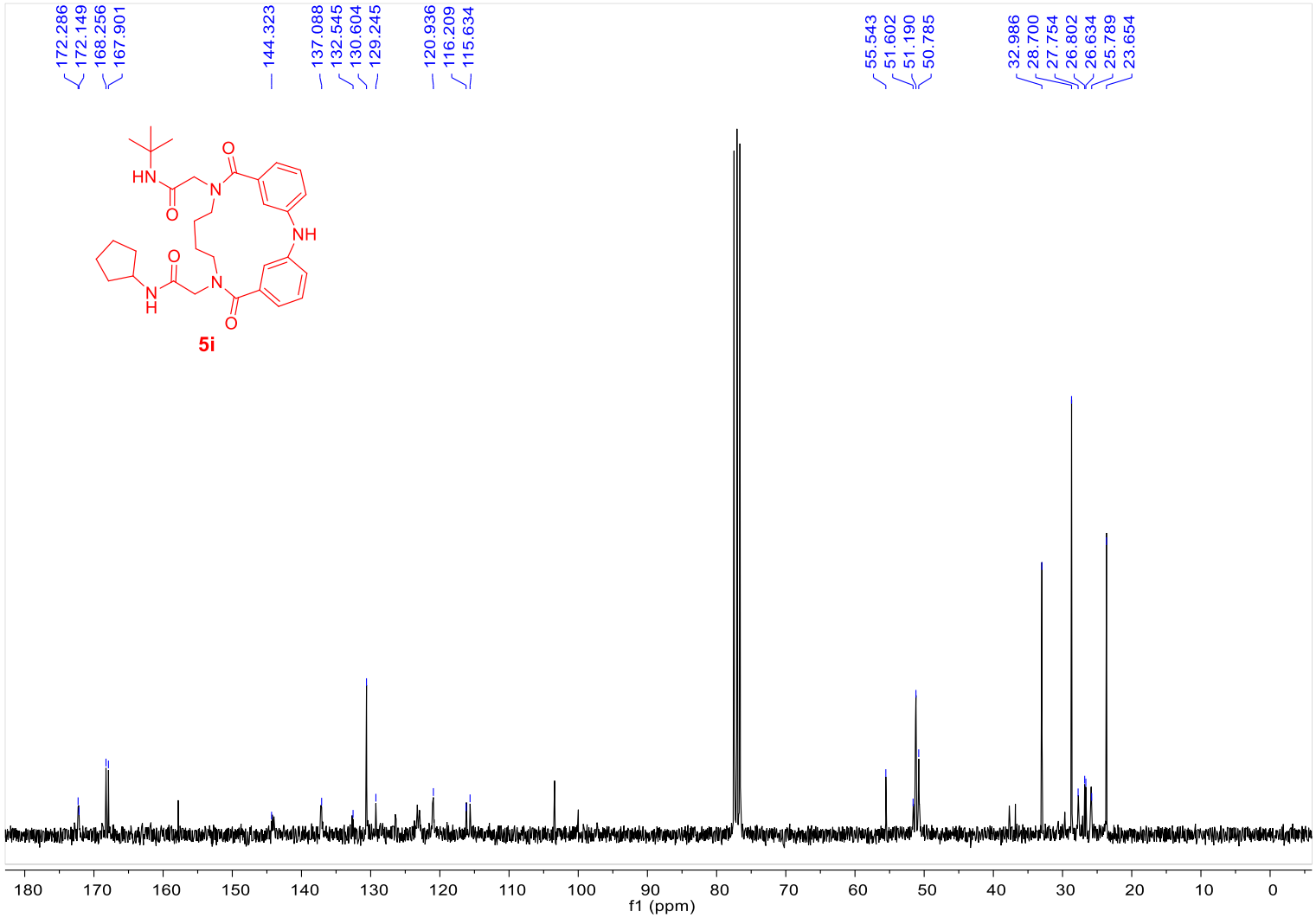


Figure 48. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-(cyclopentylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5i**)

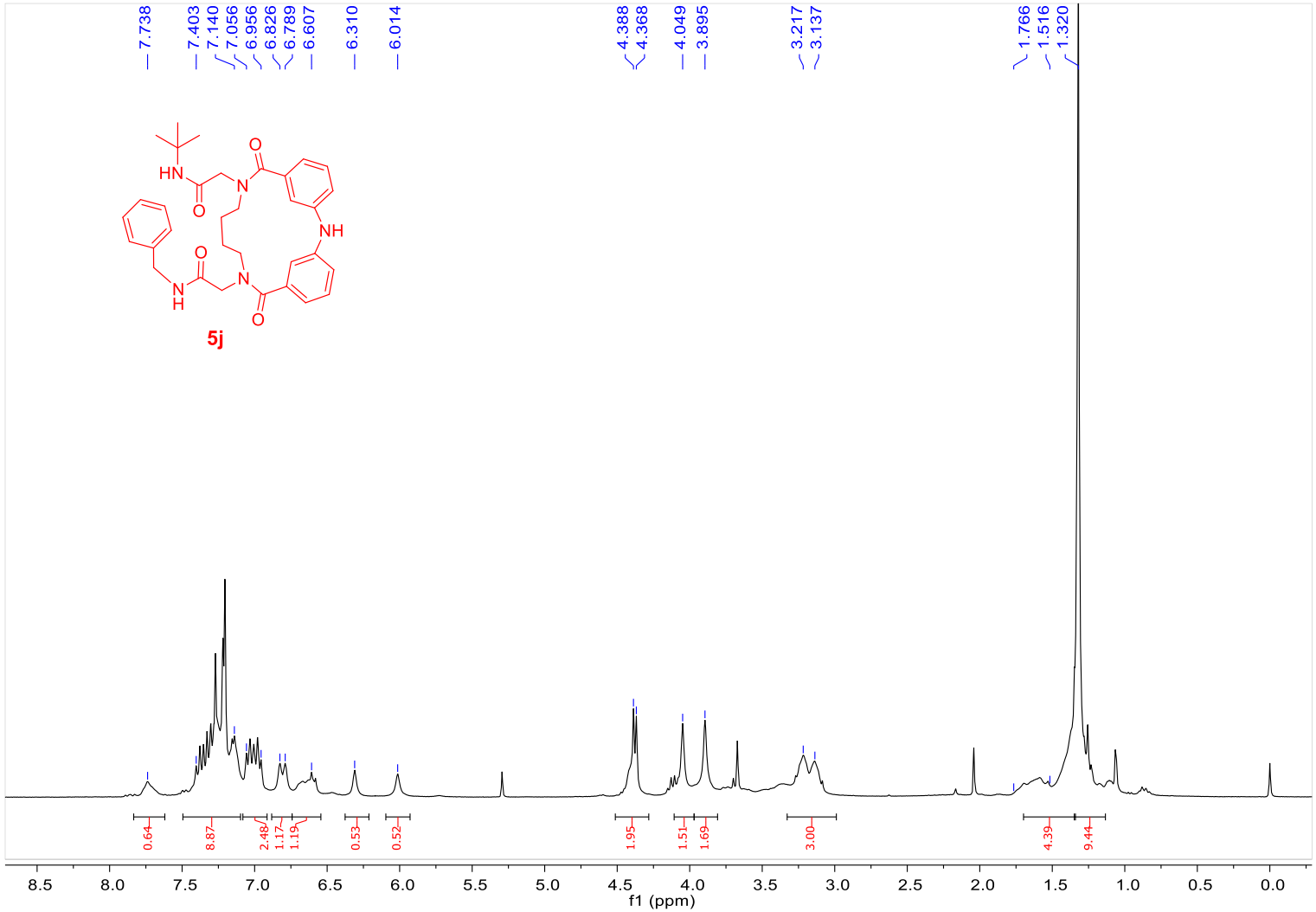


Figure 49. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of N-benzyl-2-(10-(2-(tert-butylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5j**)

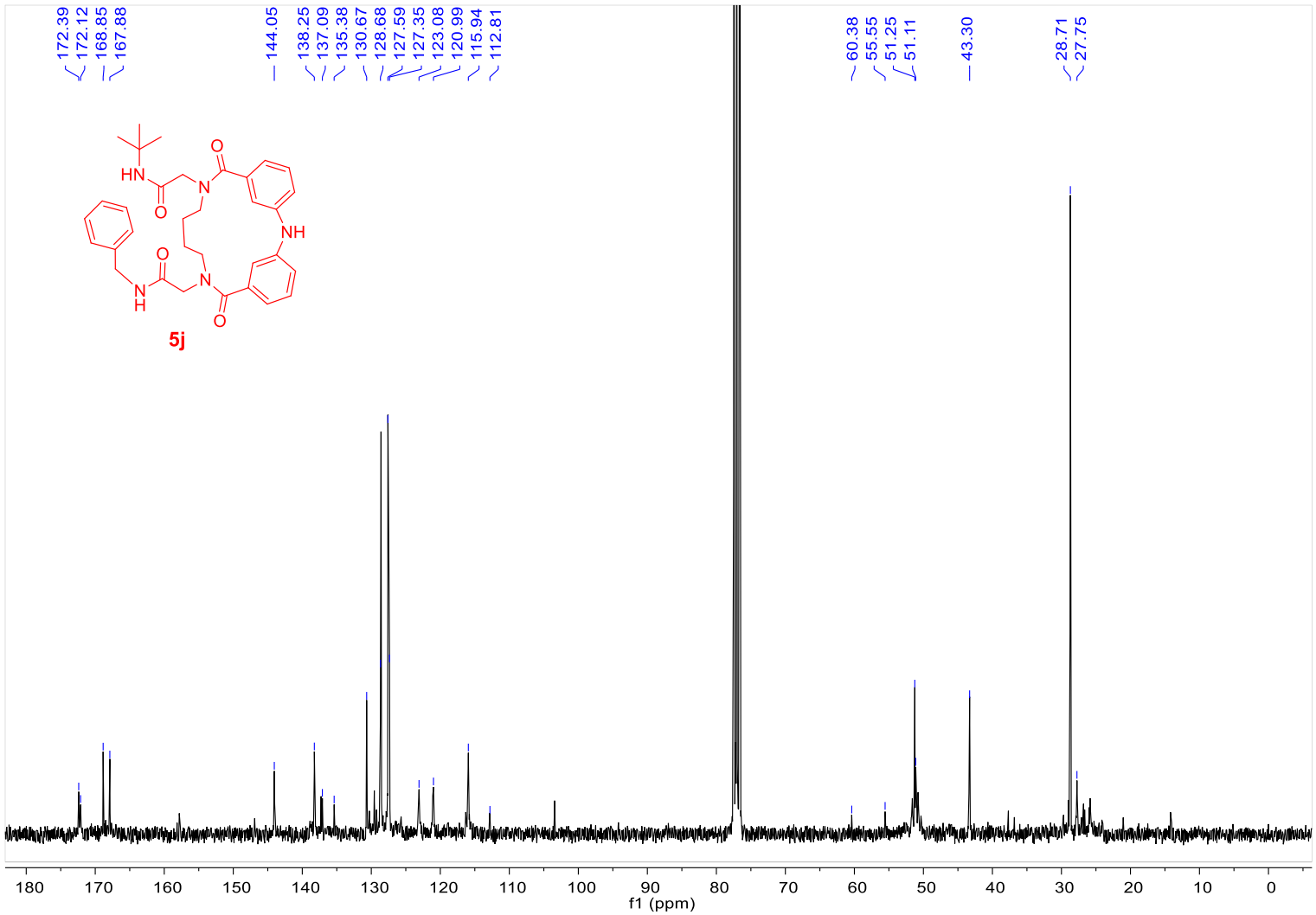


Figure 50. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of N-benzyl-2-(10-(2-(tert-butylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5j**)

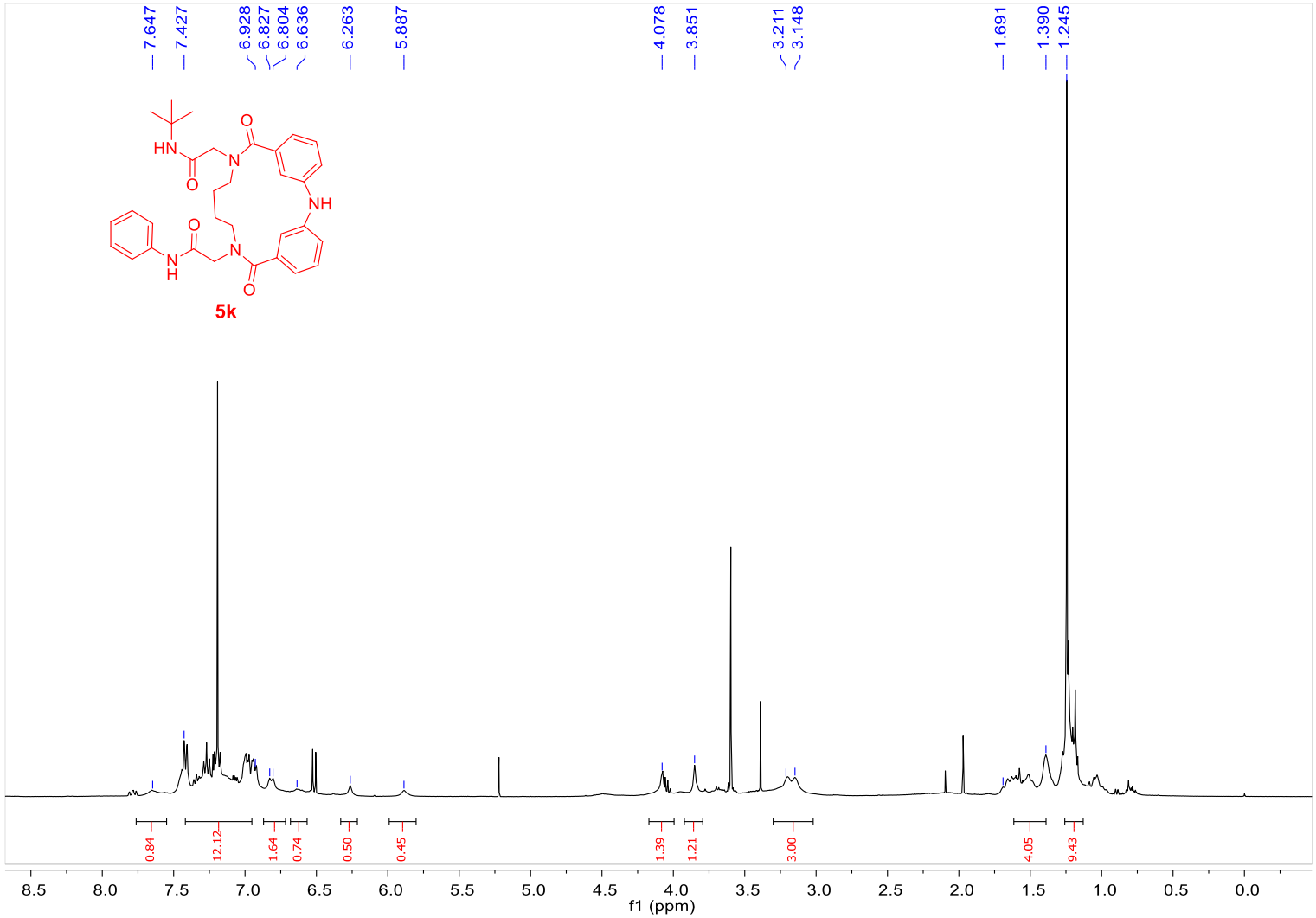


Figure 51. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(4,11-dioxo-10-(2-oxo-2-(phenylamino)ethyl)-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5k**)

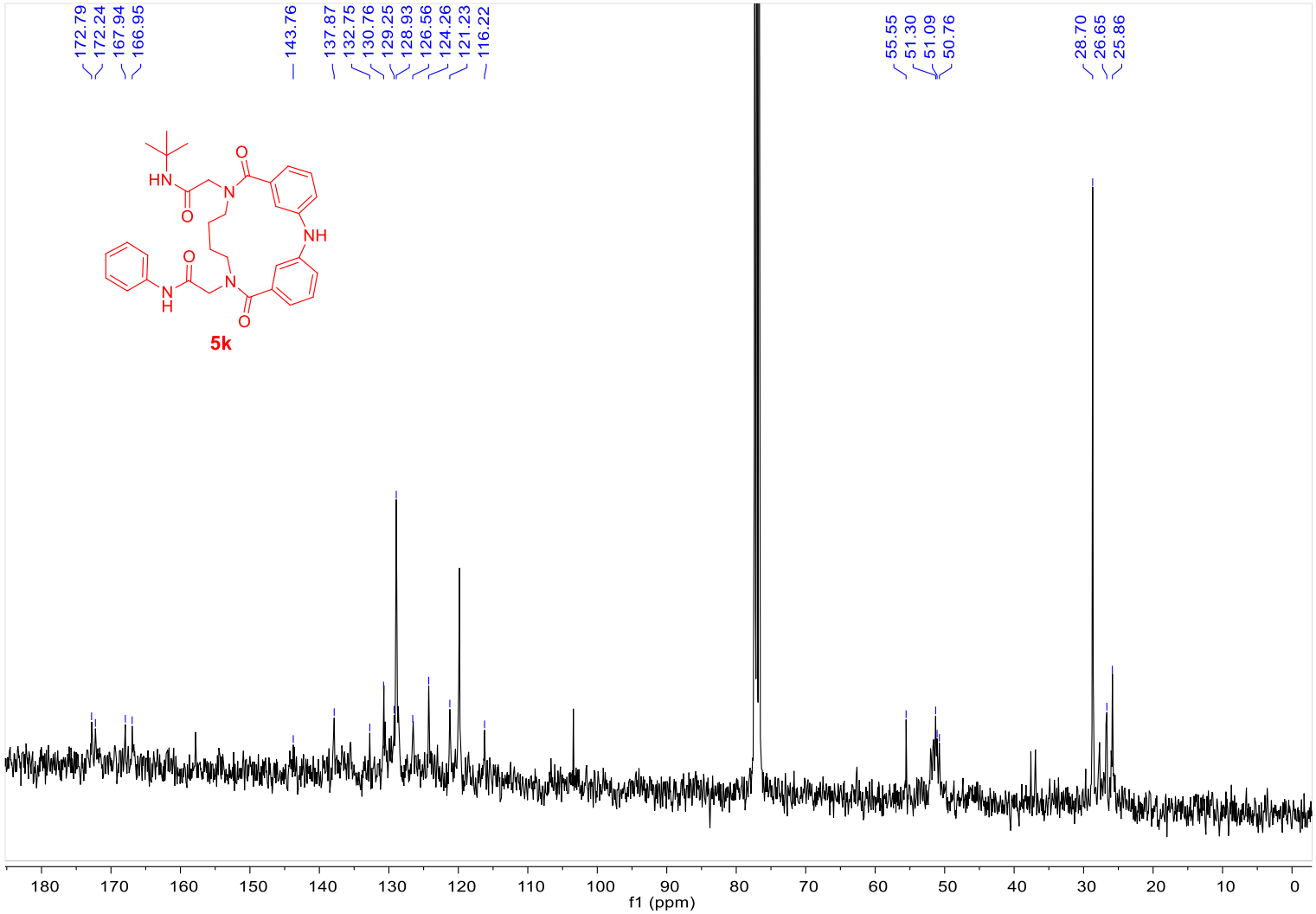


Figure 52. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(4,11-dioxo-10-(2-oxo-2-(phenylamino)ethyl)-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5k**)

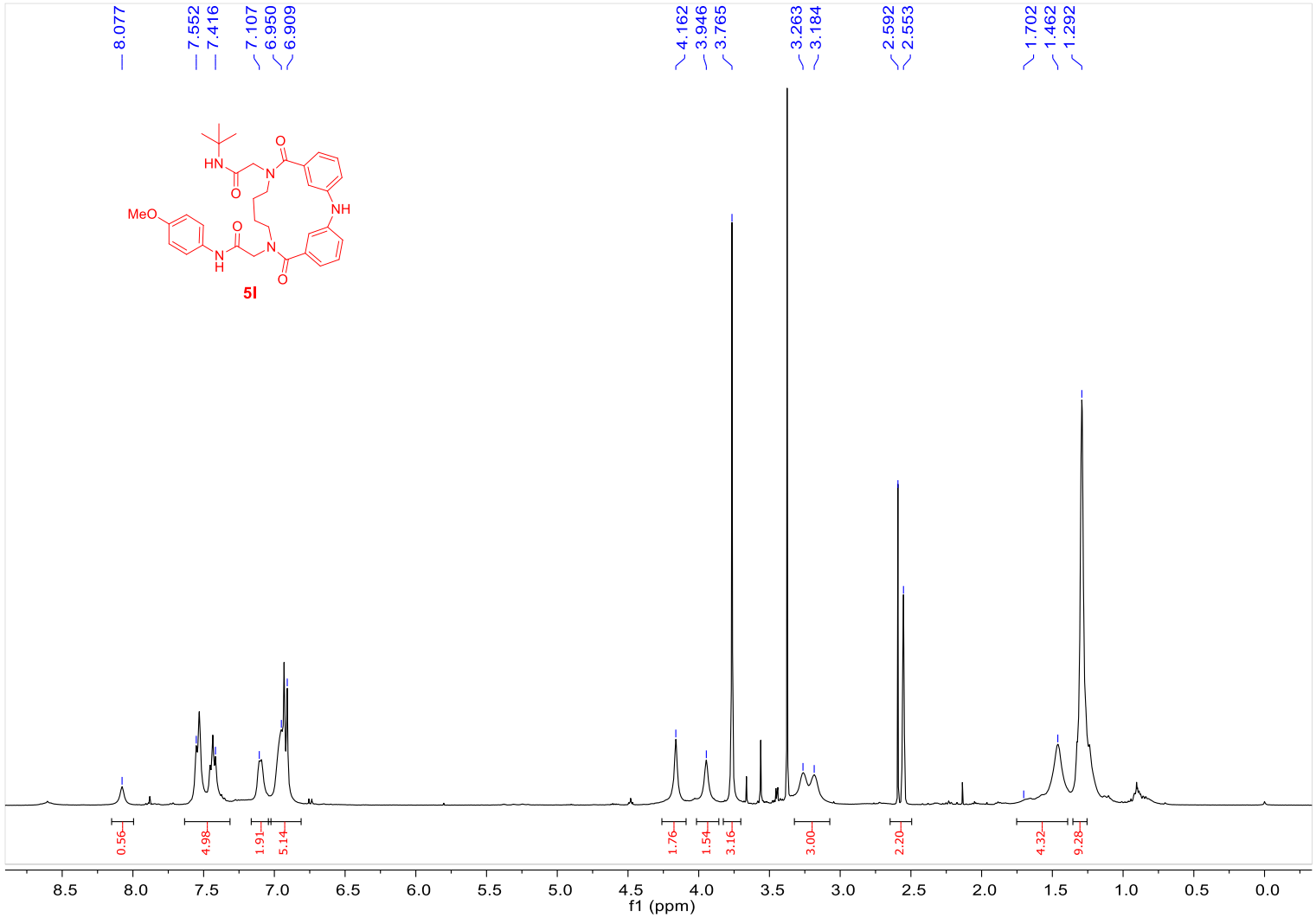


Figure 53. 400 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-((4-methoxyphenyl)amino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5l**)

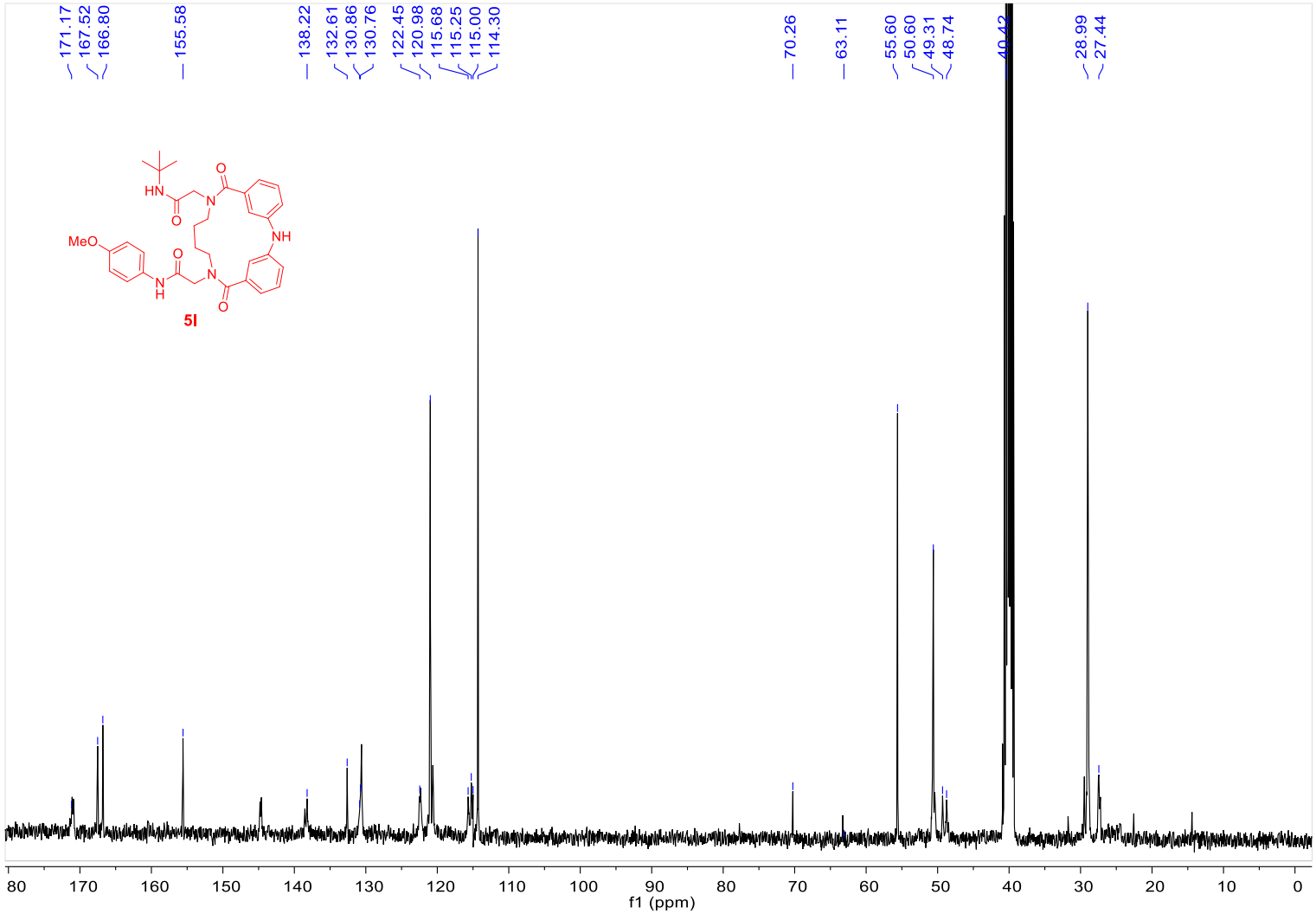


Figure 54. 100 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-((4-methoxyphenyl)amino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5l**)

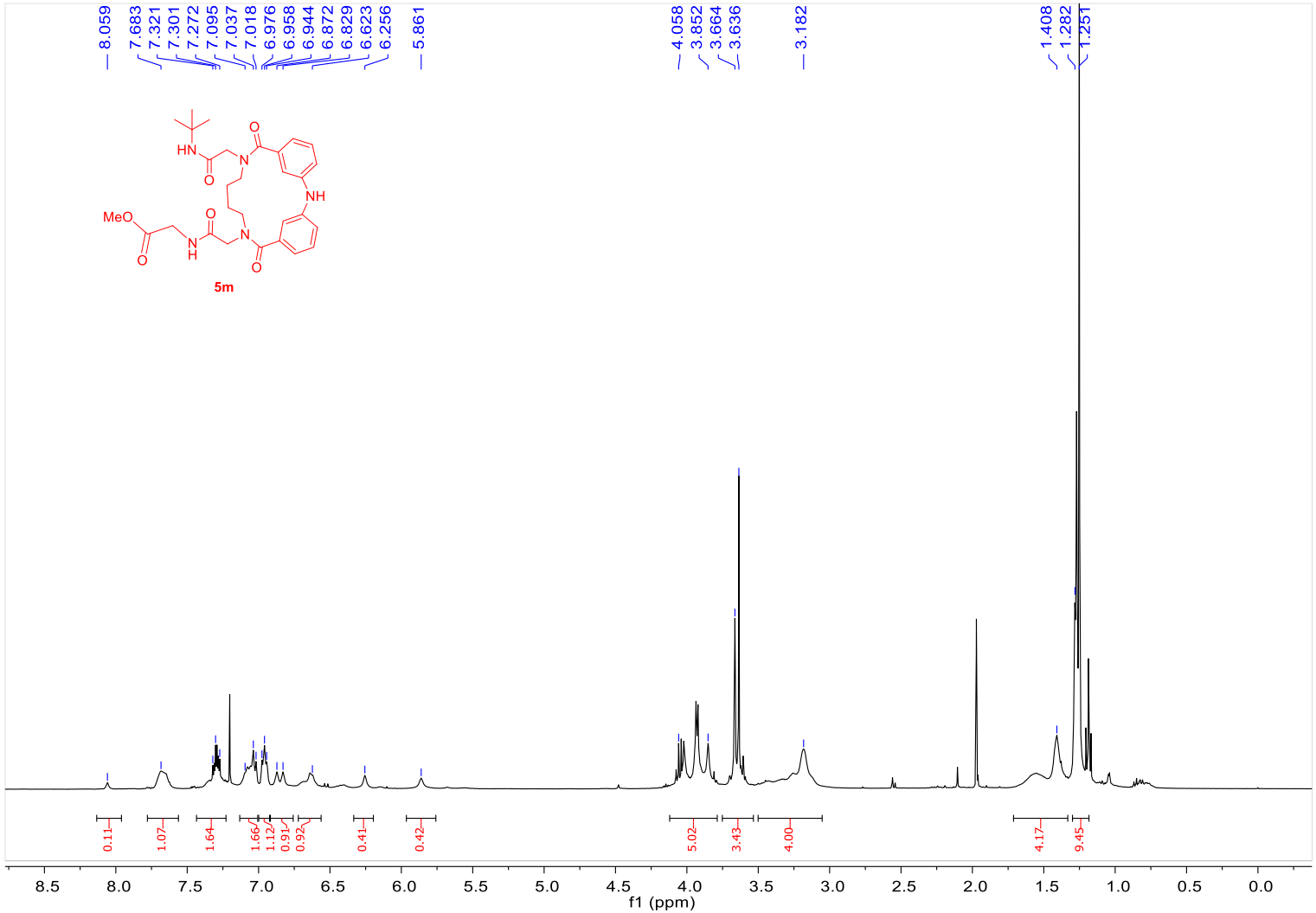


Figure 55. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of methyl(2-(10-(2-(tert-butylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetyl)glycinate (**5m**)

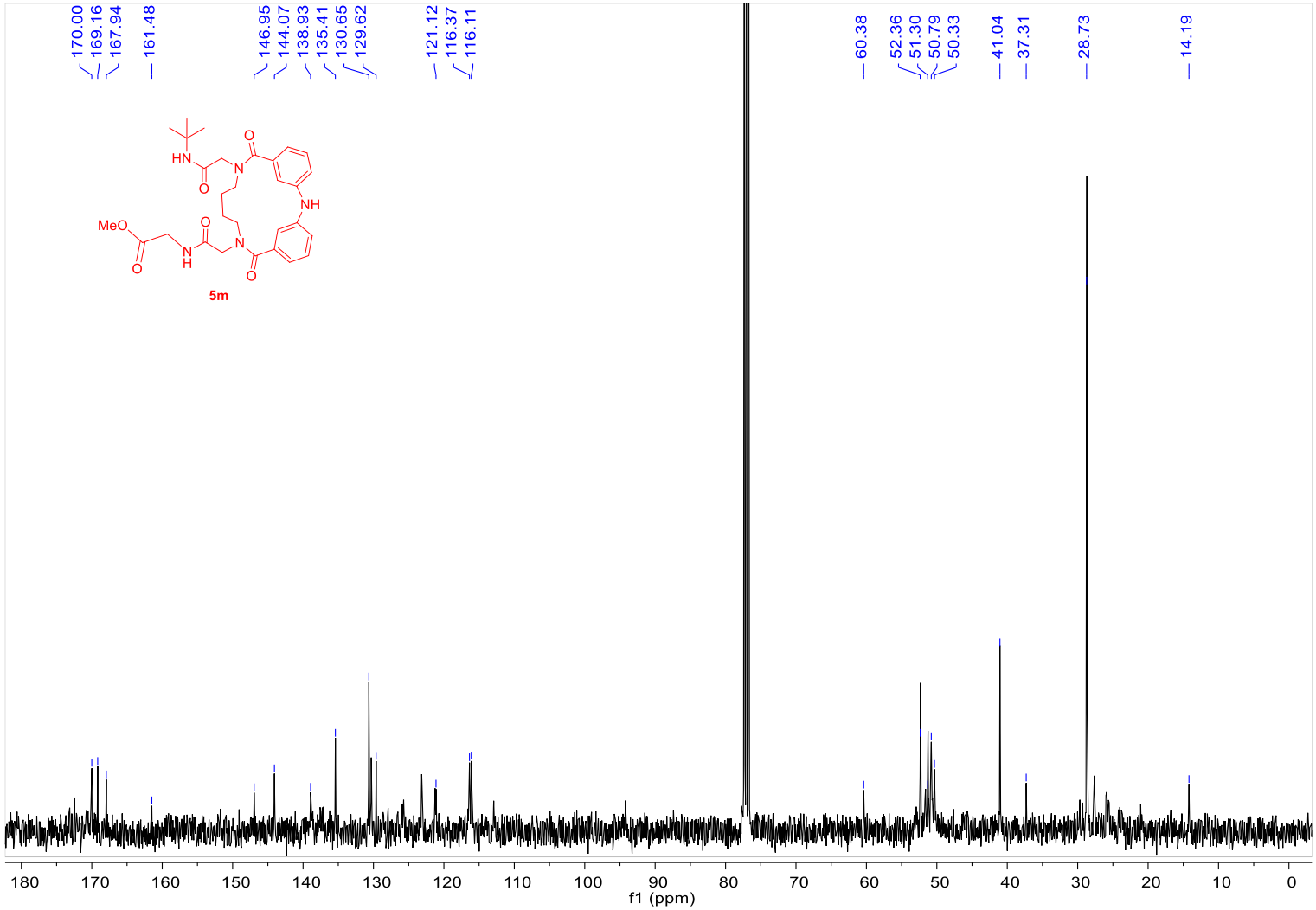


Figure 56. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of methyl(2-(10-(2-(tert-butylamino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetyl)glycinate (**5m**)

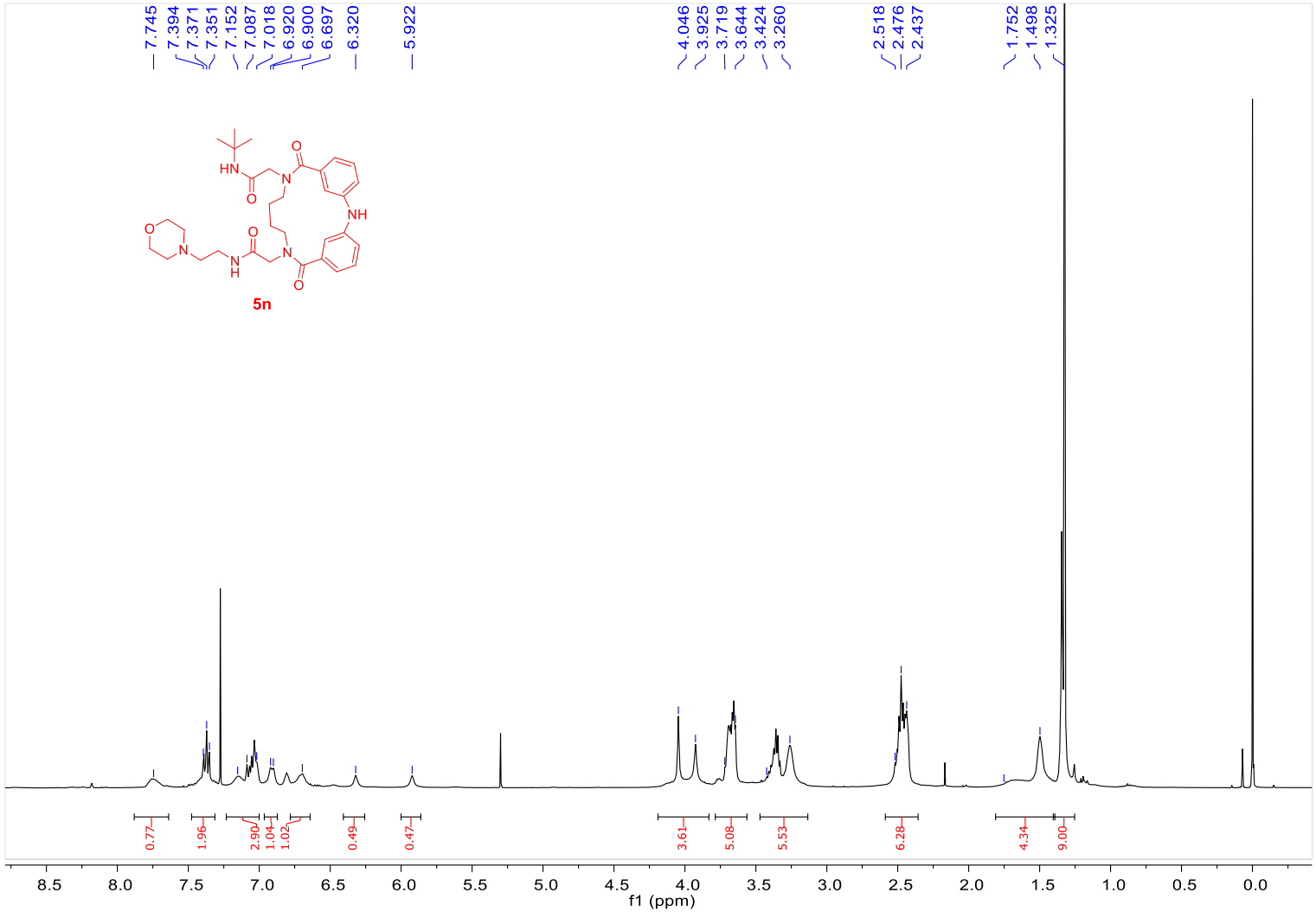


Figure 57. 300 MHz ^{1}H NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-((2-morpholinoethyl)amino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5n**)

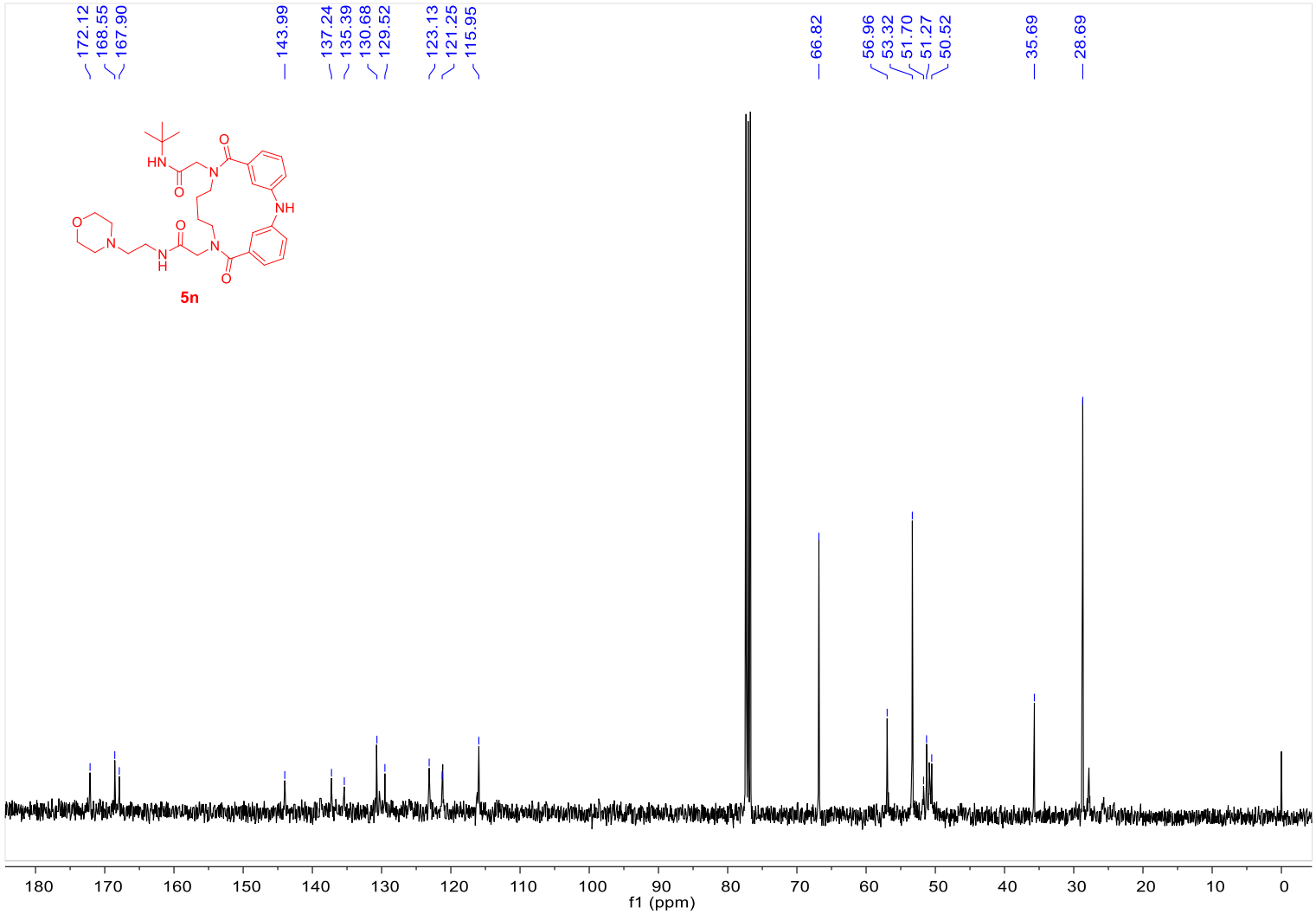
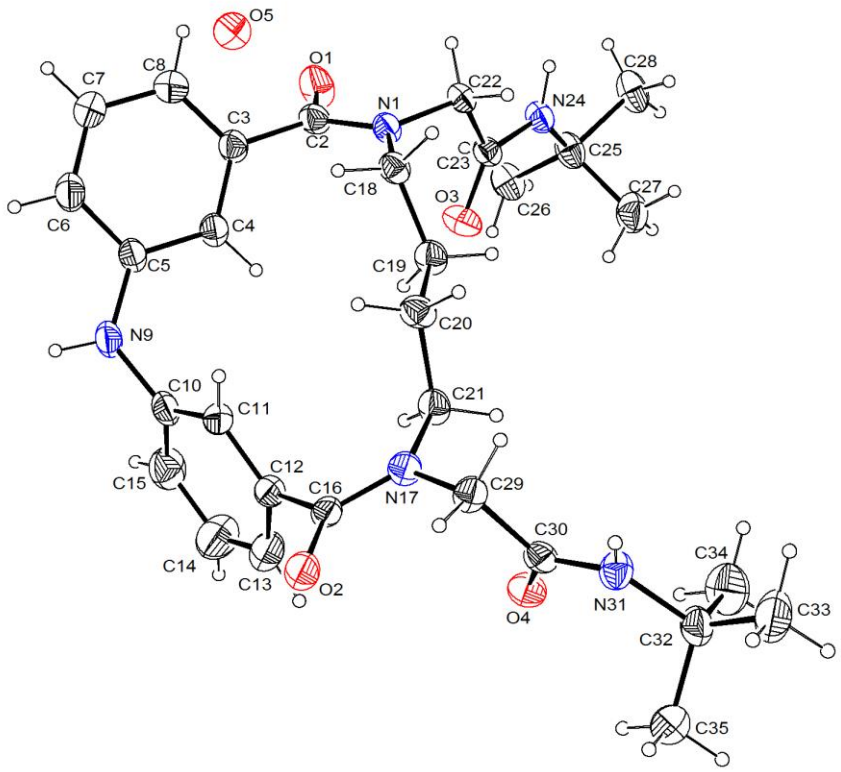


Figure 58. 75 MHz ^{13}C NMR spectra in $CDCl_3$ of N-(tert-butyl)-2-(10-(2-((2-morpholinoethyl)amino)-2-oxoethyl)-4,11-dioxo-2,5,10-triaza-1,3(1,3)-dibenzenacycloundecaphane-5-yl)acetamide (**5n**)

Crystal data and structure refinement for Macrocycle 5a.

Identification code	337MGL17	
Empirical formula	C_{120} H_{164} N_{20} O_{18}	
Formula weight	2174.70	
Temperature	298(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	Fdd2	
Unit cell dimensions	a = 27.0176(6) Å	α= 90°.
	b = 36.9735(8) Å	β= 90°.
	c = 14.7072(3) Å	γ = 90°.
Volume	14691.5(5) $Å^3$	
Z	4	
Density (calculated)	0.983 Mg/m^3	
Absorption coefficient	0.540 mm^{-1}	
F(000)	4672	
Crystal size	0.330 x 0.289 x 0.179 mm^3	
Theta range for data collection	3.624 to 68.386°.	
Index ranges	-32<=h<=32, -40<=k<=44, -16<=l<=17	
Reflections collected	27876	
Independent reflections	6518 [R(int) = 0.0443]	
Completeness to theta = 67.679°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7531 and 0.6681	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6518 / 70 / 414	
Goodness-of-fit on F^2	1.123	
Final R indices [I>2sigma(I)]	R1 = 0.0701, wR2 = 0.1767	
R indices (all data)	R1 = 0.0866, wR2 = 0.1927	
Absolute structure parameter	0.15(11)	
Largest diff. peak and hole	0.430 and -0.172 e.$Å^{-3}$	


O5
O1
C8
C7
C3
C2
N1
C22
N24
C28
C25
C23
C18
O3
C26
C27
C6
C4
C5
C19
C20
N9
C10
C11
C21
C15
C12
C16
N17
C29
C30
C14
C13
O2
O4
N31
C34
C33
C32
C35