SUPPORTING INFORMATION

“Canopy Catalysts” for Alkyne Metathesis: Molybdenum Alkylidyne Complexes with a Tripodal Ligand Framework

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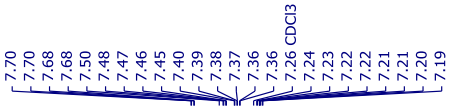
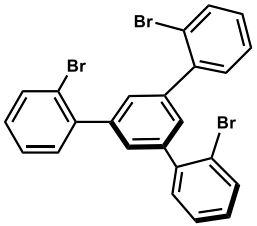
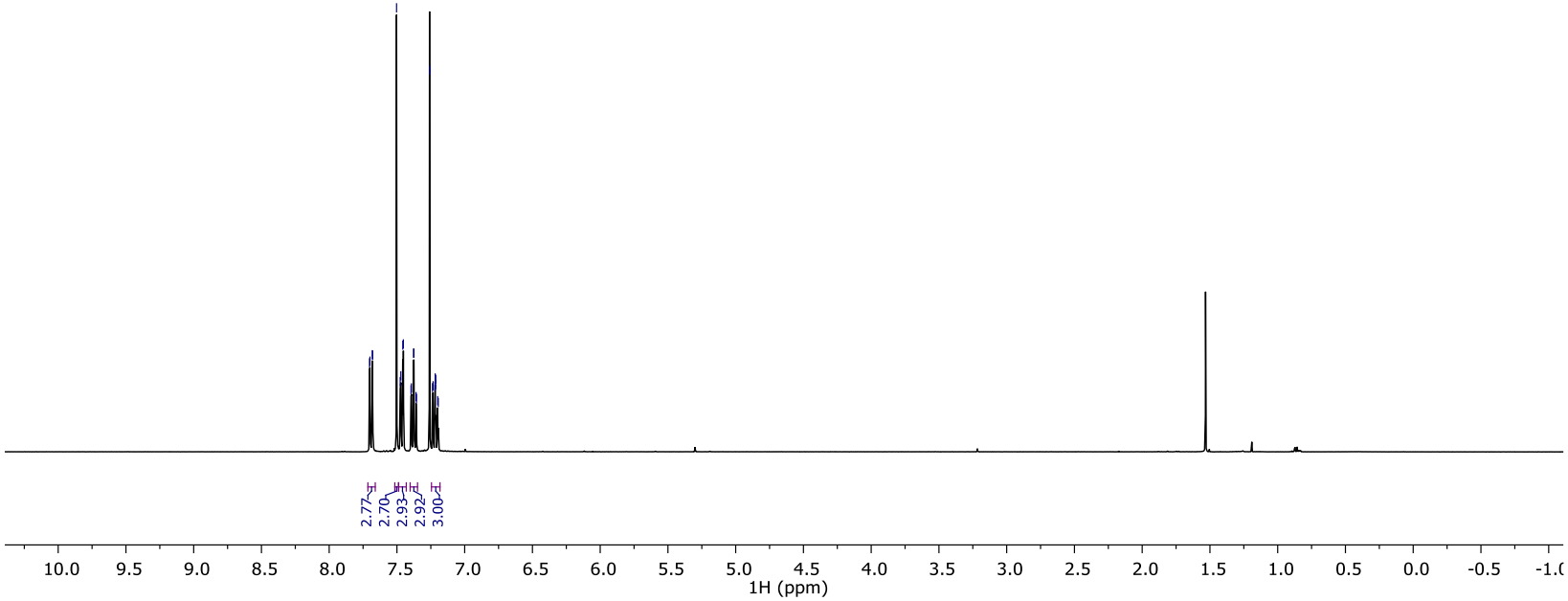
COPIES OF SPECTRA

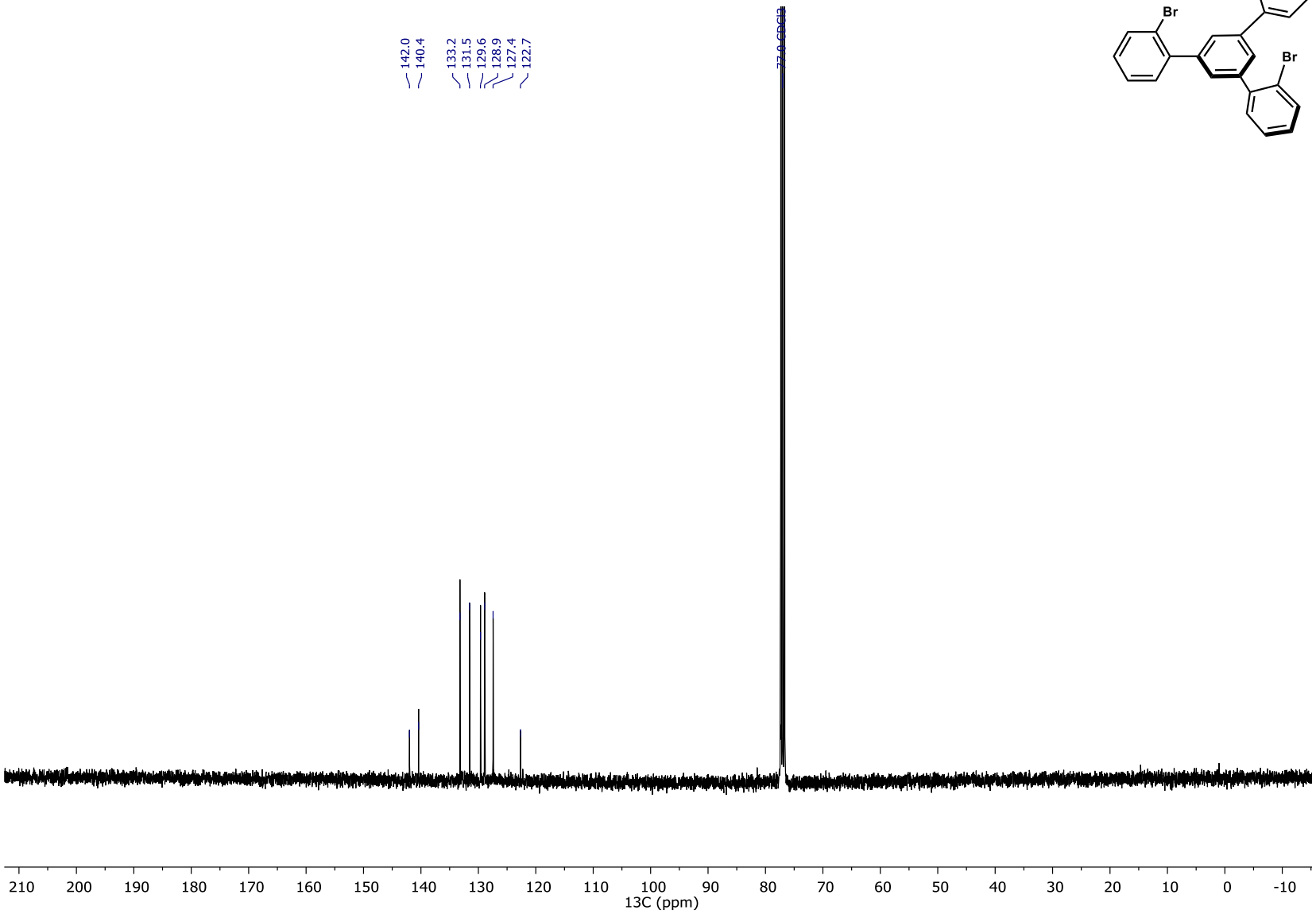
^{1}H NMR of 1,3,4-Tris-2'-bromophenylbenzene (8a), 400 MHz, $CDCl_3$, 25ºC



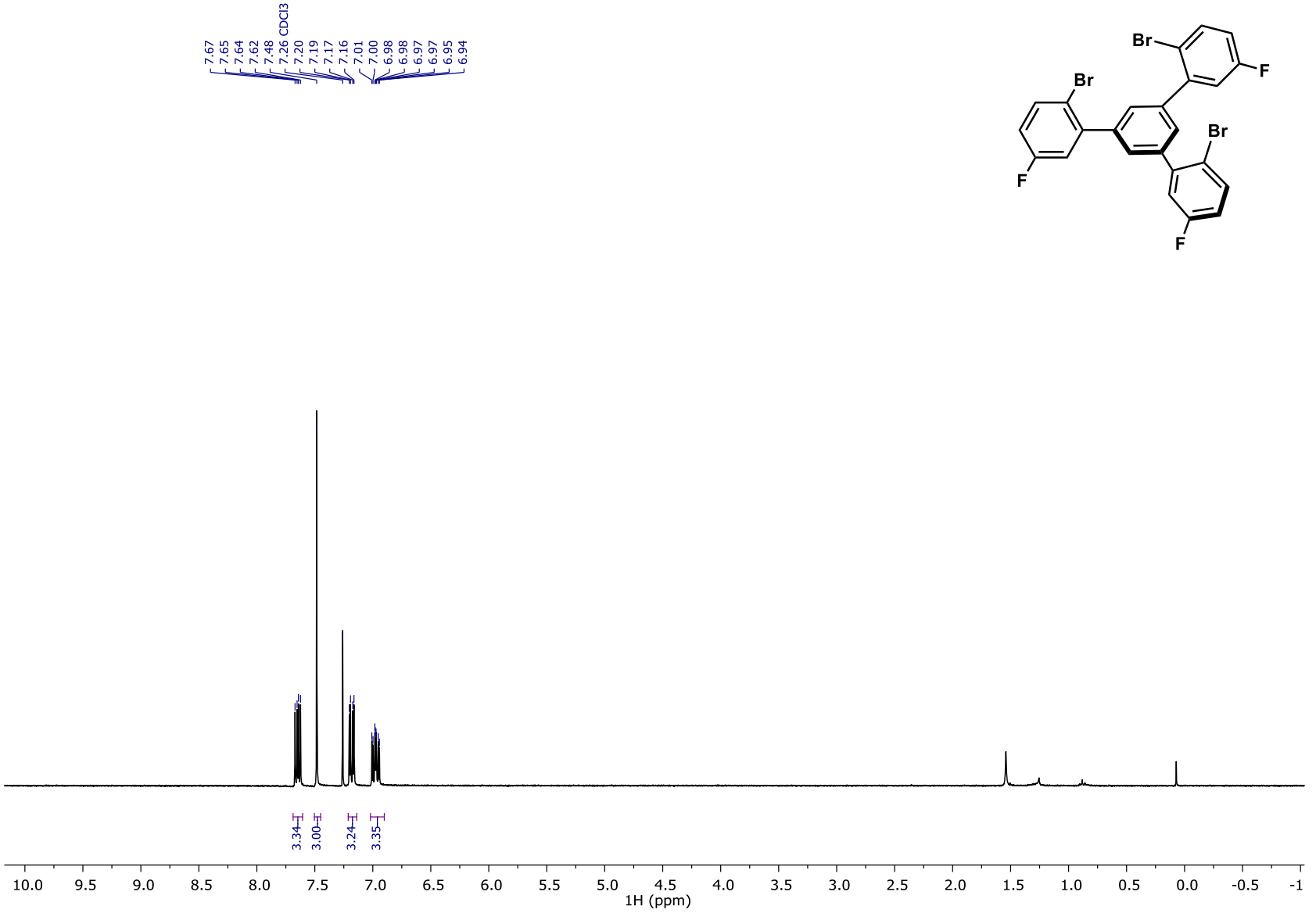




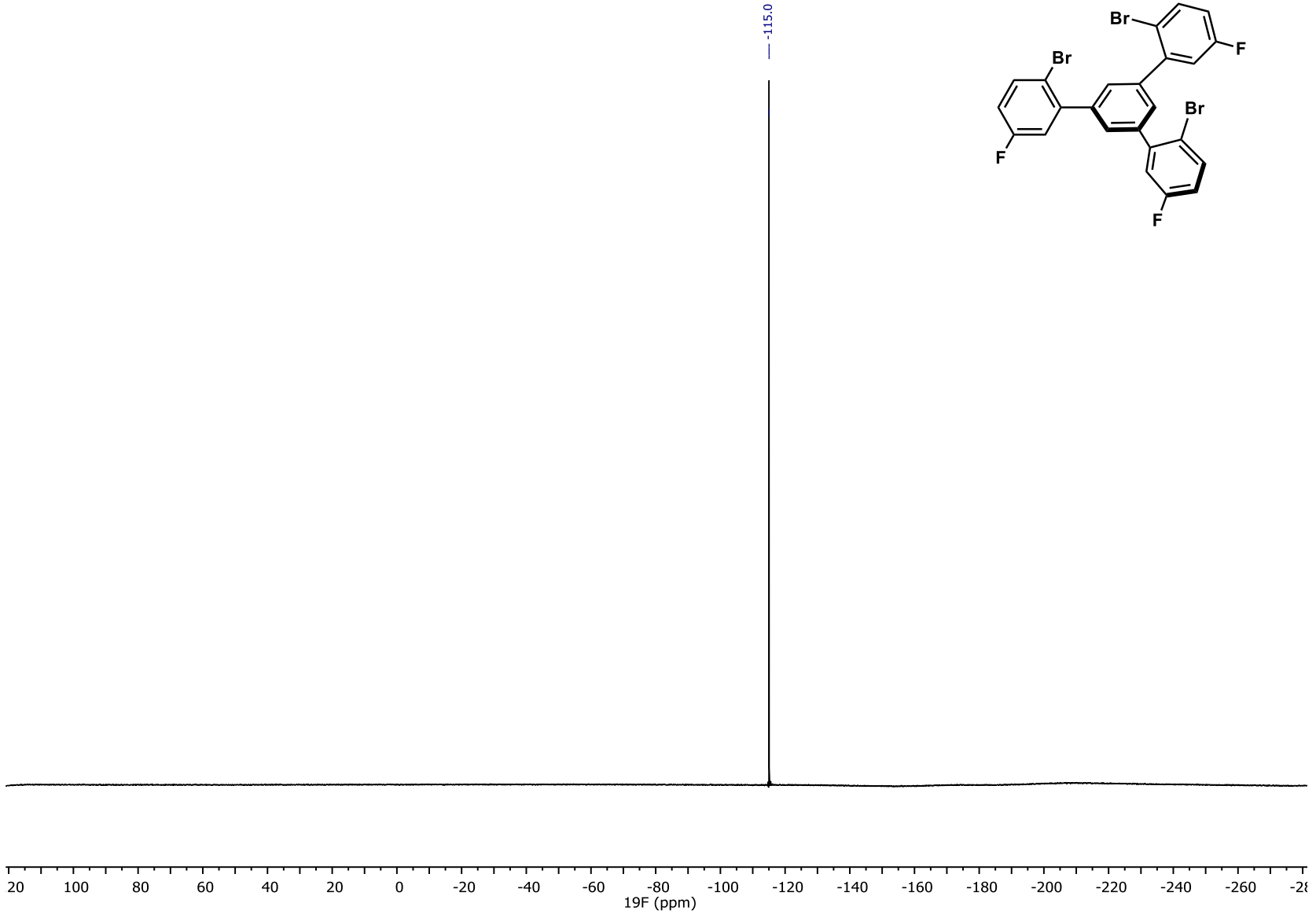
^{13}C NMR of 1,3,4-Tris-2'-bromophenylbenzene (8a), 101 MHz, $CDCl_3$, 25ºC



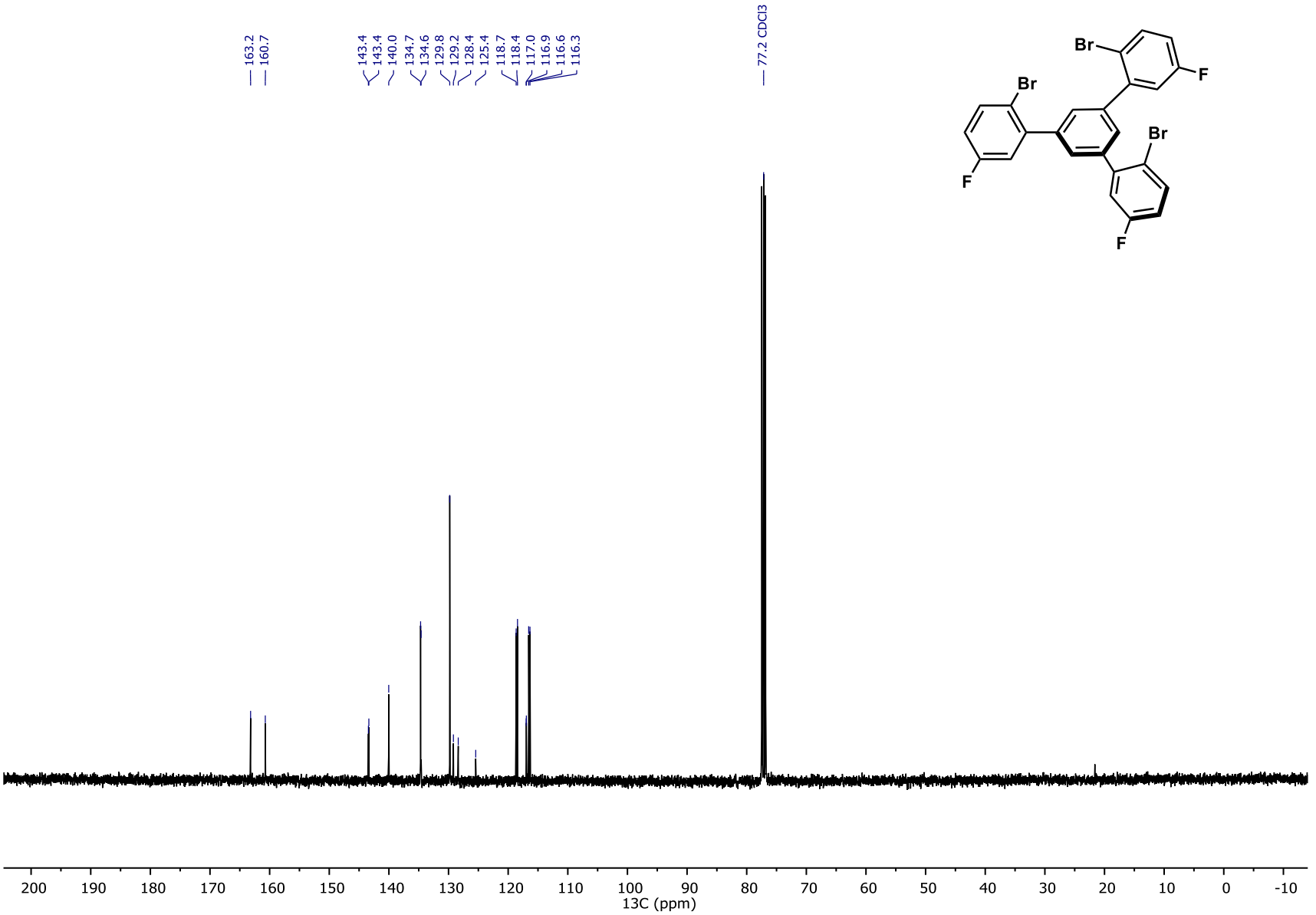
^{1}H NMR of 2,2''-Dibromo-5'-(2-bromo-5-fluorophenyl)-5,5''-difluoro-1,1':3',1''-terphenyl (8b)[6], 300 MHz, $CDCl_3$, 25ºC



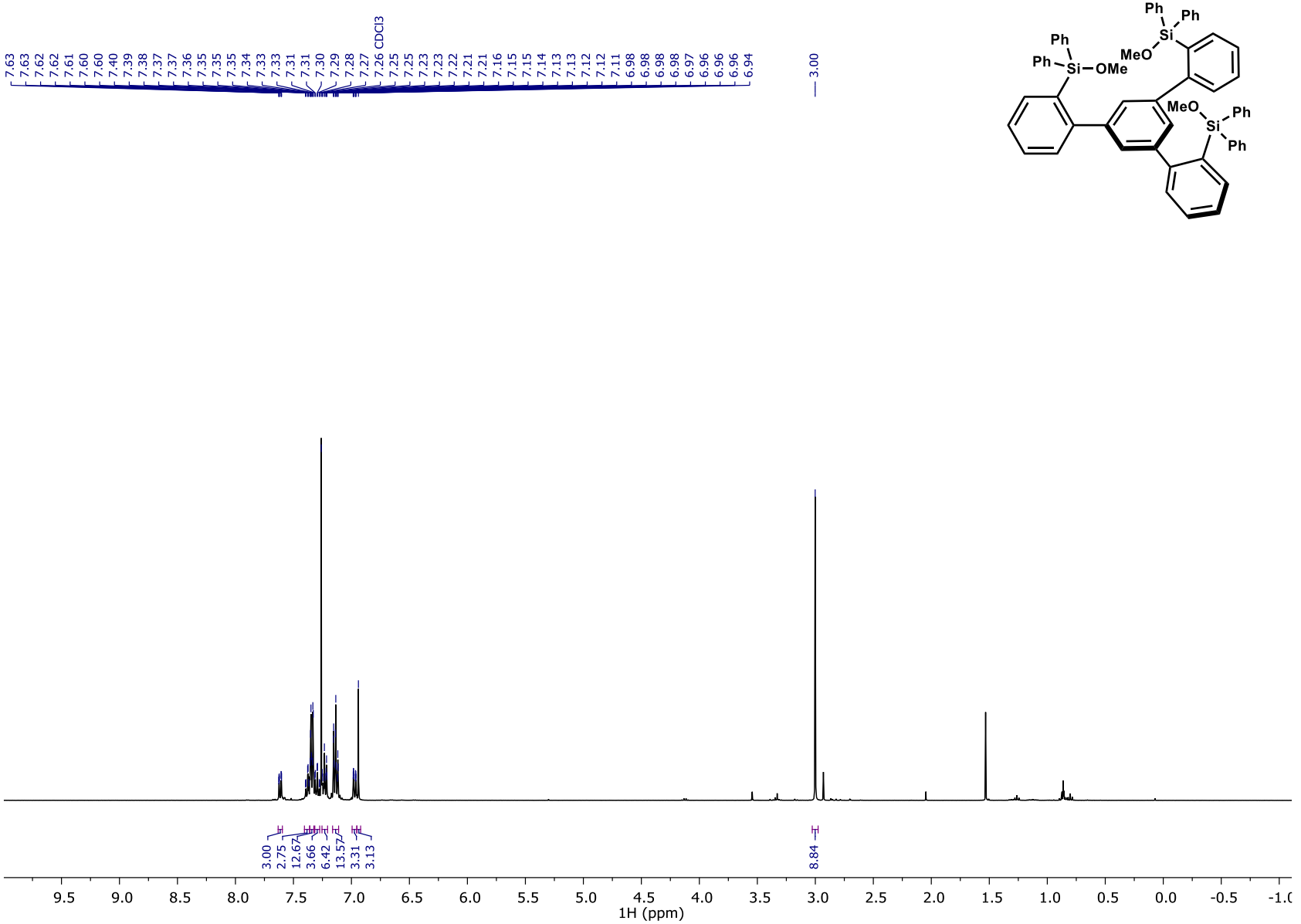
^{19}F NMR of 2,2''-Dibromo-5'-(2-bromo-5-fluorophenyl)-5,5''-difluoro-1,1':3',1''-terphenyl (8b)[6], 282 MHz, $CDCl_3$, 25ºC



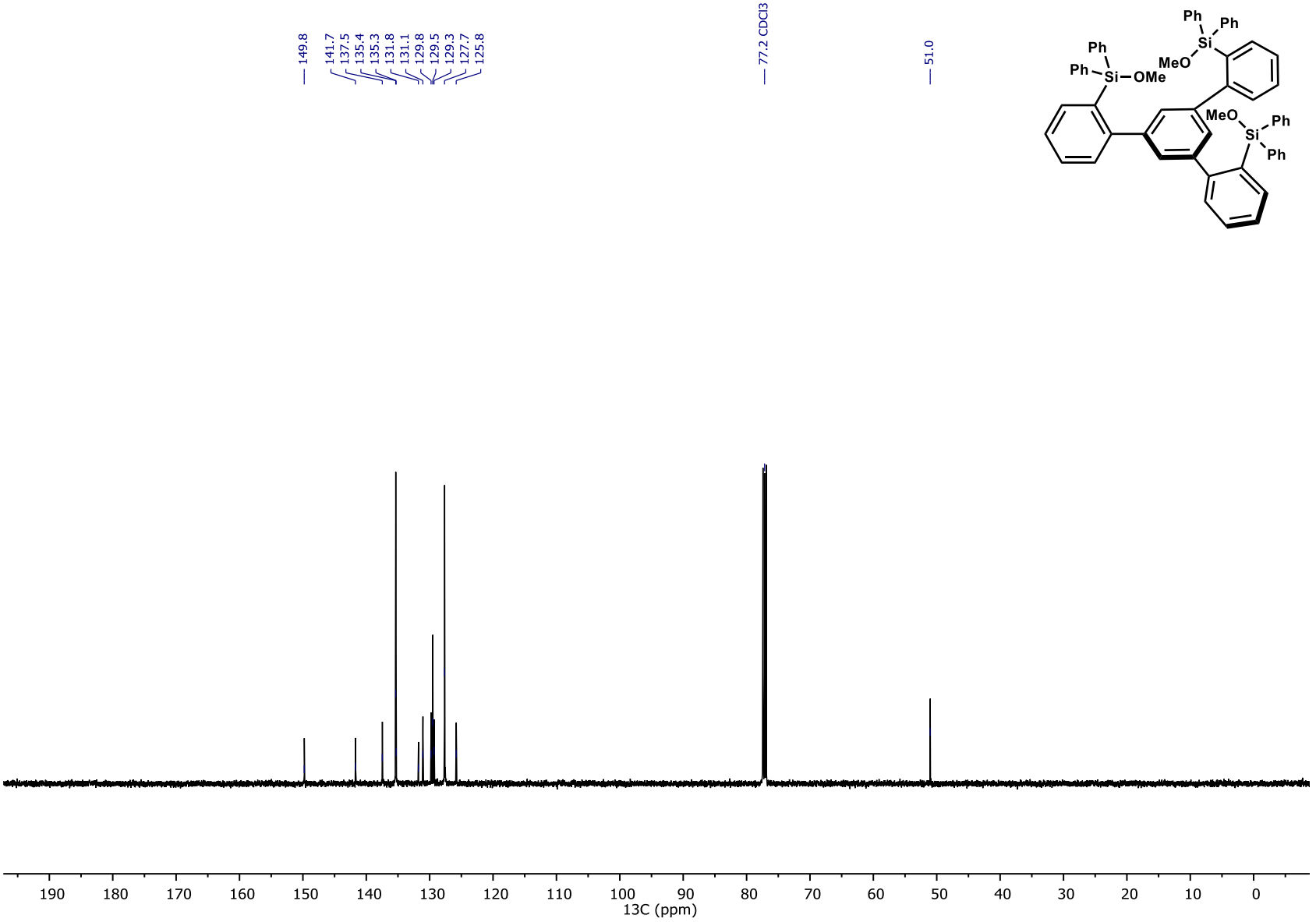
^{13}C NMR of 2,2''-Dibromo-5'-(2-bromo-5-fluorophenyl)-5,5''-difluoro-1,1':3',1''-terphenyl (8b)[6], 101 MHz, $CDCl_3$, 25ºC



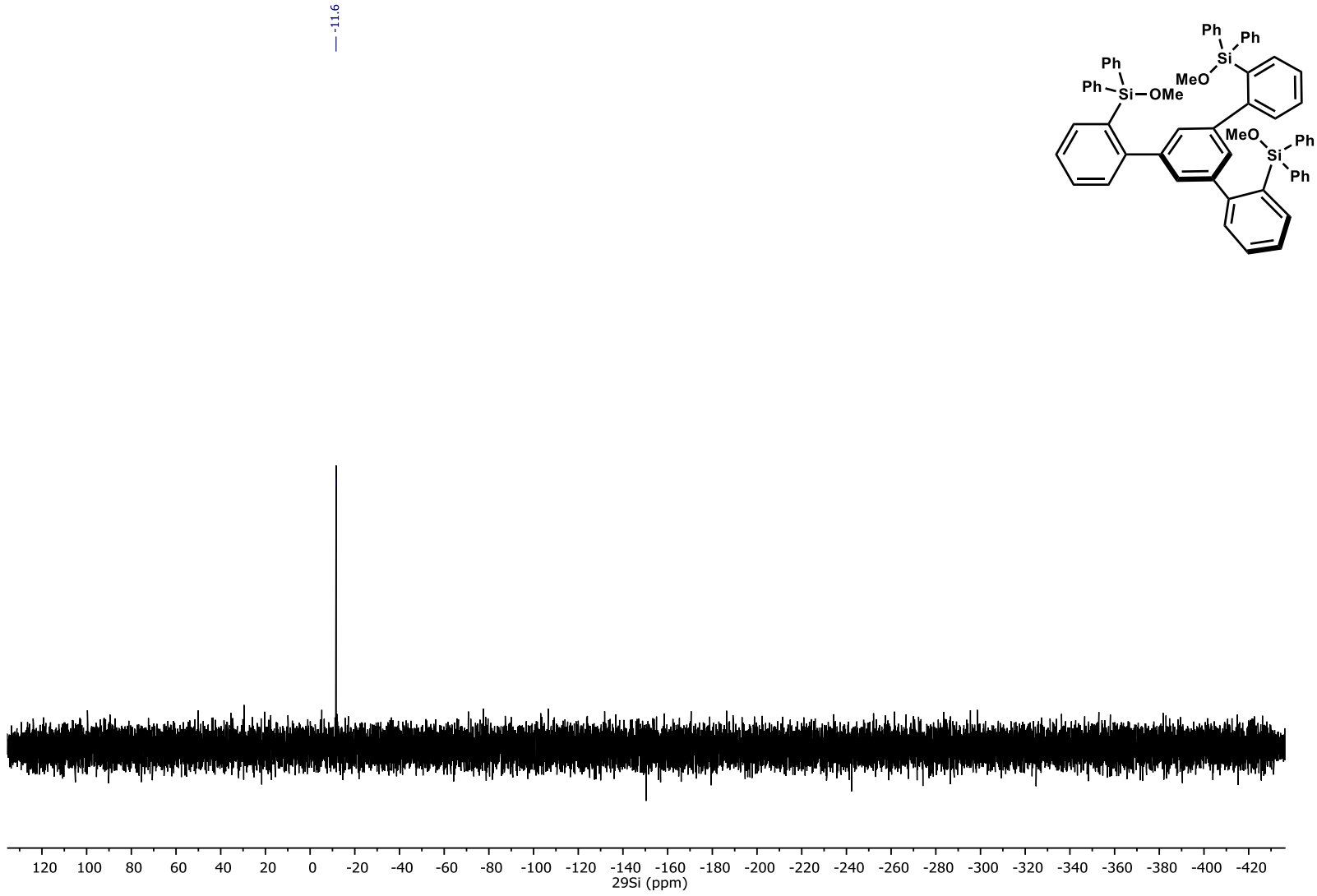
H NMR of (5'-(2-(Methoxydiphenylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxydiphenylsilane) (9a), 500 MHz**,** $CDCl_3$, 25ºC



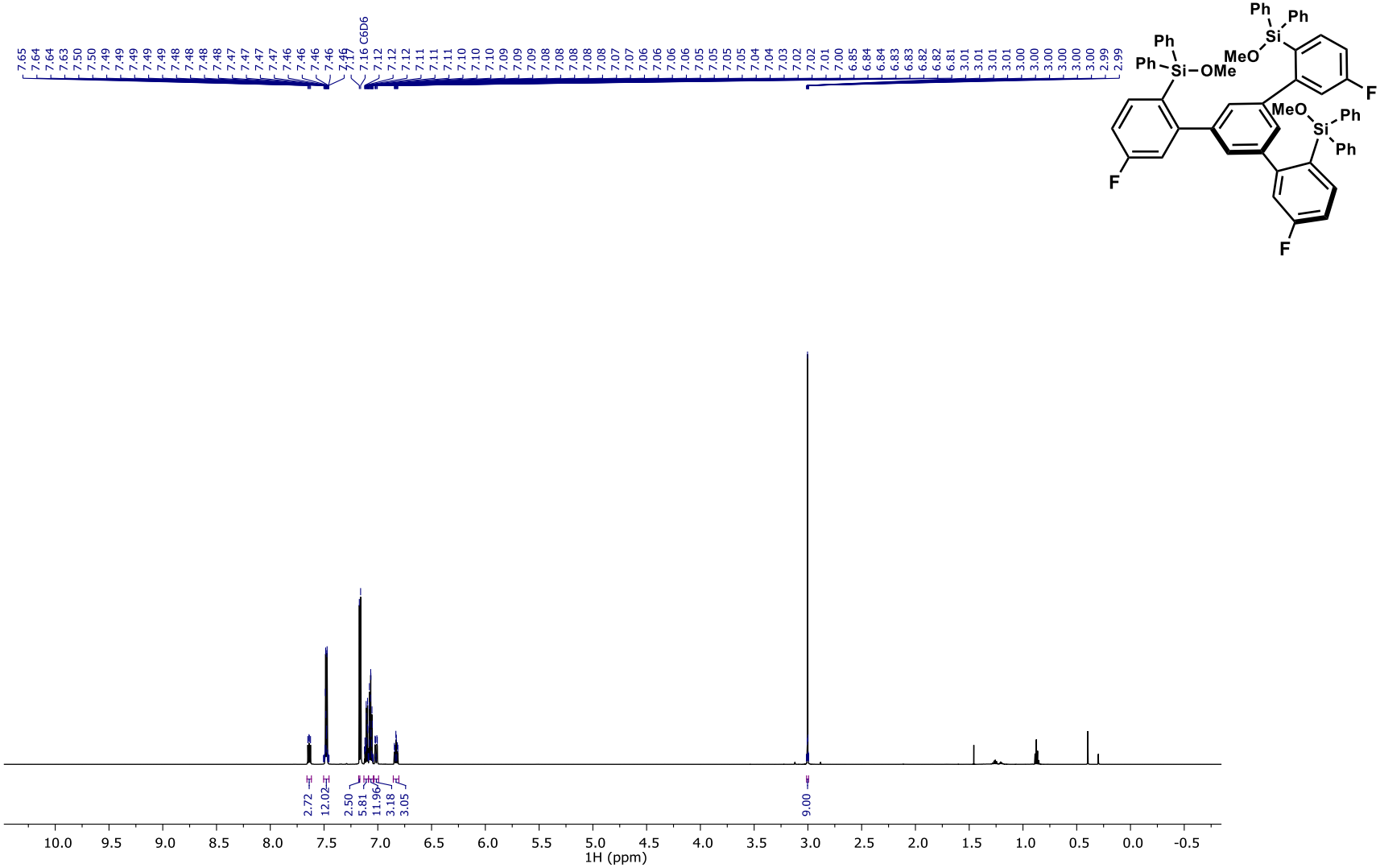
^{13}C NMR of (5'-(2-(Methoxydiphenylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxydiphenylsilane) (9a), 126 MHz, $CDCl_3$, 25ºC



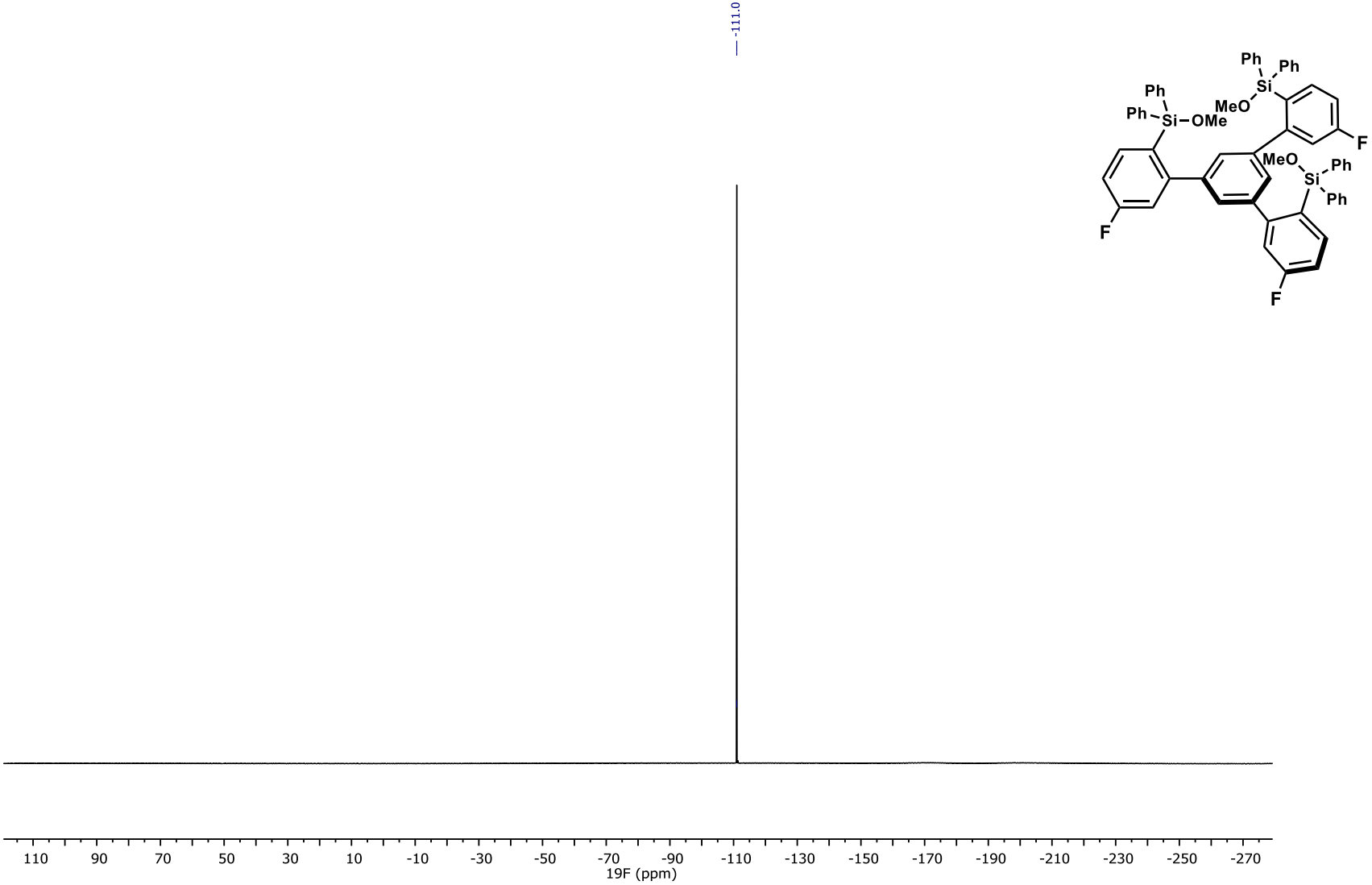
^{29}Si NMR of (5'-(2-(Methoxydiphenylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxydiphenylsilane) (9a), 99 MHz, $CDCl_3$, 25ºC



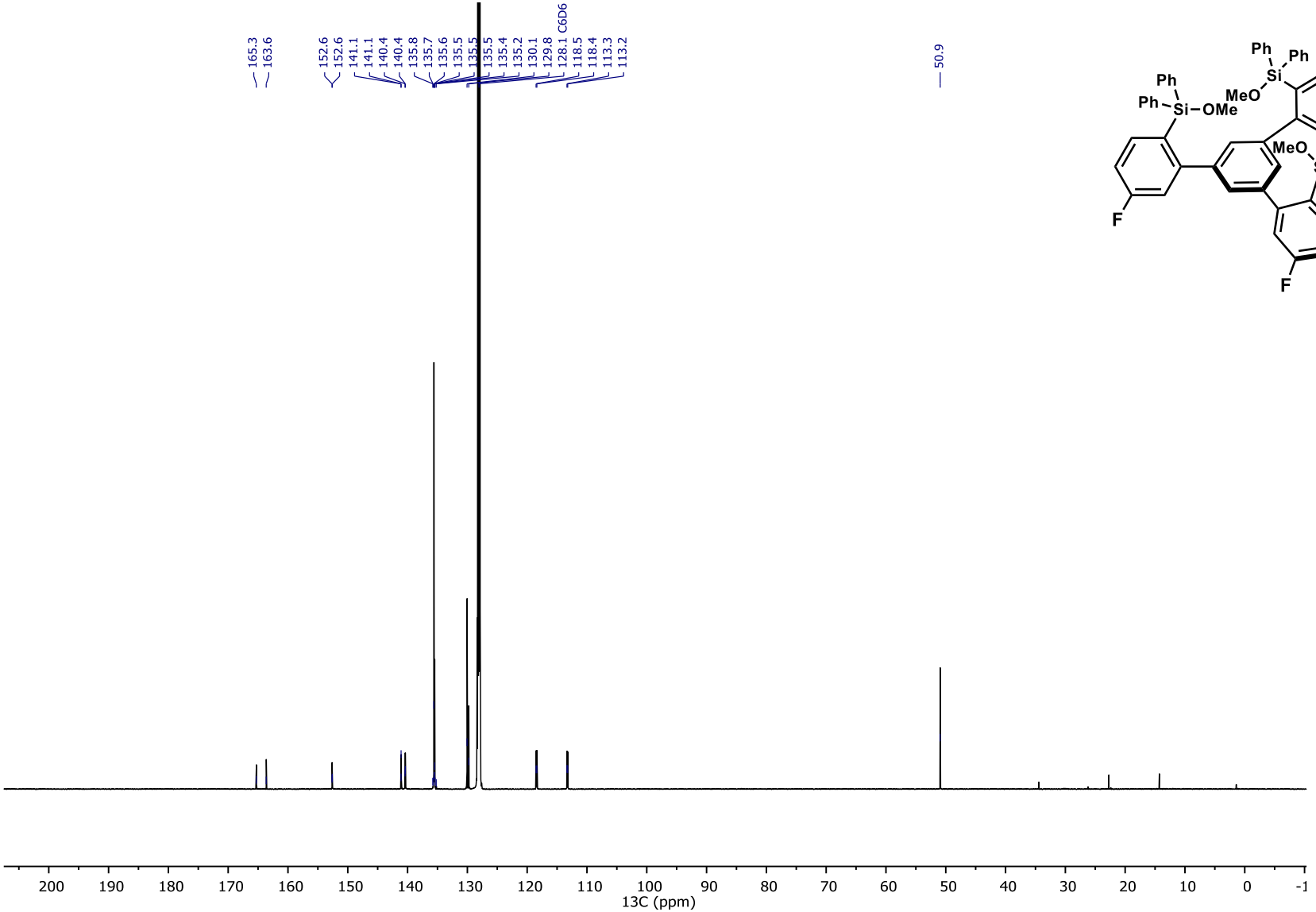
^{1}H NMR of (5,5''-Difluoro-5'-(5-fluoro-2-(methoxydiphenylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxydiphenylsilane) (9b), 500 MHz, C_6D_6, 25ºC



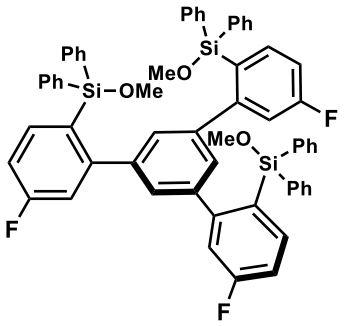
^{19}F NMR of (5,5''-Difluoro-5'-(5-fluoro-2-(methoxydiphenylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxydiphenylsilane) (9b), 470 MHz, C_6D_6, 25ºC



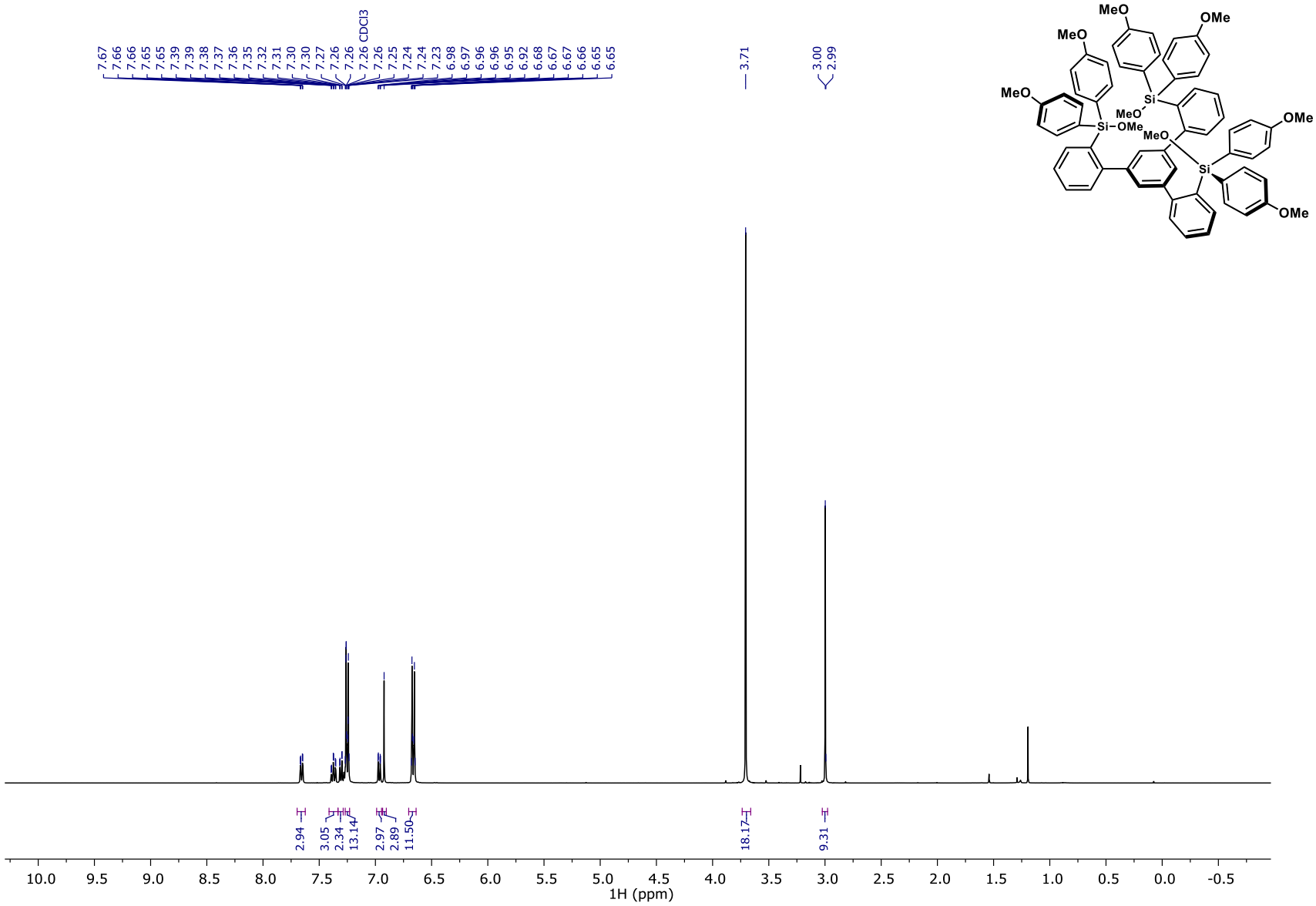
^{13}C NMR of (5,5''-Difluoro-5'-(5-fluoro-2-(methoxydiphenylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxydiphenylsilane) (9b), 126 MHz, C_6D_6, 25ºC



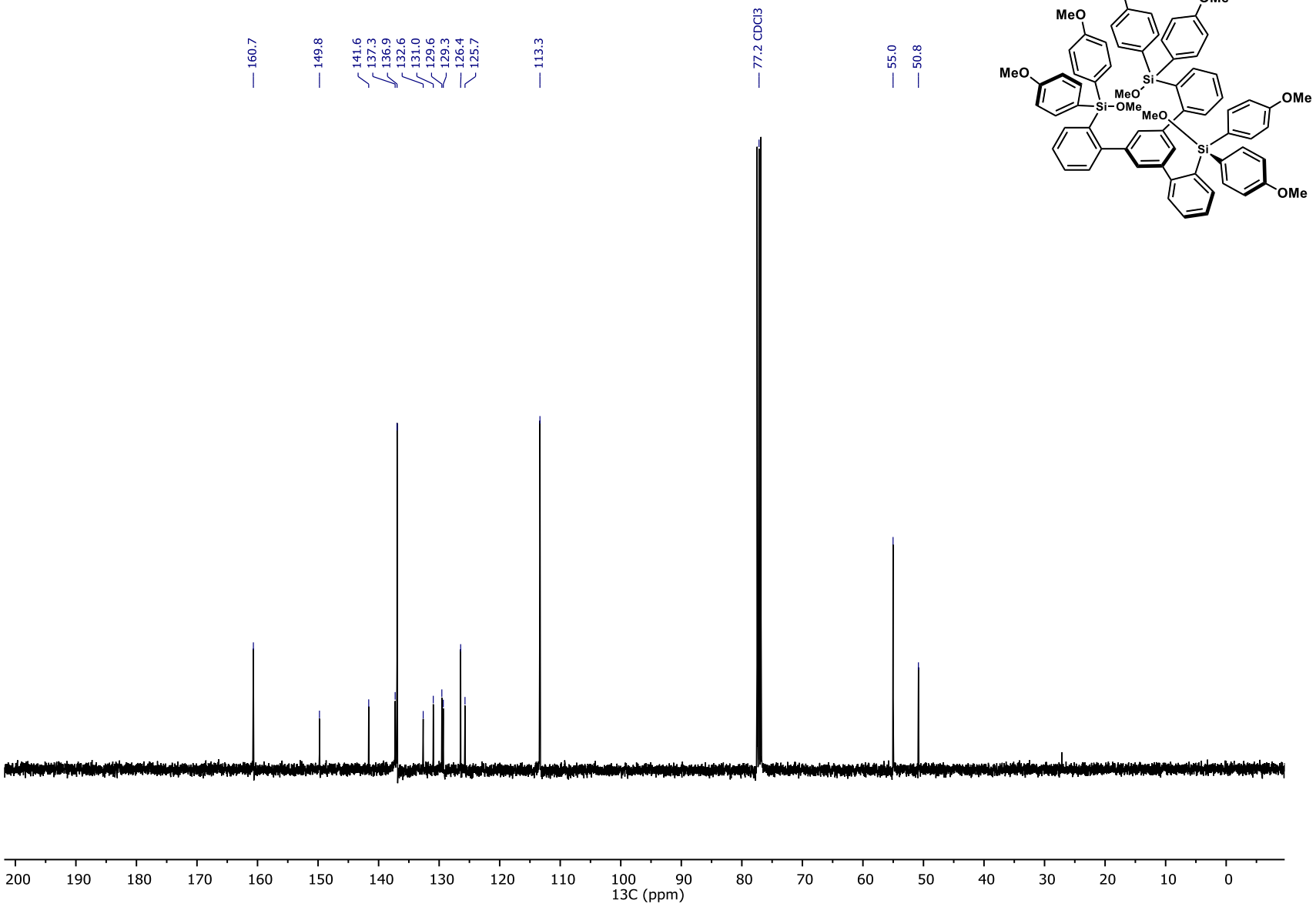
^{29}Si NMR of (5,5''-Difluoro-5'-(5-fluoro-2-(methoxydiphenylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxydiphenylsilane) (9b), 99 MHz, C_6D_6, 25ºC



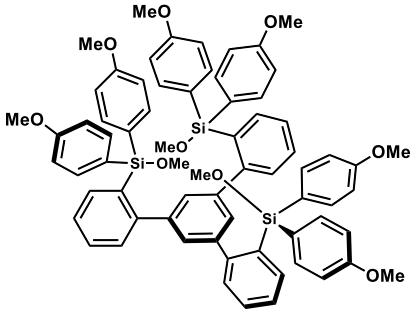
^{1}H NMR of (5'-(2-(Methoxybis(4-methoxyphenyl)silyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxybis(4-methoxyphenyl)silane) (9c), 400 MHz, $CDCl_3$, 25ºC



^{13}C NMR of (5'-(2-(Methoxybis(4-methoxyphenyl)silyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxybis(4-methoxyphenyl)silane) (9c), 126 MHz, $CDCl_3$, 25ºC

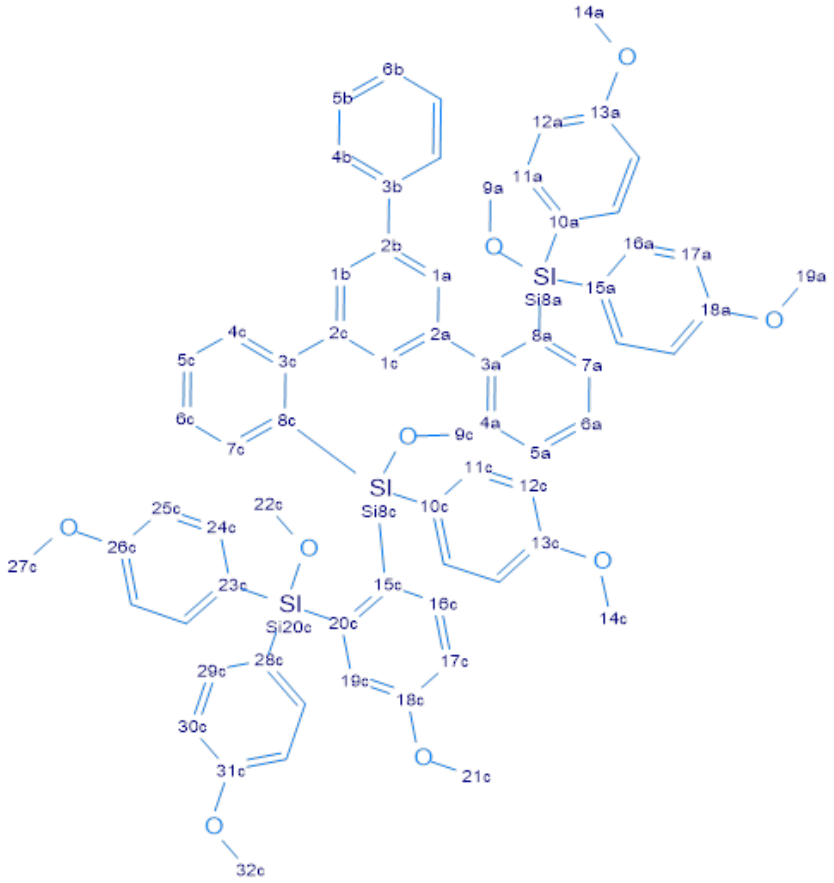


^{29}Si NMR of (5'-(2-(Methoxybis(4-methoxyphenyl)silyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(methoxybis(4-methoxyphenyl)silane) (9c), 99 MHz**,** $CDCl_3$, 25ºC



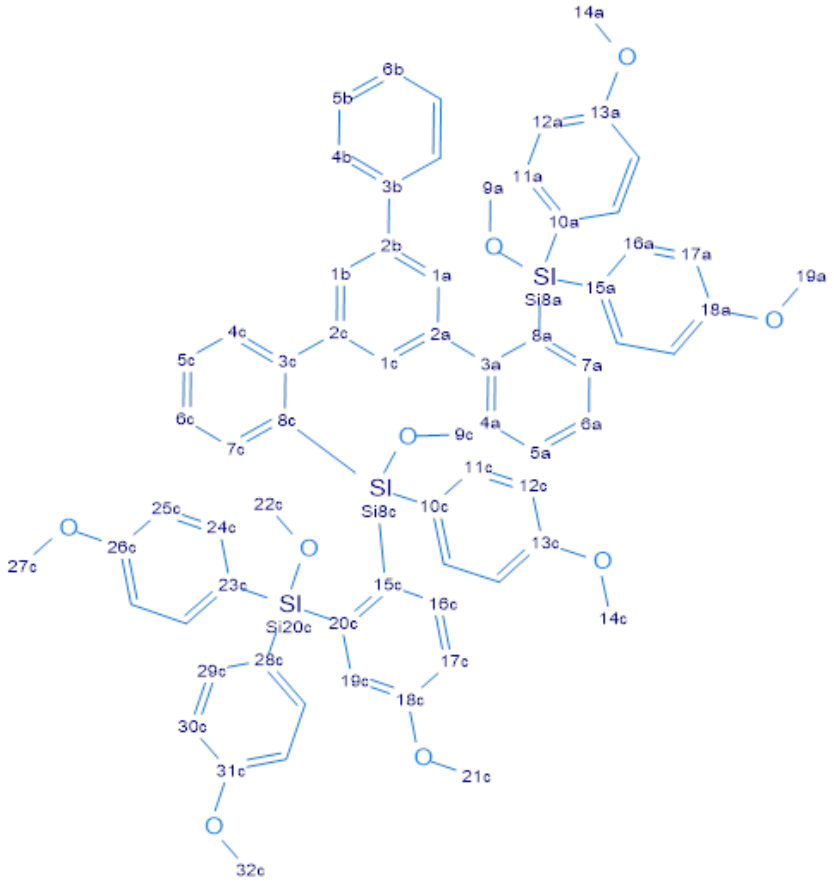
NMR assignment table of constitutional isomer S1:

Atom	δ	J	COSY	HSQC	HMQC(MB)	Si-HMQC(MB)	ROESY
C1a	126.26			1a			
H1a	6.97	t 1.7(1b,1c)	1b, 1c	1a	1b, 1c, 3a, 3b		4a, 4b, 9a, 11a, 16a
C1b	126.58			1b	1a		
H1b	7.03	(br) s	1a, 1c	1b	1c, 3b, 3c		4b, 9c, 11c
C1c	128.97			1c	1a, 1b		
H1c	6.51	(br) s	1a, 1b	1c	3a, 3c		9c, 11a
C2a	142.85				4a		
C2b	138.31				4b		
C2c	143.74				4c		
C3a	149.79				1a, 1c, 5a, 7a		
C3b	140.25				1a, 1b, 5b		
C3c	148.77				1b, 1c, 4c, 5c, 7c		
C4a	130.12			4a	6a		
H4a	6.65	m	5a, 6a, 7a	4a	2a, 6a, 8a		1a, 9c
C4b	126.78			4b	6b		
H4b	6.52	m (AA'XX'Y)	5b, 6b	4b	2b, 6b		1a, 1b
C4c	129.84			4c	6c		
H4c	6.57	dm 7.0(5c)	5c, 6c, 7c	4c	2c, 3c, 6c, 8c		
C5a	129.50			5a	7a		
H5a	7.35	m	4a, 6a, 7a	5a	3a, 7a		
C5b	128.12			5b	5b		
H5b	7.10	m (AA'XX'Y)	4b, 6b	5b	3b, 5b		
C5c	128.38			5c	7c		
H5c	7.29	m	4c, 7c	5c	3c, 7c		
C6a	125.99			6a	4a		
H6a	7.41	tm 7.5(7a,5a)	4a, 5a, 7a	6a	4a, 8a		
C6b	126.68			6b	4b		
H6b	7.17	m	4b, 5b	6b	4b		
C6c	125.56			6c	4c		
H6c	7.26	m	4c, 7c	6c	4c, 8c		
C7a	136.49			7a	5a		
H7a	7.99	dm 7.5(6a)	4a, 5a, 6a	7a	3a, 5a, 8a	Si8a	9a
C7c	136.75			7c	5c		
H7c	7.60	dm 6.7(6c)	4c, 5c, 6c	7c	3c, 5c, 8c	Si8c	9c, 11c, 22c
C8a	133.21				4a, 6a, 7a		
Si8a	-11.70					7a, 9a, 11a, 16a	
C8c	135.28				4c, 6c, 7c		
Si8c	-11.12					7c, 9c, 11c, 16c	
C9a	51.01			9a	9a		
H9a	3.36	s		9a	9a	Si8a	1a, 7a, 11a, 16a
C9c	50.79			9c	9c		
H9c	2.82	s		9c	9c	Si8c	1b, 1c, 4a, 7c, 11c, 16c, 24c, 29c
C10a	125.68				11a, 12a		
C10c	128.12				11c, 12c		
C11a	136.99			11a	11a		
H11a	7.34	m (AA'XX')	12a	11a	10a, 11a, 12a, 13a	Si8a	1a, 1c, 9a
C11c	137.55			11c	11c		
H11c	7.34	m (AA'XX')	12c	11c	10c, 11c, 12c, 13c	Si8c	1b, 7c, 9c, 16c, 22c

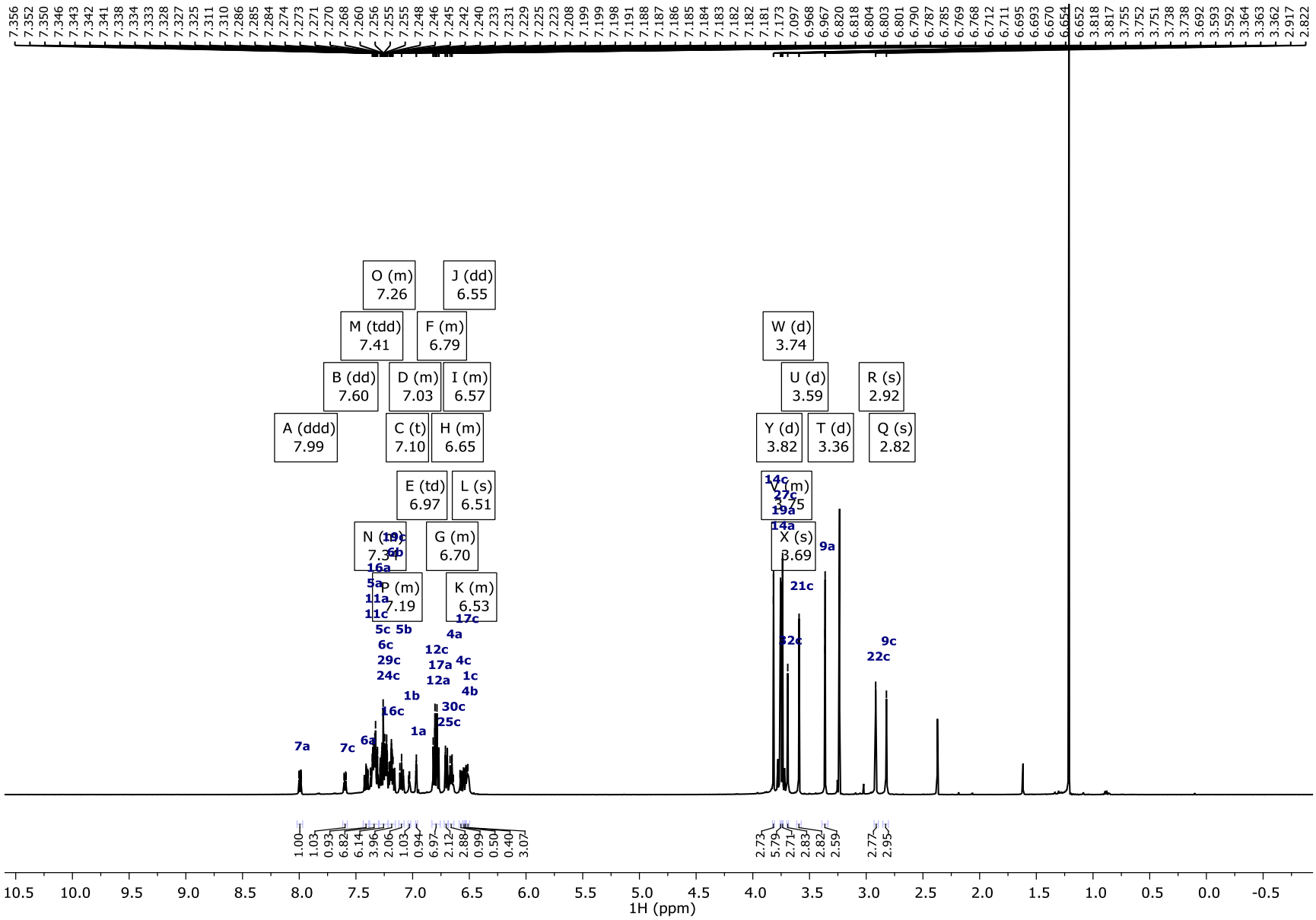


NMR assignment table of constitutional isomer S1:

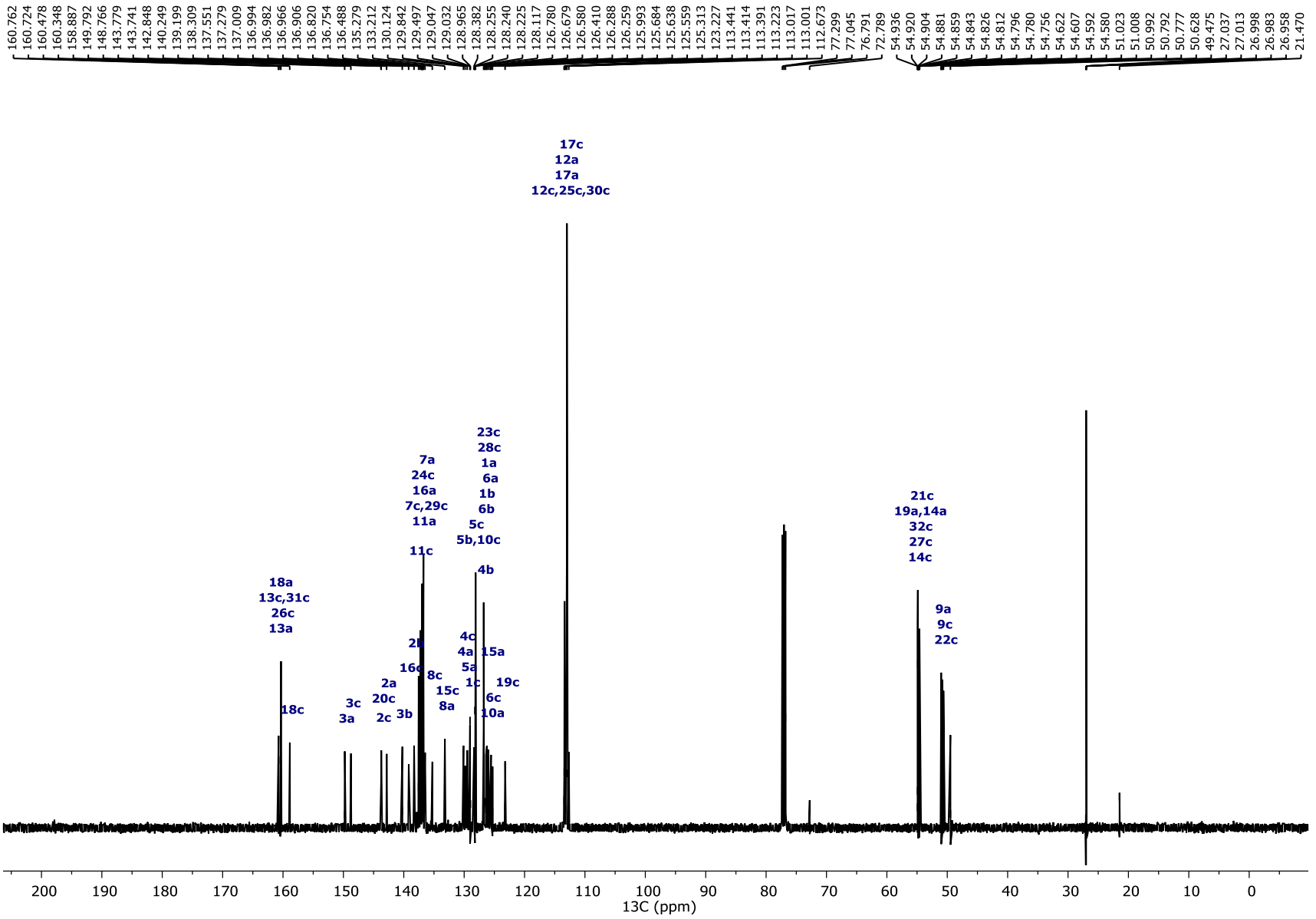
Atom	δ	J	COSY	HSQC	HMQC(MB)	Si-HMQC(MB)	ROESY
C12a	113.41			12a	11a, 12a		
H12a	6.80	m (AA'XX')	11a, 14a	12a	10a, 12a, 13a		14a
C12c	113.00			12c	11c, 12c		
H12c	6.81	m (AA'XX')	11c, 14c	12c	10c, 12c, 13c		14c
C13a	160.75				11a, 12a, 14a		
C13c	160.35				11c, 12c, 14c		
C14a	54.88			14a			
H14a	3.76	s	12a	14a	13a		12a
C14c	54.92			14c			
H14c	3.82	s	12c	14c	13c		12c
C15a	125.64				16a, 17a		
C15c	133.20				16c, 17c		
C16a	136.98			16a	16a		
H16a	7.32	m (AA'XX')	17a	16a	15a, 16a, 17a, 18a	Si8a	1a, 9a
C16c	139.20			16c	16c		
H16c	7.20	m	17c	16c	15c, 16c, 18c, 20c	Si8c	9c, 11c
C17a	113.39			17a	16a, 17a		
H17a	6.78	m (AA'XX')	16a, 19a	17a	15a, 17a, 18a		19a
C17c	112.67			17c	17c, 19c		
H17c	6.54	dd 8.5(16c),2.8(19c)	16c, 19c, 21c	17c	15c, 17c, 18c, 19c		21c
C18a	160.72				16a, 17a, 19a		
C18c	158.89				16c, 17c, 21c		
C19a	54.88			19a			
H19a	3.75	s	17a	19a	18a		17a
C19c	123.23			19c	17c, 19c		
H19c	7.19	m	17c, 21c	19c	17c, 19c	Si20c	21c, 22c
C20c	143.78				16c		
Si20c	-11.26					19c, 22c, 24c, 29c	
C21c	54.61			21c			
H21c	3.59	s	17c, 19c	21c	18c		17c, 19c
C22c	50.63			22c	22c		
H22c	2.92	s		22c	22c	Si20c	7c, 11c, 19c, 24c, 29c
C23c	126.41				24c, 25c		
C24c	137.28			24c	24c		
H24c	7.24	m (AA'XX')	25c	24c	23c, 24c, 25c, 26c	Si20c	9c, 22c
C25c	113.00			25c	24c, 25c		
H25c	6.70	m (AA'XX')	24c, 27c	25c	23c, 25c, 26c		27c
C26c	160.48				24c, 25c, 27c		
C27c	54.84			27c			
H27c	3.74	s	25c	27c	26c		25c
C28c	126.29				29c, 30c		
C29c	136.75			29c	29c		
H29c	7.23	m (AA'XX')	30c	29c	28c, 29c, 30c, 31c	Si20c	9c, 22c
C30c	113.00			30c	29c, 30c		
H30c	6.66	m (AA'XX')	29c, 32c	30c	28c, 30c, 31c		32c
C31c	160.35				29c, 30c, 32c		
C32c	54.80			32c			
H32c	3.69	s	30c	32c	31c		30c



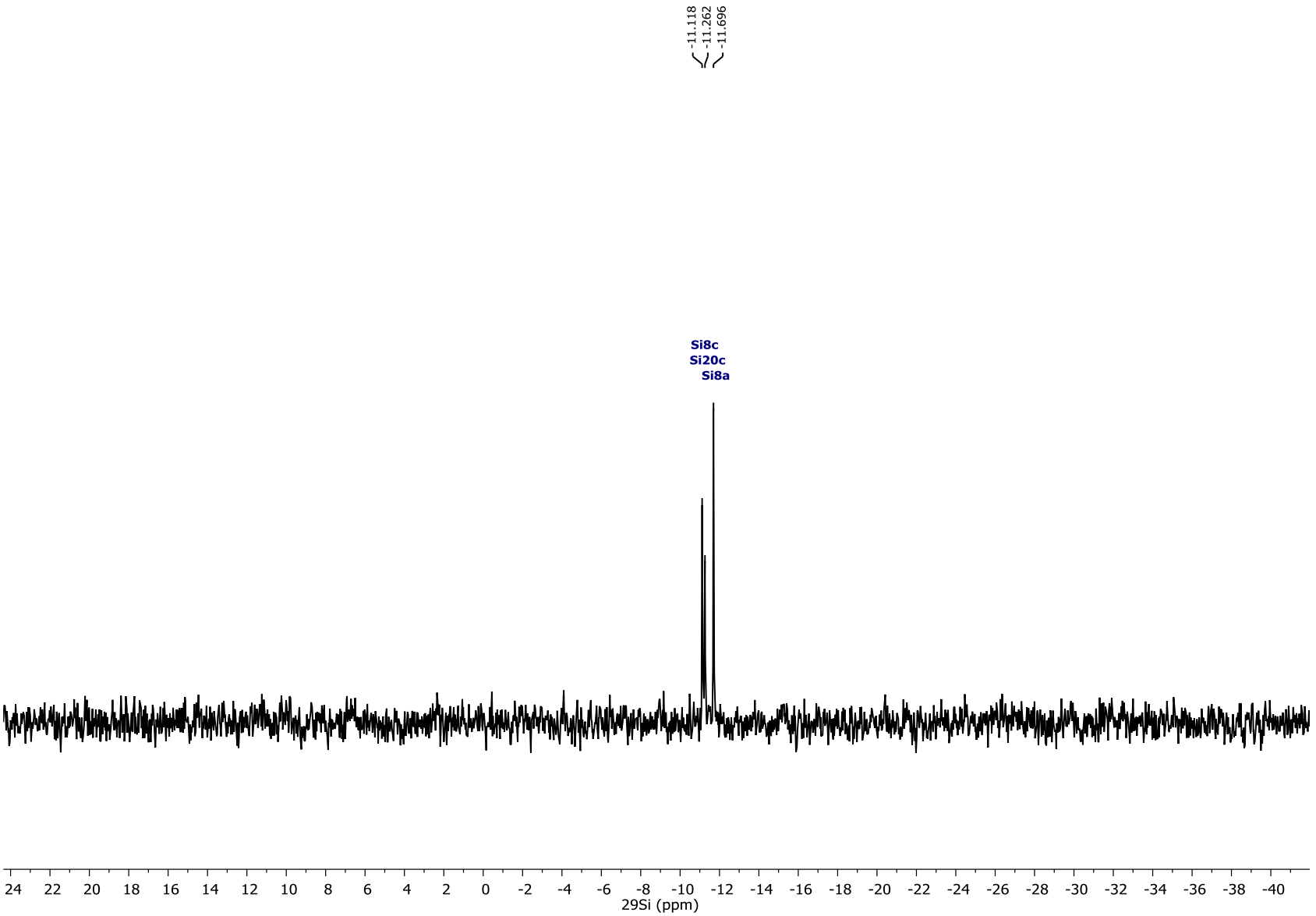
^{1}H NMR of Constitutional Isomer S1, 500 MHz, $CDCl_3$, 25ºC



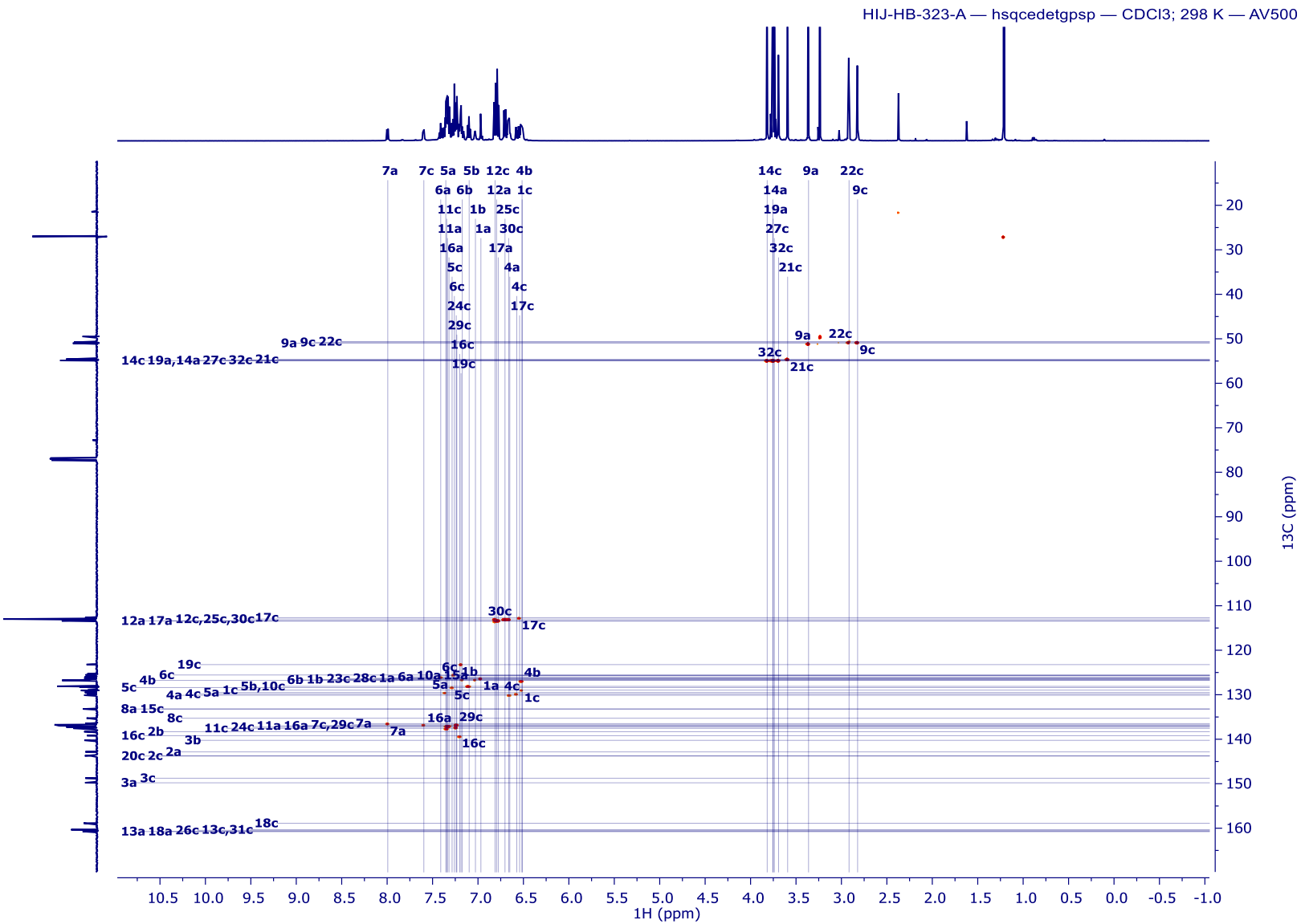
^{13}C NMR of Constitutional Isomer S1, 126 MHz, $CDCl_3$, 25ºC



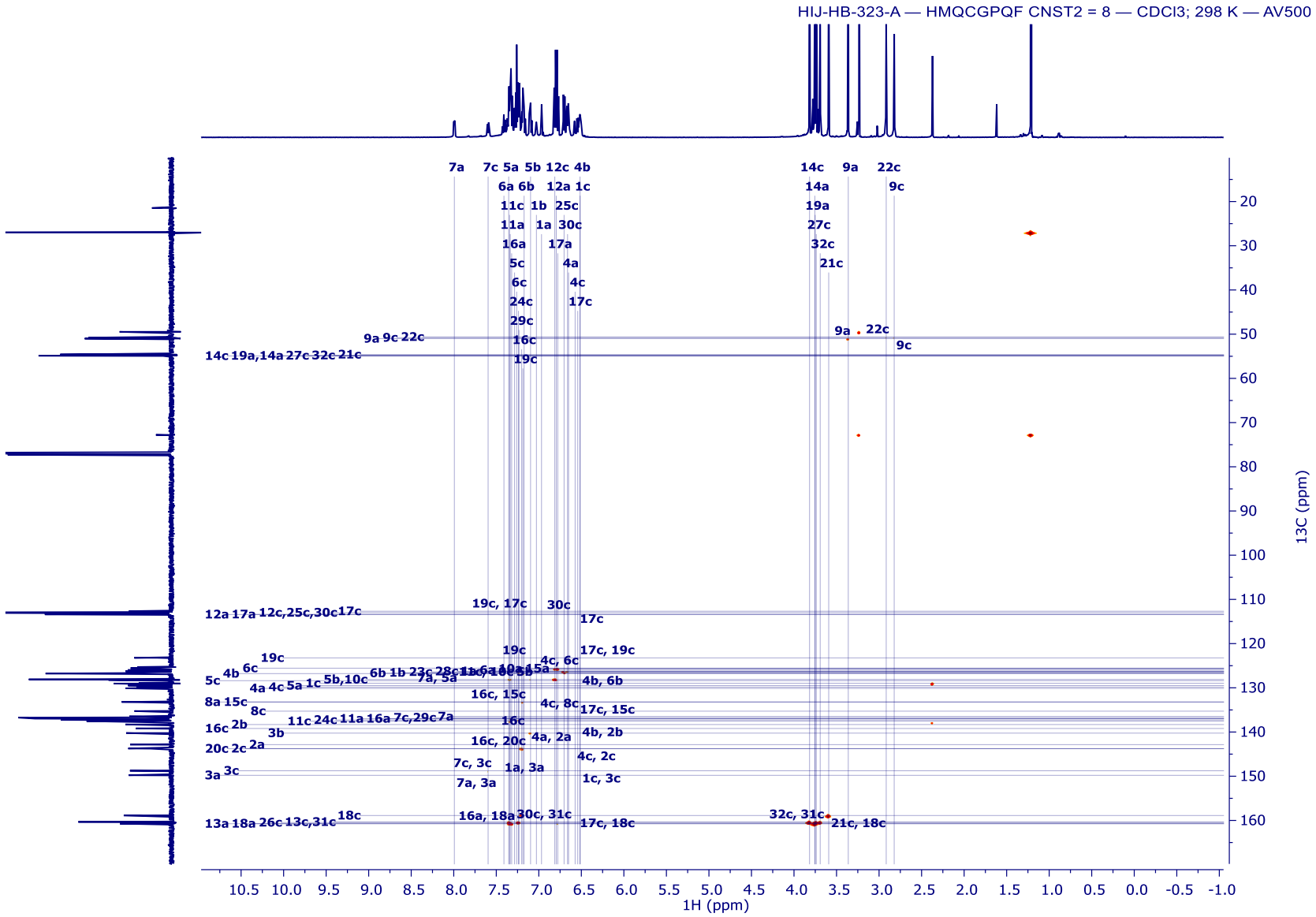
^{29}Si NMR of Constitutional Isomer S1, 99 MHz, $CDCl_3$, 25ºC



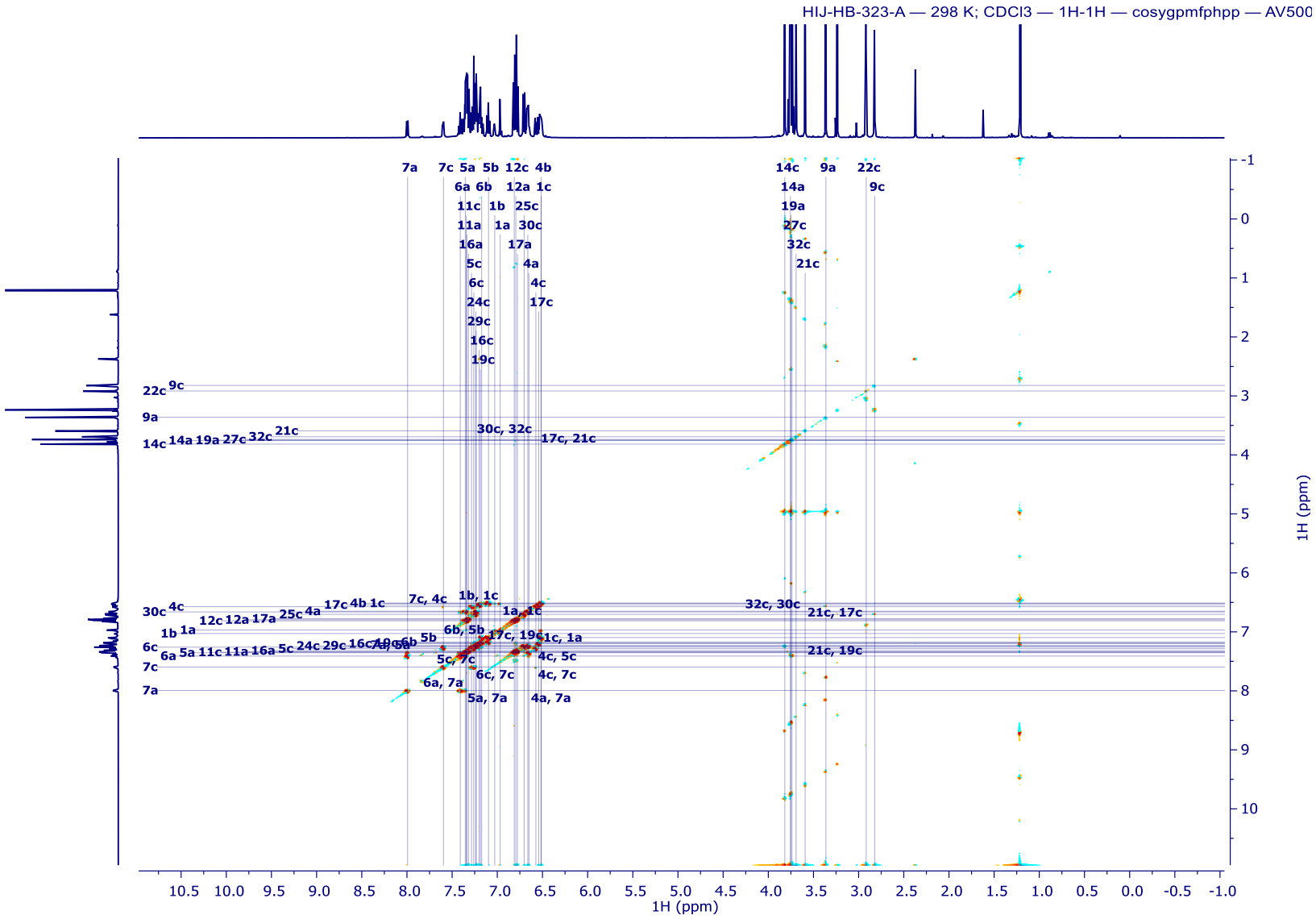
^{1}H-^{13}C HSQC NMR of Constitutional Isomer S1, $CDCl_3$, 25ºC



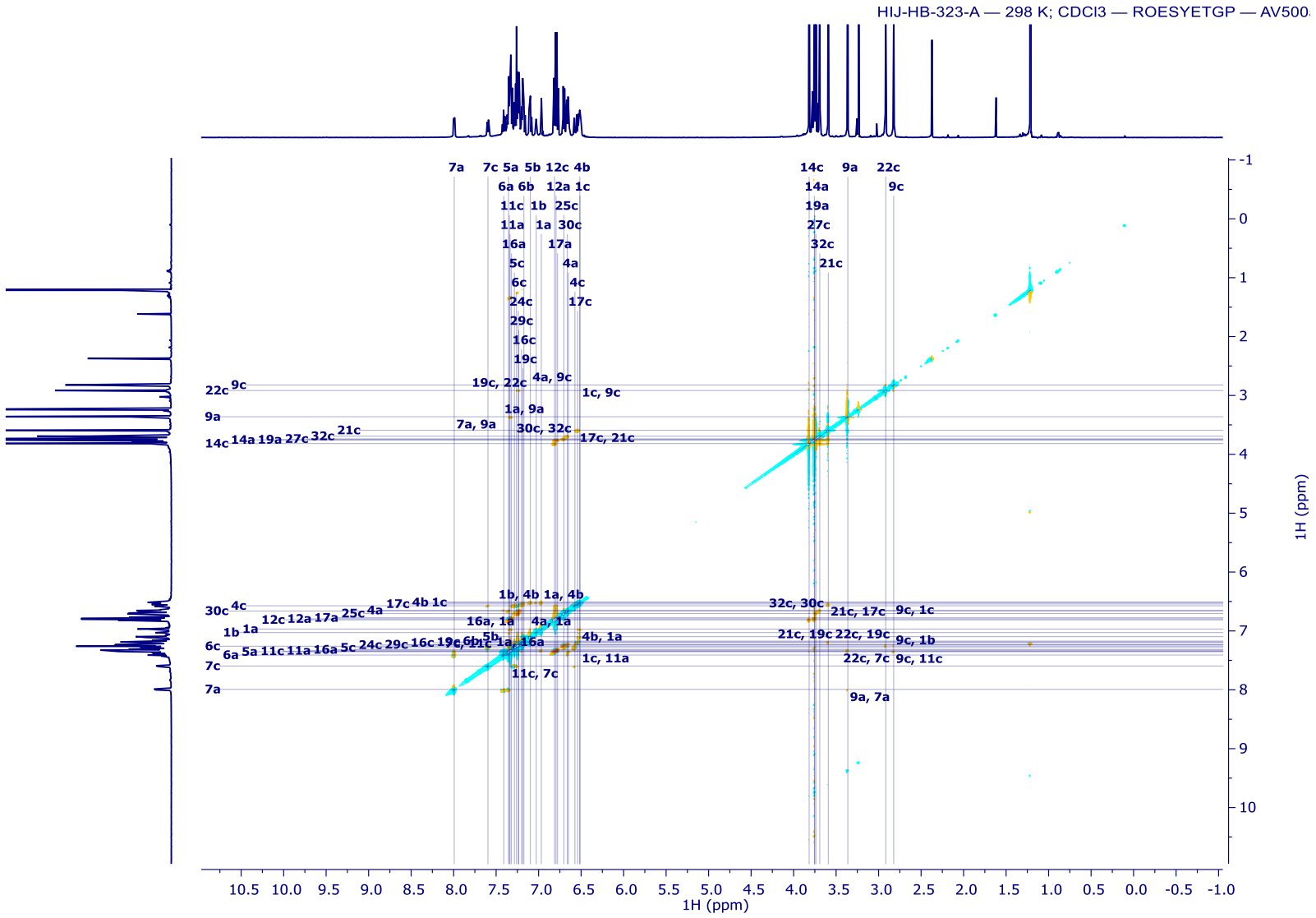
^{1}H-^{13}C HMBC NMR of Constitutional Isomer S1, $CDCl_3$, 25ºC



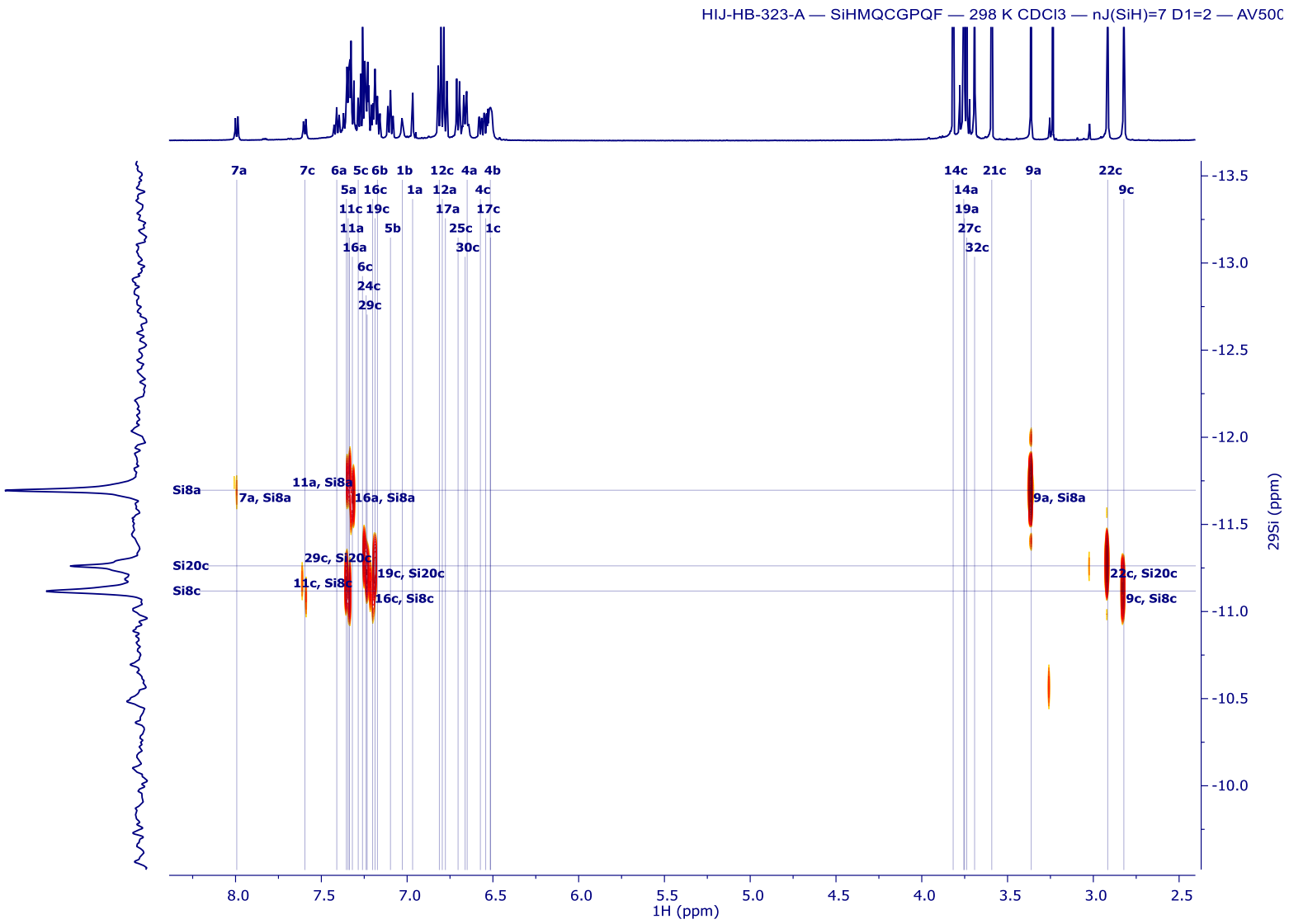
^{1}H-^{1}H COSY NMR of Constitutional Isomer S1, $CDCl_3$, 25ºC



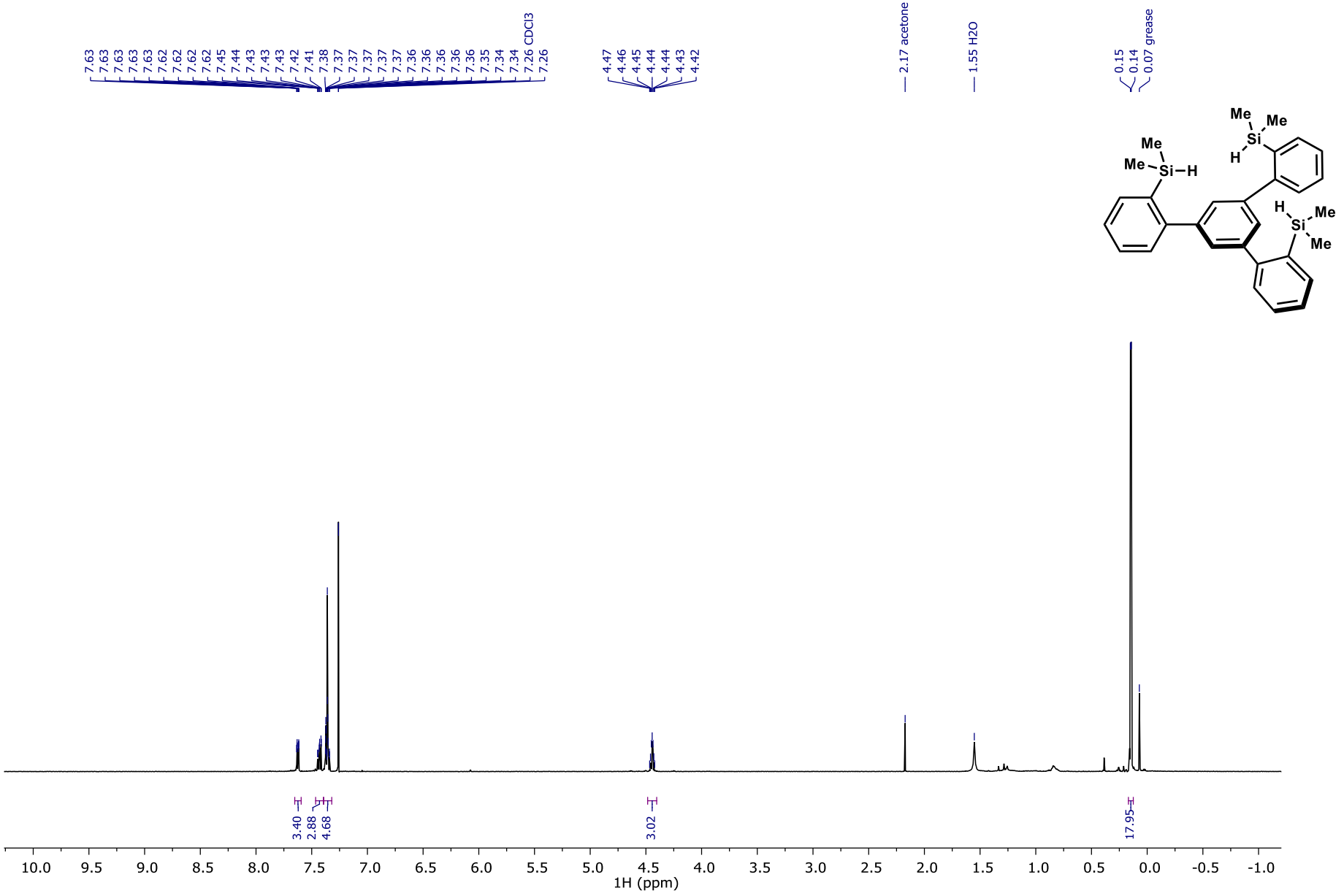
^{1}H-^{1}H ROESY NMR of Constitutional Isomer S1, $CDCl_3$, 25ºC



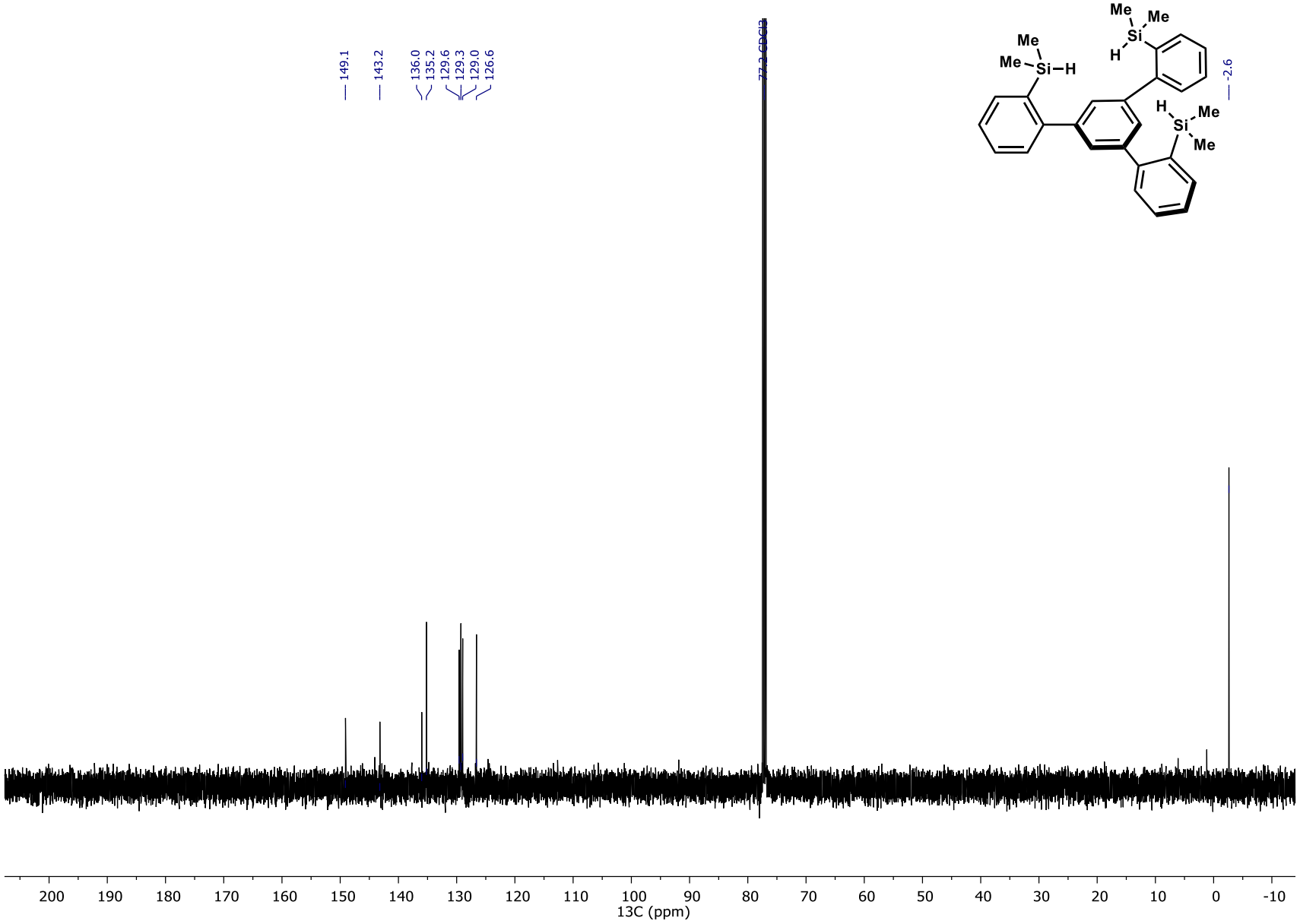
^{1}H-^{29}Si HMBC NMR of Constitutional Isomer S1, $CDCl_3$, 25ºC



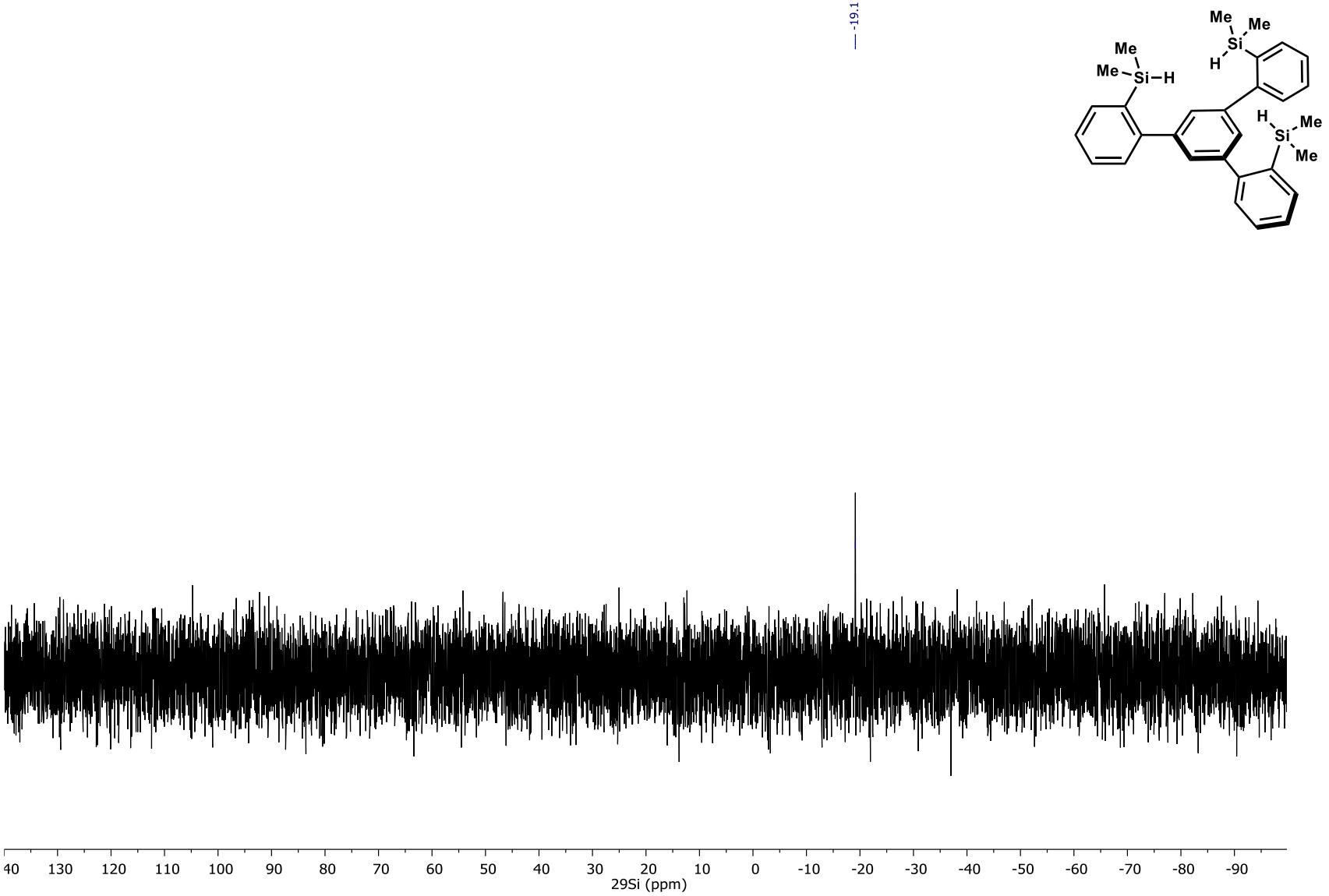
^{1}H NMR of 5'-(2-(Dimethylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(dimethylsilane) (10d), 500 MHz, $CDCl_3$, 25ºC



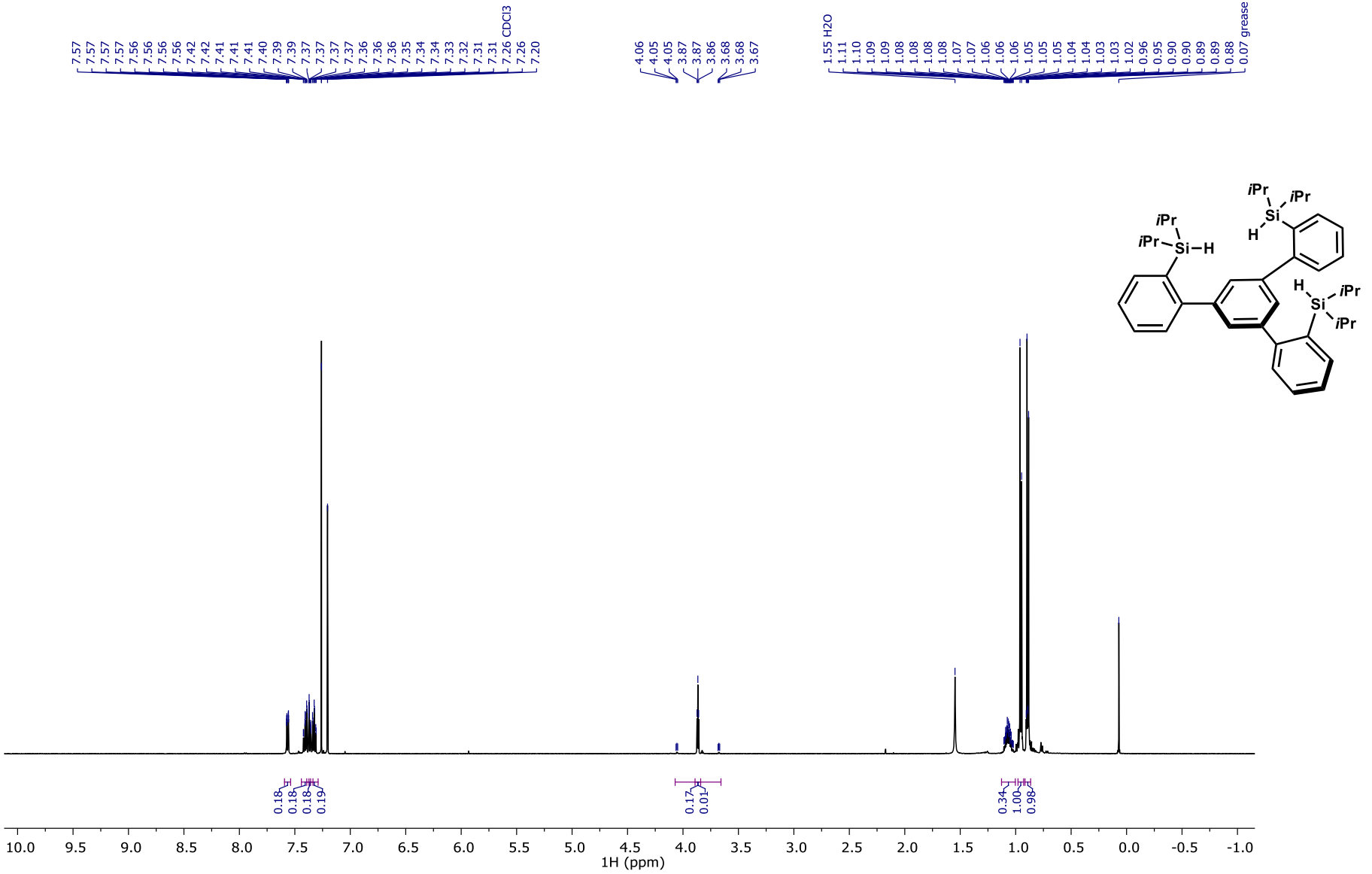
^{13}C NMR of 5'-(2-(Dimethylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(dimethylsilane) (10d), 126 MHz, $CDCl_3$, 25ºC



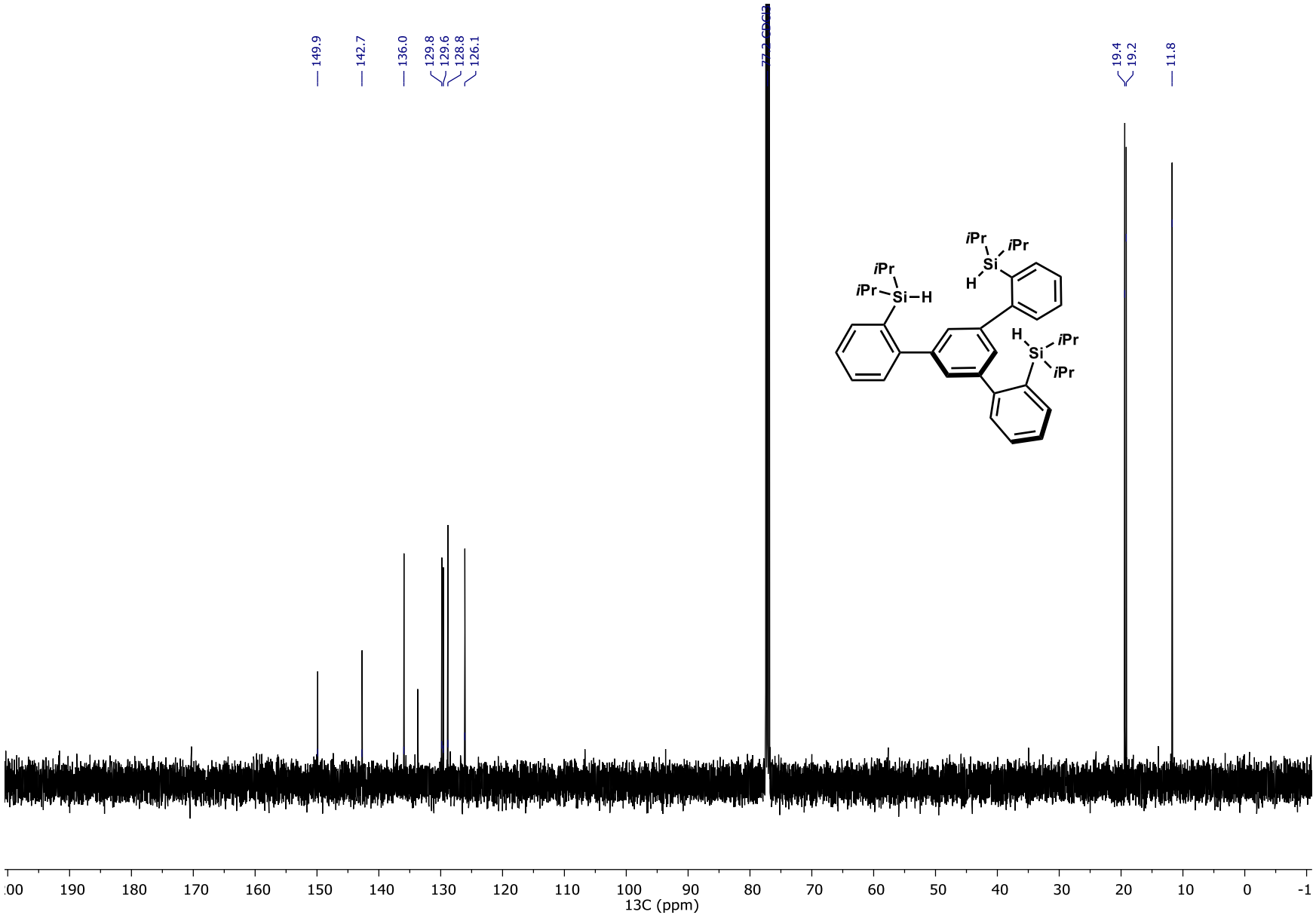
^{29}Si NMR of 5'-(2-(Dimethylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(dimethylsilane) (10d), 99 MHz, $CDCl_3$, 25ºC



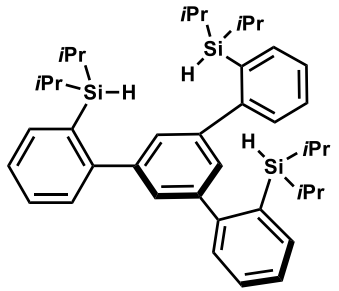
^{1}H NMR of 5'-(2-(Diisopropylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(diisopropylsilane) (10e), 500 MHz, $CDCl_3$, 25ºC



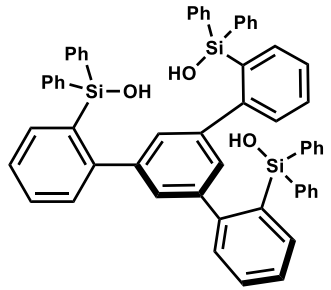
^{13}C NMR of 5'-(2-(Diisopropylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(diisopropylsilane) (10e), 126 MHz, $CDCl_3$, 25ºC

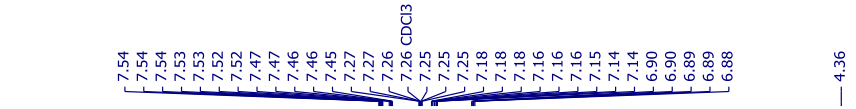


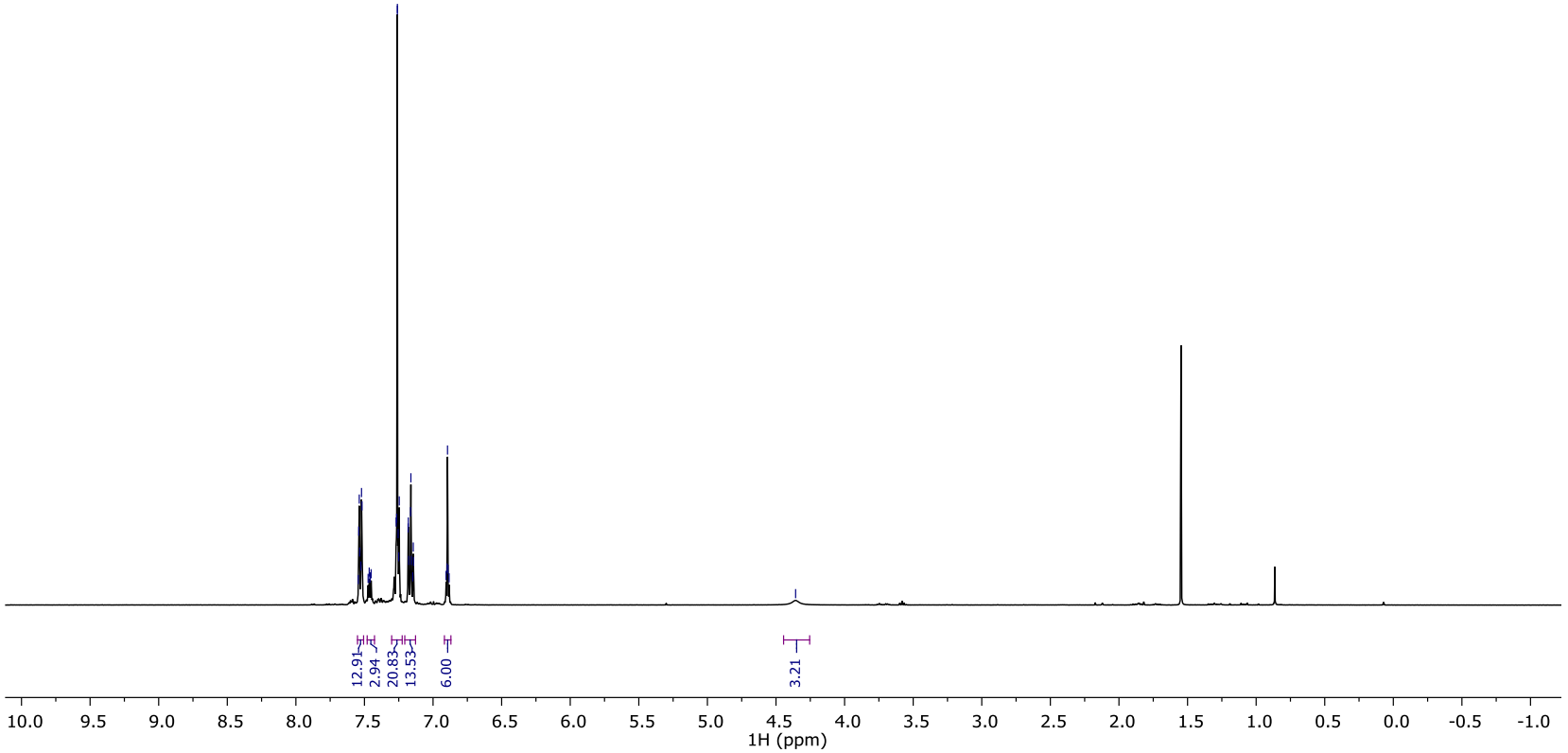
^{29}Si NMR of 5'-(2-(Diisopropylsilyl)phenyl)-[1,1':3',1''-terphenyl]-2,2''-diyl)bis(diisopropylsilane) (10e), 99 MHz, $CDCl_3$, 25ºC



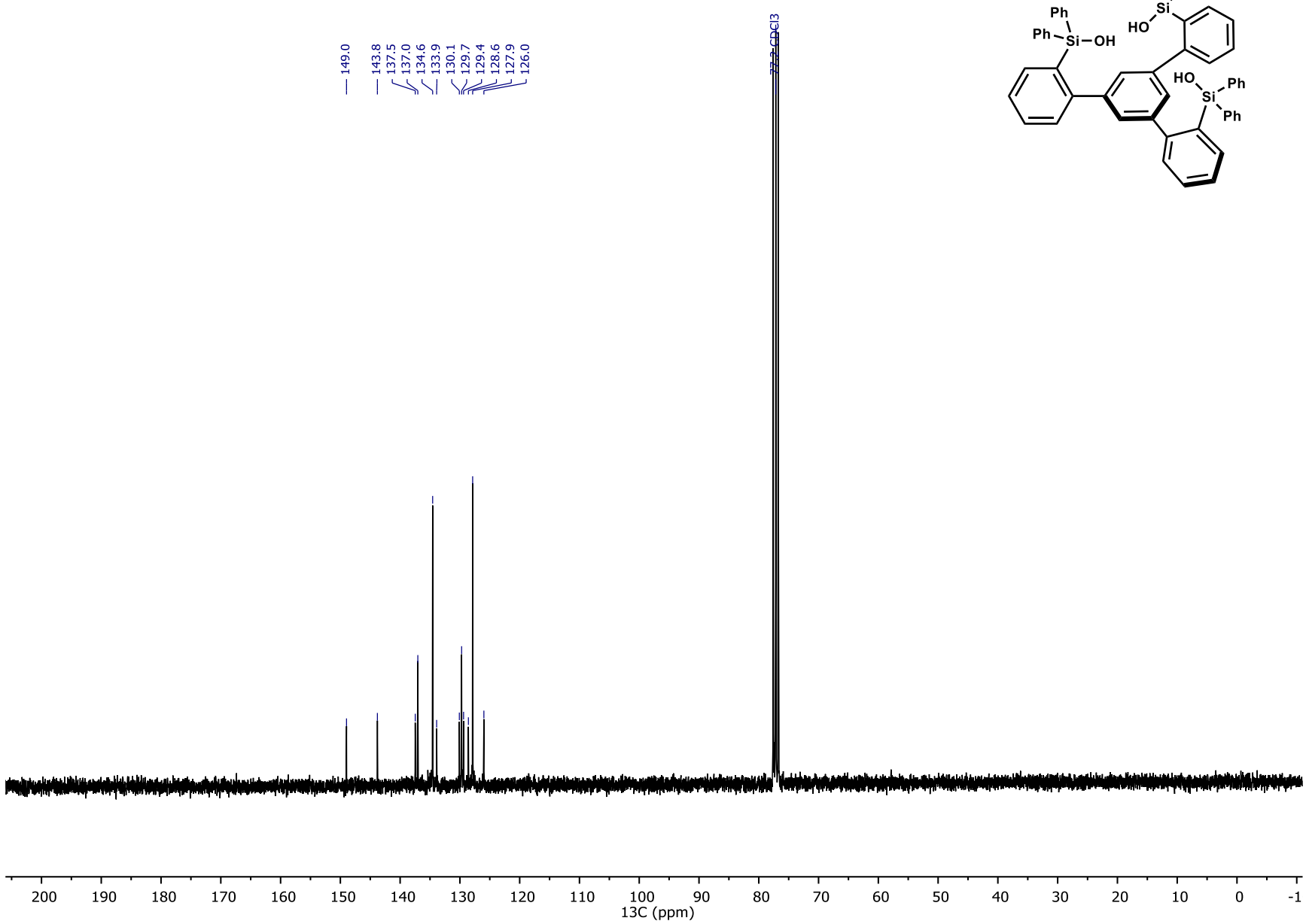
^{1}H NMR of Ligand 11a, 400 MHz**,** $CDCl_3$, 25 ºC



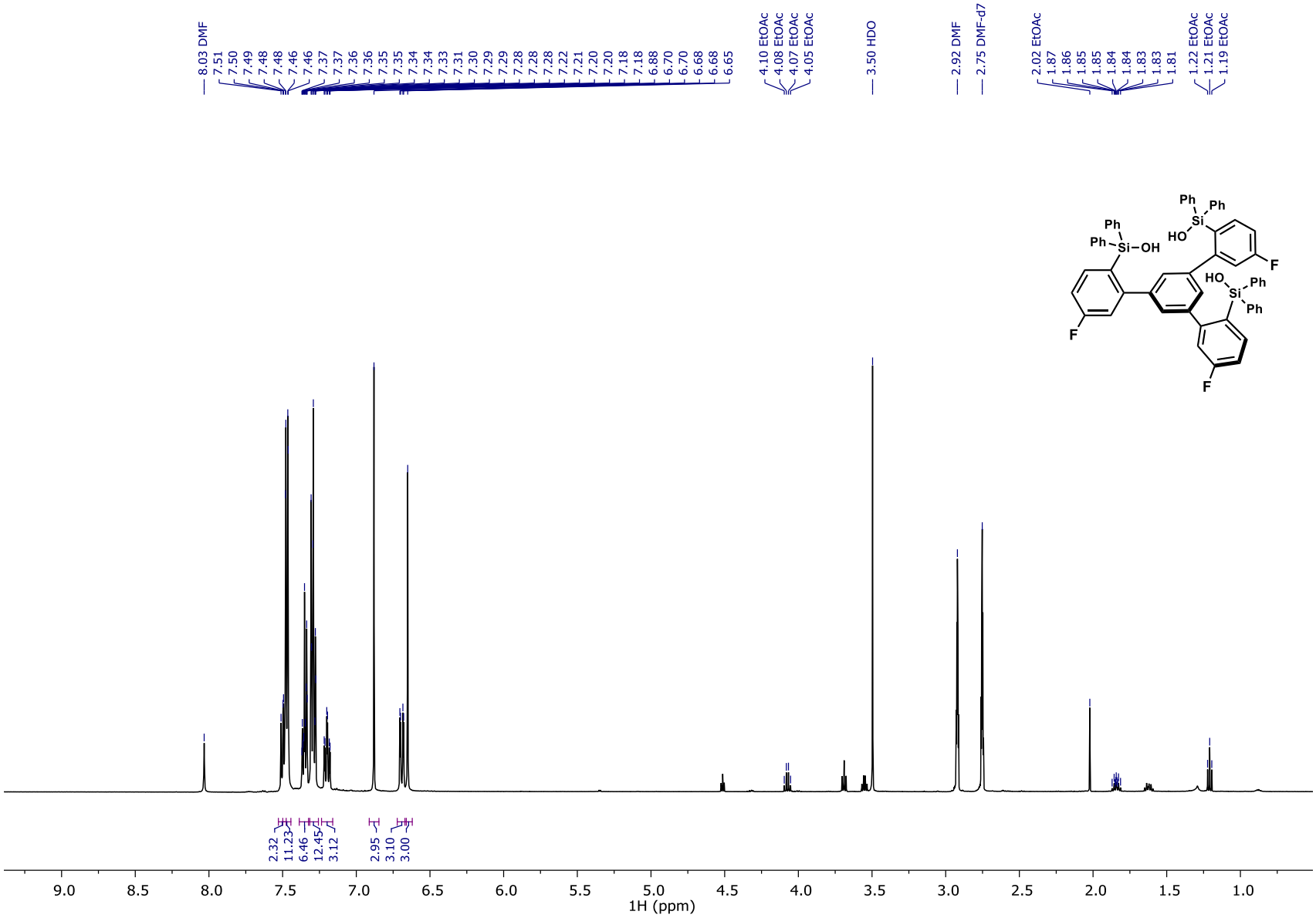




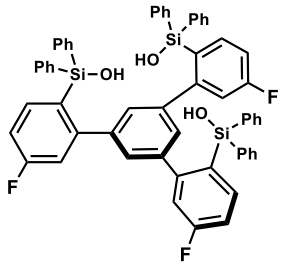
^{13}C NMR of Ligand 11a, 126 MHz, $CDCl_3$, 25ºC



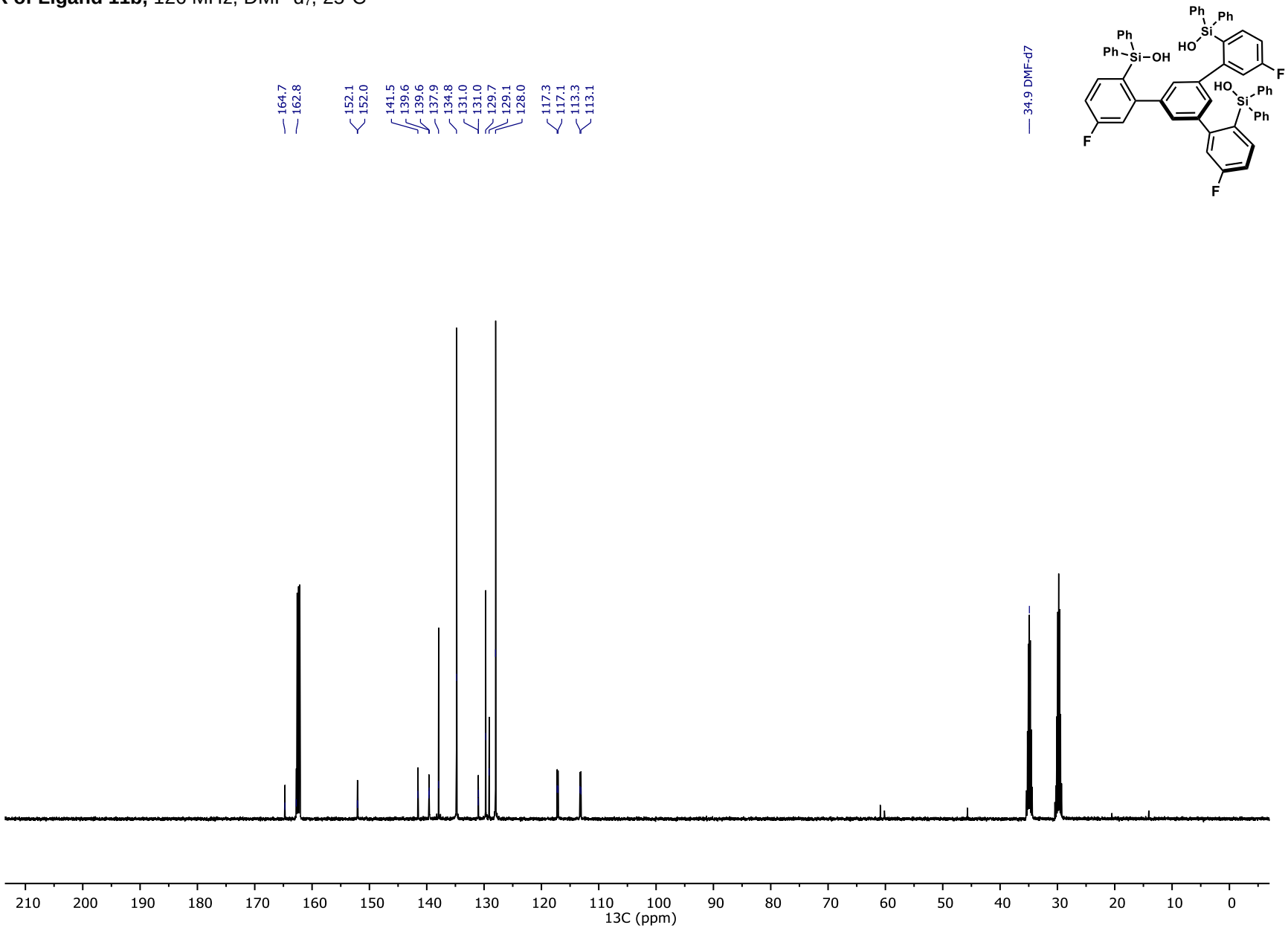
^{1}H NMR of Ligand 11b, 500 MHz, DMF-d_7, 25ºC



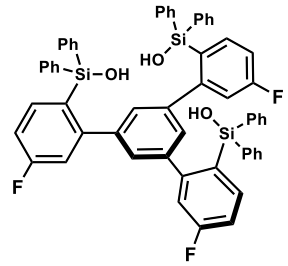
^{19}F NMR of Ligand 11b, 470 MHz, DMF-d_7, 25ºC



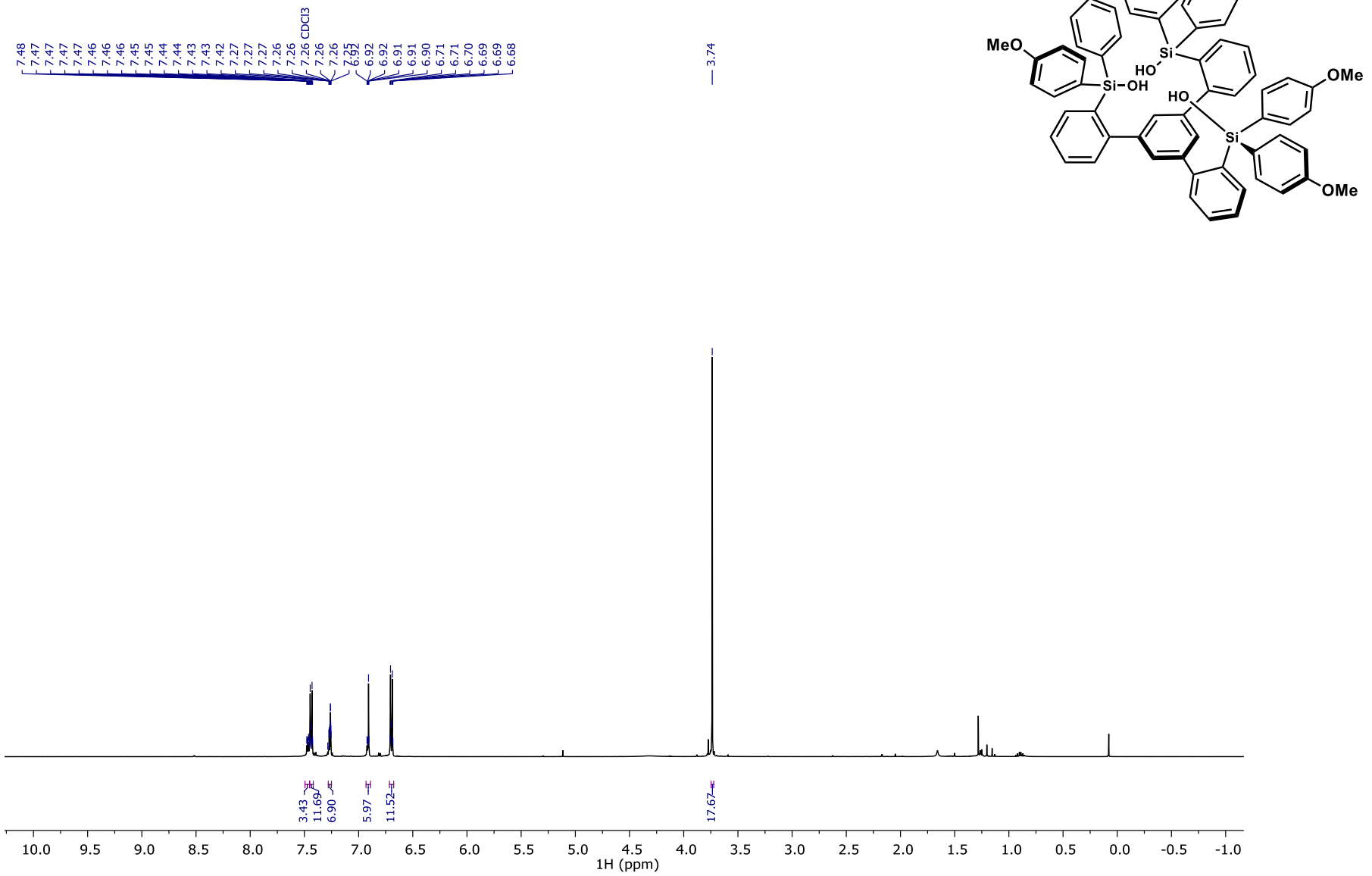
^{13}C NMR of Ligand 11b, 126 MHz, DMF-d_7, 25ºC



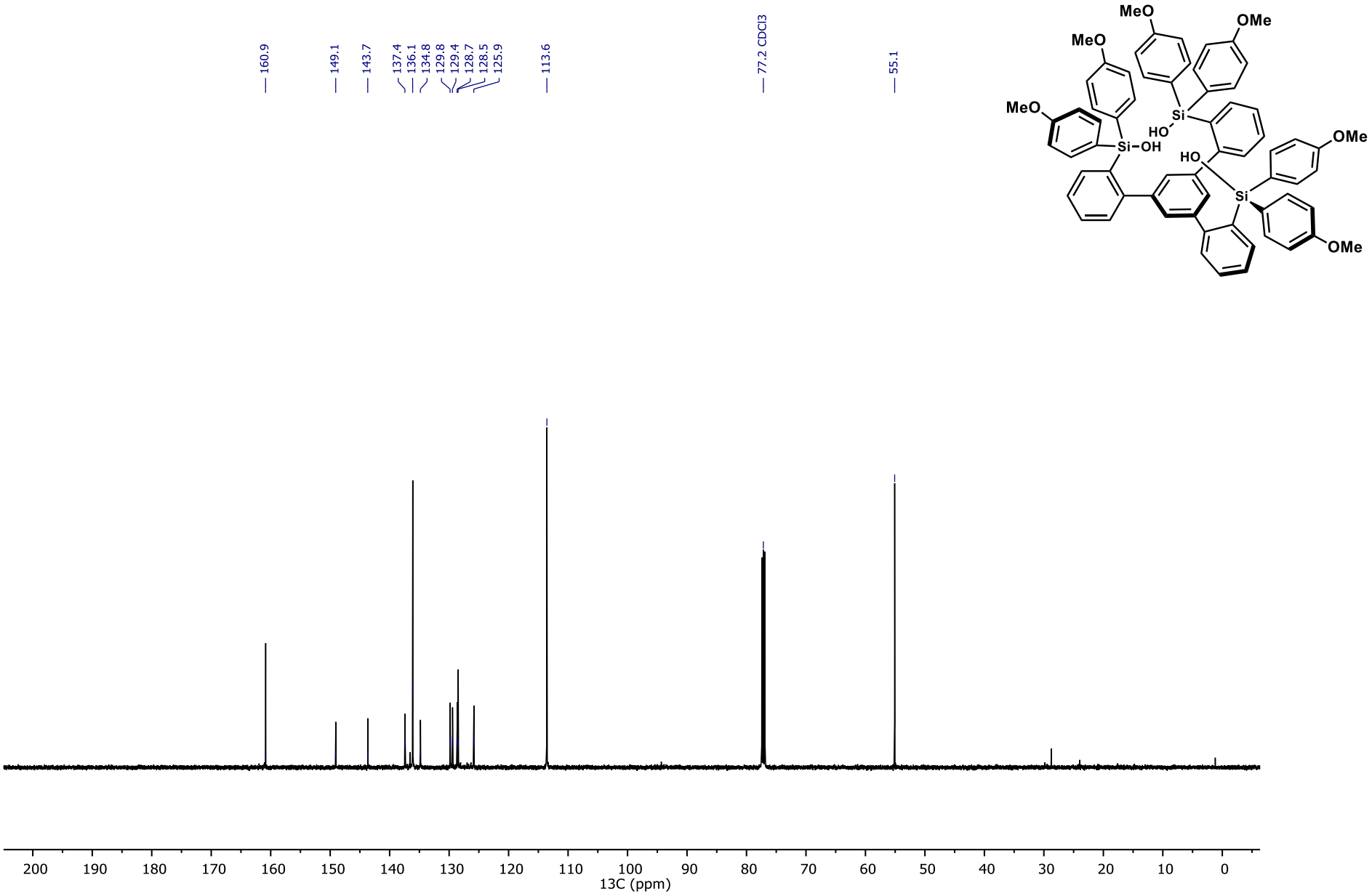
^{29}Si NMR of Ligand 11b, 99 MHz, DMF-d_7, 25ºC



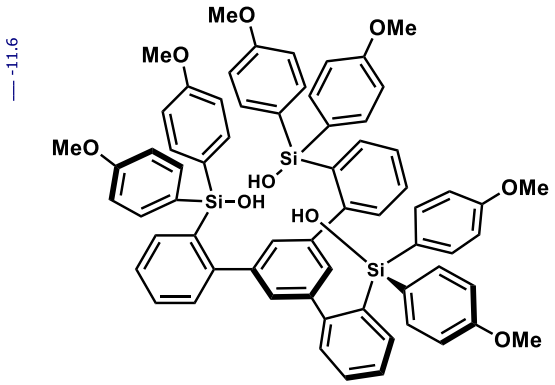
^{1}H NMR of Ligand 11c, 500 MHz, $CDCl_3$, 25ºC

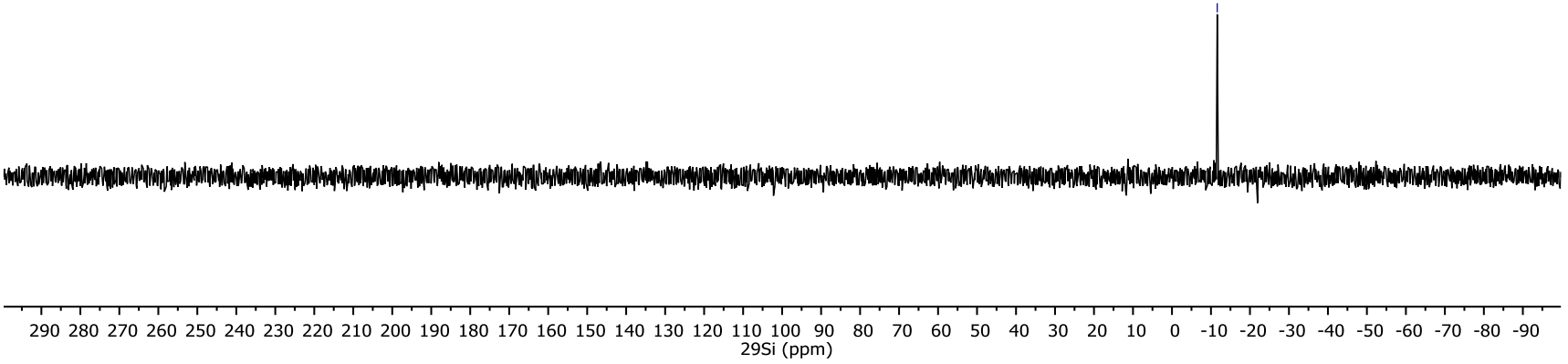


^{13}C NMR of Ligand 11c, 126 MHz, $CDCl_3$, 25ºC

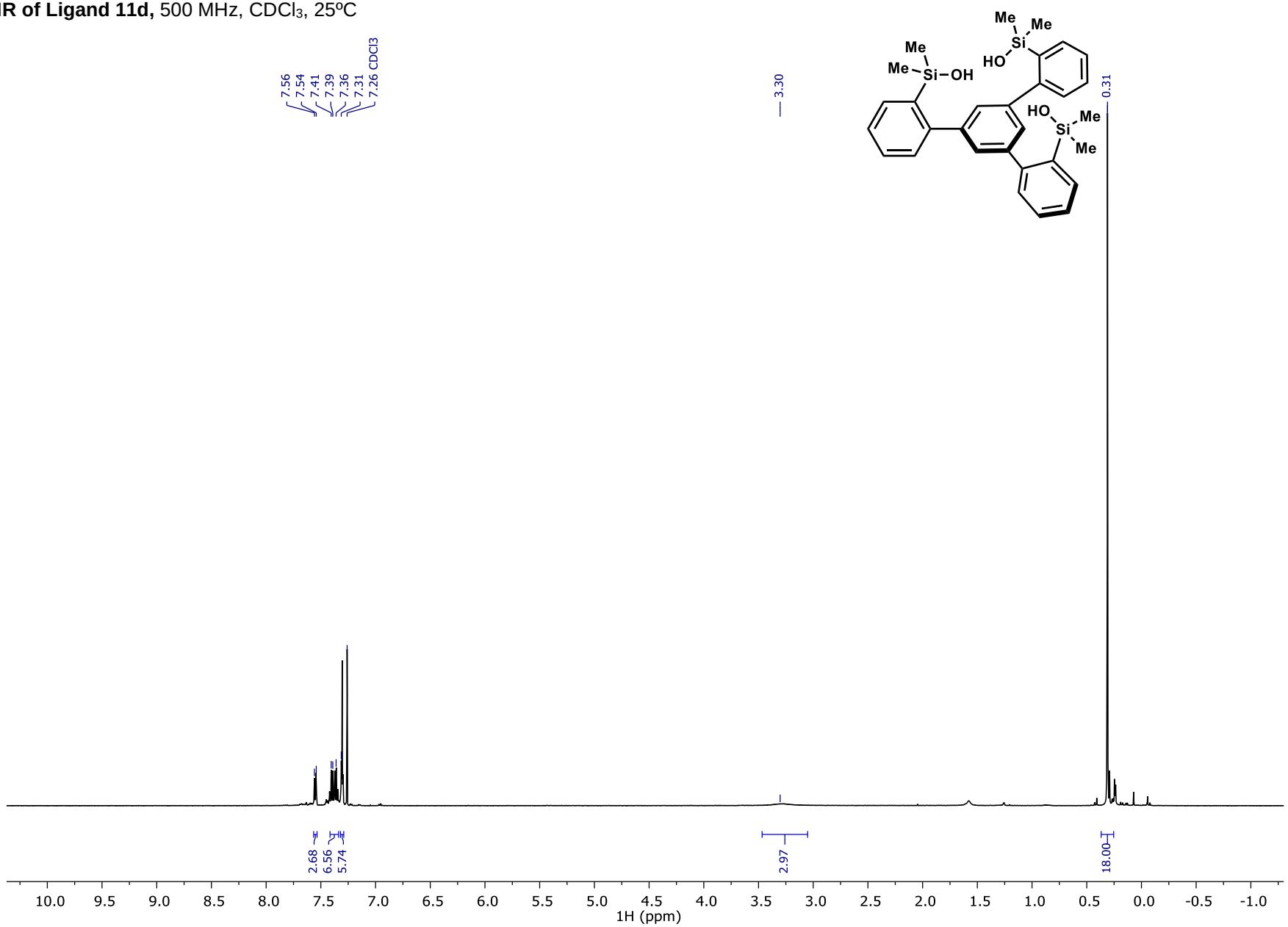


^{29}Si NMR of Ligand 11c, 99 MHz, $CDCl_3$, 25ºC

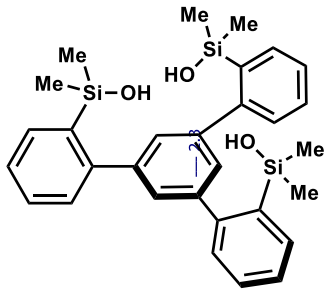




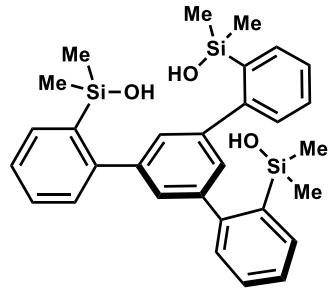
^{1}H NMR of Ligand 11d, 500 MHz, $CDCl_3$, 25ºC



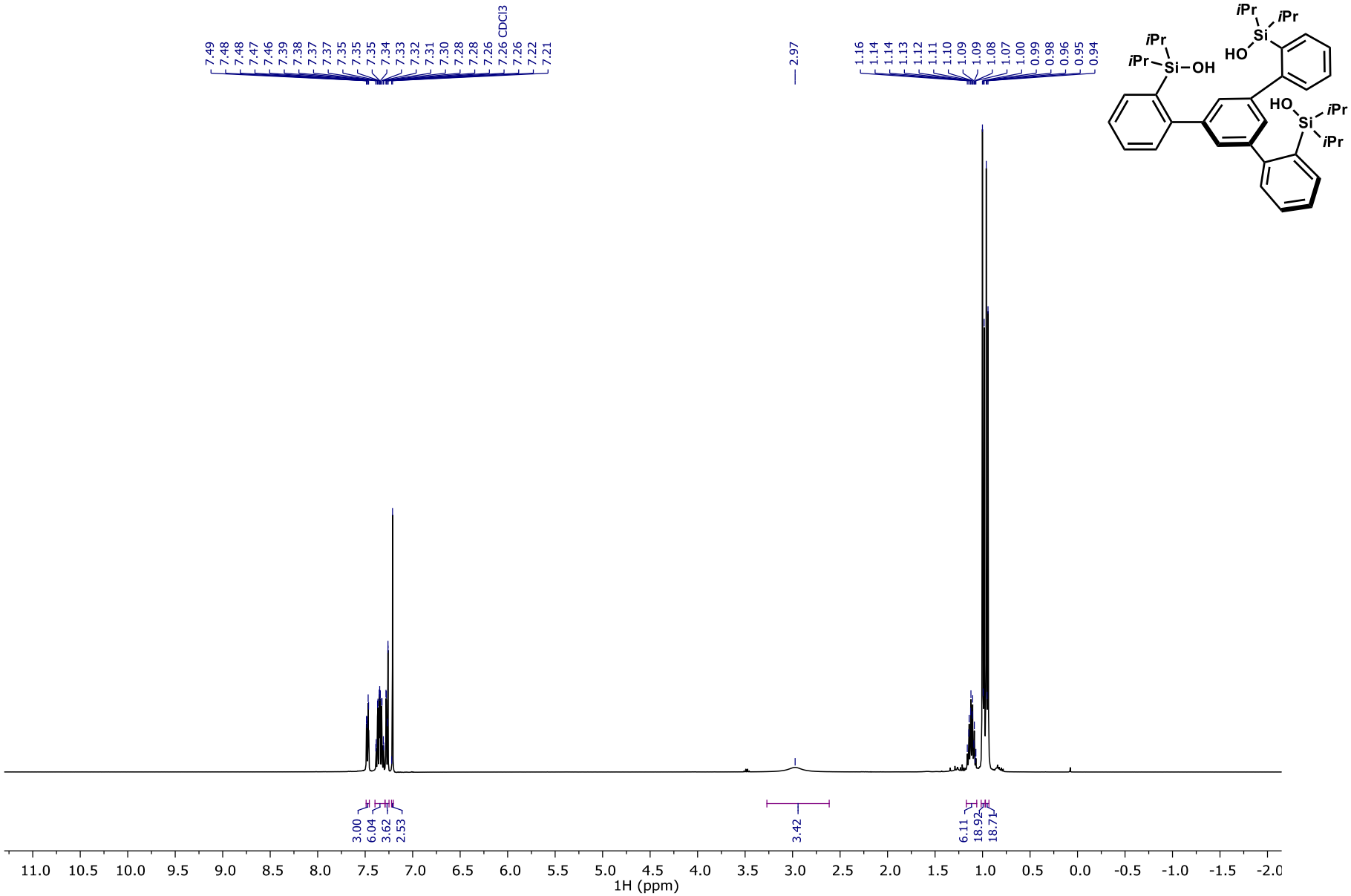
^{3}C NMR of Ligand 11d, 126 MHz, $CDCl_3$, 25ºC



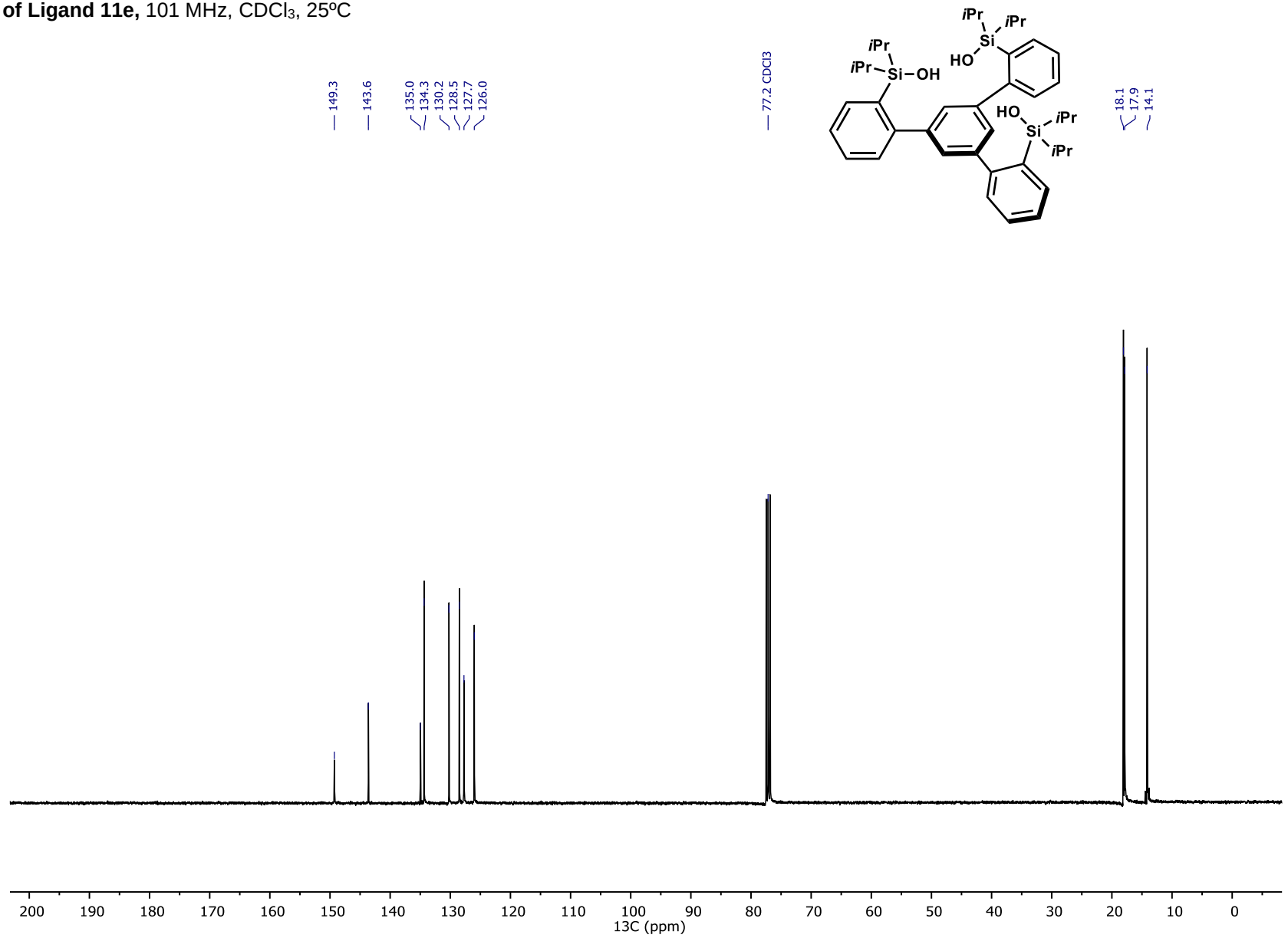
^{29}Si NMR of Ligand 11d, 99 MHz, $CDCl_3$, 25ºC



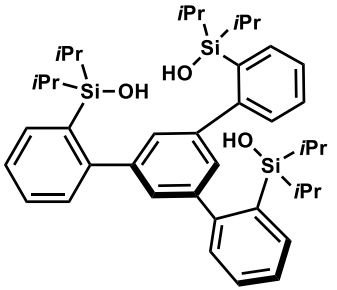
^{1}H NMR of Ligand 11e, 400 MHz, $CDCl_3$, 25ºC

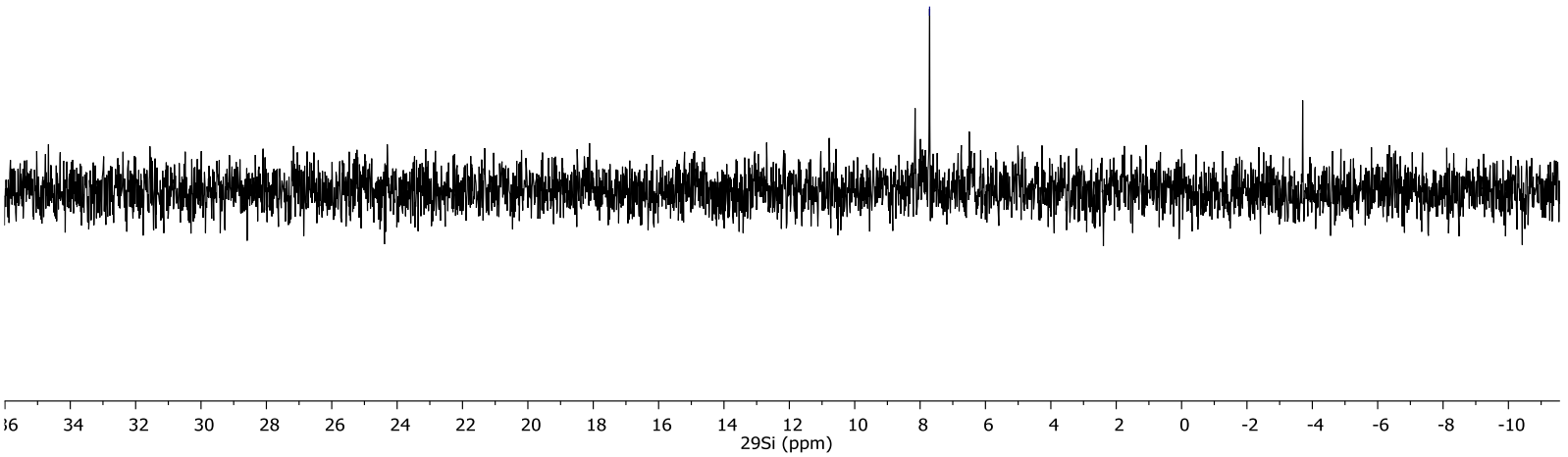


^{13}C NMR of Ligand 11e, 101 MHz, $CDCl_3$, 25ºC

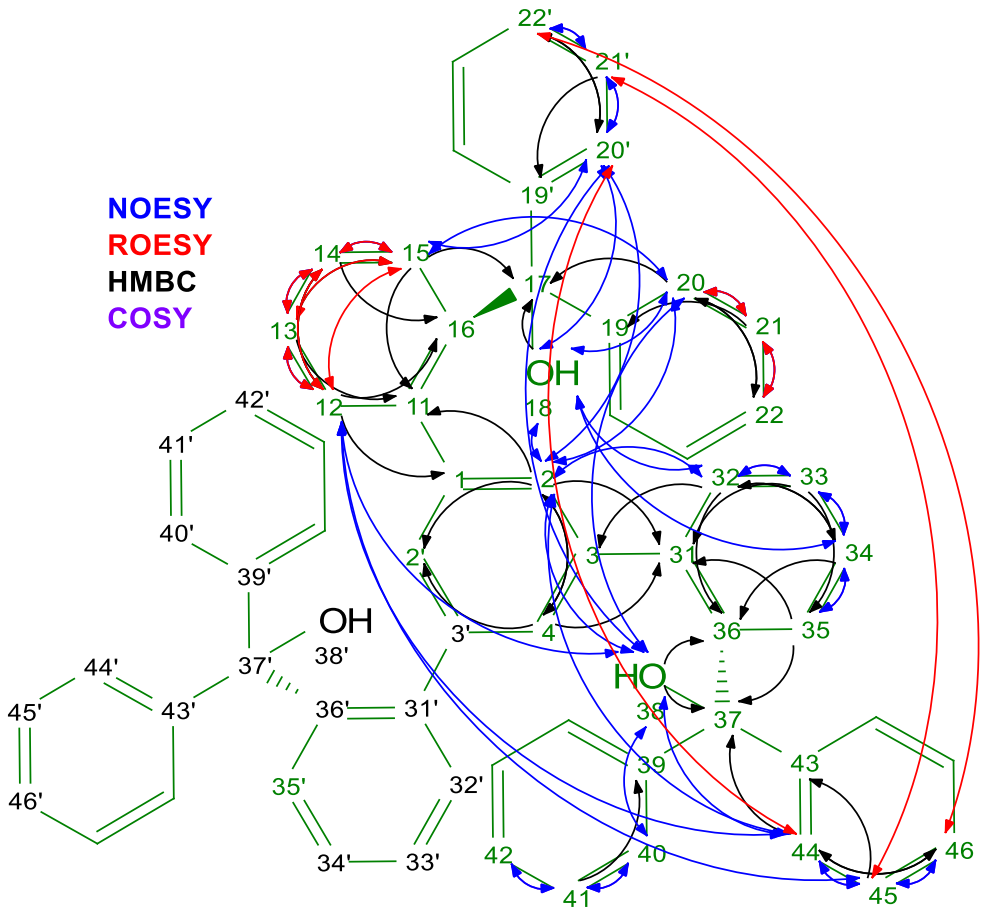


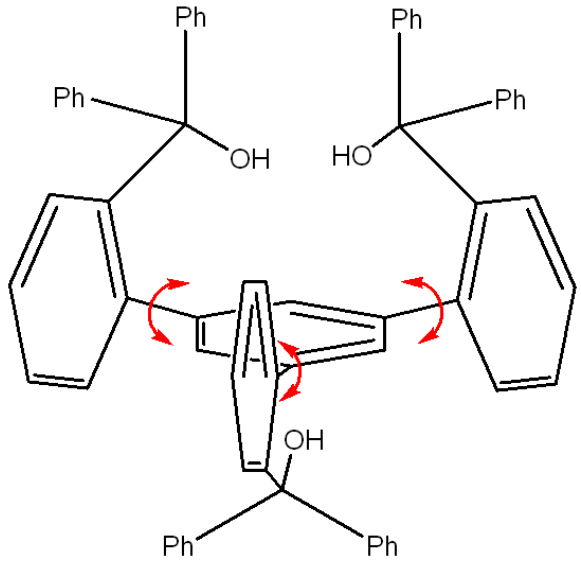
^{29}Si NMR of Ligand 11e, 99 MHz, $CDCl_3$, 25ºC





NMR Studies of Ligand 12, 600 MHz, CD_2Cl_2





C2-symmetry with conformational exchange

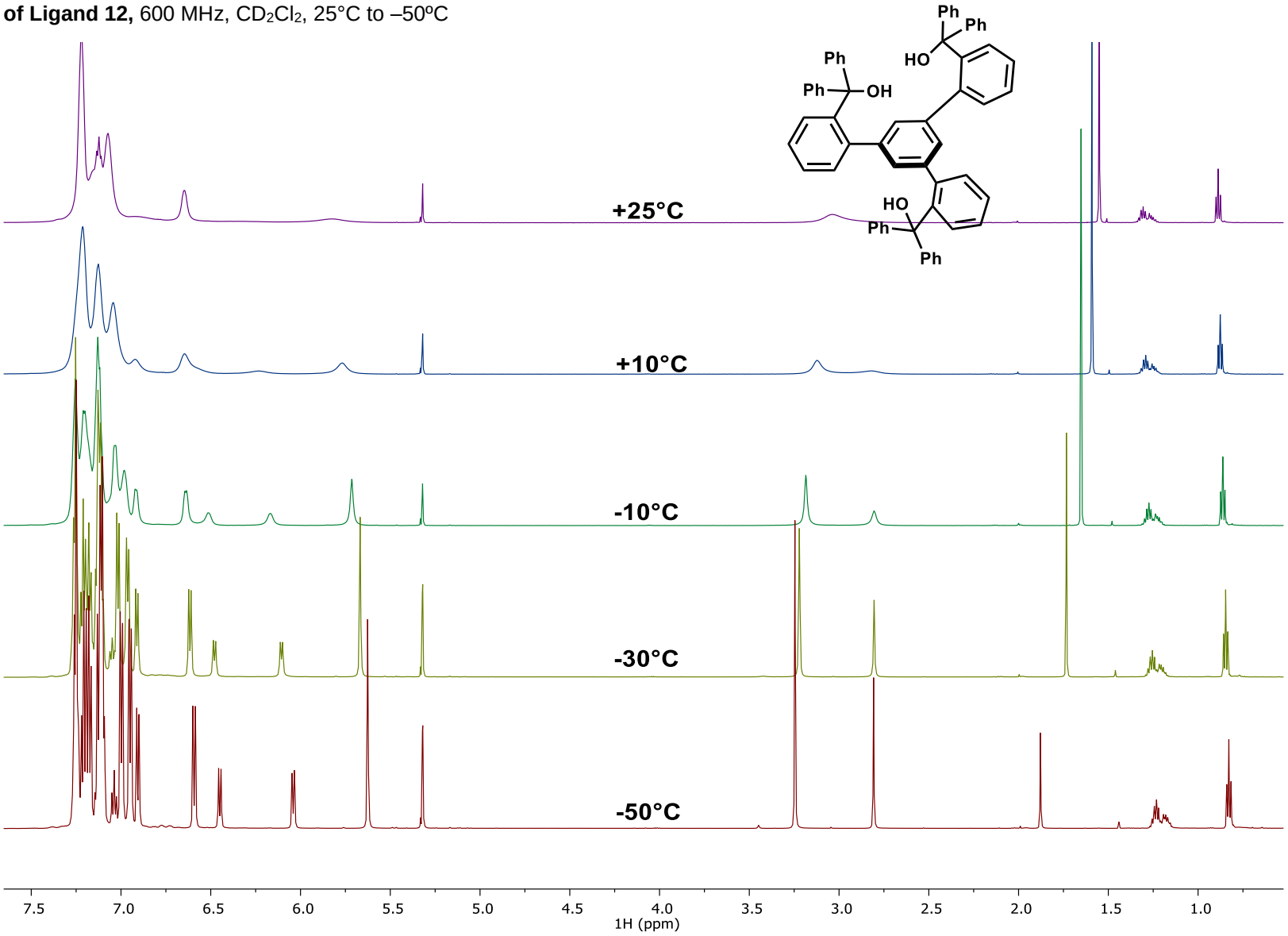
Although this molecule might be expected to have 3-fold symmetry, the NMR results clearly indicate that a 2-fold symmetry is present in CD_2Cl_2 at low temperature (-50°C). The axis of symmetry is indicated by the numbering of the atoms (e.g. 2 and 2'). This symmetry is clearly visible through 2:1 integrals for corresponding 1Hs (e.g. 38OH:18:OH = 2:1). Importantly, this molecule shows int-slow dynamics (on the NMR timescale) which causes the lines to broadened at ambient temperature. At low T, one can differentiate the exchanging nuclei.The symmetry, dynamic exchange and

NOEs are compatible with a model, where the 3 equivalent side-groups are oriented in a [2-up, 1-down]-conformation relative to the central aromatic plane, and where any one of the "up"-side-groups can flips to "down" position, giving a conformation equivalent to the outset (see model).

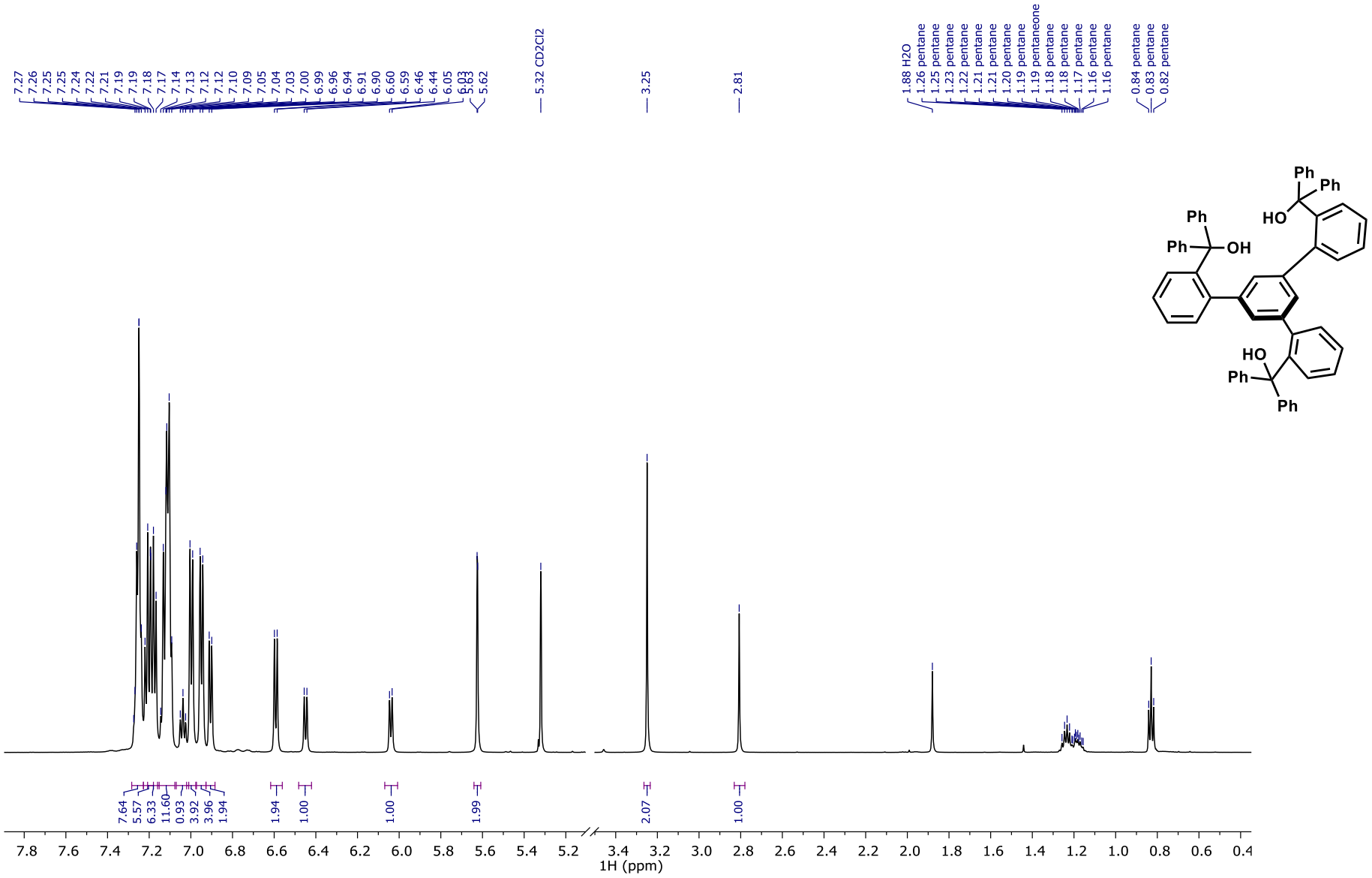
Atom	δ (ppm)	Predicted Shift	J	COSY	HSQC	HMBC	ROESY	EXSY
1 C	139.25	138.79				12		
2 C	129.25	130.27			2	4		
2H	5.62	7.2	2.00(4)		2	2', 4, 11, 31	**18, 20, 20', 32, 38, 44**	
3 C	141.15	138.79				32		
4 C	129.94	130.27			4	2		
4H	7.13	7.2	2.00(2)		4	2, 2', 31		
11 C	139.19	140.78				2, 13, 15		
12 C	133.81	129.56			12	14		
12H	6.04	8.17	7.50(13)	**13**	12	1, 14, 16	**13, 38, 44, 45**	**32**
13 C	126.17	127.94			13			
13H	7.13	7.4	7.50(12)	**12, 14**	13	11, 15	**12, 14**	**33**
14 C	126.06	128.36			14	12		
14H	7.04	7.44	7.90(15)	**13, 15**	14	12, 16	**13, 15**	**34**
15 C	129.45	128.25			15	13		
15H	6.45	7.44	7.90(14)	**14**	15	11, 17	**14, 20, 20'**	**35**
16 C	143.97	145.67				12, 14		
17 C	82.99	79.44				15, 18, 20		
18OH	2.81	2.75				17, 18	**2, 20, 20', 32, 34**	**38**
19 C	147.64	143.04				21		
20 C	127.66	128.27			20	22		
20H	6.95	7.25	7.70(21)	**21**	20	17, 22	**2, 15, 18, 21, 38**	**40**
21 C	127.94	128.63			21			
21H	7.18	7.23	7.70(20)	**20, 22**	21	19	**20, 22**	**41**
22 C	127	127.56			22	20		
22H	7.1	7.24		**21**	22	20	**21**	**42**
19' C	147.64	143.04				21'		
20' C	127.66	128.27			20'	22'		
20'H	6.95	7.25	7.70(21')	**21'**	20'	22'	**2, 15, 18, 21', 38**	**44**
21' C	127.94	128.63			21'			

Atom	δ (ppm)	Predicted Shift	J	COSY	HSQC	HMBC	ROESY	EXSY
21'H	7.18	7.23	7.70(20')	**20', 22**	21'	19'	**20', 22'**	**45**
22' C	127	127.56			22'	20'		
22'H	7.1	7.24		**21'**	22'	20'	**21'**	**46**
31 C	140.78	140.78				2, 4, 33, 35		
32 C	132.52	129.55			32	34		
32H	6.91	8.17	7.40(33)	**33**	32	3, 34, 36	**2, 18, 33**	**12**
33 C	126.82	127.94			33			
33H	7.25	7.4	7.40(32)	**32, 34**	33	31, 35	**32, 34**	**13**
34 C	126.31	128.36			34	32		
34H	7.12	7.44	7.90(35')	**33, 35**	34	32, 36	**18, 33, 35**	**14**
35 C	129.66	128.25			35	33		
35H	6.59	7.44		**34**	35	31, 37	**34**	**15**
36 C	144.38	145.67				32, 34, 38		
37 C	83.07	79.44				35, 38, 44		
38OH	3.25	2.75				36, 37	**2, 12, 20, 20', 40, 44**	**18**
39 C	147.1	143.04				41		
40 C	128.29	128.27			40			
40H	7.11	7.25		**41**	40		**38, 41**	**20**
41 C	127.87	128.63			41			
41H	7.26	7.23		**40, 42**	41	39	**40, 42**	**21**
42 C	127.43	127.56			42			
42H	7.25	7.24		**41**	42		**41**	**22**
43 C	147.55	143.04				45		
44 C	127.35	128.27			44	46		
44H	7	7.25	7.70(45)	**45**	44	37, 46	**2, 12, 38, 45**	**20'**
45 C	127.95	128.63			45			
45H	7.21	7.23	7.70(44)	**44, 46**	45	43	**12, 44, 46**	**21'**
46 C	127.02	127.56			46	44		
46H	7.1	7.24		**45**	46	44	**45**	**22'**

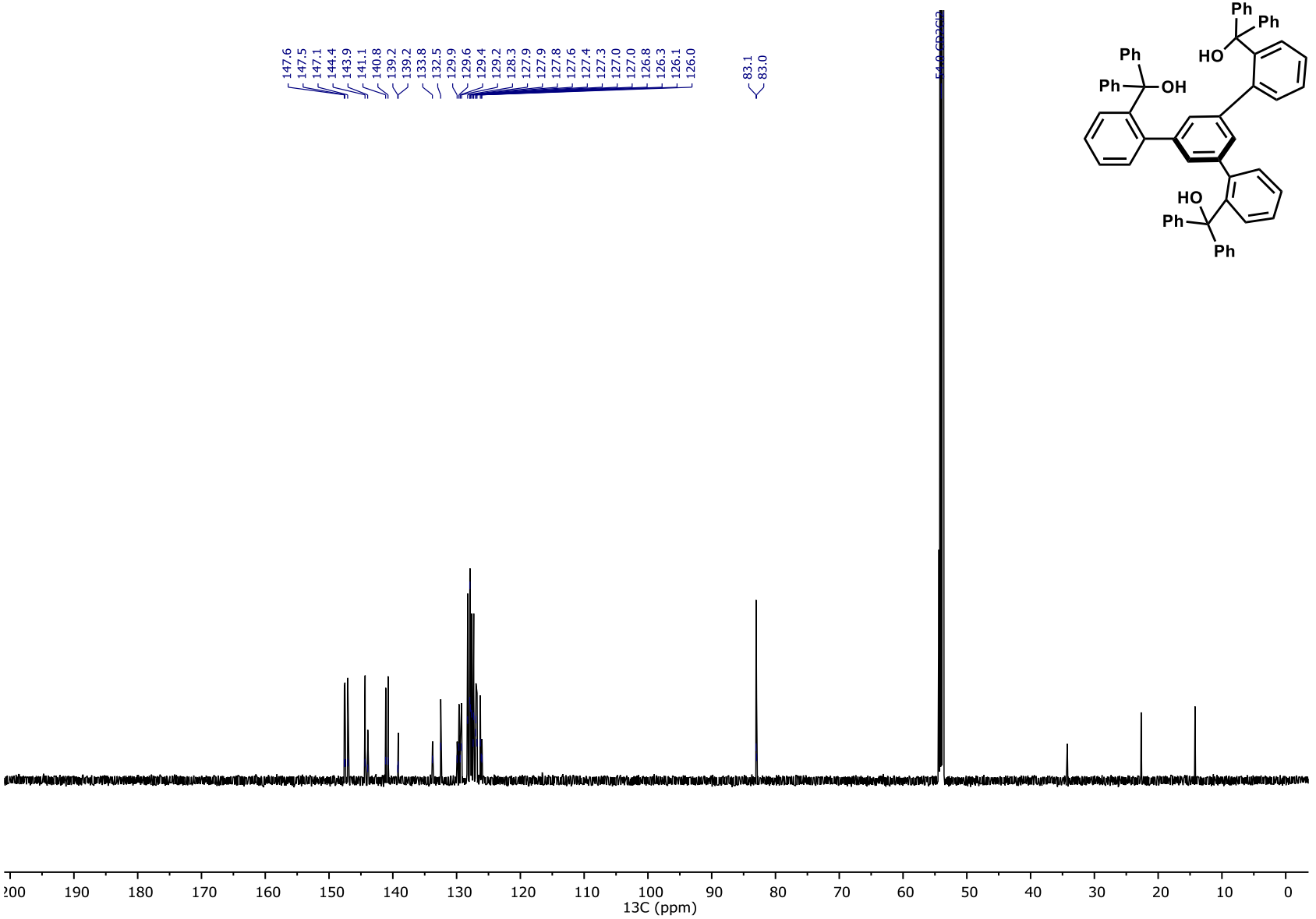
VT ^{1}H NMR of Ligand 12, 600 MHz, CD_2Cl_2, 25°C to –50ºC



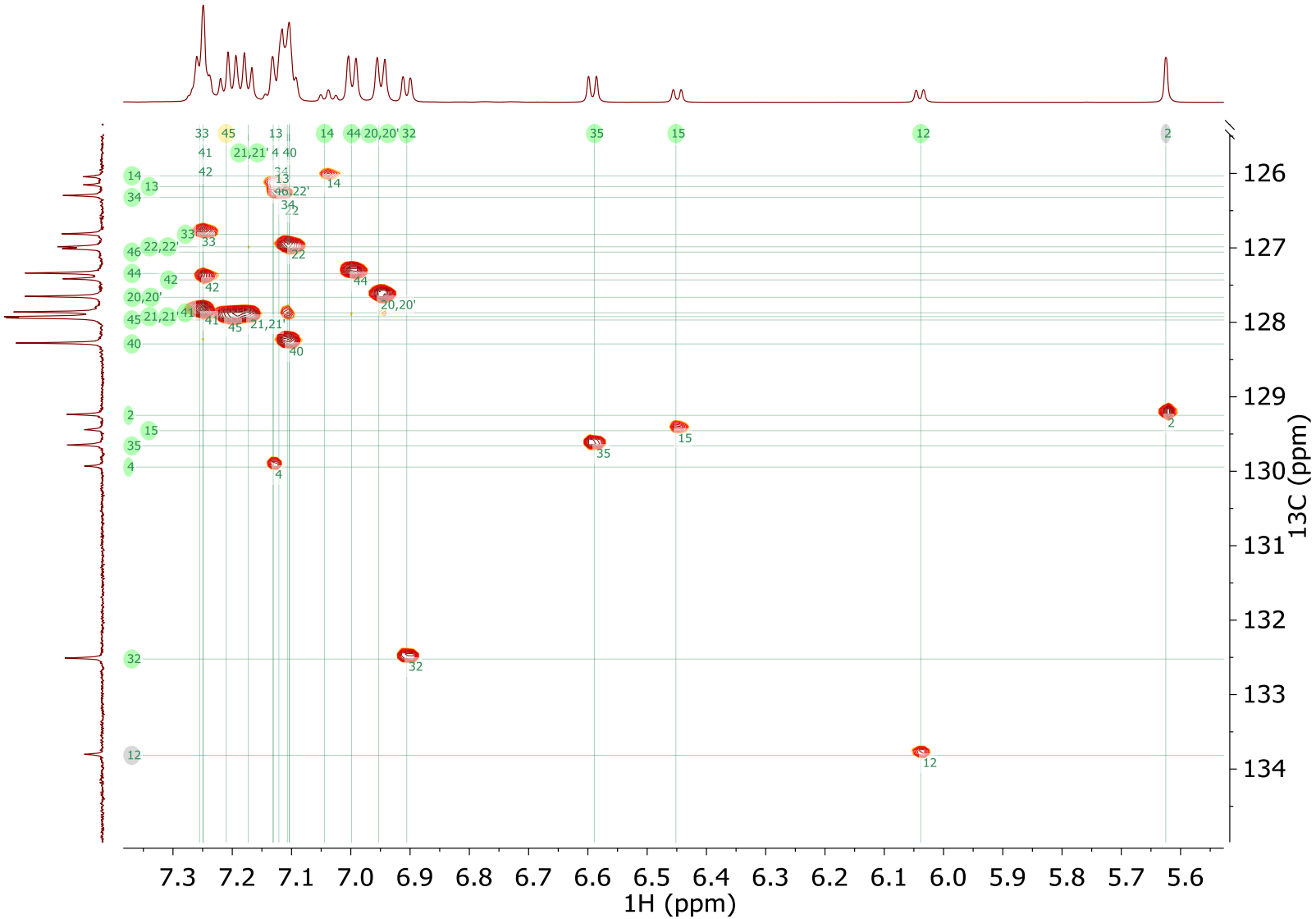
^{1}H NMR of Ligand 12, 600 MHz, CD_2Cl_2, –50ºC



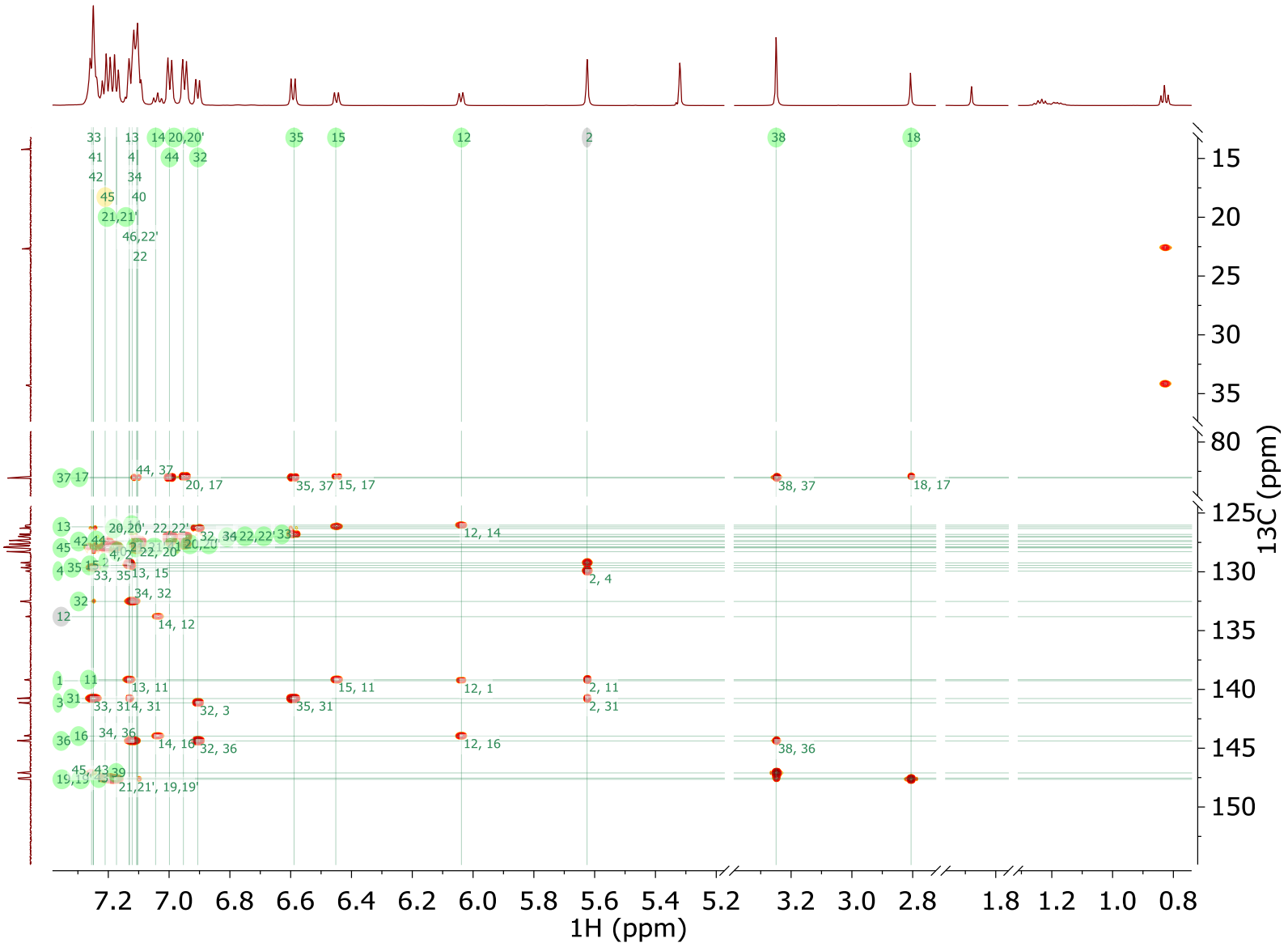
^{13}C NMR of Ligand 12, 151 MHz, CD_2Cl_2, –50ºC



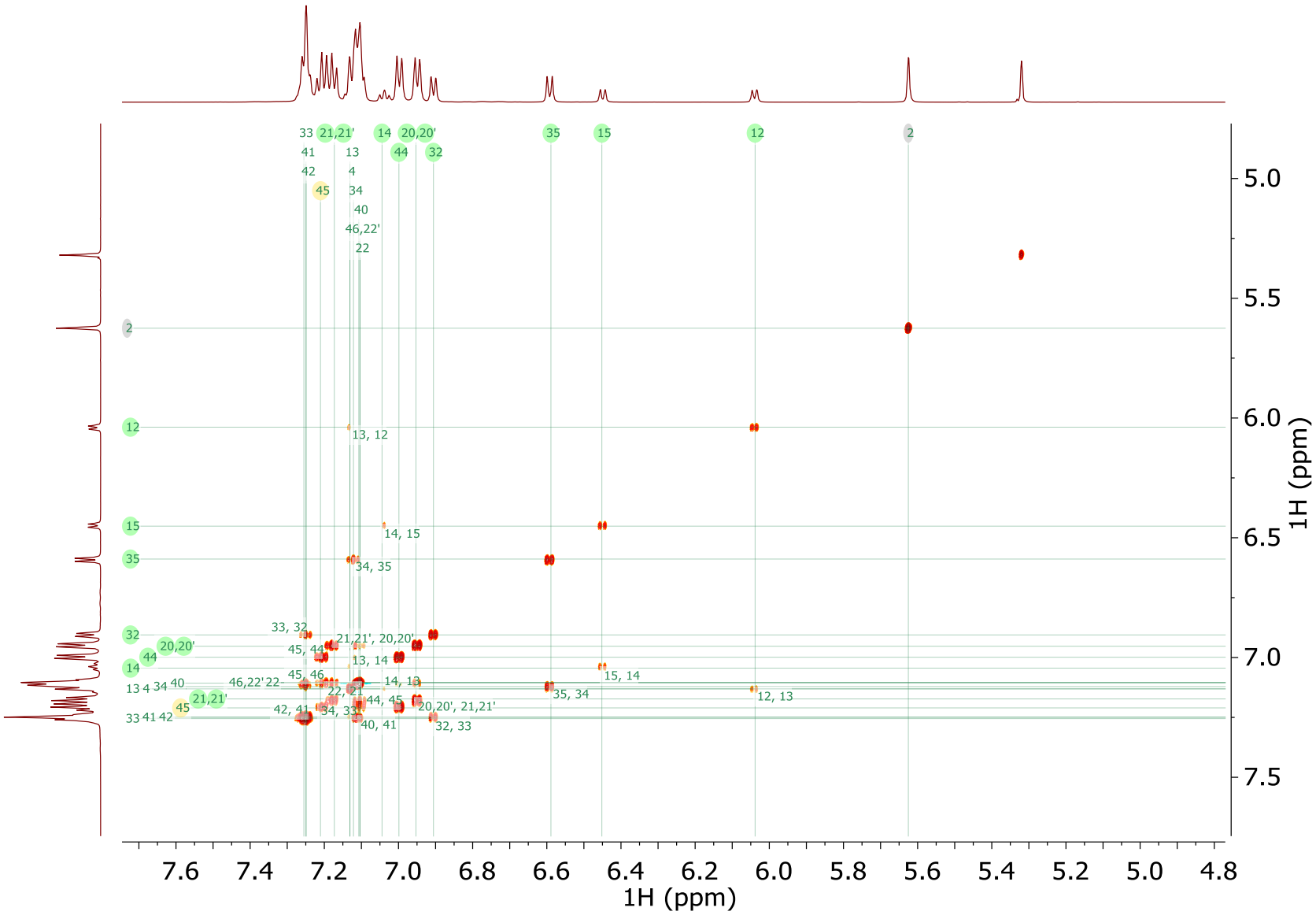
^{1}H-^{13}C HSQC NMR of Ligand 12, CD_2Cl_2, –50ºC



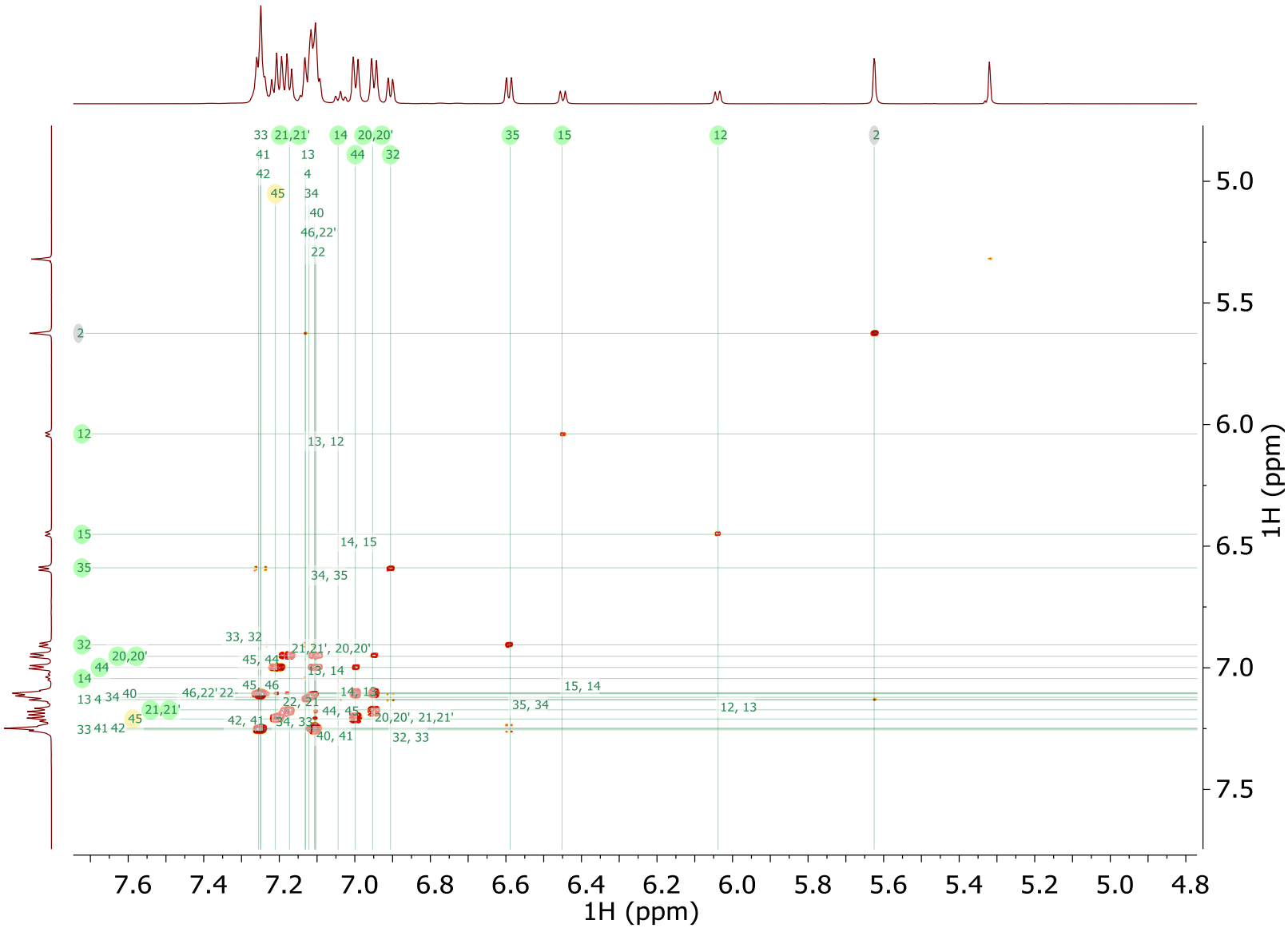
^{1}H-^{13}C HMBC NMR of Ligand 12, CD_2Cl_2, –50ºC



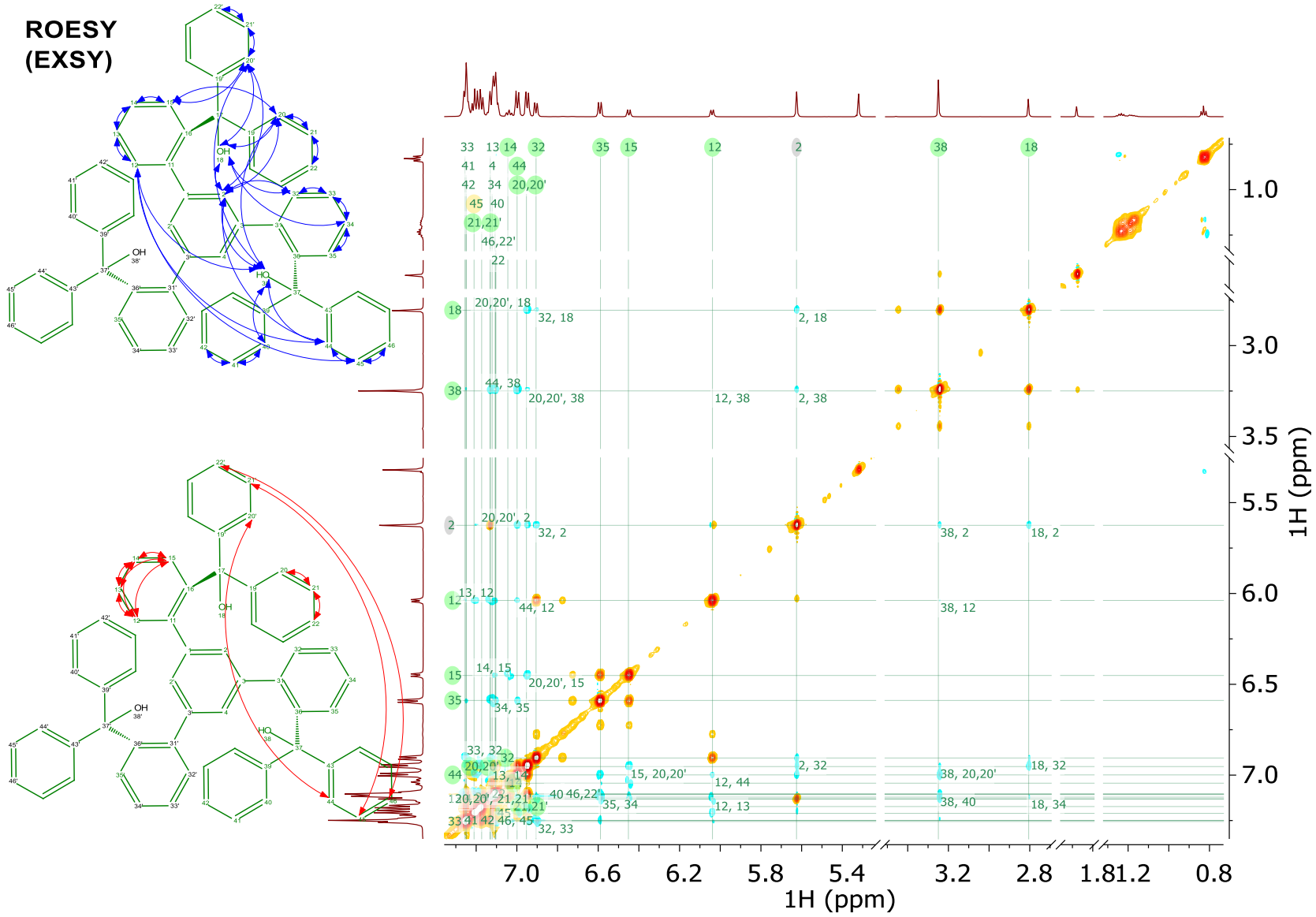
^{1}H COSY NMR of Ligand 12, CD_2Cl_2, –50ºC



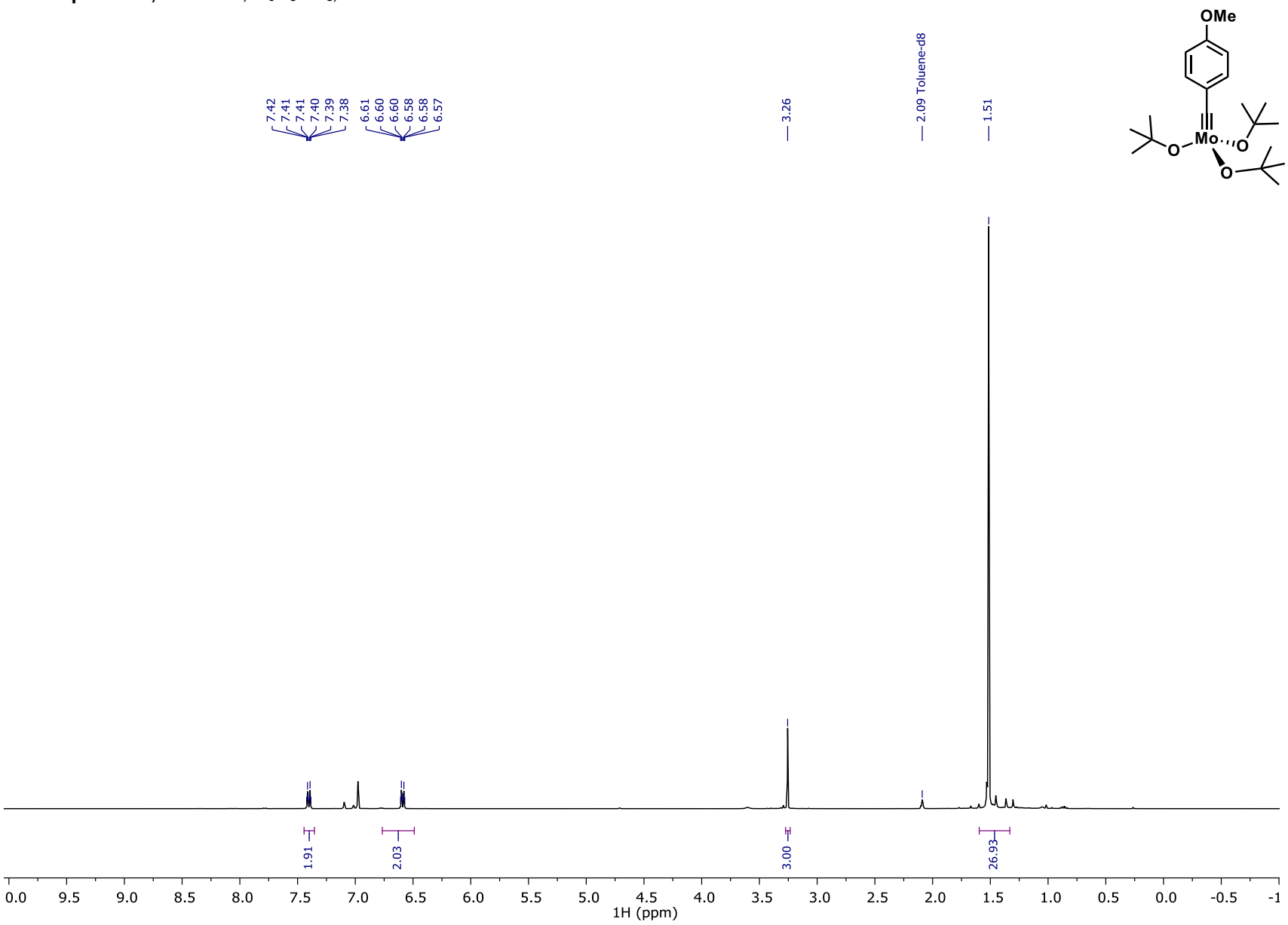
^{1}H TOSY NMR of Ligand 12, CD_2Cl_2, –50ºC



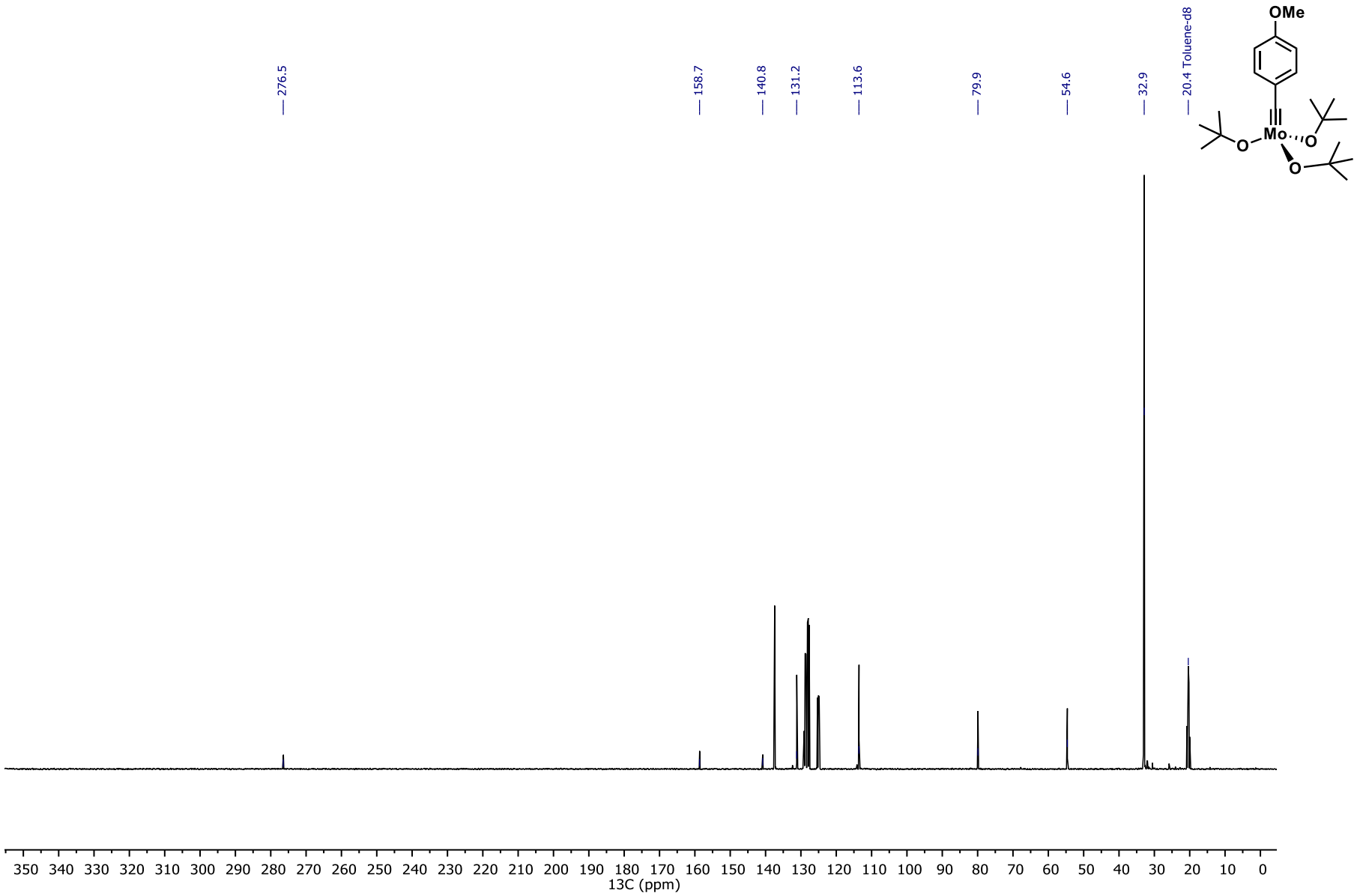
^{1}H ROESY NMR of Ligand 12, CD_2Cl_2, –50ºC



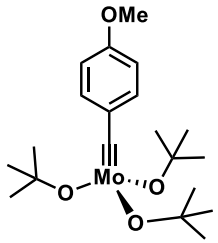
^{1}H NMR of Complex 14a, 400 MHz, $C_6D_5CD_3$, 25ºC



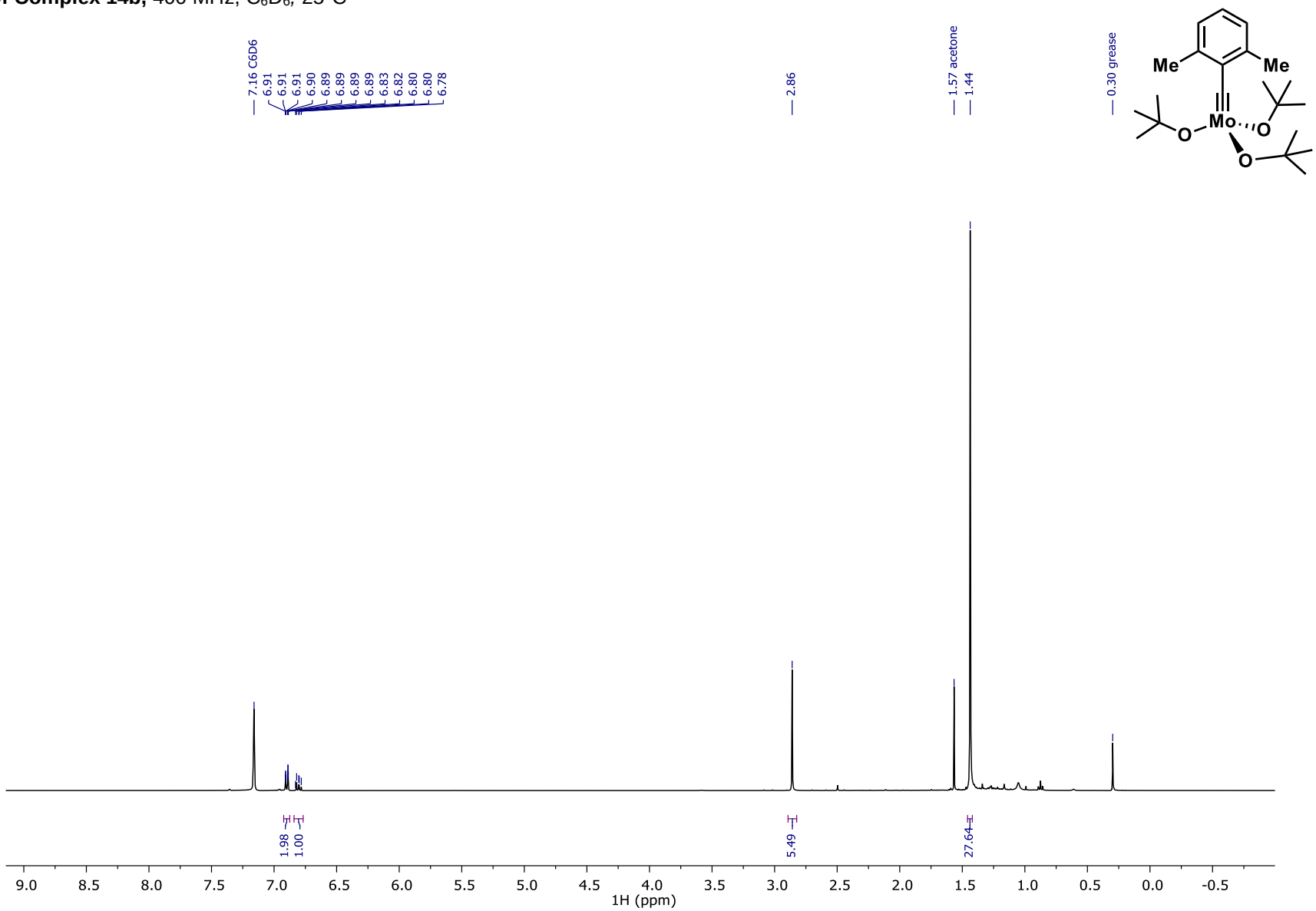
^{13}C NMR of Complex 14a, 101 MHz, $C_6D_5CD_3$, 25°C



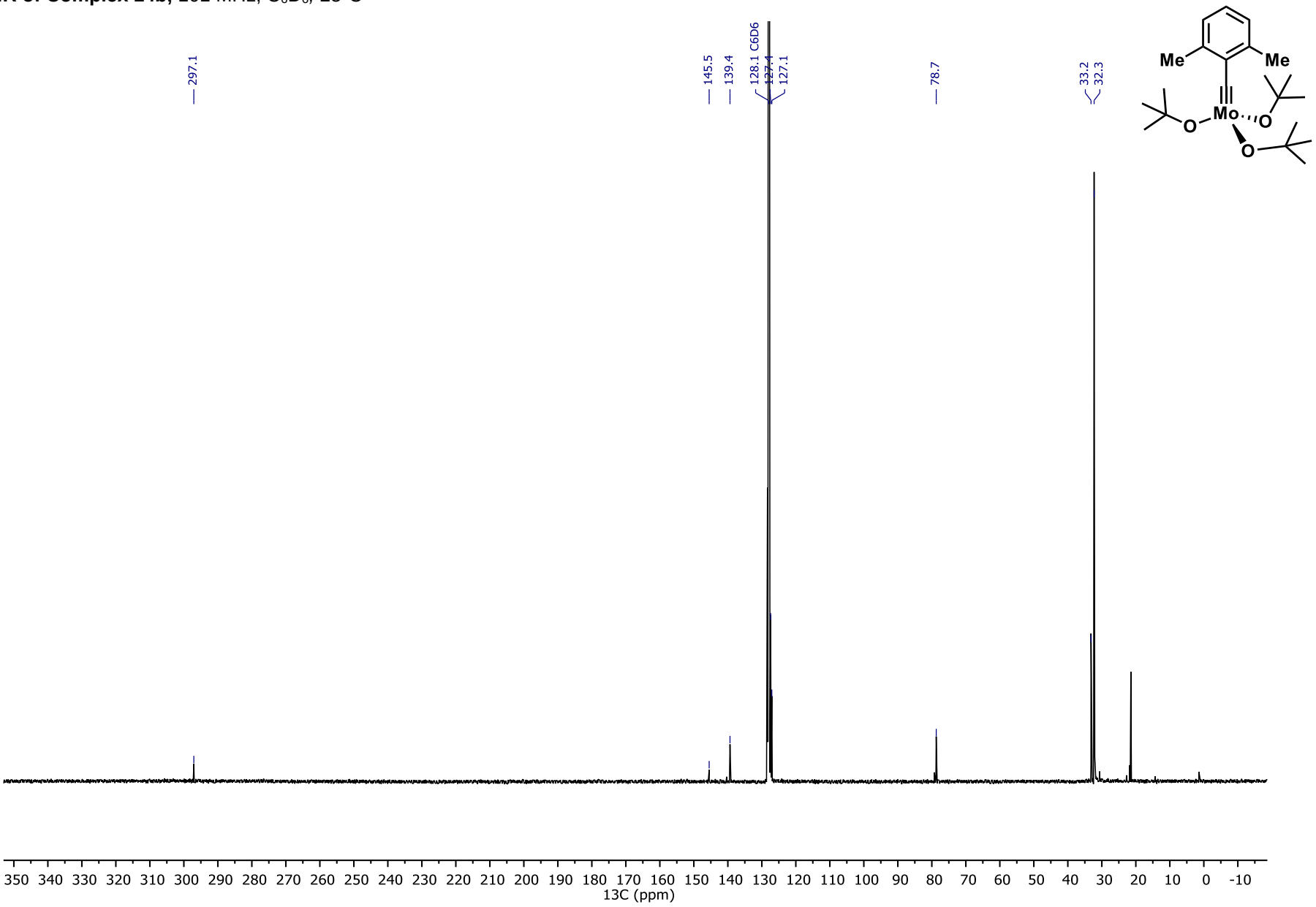
^{95}Mo NMR of Complex 14a, 26 MHz, $C_6D_5CD_3$, 60^{o}C



^{1}H NMR of Complex 14b, 400 MHz, C_6D_6, 25ºC



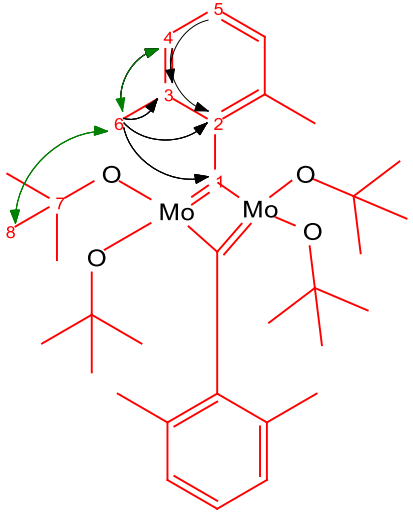
^{13}C NMR of Complex 14b, 101 MHz, C_6D_6, 25ºC

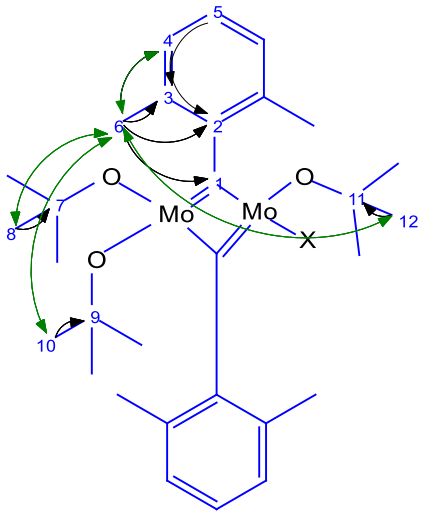


NMR Assignment Table of Dinuclear Complex 15b, $C_6D_5CD_3$, 25 ºC

Atom	δ (ppm)	HSQC	HMBC	ROESY
1 C	341.28		6	
2 C	151.35		4, 6	
3 C	131.93		5, 6	
4 C	128.58	4	4, 6	
H	7.18	4	2, 4	6
5 C	126.62	5		
H	7.02	5	3	
6 C	24.74	6		
H3	2.98	6	1, 2, 3, 4	4, 8
7 C	80.78			
8 C	31.85	8	8	
H3	0.98	8	8	6

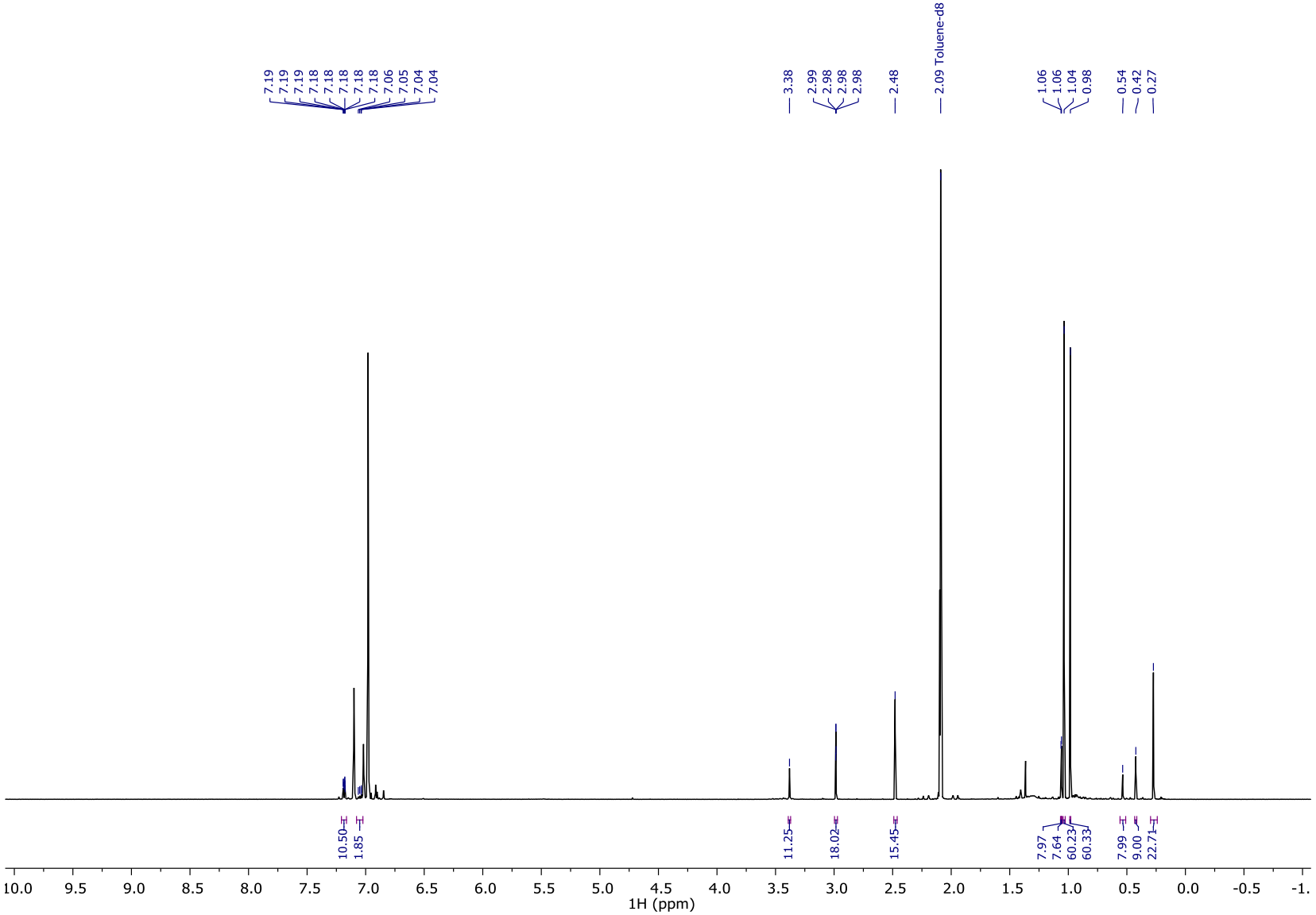
HMBC cross peaks
ROESY cross peaks



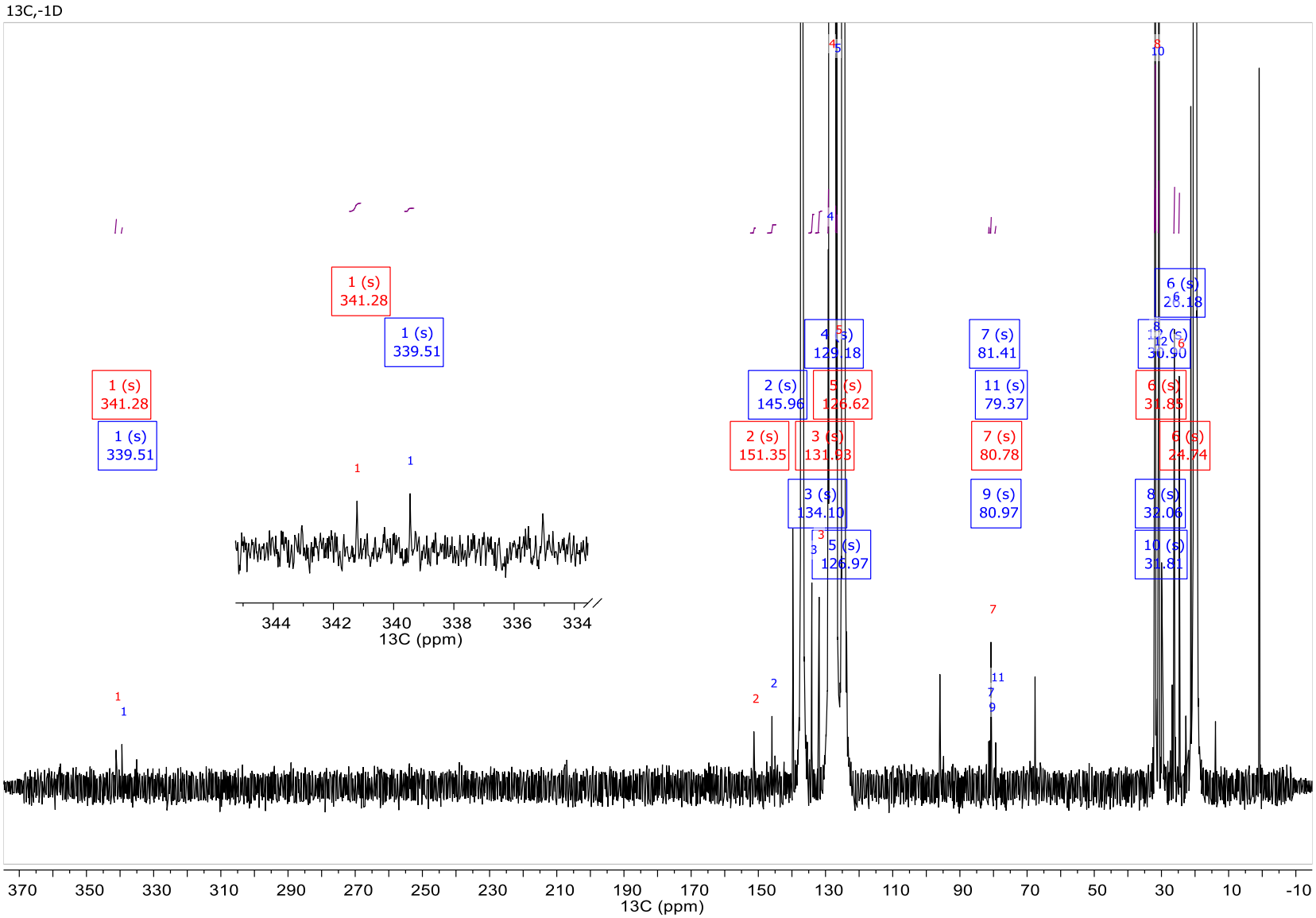


Atom	δ (ppm)	HSQC	HMBC	ROESY
1 C	339.51		6	
2 C	145.96		4, 6	
3 C	134.1		5, 6	
4 C	129.18	4	4, 6	
H	7.18	4	2, 4	6
5 C	126.97	5		
H	7.05	5	3	
6 C	26.18	6		
H3	3.38	6	1, 2, 3, 4	4, 8, 10, 12
7 C	81.41		8	
8 C	32.06	8	8	
H3	1.06	8	7, 8	6
9 C	80.97		10	
10 C	31.81	10	10	
H3	1.06	10	9, 10	6
11 C	79.37		12	
12 C	30.9	12	12	
H3	0.42	12	11, 12	6

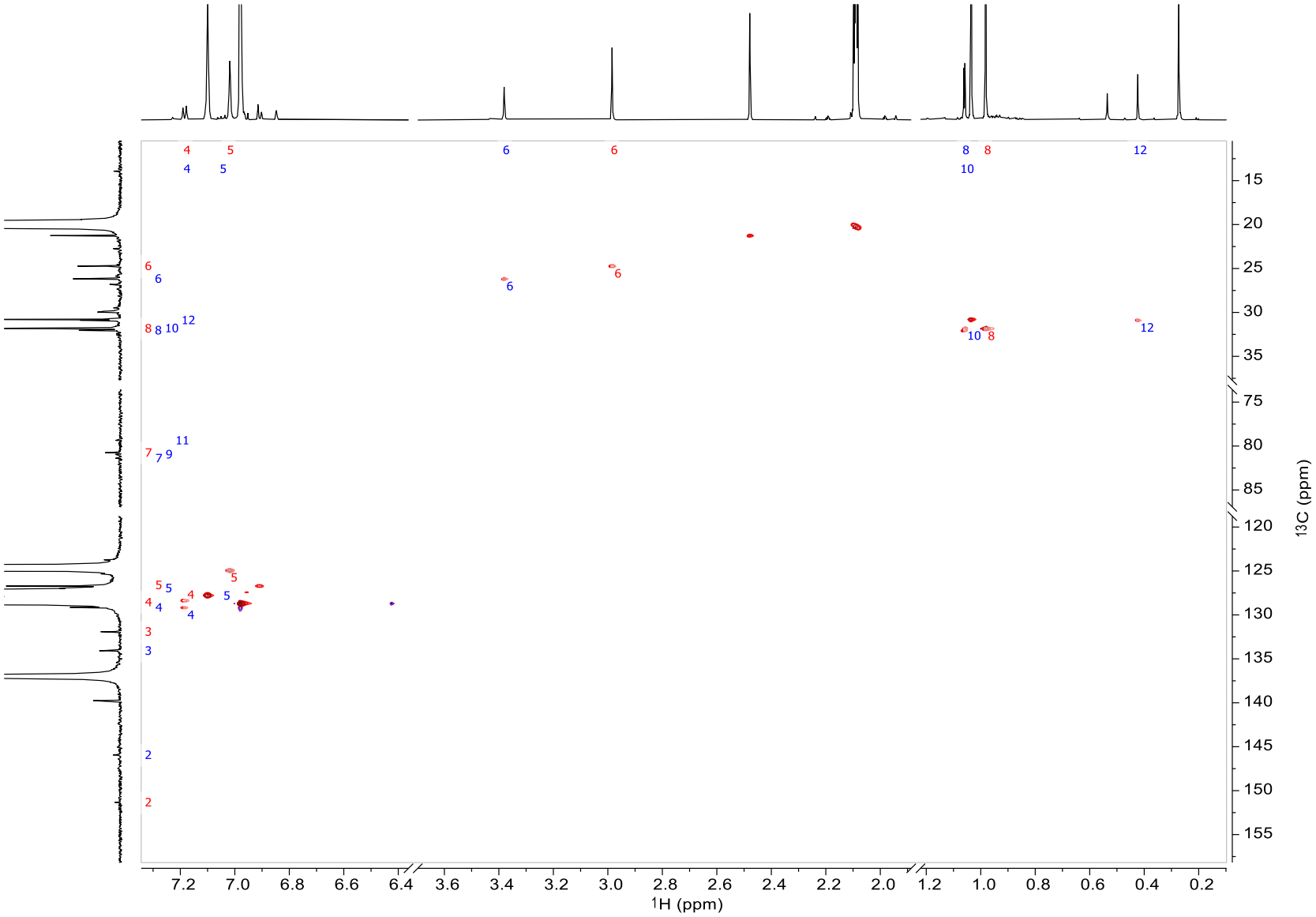
^{1}H NMR of Dinuclear Complex 15b, 600 MHz**,** $C_6D_5CD_3$, 25ºC



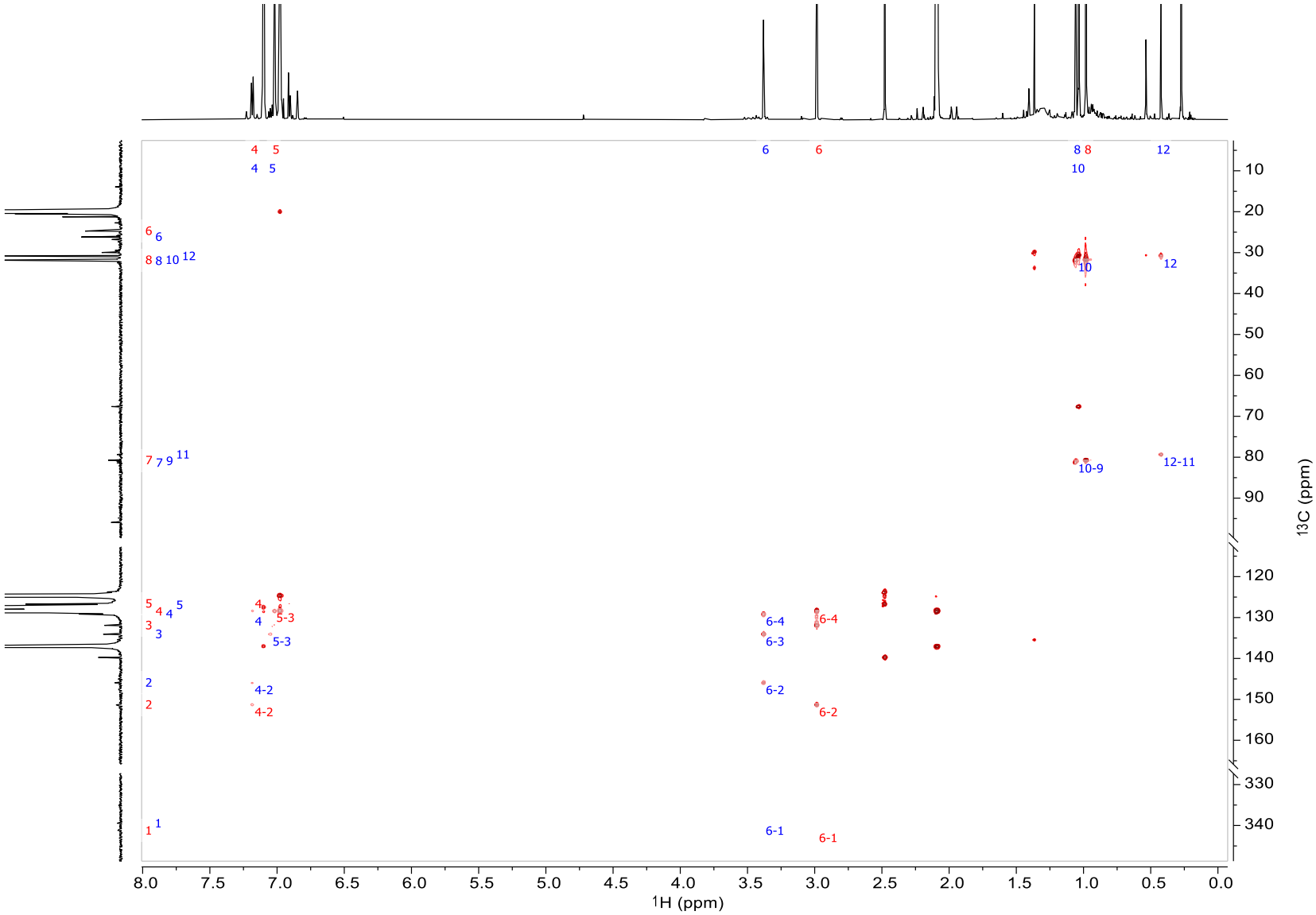
^{13}C NMR of Dinuclear Complex 15b, 151 MHz, $C_6D_5CD_3$, 25°C



^{1}H-^{13}C edited HSQC NMR of Dinuclear Complexe 15b, $C_6D_5CD_3$, 25 °C

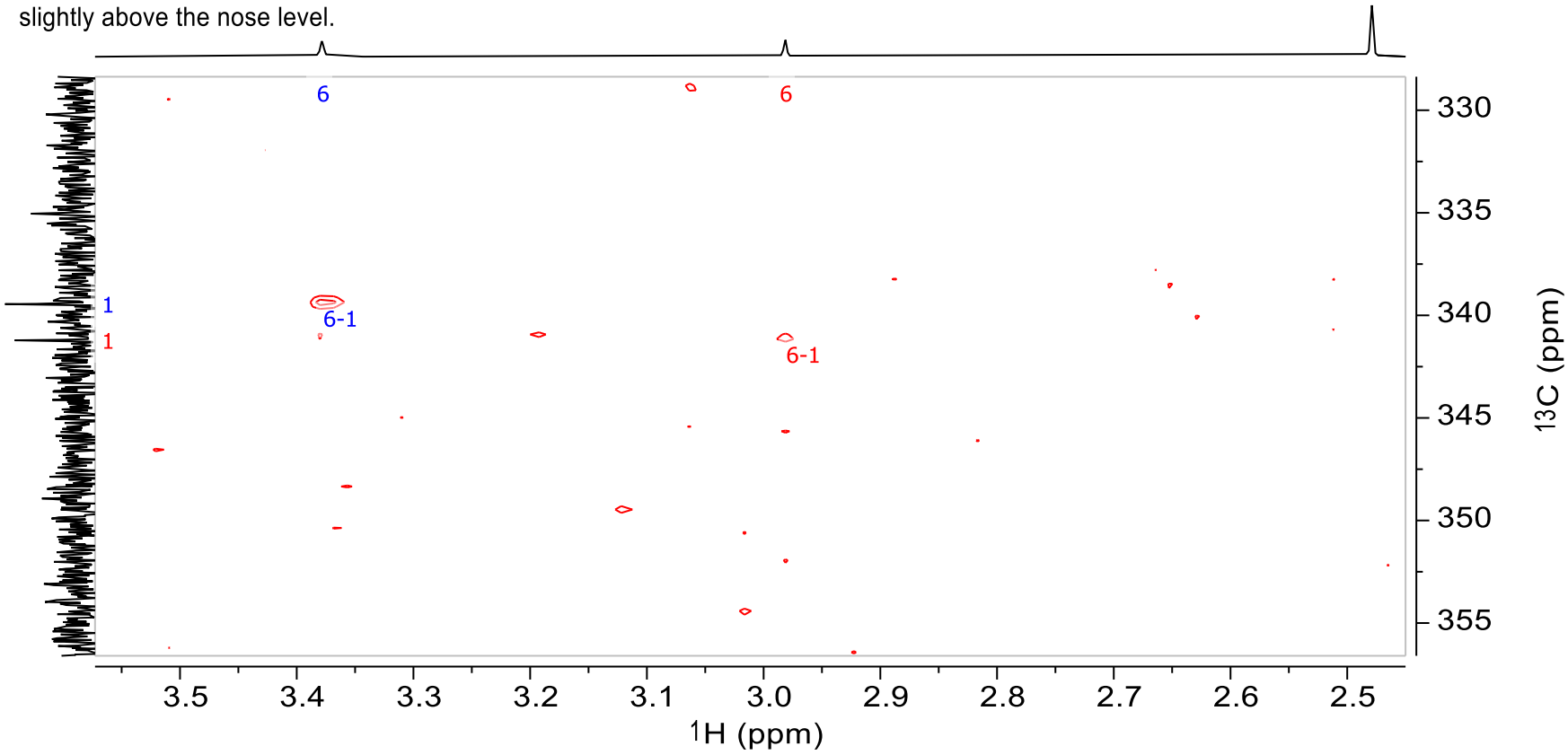


^{1}H-^{13}C HMBC NMR of Dinuclear Complex 15b, $C_6D_5CD_3$, 25 °C

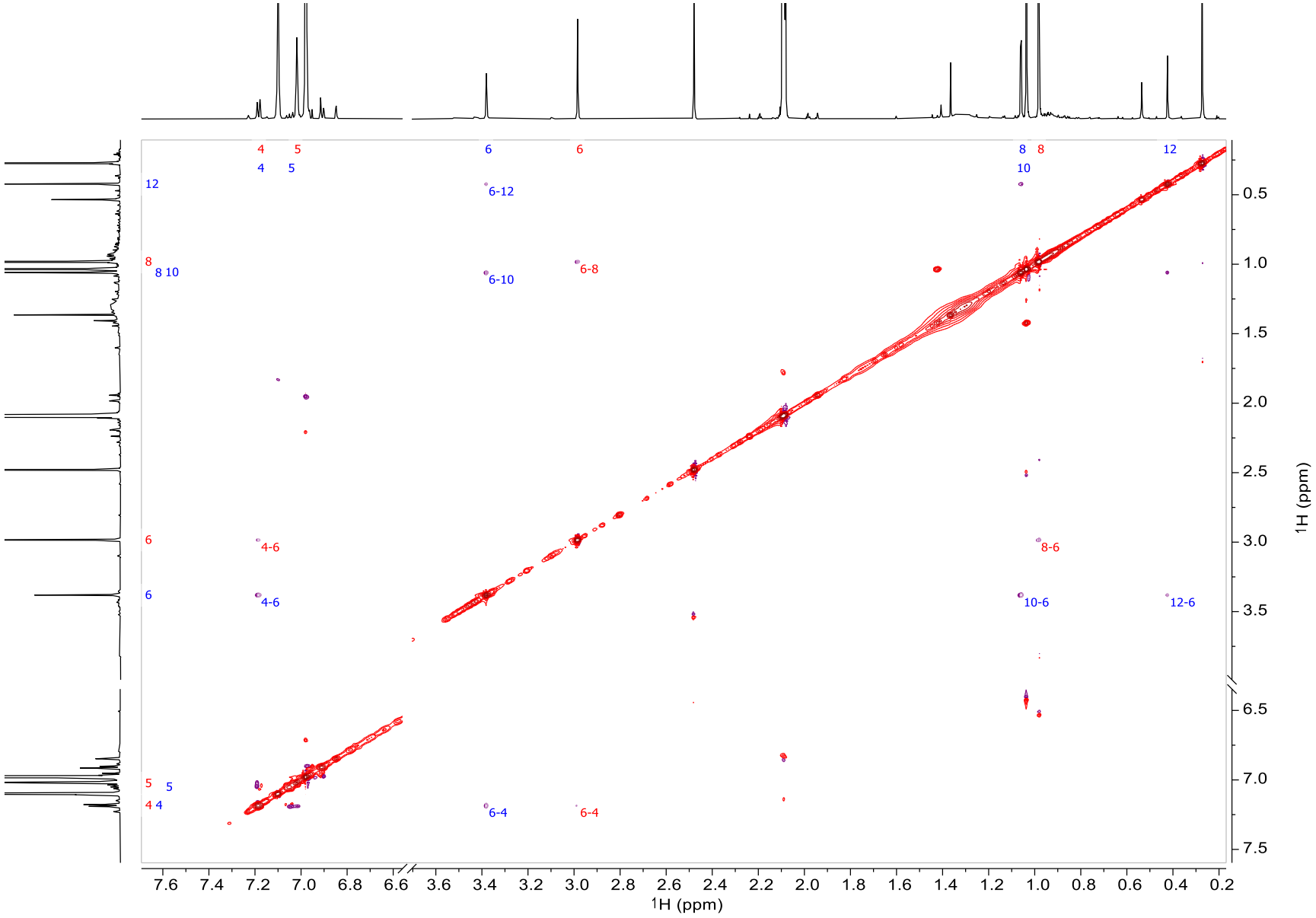


^{1}H-^{13}C HMBC NMR Studies of Dinuclear Complex 15b, $C_6D_5CD_3$, 25 °C

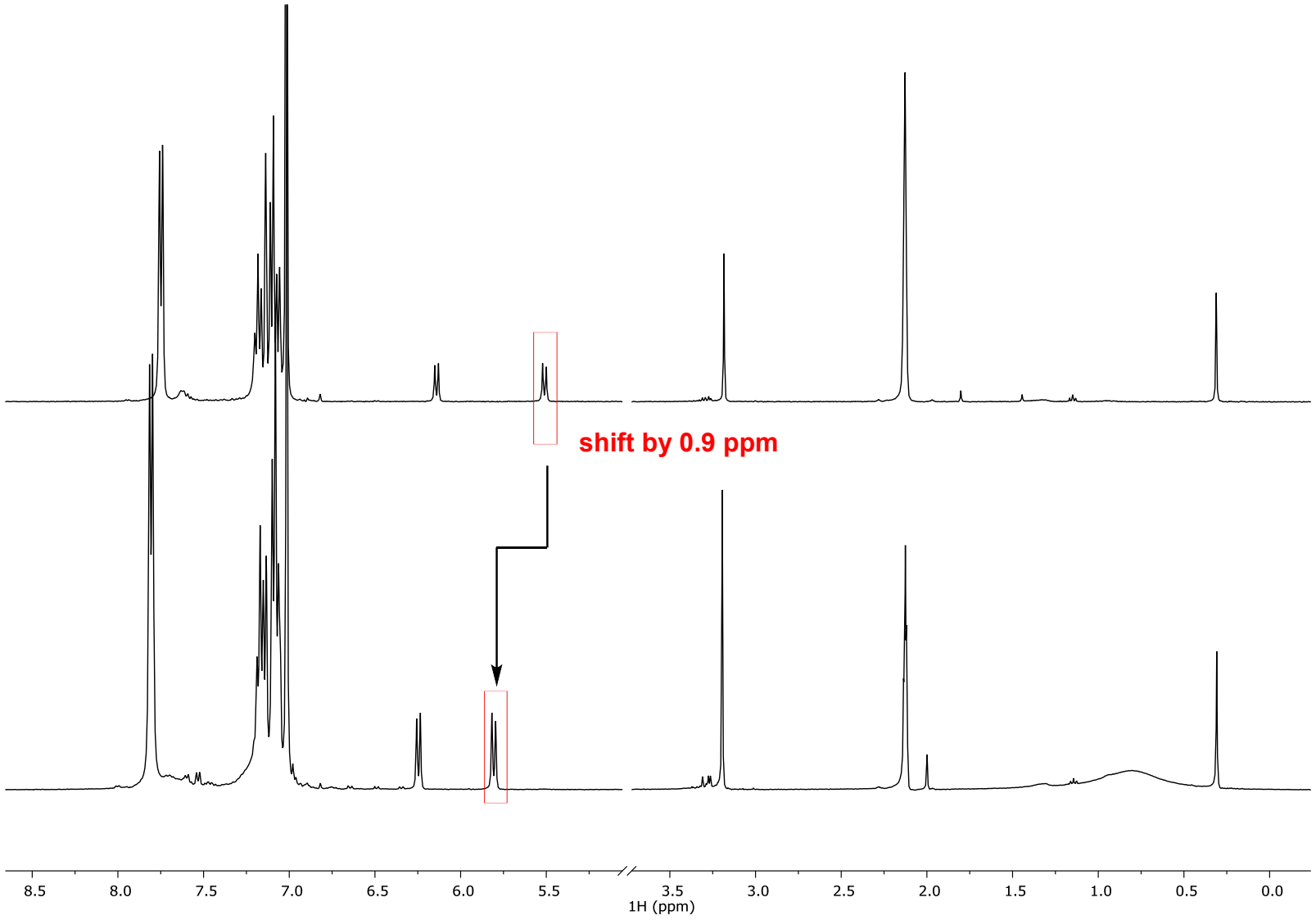
This HMBC was measured at the with optimized offset and transfer delay for 16h, where the very weak peaks become visible slightly above the nose level.

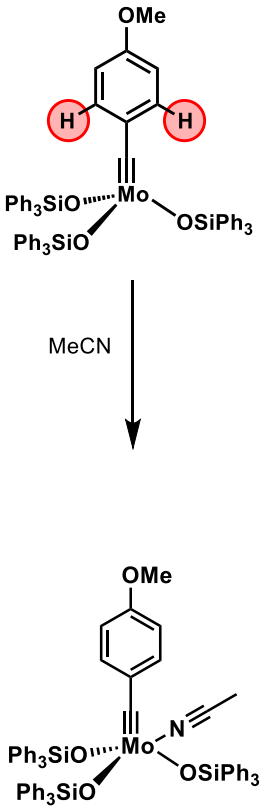


H EASY ROESY NMR Studies of Dinuclear Complex 15b, $C_6D_5CD_3$, 25ºC

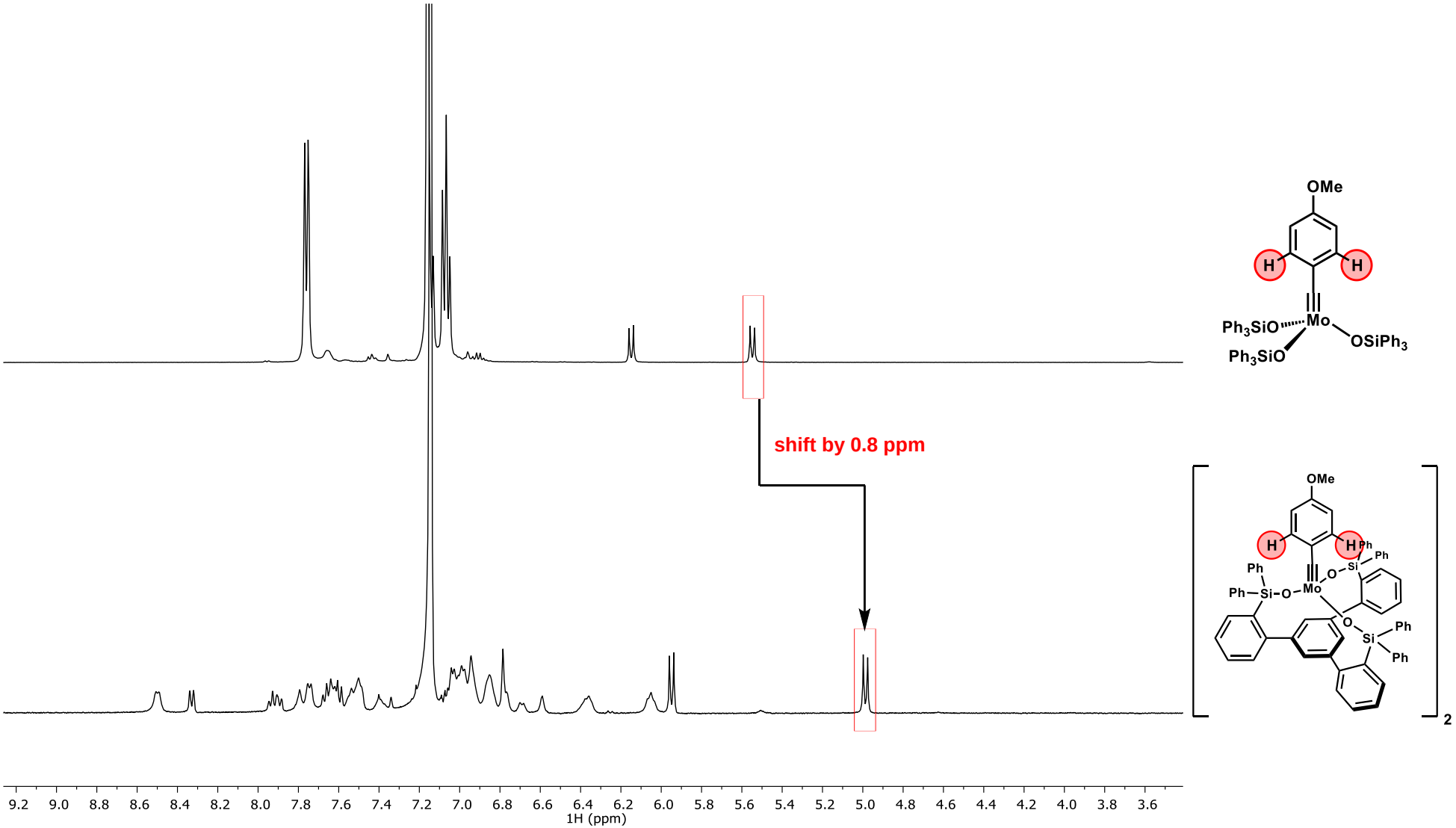


^{1}H NMR Studies of Complex 2a: Disturbing of C–H/π interaction by addition of MeCN, $C_6D_5CD_3$, 25°C

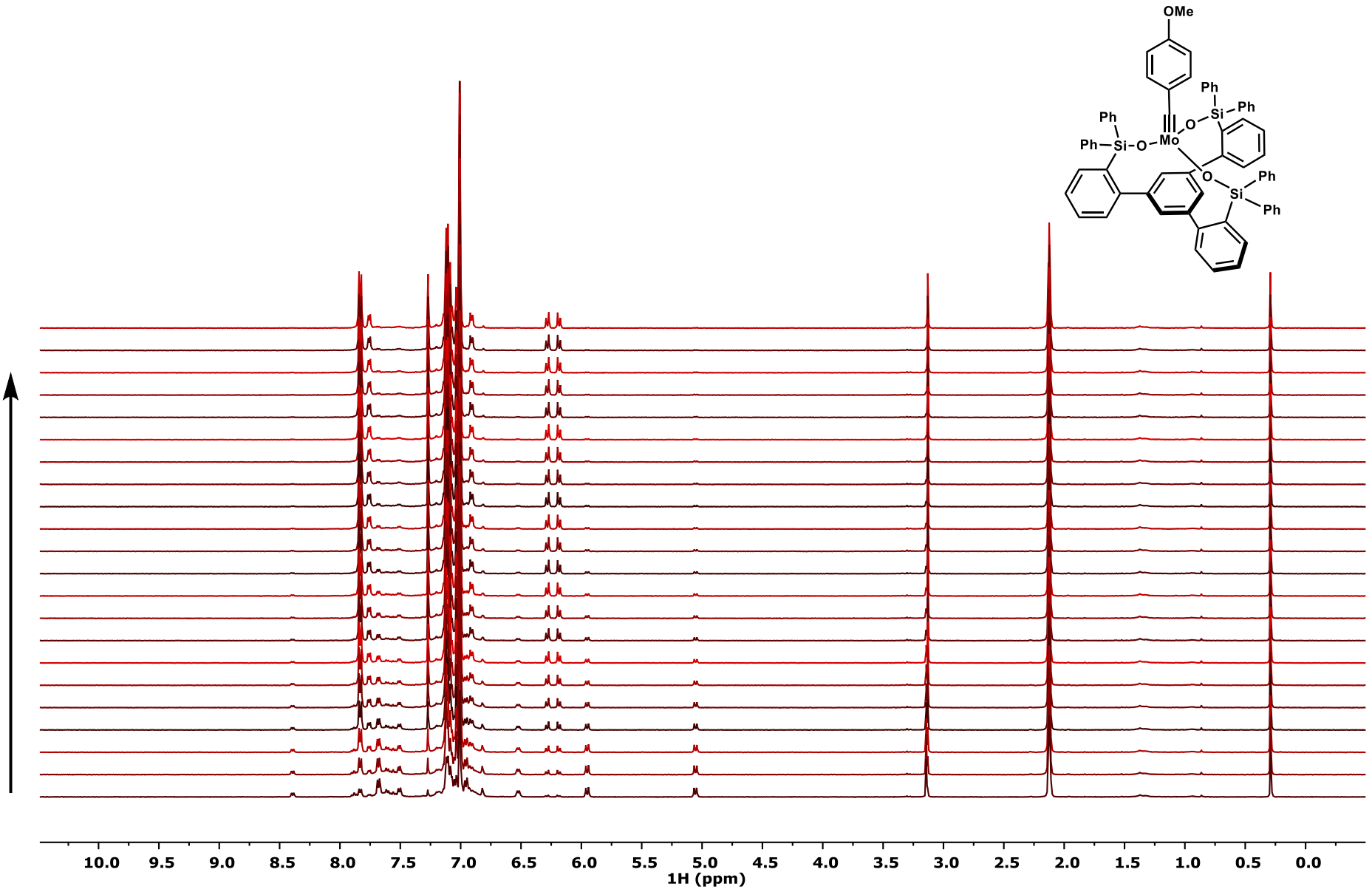




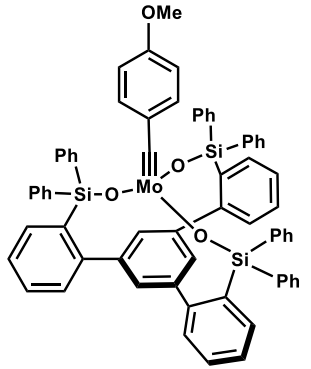
^{1}H NMR Studies of Complexes 1a and [2a]$_2$, 400 MHz, $C_6D_5CD_3$, 25ºC



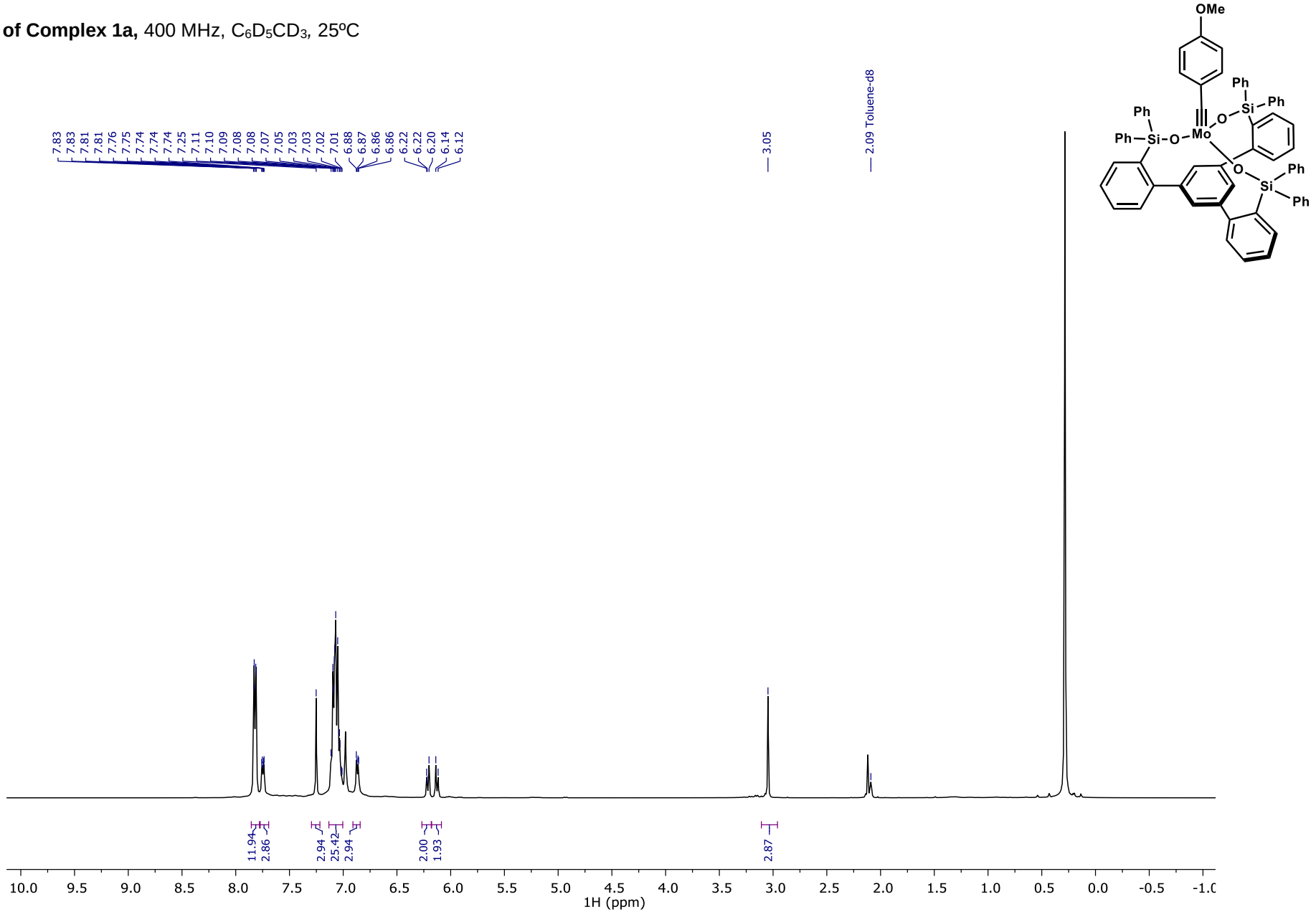
^{1}H NMR Conversion Studies of Complex $[1a]_2$ into Complex 1a, 400 MHz, $C_6D_5CD_3$, 50ºC



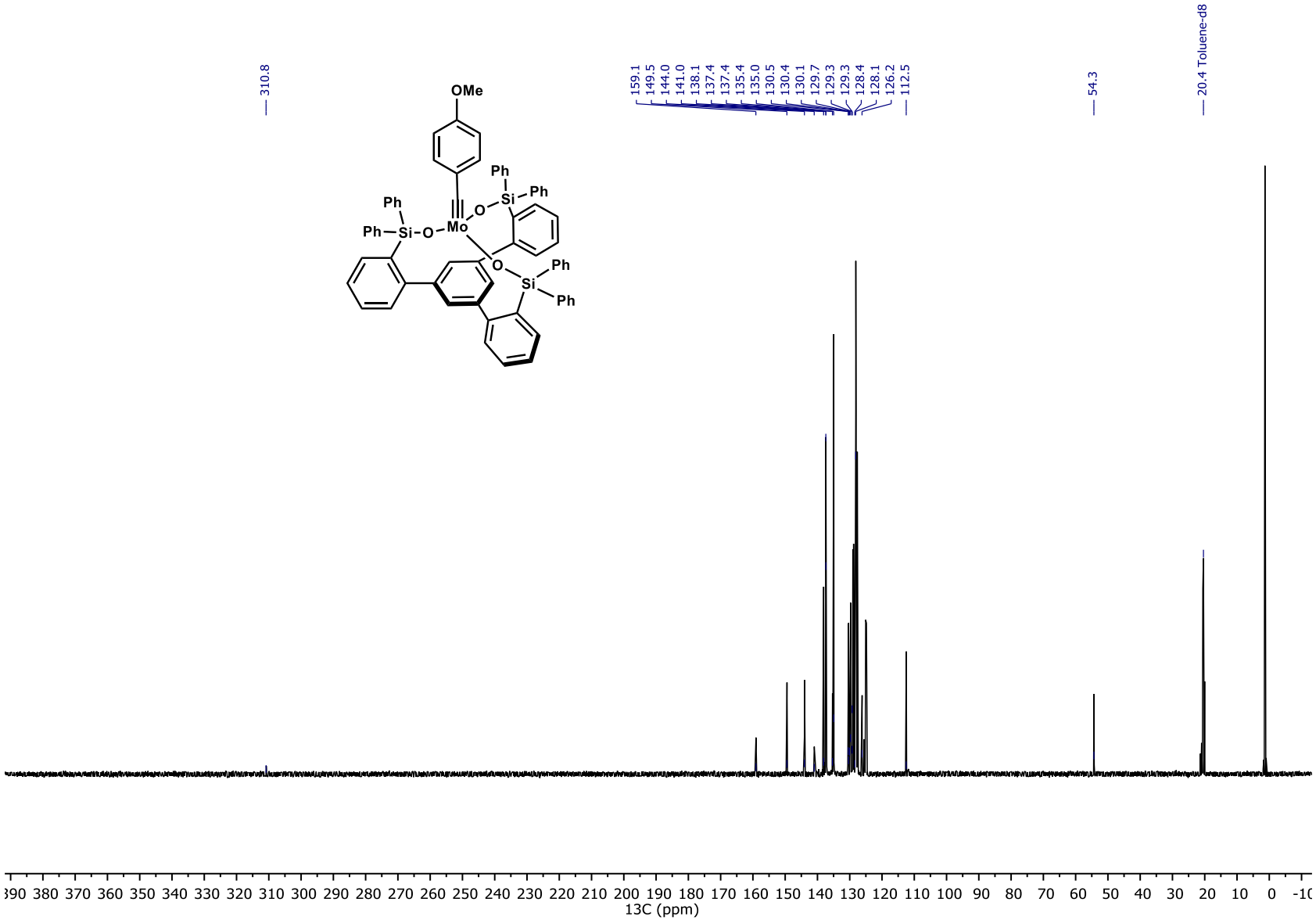
^{1}H NMR Conversion Studies of Complex $[1a]_2$ into Complex 1a, 400 MHz, $C_6D_5CD_3$, 50ºC



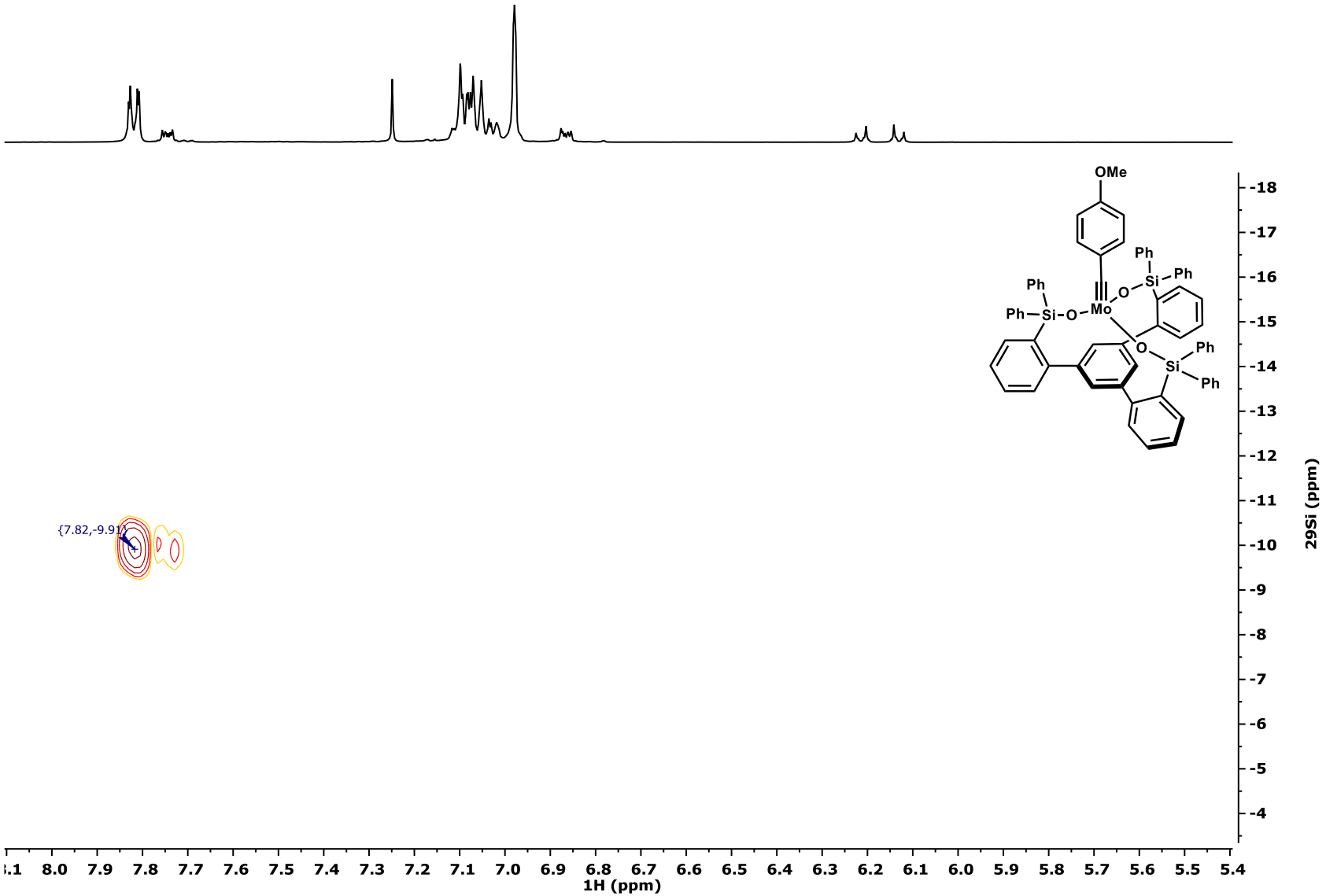
^{1}H NMR of Complex 1a, 400 MHz, $C_6D_5CD_3$, 25ºC



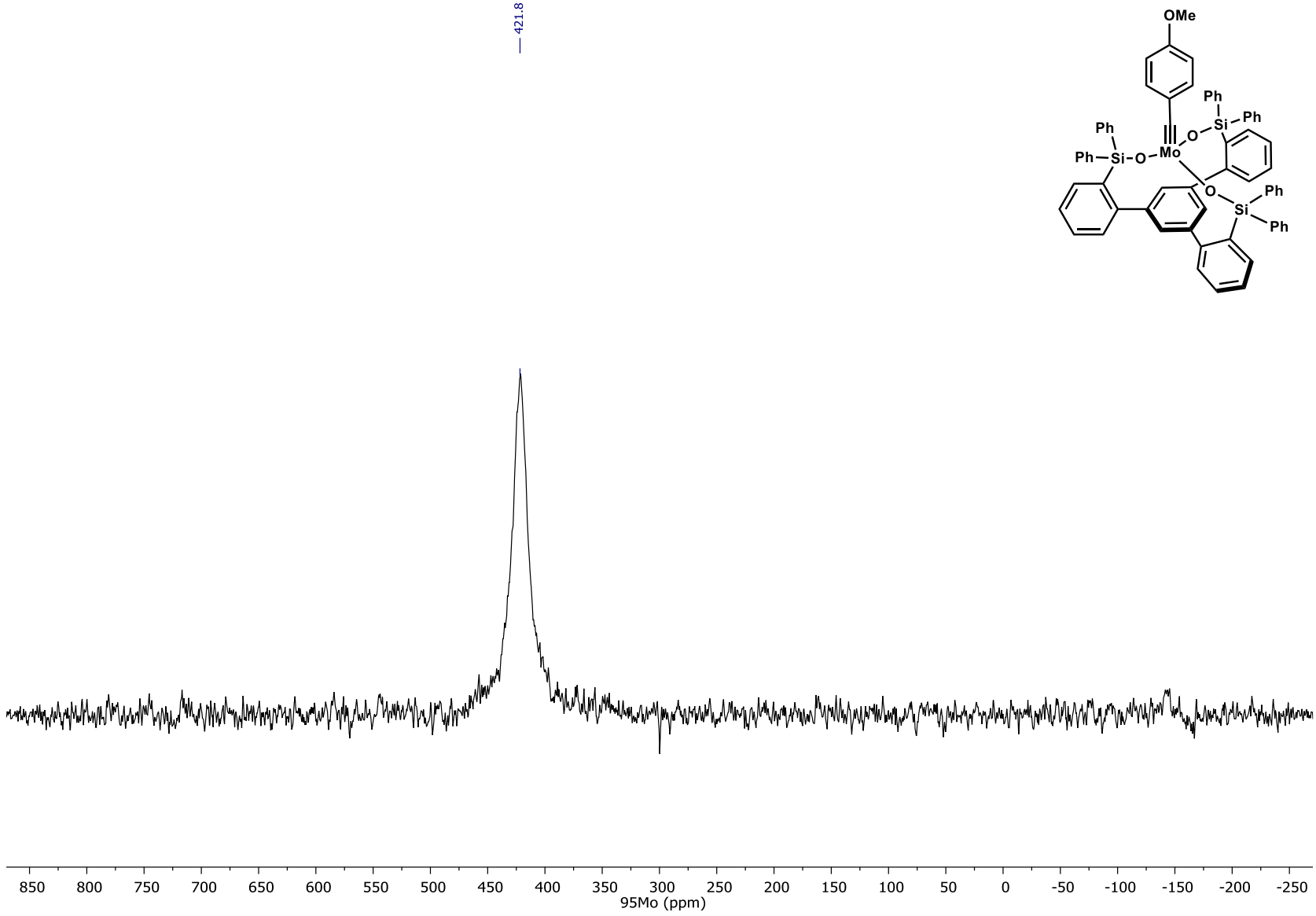
^{13}C NMR of Complex 1a, 101 MHz, $C_6D_5CD_3$, 25ºC



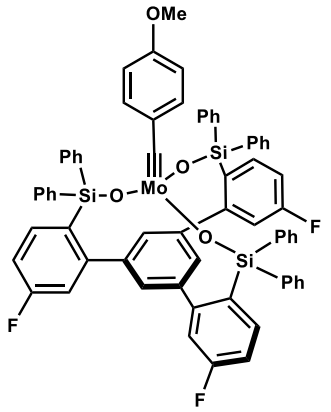
^{1}H-^{29}Si-HMBC NMR of Complex 1a, $C_6D_5CD_3$, 25ºC



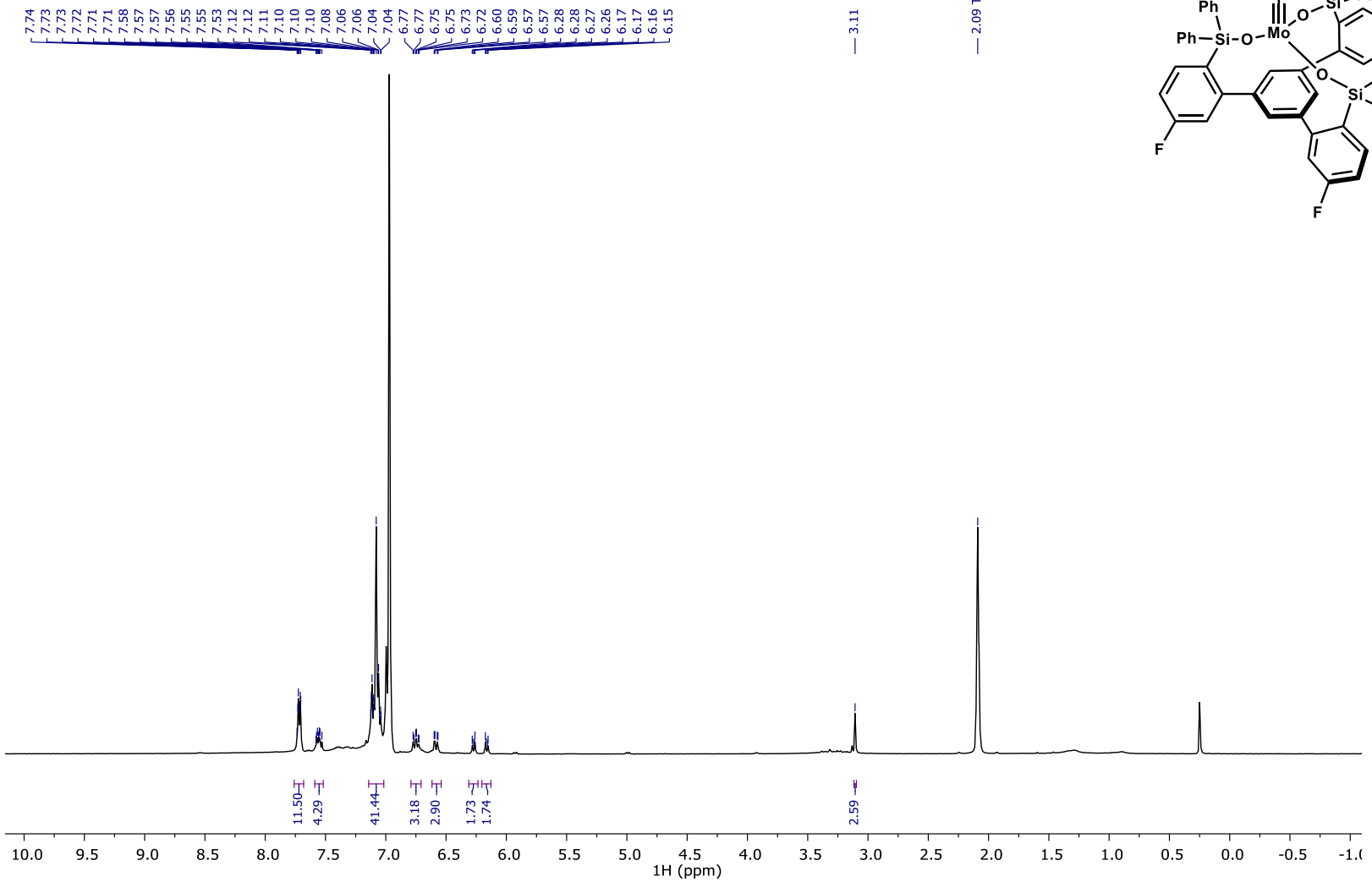
^{95}Mo NMR of Complex 1a, 26 MHz, $C_6D_5CD_3$, 60ºC



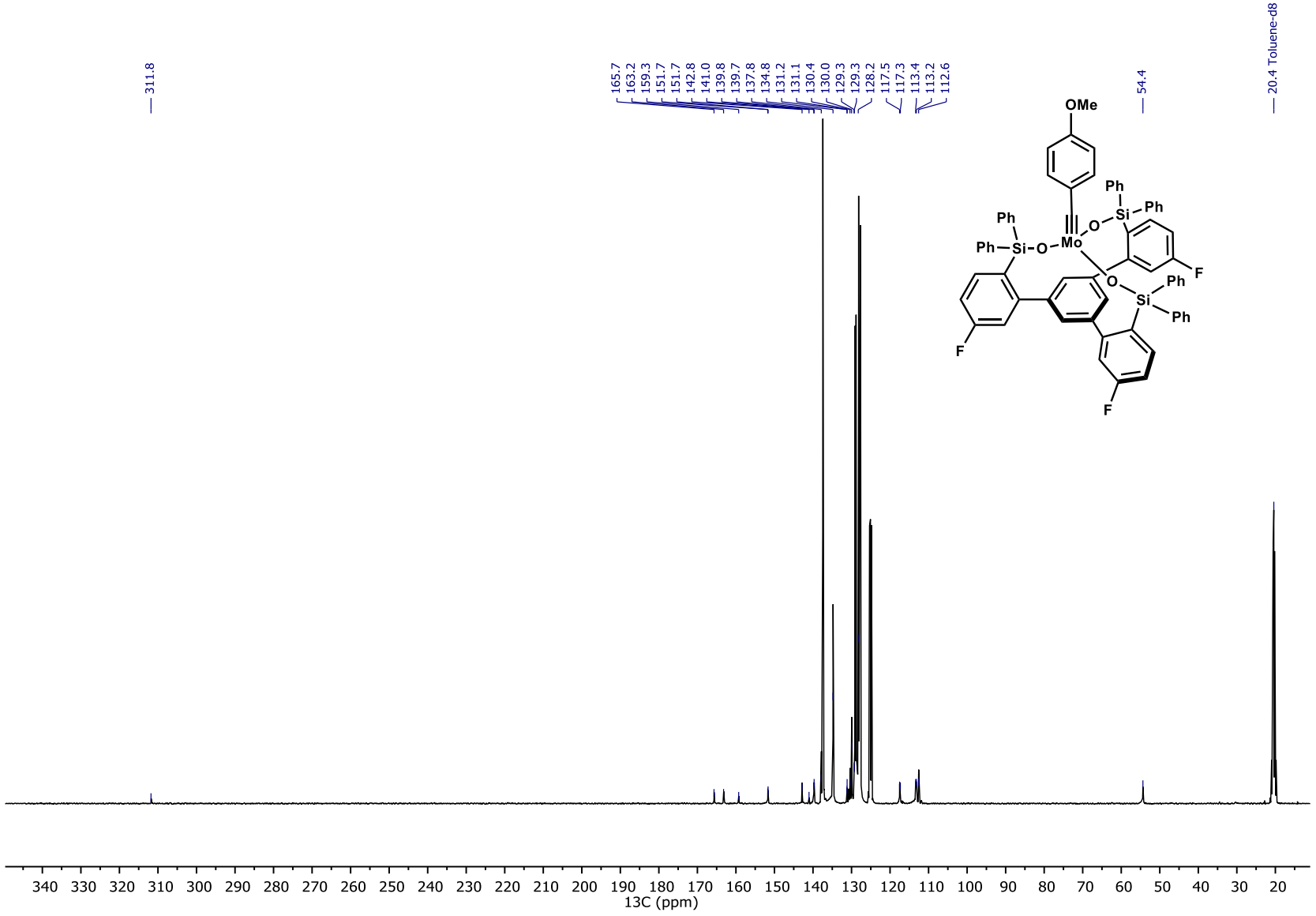
^{1}H NMR Conversion Studies of Complex [1b]$_2$ to Complex 1b, 400 MHz, $C_6D_5CD_3$, 60ºC



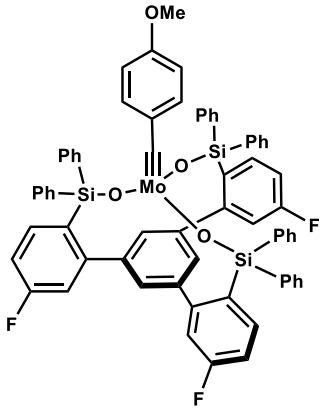
^{1}H NMR of Complex 1b, 400 MHz, $C_6D_5CD_3$, 25ºC



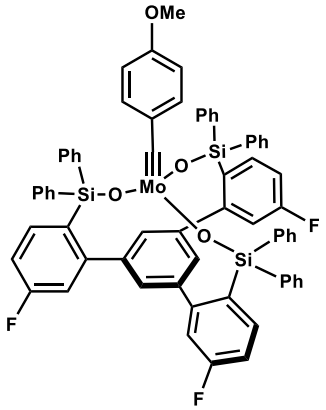
^{13}C NMR of Complex 1b, 101 MHz, $C_6D_5CD_3$, 25°C



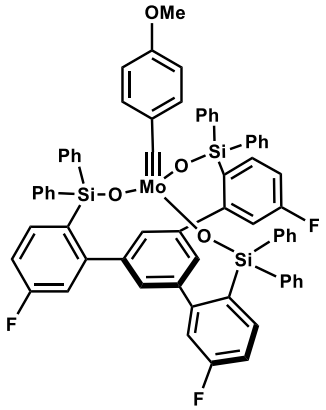
^{19}F NMR of Complex 1b, 376 MHz, $C_6D_5CD_3$, 25ºC



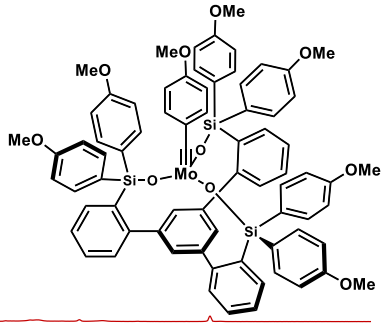
^{29}Si NMR of Complex 1b, 79 MHz, $C_6D_5CD_3$, 25ºC



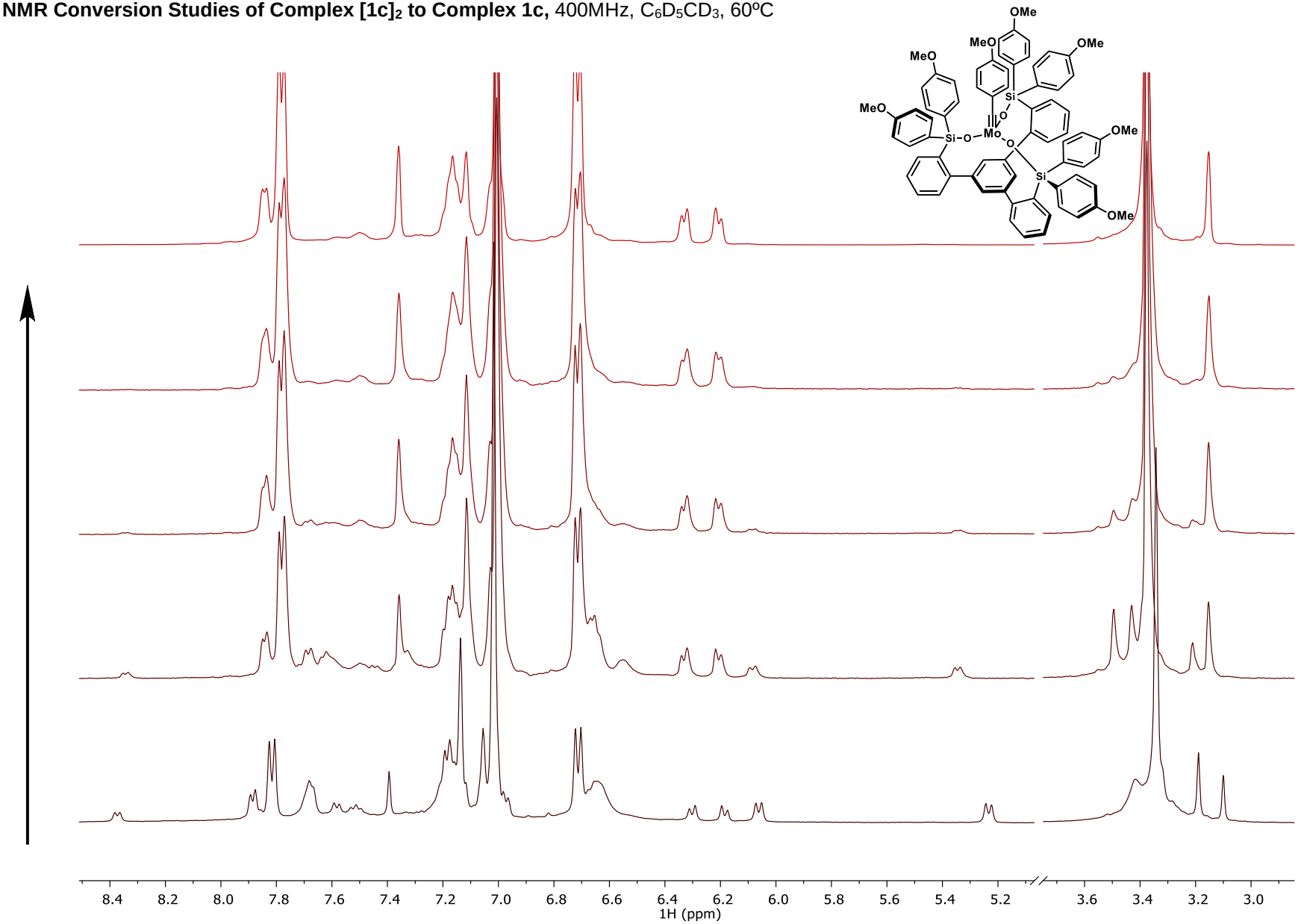
^{95}Mo NMR of Complex 1b, 26 MHz, $C_6D_5CD_3$, 60ºC



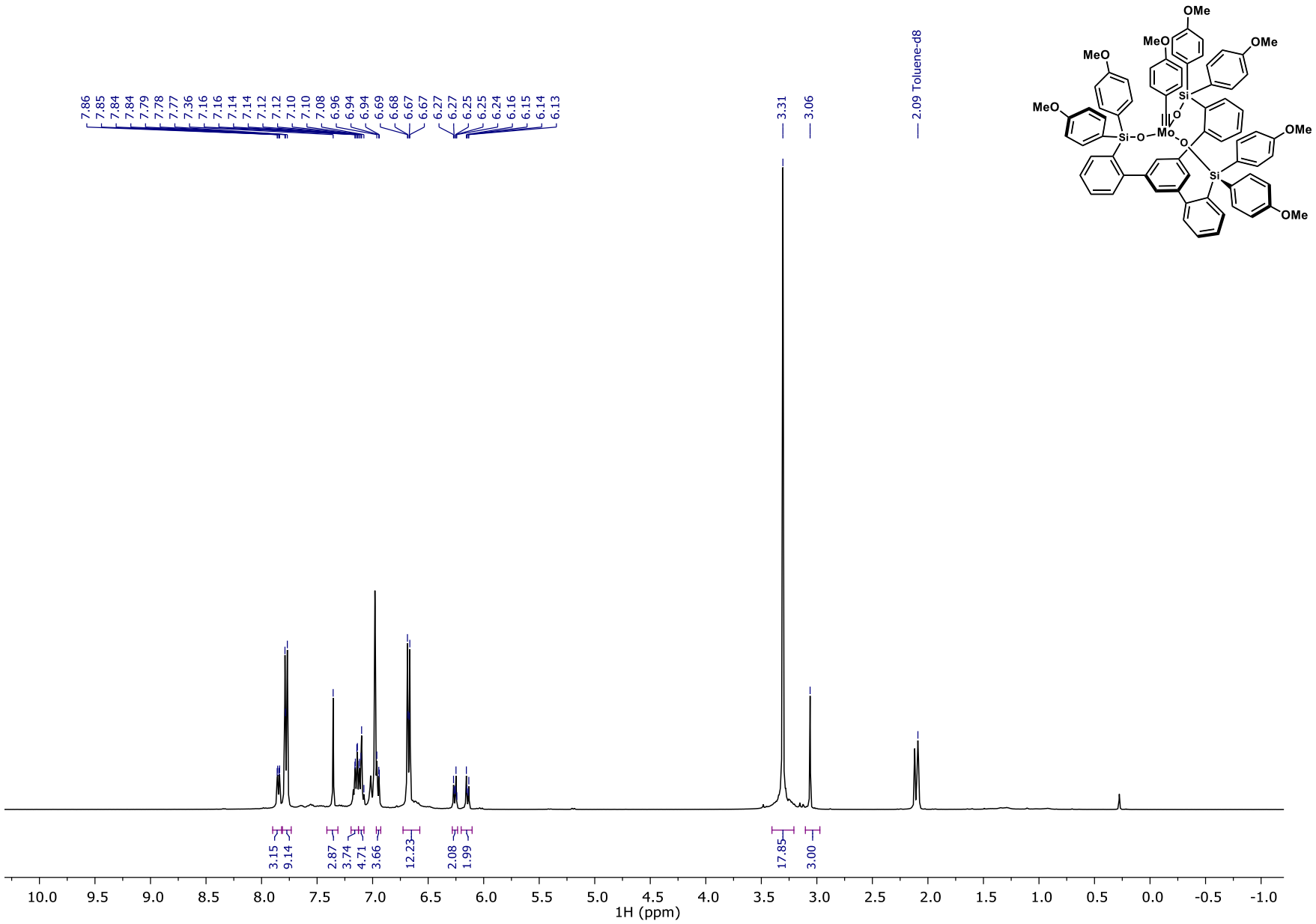
^{1}H NMR Conversion Studies of Complex $[1c]_2$ to Complex 1c, 400 MHz, $C_6D_5CD_3$, 60ºC



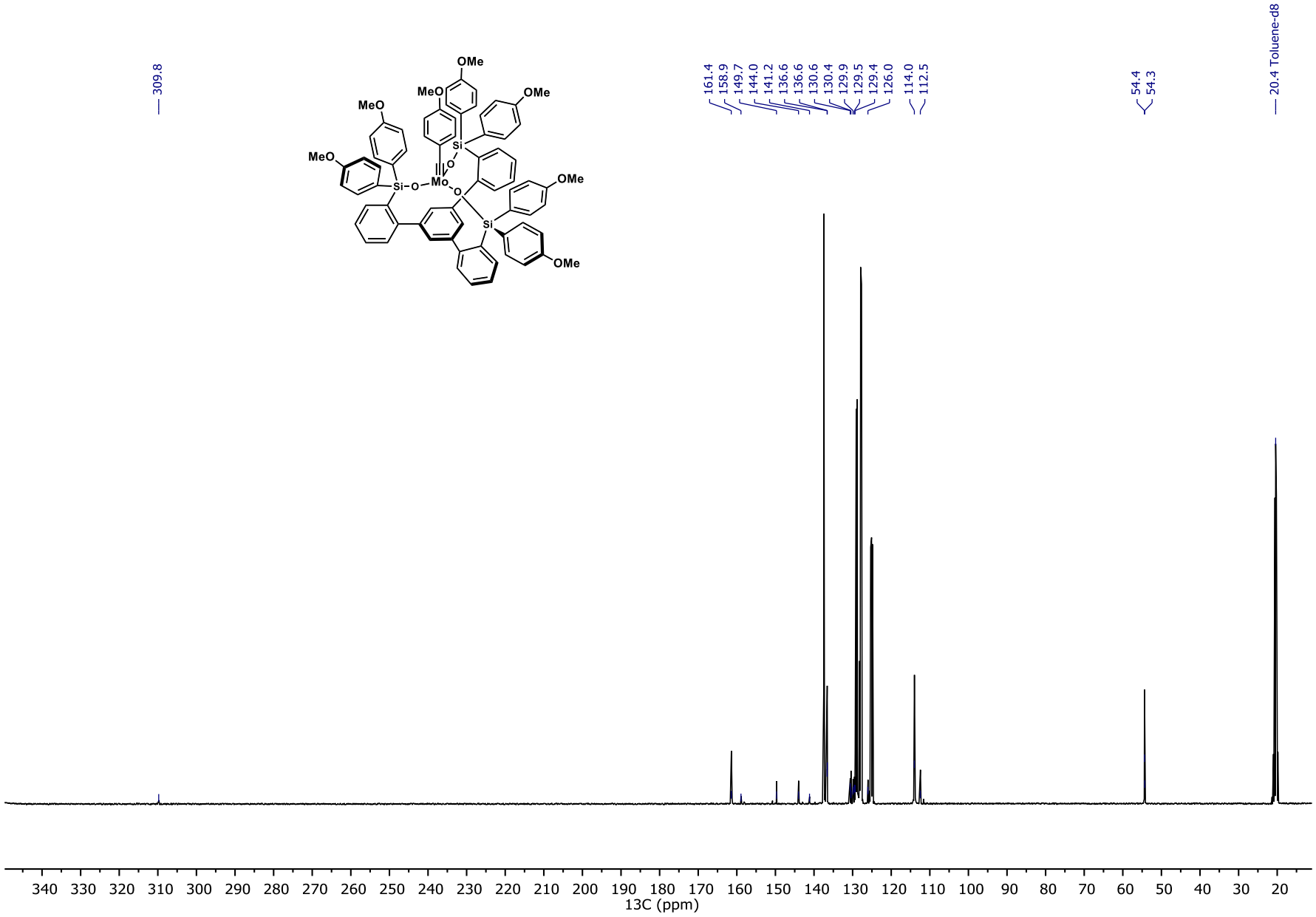
^{1}H NMR Conversion Studies of Complex $[1c]_2$ to Complex 1c, 400MHz, $C_6D_5CD_3$, 60ºC



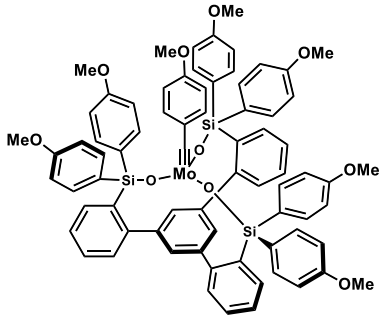
^{1}H NMR of Complex 1c, 400 MHz, $C_6D_5CD_3$, 25ºC



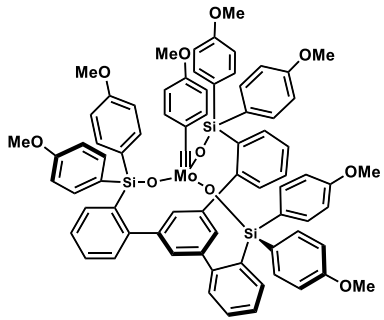
^{1}H NMR of Complex 1c, 101 MHz, $C_6D_5CD_3$, 25ºC



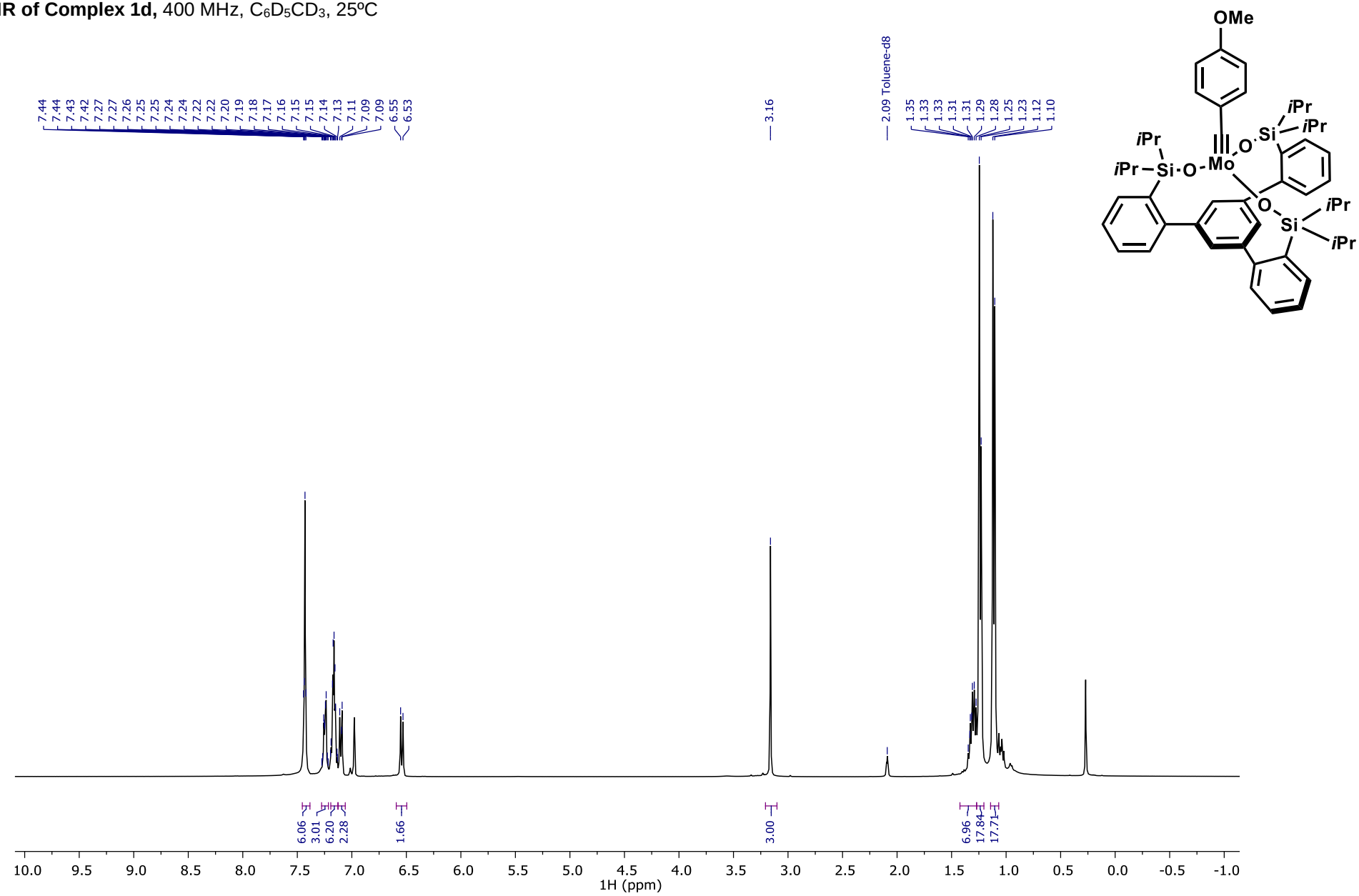
^{29}Si NMR of Complex 1c, 79 MHz, $C_6D_5CD_3$, 25ºC



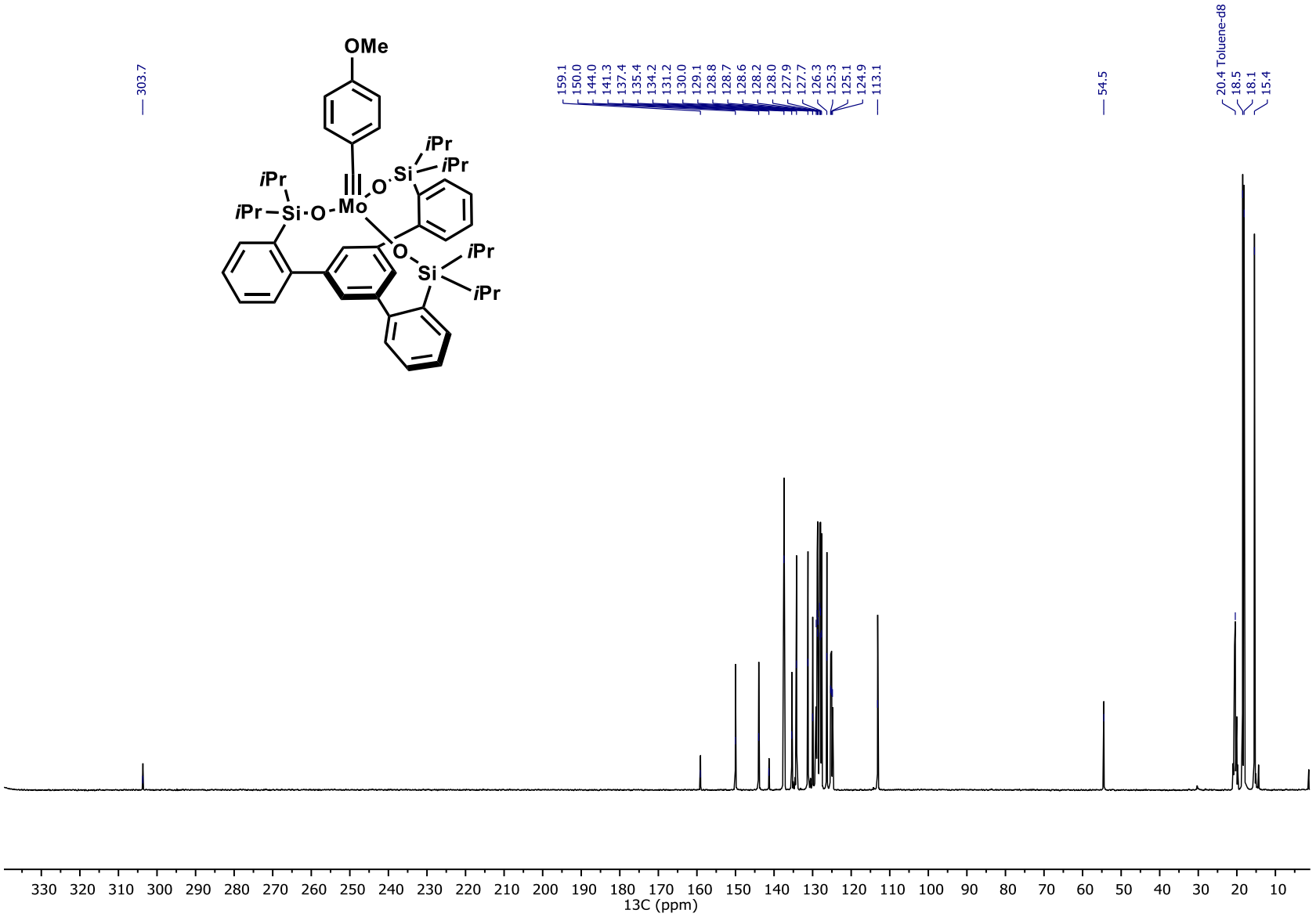
^{95}Mo NMR of Complex 1c, 26 MHz, $C_6D_5CD_3$, 60ºC



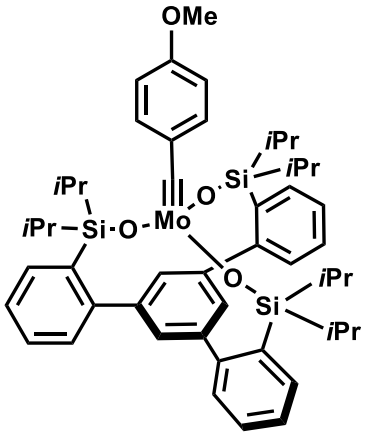
^{1}H NMR of Complex 1d, 400 MHz, $C_6D_5CD_3$, 25ºC



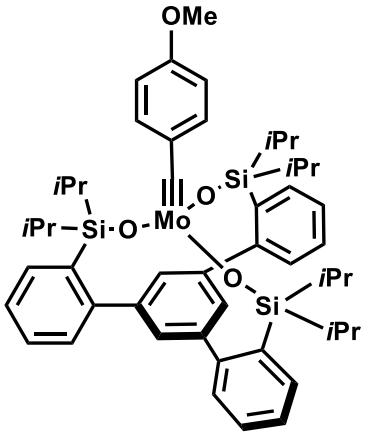
^{13}C NMR of Complex 1d, 101 MHz, $C_6D_5CD_3$, 25ºC



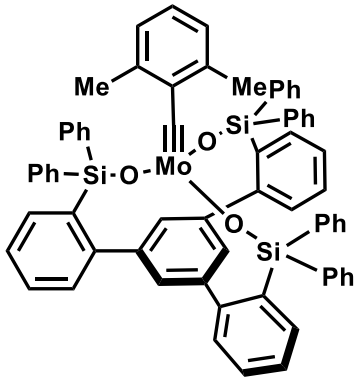
^{29}Si NMR of Complex 1d, 79 MHz, $C_6D_5CD_3$, 25ºC



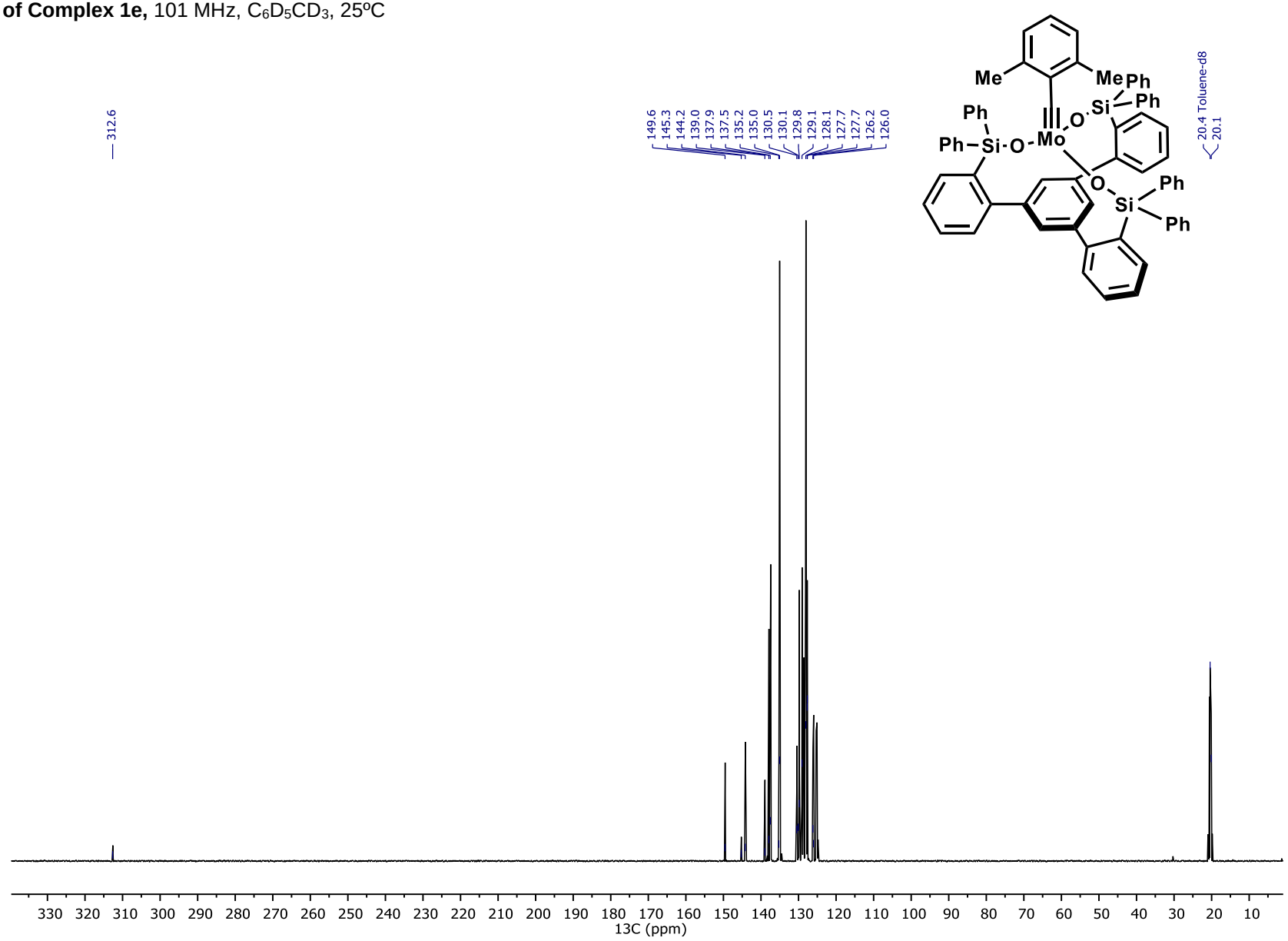
^{95}Mo NMR of Complex 1d, 26 MHz, $C_6D_5CD_3$, 60ºC



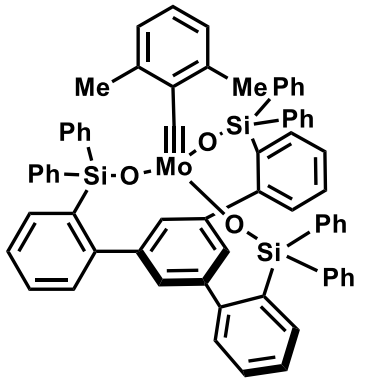
^{1}H NMR of Complex 1e, 400 MHz, $C_6D_5CD_3$, 25ºC



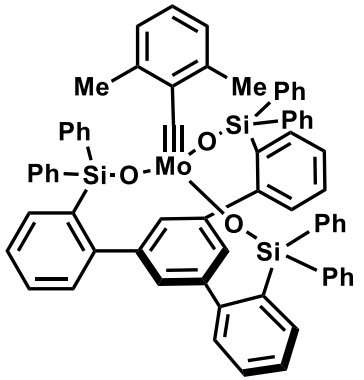
^{13}C NMR of Complex 1e, 101 MHz, $C_6D_5CD_3$, 25ºC



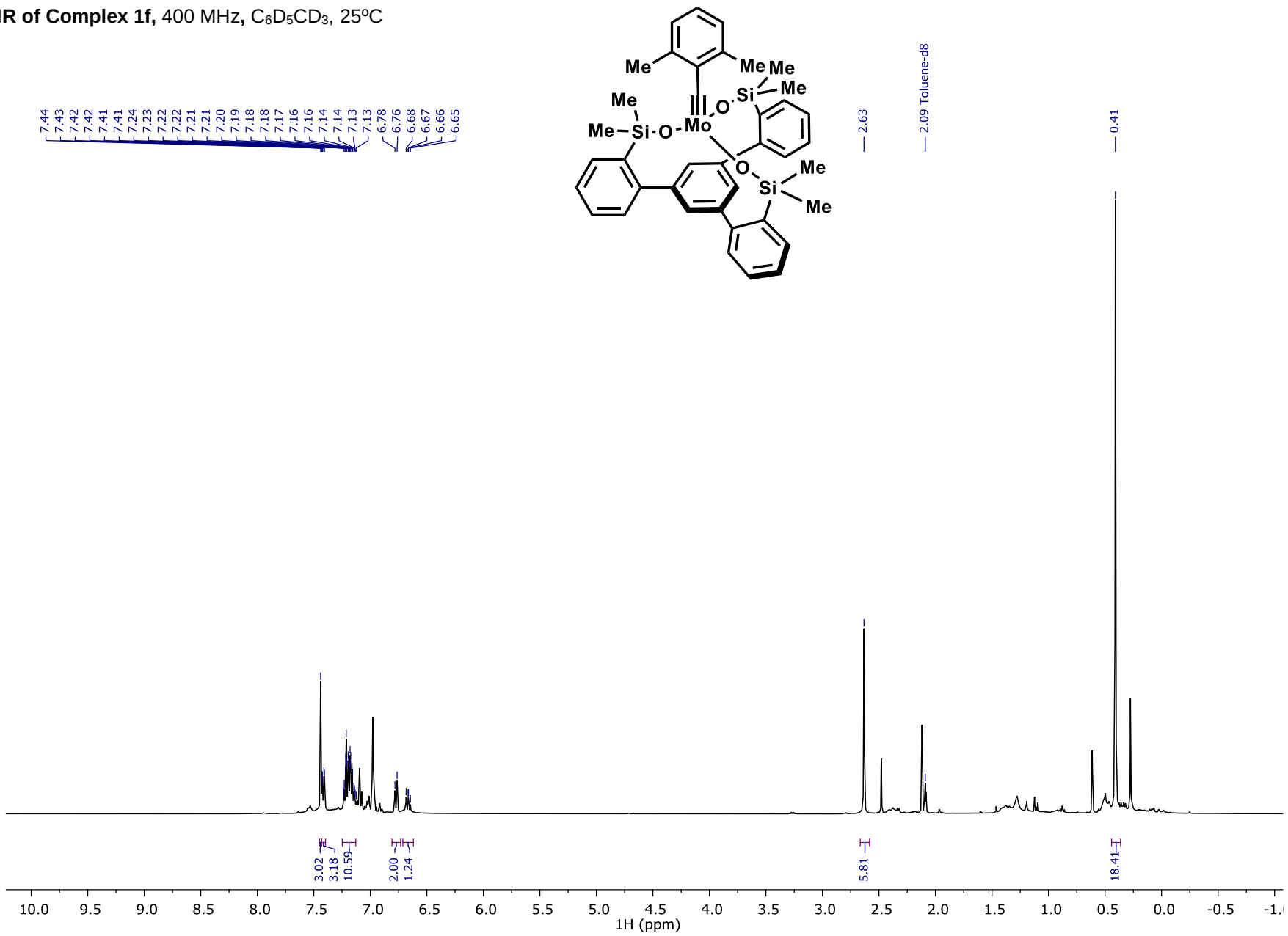
^{29}Si NMR of Complex 1e, 79 MHz, $C_6D_5CD_3$, 25ºC



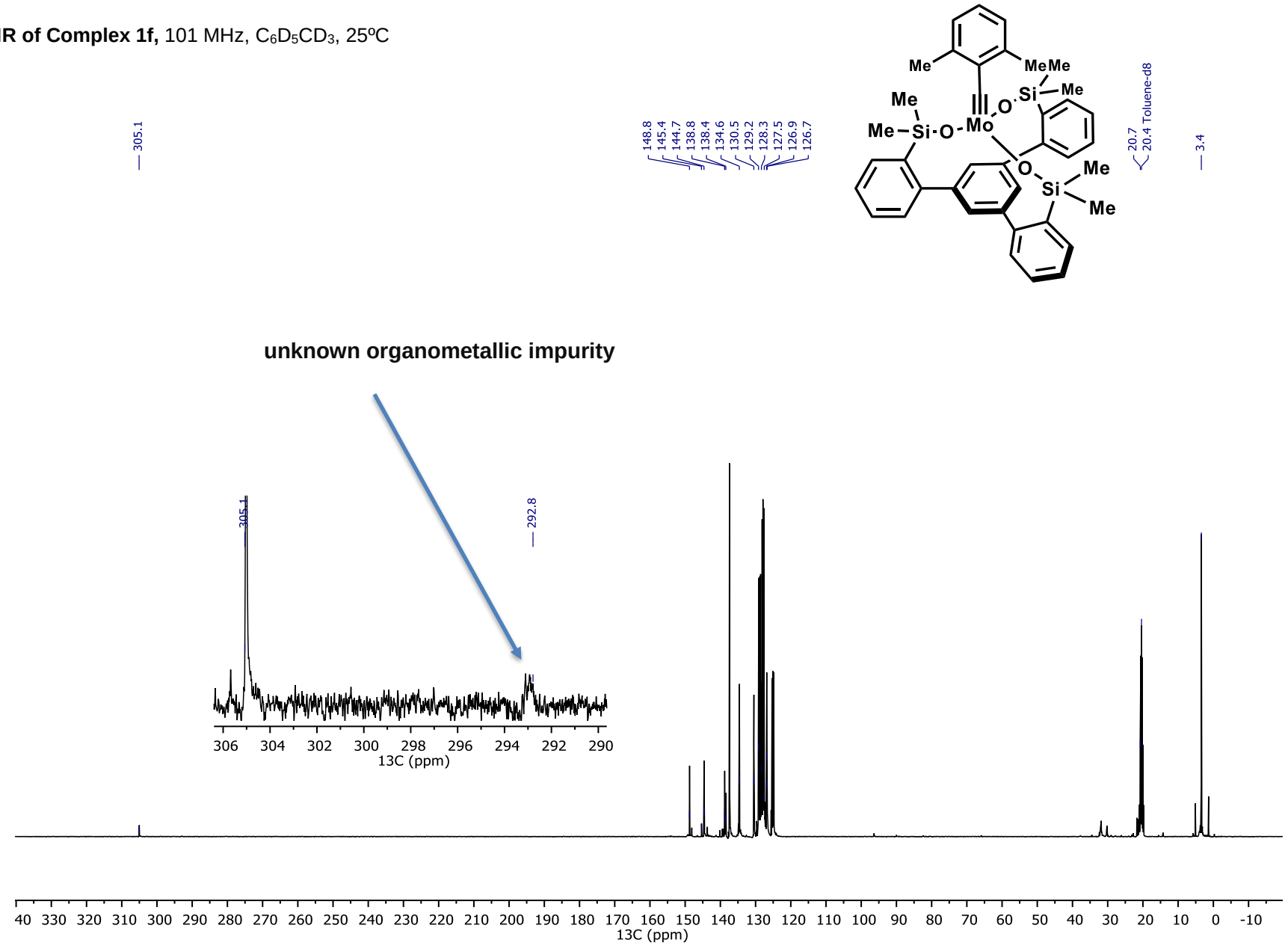
^{95}Mo NMR of Complex 1e, 26 MHz, $C_6D_5CD_3$, 60ºC



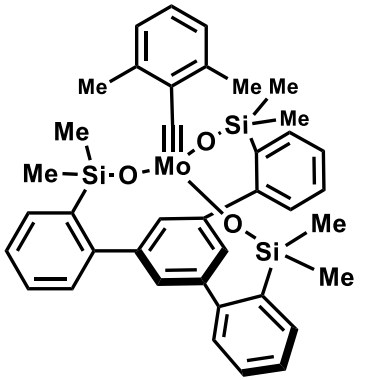
^{1}H NMR of Complex 1f, 400 MHz, $C_6D_5CD_3$, 25ºC



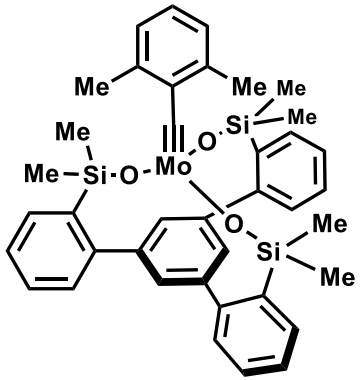
^{13}C NMR of Complex 1f, 101 MHz, $C_6D_5CD_3$, 25ºC



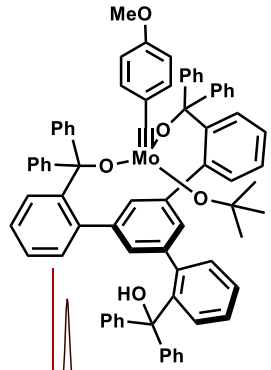
^{29}Si NMR of Complex 1f, 79 MHz, $C_6D_5CD_3$, 25ºC



^{95}Mo NMR of Complex 1f, 26 MHz, $C_6D_5CD_3$, 25ºC

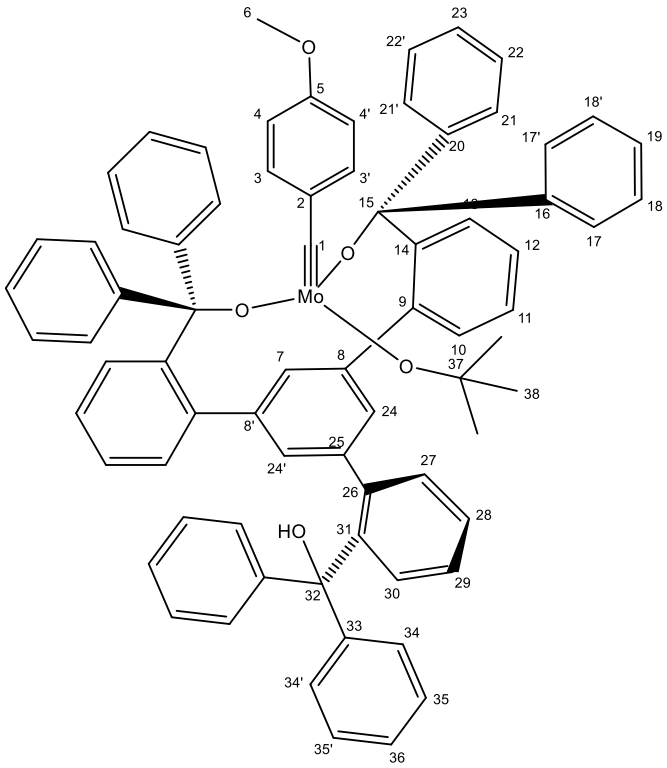


VT NMR Studies of Complex 16, 400 MHz, $C_6D_5CD_3$, 25°C to –20ºC



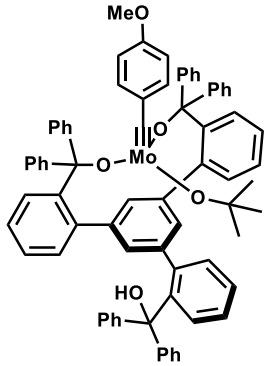
NMR assignment table of Complex 16, $C_6D_5CD_3$, –20ºC

Atom	δ (ppm)	COSY	HSQC	HMBC	ROESY	Atom	δ (ppm)	COSY	HSQC	HMBC	ROESY
1 C	287.08			3, 3'		**21' C**	129.25		21'	21, 23	
2 C	138.84			4, 4'		**H**	7.57	22', 23	21'	15, 21, 23	3, 3', 7
3 C	131		3	3'		**22 C***	127.54		22	22'	
H	5.42	4	3	1, 3', 5	21, 21', 38	**H***	7	21, 23	22	20, 22'	
3' C	131		3'	3		**22' C***	127.54		22'	22	
H	5.42	3'	3'	1, 3, 5	21, 21', 38	**H***	7	21', 23	22'	20, 22	
4 C	112.08		4	4'		**23 C***	127.44		23	21, 21'	
H	6.32	3	4	2, 4', 5	6	**H***	6.92	21, 21', 22, 22'	23	21, 21'	
4' C	112.08		4'	4		**24 C***	127.5		24	7, 24'	
H	6.32		4'	2, 4, 5	6	**H**	6.71	7	24	7, 9, 24', 26	17, 27, 34, 34', 200
5 C	158.29			3, 3', 4, 4', 6		**24' C***	127.5		24'	7, 24	
6 C	53.91		6			**H**	6.71	7	24'	9, 24, 26	17, 27, 34, 34', 200
H3	3.09		6	5	4, 4'	**25 C**	139.23			27	
7 C*	127.71		7	24		**26 C**	141.22			24, 24', 28, 30	
H	6.75	24, 24'	7	9, 24, 24'	21, 21'	**27 C**	133.89		27	29	
8 C	144.48			10		**H**	7.71	28, 29	27	25, 29, 31	24, 24', 38
8' C	144.48					**28 C**	126.11		28	30	
9 C	141.13			7, 11, 13, 24, 24'		**H**	7.28	27	28	26, 30	38
10 C	132.51		10	12		**29 C**	126.11			27	
H	6.66	11, 12	10	8, 12, 14		**H**	7	27, 30		27, 31	
11 C	126.48		11	13		**30 C**	129.95		30	28	
H*	6.93	10, 12	11	9, 13		**H***	6.88	29	30	26, 28, 32	
12 C	125.72		12	10		**31 C**	144.93			27, 29	
H*	6.88	10, 11, 13	12	10, 14		**32 C**	83.59			30, 34, 34', 200	

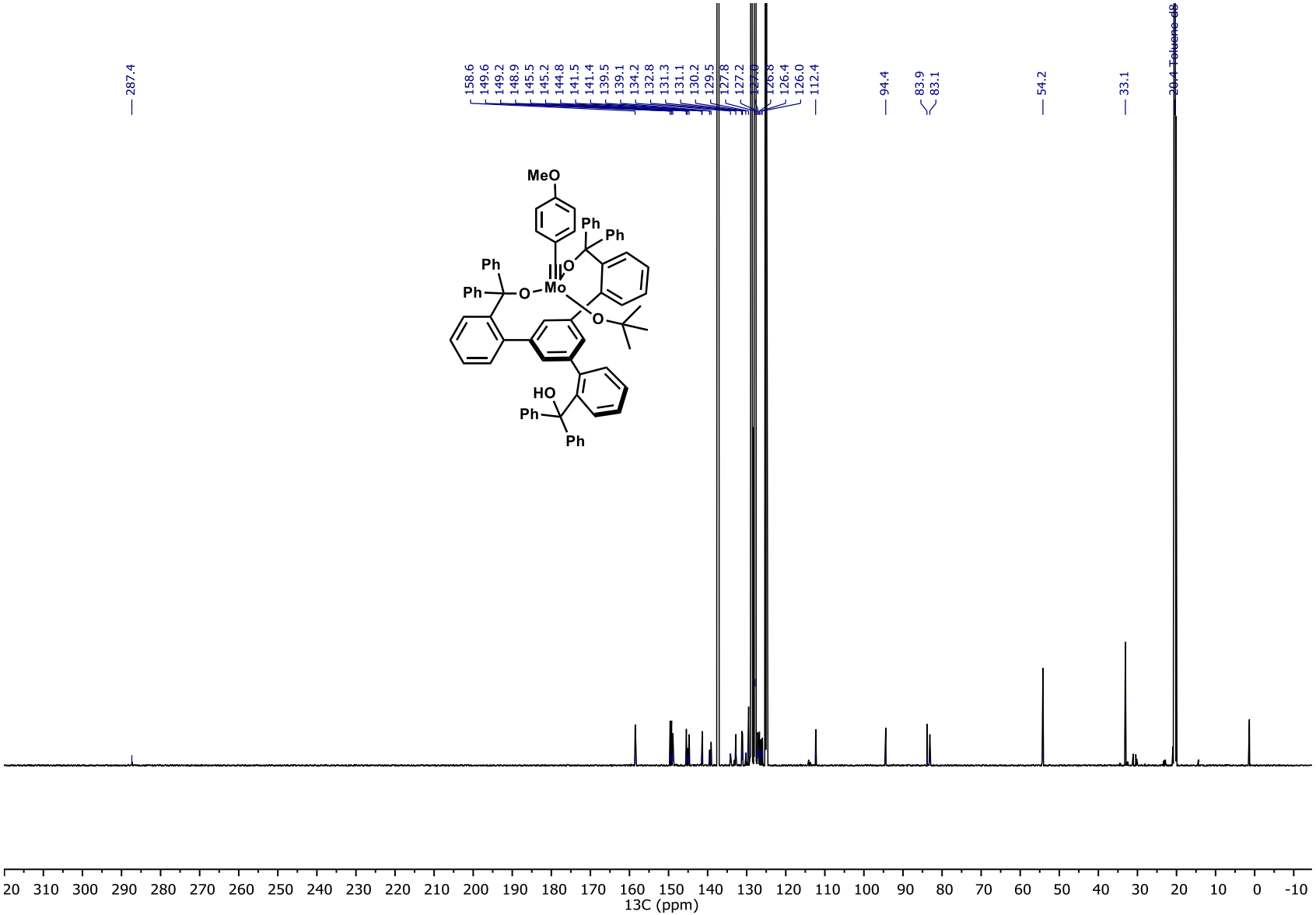


13 C	130.81		13	11		**33 C**	148.65			35, 35', 200	
H*	6.87	12	13	9, 11, 15		**34 C***	128.51		34	34', 36	
14 C	145.19			10, 12		**H**	7.31	35, 35', 36	34	32, 34', 36	24, 24', 200
15 C	94.15			13, 17, 17', 21, 21'		**34' C***	128.51		34'	34, 36	
16 C	149.34			18, 18'		**H**	7.31	35', 36	34'	32, 34, 36	24, 24', 200
17 C*	128.68		17	17', 19		**35 C***	127.6		35	35'	
H	7.38	18, 19	17	15, 17', 19	24, 24', 38	**H***	6.94	34	35	33, 35'	
17' C*	128.68		17'	17, 19		**35' C**	127.6		35'	35	
H	7.38	18', 19	17'	15, 17, 19	38	**H***	6.94	34, 34'	35'	33, 35	
18 C*	127.5		18	18'		**36 C**	126.74			34, 34'	
H	7.11	17, 19	18	16, 18'	38	**H**	6.98	34, 34'		34, 34'	
18' C*	127.5		18'	18		**37 C**	82.86			38	
H	7.11	17', 19	18'	16, 18	38	**38 C**	32.81		38	38	
19 C	126.96		19	17, 17'		**H3**	1.46		38	37, 38	3, 3', 17, 17', 18, 18', 27, 28
H*	7.03	17, 17', 18, 18'	19	17, 17'		**100 Mo**	118.3				
20 C	148.97			22, 22'		**200 O**					
21 C	129.25		21	21', 23		**H**	3.66			32, 33	24, 24', 34, 34'
H	7.57	22, 23	21	15, 21', 23	3, 3', 7						

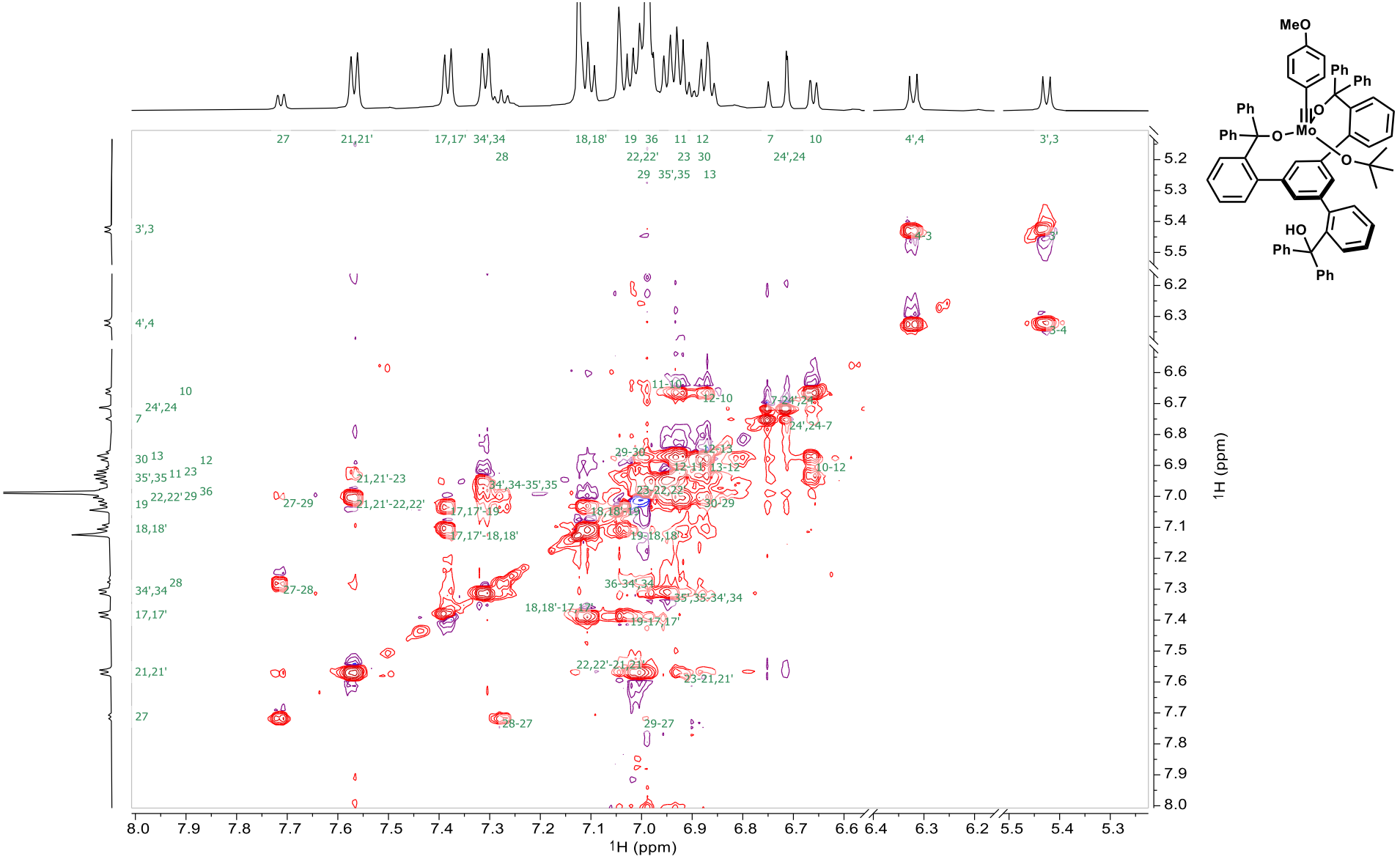
^{1}H NMR of Complex 16, 600 MHz, $C_6D_5CD_3$, –20°C



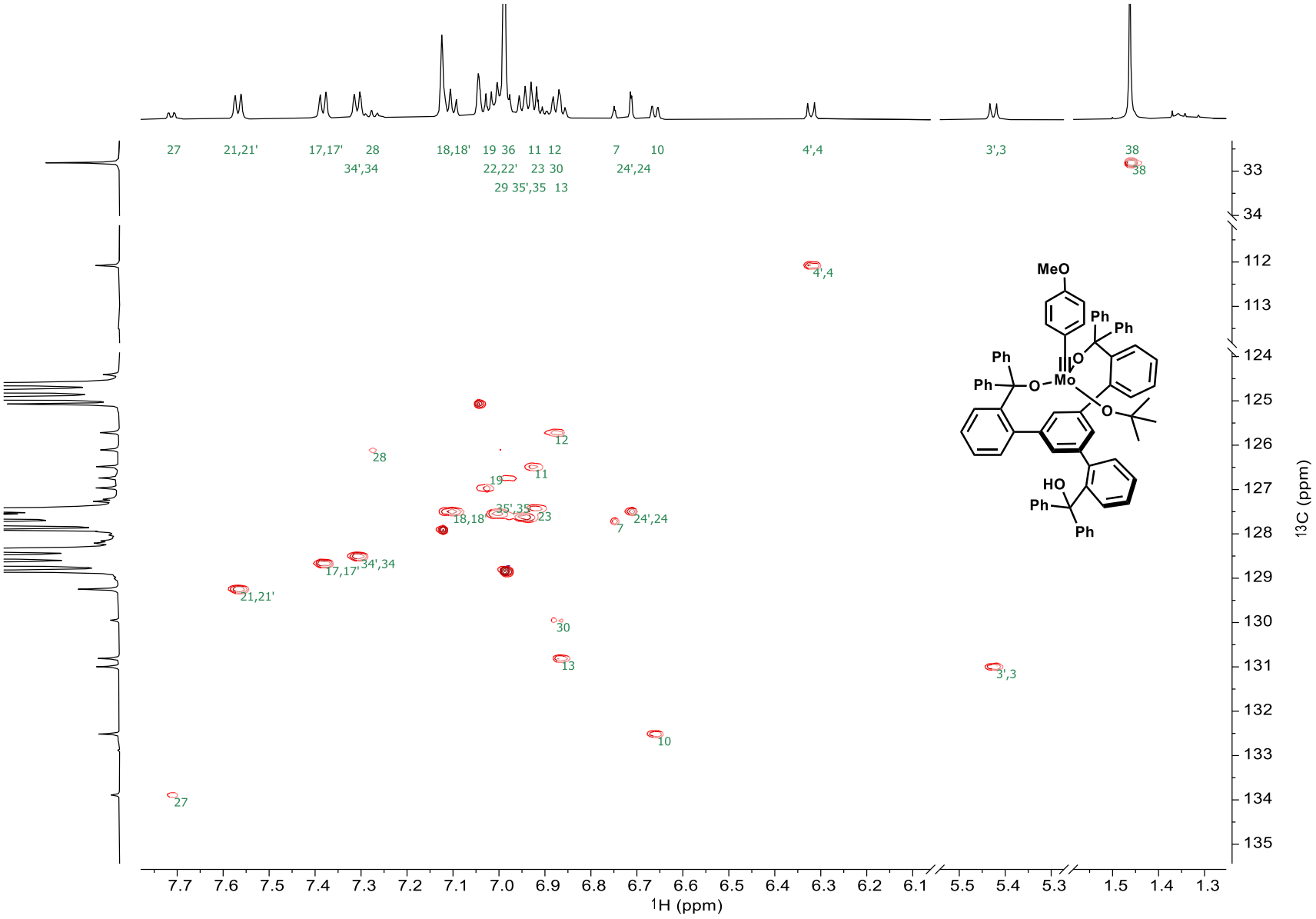
^{13}C NMR of Complex 16, 151 MHz, $C_6D_5CD_3$, –20°C



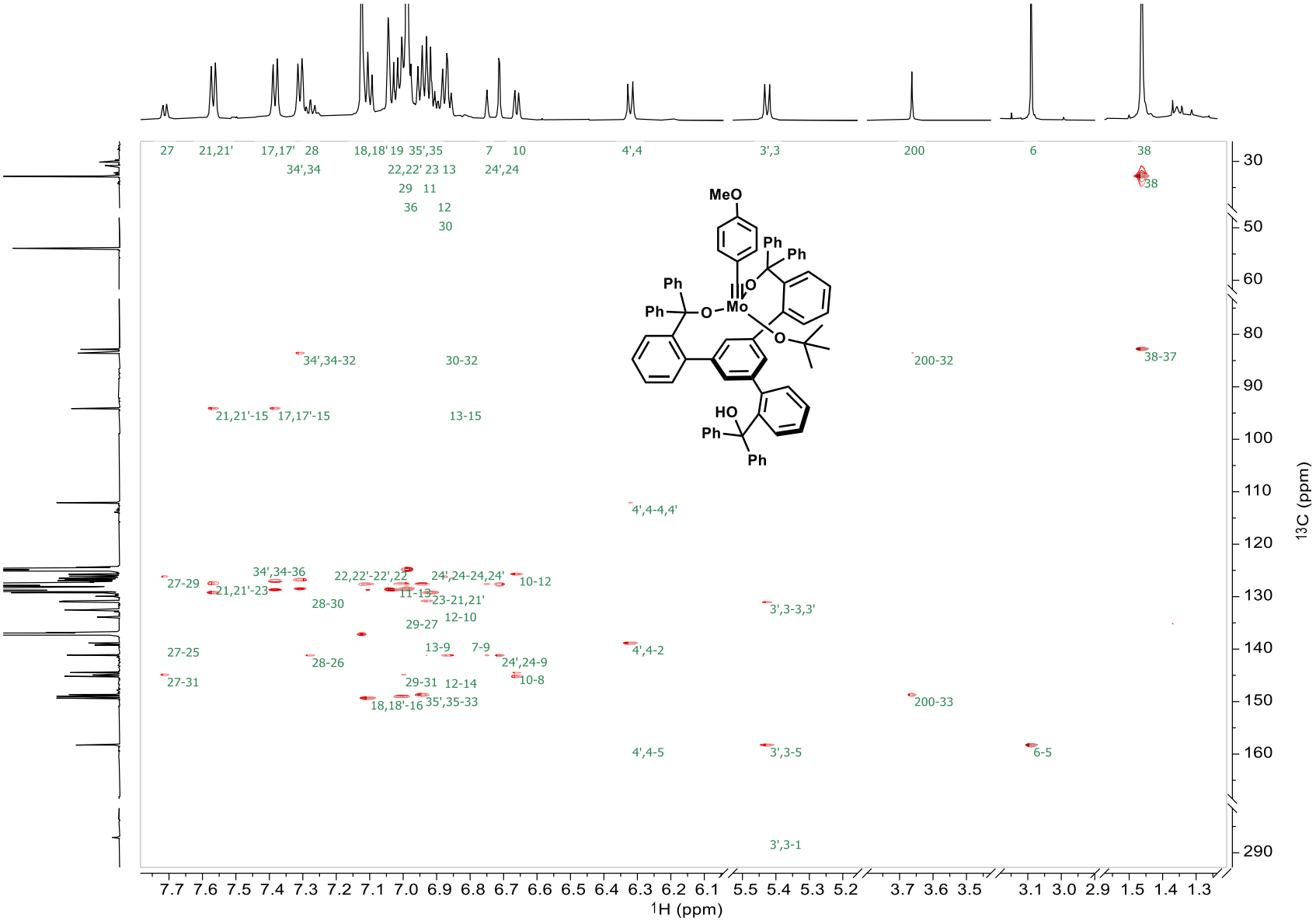
^{1}H CLIP COSY of Complex 16, $C_6D_5CD_3$, -20°C



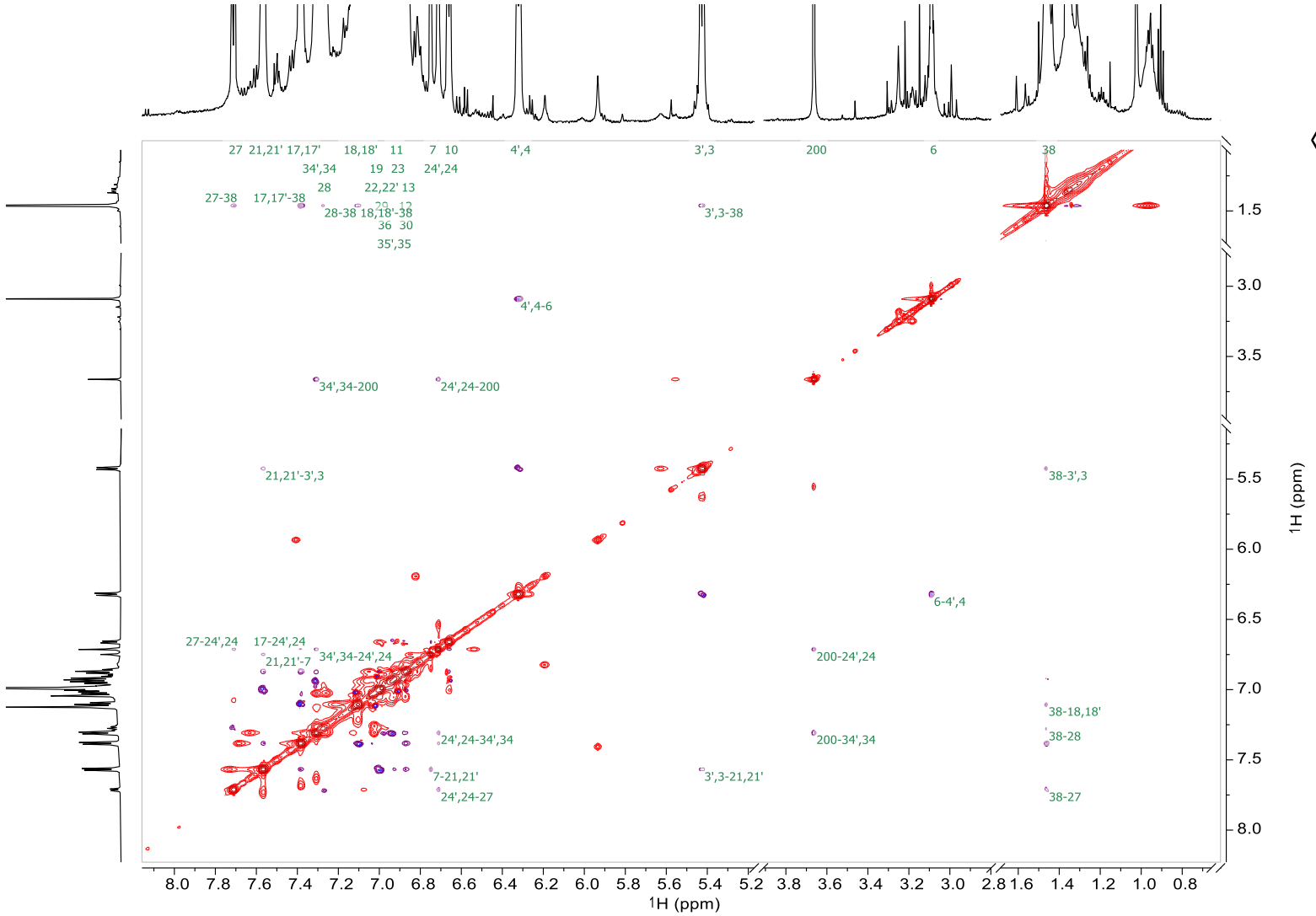
^{1}H-^{13}C edited HSQC of Complex 16, $C_6D_5CD_3$, -20°C

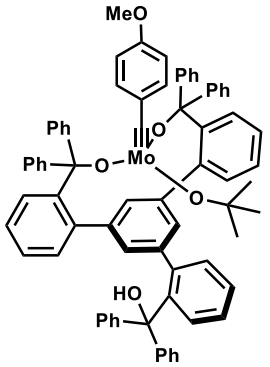


^{1}H-^{13}C HMBC of Complex 16, $C_6D_5CD_3$, -20°C

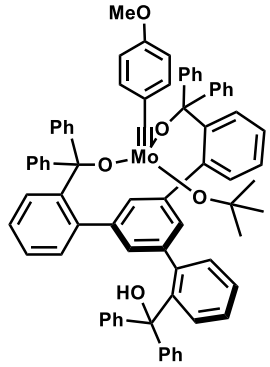


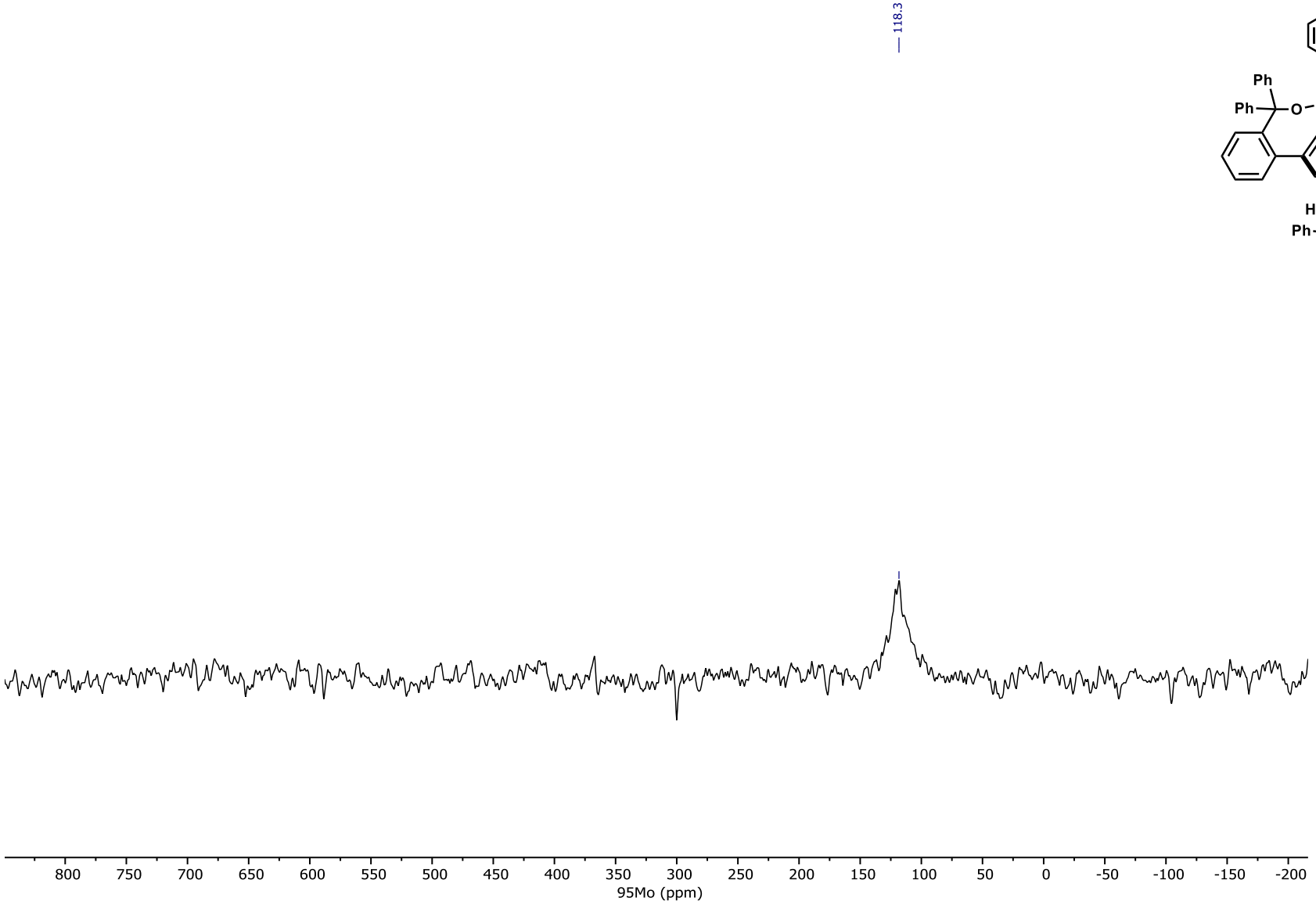
^{1}H EASY ROESY of Complex 16, $C_6D_5CD_3$, -20°C



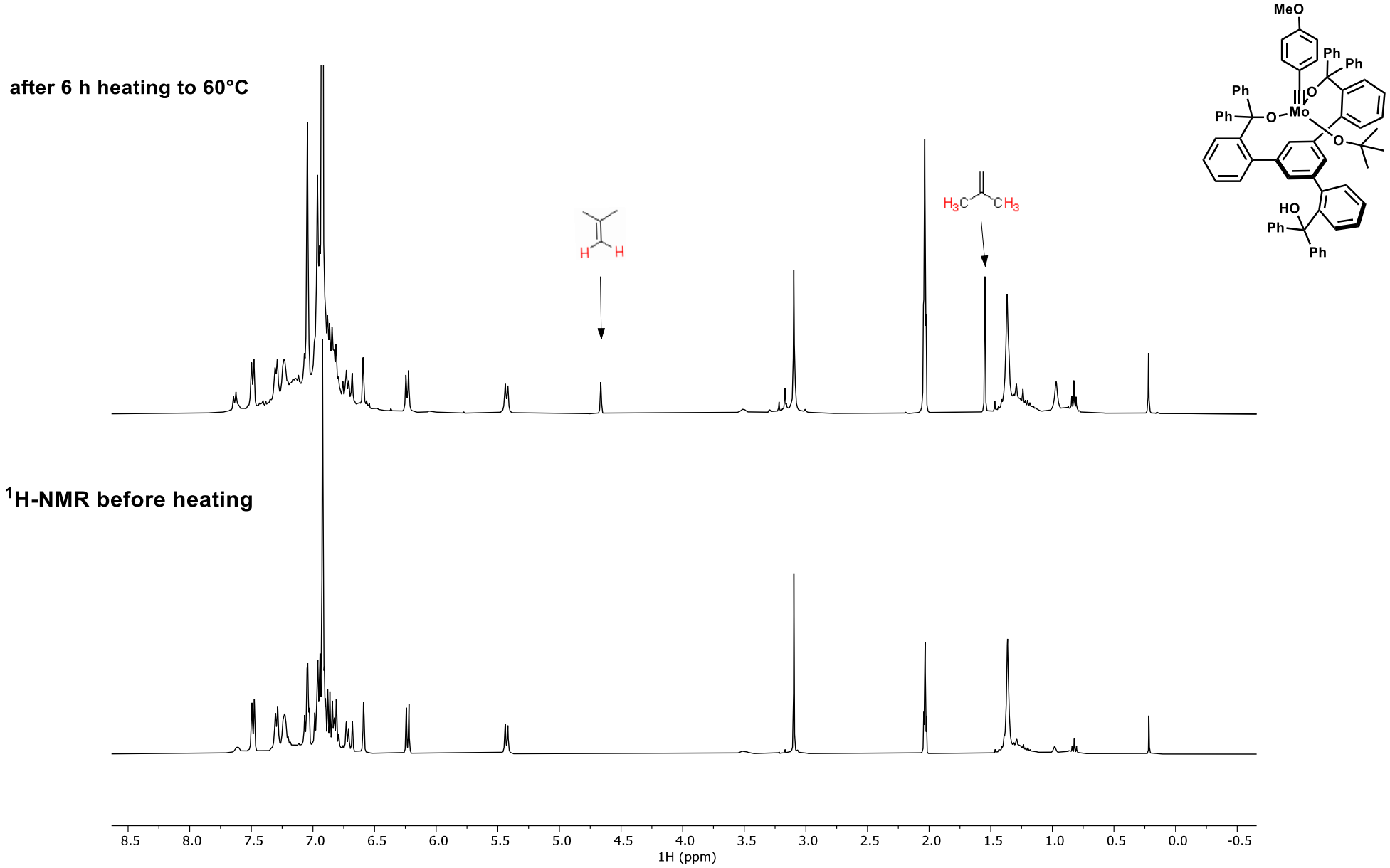


^{95}Mo NMR of Complex 16, 26 MHz, $C_6D_5CD_3$, 60ºC; the spectrum was processed with lb = 50 Hz

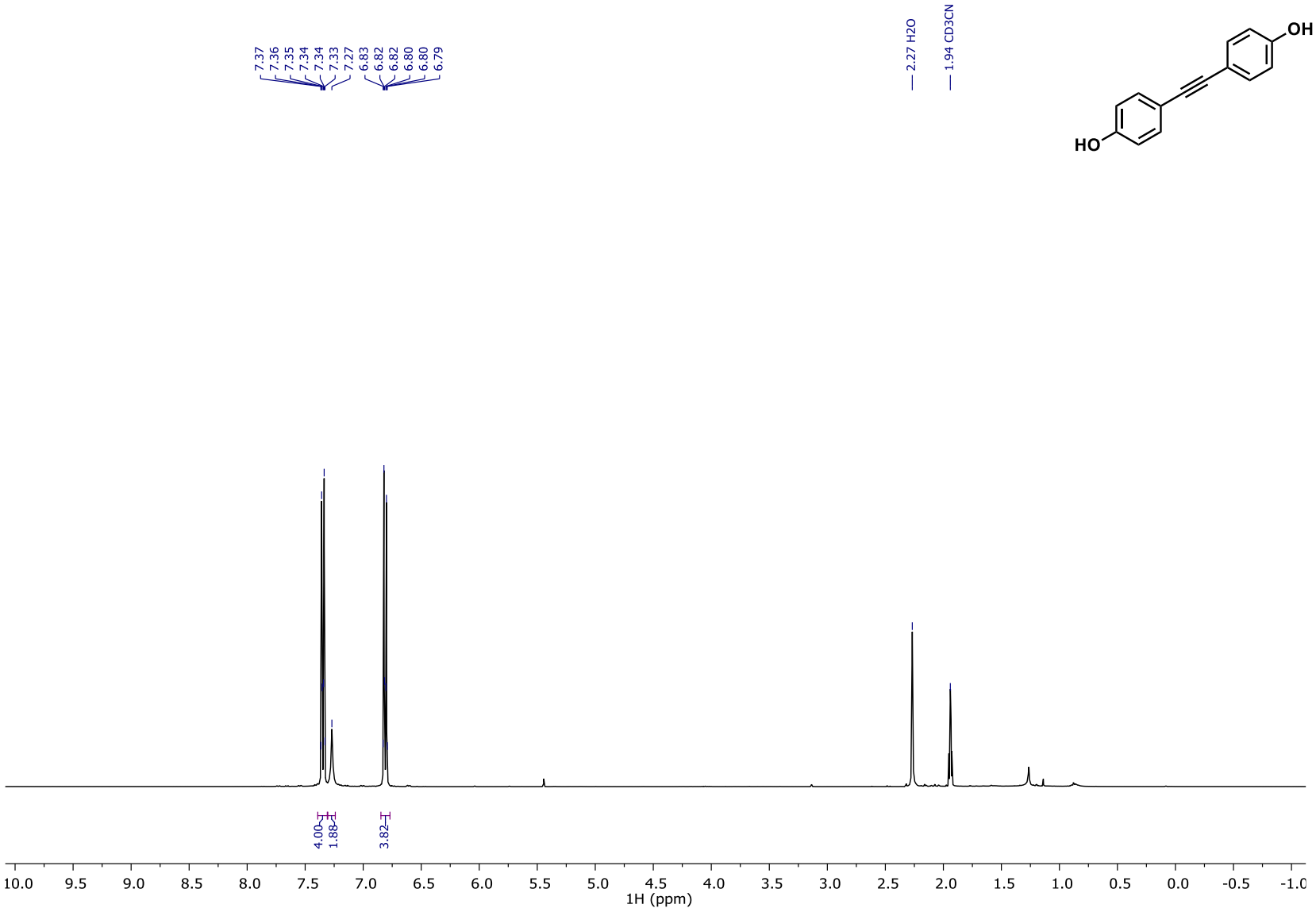




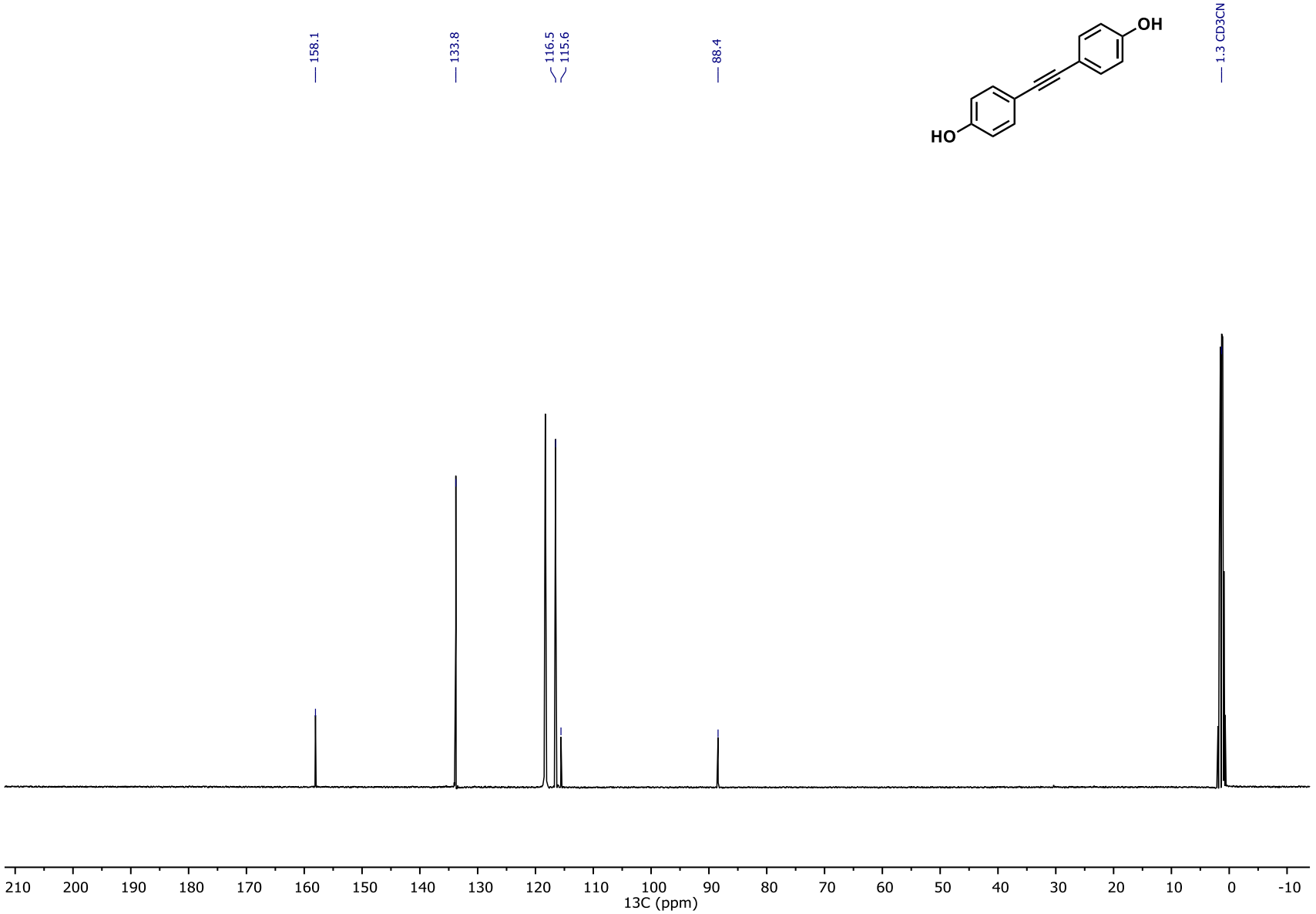
Decomposition of Complex 16 observed after 6h heating to 60°C (Formation of isobutylene), 400 MHz, $C_6D_5CD_3$, 25ºC



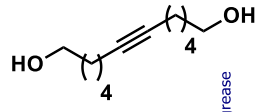
^{1}H NMR of 4,4'-(Ethyne-1,2-diyl)diphenol (20b), 400 MHz, CD_3CN, 25ºC

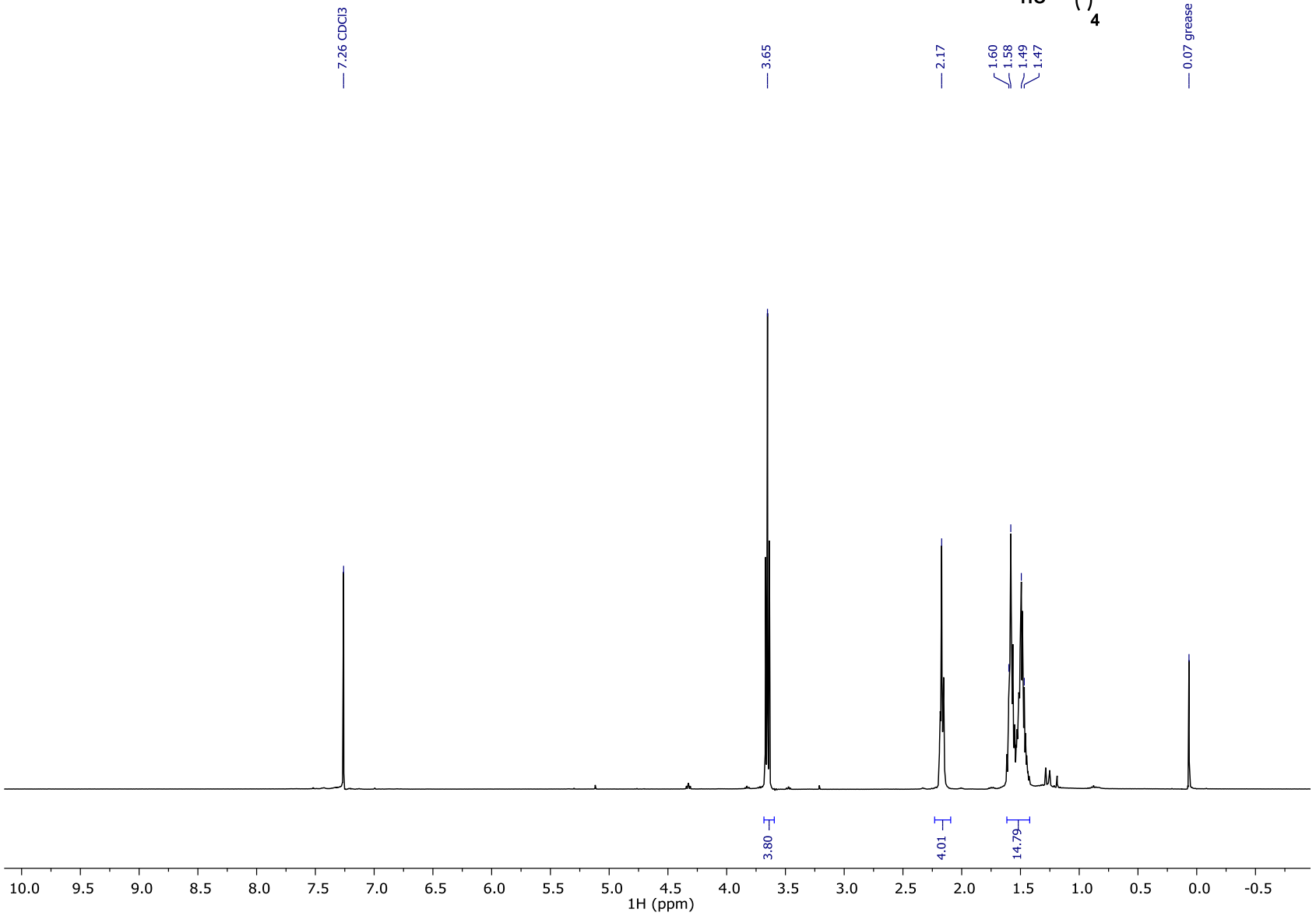


^{13}C NMR of 4,4'-(Ethyne-1,2-diyl)diphenol (20b), 101 MHz, CD_3CN, 25ºC

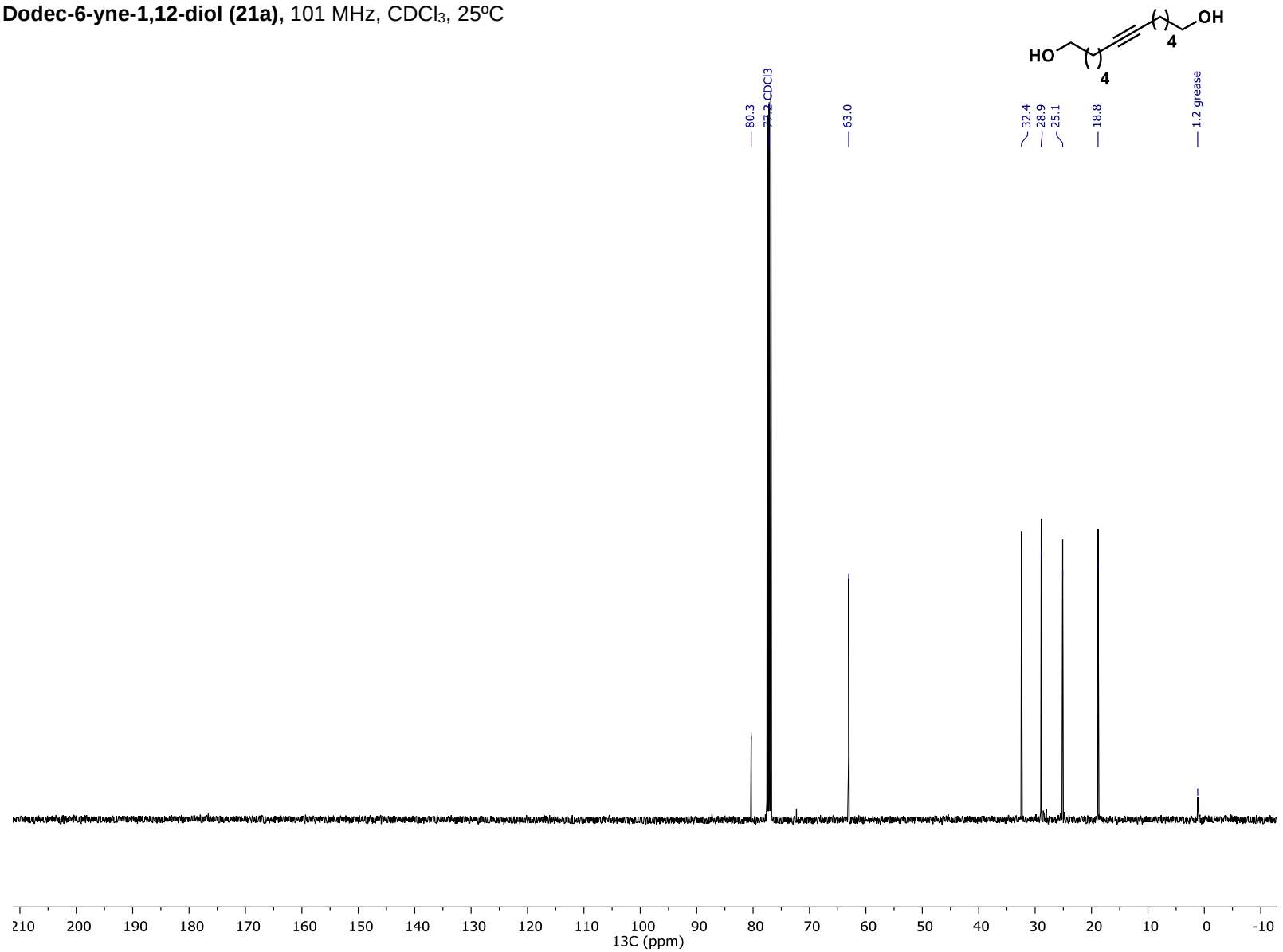


^{1}H NMR of Dodec-6-yne-1,12-diol (21a), 400 MHz, $CDCl_3$, 25ºC

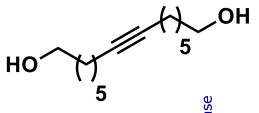


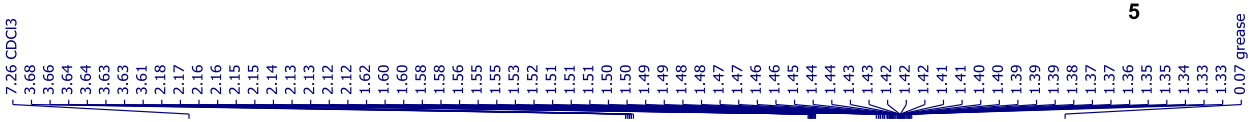


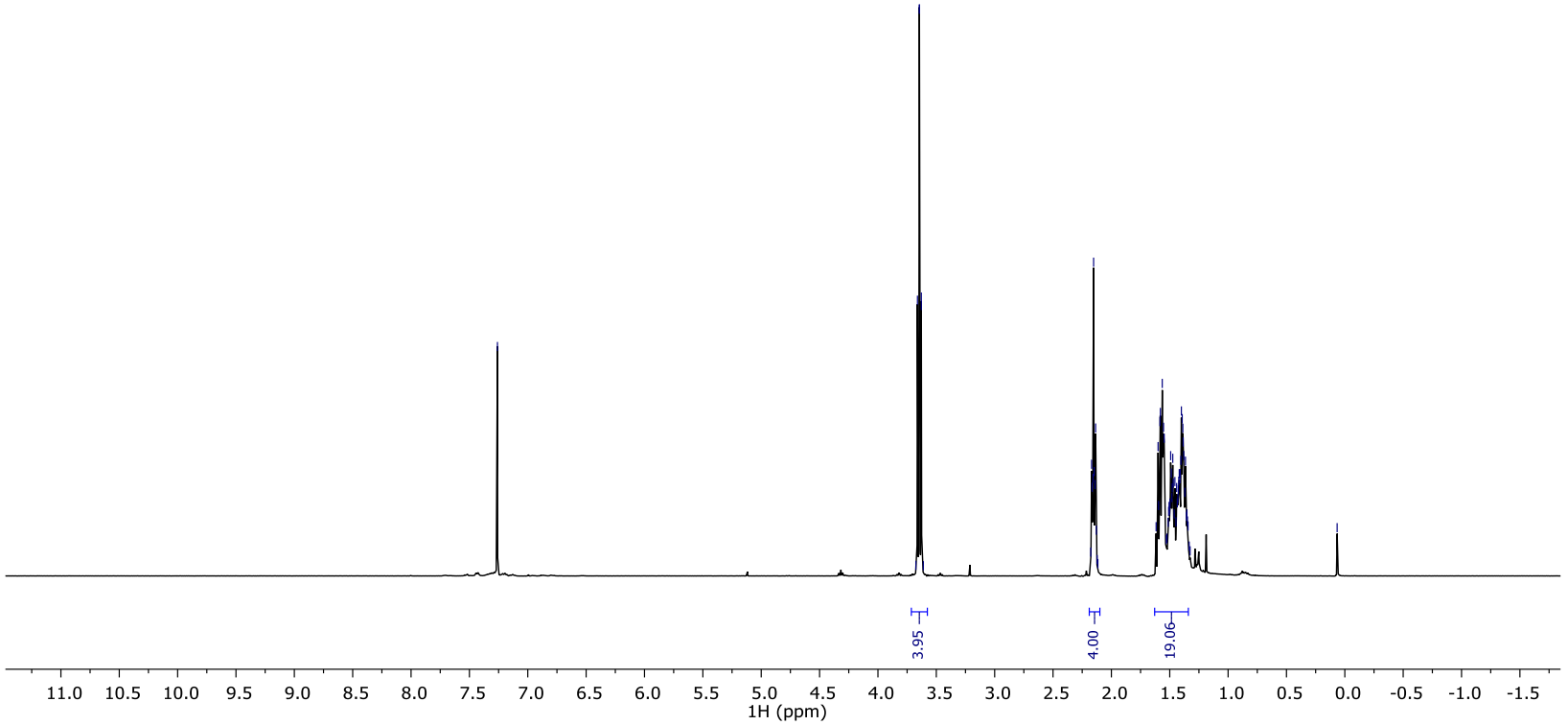
^{13}C NMR of Dodec-6-yne-1,12-diol (21a), 101 MHz, $CDCl_3$, 25ºC



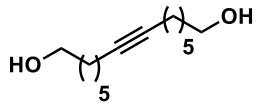
^{1}H NMR of Tetradec-7-yne-1,14-diol (21b), 400 MHz, $CDCl_3$, 25ºC

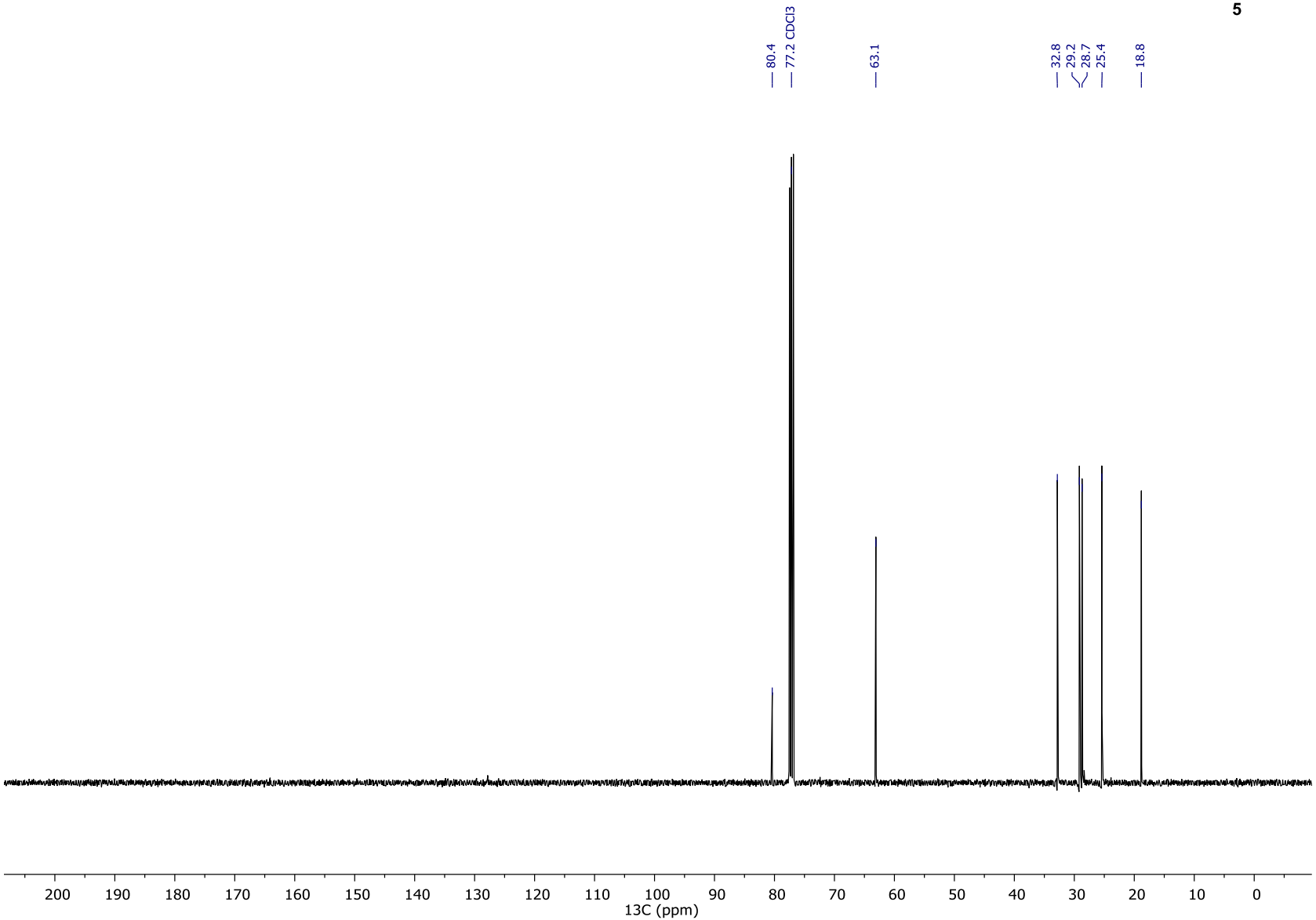




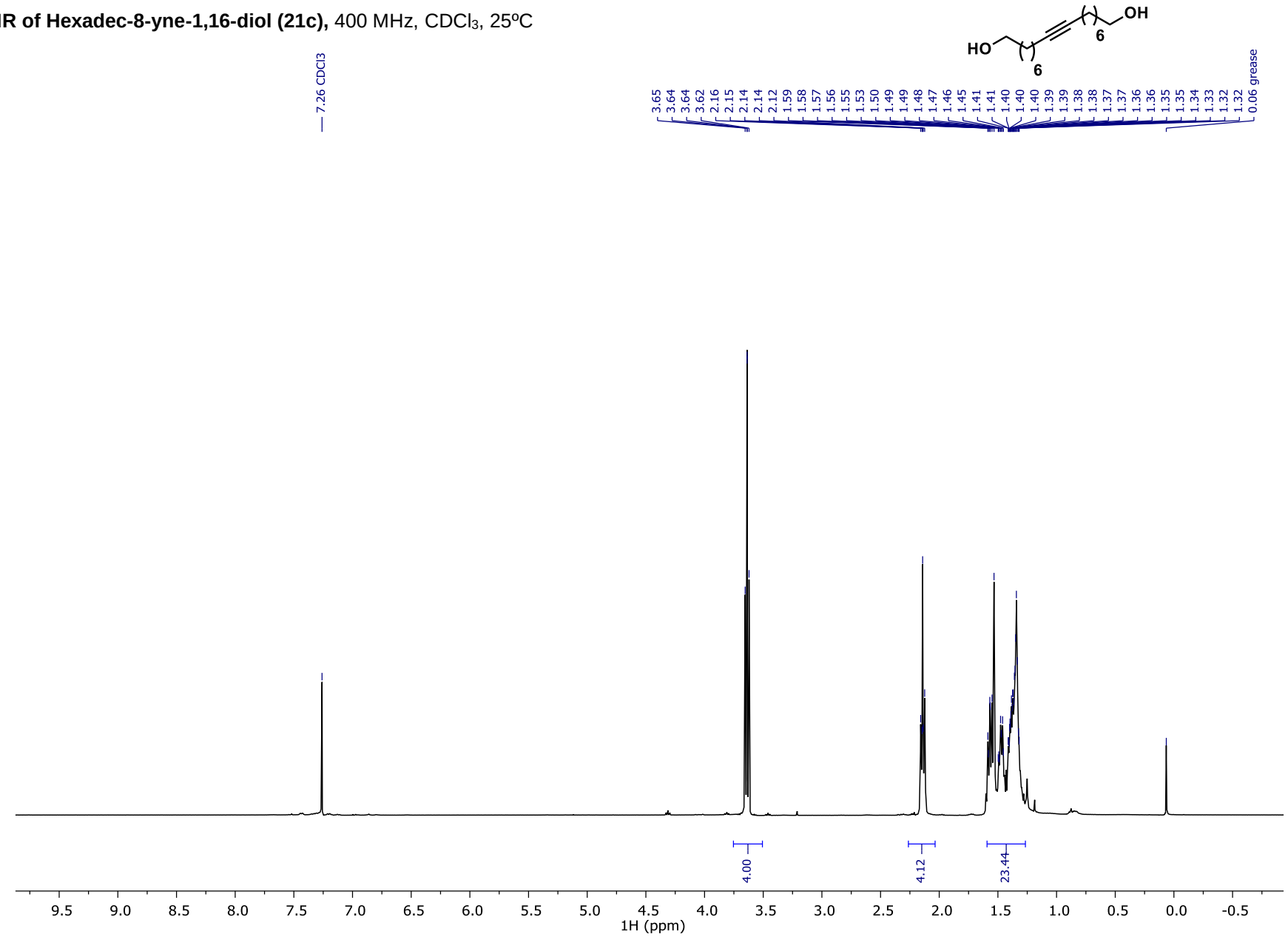


^{13}C NMR of Tetradec-7-yne-1,14-diol (21b), 101 MHz, $CDCl_3$, 25ºC

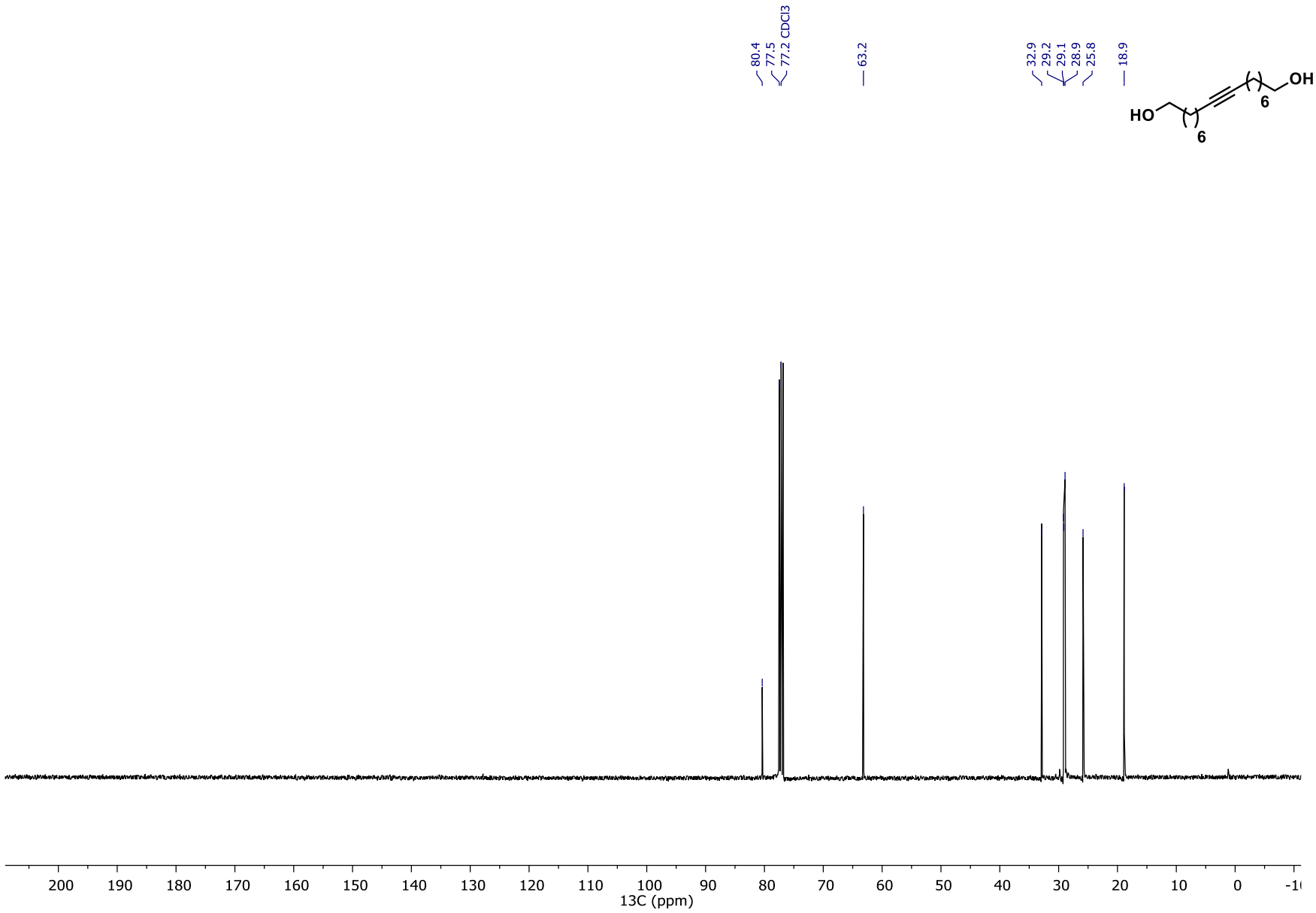




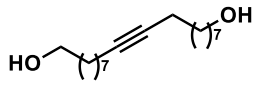
^{1}H NMR of Hexadec-8-yne-1,16-diol (21c), 400 MHz, $CDCl_3$, 25ºC

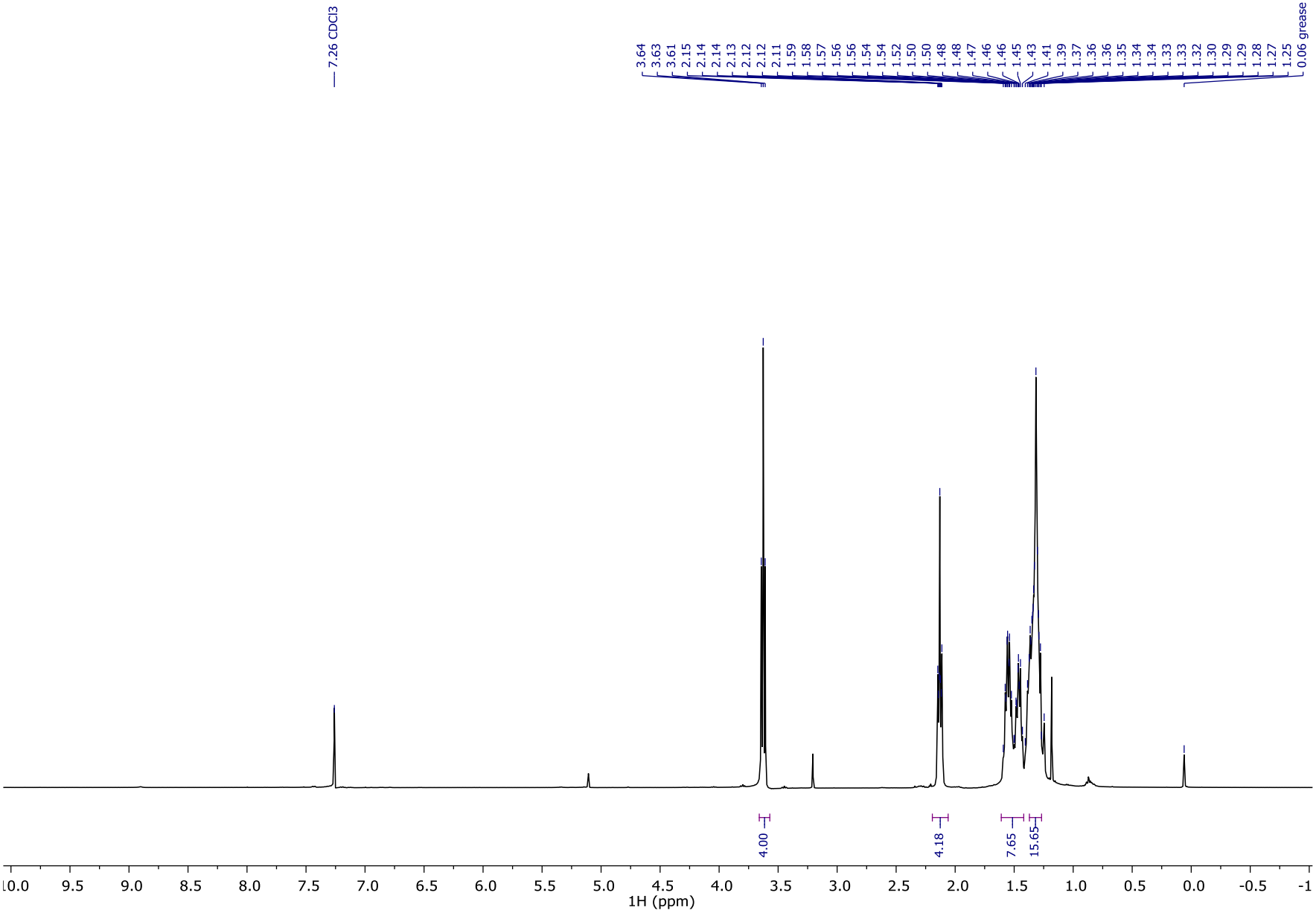


^{13}C NMR of Hexadec-8-yne-1,16-diol (21c), 101 MHz, $CDCl_3$, 25ºC

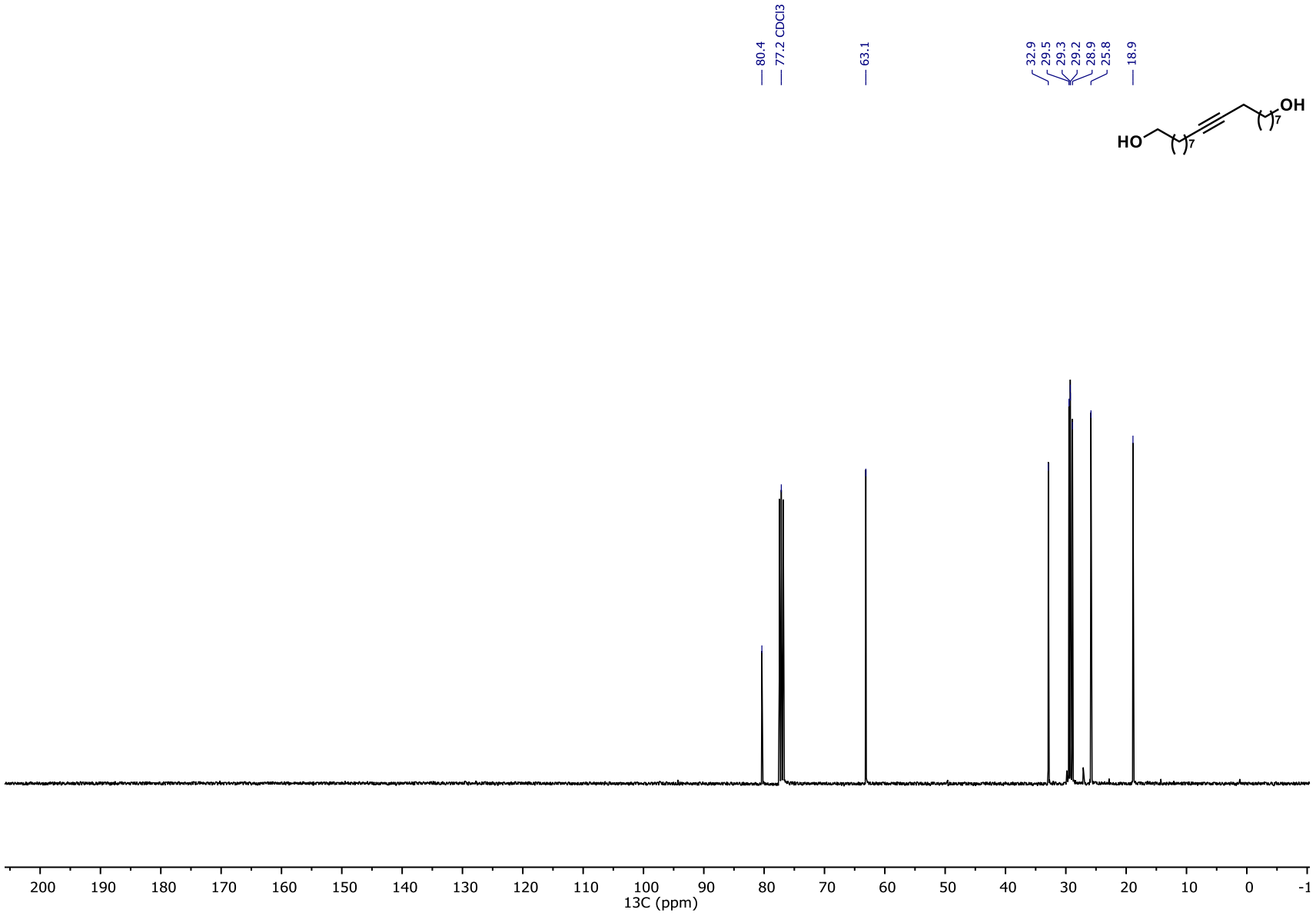


^{1}H NMR of Octadec-9-yne-1,18-diol (21d), 400 MHz, $CDCl_3$, 25ºC

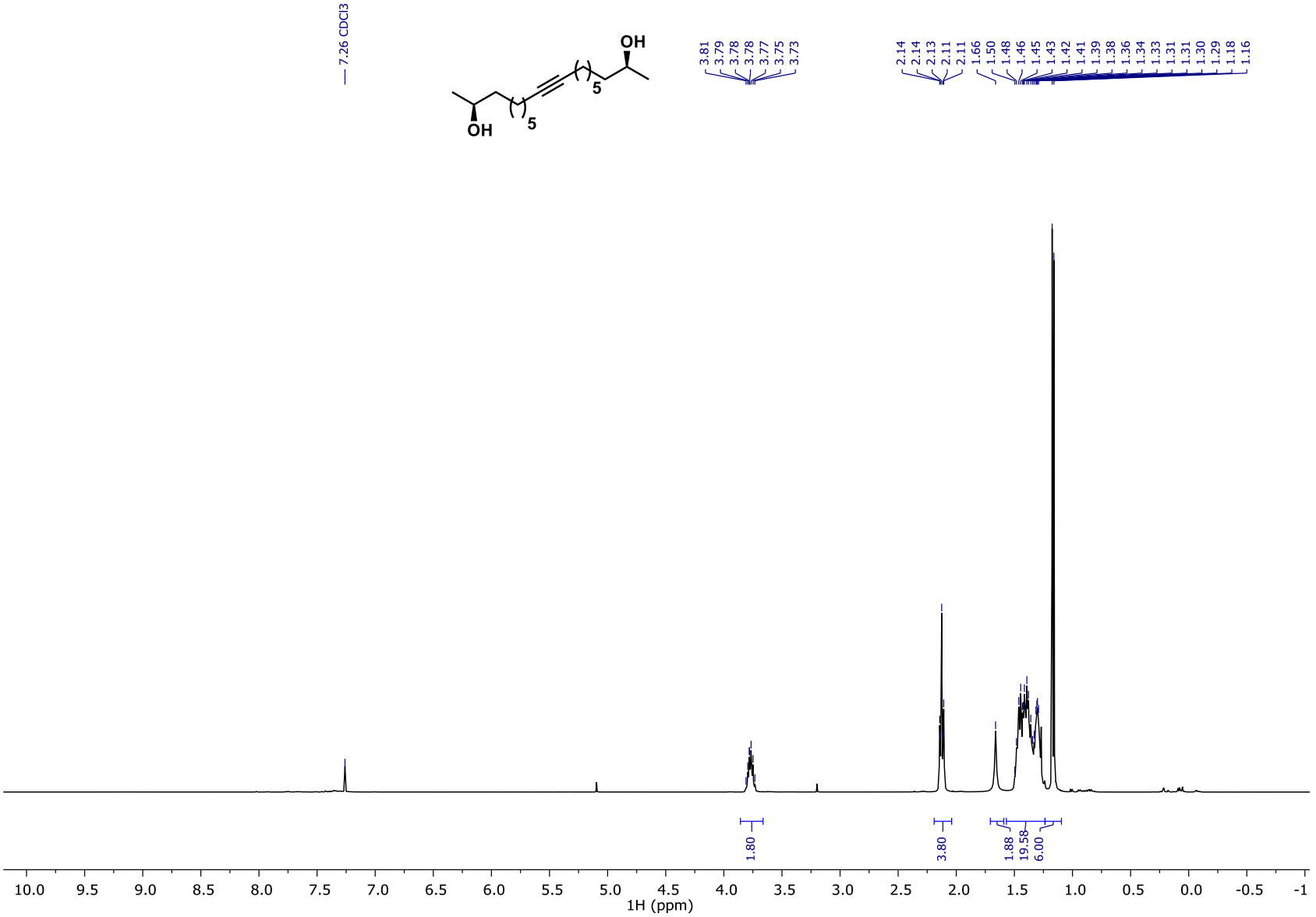




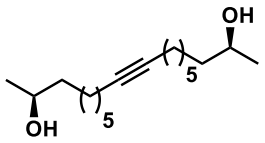
^{13}C NMR of Octadec-9-yne-1,18-diol (21d), 101 MHz, $CDCl_3$, 25ºC

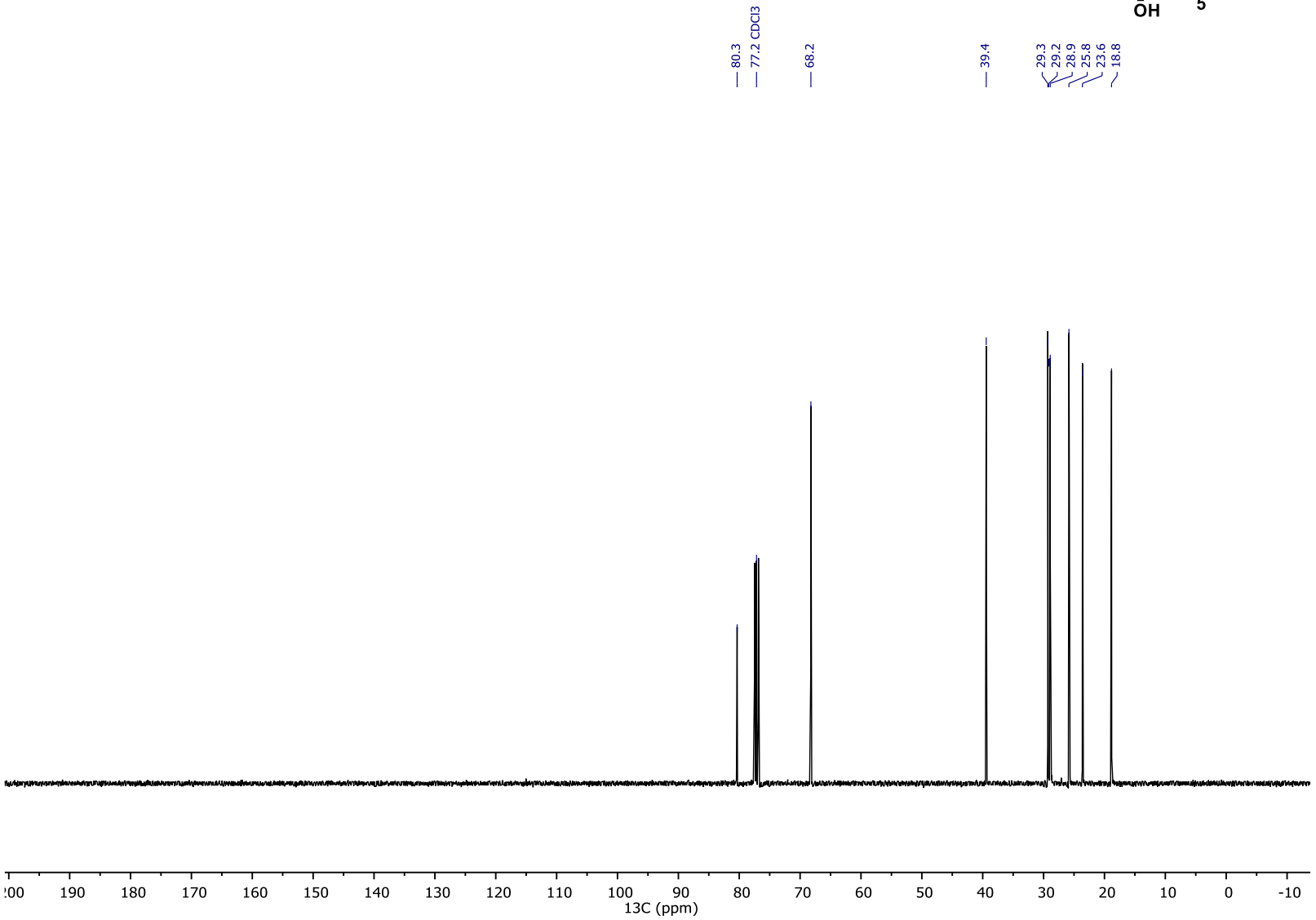


^{1}H NMR of (2S,17S)-octadec-9-yne-2,17-diol (22), 400 MHz, C_6D_6, 25ºC

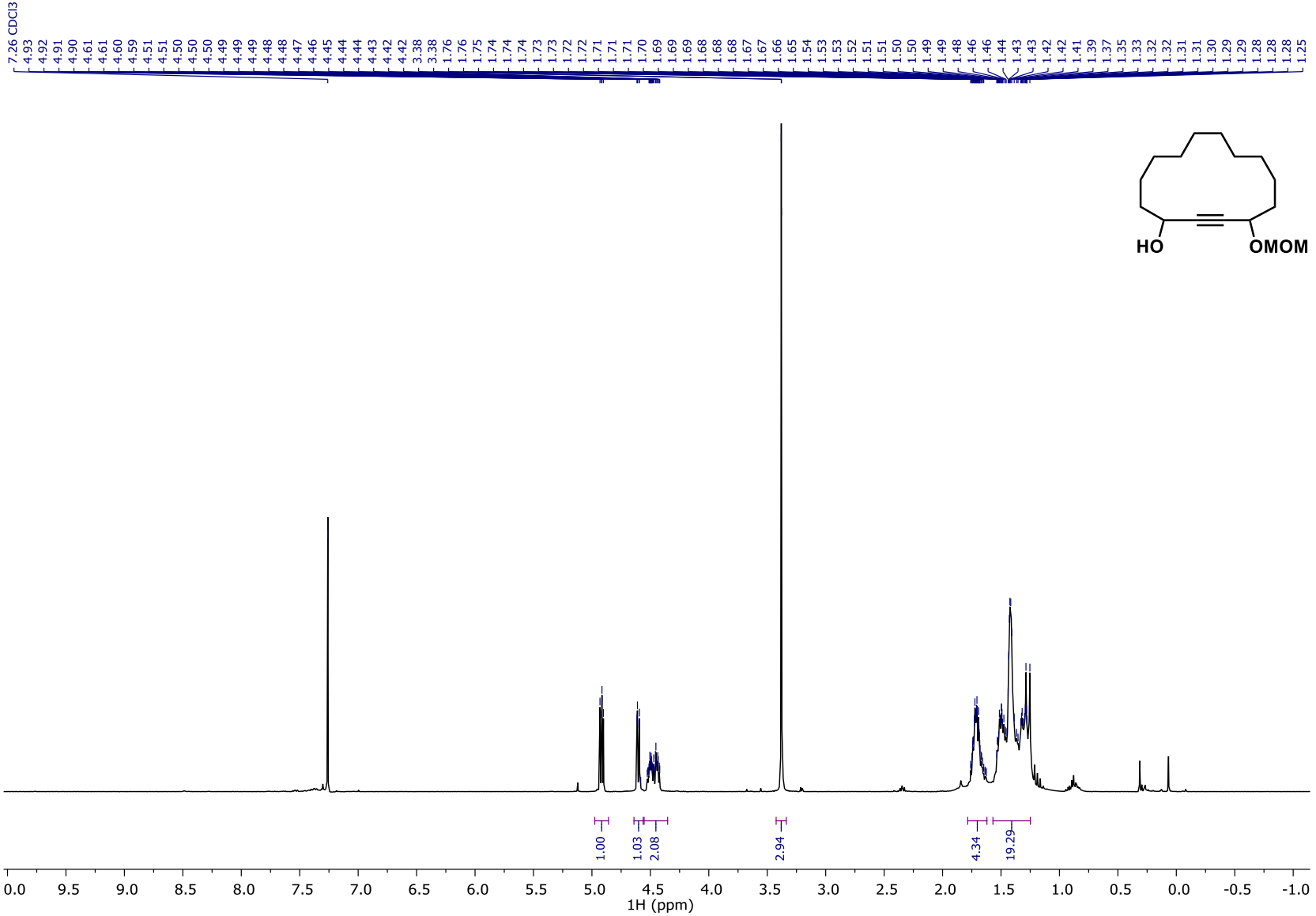


^{13}C NMR of (2S,17S)-octadec-9-yne-2,17-diol (22), 101 MHz, C_6D_6, 25ºC

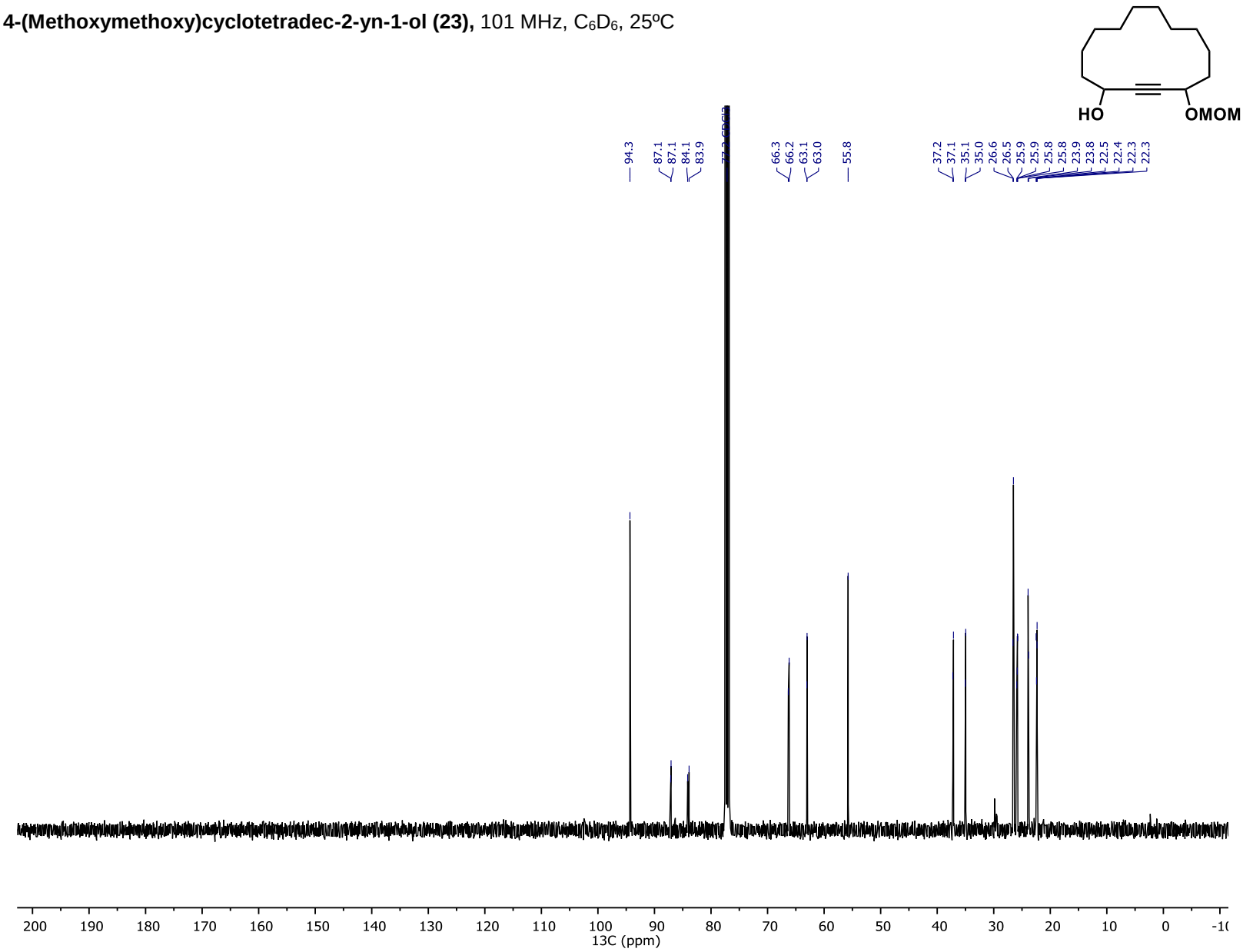




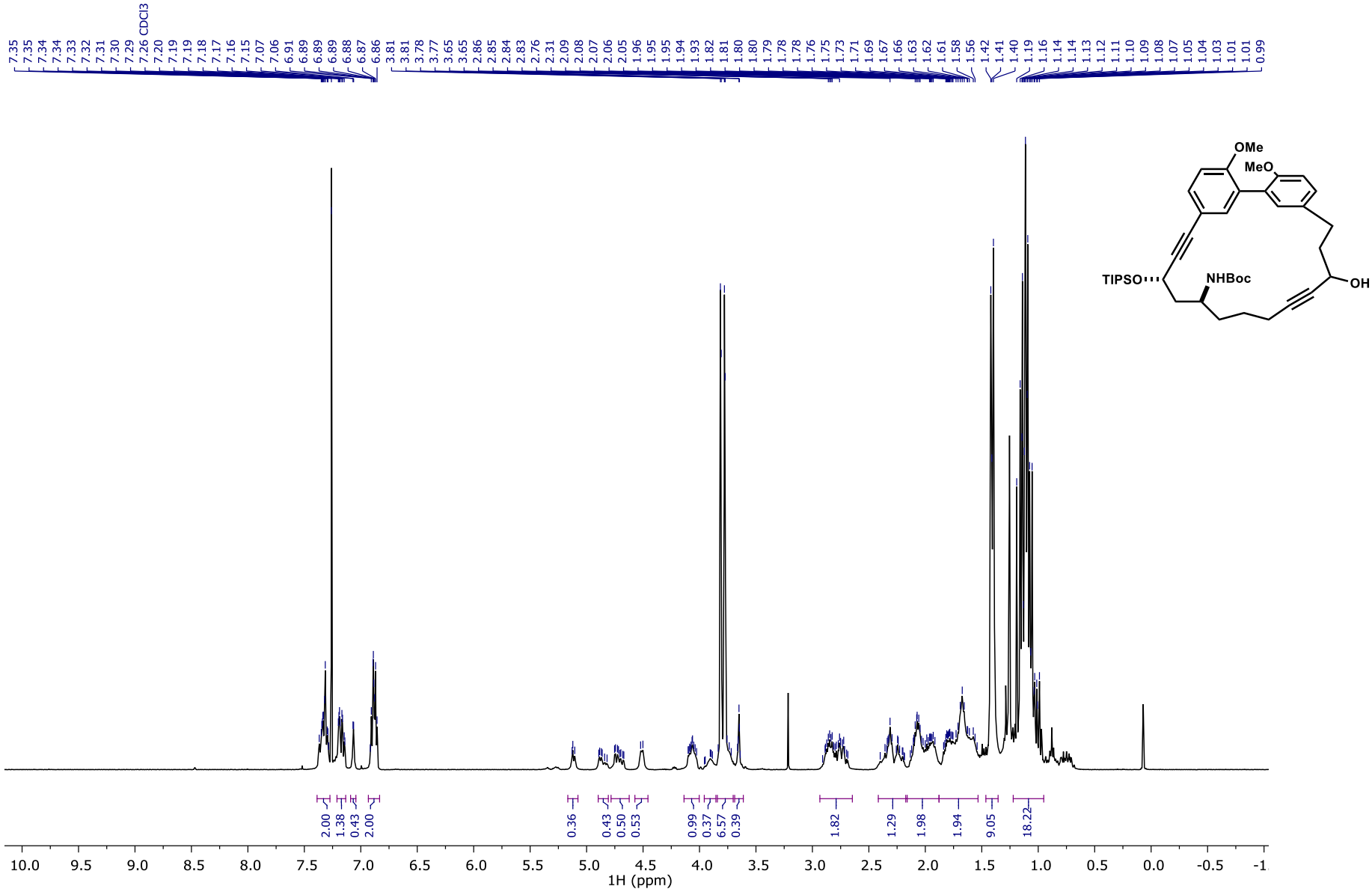
^{1}H NMR of 4-(Methoxymethoxy)cyclotetradec-2-yn-1-ol (23), 101 MHz, C_6D_6, 25ºC



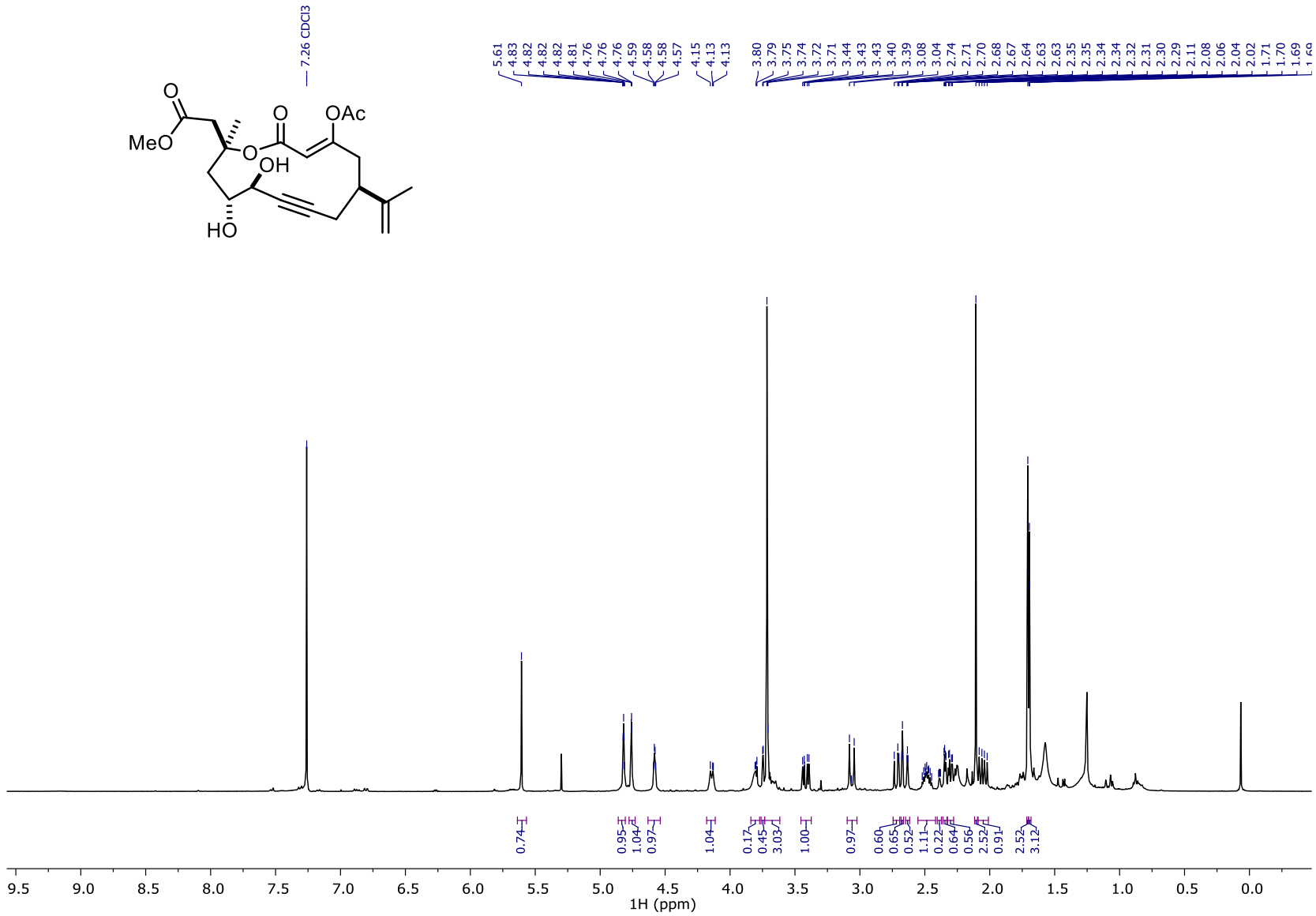
^{13}C NMR of 4-(Methoxymethoxy)cyclotetradec-2-yn-1-ol (23), 101 MHz, C_6D_6, 25ºC



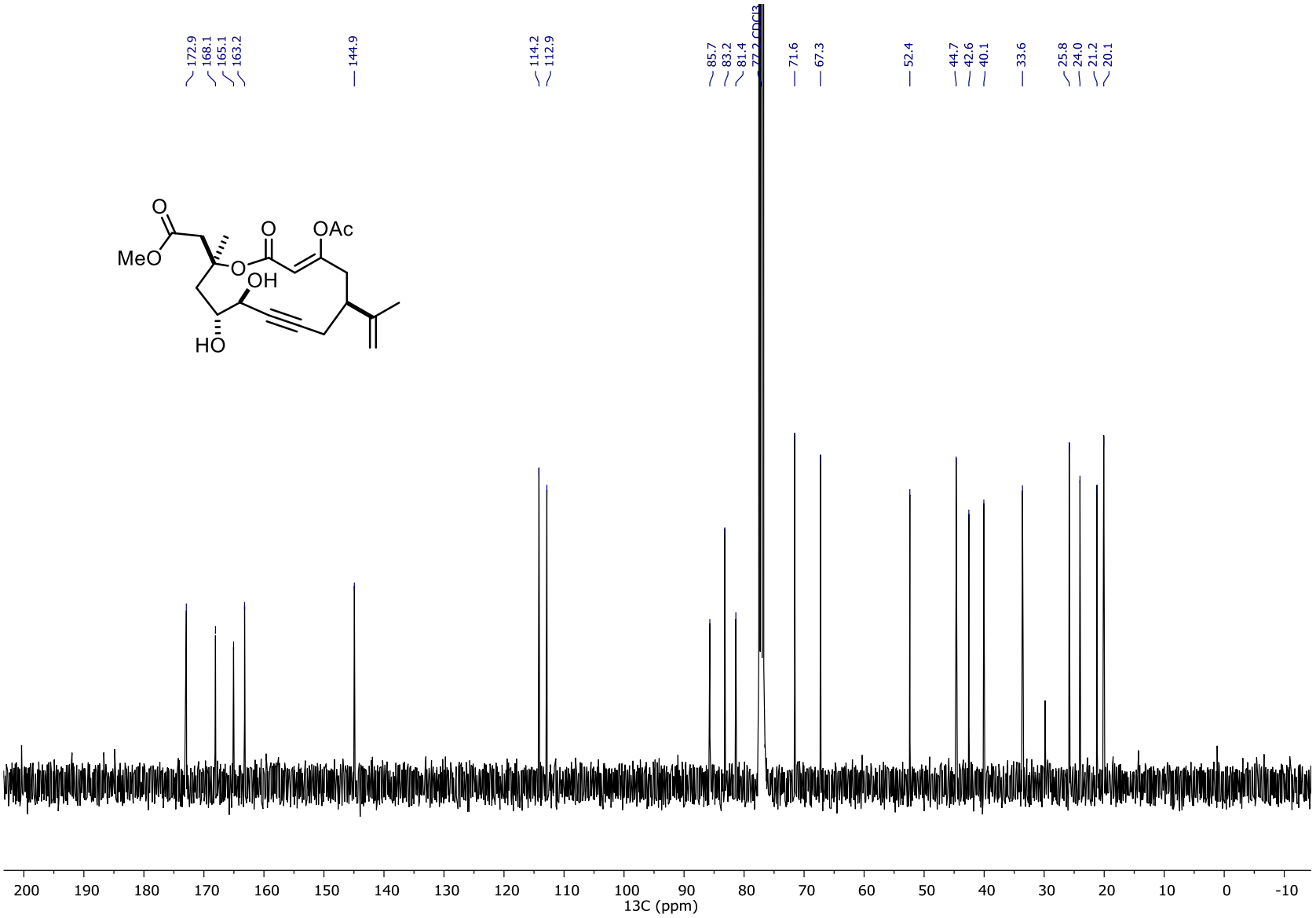
^{1}H NMR of Compound (24), 101 MHz, $CDCl_3$, 25ºC



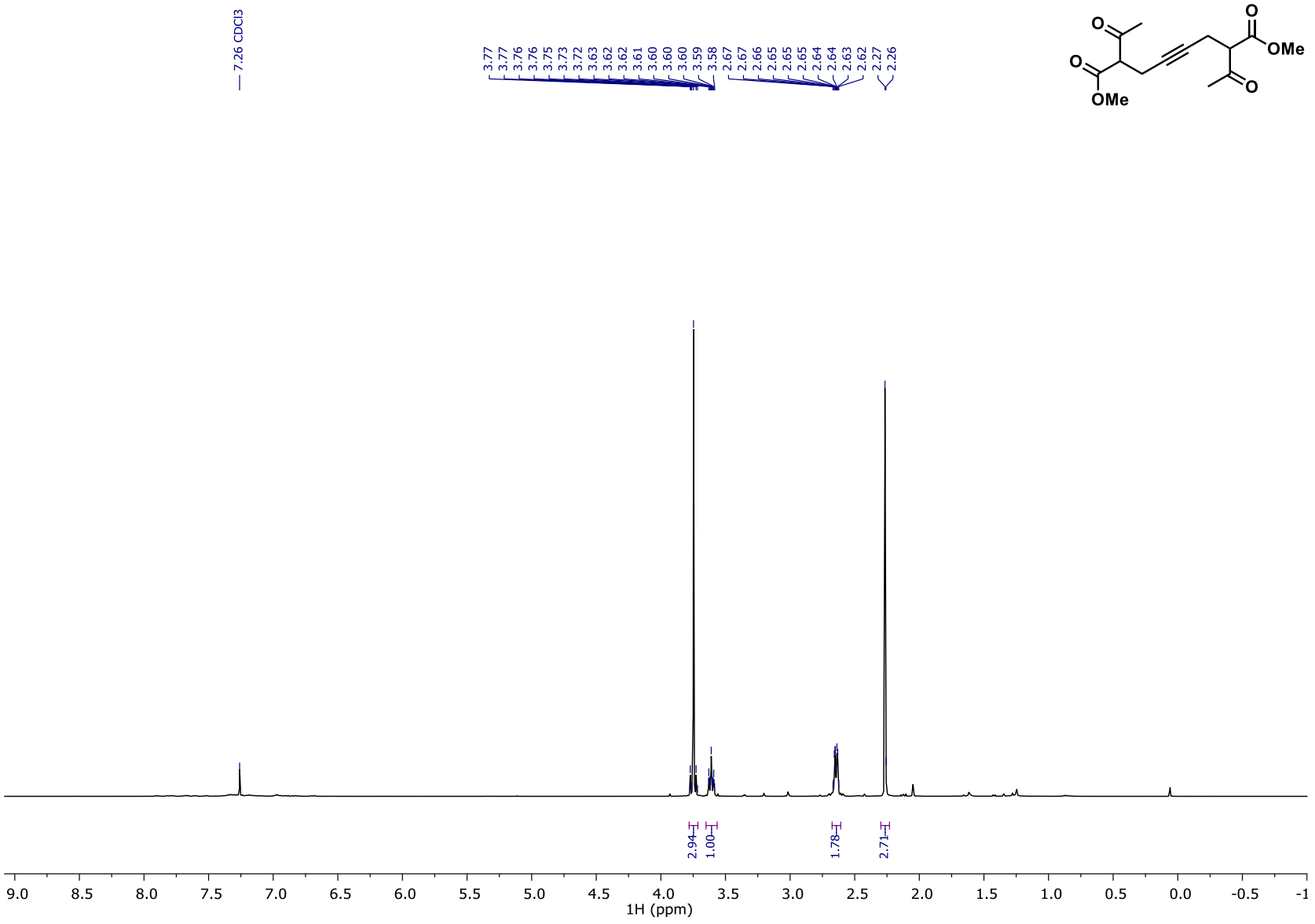
^{1}H NMR of compound 25, 400 MHz**,** $CDCl_3$, 25°C



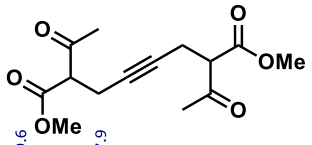
^{13}C NMR of compound 25, 101 MHz, $CDCl_3$, 25°C

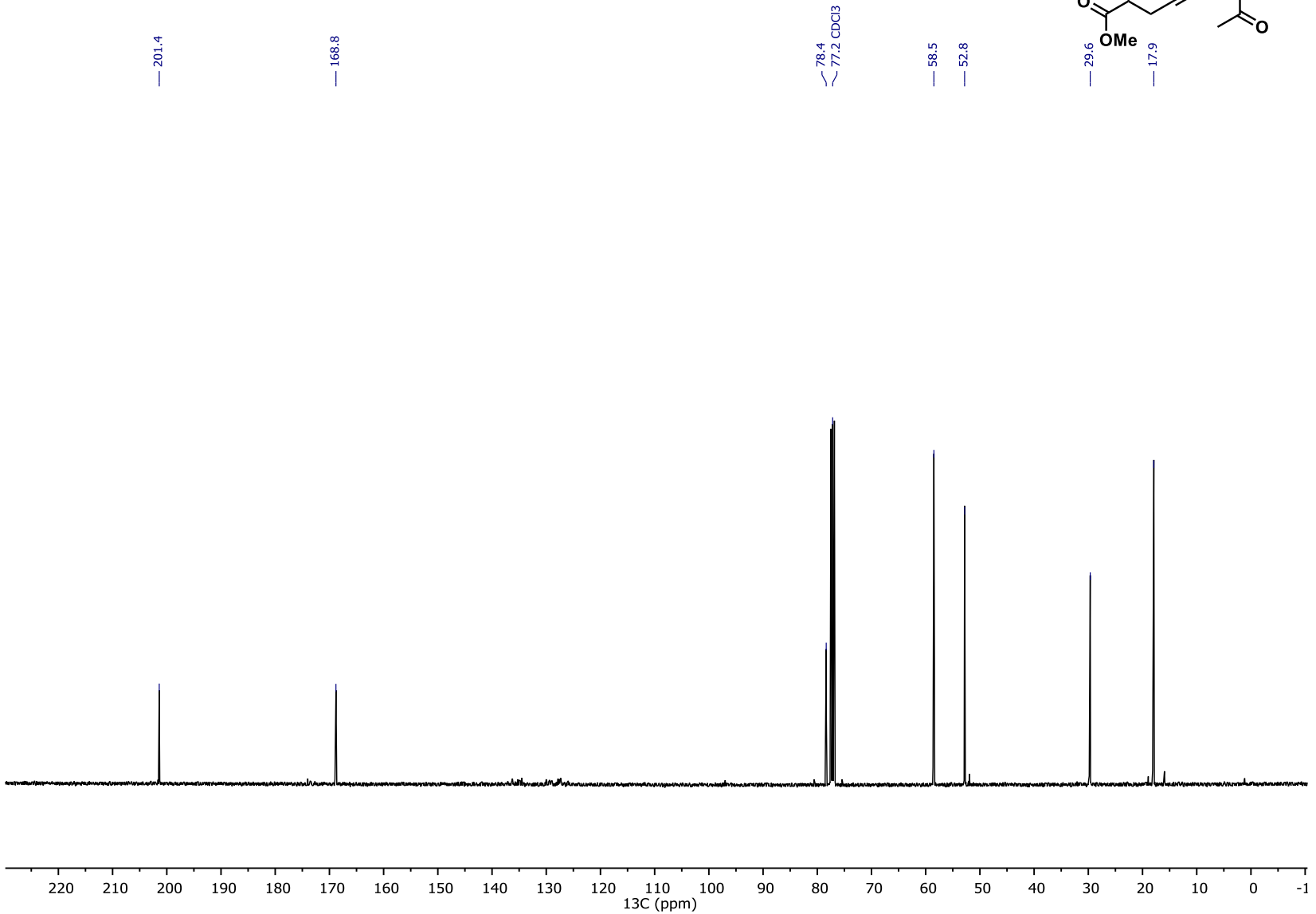


^{1}H NMR of Dimethyl 2,7-diacetyloct-4-ynedioate (26), 400 MHz, $CDCl_3$, 25ºC

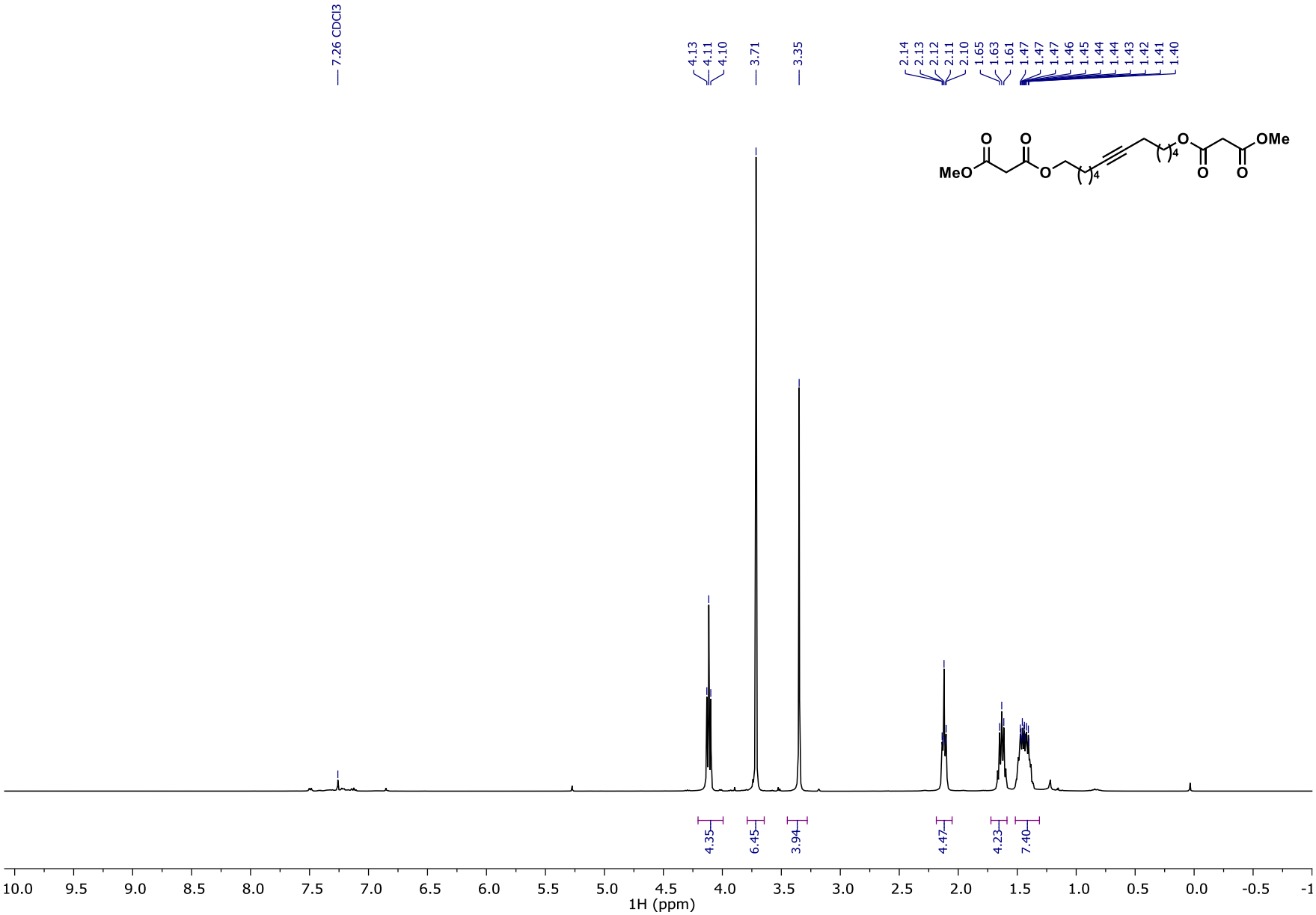


^{13}C NMR of Dimethyl 2,7-diacetyloct-4-ynedioate (26), 101 MHz, $CDCl_3$, 25ºC

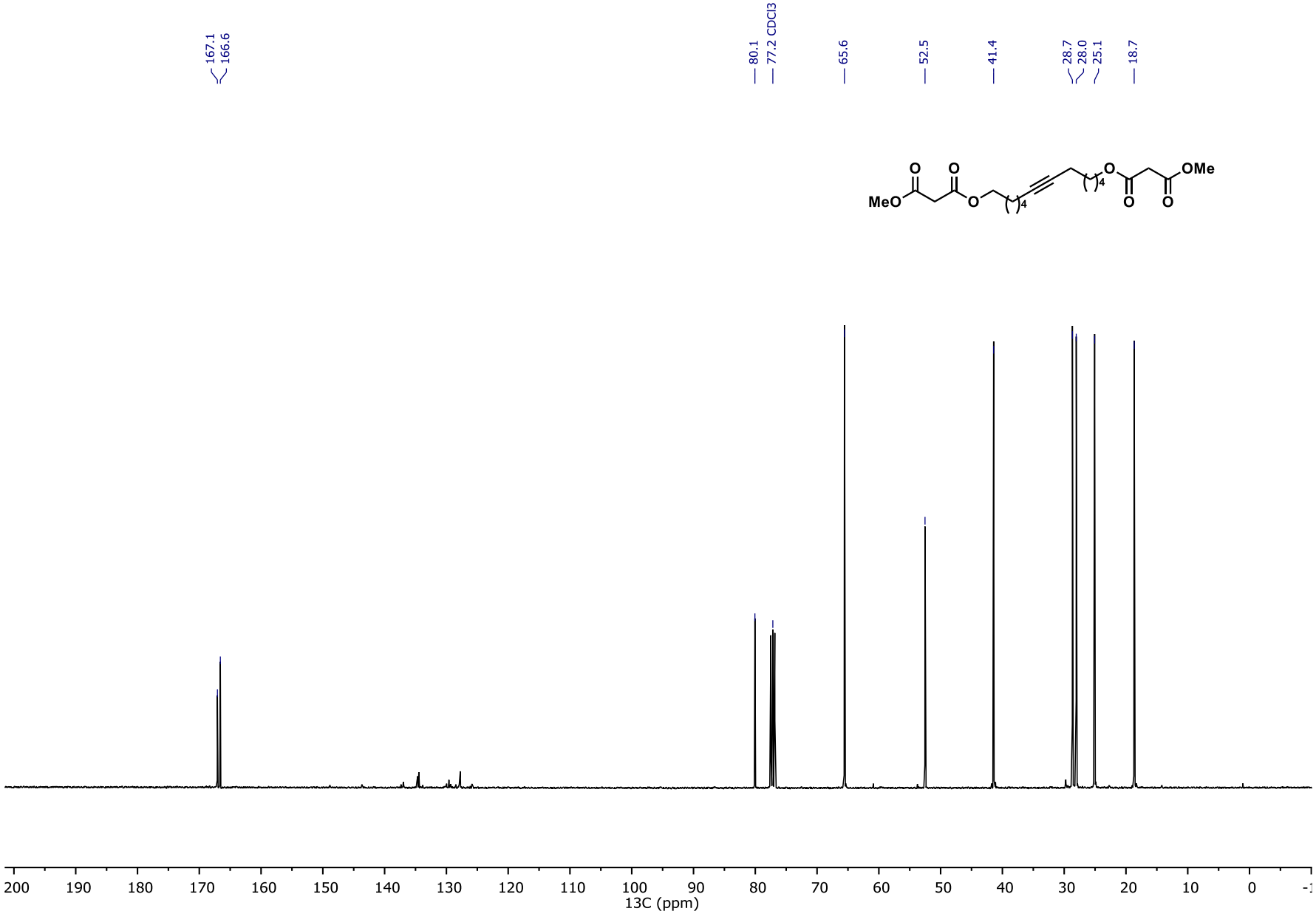




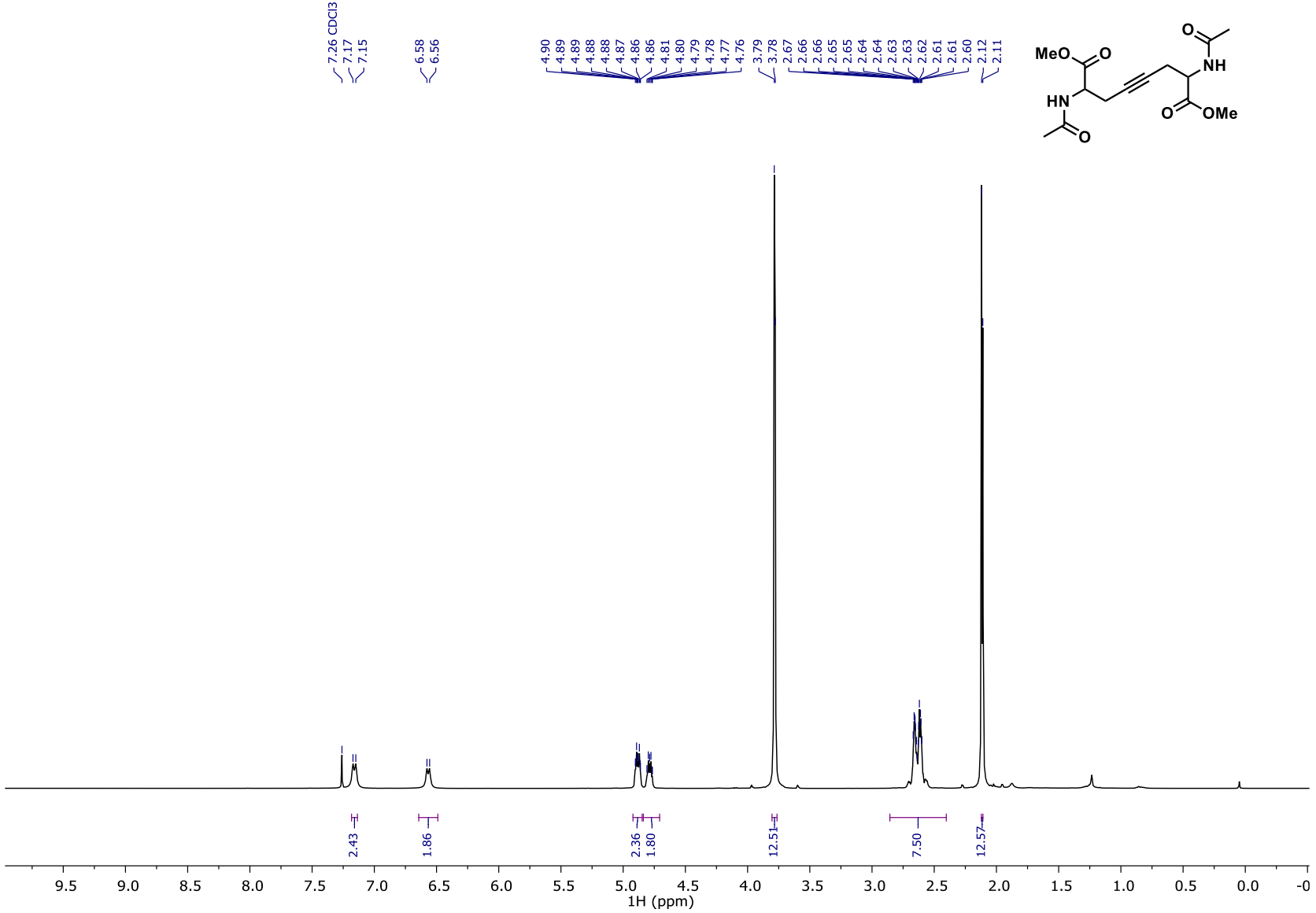
^{1}H NMR of *O*,*O*'-(Dodec-6-yne-1,12-diyl) dimethyl dimalonate (27), 400 MHz, $CDCl_3$, 25ºC



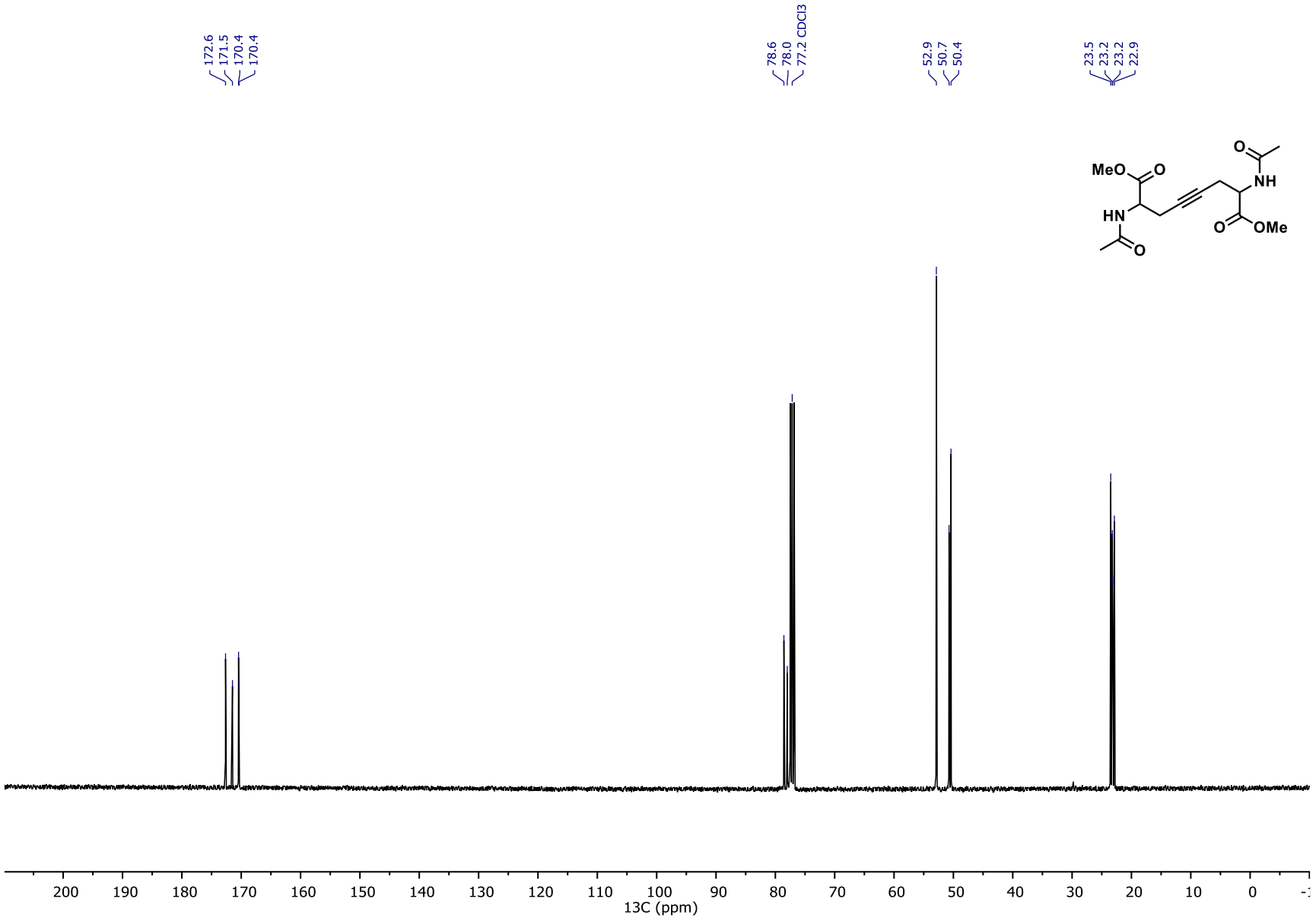
^{13}C NMR of O,O'-(Dodec-6-yne-1,12-diyl) dimethyl dimalonate (27), 101 MHz, $CDCl_3$, 25ºC



^{1}H NMR of Dimethyl 2,7-diacetamidooct-4-ynedioate (28), 400 MHz, $CDCl_3$, 25ºC

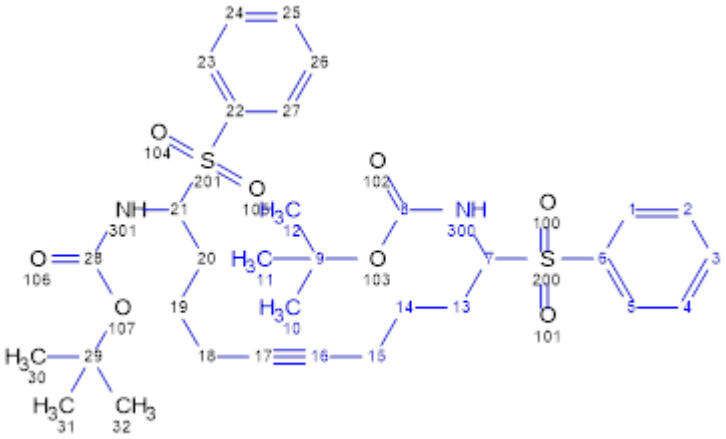


^{13}C NMR of Dimethyl 2,7-diacetamidooct-4-ynedioate (28), 101 MHz, $CDCl_3$, 25ºC



NMR Analysis of Di-tert-butyl (1,10-bis(phenylsulfonyl)dec-5-yne-1,10-diyl)dicarbamate Diastereomer 1 (29), C_6D_6, 25ºC

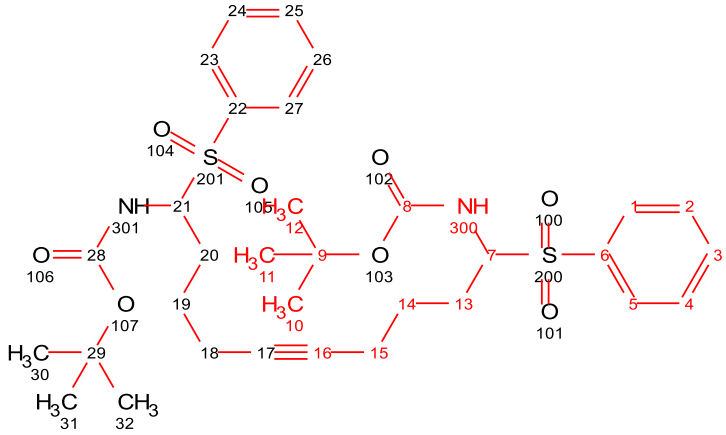
Atom	Chemical Shift	Quality	Predicted Shift	J	COSY	HSQC	HMBC	NOESY
1 C	129.73		127.92			1	3, 5	
H	8.12	0.71	7.87	7.82(2)	2	1	3, 5	2, 3, 7
2 C	128.52		129.23			2	4	
H	7.06	-0.7	7.58	7.82(1)	1, 3	2	4, 6	1, 10, 11, 12
3 C	133.09		134.19			3	1, 5	
H	7.06	-0.7	7.64		2, 4	3	1, 5	1, 5, 10, 11, 12
4 C	128.52		129.23			4	2	
H	7.06	-0.7	7.58		3, 5	4	2, 6	5, 10, 11, 12
5 C	129.73		127.92			5	1, 3	
H	8.12	0.71	7.87		4	5	1, 3	3, 4, 7
6 C	138.09		136.77				2, 4	
7 C	71.18		71.24			7	13", 14'	
H	5.19	0.71	4.96	5.00(13'), 5.00(13")	13', 300	7	8, 13, 14	1, 5, 13", 14'
8 C	154.27		156.94				7	
9 C	79.4		79.18				10, 11, 12	
10 C	27.73		28.31			10	11, 12	
H3	1.14	0.71	1.4			10	9, 11, 12	2, 3, 4
11 C	27.73		28.31			11	10, 12	
H3	1.14	0.71	1.4			11	9, 10, 12	2, 3, 4
12 C	27.73		28.31			12	10, 11	
H3	1.14	0.71	1.4			12	9, 10, 11	2, 3, 4
13 C	26.4		27.48			13', 13"	7, 14', 15', 15"	
H'	2.07	0.71	2.08, 2.00	5.00(7)	7, 13", 14', 14"	13	14	13"
H"	2.44	0.71	2.08, 2.00	5.00(7)	13', 14', 14"	13	7, 14, 15	7, 13'
14 C	24.43		24.25			14', 14"	7, 13', 13", 15', 15"	
H'	1.37	0.5	1.66, 1.63		13', 13", 14"	14	7, 13, 15, 16	7, 14"
H"	1.48	0.71	1.66, 1.63		13', 13", 14', 15', 15"	14		14'



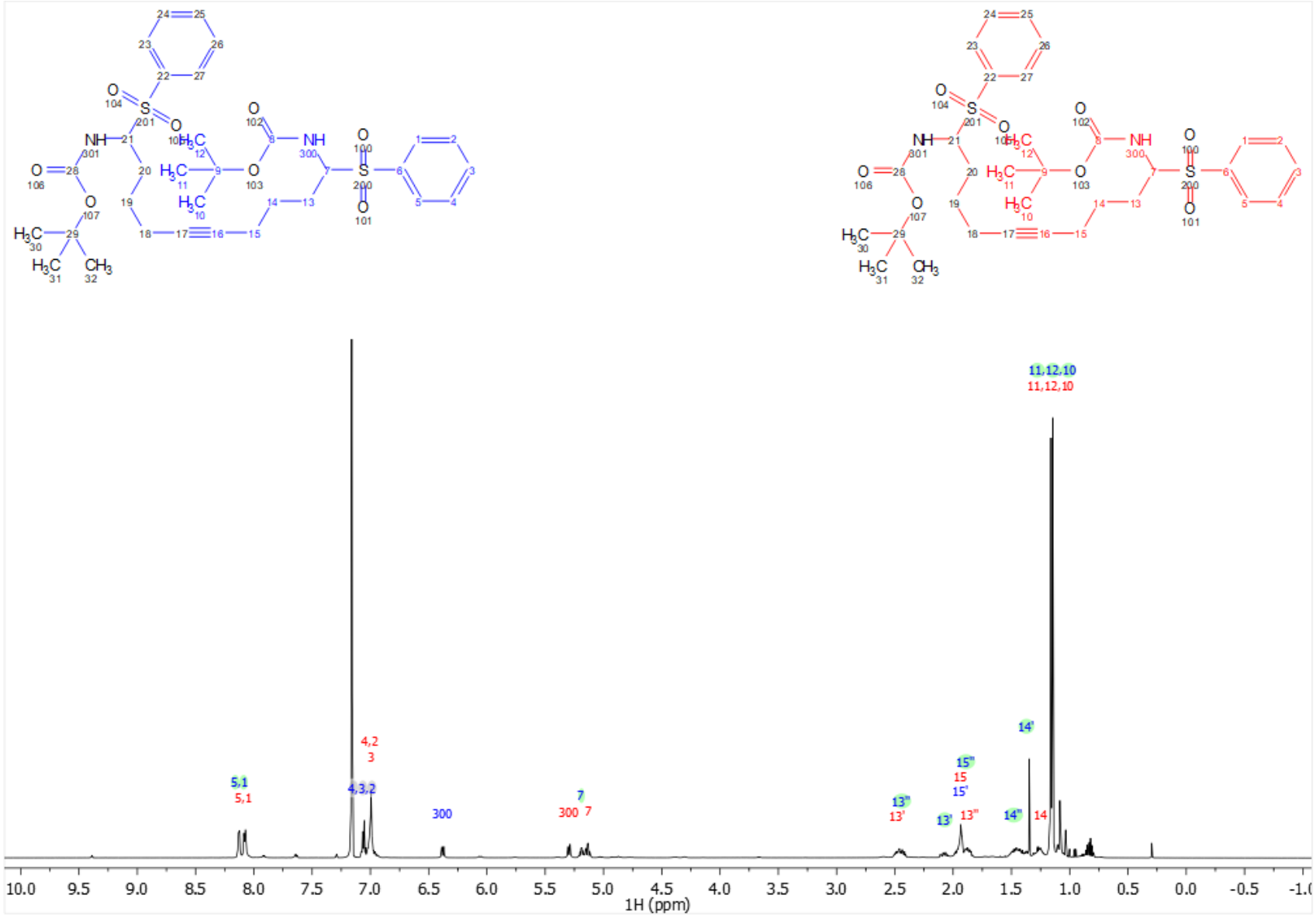
Atom	Chemical Shift	Quality	Predicted Shift	J	COSY	HSQC	HMBC	NOESY
15 C	18.29		18.24			15', 15"	13", 14'	
H'	1.93		2.34, 2.31		14"	15	13, 14, 16	
H"	1.89	0.71	2.34, 2.31		14"	15	13, 14, 16	
16 C	80.34		78.77				14', 15', 15"	

NMR Analysis of Di-tert-butyl (1,10-bis(phenylsulfonyl)dec-5-yne-1,10-diyl)dicarbamate Diastereomer 2 (29), C_6D_6, 25ºC

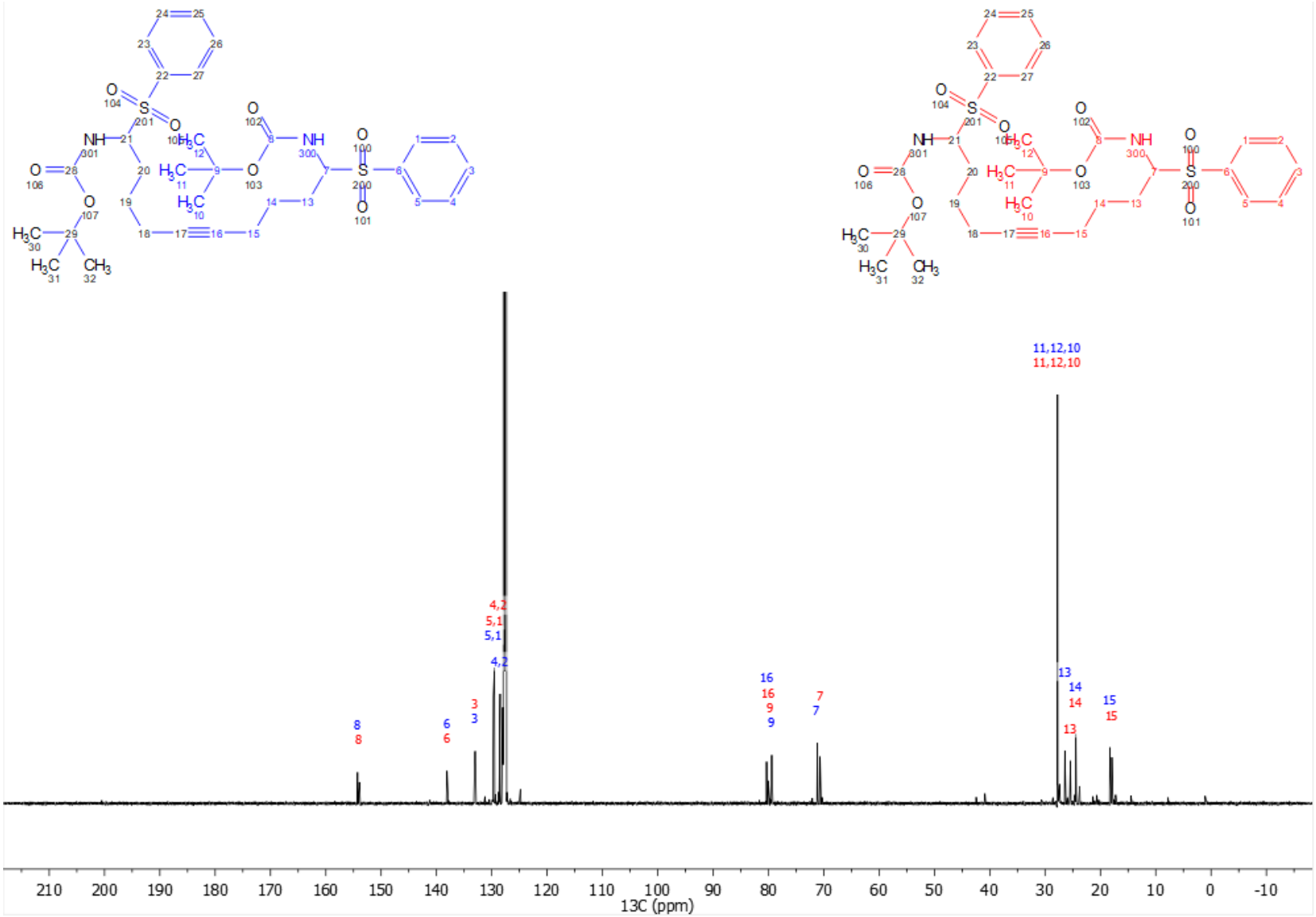
Atom	Chemical Shift	Predicted Shift	COSY	HSQC	HMBC	NOESY
1 C	129.54	127.92		1	3, 5	
H	8.08	7.87		1	3, 5	7, 300
2 C	128.64	129.23		2	4	
H	7	7.58		2	4, 6	
3 C	132.98	134.19		3	1, 5	
H	6.99	7.64		3	1, 5	
4 C	128.64	129.23		4	2	
H	7	7.58		4	2, 6	
5 C	129.54	127.92		5	1, 3	
H	8.08	7.87		5	1, 3	7, 300
6 C	138.11	136.77			2, 4	
7 C	70.68	71.24		7	13", 14'	
H	5.13	4.96	300	7	8, 13, 14	1, 5
8 C	153.95	156.94			7	
9 C	79.56	79.18			10, 11, 12	
10 C	27.74	28.31		10	11, 12	
H3	1.16	1.4		10	9, 11, 12	
11 C	27.74	28.31		11	10, 12	
H3	1.16	1.4		11	9, 10, 12	
12 C	27.74	28.31		12	10, 11	
H3	1.16	1.4		12	9, 10, 11	
13 C	25.43	27.48		13', 13"	7, 14', 15	
H'	2.48	2.08, 2.00		13	14	13"
H"	1.86	2.08, 2.00		13	7, 14, 15	13'
Atom	Chemical Shift	Predicted Shift	COSY	HSQC	HMBC	NOESY
14 C	24.48	24.25		14', 14"	7, 13', 13", 15	
H'	1.25	1.66, 1.63		14	7, 13, 15, 16	
H"	1.25	1.66, 1.63		14		
15 C	17.92	18.24		15	13", 14'	
H2	1.93	2.34, 2.31		15	13, 14, 16	
16 C	80.01	78.77			14', 15	



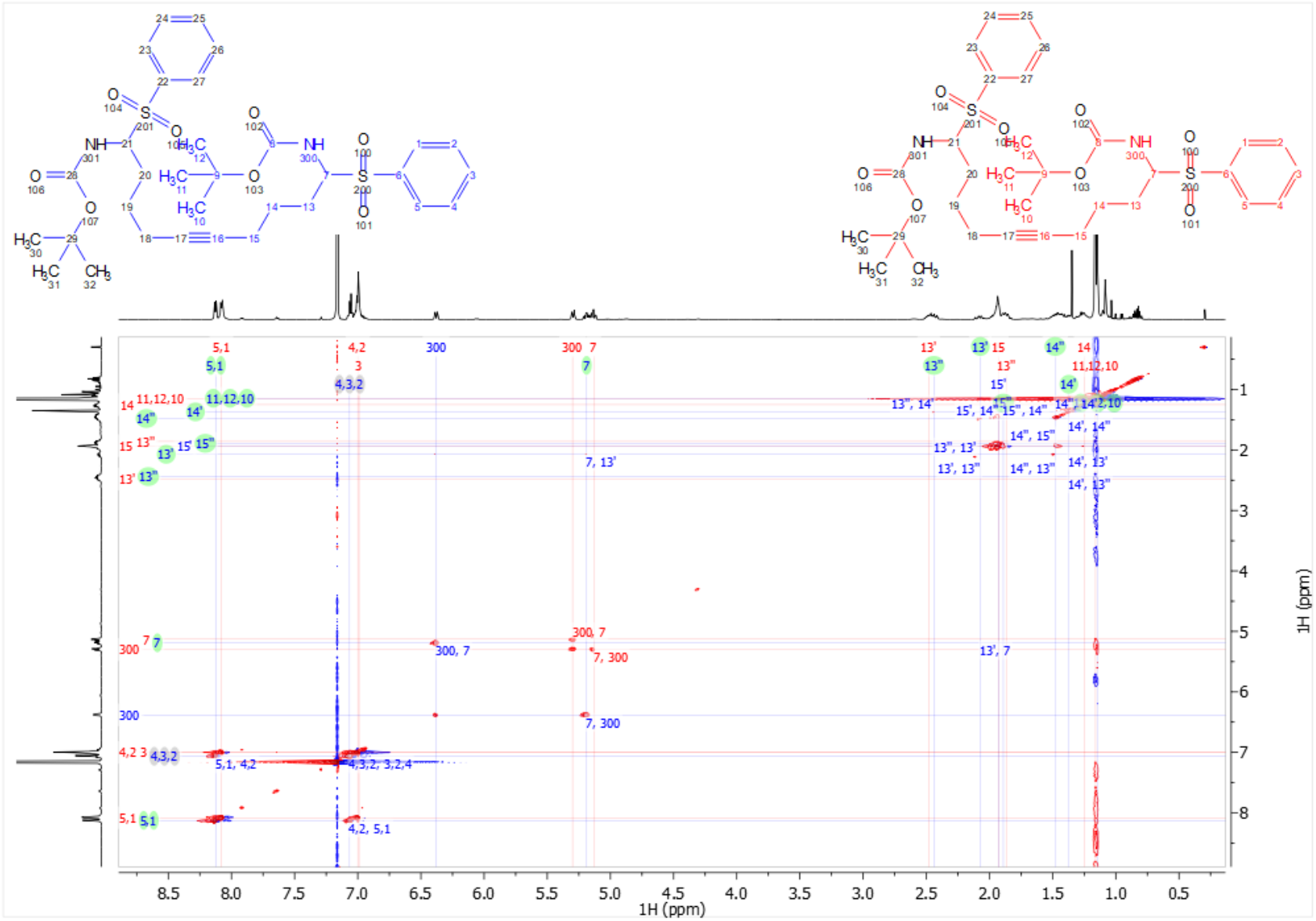
^{1}H NMR of Di-tert-butyl (1,10-bis(phenylsulfonyl)dec-5-yne-1,10-diyl)dicarbamate (29), 600 MHz, C_6D_6, 25ºC



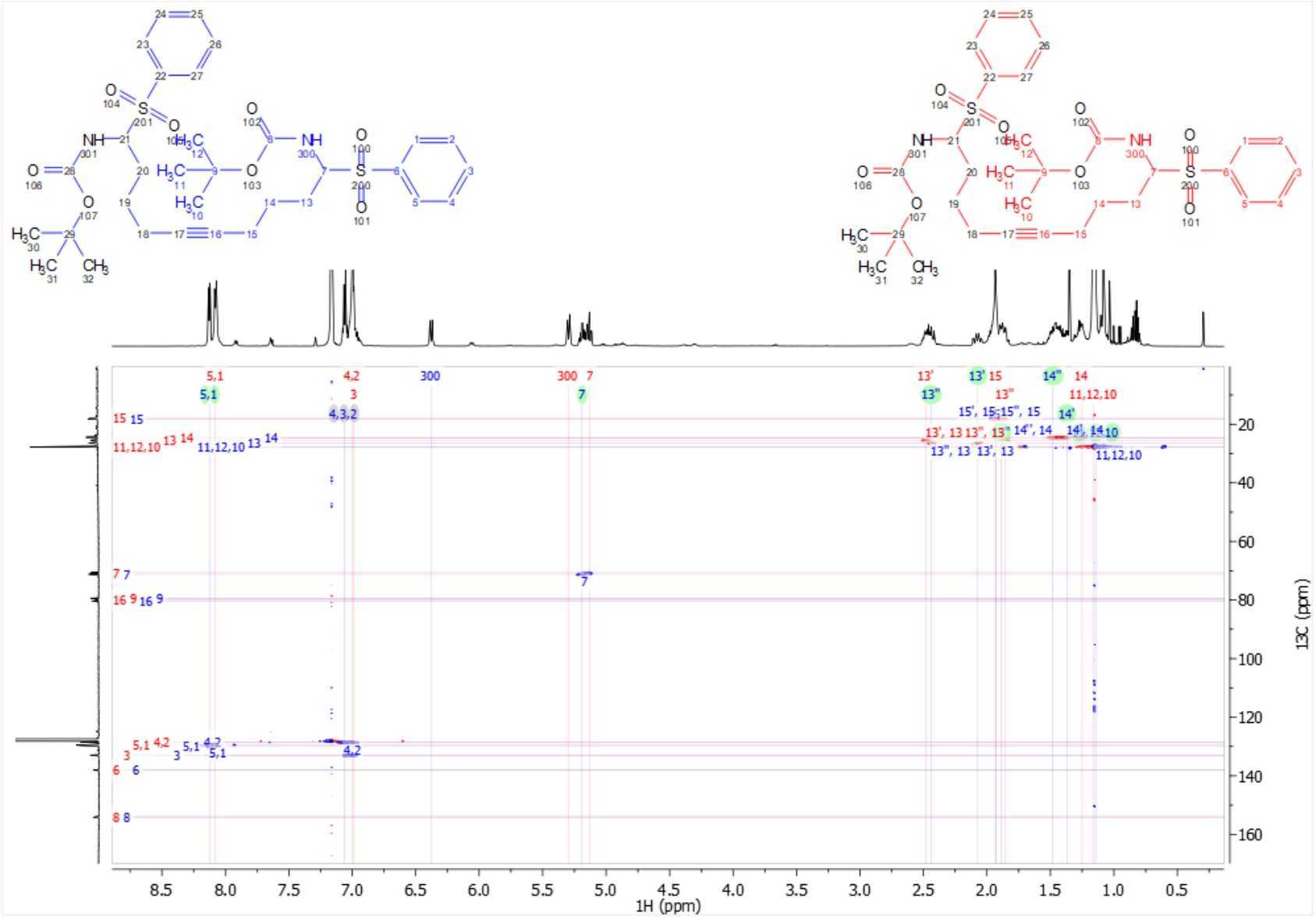
^{13}C NMR of Di-tert-butyl (1,10-bis(phenylsulfonyl)dec-5-yne-1,10-diyl)dicarbamate (29), 151 MHz, C_6D_6, 25ºC



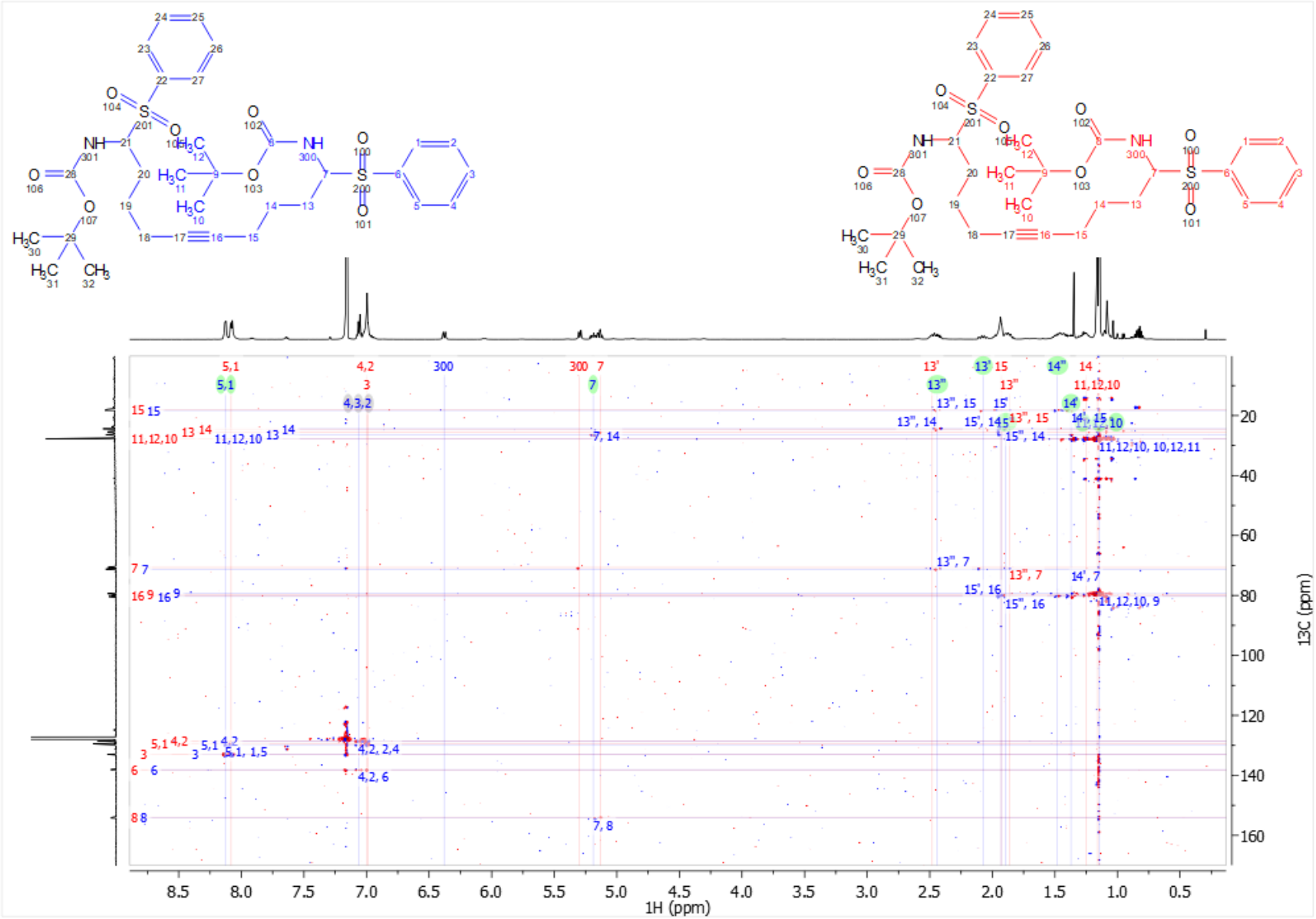
^{1}H ^{1}H COSY NMR of Di-tert-butyl (1,10-bis(phenylsulfonyl)dec-5-yne-1,10-diyl)dicarbamate (29), 600 MHz, C_6D_6, 25ºC



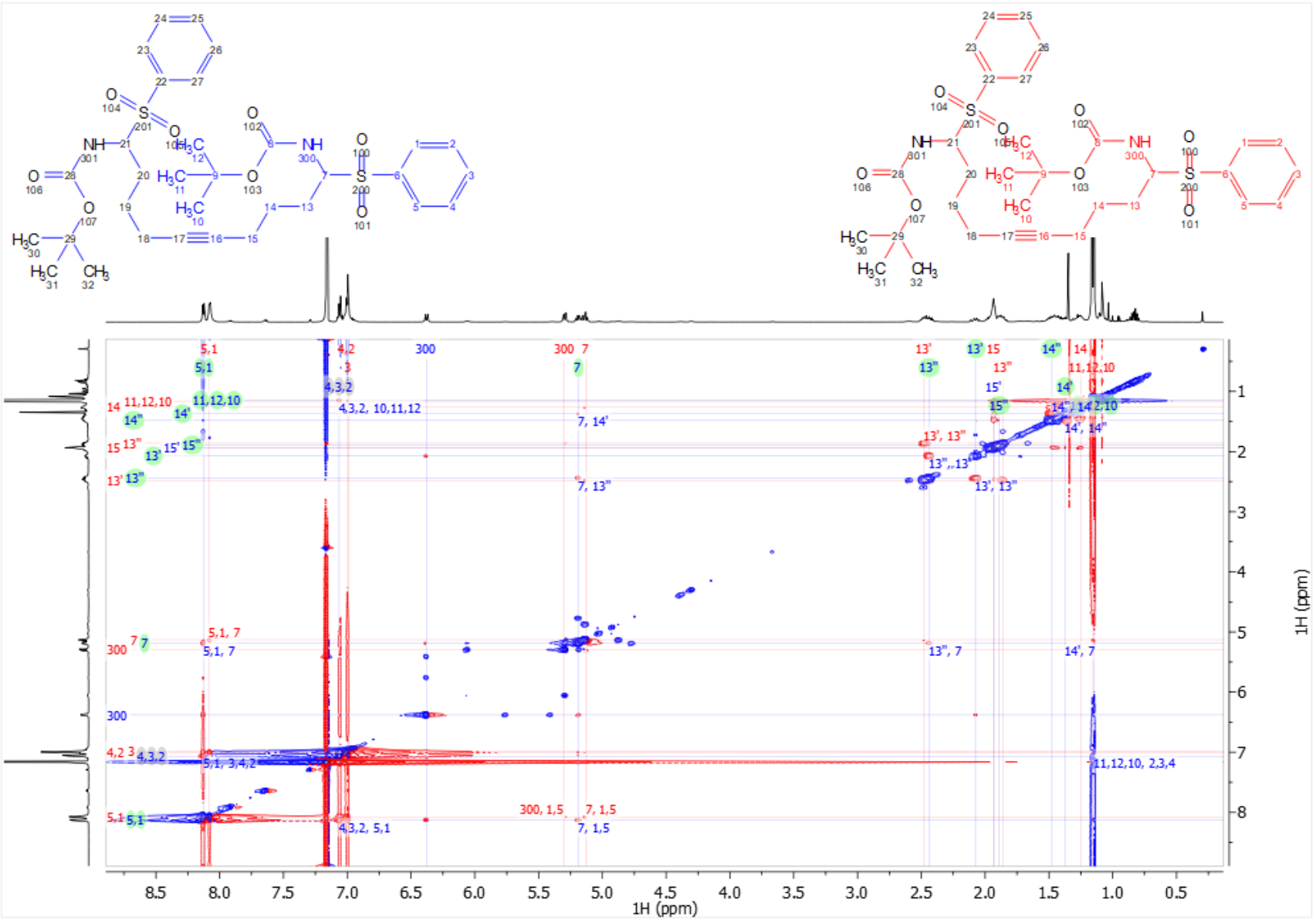
^{1}H ^{13}C HSQC NMR of Di-tert-butyl (1,10-bis(phenylsulfonyl)dec-5-yne-1,10-diyl)dicarbamate (29), 600 MHz, C_6D_6, 25°C



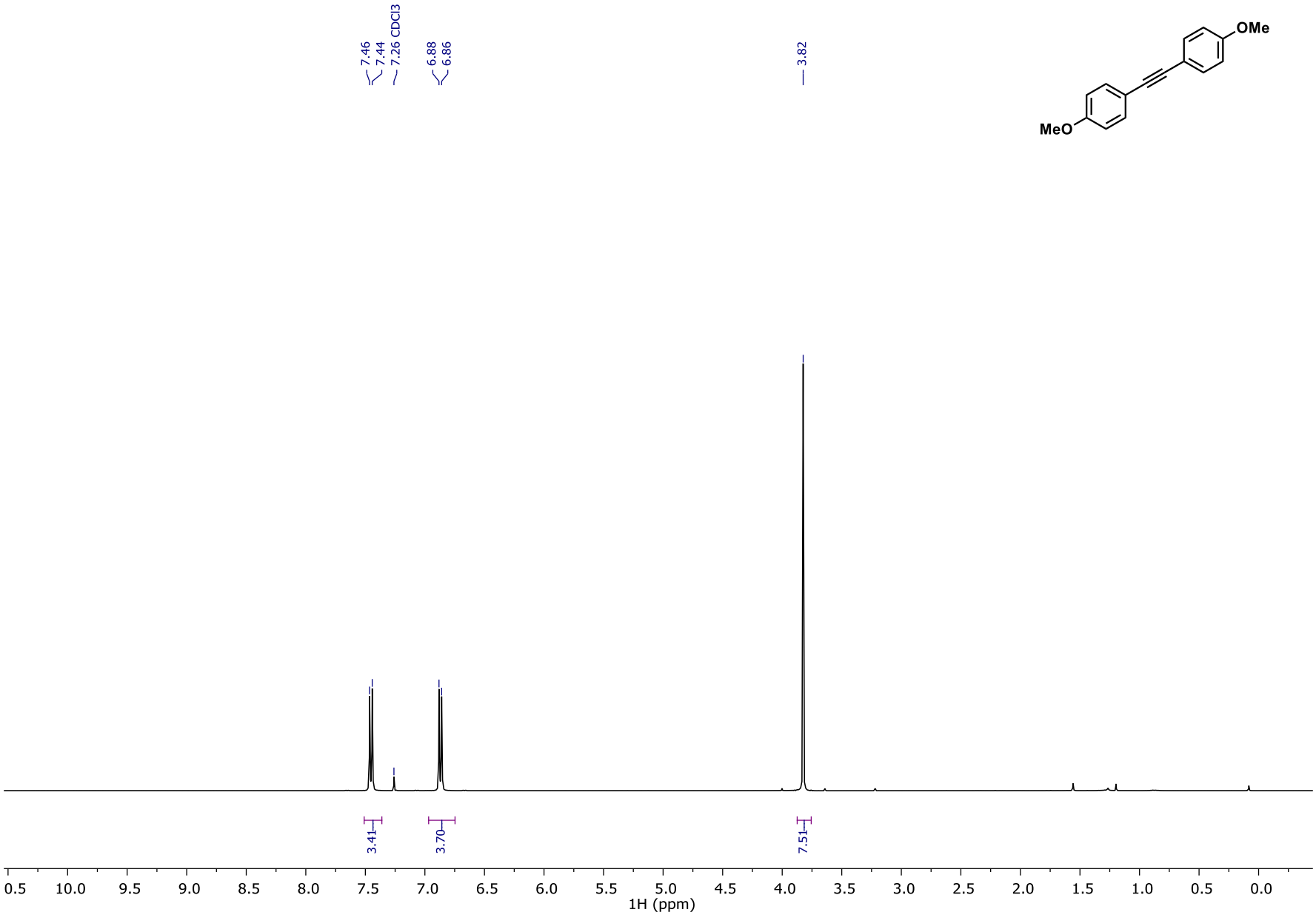
^{1}H ^{13}C HMBC NMR of Di-tert-butyl (1,10-bis(phenylsulfonyl)dec-5-yne-1,10-diyl)dicarbamate (29), 600 MHz, C_6D_6, 25ºC


1H (ppm)
13C (ppm)

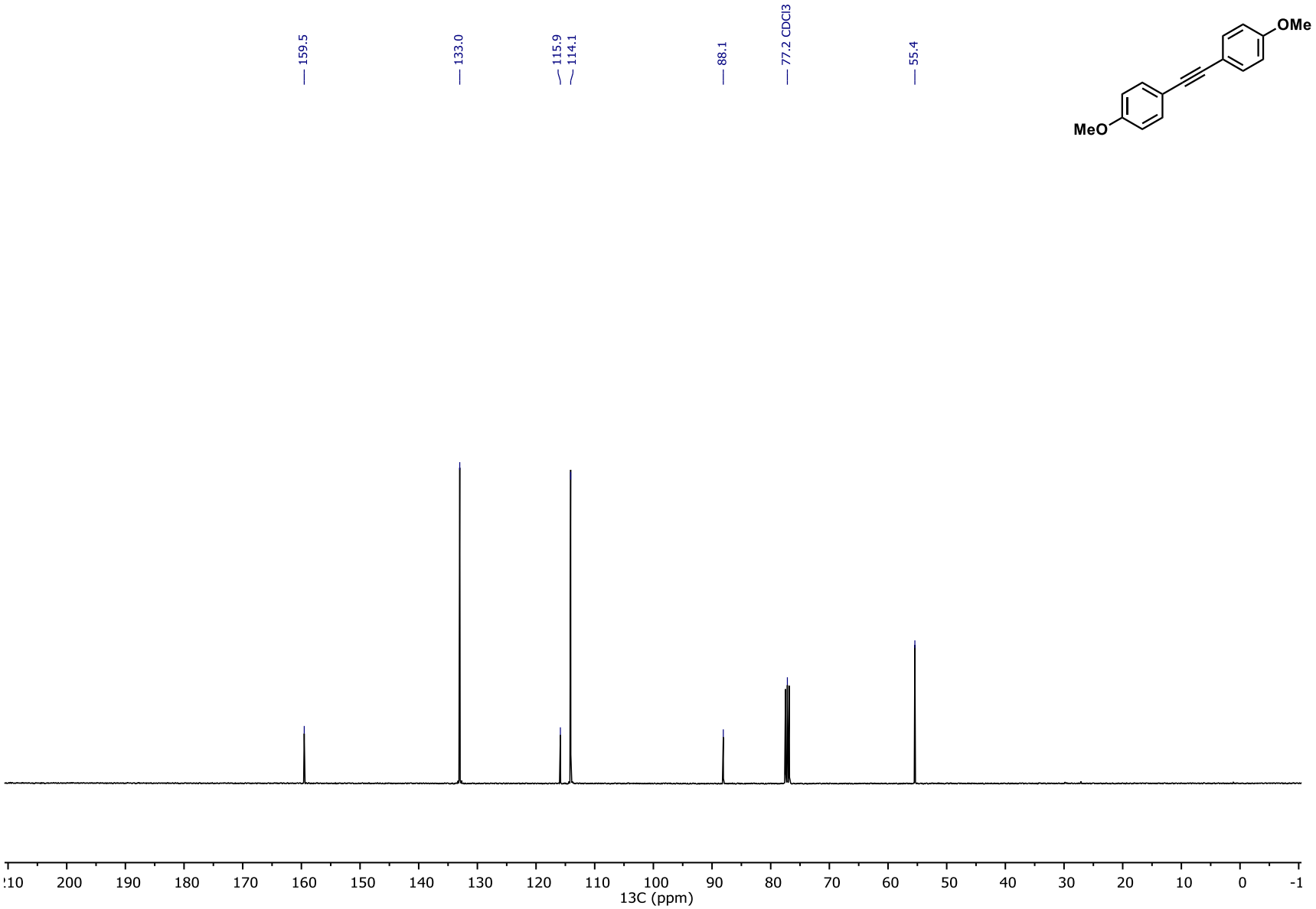
^{1}H ^{1}H NOESY NMR of Di-tert-butyl (1,10-bis(phenylsulfonyl)dec-5-yne-1,10-diyl)dicarbamate (29), 600 MHz, C_6D_6, 25ºC



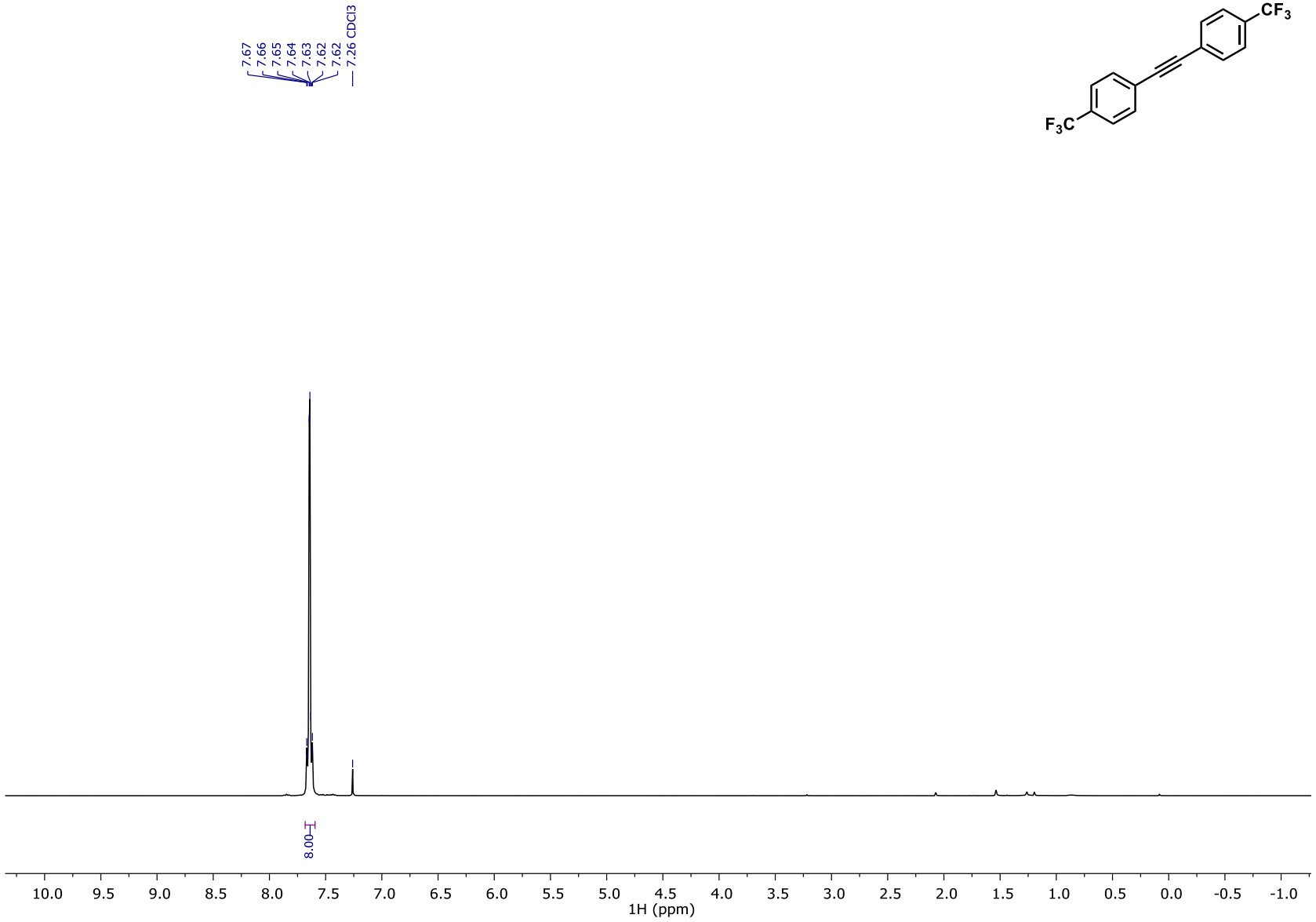
^{1}H NMR of 1,2-Bis(4-methoxyphenyl)ethyne (20a), 400 MHz, $CDCl_3$, 25ºC



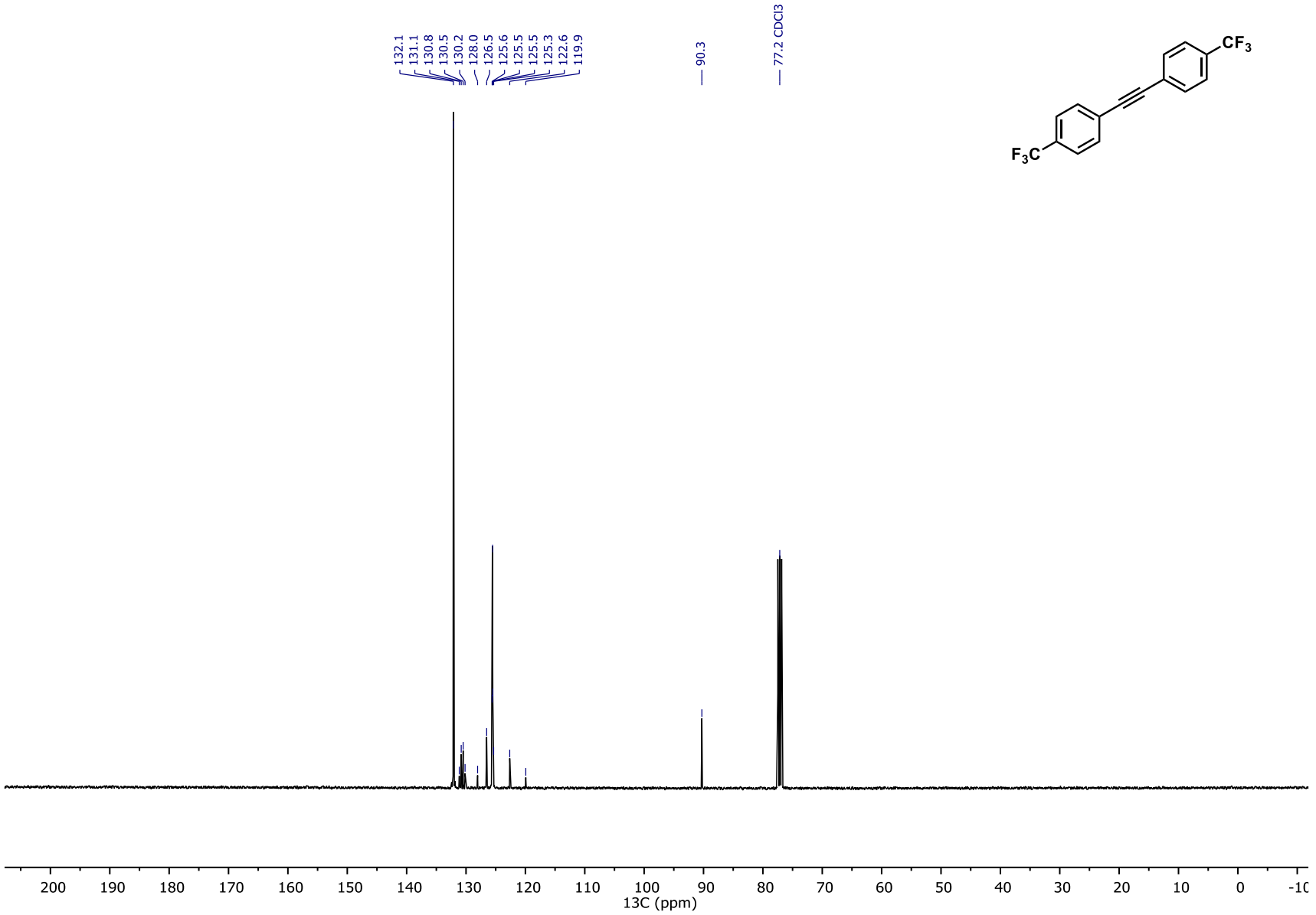
^{13}C NMR of 1,2-Bis(4-methoxyphenyl)ethyne (20a), 101 MHz, $CDCl_3$, 25ºC



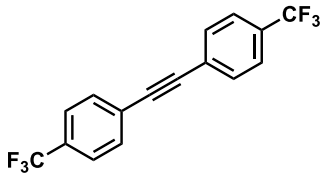
^{1}H NMR of 1,2-Bis(4-(trifluoromethyl)phenyl)ethyne (20c), 400 MHz, $CDCl_3$, 25ºC



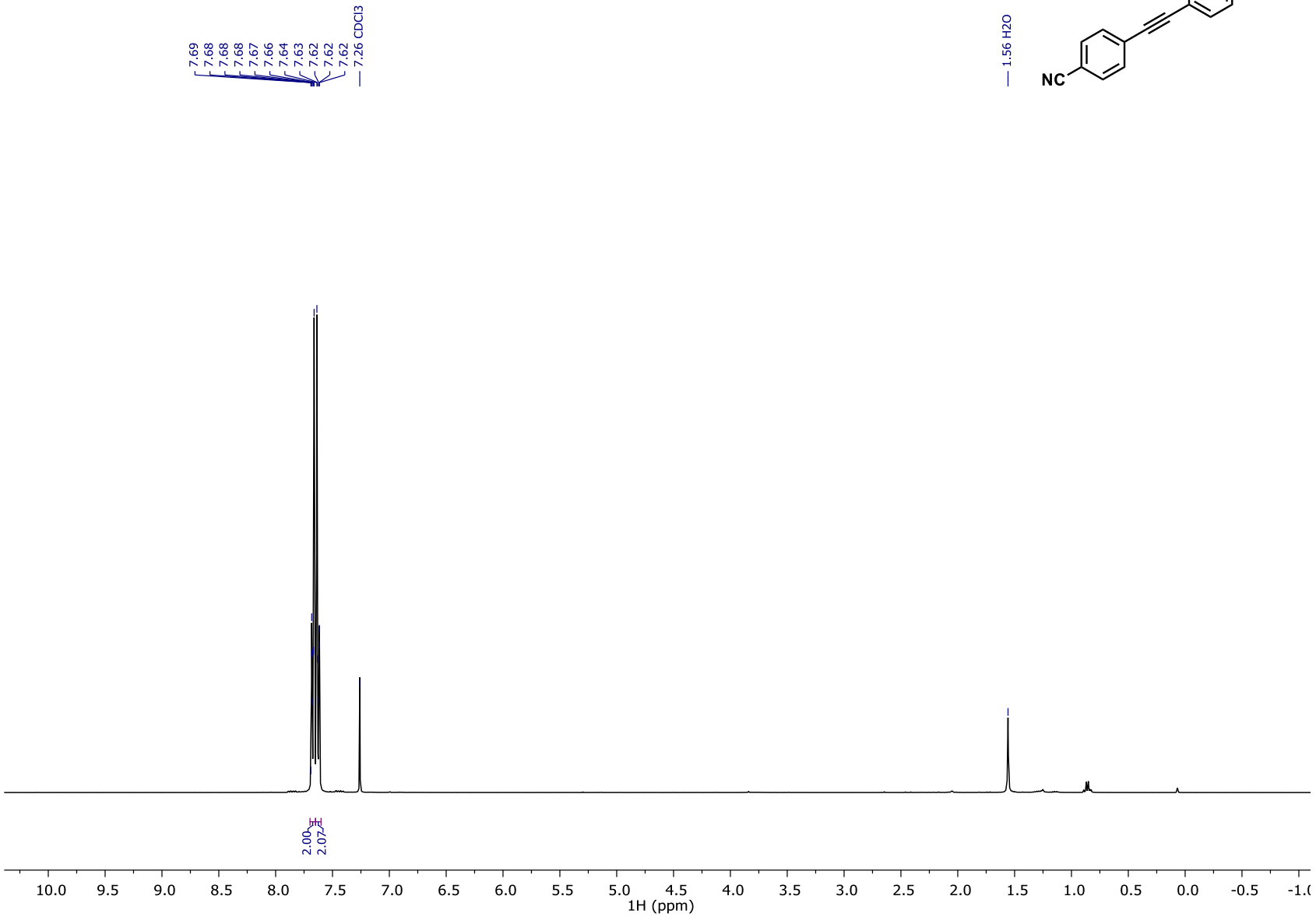
^{13}C NMR of 1,2-Bis(4-(trifluoromethyl)phenyl)ethyne (20c), 101 MHz, $CDCl_3$, 25ºC



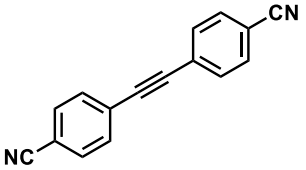
^{19}F NMR of 1,2-Bis(4-(trifluoromethyl)phenyl)ethyne (20c), 282 MHz, $CDCl_3$, 25ºC



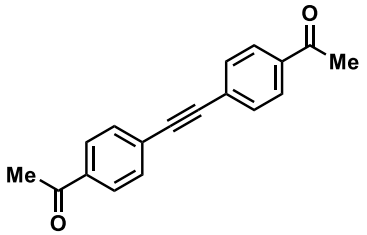
^{1}H NMR of 4,4'-(Ethyne-1,2-diyl)dibenzonitrile (20d), 400 MHz, $CDCl_3$, 25ºC



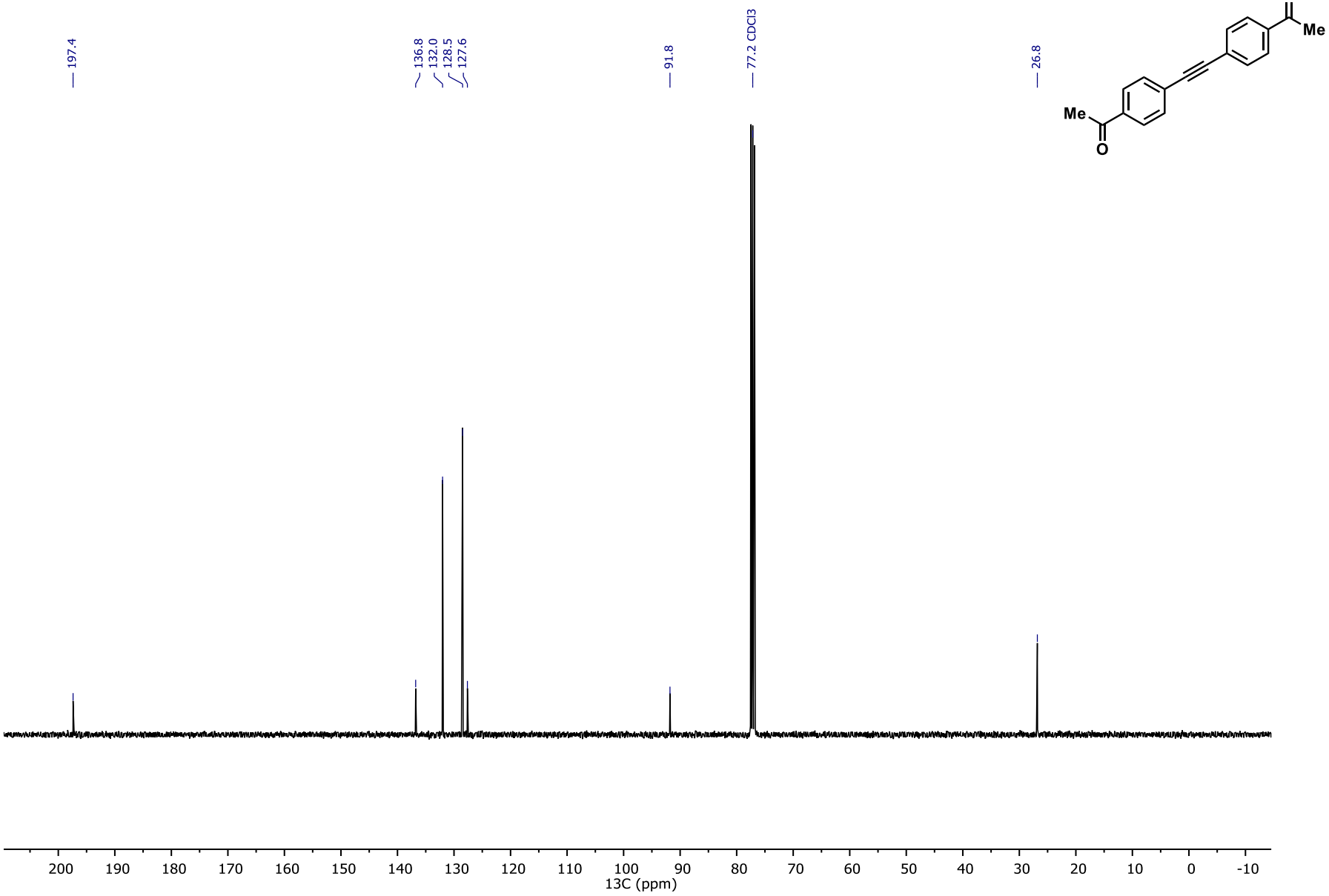
^{13}C NMR of 4,4'-(Ethyne-1,2-diyl)dibenzonitrile (20d), 101 MHz, $CDCl_3$, 25ºC



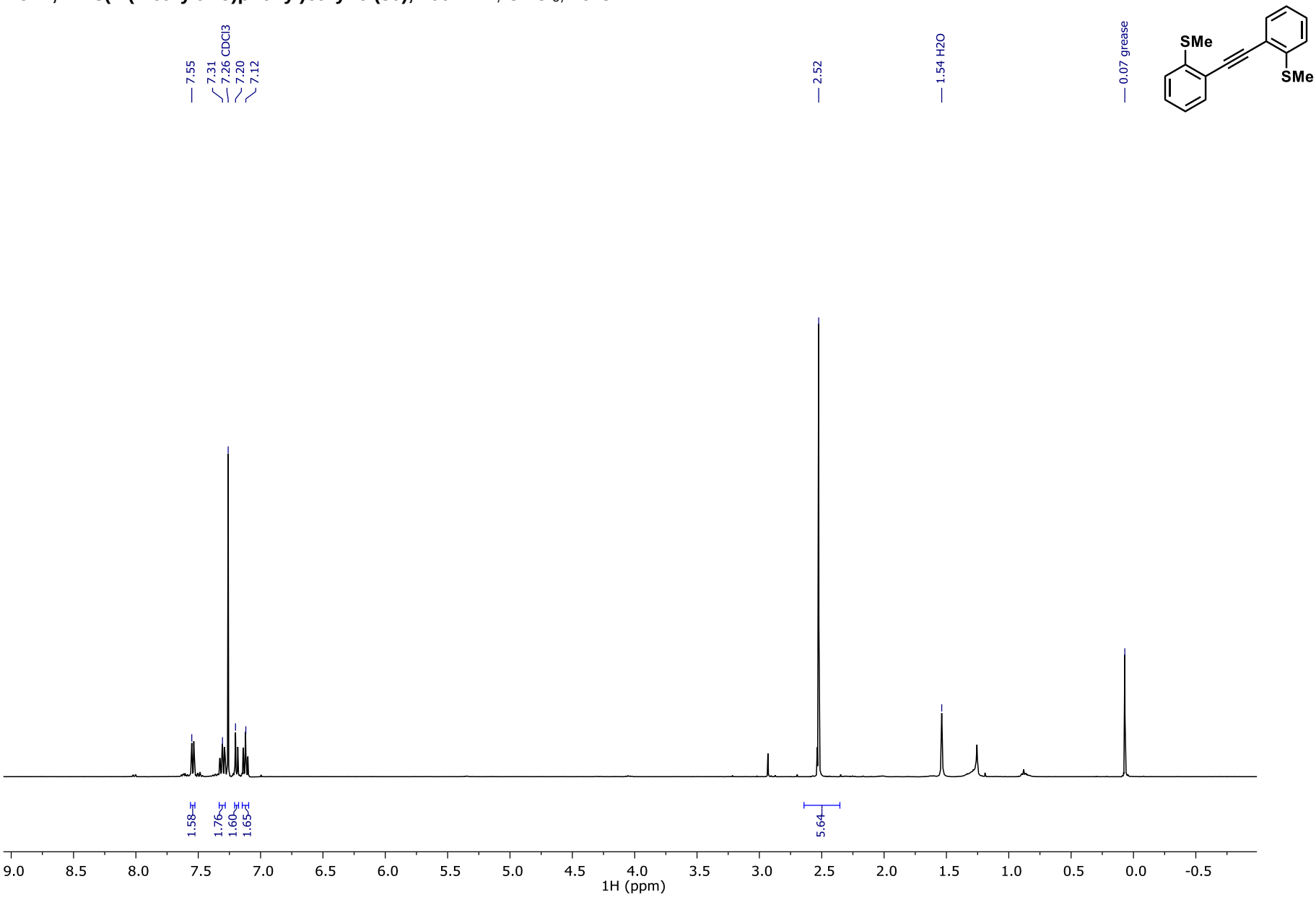
^{1}H NMR of 1,1'-(Ethyne-1,2-diylbis(4,1-phenylene))bis(ethan-1-one) (20e), 400 MHz, $CDCl_3$, 25ºC



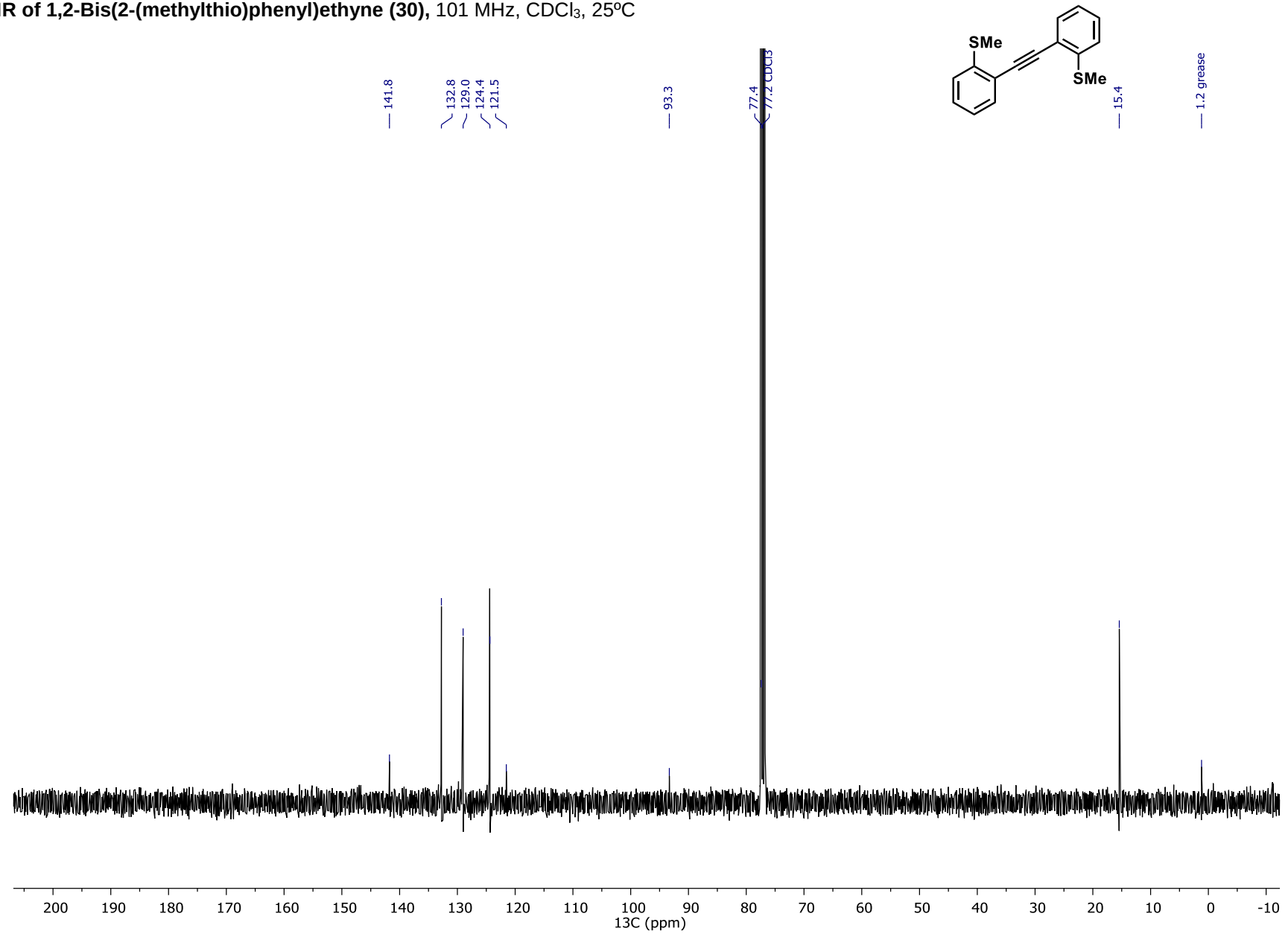
^{13}C NMR of 1,1'-(Ethyne-1,2-diylbis(4,1-phenylene))bis(ethan-1-one) (20e), 101 MHz, $CDCl_3$, 25ºC



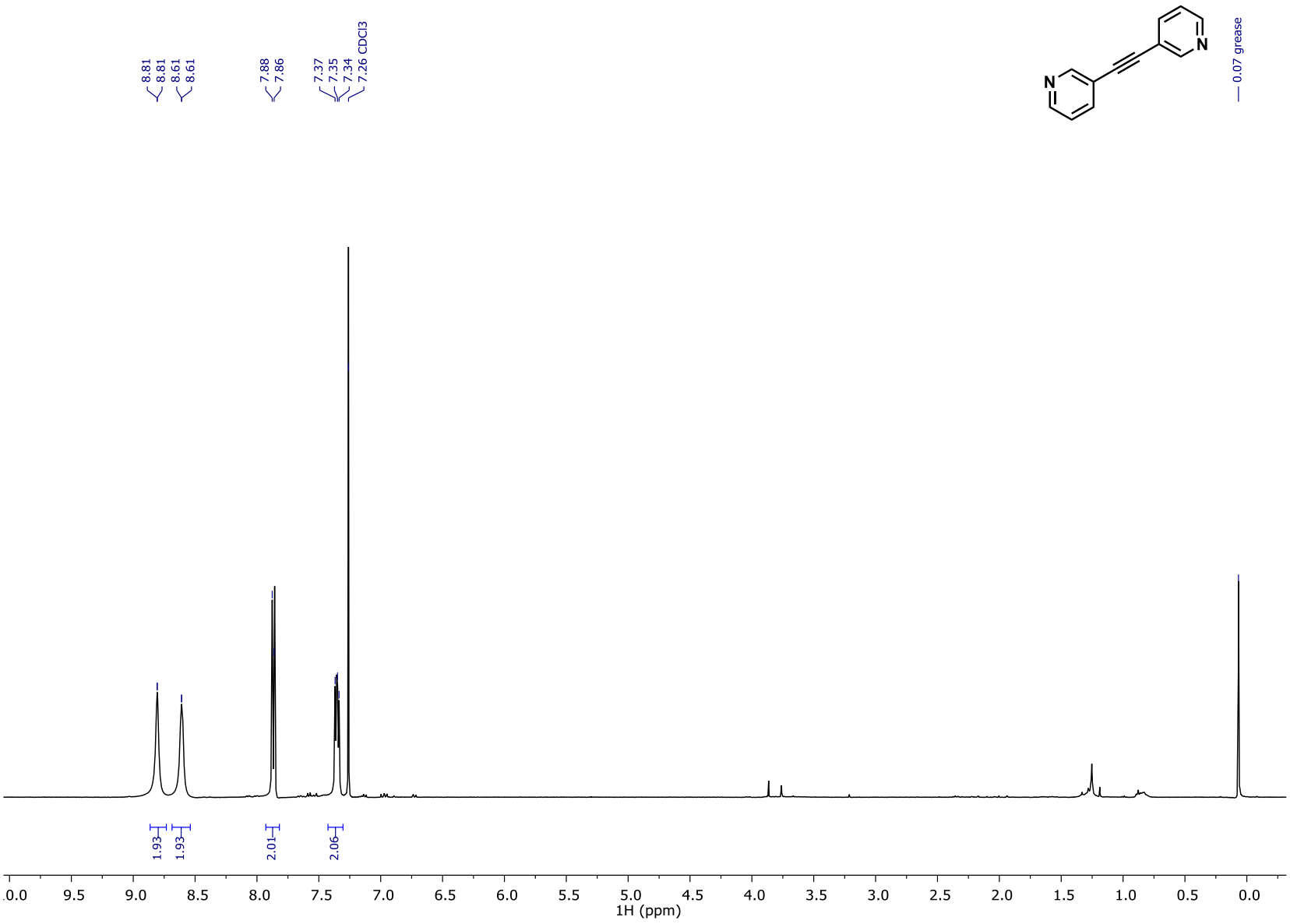
^{1}H NMR of 1,2-Bis(2-(methylthio)phenyl)ethyne (30), 400 MHz, $CDCl_3$, 25ºC



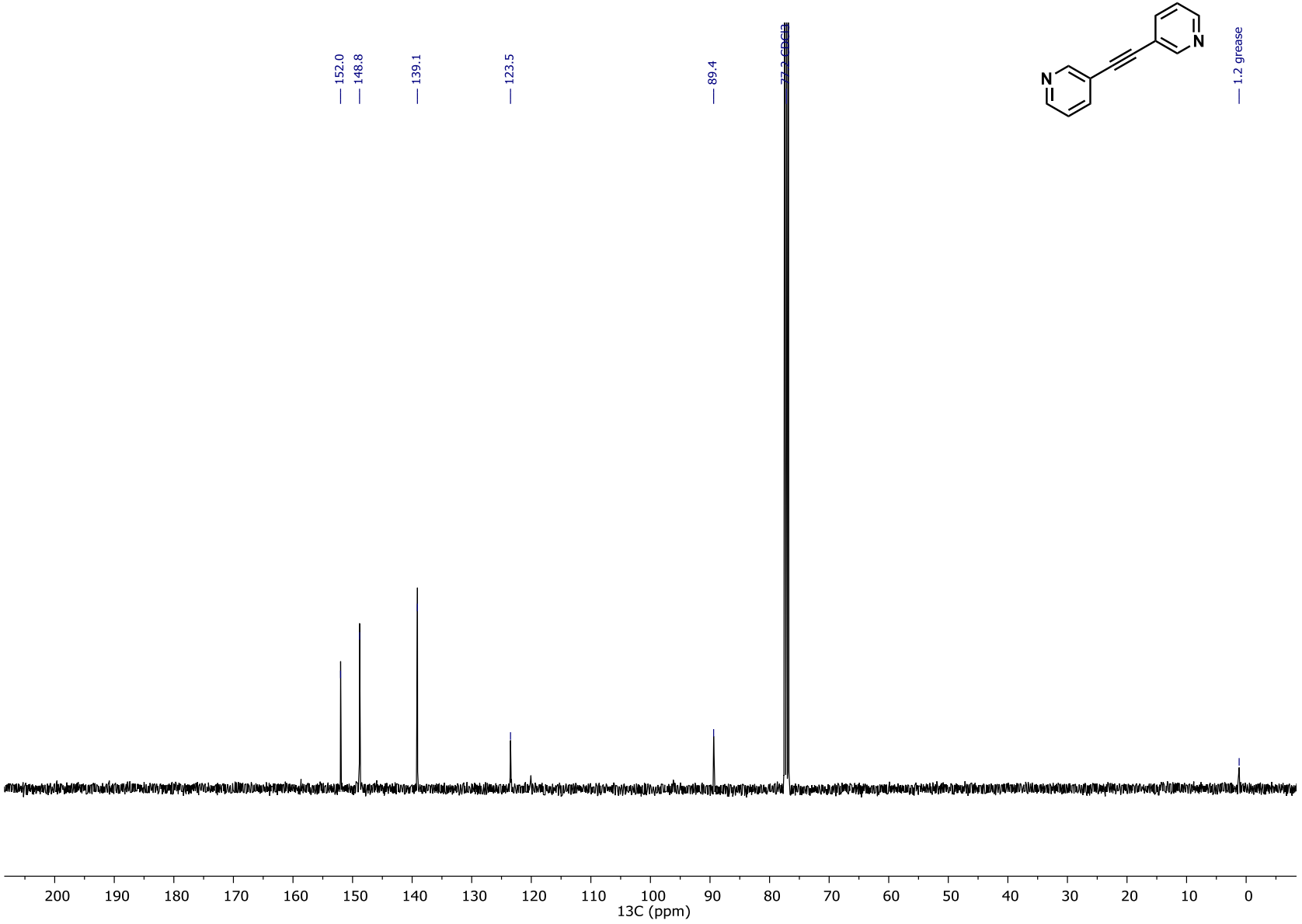
^{13}C NMR of 1,2-Bis(2-(methylthio)phenyl)ethyne (30), 101 MHz, $CDCl_3$, 25ºC



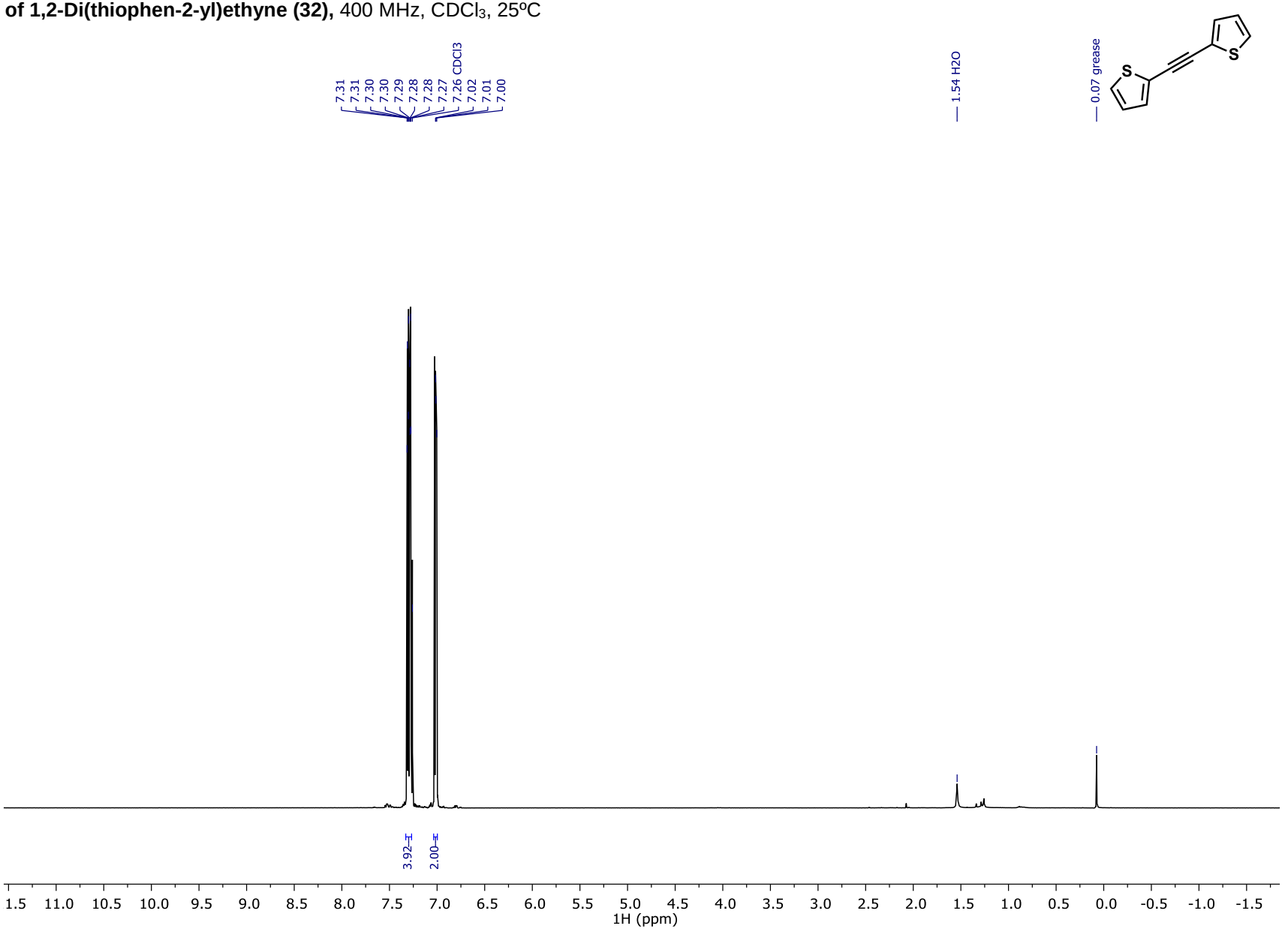
^{1}H NMR of 1,2-Di(pyridin-3-yl)ethyne (31), 400 MHz, $CDCl_3$, 25ºC



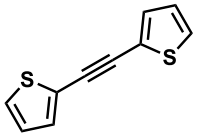
^{13}C NMR of 1,2-Di(pyridin-3-yl)ethyne (31), 101 MHz, $CDCl_3$, 25ºC



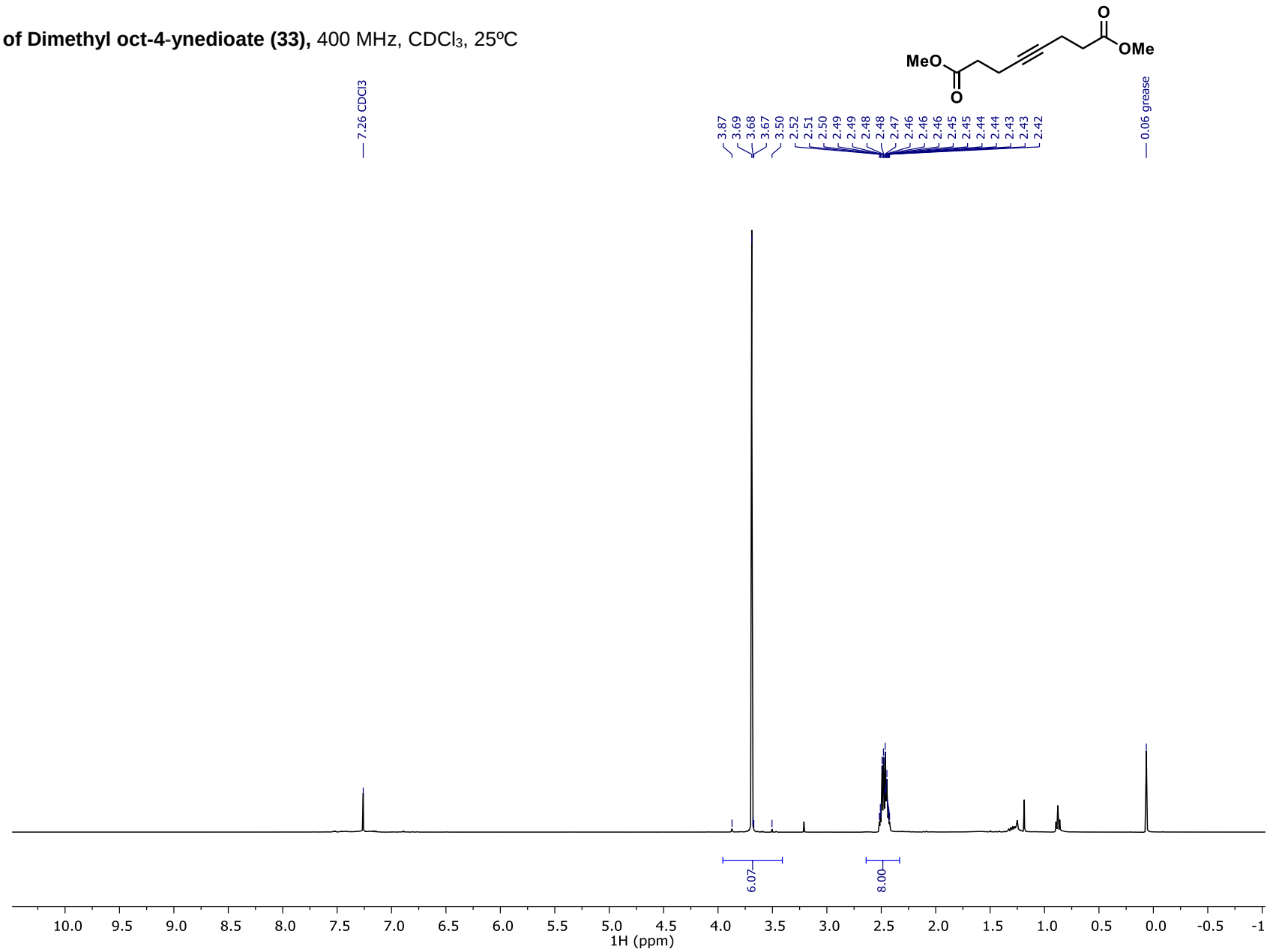
^{1}H NMR of 1,2-Di(thiophen-2-yl)ethyne (32), 400 MHz, $CDCl_3$, 25ºC



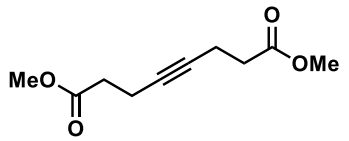
^{13}C NMR of 1,2-Di(thiophen-2-yl)ethyne (32), 101 MHz, $CDCl_3$, 25ºC

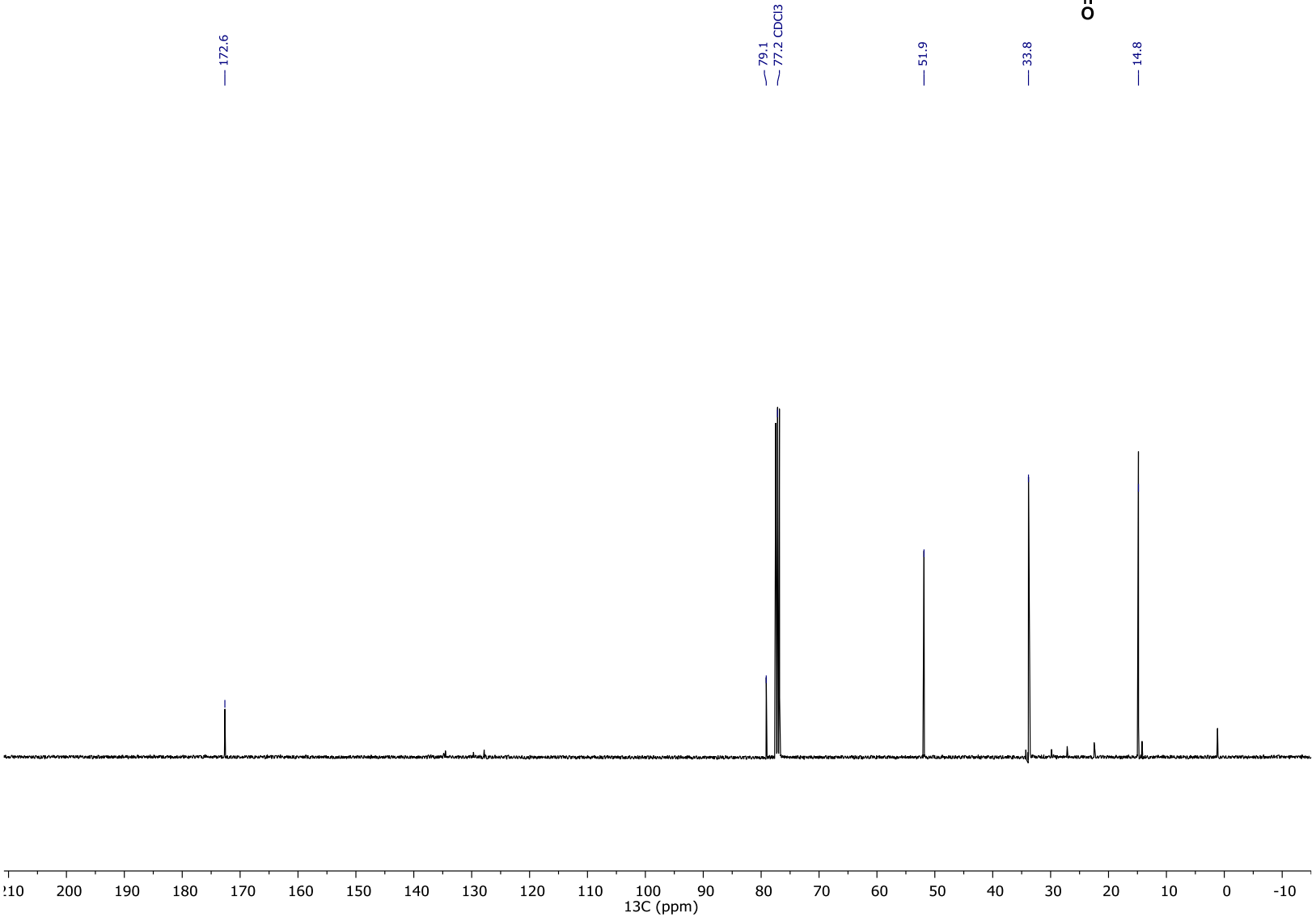


^{1}H NMR of Dimethyl oct-4-ynedioate (33), 400 MHz, $CDCl_3$, 25ºC

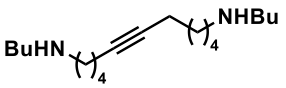


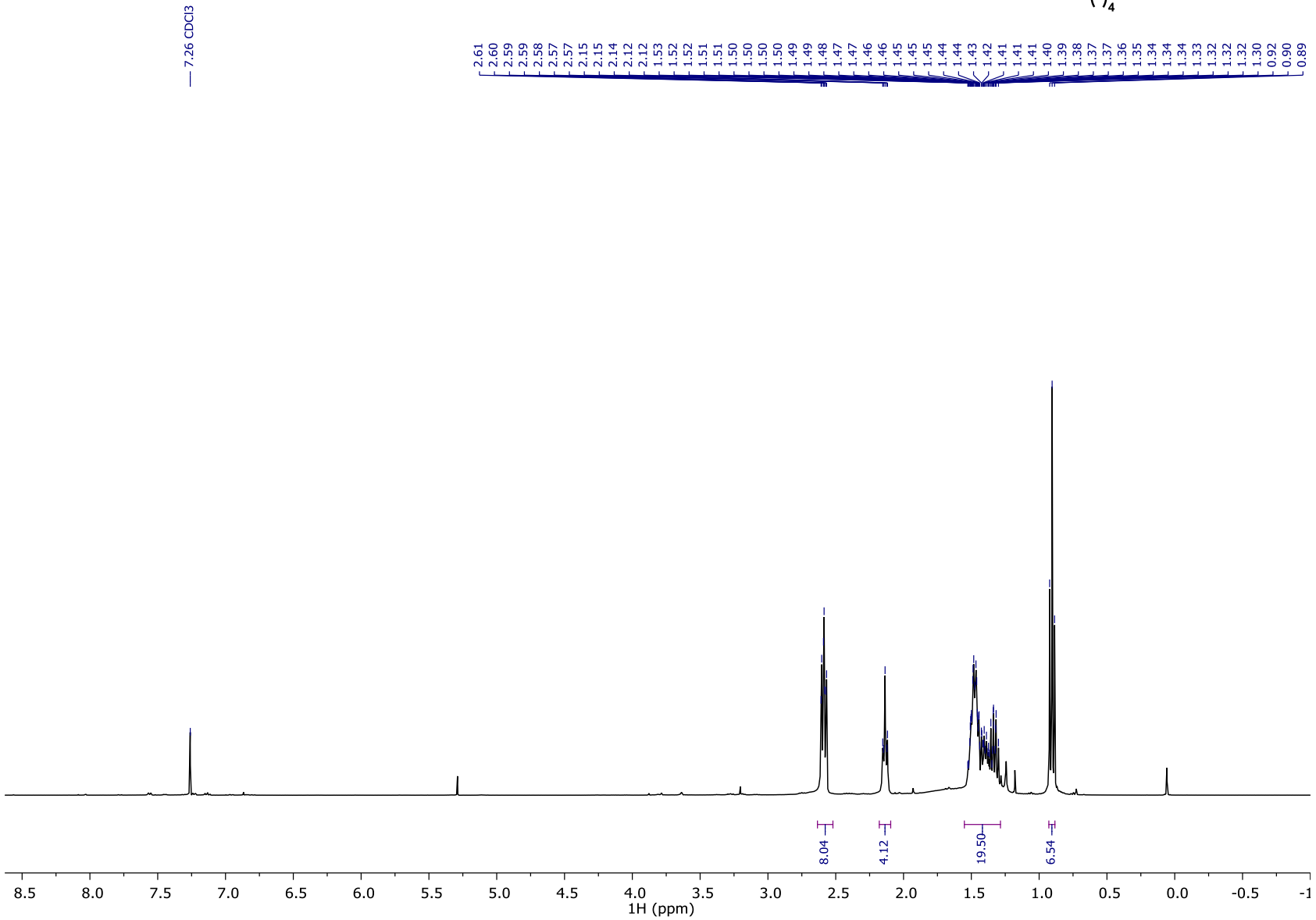
^{13}C NMR of Dimethyl oct-4-ynedioate (33), 101 MHz, $CDCl_3$, 25ºC



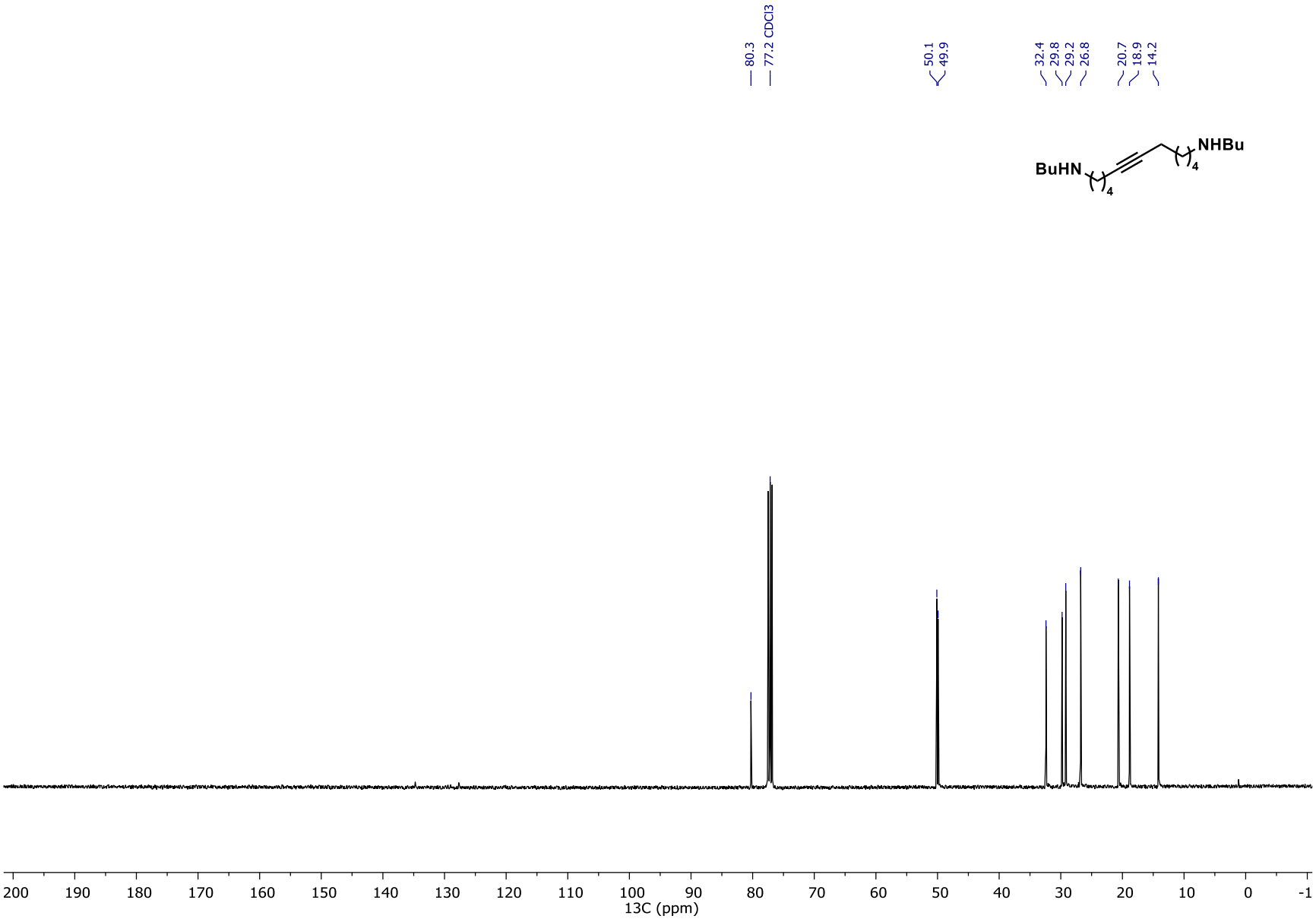


^{1}H NMR of *N*^{1},*N*^{12}-Dibutyldodec-6-yne-1,12-diamine (34), 400 MHz, $CDCl_3$, 25ºC

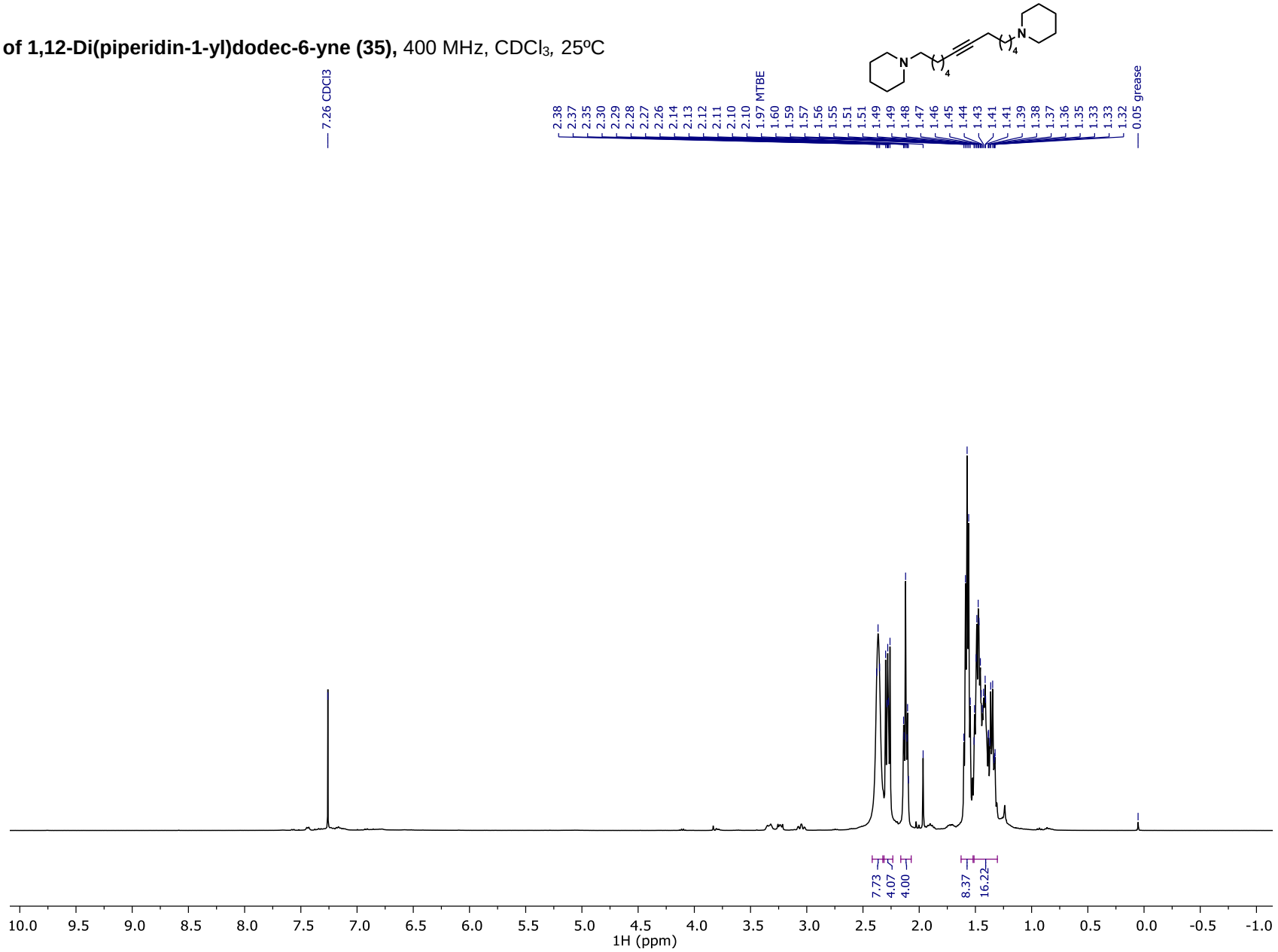




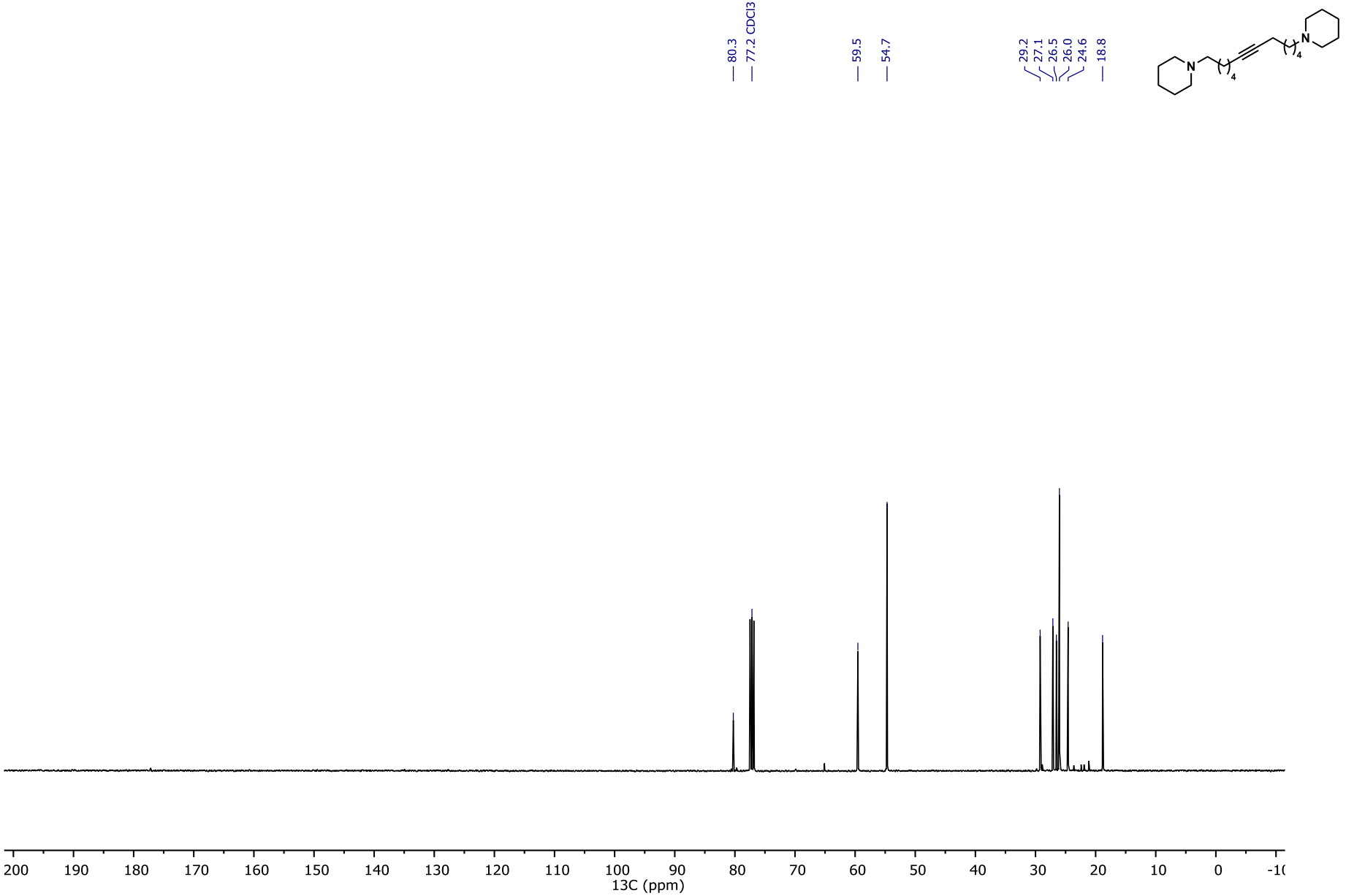
^{13}C NMR of *N*^{1},*N*^{12}-dibutyldodec-6-yne-1,12-diamine (34), 101 MHz, $CDCl_3$, 25ºC



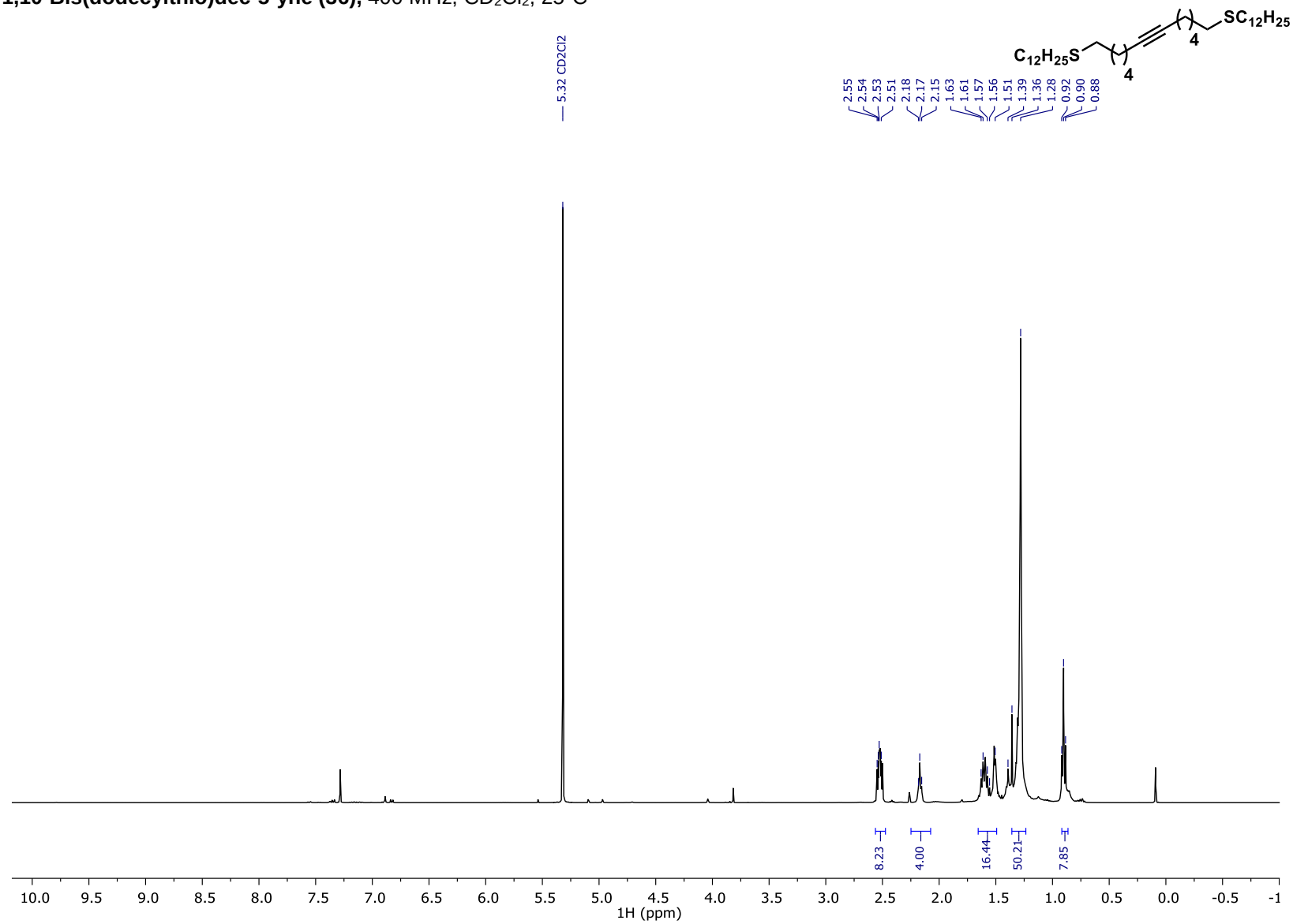
^{1}H NMR of 1,12-Di(piperidin-1-yl)dodec-6-yne (35), 400 MHz, $CDCl_3$, 25ºC



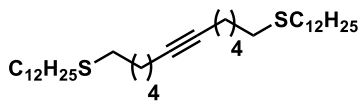
^{13}C NMR of 1,12-Di(piperidin-1-yl)dodec-6-yne (35), 101 MHz, $CDCl_3$, 25ºC

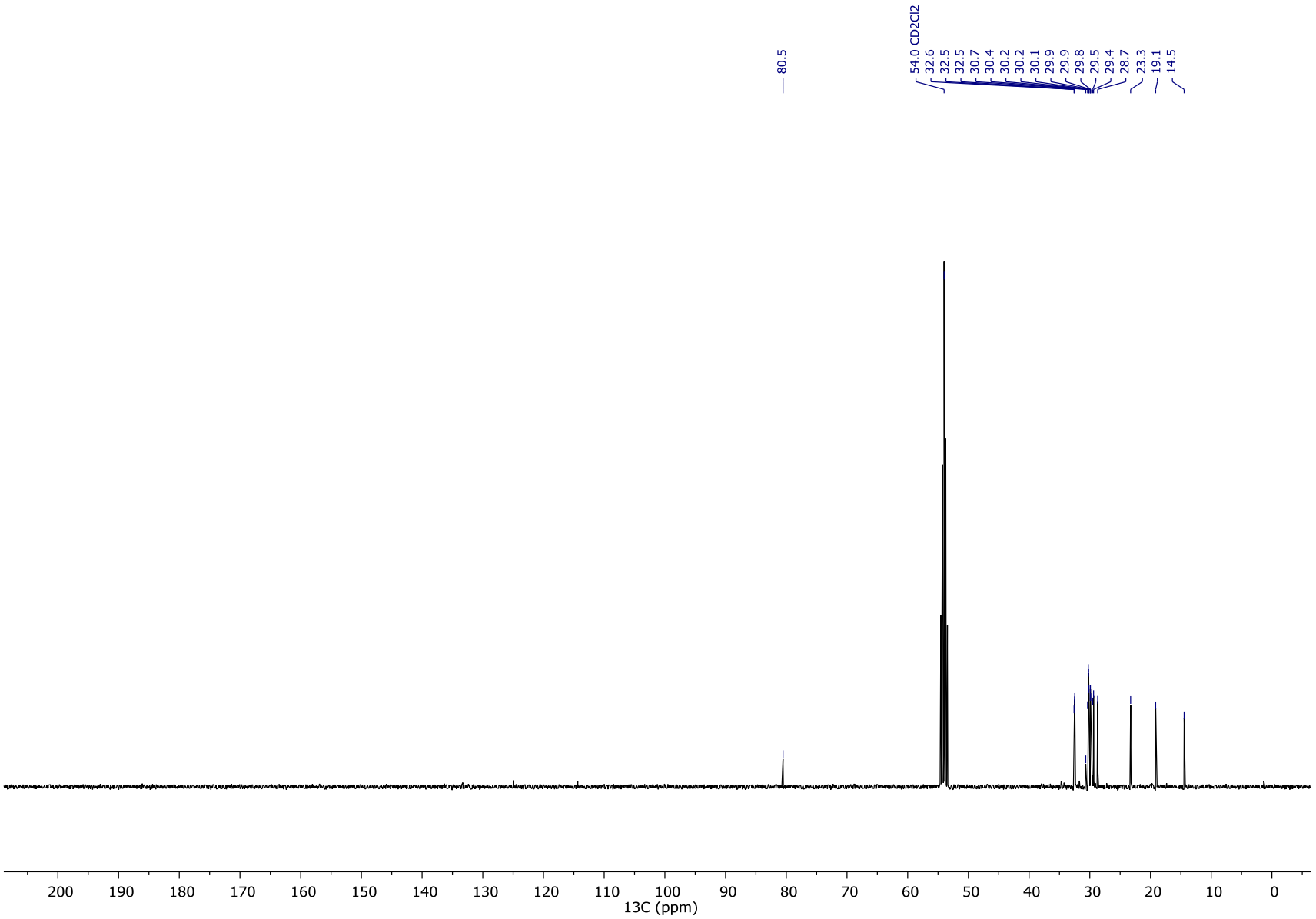


^{1}H NMR of 1,10-Bis(dodecylthio)dec-5-yne (36), 400 MHz, CD_2Cl_2, 25ºC

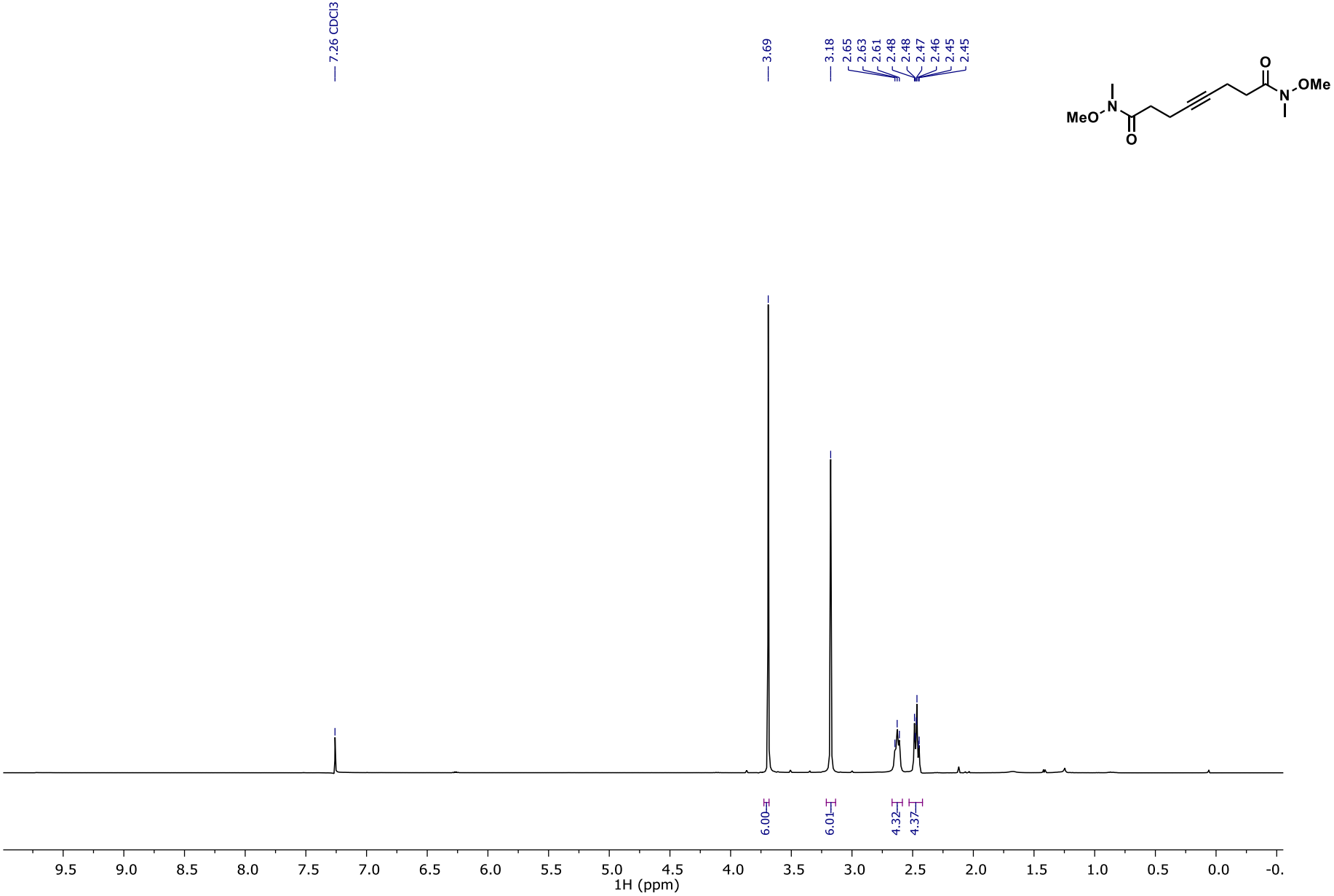


^{13}C NMR of 1,10-Bis(dodecylthio)dec-5-yne (36), 101 MHz, CD_2Cl_2, 25ºC

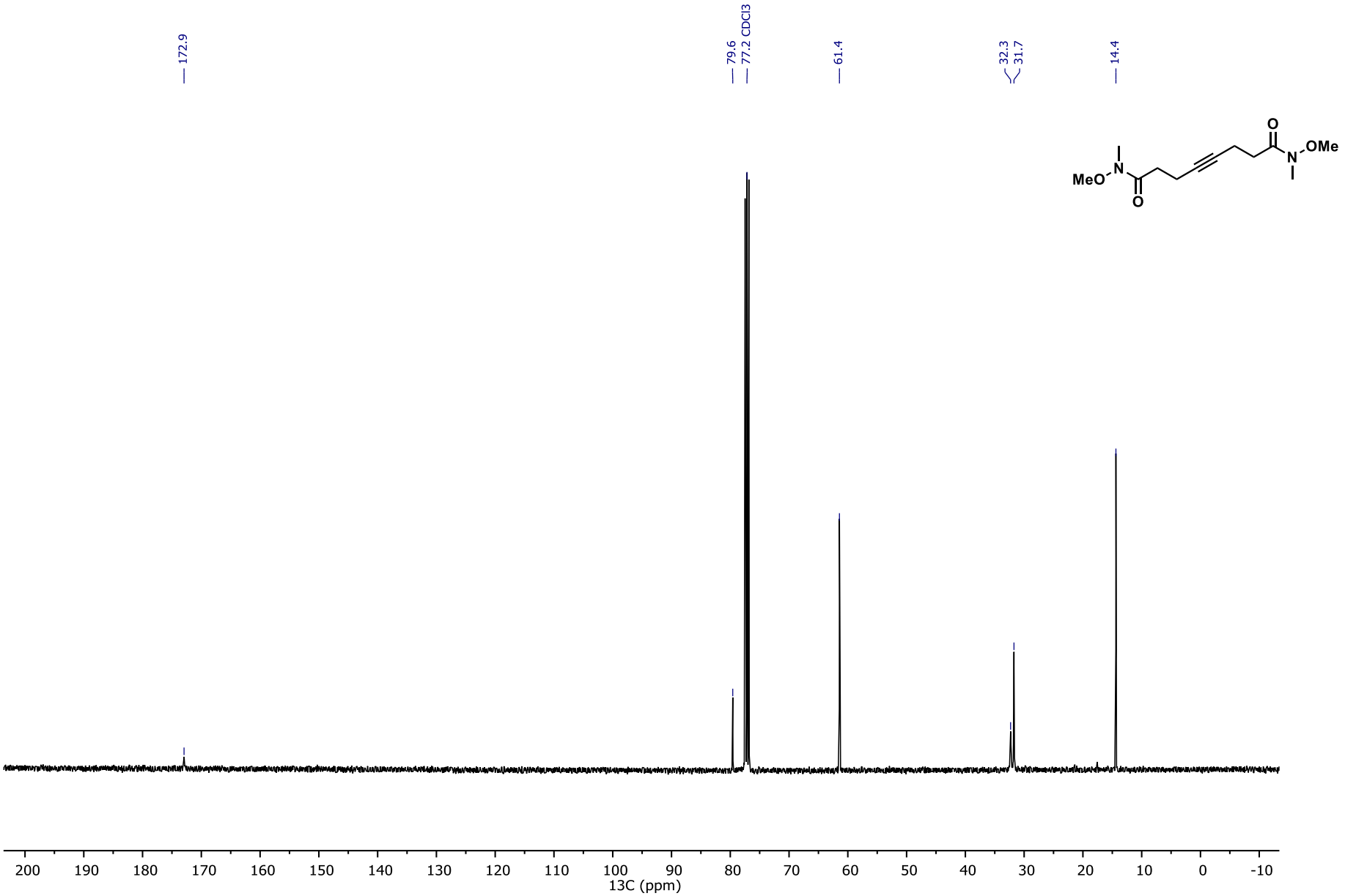




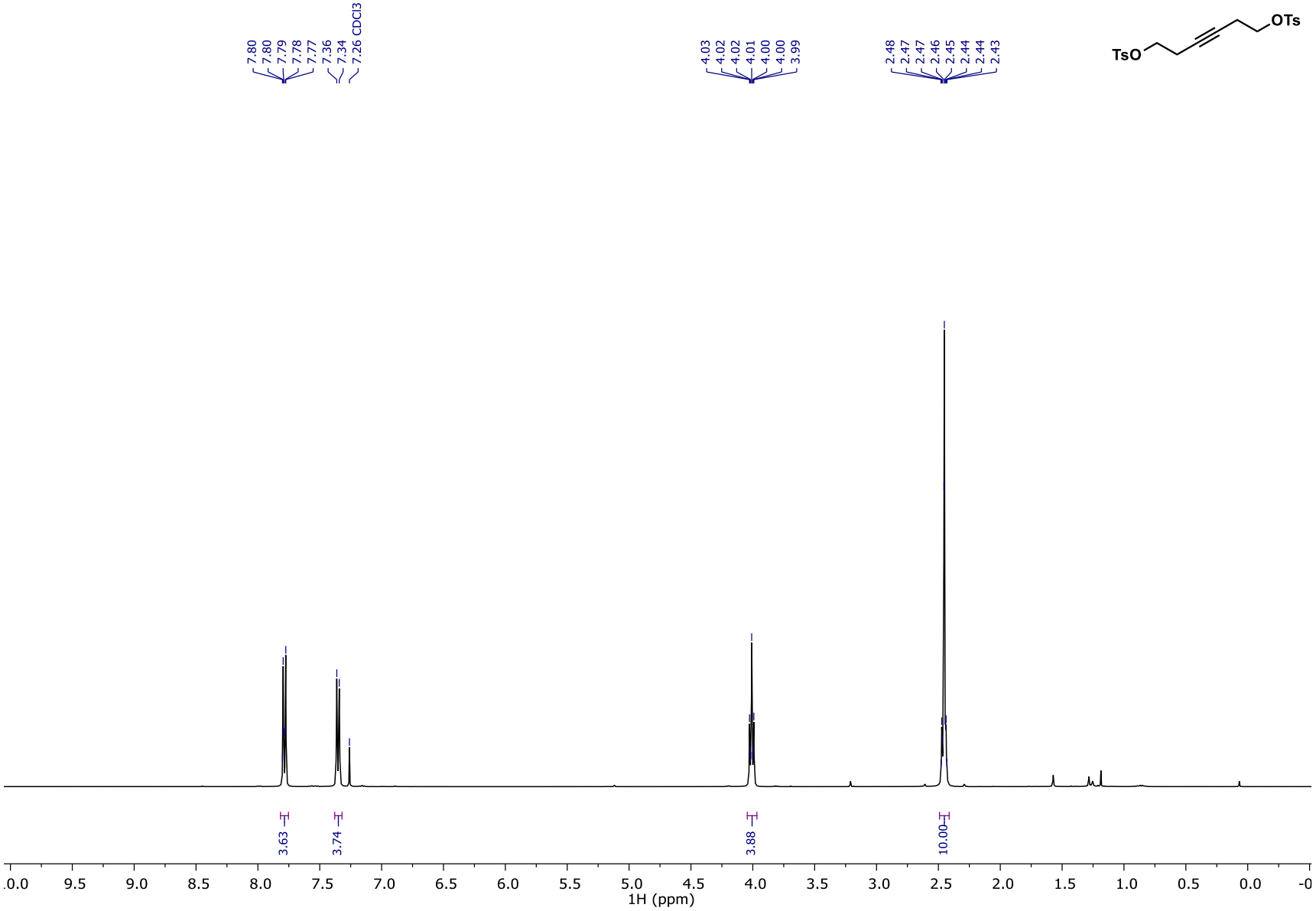
^{1}H NMR of *N*^{1},*N*^{8}-dimethoxy-*N*^{1},*N*^{8}-dimethyloct-4-ynediamide (37), 400 MHz, $CDCl_3$, 25ºC



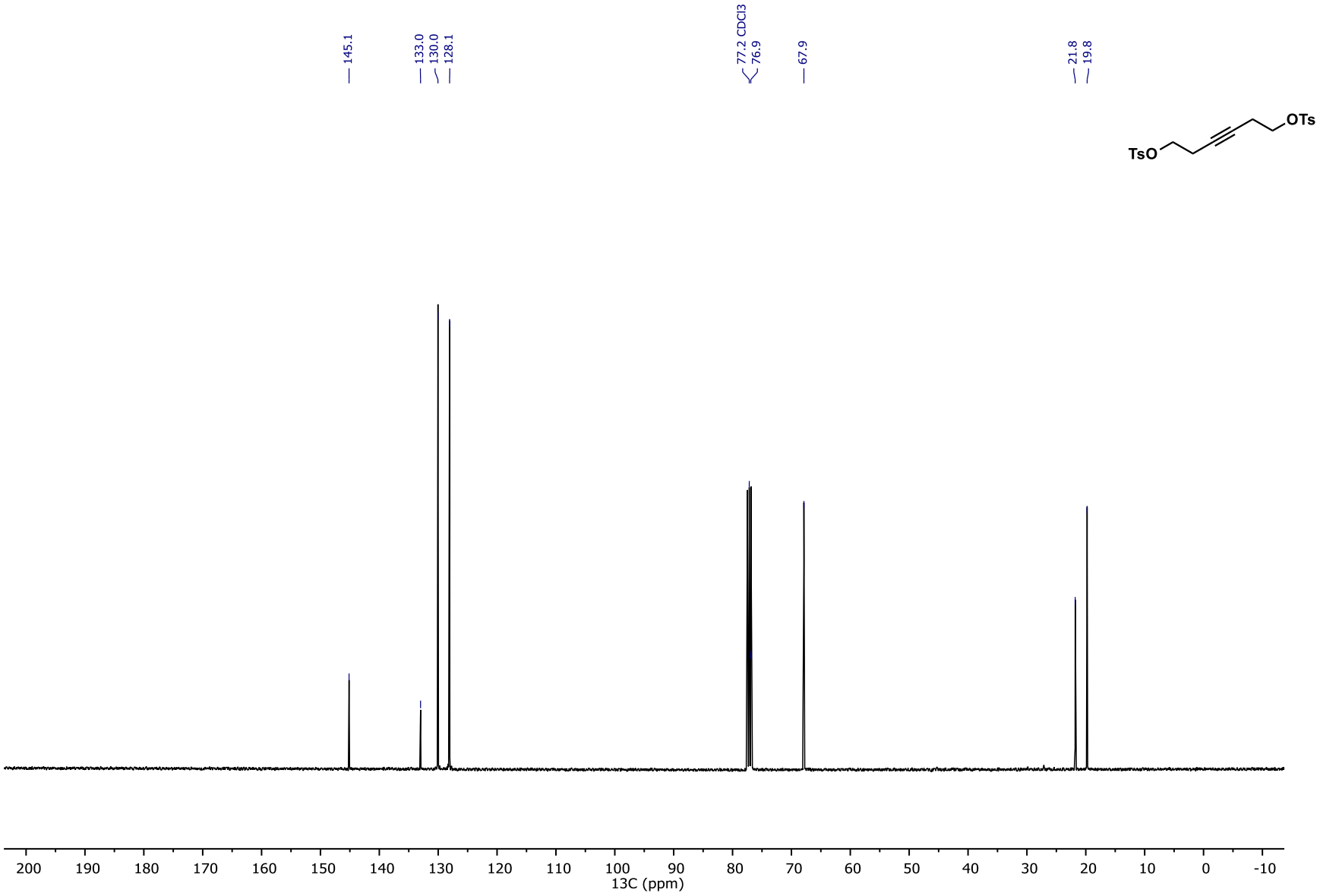
^{13}C NMR of *N*^{1},*N*^{8}-dimethoxy-*N*^{1},*N*^{8}-dimethyloct-4-ynediamide (37), 101 MHz, $CDCl_3$, 25ºC



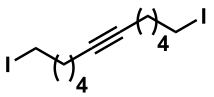
^{1}H NMR of Hex-3-yne-1,6-diyl bis(4-methylbenzenesulfonate) (38), 400 MHz, $CDCl_3$, 25ºC

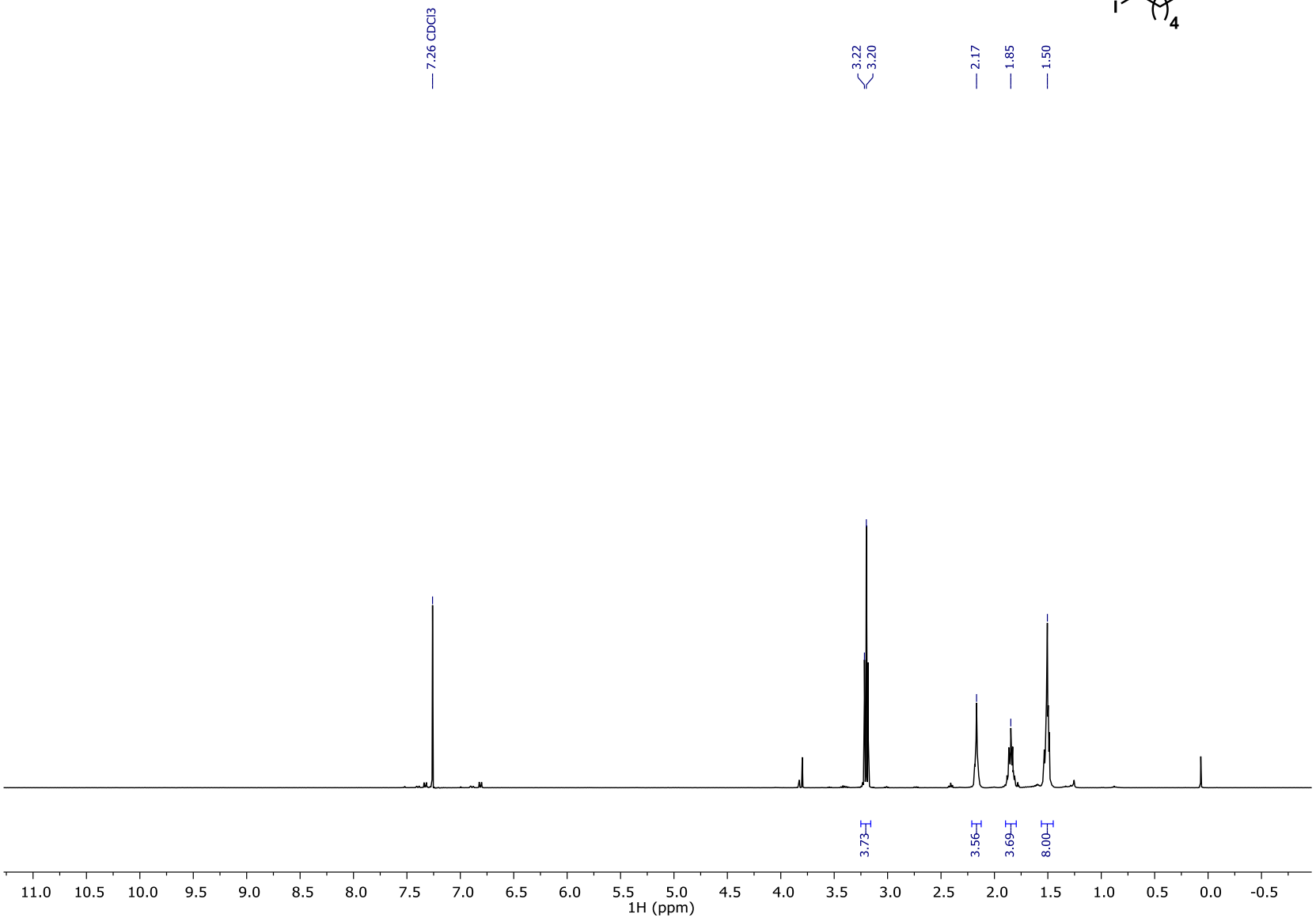


^{13}C NMR of Hex-3-yne-1,6-diyl bis(4-methylbenzenesulfonate) (38), 101 MHz, $CDCl_3$, 25ºC

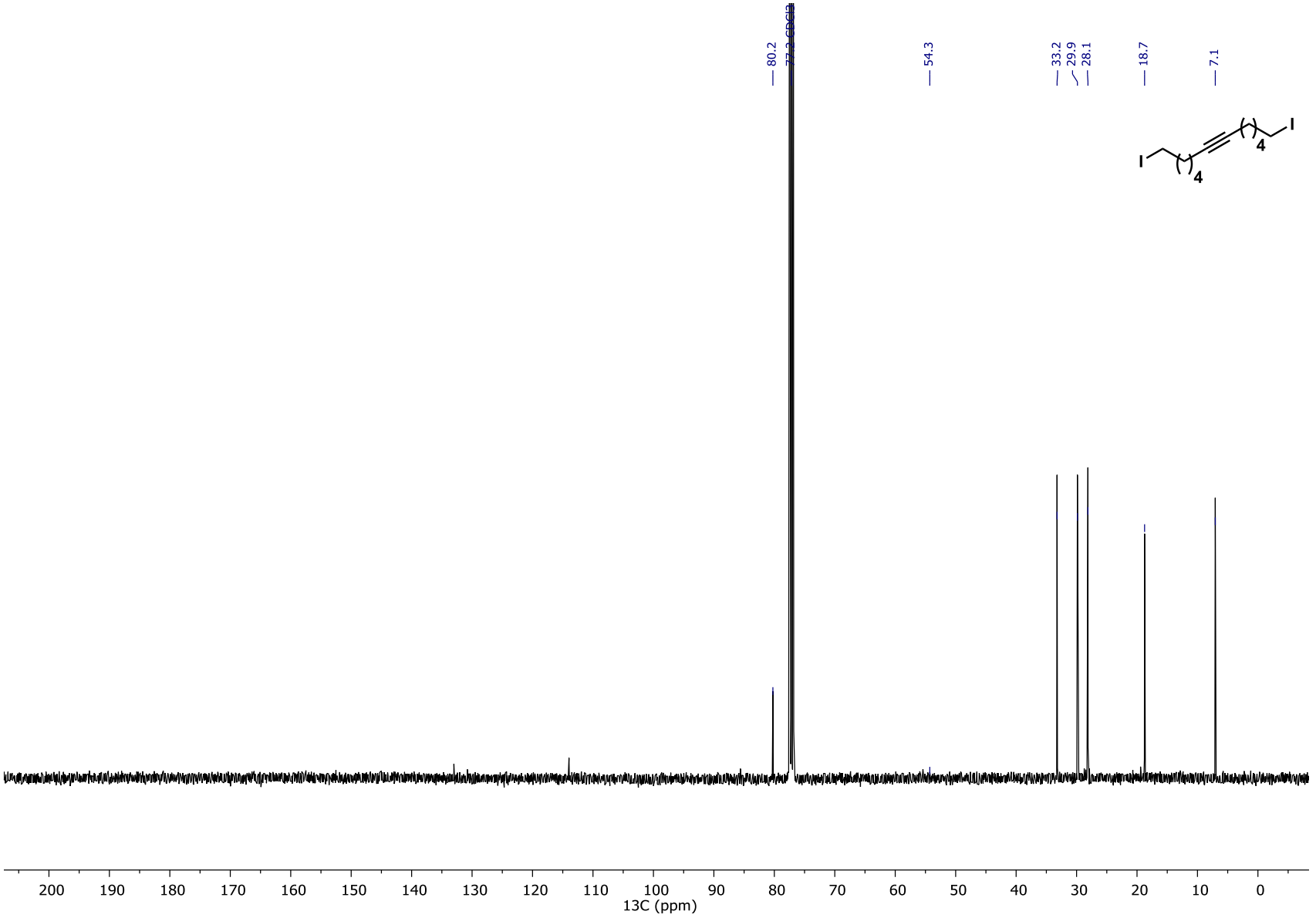


^{1}H NMR of 1,12-Diiodododec-6-yne (39), 400 MHz, $CDCl_3$, 25ºC

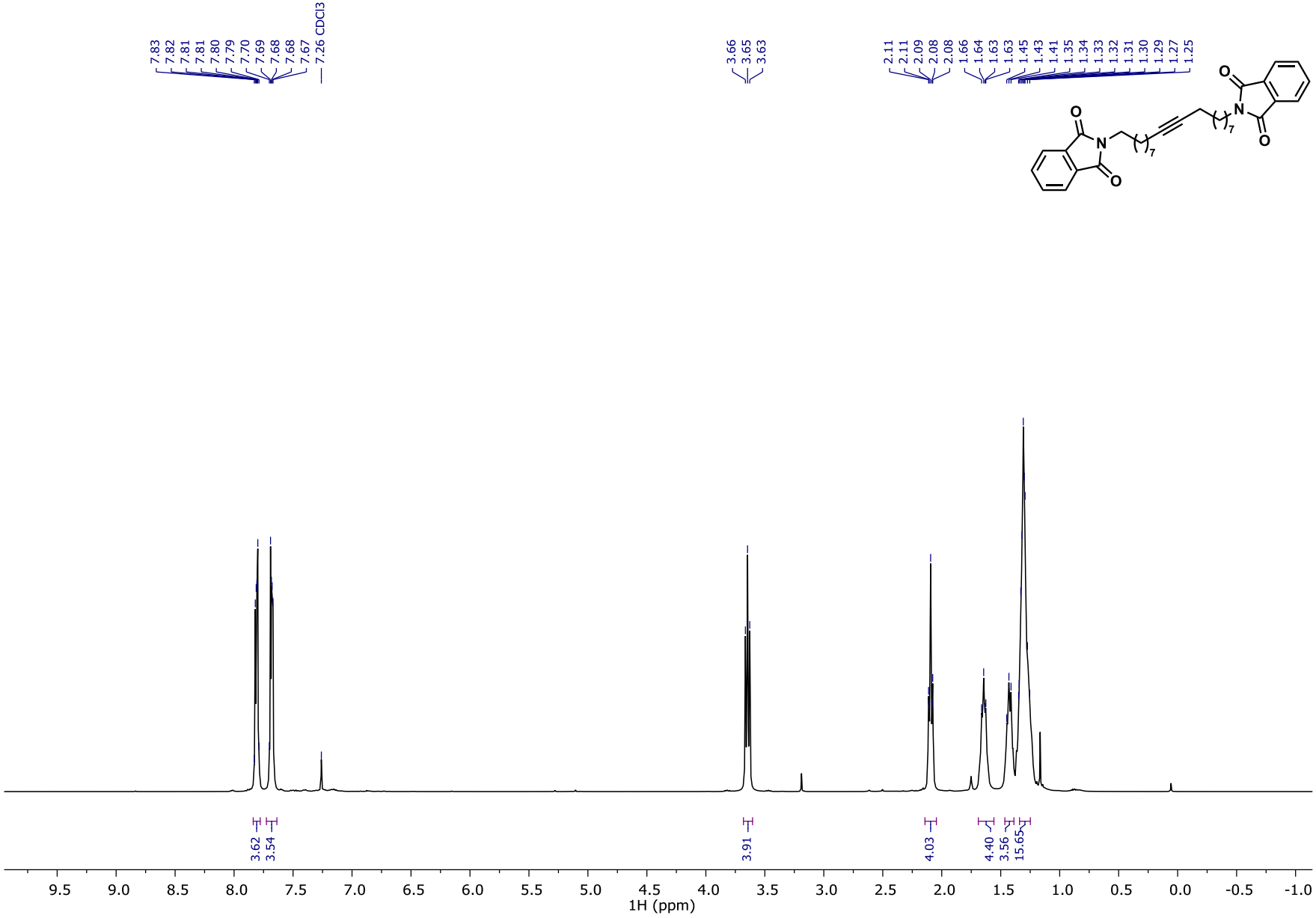




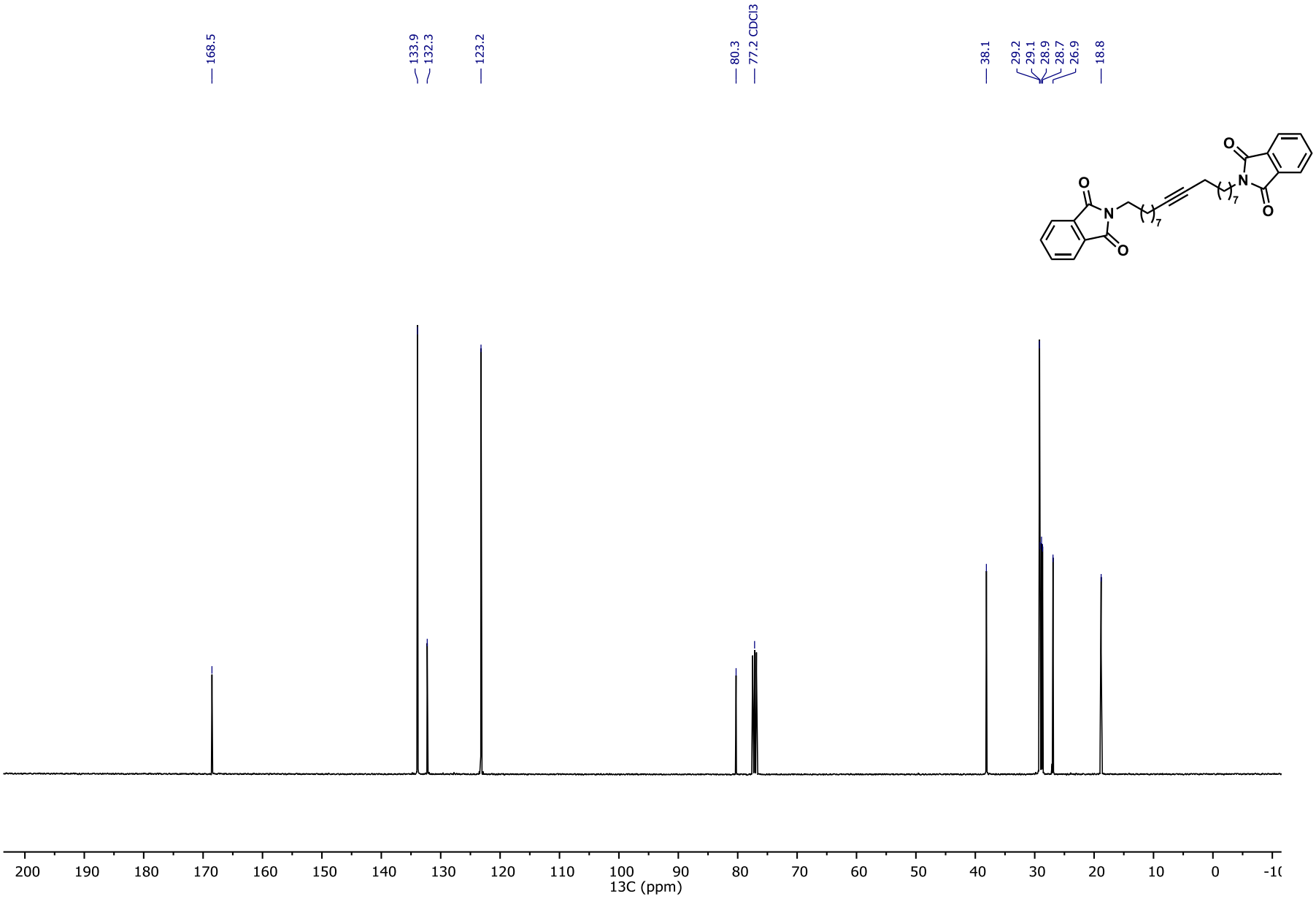
^{13}C NMR of 1,12-Diiodododec-6-yne (39), 101 MHz**,** $CDCl_3$, 25ºC



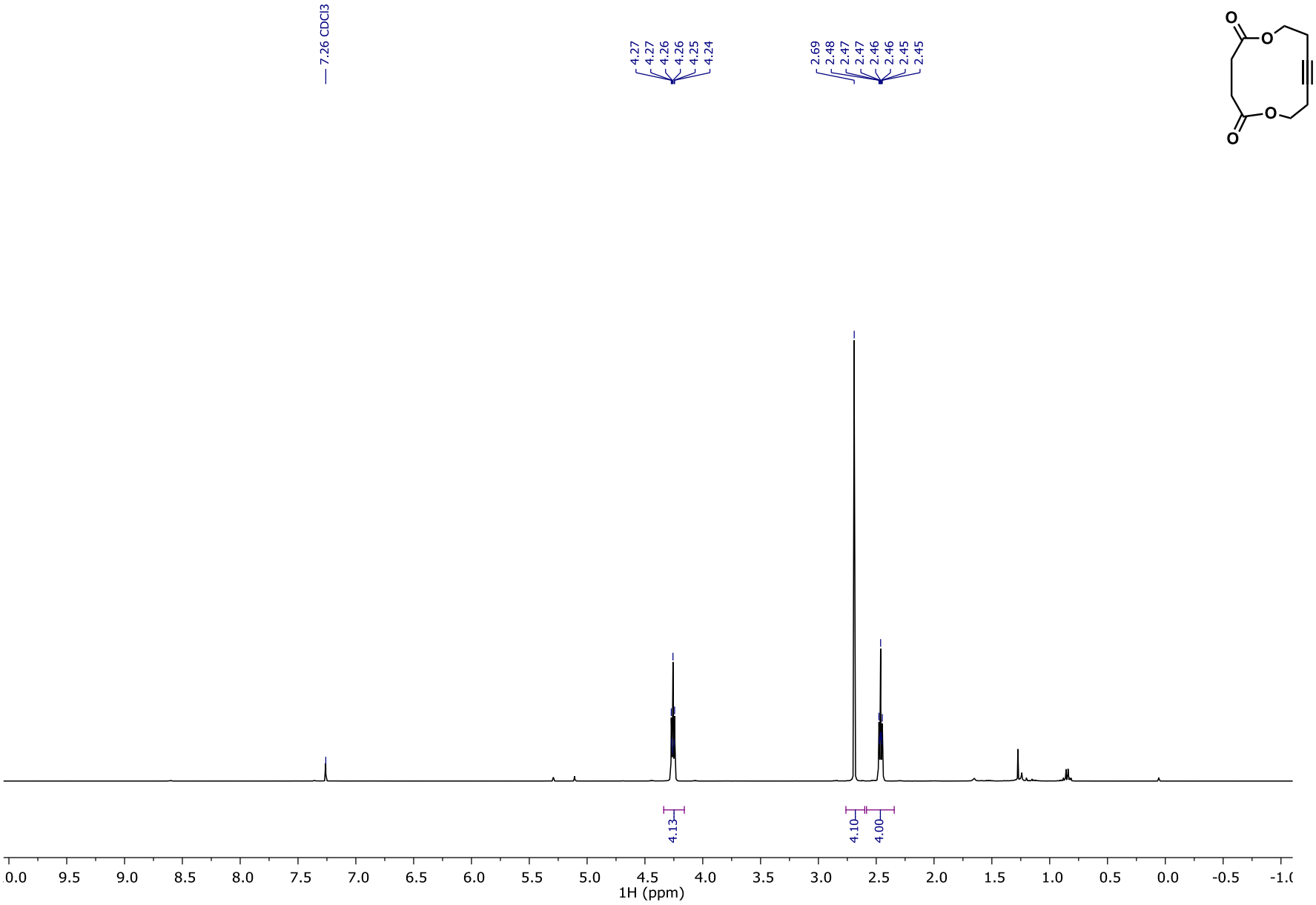
^{1}H NMR of 2,2'-(Octadec-9-yne-1,18-diyl)bis(isoindoline-1,3-dione) (40), 400 MHz, $CDCl_3$, 25ºC



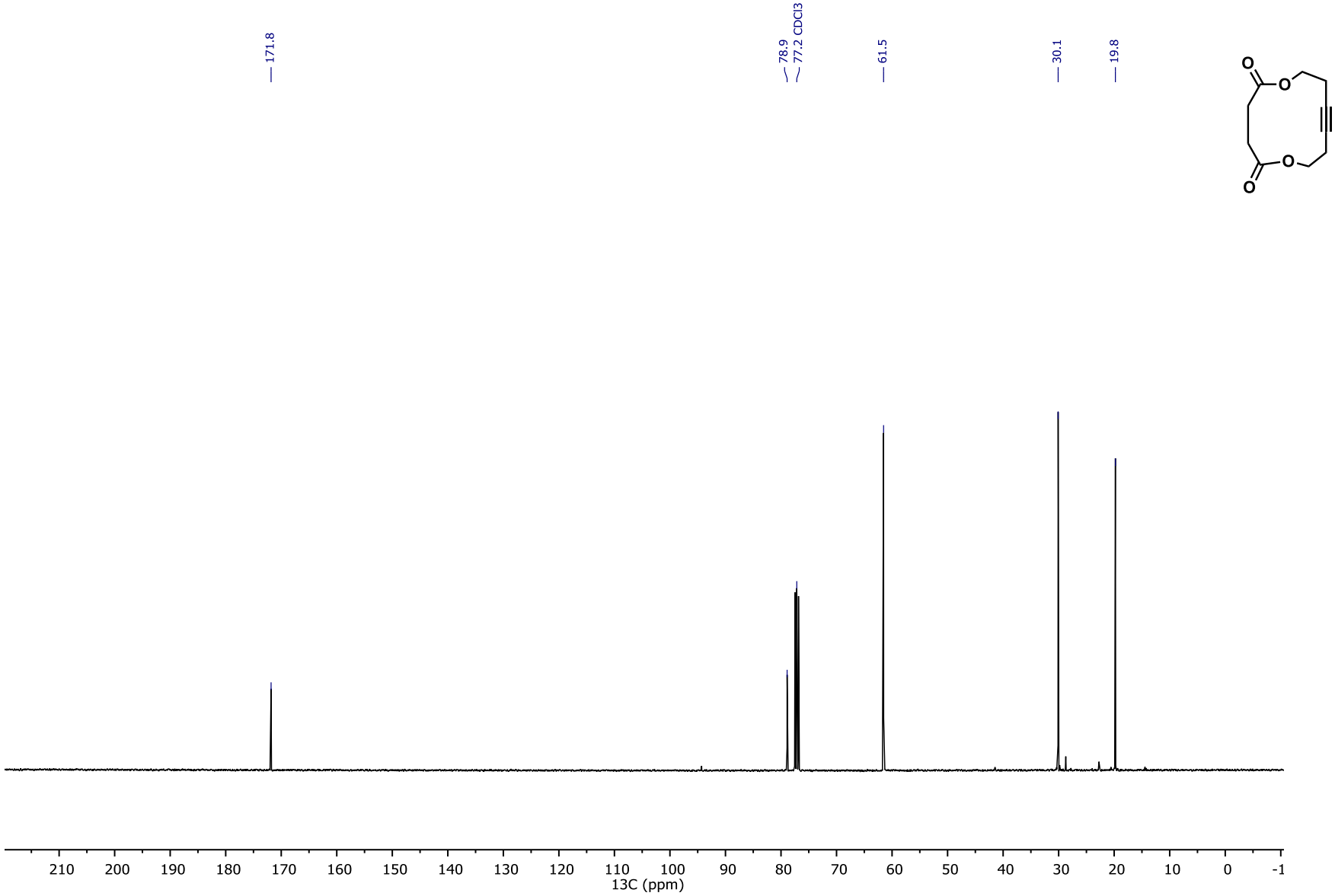
^{13}C NMR of 2,2'-(Octadec-9-yne-1,18-diyl)bis(isoindoline-1,3-dione) (40), 101 MHz, $CDCl_3$, 25ºC



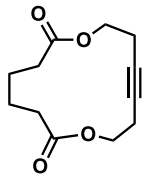
^{1}H NMR of 1,6-Dioxacyclododec-9-yne-2,5-dione (41a), 400 MHz, $CDCl_3$, 25ºC



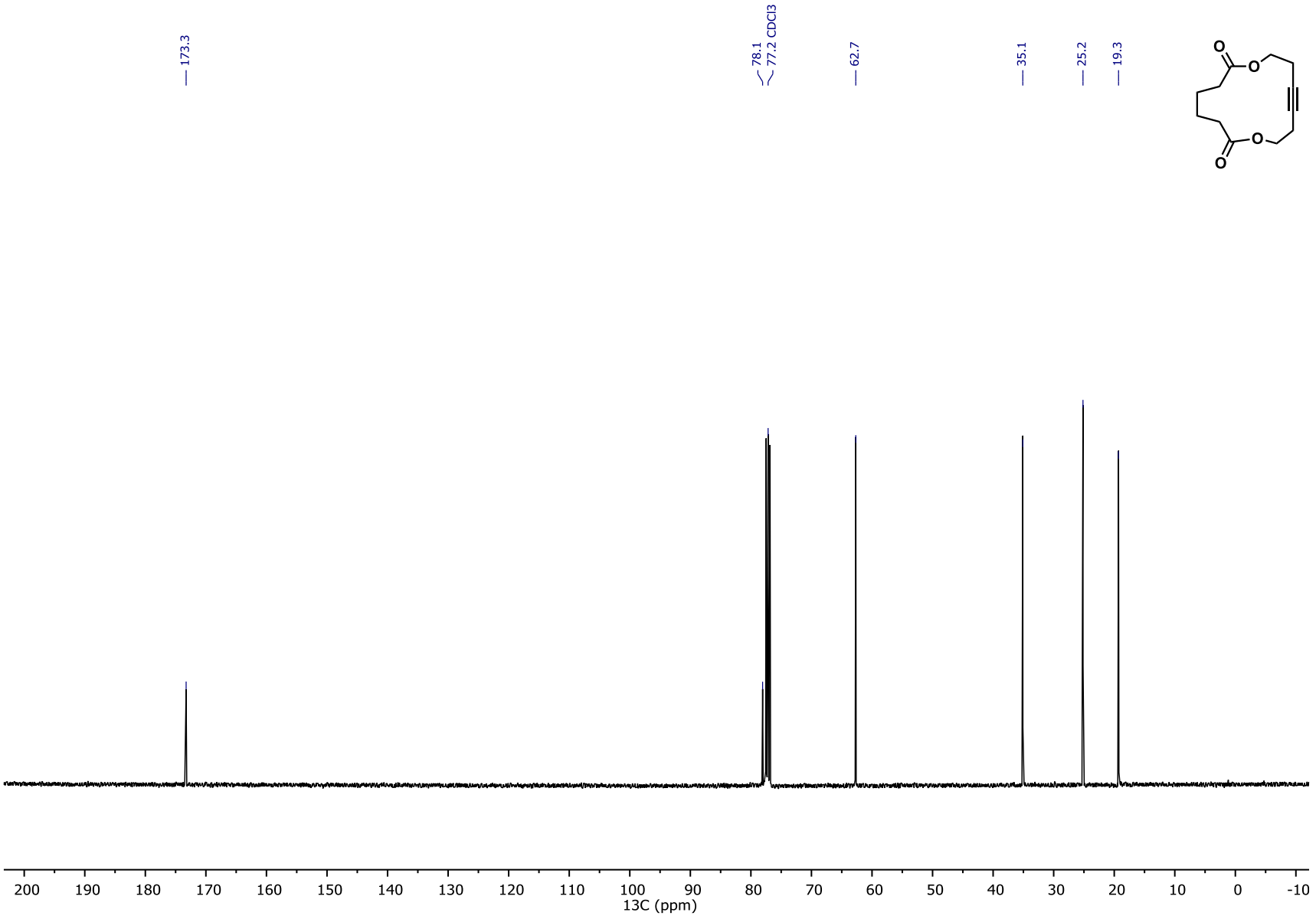
^{13}C NMR of 1,6-Dioxacyclododec-9-yne-2,5-dione (41a), 101 MHz, $CDCl_3$, 25ºC



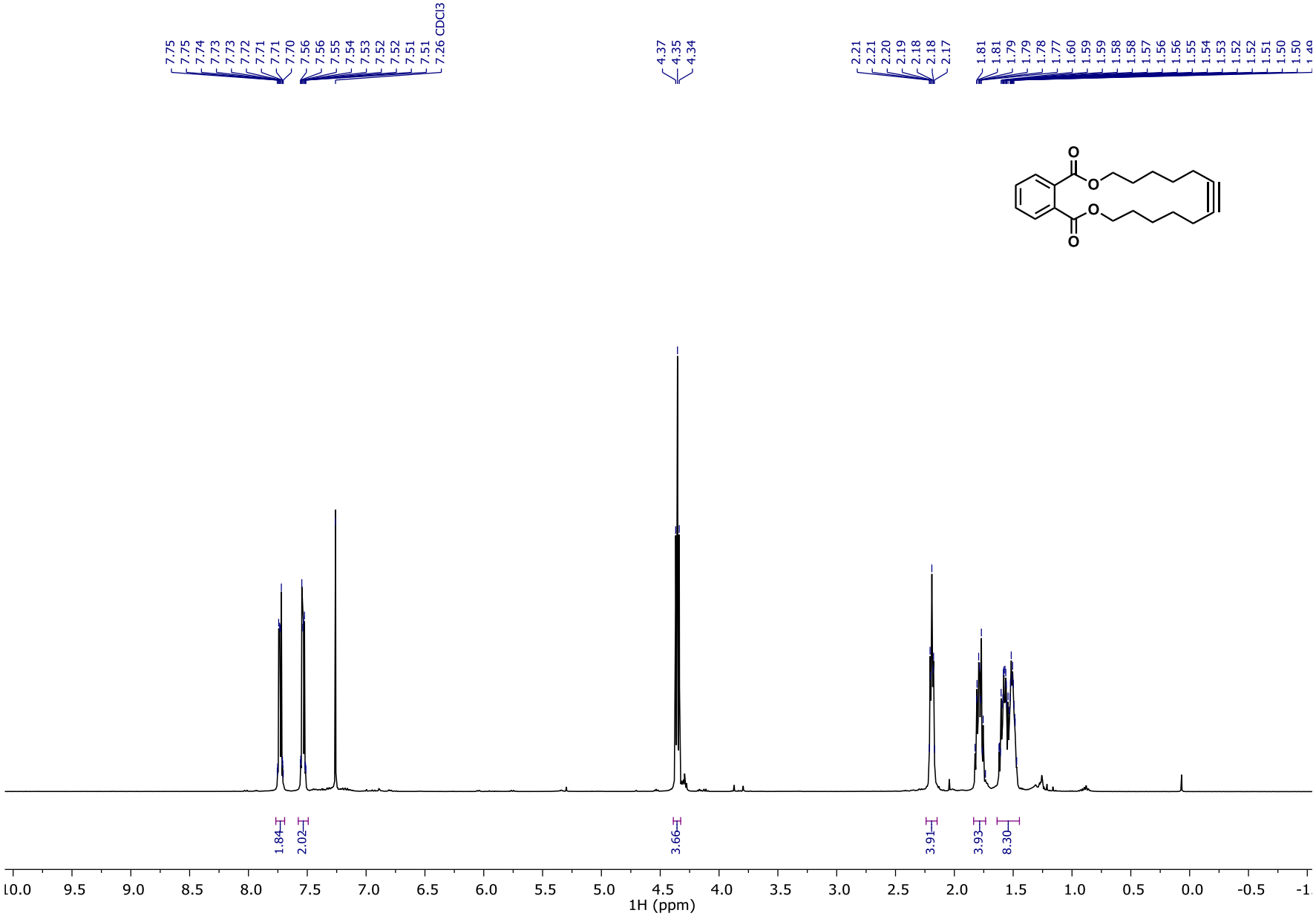
^{1}H NMR of 1,8-Dioxacyclotetradec-11-yne-2,7-dione (41b), 400 MHz, $CDCl_3$, 25ºC



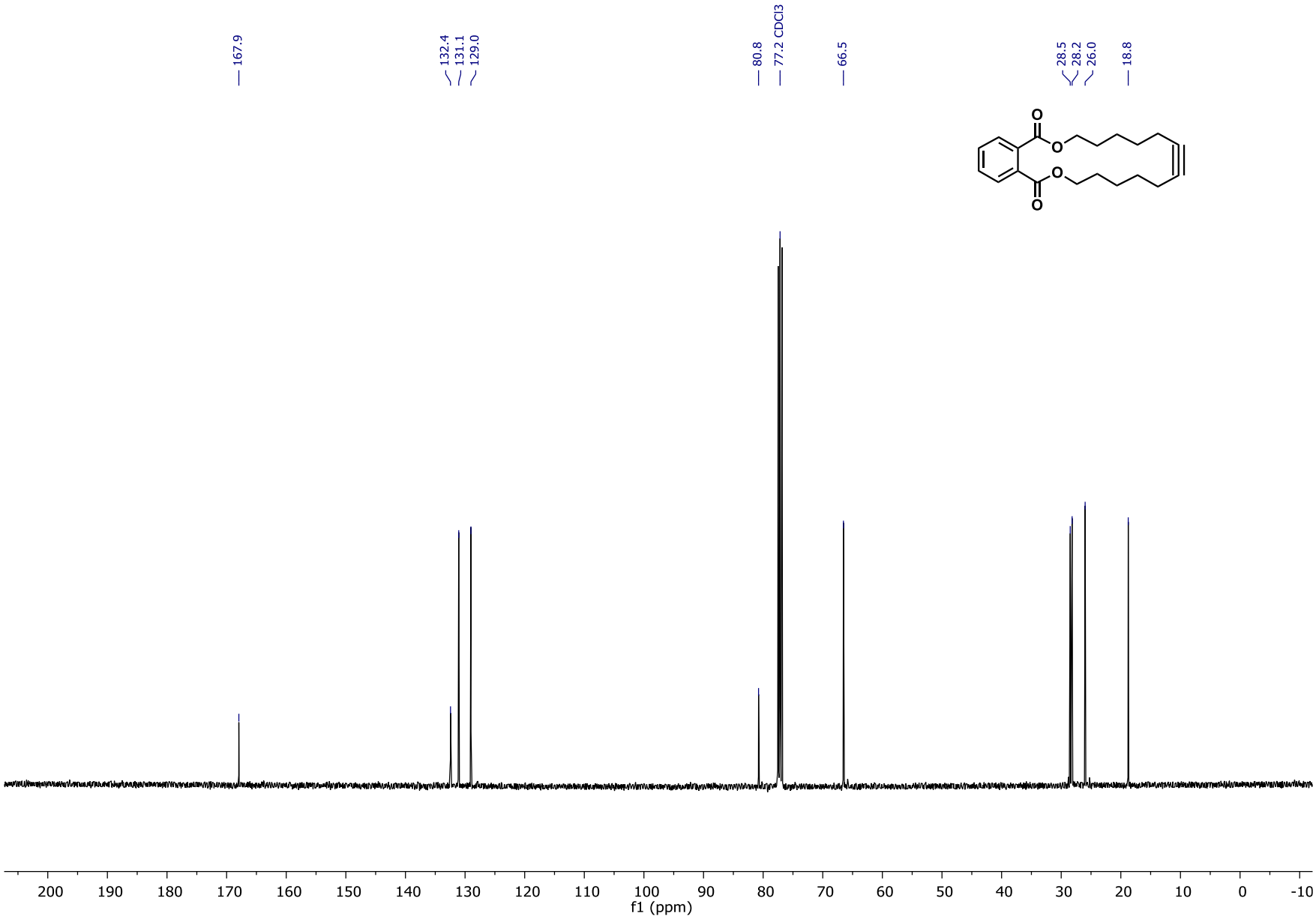
^{13}C NMR of 1,8-Dioxacyclotetradec-11-yne-2,7-dione (41b), 101 MHz, $CDCl_3$, 25ºC



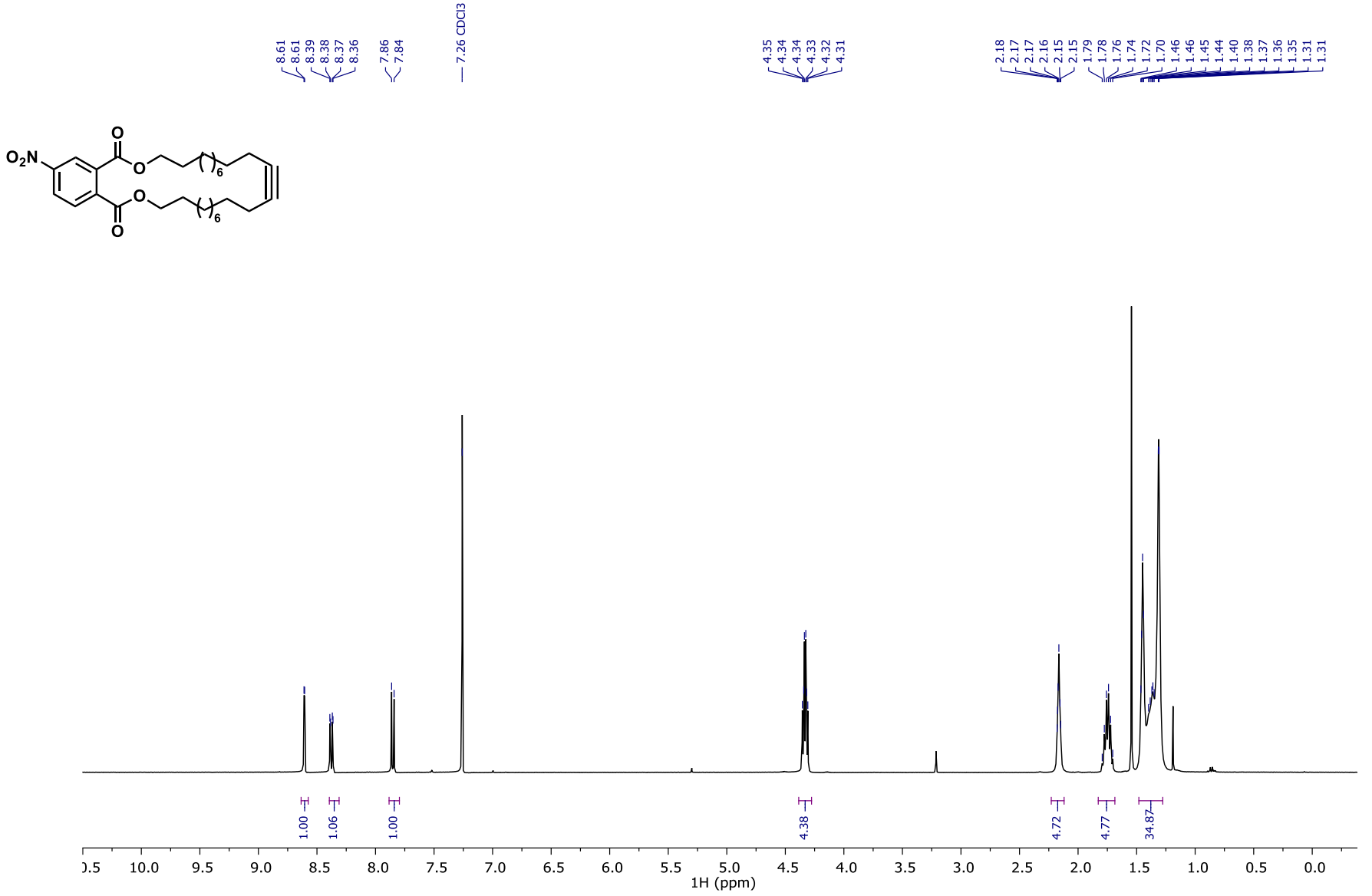
^{1}H NMR of Compound 42, 400 MHz, $CDCl_3$, 25°C



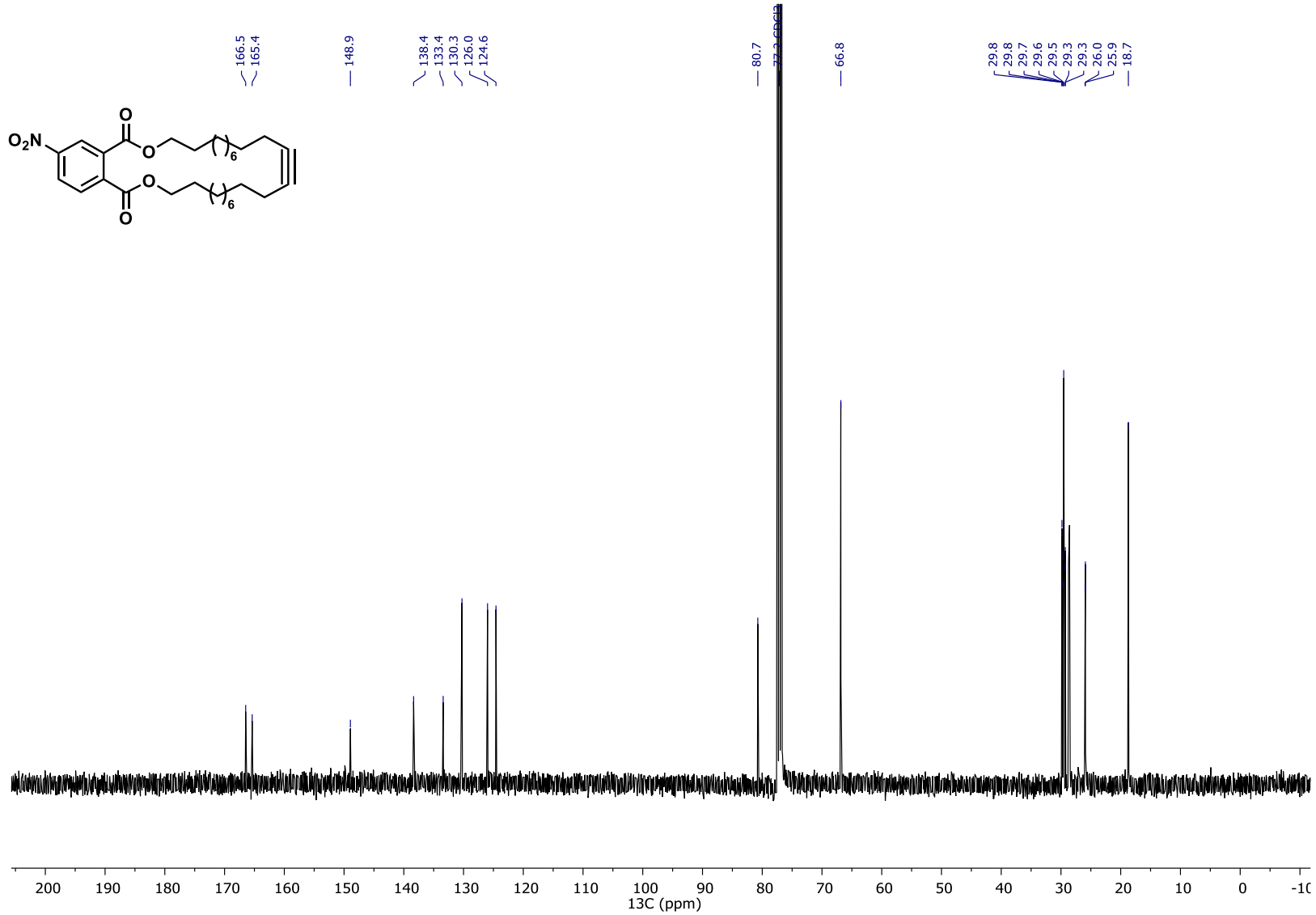
^{3}C NMR of Compound 42, 101 MHz**,** $CDCl_3$, 25ºC



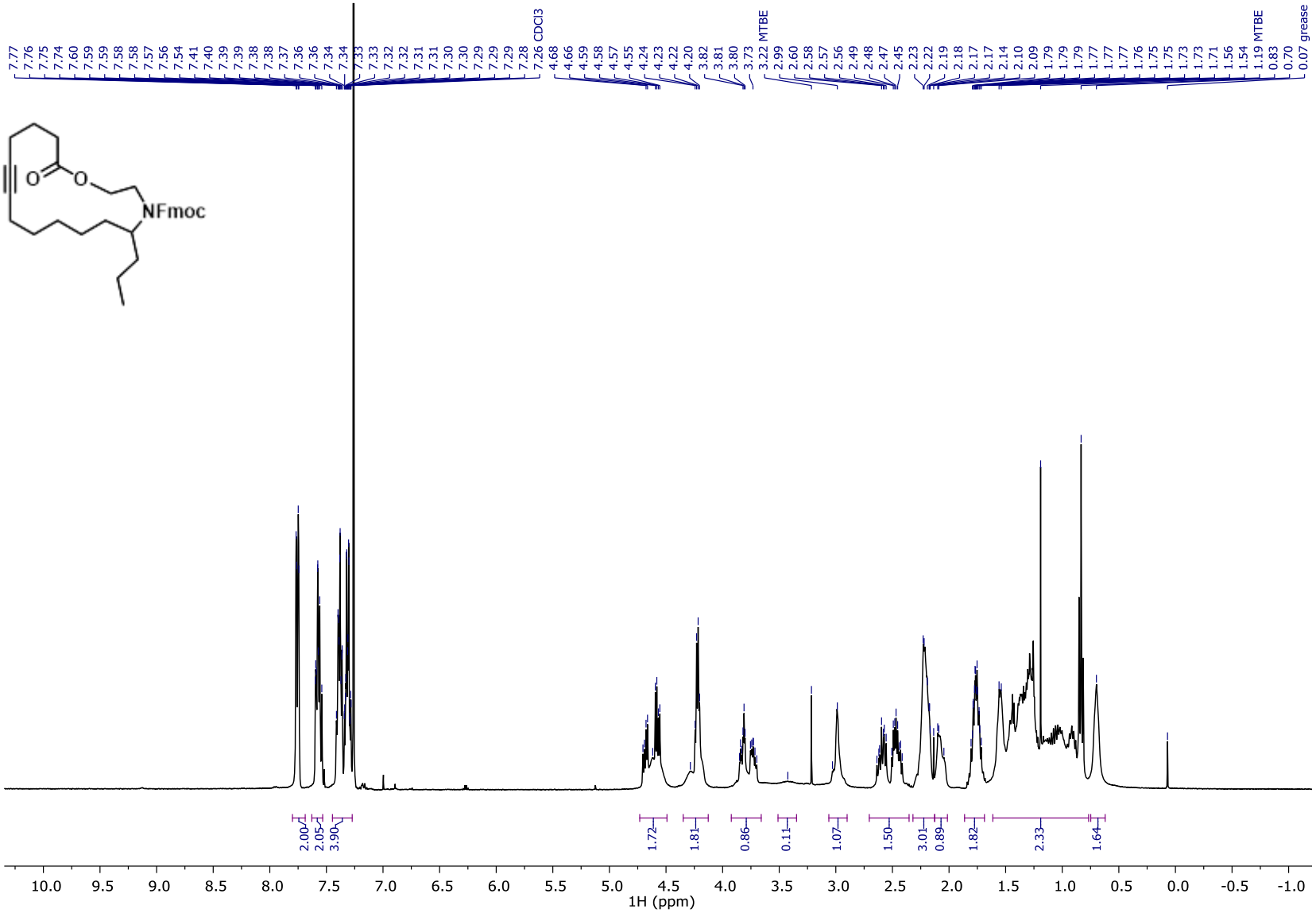
^{1}H NMR of compound 43, 400 MHz, $CDCl_3$, 25ºC



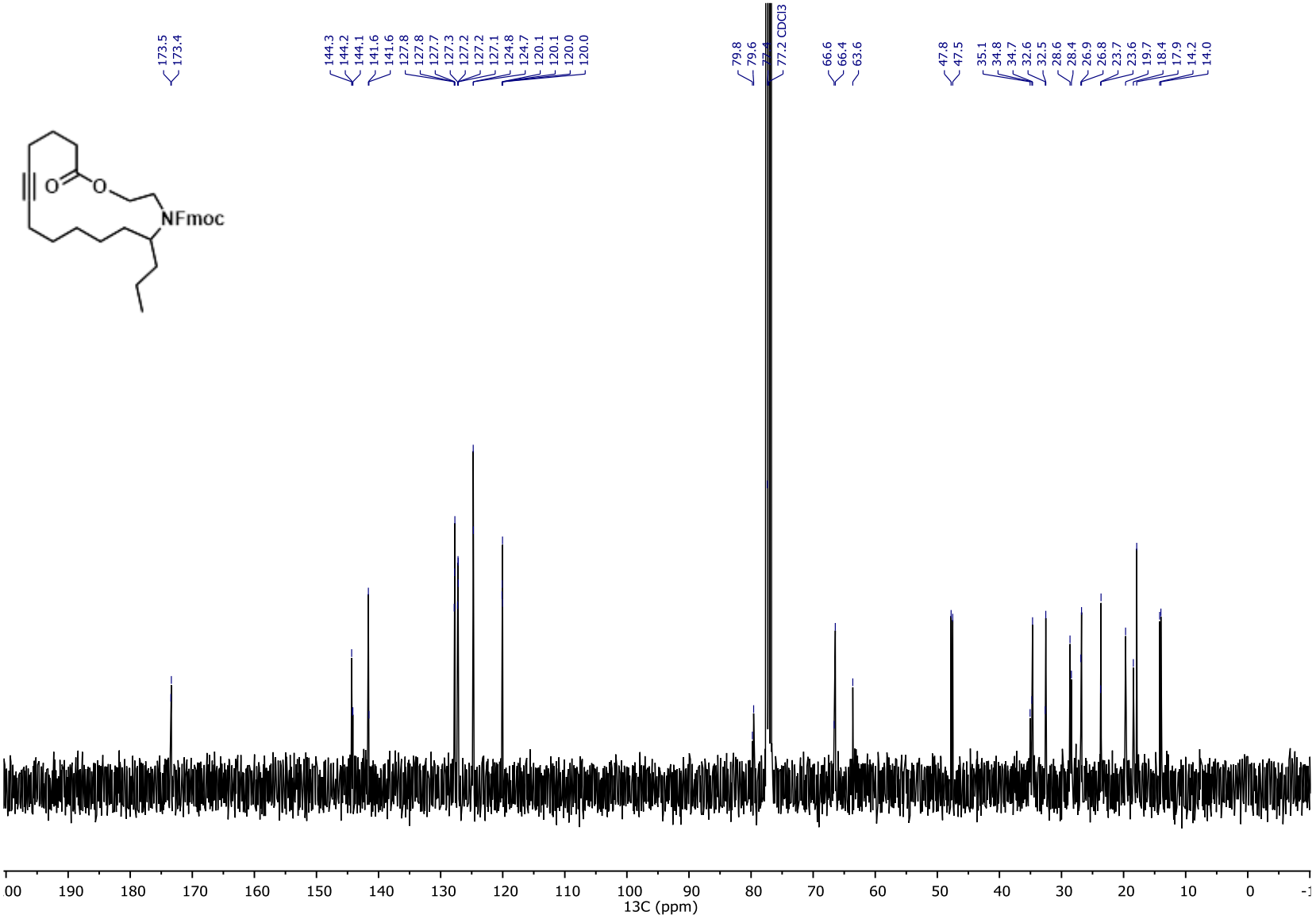
^{13}C NMR of compound 45, 101 MHz, $CDCl_3$, 25ºC



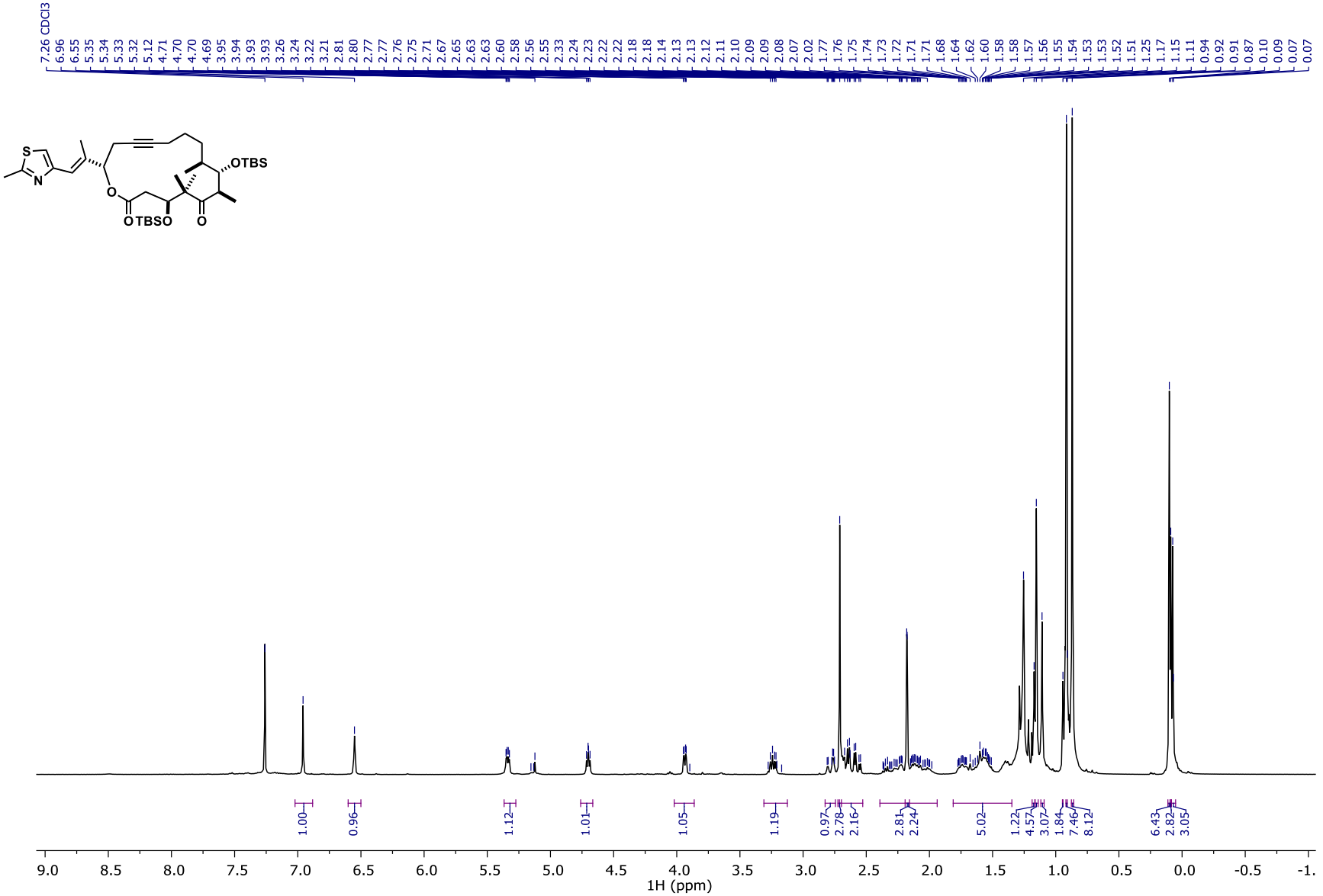
^{1}H NMR of Compound 45, 400 MHz, $CDCl_3$, 25ºC



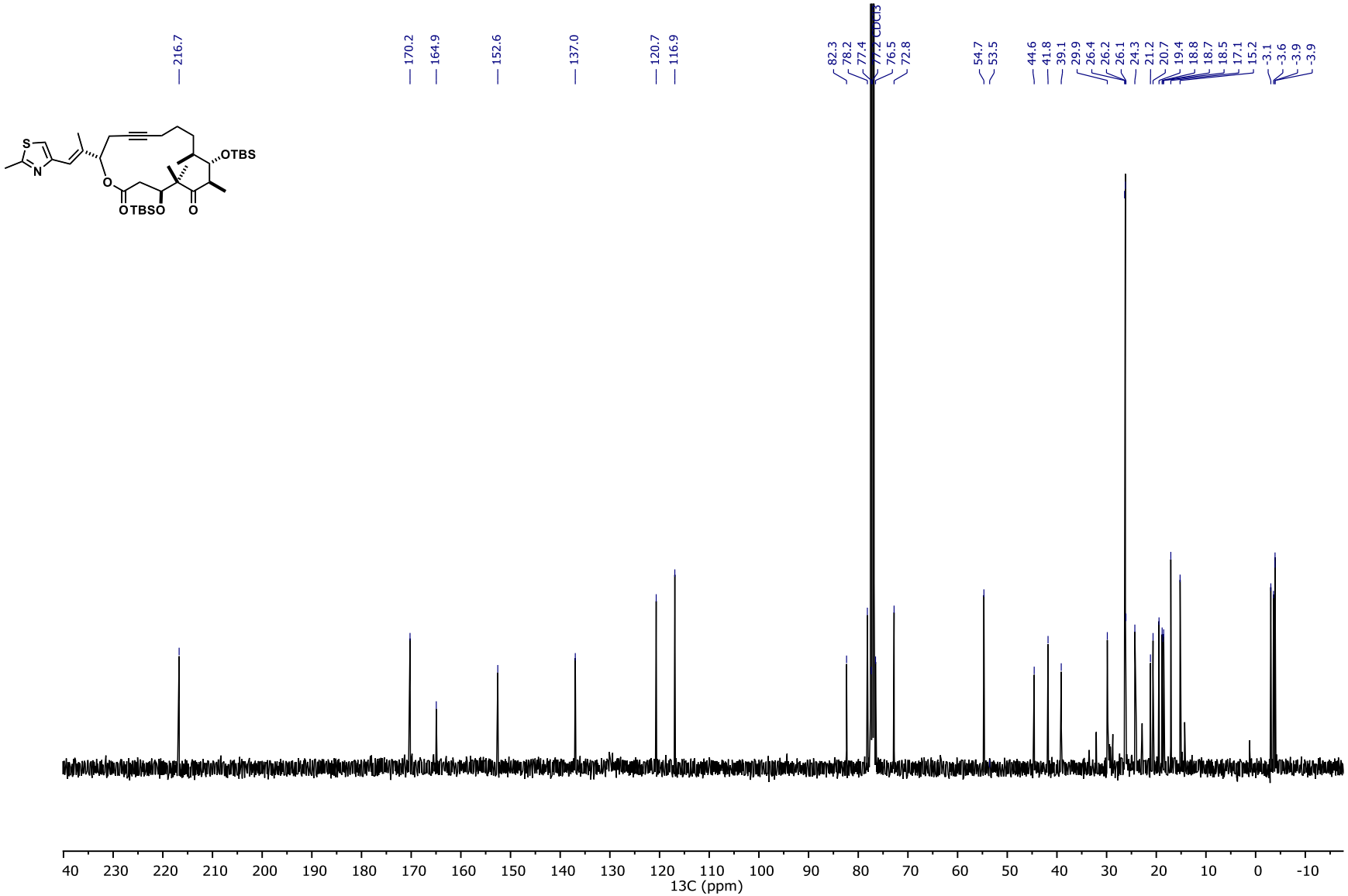
^{13}C NMR of compound 45, 101 MHz, $CDCl_3$, 25ºC



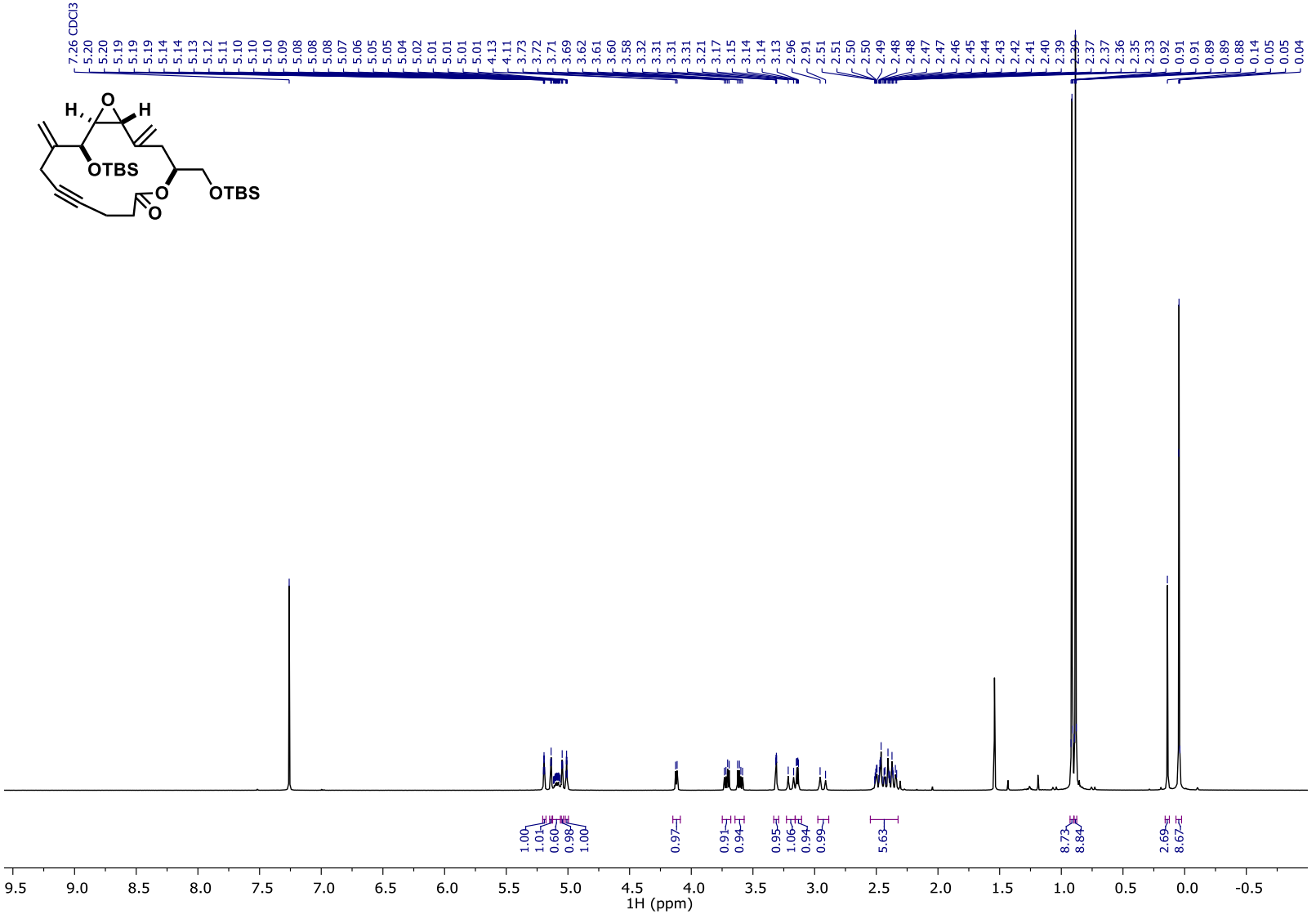
^{1}H NMR of compound 47, 400 MHz**,** $CDCl_3$, 25ºC



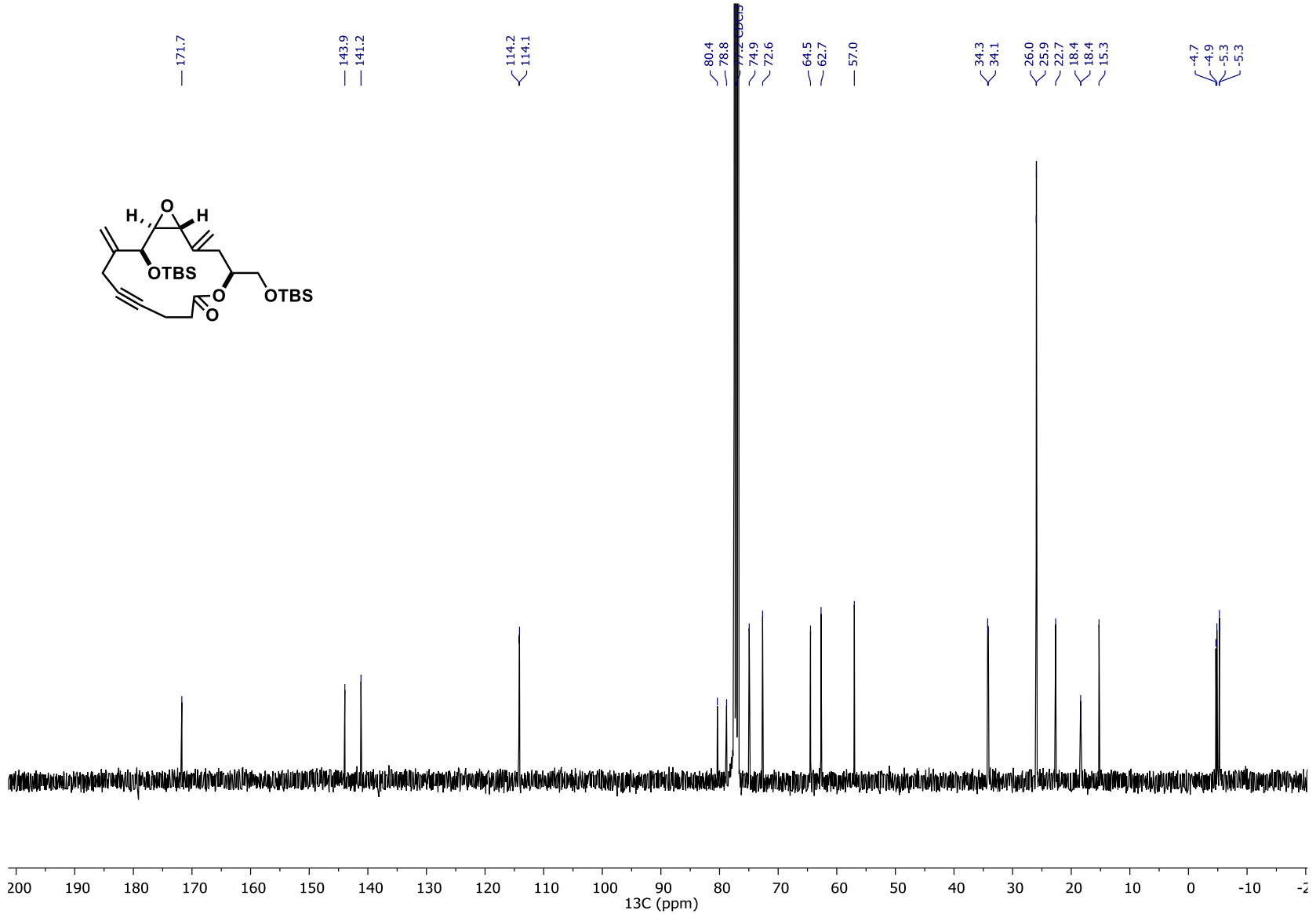
^{13}C NMR of compound 47, 101 MHz, $CDCl_3$, 25ºC



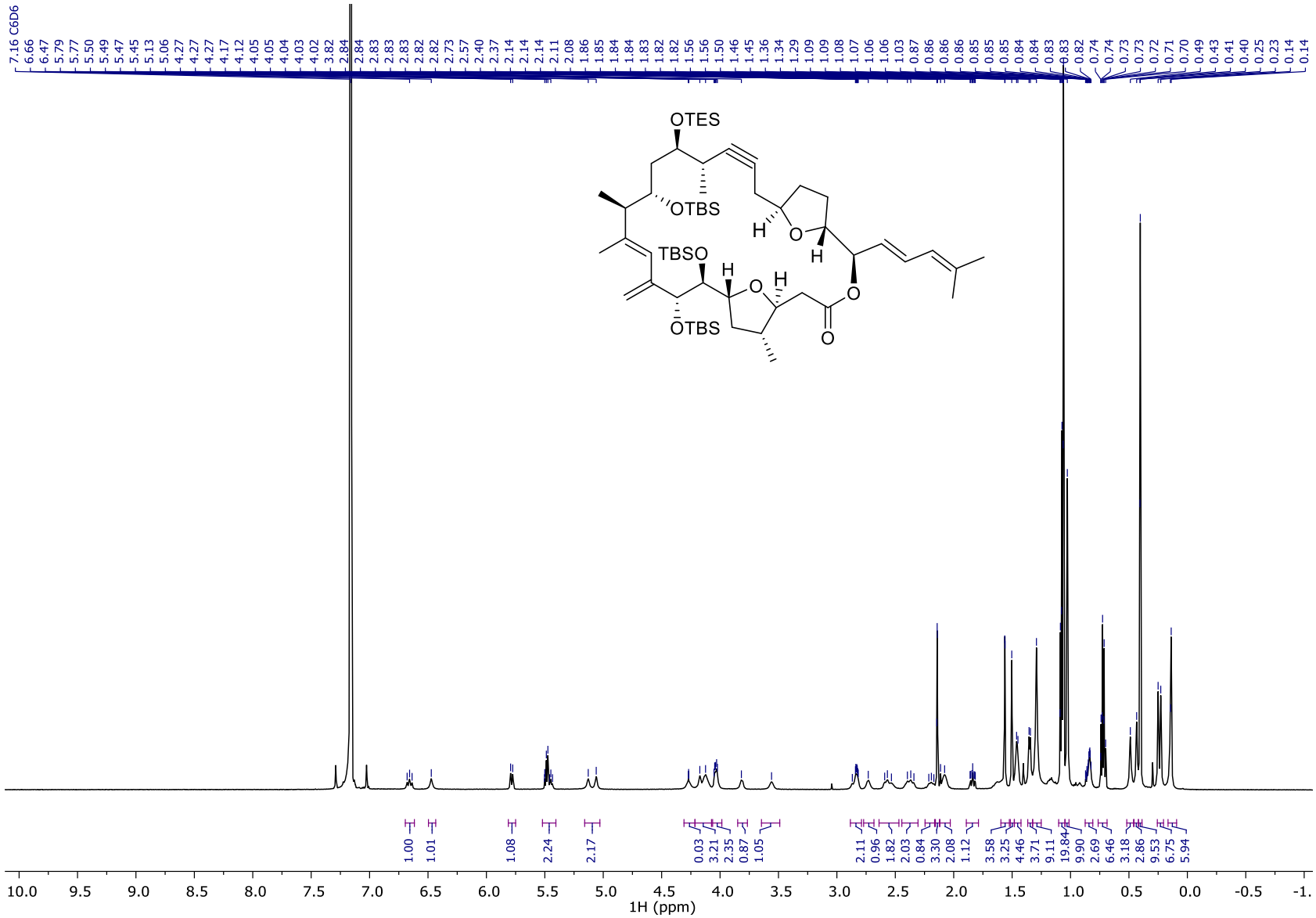
^{1}H NMR of compound 49, 400 MHz, $CDCl_3$, 25ºC



^{13}C NMR of compound 49, 101 MHz, $CDCl_3$, 25ºC



^{1}H NMR of Compound 50, 600 MHz, C_6D_6, 25ºC



^{13}C NMR of Compound 50, 151 MHz, C_6D_6, 25ºC