Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Electronic Supplementary Information

Disila-analogues of the synthetic retinoids EC23 and TTNN: synthesis, structure and biological evaluation

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Table S1 Crystallographic data and experimental parameters for the crystal structure analyses of **5a**, **5b** and **8b**.

Compound	**5a**	**5b**	**8b**
Empirical formula	$C_{22}H_{22}O_2$	$C_{20}H_{22}O_2Si_2$	$C_{22}H_{24}O_2Si_2$
Formula mass [g mol^{-1}]	318.40	350.56	376.59
Collection T [K]	173(2)	173(2)	173(2)
λ(Mo$_{K\alpha}$) [Å]	0.71073	0.71073	0.71073
Crystal system	triclinic	orthorhombic	orthorhombic
Space group (No.)	*P*1 (1)	*Pbca* (61)	*Pbca* (61)
a [Å]	6.6502(19)	9.7328(10)	10.3734(6)
b [Å]	8.0811(19)	11.2756(8)	11.1736(7)
c [Å]	18.466(5)	35.715(4)	36.103(2)
α [°]	80.28(3)	90	90
β [°]	84.36(3)	90	90
γ [°]	66.24(3)	90	90
V [Å^3]	894.7(4)	3919.5(6)	4184.6(4)
Z	2	8	8
D_{calcd} [g cm^{-3}]	1.182	1.188	1.196
μ [mm^{-1}]	0.074	0.190	0.182
F(000)	340	1488	1600
Crystal dimensions [mm]	0.50 × 0.20 × 0.02	0.5 × 0.4 × 0.1	0.4 × 0.3 × 0.2
2θ range [°]	5.56–58.34	4.76–53.84	4.52–53.72
Index ranges	$-9 \le h \le 9$,	$-12 \le h \le 12$,	$-13 \le h \le 13$,
	$-10 \le k \le 10$,	$-13 \le k \le 12$,	$-14 \le k \le 14$,
	$-25 \le l \le 25$	$-45 \le l \le 26$	$-45 \le l \le 45$
No. of collected reflections	12799	19094	30629
No. of independent reflections	4485	3971	4474
R_{int}	0.0927	0.0456	0.0654
No. of reflections used	4485	3971	4474
No. of parameters	232	224	240
No. of restraints	12	0	0
S^a	0.900	1.014	1.030
Weight parameters a/b^b	0.1005/0.0000	0.0665/0.2200	0.0540/2.6001
R_1^c [$I > 2\sigma(I)$]	0.0579	0.0402	0.0469
wR_2^d (all data)	0.1664	0.1109	0.1229
Max./min. residual electron density [e Å^{-3}]	+0.368/−0.261	+0.232/−0.284	+0.271/–0.301

[a] $S = \{\sum[w(F_o^2 - F_c^2)^2]/(n-p)\}^{0.5}$; n = no. of reflections; p = no. of parameters. [b] $w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$, with $P = [\max(F_o^2,0) + 2F_c^2]/3$. [c] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$. [d] $wR_2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]\}^{0.5}$.

Table S2 Crystallographic data and experimental parameters for the crystal structure analyses of **15**, **17** and **28**.

Compound	**15**	**17**	**28**
Empirical formula	$C_{23}H_{24}O_2$	$C_{20}H_{22}O_3Si_2$	$C_{14}H_{26}Si_3$
Formula mass [g mol^{-1}]	332.42	366.56	278.62
Collection T [K]	100(2)	100(2)	173(2)
λ(Mo$_{K\alpha}$) [Å]	0.71073	0.71073	0.71073
Crystal system	monoclinic	monoclinic	monoclinic
Space group (No.)	$P2_1/c$ (14)	$C2$ (5)	$P2_1/c$ (14)
a [Å]	20.5765(9)	60.024(5)	14.635(4)
b [Å]	11.1441(5)	11.4478(8)	10.5599(16)
c [Å]	8.1245(4)	8.7407(6)	11.643(3)
β [°]	98.9270(10)	95.534(4)	94.50(3)
V [Å^3]	1840.43(15)	5978.1(7)	1793.9(7)
Z	4	12	4
D_{calcd} [g cm^{-3}]	1.200	1.222	1.032
μ [mm^{-1}]	0.075	0.193	0.247
F(000)	712	2328	608
Crystal dimensions [mm]	0.30 × 0.22 × 0.10	0.47 × 0.20 × 0.03	0.50 × 0.30 × 0.15
2θ range [°]	2.00−66.28	2.72−52.74	4.76–54.96
Index ranges	$-31 \le h \le 31$, $-17 \le k \le 16$, $-9 \le l \le 12$	$-72 \le h \le 74$, $-14 \le k \le 14$, $-10 \le l \le 10$	$-19 \le h \le 19$, $-12 \le k \le 12$, $-15 \le l \le 15$
No. of collected reflections	38468	39207	21879
No. of independent reflections	6772	12031	3958
R_{int}	0.0313	0.0454	0.0949
No. of reflections used	6772	12031	3958
No. of parameters	231	692	161
No. of restraints	0	1	0
S^a	1.072	1.020	0.913
Weight parameters a/b^b	0.0565/0.5356	0.0909/2.7879	0.0588/0.0000
R_1^c [$I > 2\sigma(I)$]	0.0422	0.0549	0.0425
wR_2^d (all data)	0.1216	0.1458	0.1084
Absolute structure parameter		0.00(15)	
Max./min. residual electron density [e Å^{-3}]	+0.558/−0.184	+1.103/−0.295	+0.357/−0.278

[a] $S = \{\sum[w(F_o^2 - F_c^2)^2]/(n-p)\}^{0.5}$; n = no. of reflections; p = no. of parameters. [b] $w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$, with $P = [\max(F_o^2,0) + 2F_c^2]/3$. [c] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$. [d] $wR_2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]\}^{0.5}$.

Table S3 Crystallographic data and experimental parameters for the crystal structure analyses of **29**, **33** and **34**.

Compound	**29**	**33**	**34**
Empirical formula	$C_{13}H_{24}OSi_3$	$C_{13}H_{20}O_2Si_2$	$C_{12}H_{18}O_2Si_2$
Formula mass [g mol^{-1}]	280.59	264.47	250.44
Collection T [K]	173(2)	173(2)	173(2)
$\lambda(Mo_{K\alpha})$ [Å]	0.71073	0.71073	0.71073
Crystal system	monoclinic	monoclinic	monoclinic
Space group (No.)	$P2_1$ (4)	$P2_1/c$ (14)	$C2/c$ (15)
a [Å]	6.2270(8)	12.8871(17)	17.973(4)
b [Å]	10.0764(18)	8.8099(11)	5.5806(8)
c [Å]	14.0512(19)	14.4156(14)	27.605(6)
β [°]	91.347(16)	116.374(12)	90.75(3)
V [Å^3]	881.4(2)	1466.3(3)	2768.5(9)
Z	2	4	8
D_{calcd} [g cm^{-3}]	1.057	1.198	1.202
μ [mm^{-1}]	0.256	0.231	0.241
F(000)	304	568	1072
Crystal dimensions [mm]	0.5 × 0.2 × 0.2	0.4 × 0.4 × 0.2	0.50 × 0.40 × 0.15
2θ range [°]	5.80–59.04	5.60−58.50	7.64−58.32
Index ranges	$-8 \le h \le 8$, $-13 \le k \le 13$, $-19 \le l \le 19$	$-17 \le h \le 17$, $-11 \le k \le 12$, $-19 \le l \le 19$	$-24 \le h \le 24$, $-7 \le k \le 7$, $-37 \le l \le 37$
No. of collected reflections	11609	18328	19009
No. of independent reflections	4562	3938	3545
R_{int}	0.0605	0.0410	0.0368
No. of reflections used	4562	3938	3545
No. of parameters	193	180	150
No. of restraints	51	0	0
S^a	1.053	1.060	1.104
Weight parameters a/b^b	0.0778/0.0000	0.0769/0.3474	0.0619/1.4934
$R_1{}^c$ [$I > 2\sigma(I)$]	0.0433	0.0446	0.0370
$wR_2{}^d$ (all data)	0.1232	0.1252	0.1080
Absolute structure parameter	0.01(13)		
Max./min. residual electron density [e Å^{-3}]	+0.365/−0.381	+0.382/−0.403	+0.364/−0.242

[a] $S = \{\sum[w(F_o^2 - F_c^2)^2]/(n-p)\}^{0.5}$; n = no. of reflections; p = no. of parameters. [b] $w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$, with $P = [\max(F_o^2,0) + 2F_c^2]/3$. [c] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$. [d] $wR_2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]\}^{0.5}$.

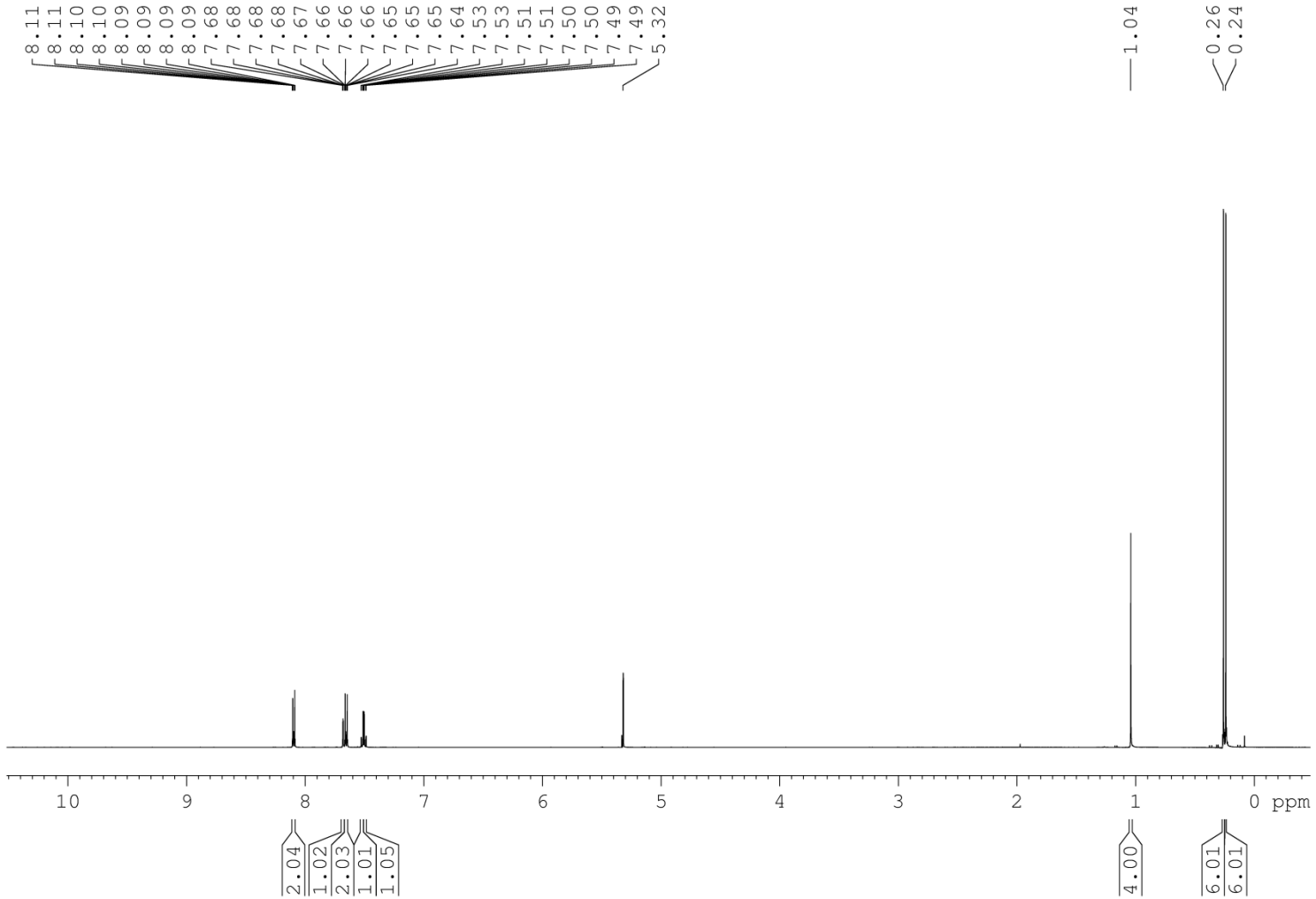

Figure S1 ^{1}H NMR spectrum of compound **4b**.

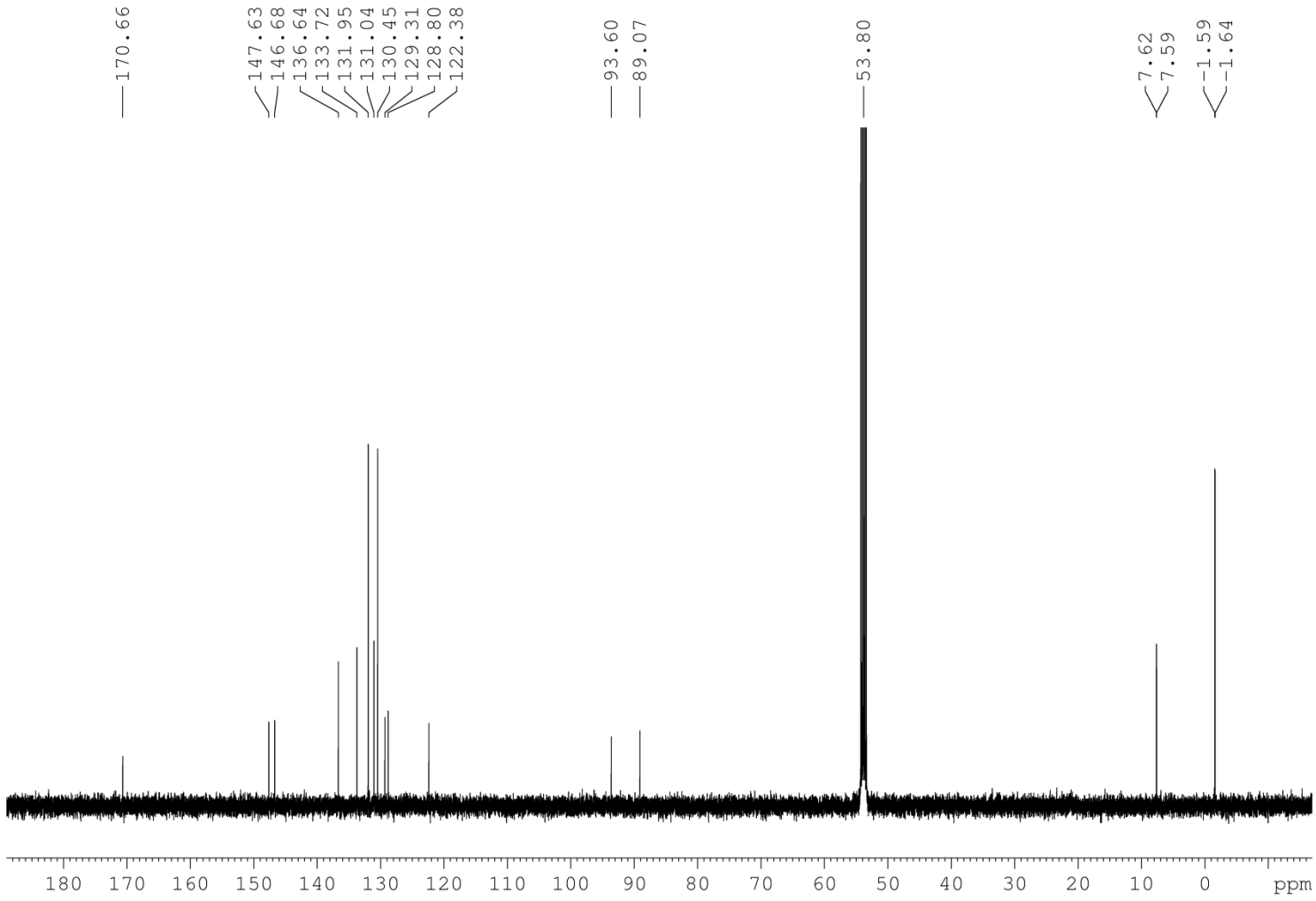

Figure S2 ^{13}C NMR spectrum of compound **4b**.

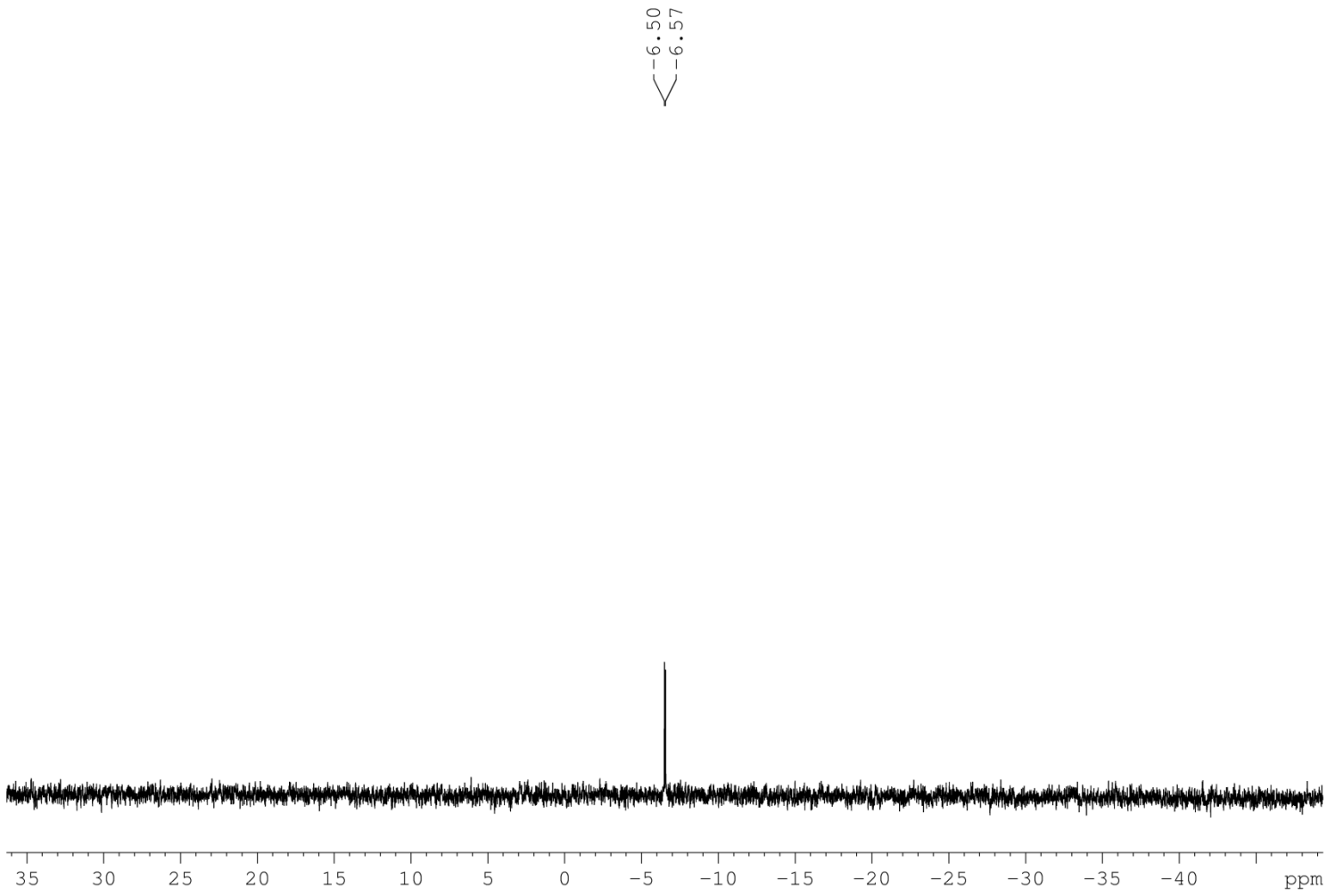

Figure S3 ^{29}Si NMR spectrum of compound **4b**.

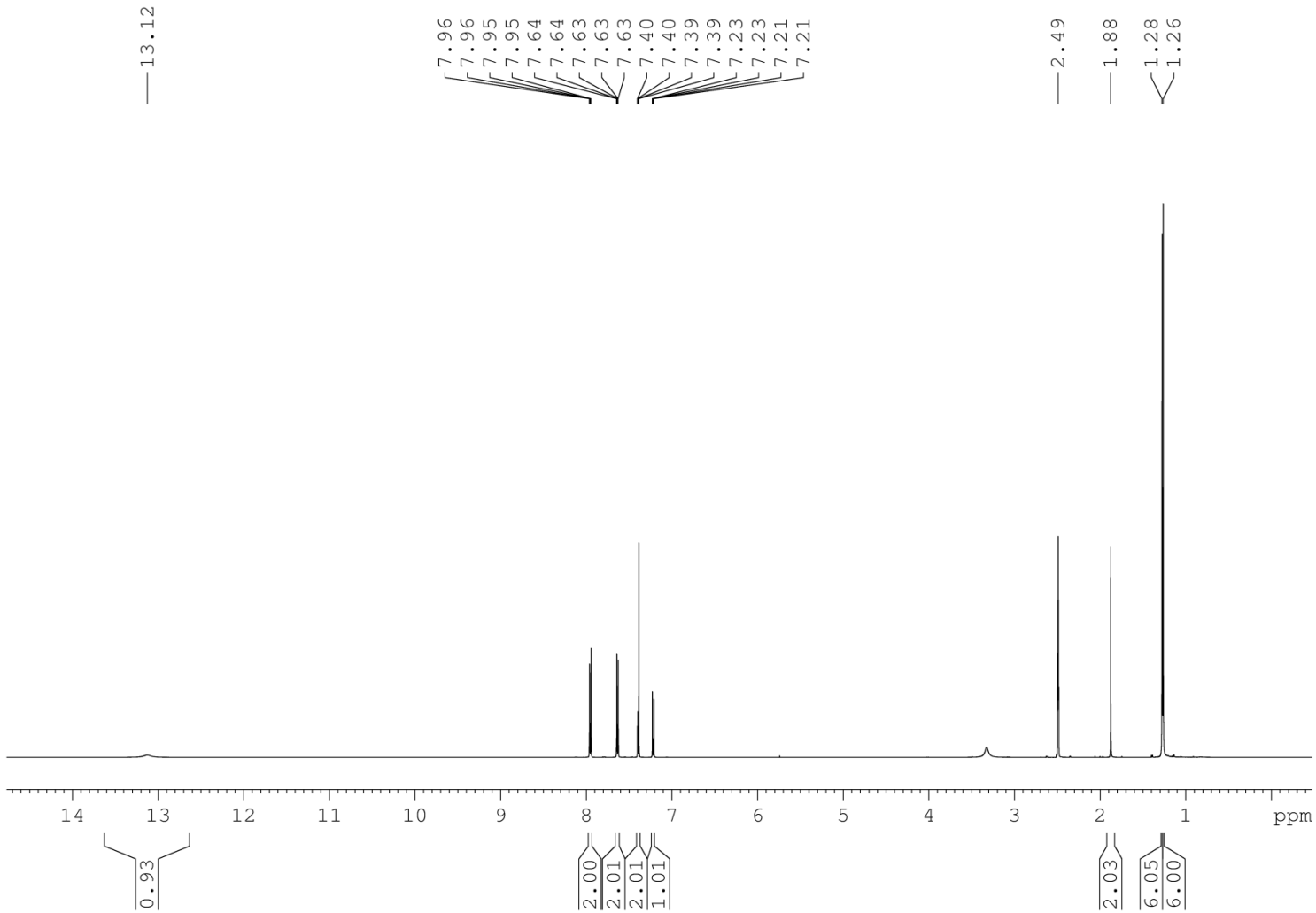

Figure S4 ^{1}H NMR spectrum of compound **5a**.

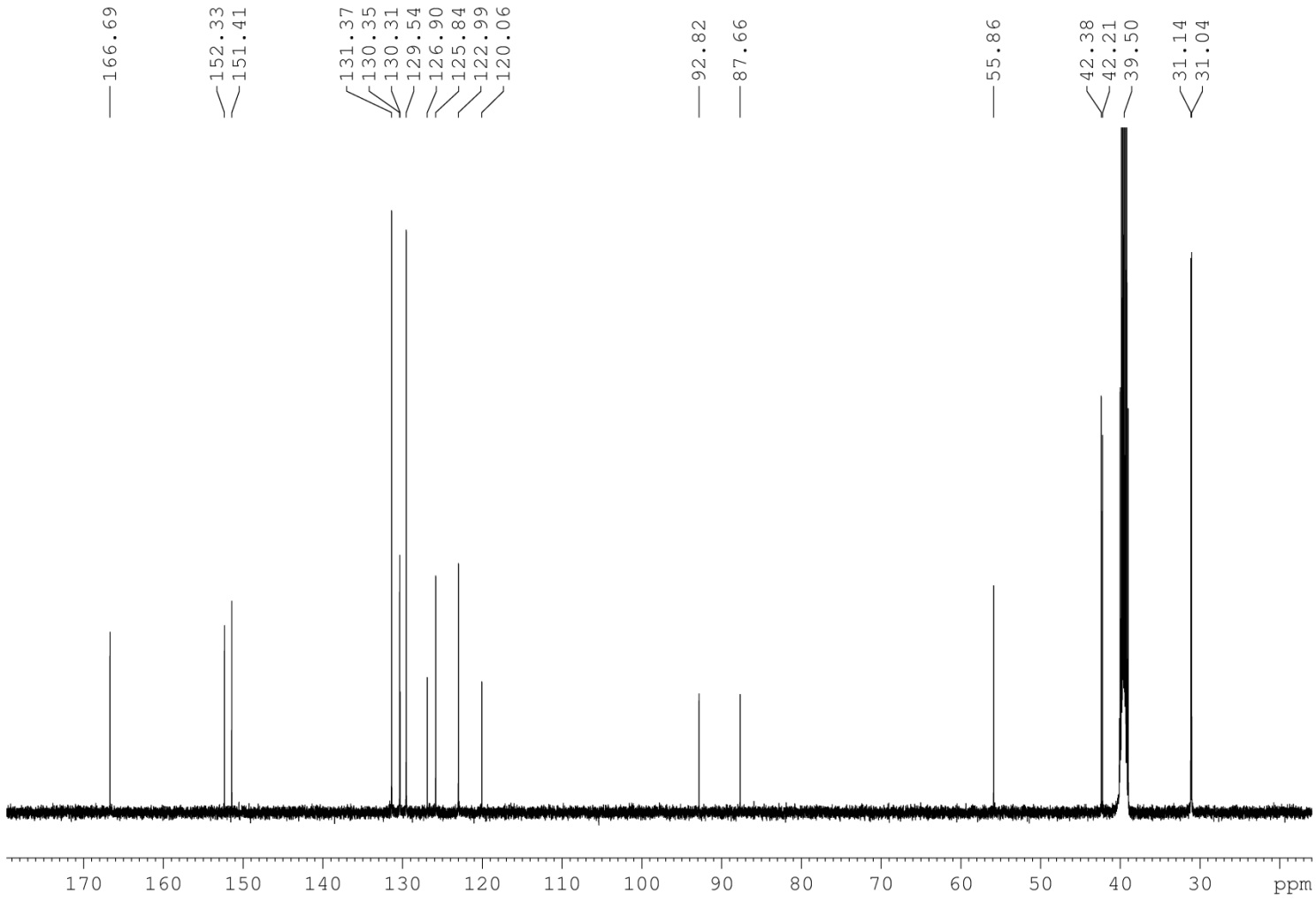

Figure S5 ^{13}C NMR spectrum of compound **5a**.

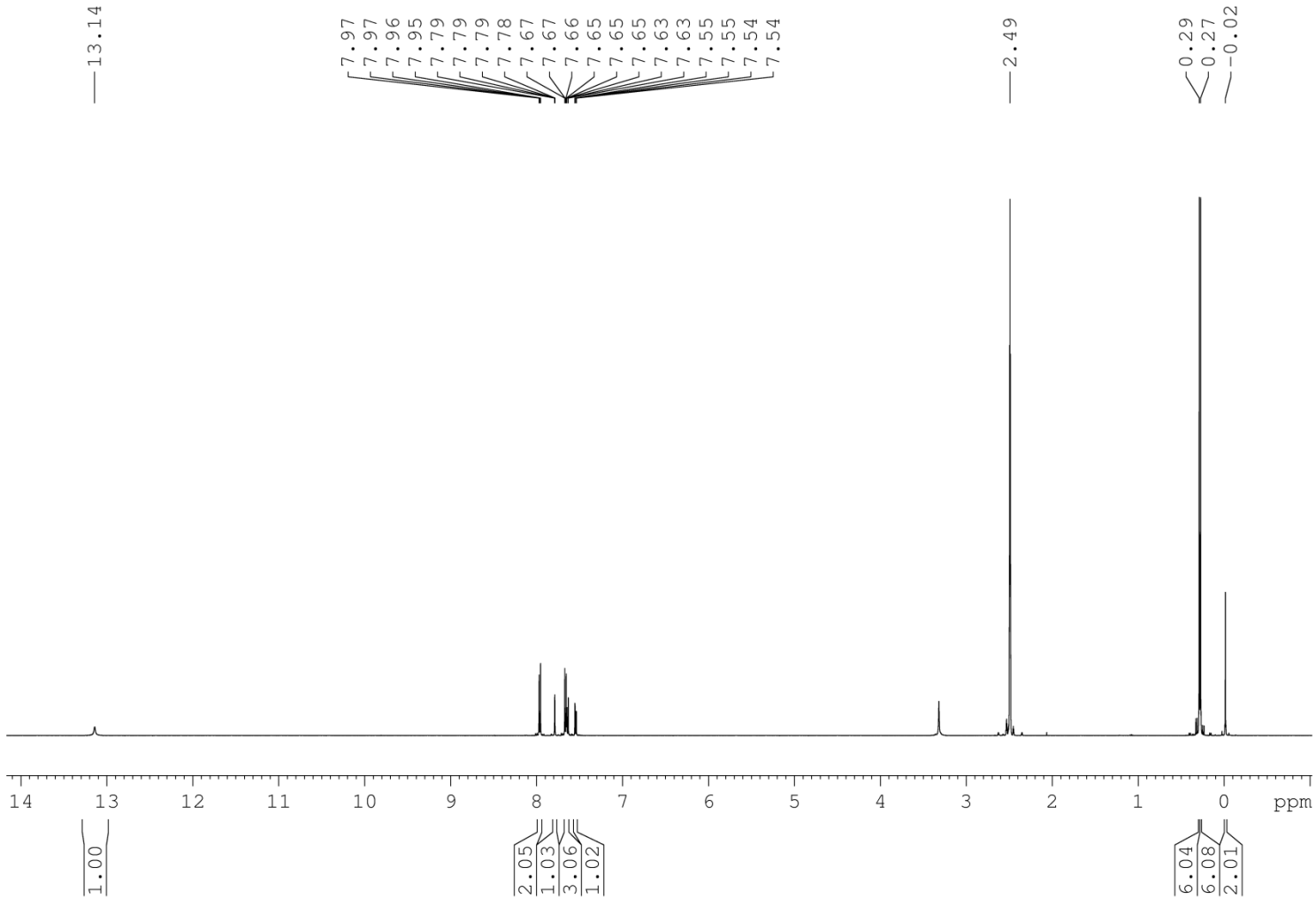

Figure S6 ^{1}H NMR spectrum of compound **5b**.

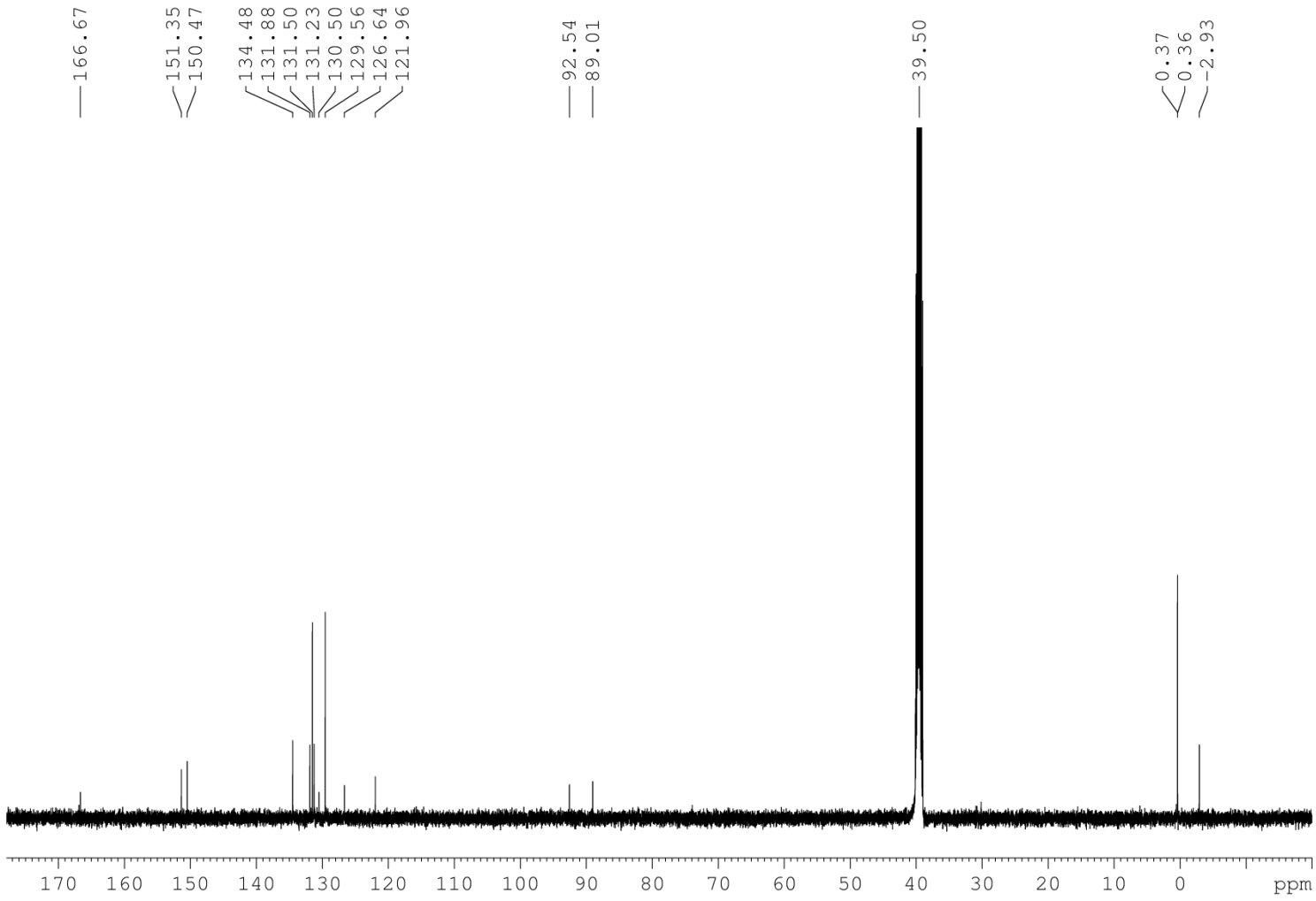

Figure S7 ^{13}C NMR spectrum of compound **5b**.

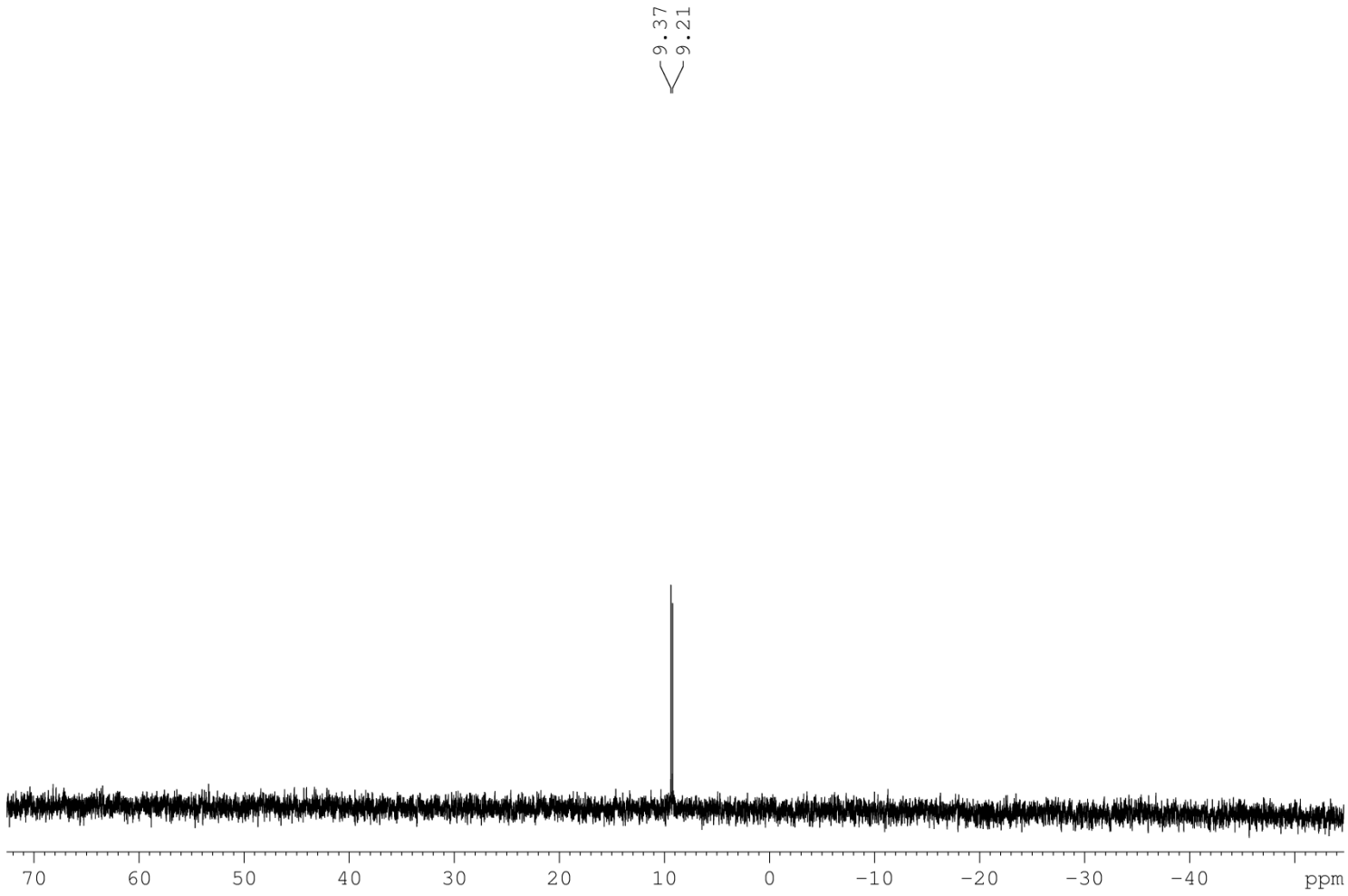

Figure S8 ^{29}Si NMR spectrum of compound **5b**.

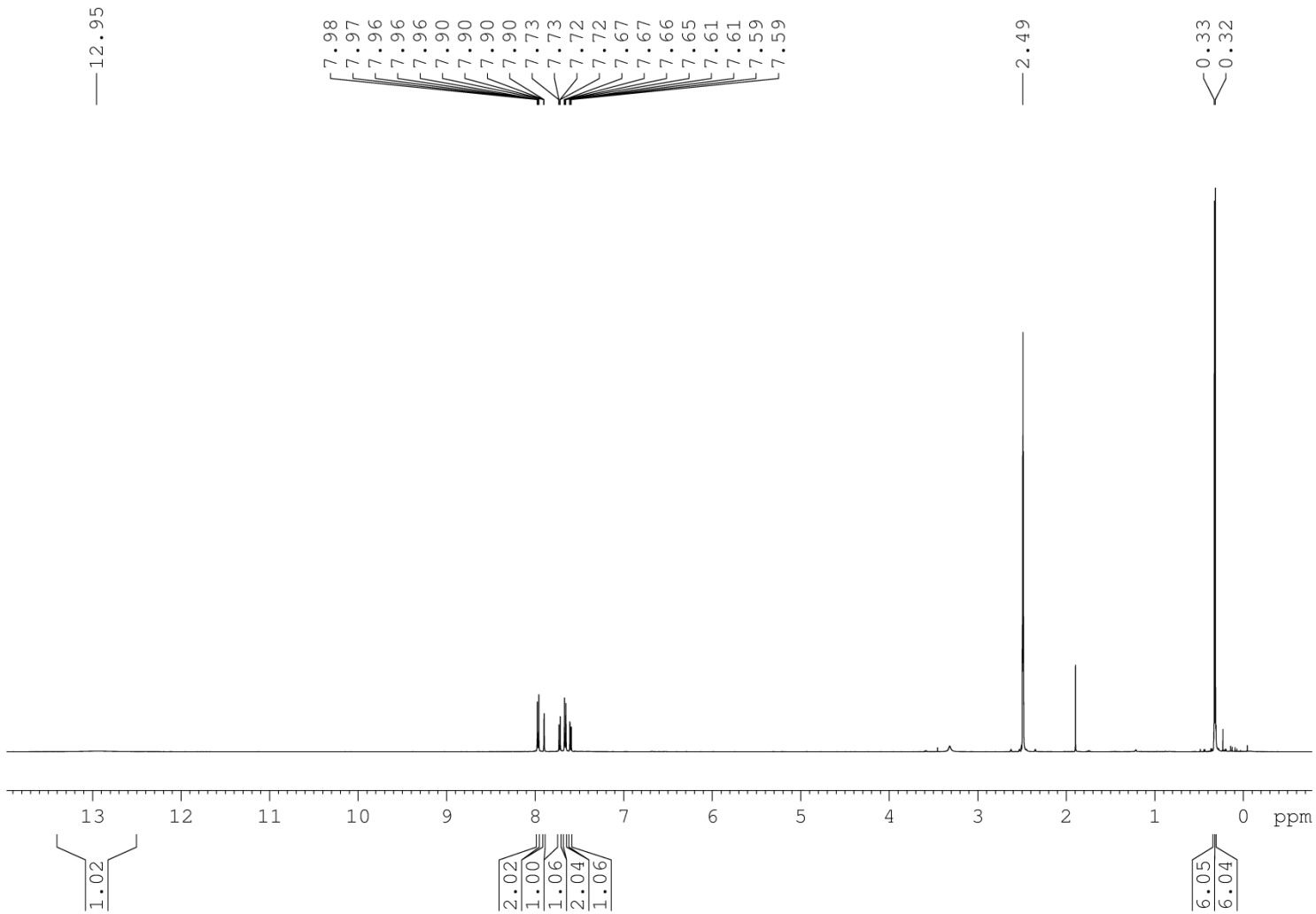

Figure S9 ^{1}H NMR spectrum of compound **6**.

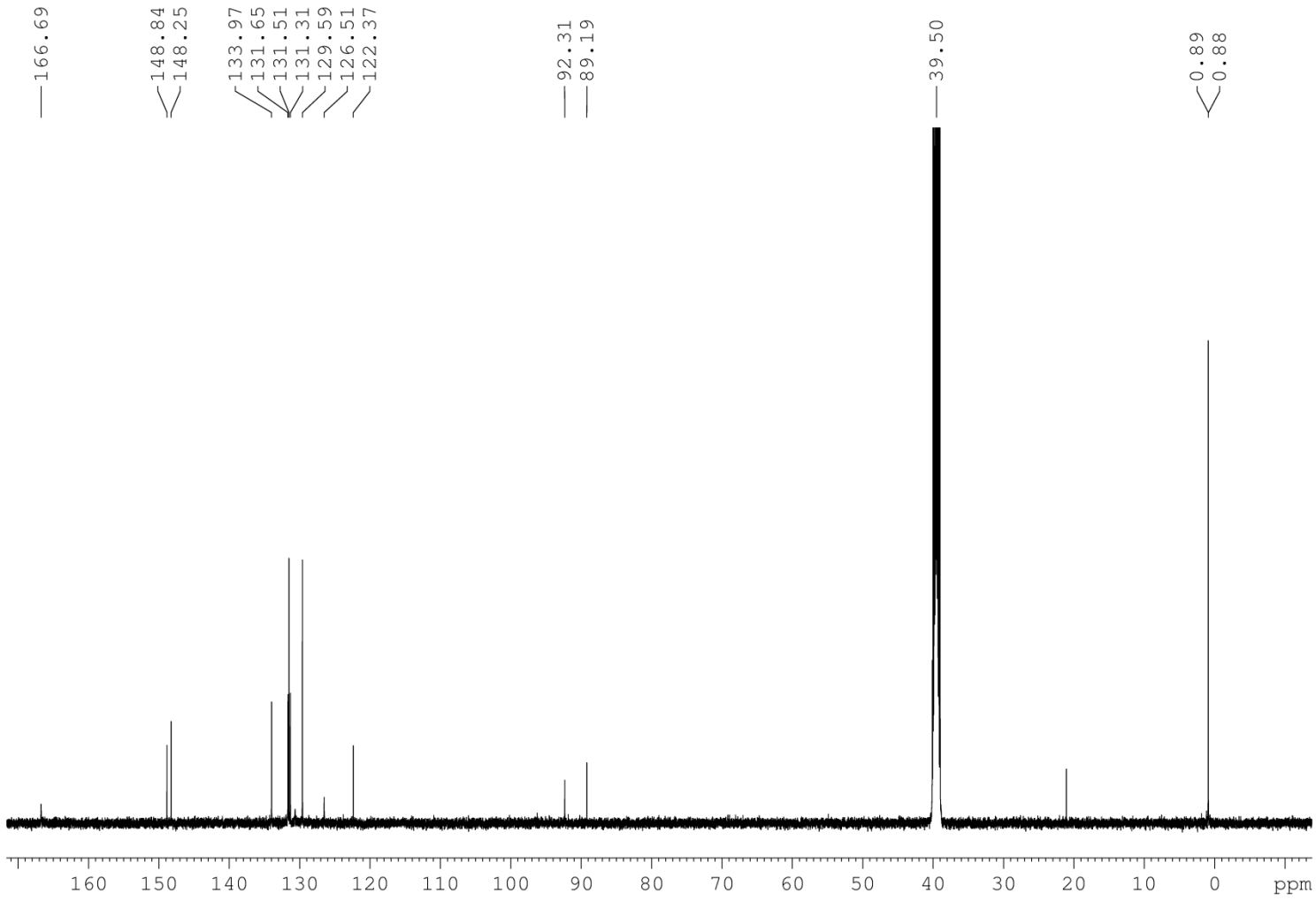

Figure S10 ^{13}C NMR spectrum of compound **6**.

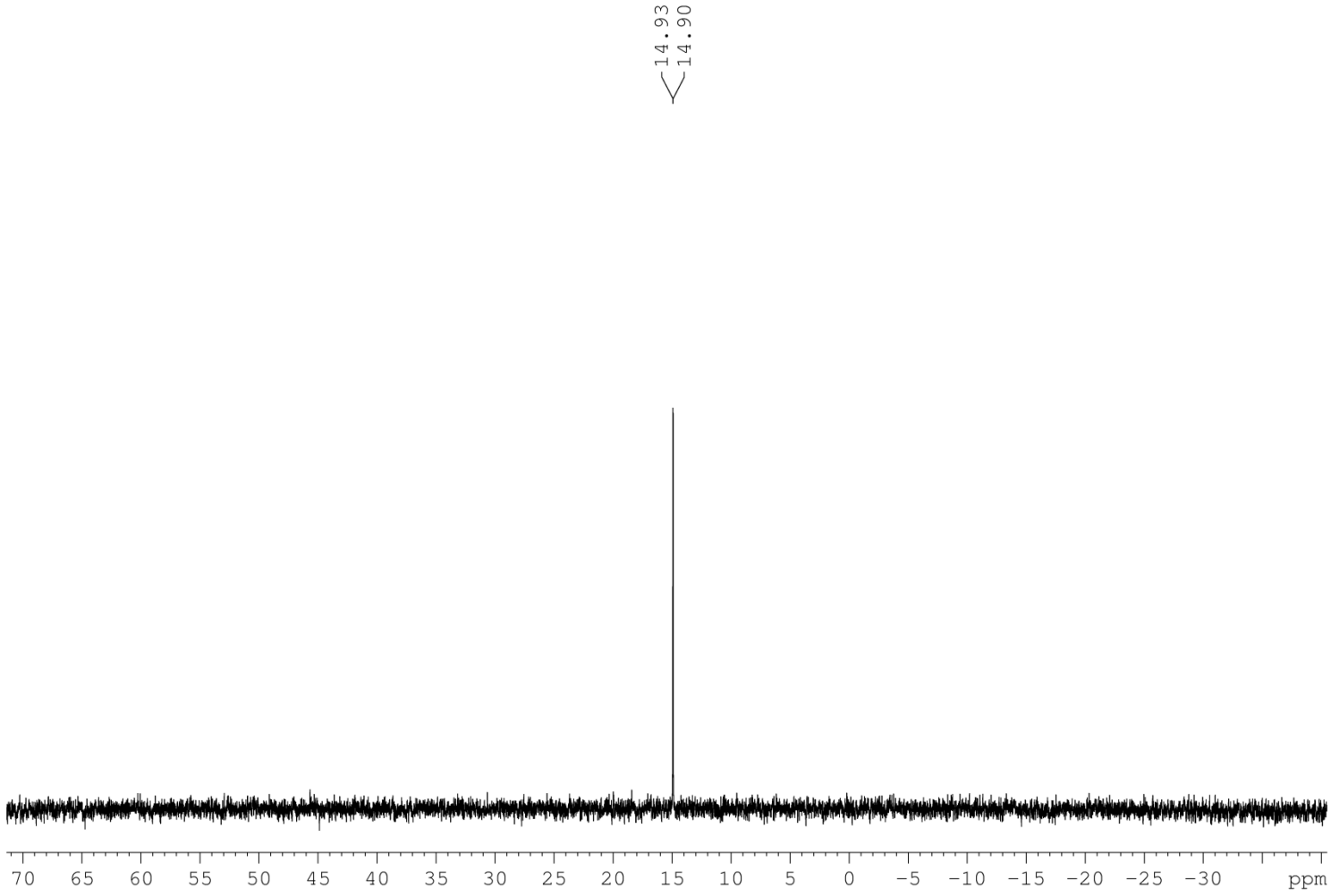

Figure S11 ^{29}Si NMR spectrum of compound **6**.

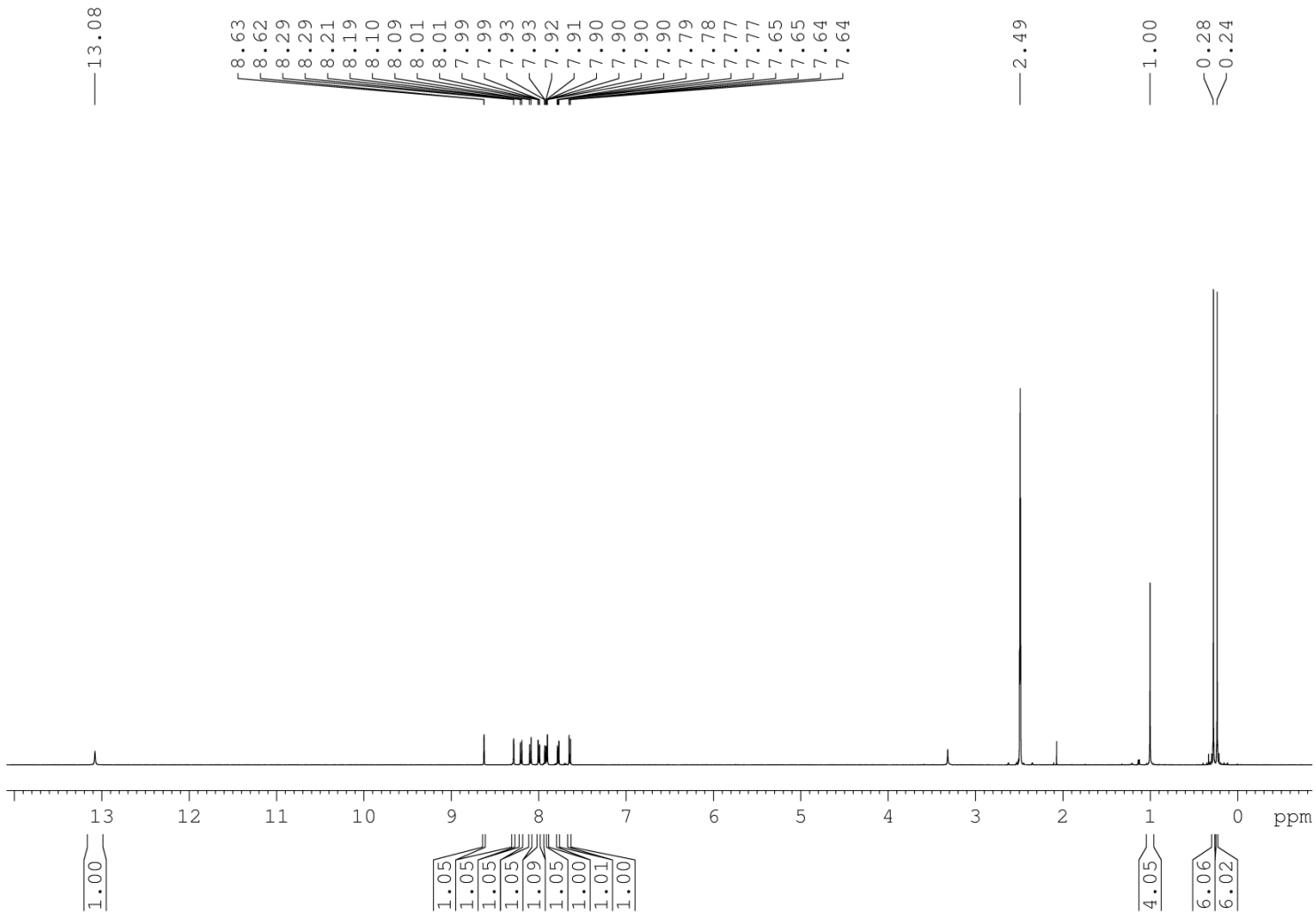

Figure S12 ^{1}H NMR spectrum of compound **7b**.

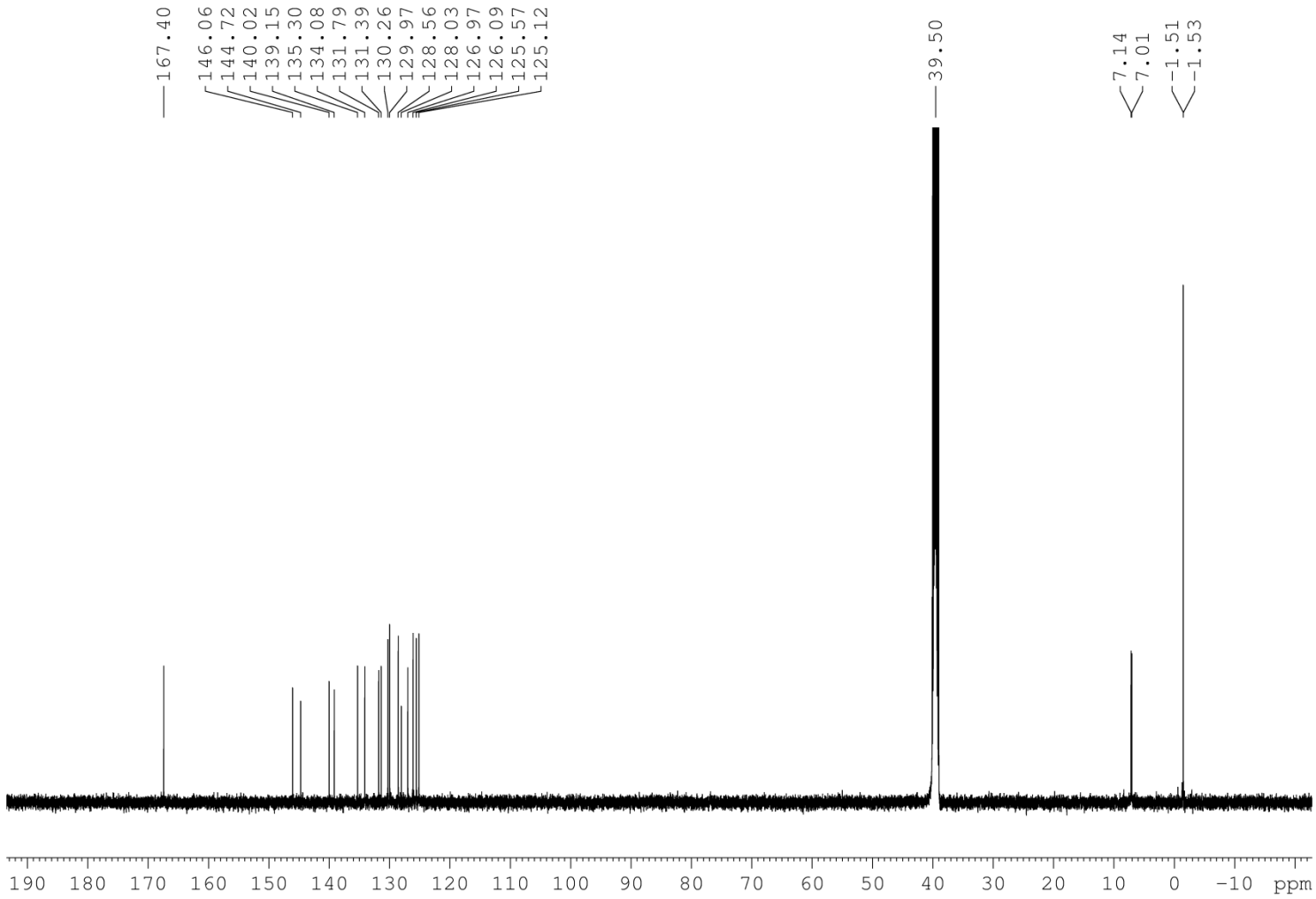

Figure S13 ^{13}C NMR spectrum of compound **7b**.

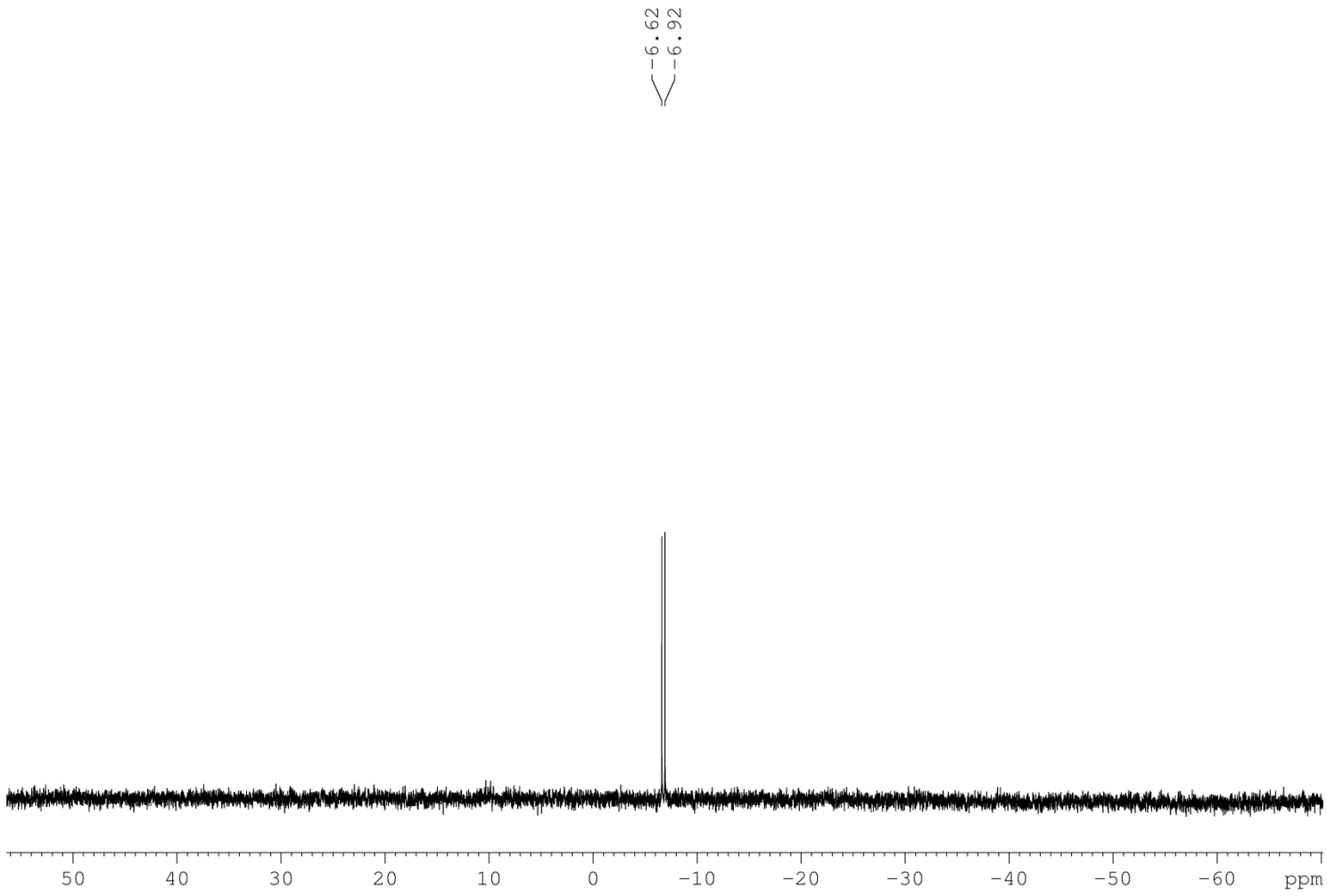

Figure S14 ^{29}Si NMR spectrum of compound **7b**.

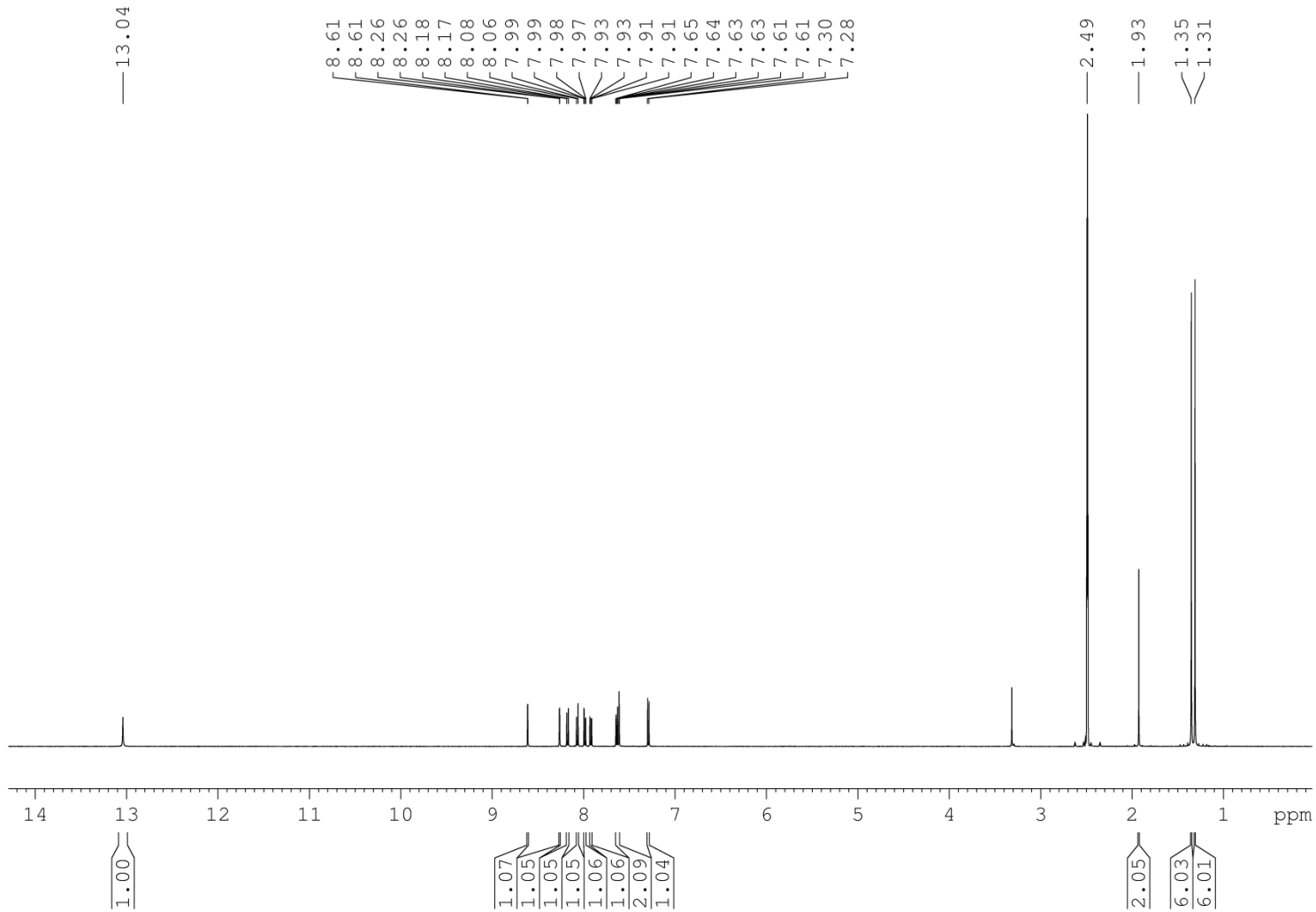

Figure S15 ^{1}H NMR spectrum of compound **8a**.

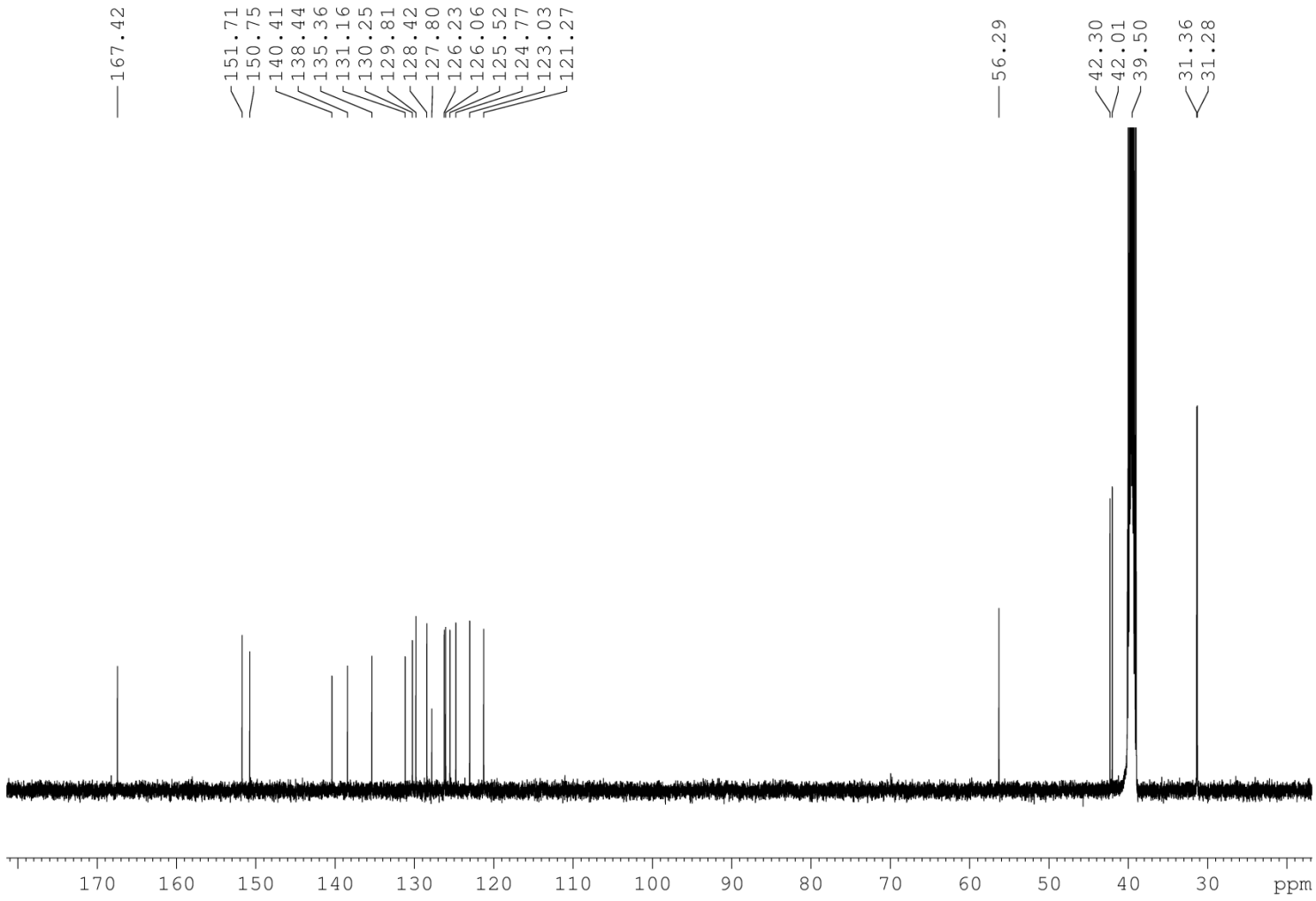

Figure S16 ^{13}C NMR spectrum of compound **8a**.

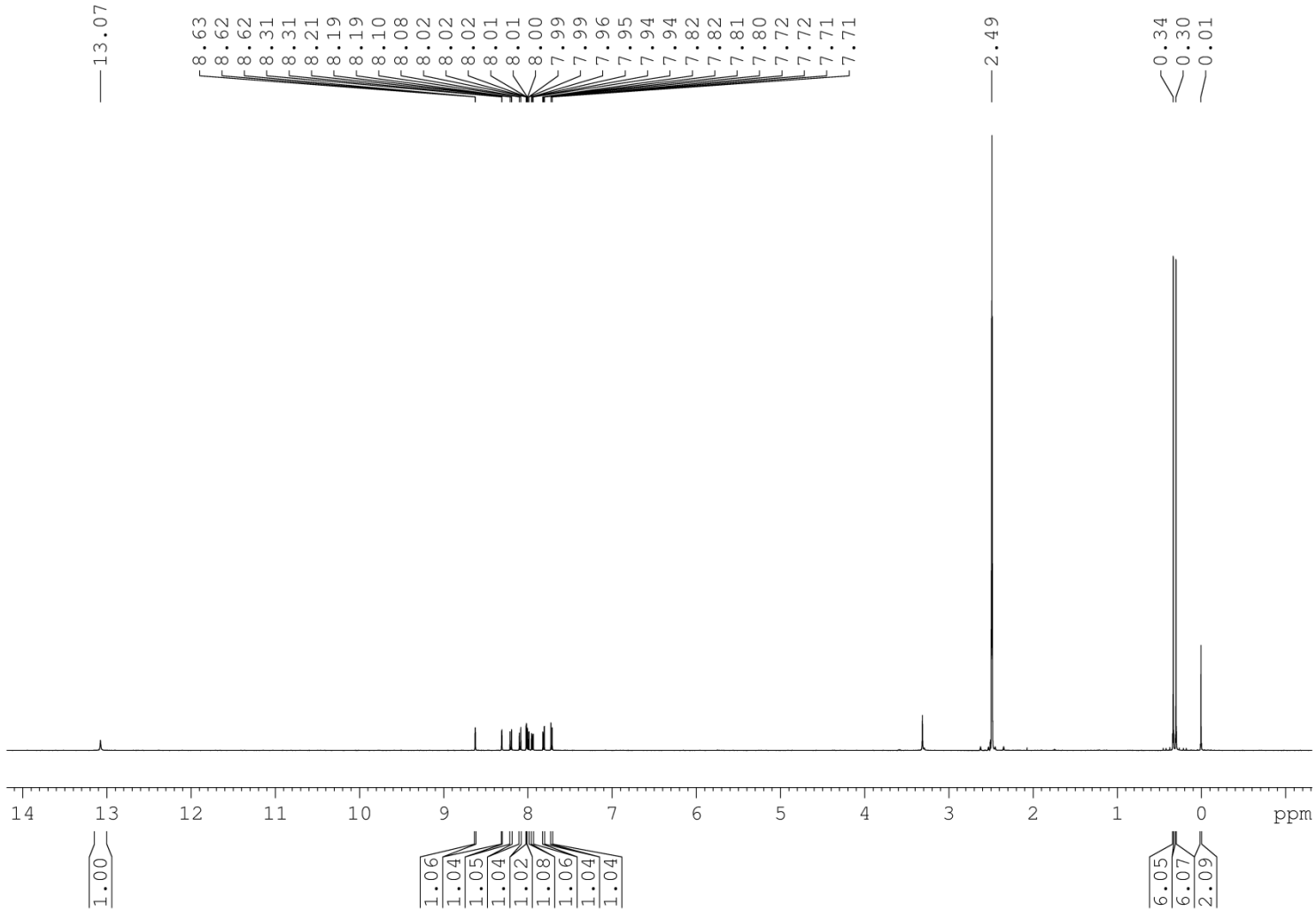

Figure S17 ^{1}H NMR spectrum of compound **8b**.

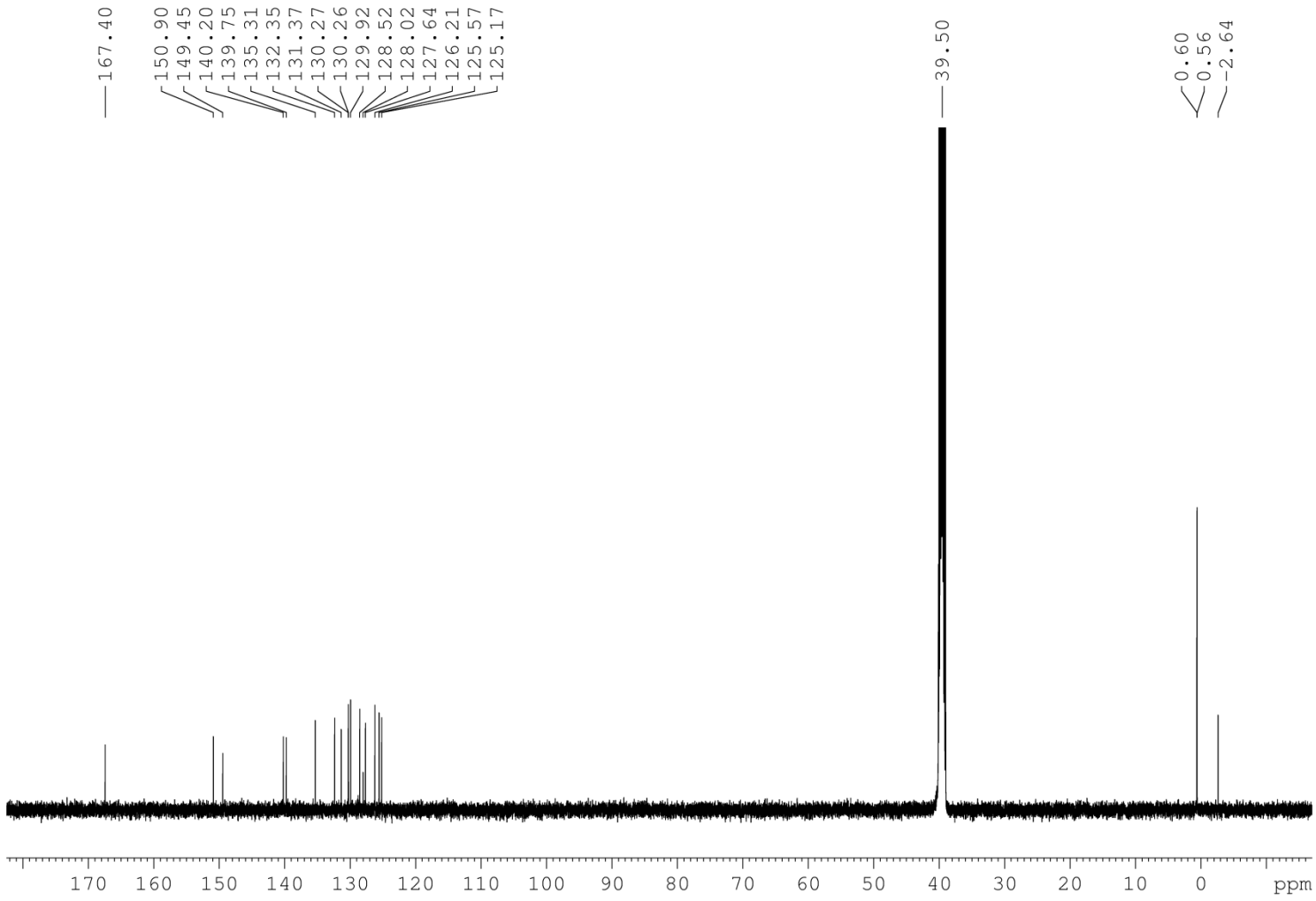

Figure S18 ^{13}C NMR spectrum of compound **8b**.

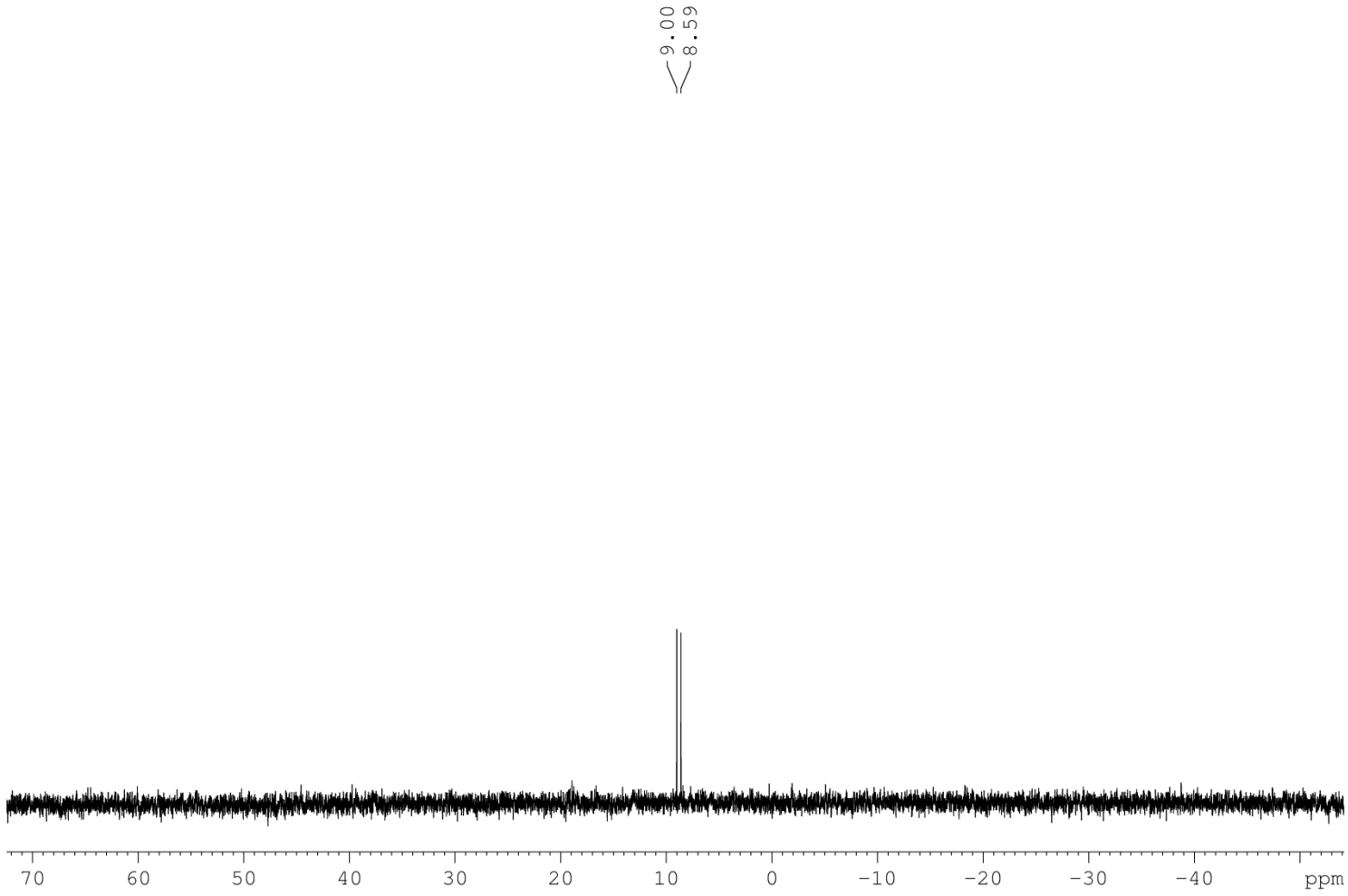

Figure S19 ^{29}Si NMR spectrum of compound **8b**.

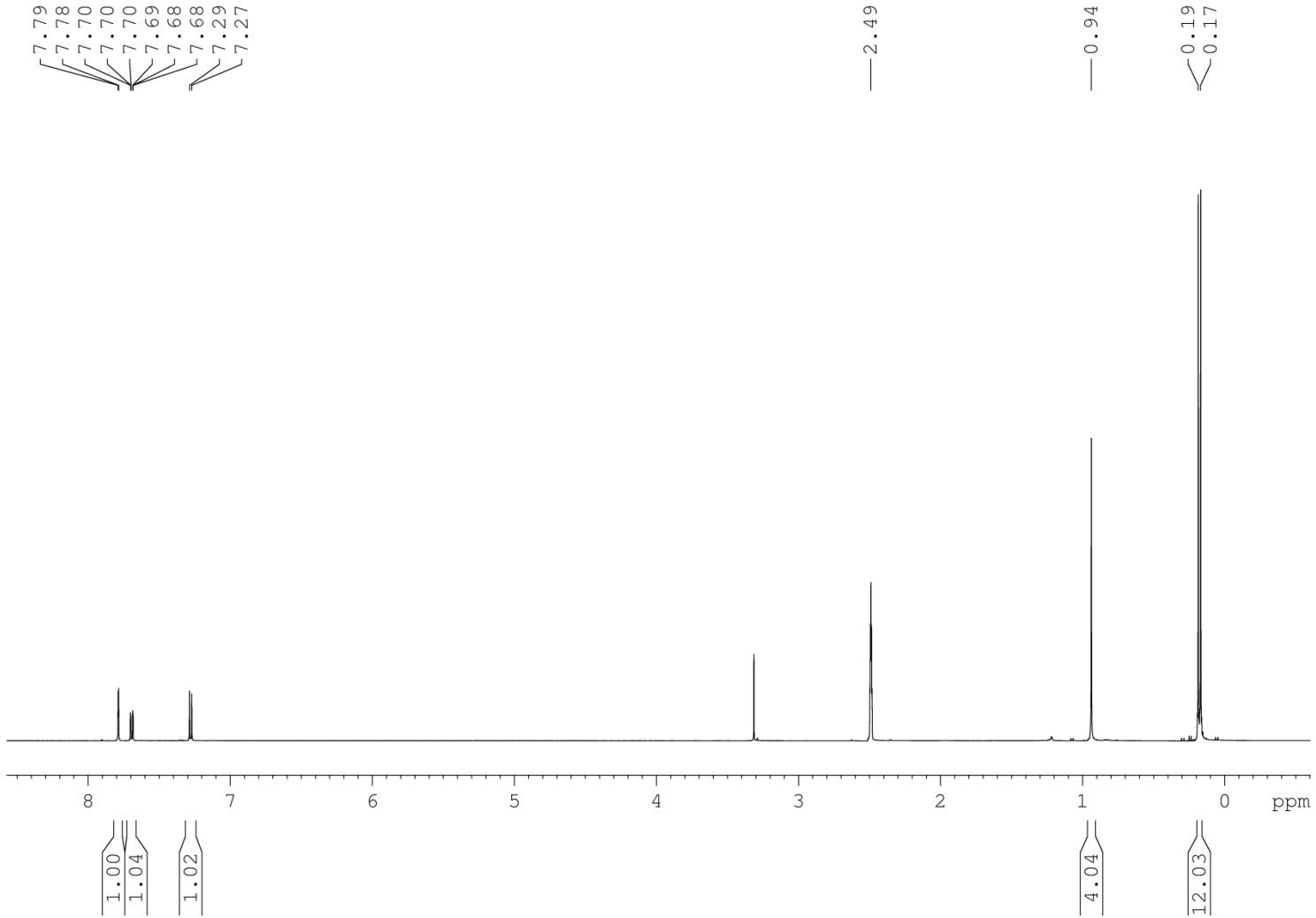

Figure S20 ^{1}H NMR spectrum of compound **9**.

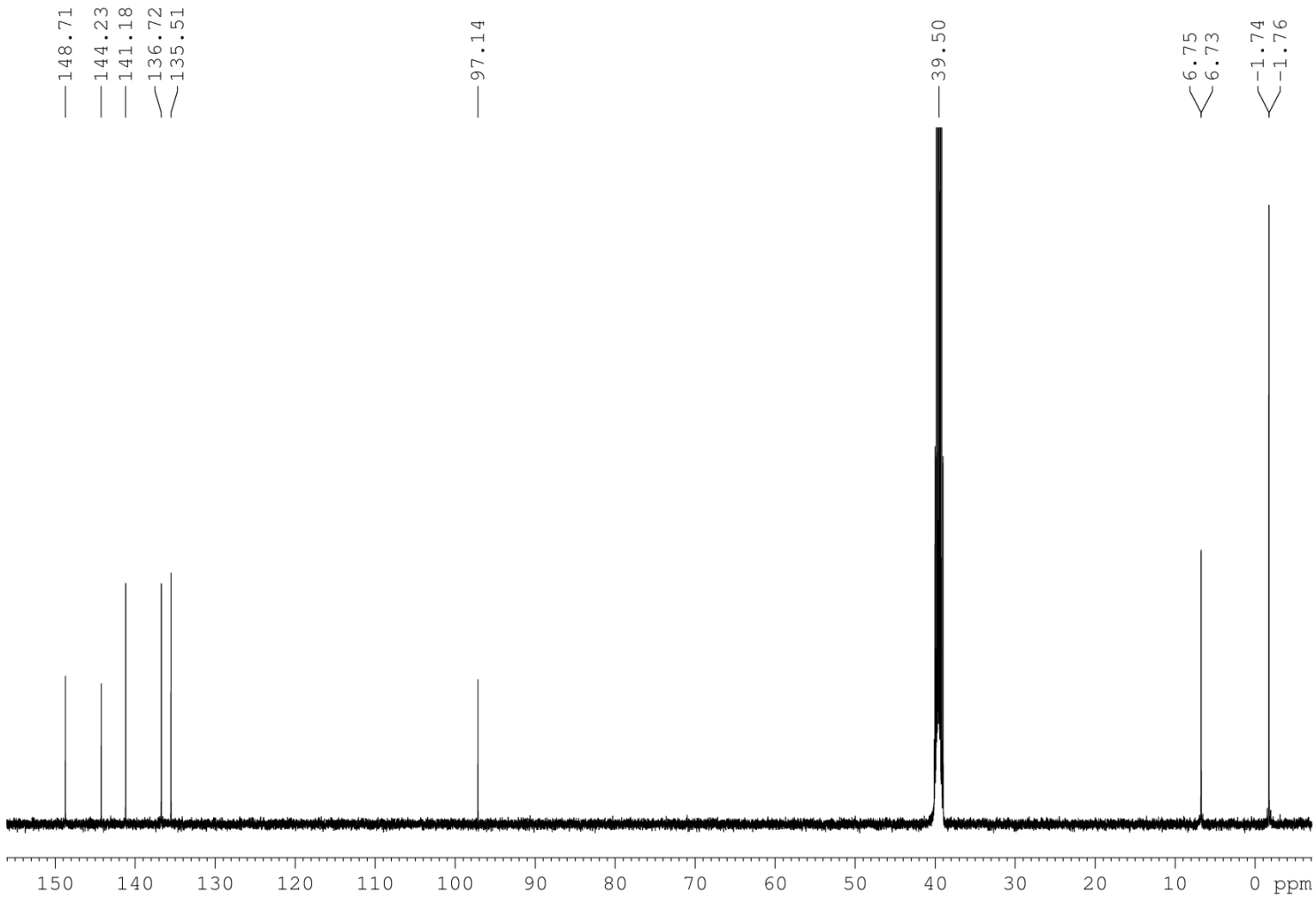

Figure S21 ^{13}C NMR spectrum of compound **9**.

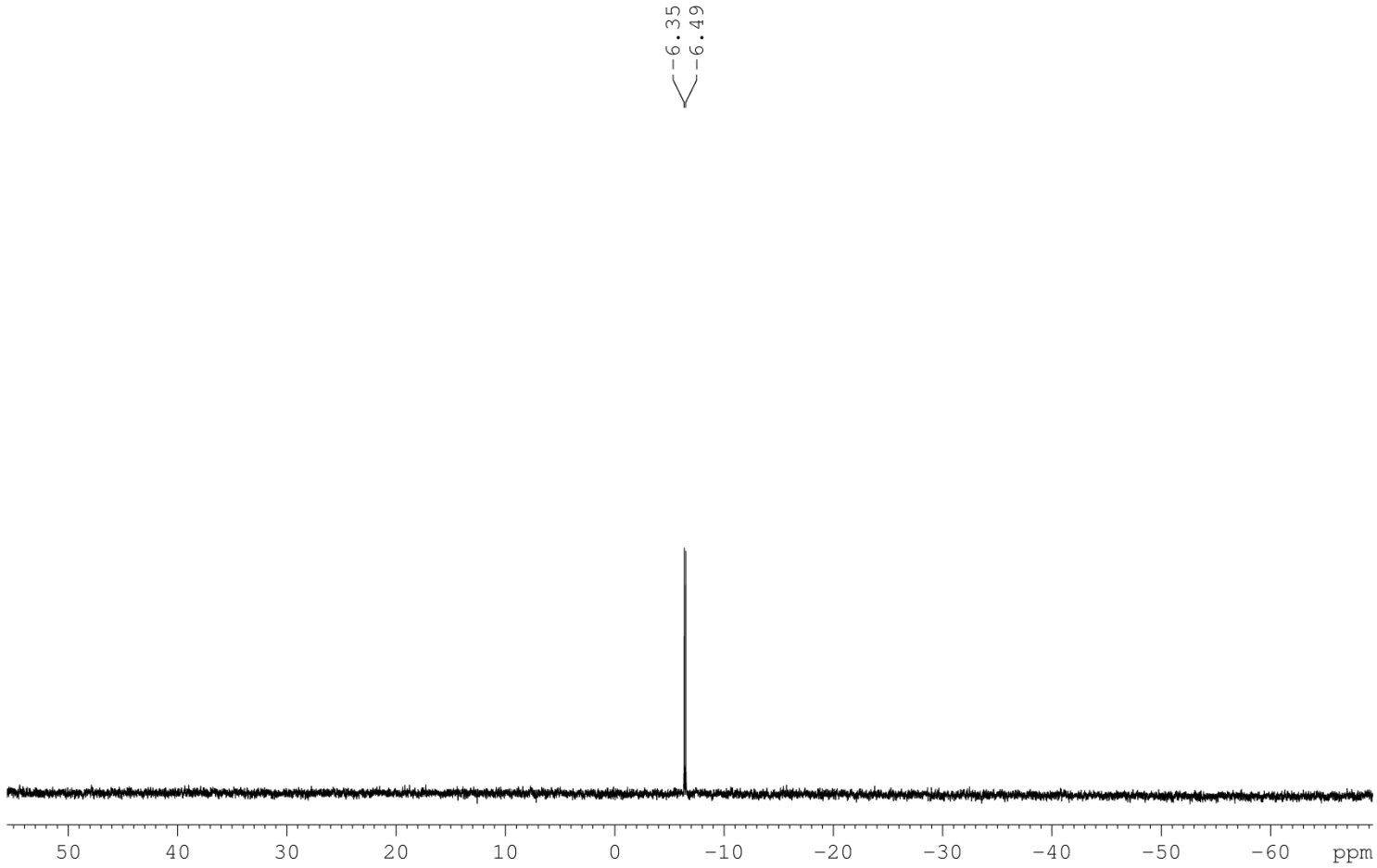

Figure S22 ^{29}Si NMR spectrum of compound **9**.

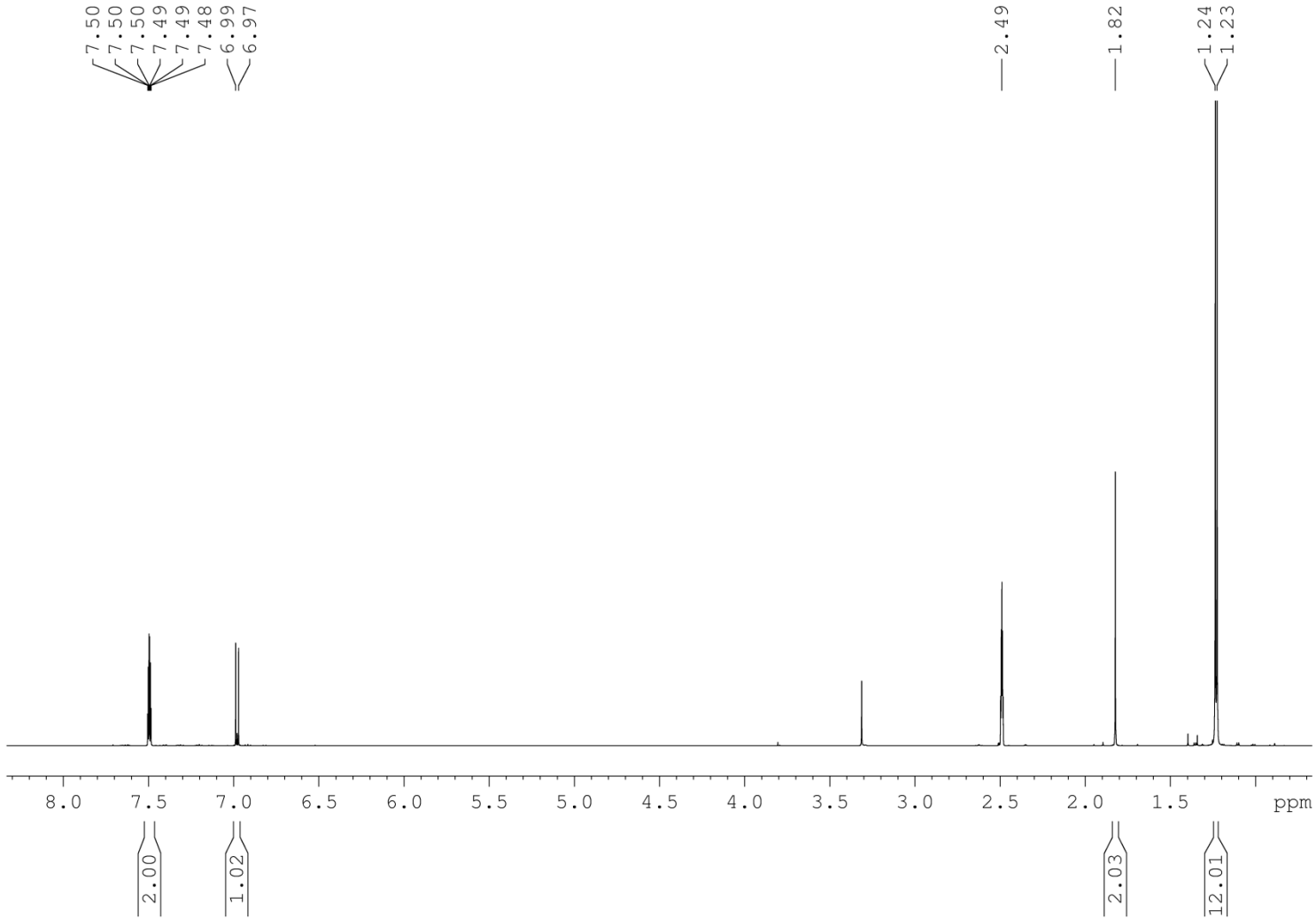

Figure S23 ^{1}H NMR spectrum of compound **10**.

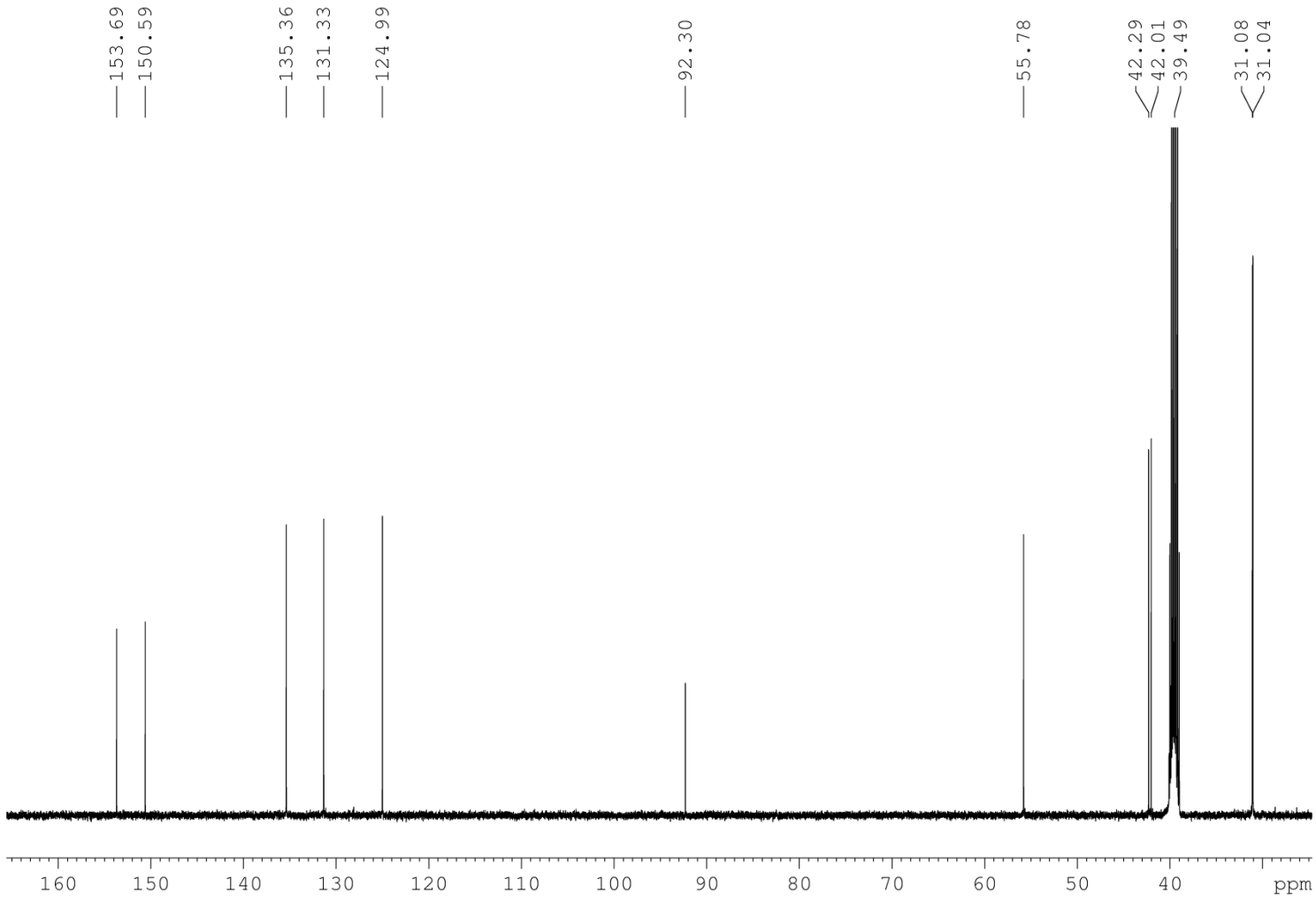

Figure S24 ^{13}C NMR spectrum of compound **10**.

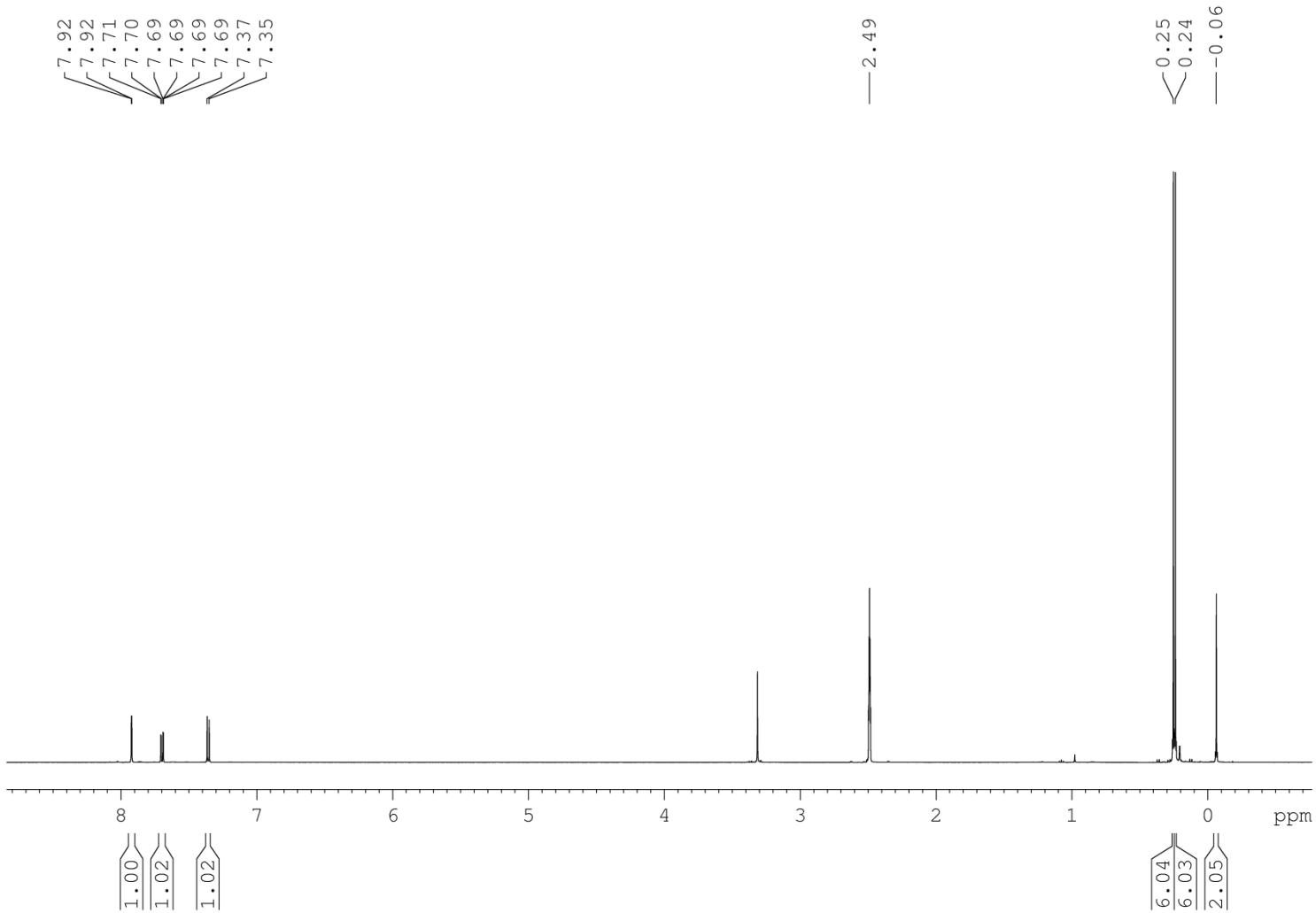

Figure S25 ^{1}H NMR spectrum of compound **11**.

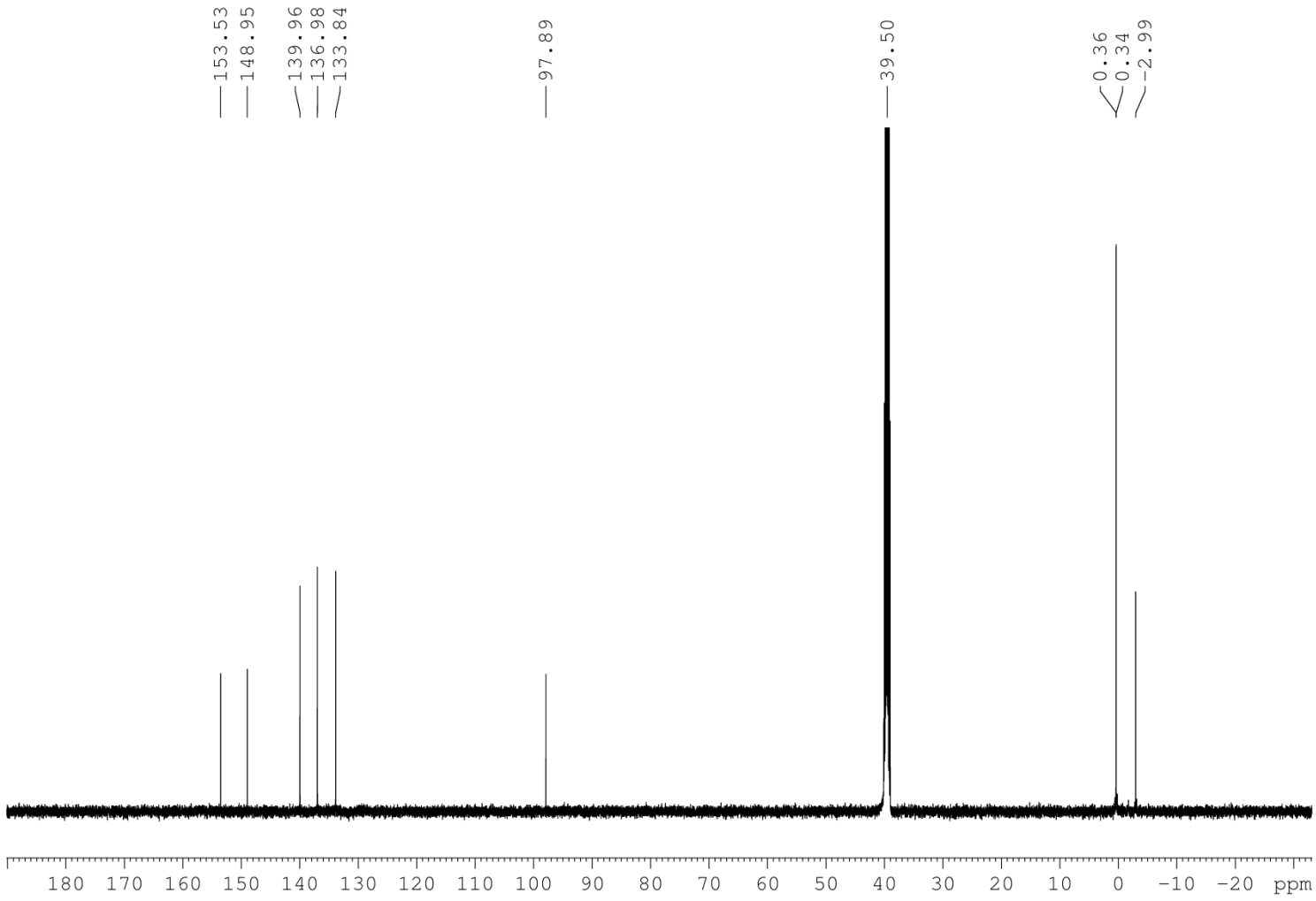

Figure S26 ^{13}C NMR spectrum of compound **11**.

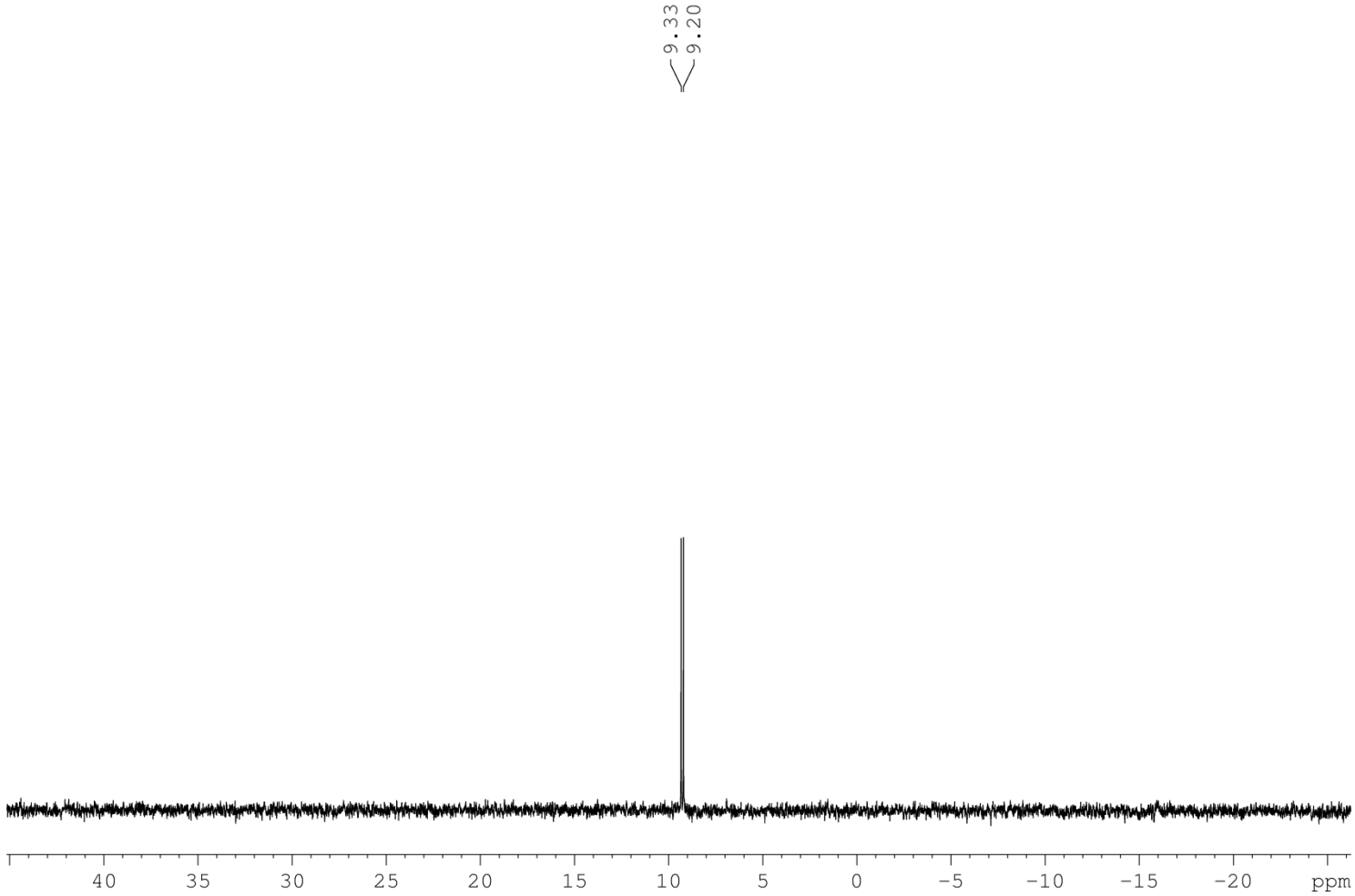

Figure S27 ^{29}Si NMR spectrum of compound **11**.

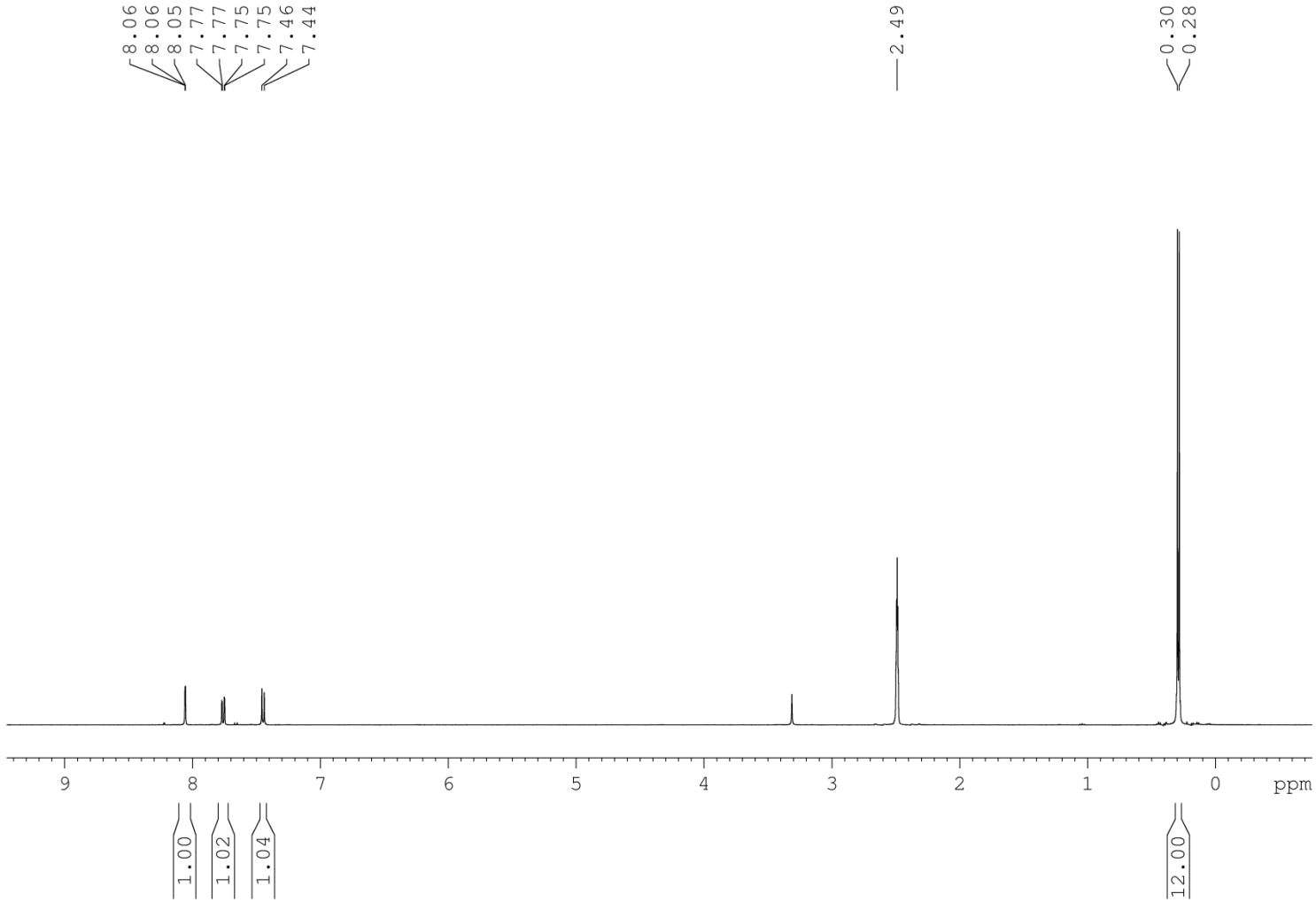

Figure S28 ^{1}H NMR spectrum of compound **12**.

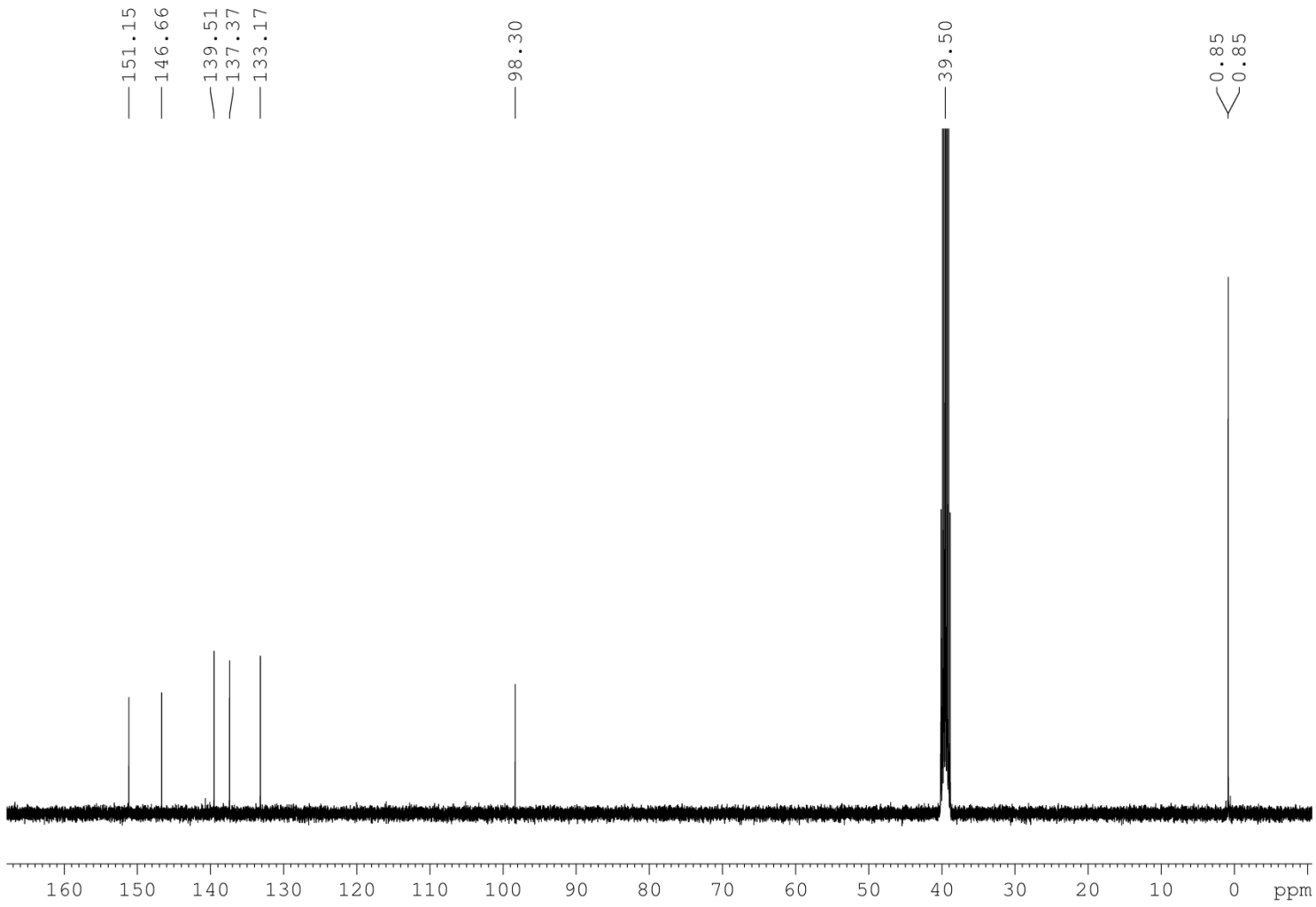

Figure S29 ^{13}C NMR spectrum of compound **12**.

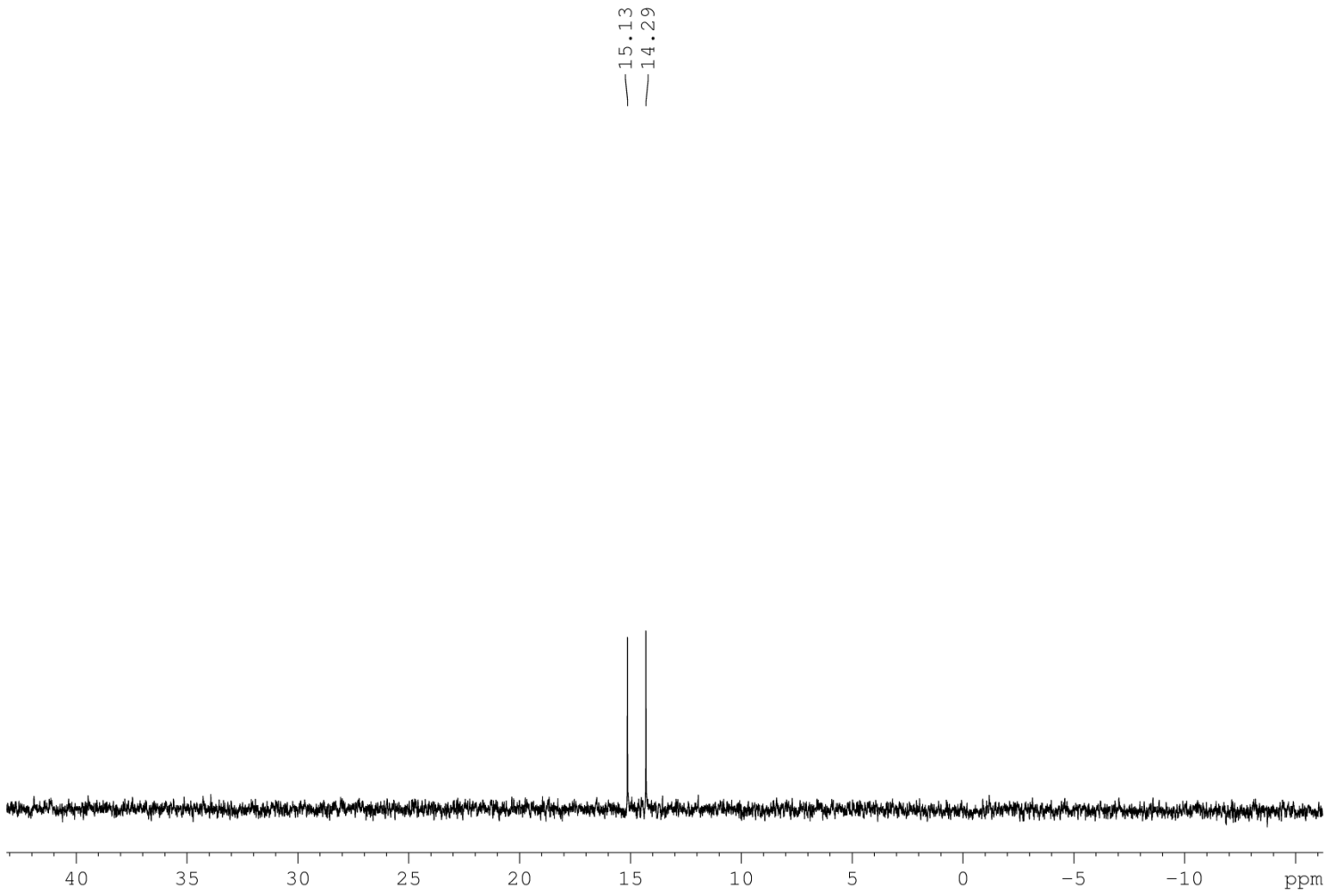

Figure S30 ^{29}Si NMR spectrum of compound **12**.

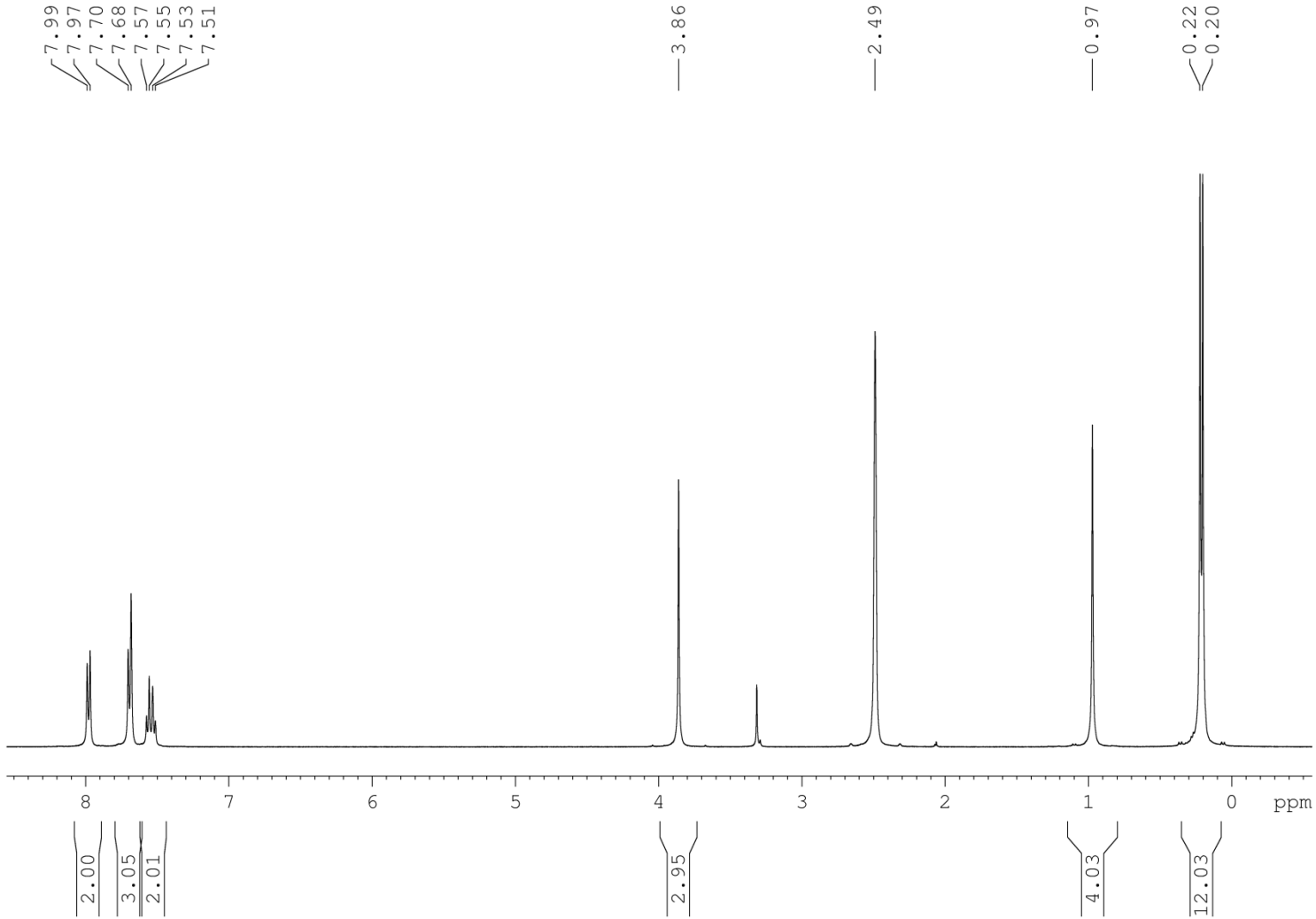

Figure S31 ^{1}H NMR spectrum of compound **14**.

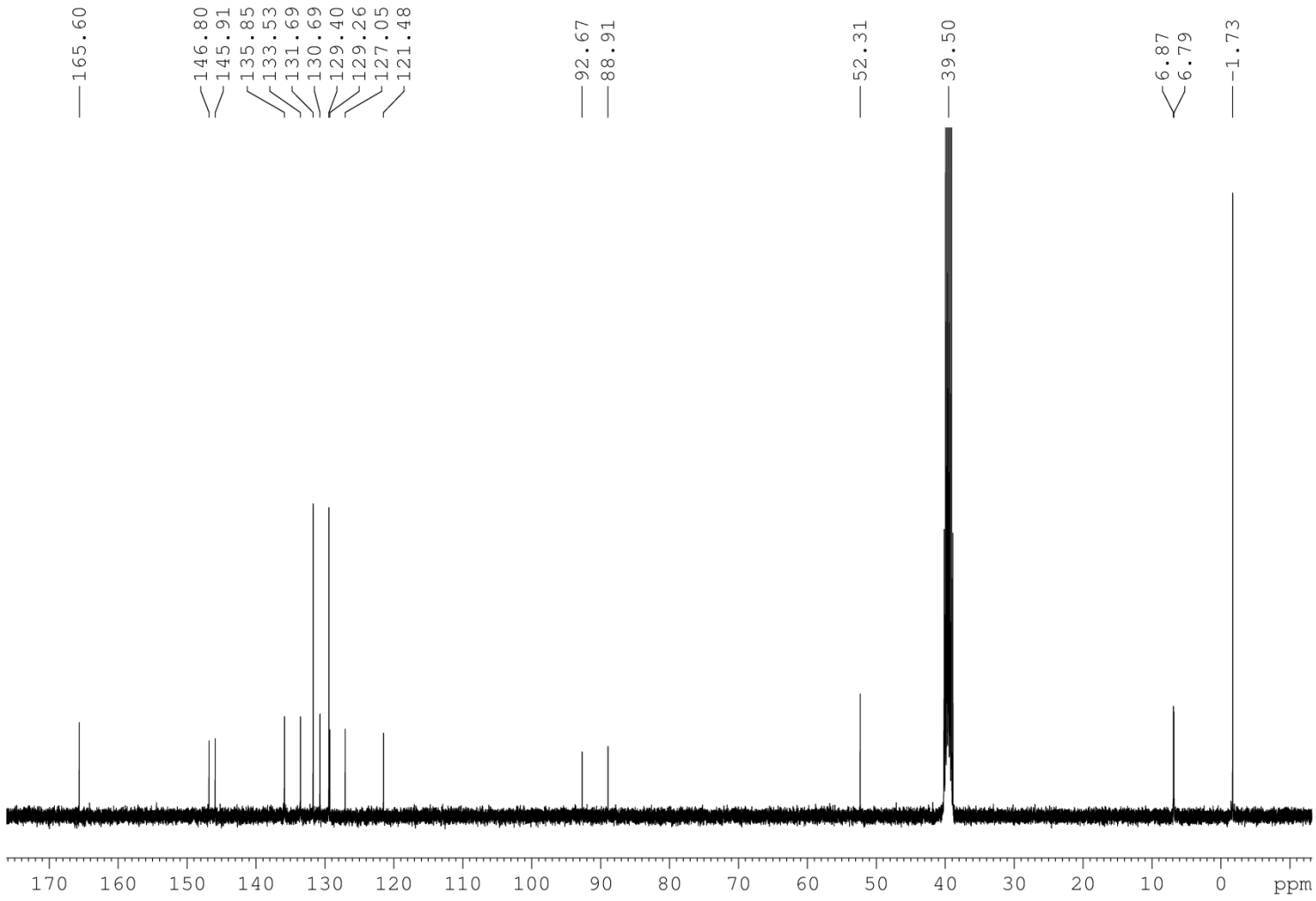

Figure S32 ^{13}C NMR spectrum of compound **14**.

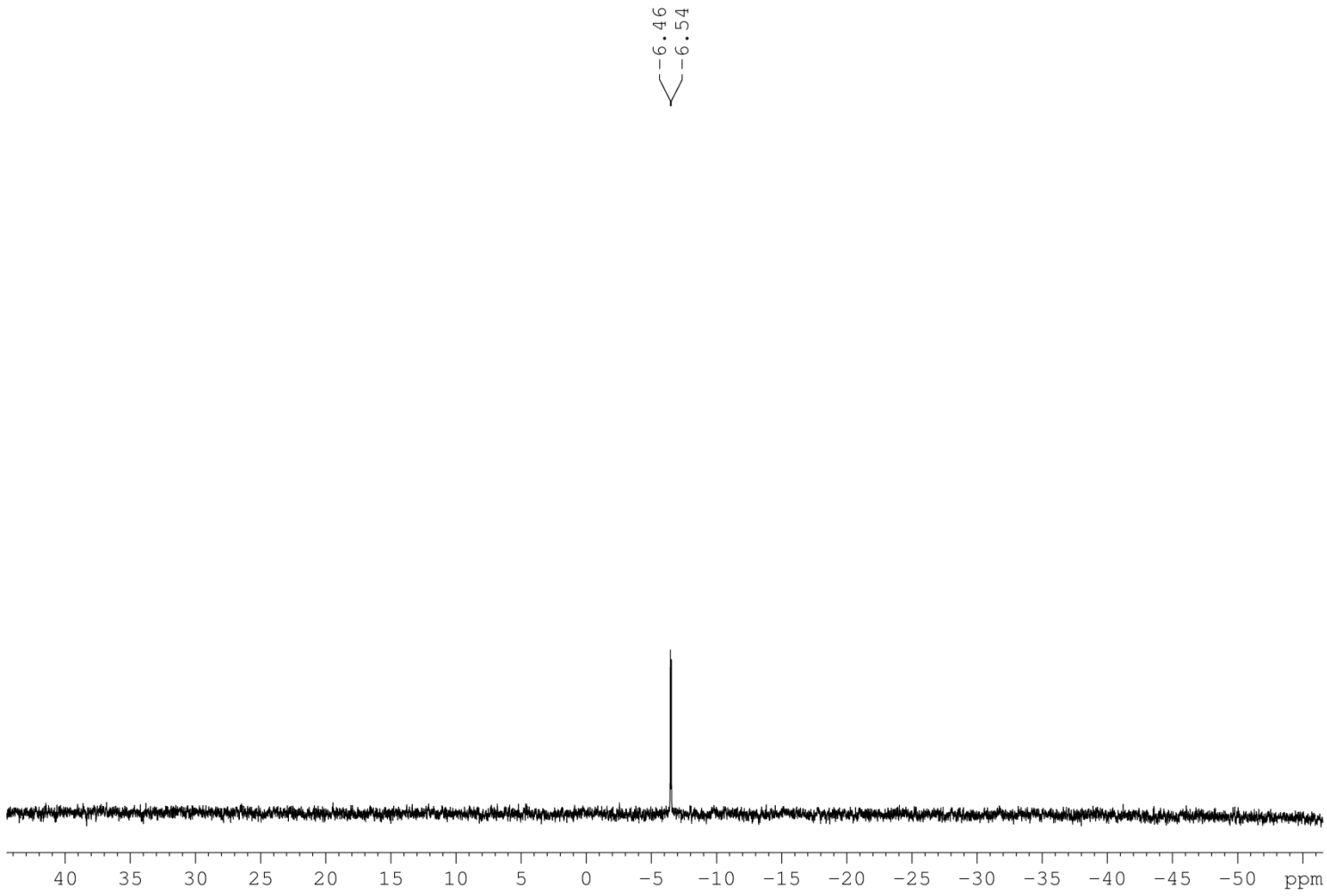

Figure S33 ^{29}Si NMR spectrum of compound **14**.

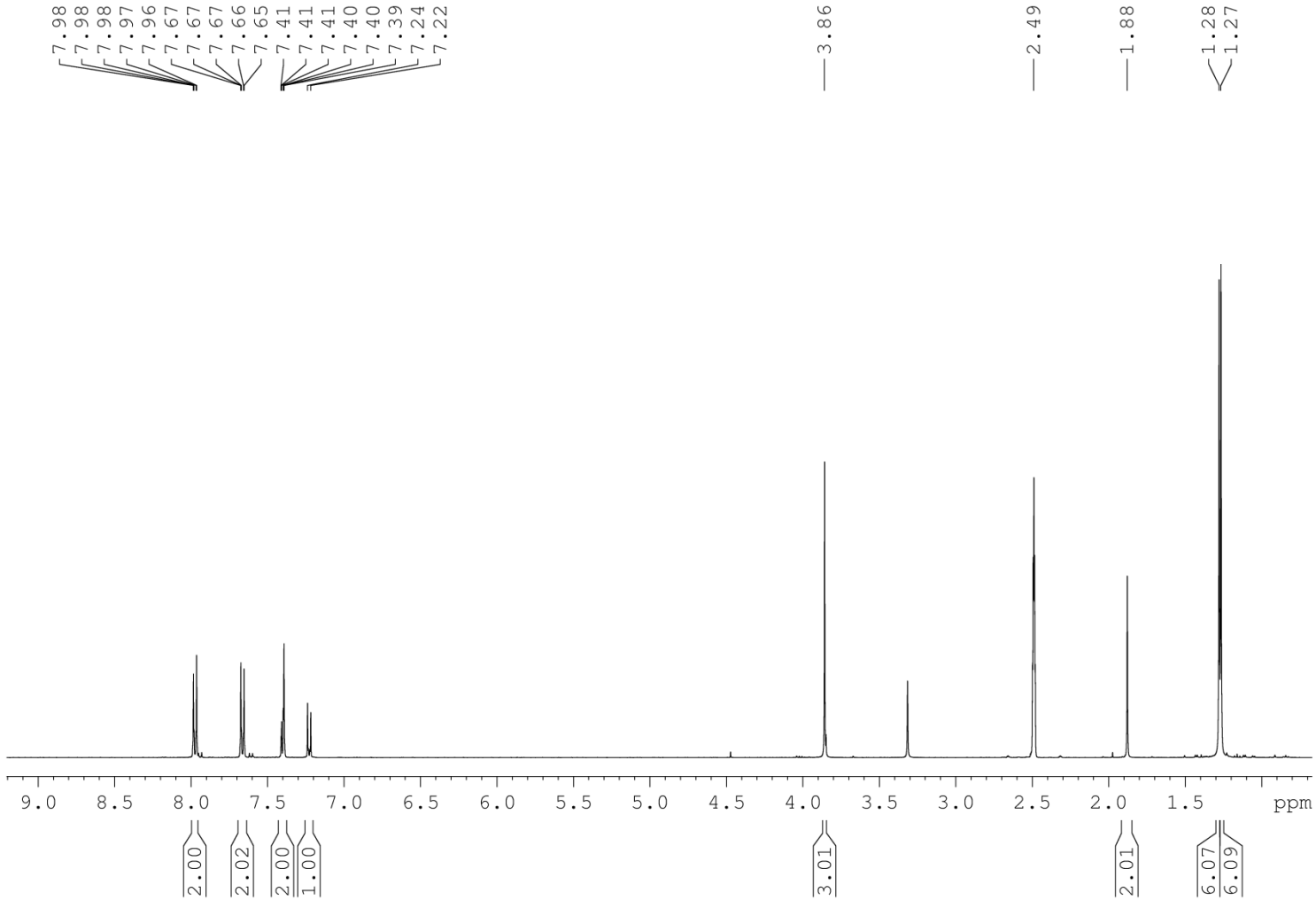

Figure S34 ^{1}H NMR spectrum of compound **15**.

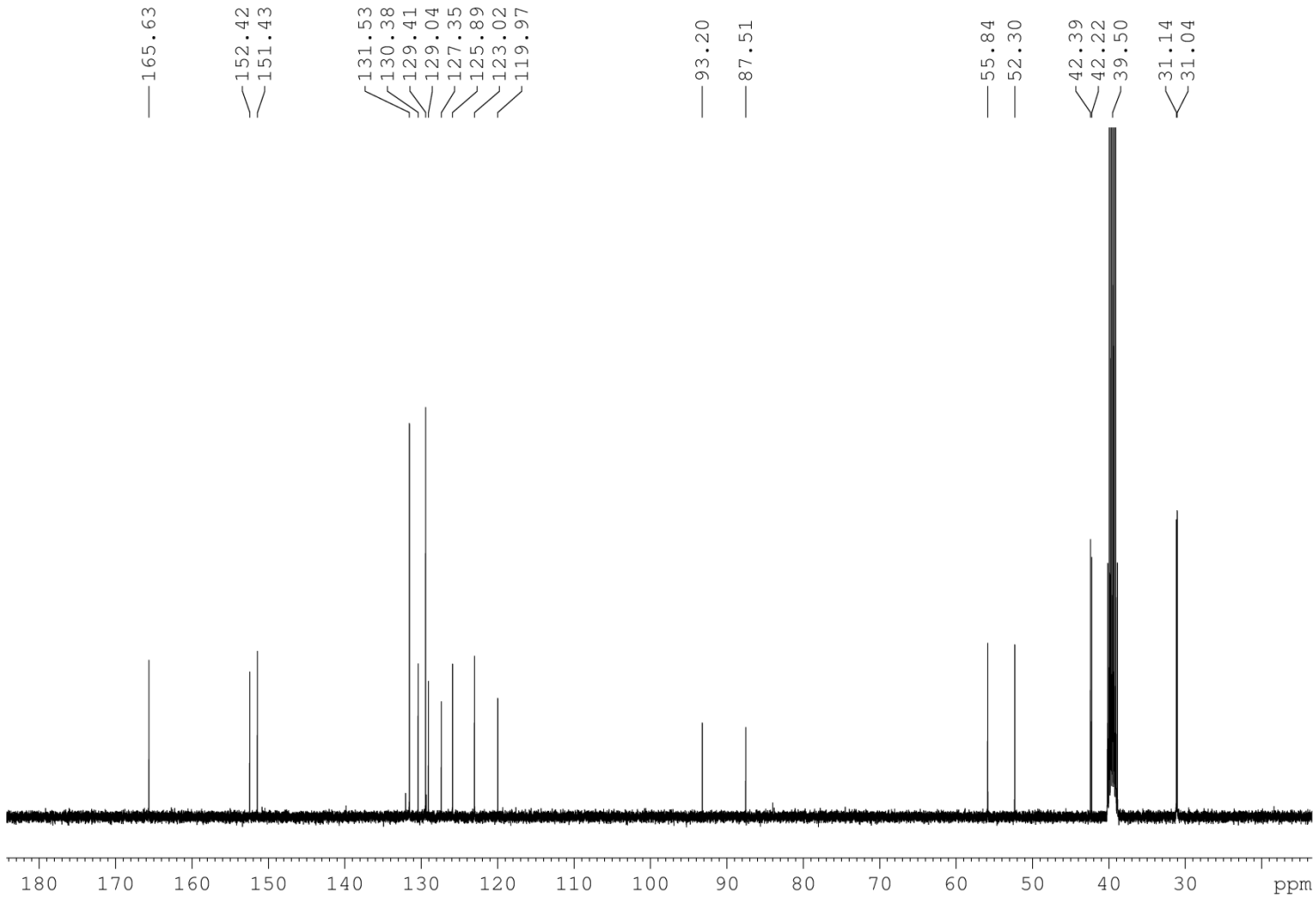

Figure S35 ^{13}C NMR spectrum of compound **15**.

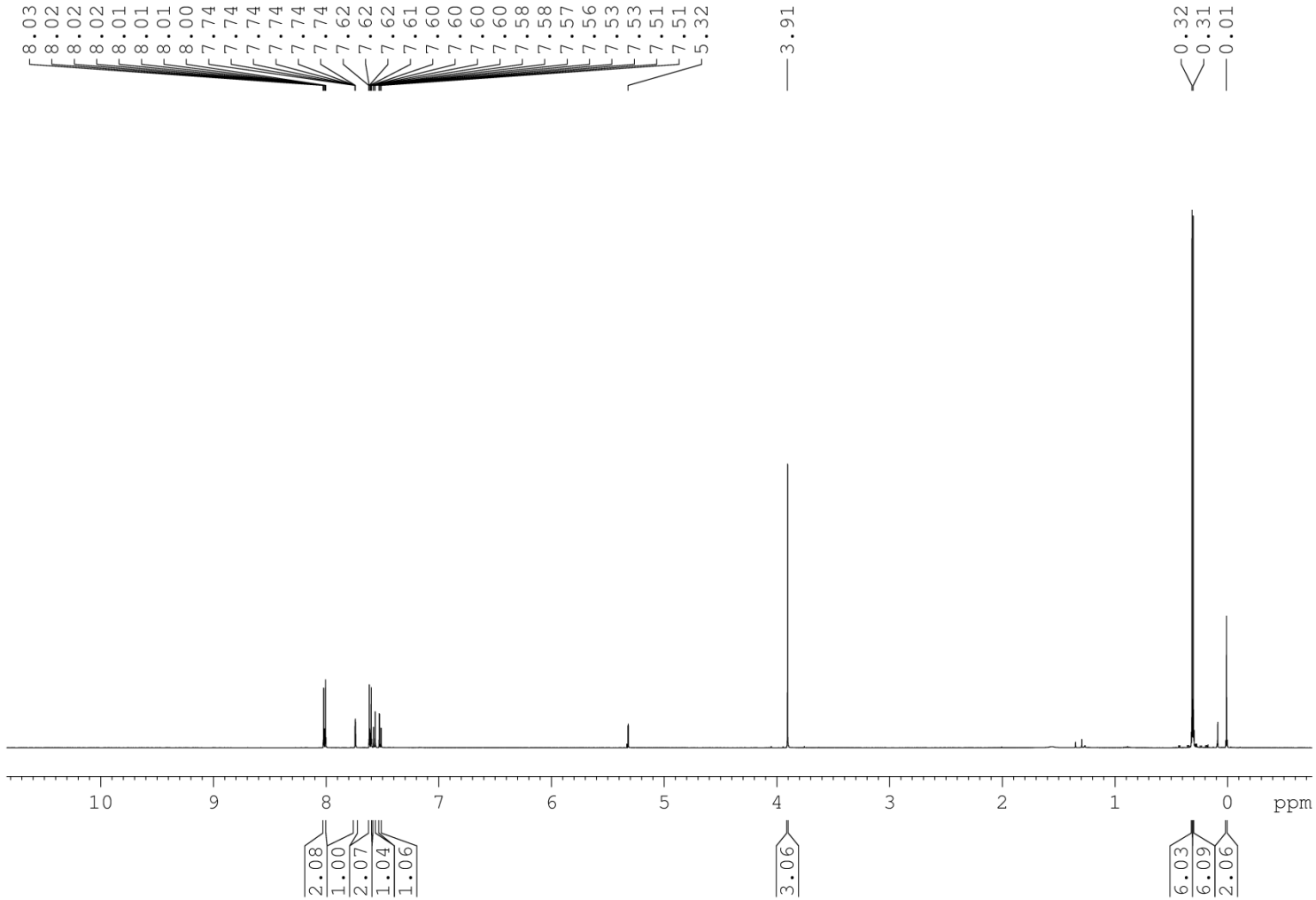

Figure S36 ^{1}H NMR spectrum of compound **16**.

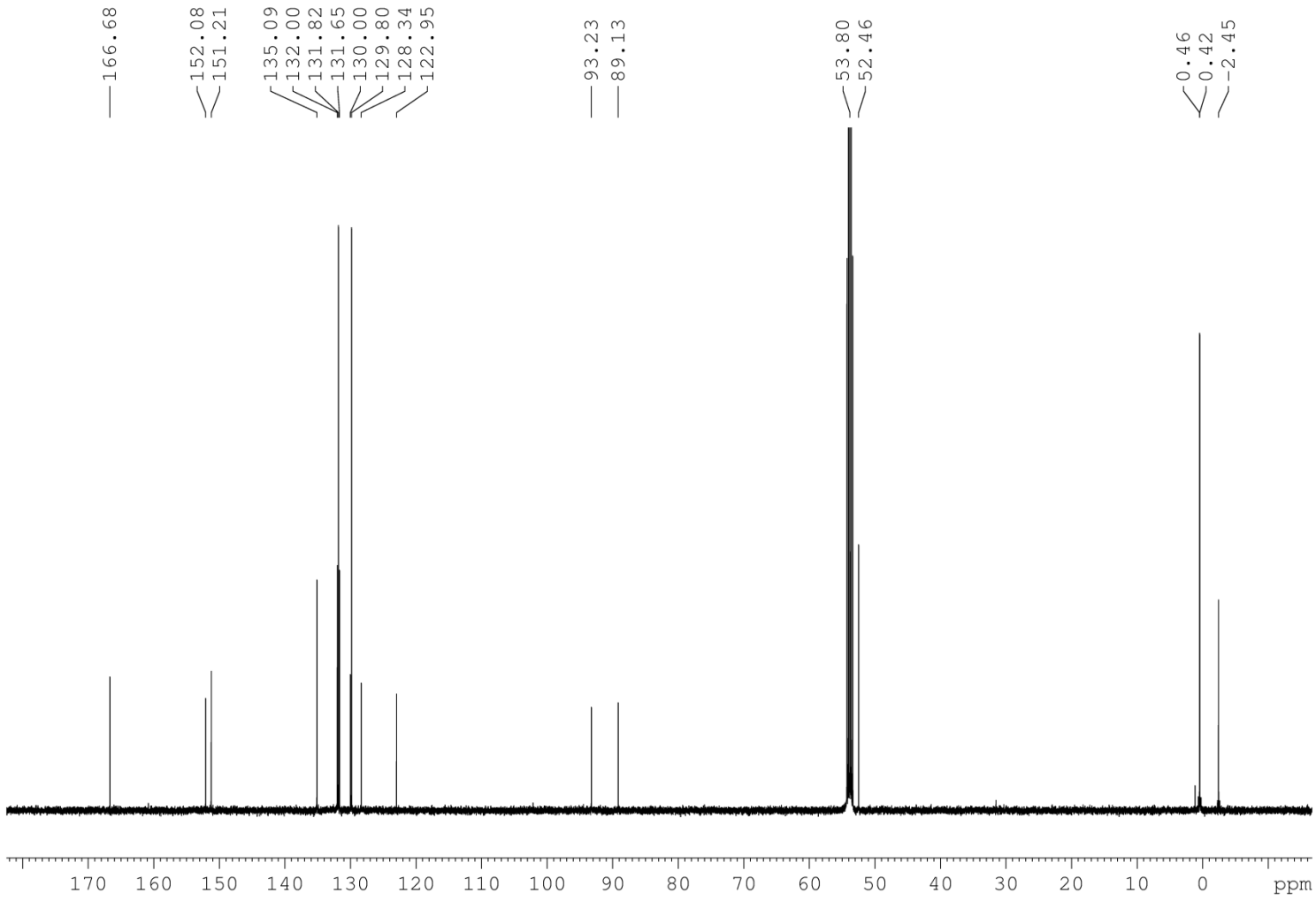

Figure S37 ^{13}C NMR spectrum of compound **16**.

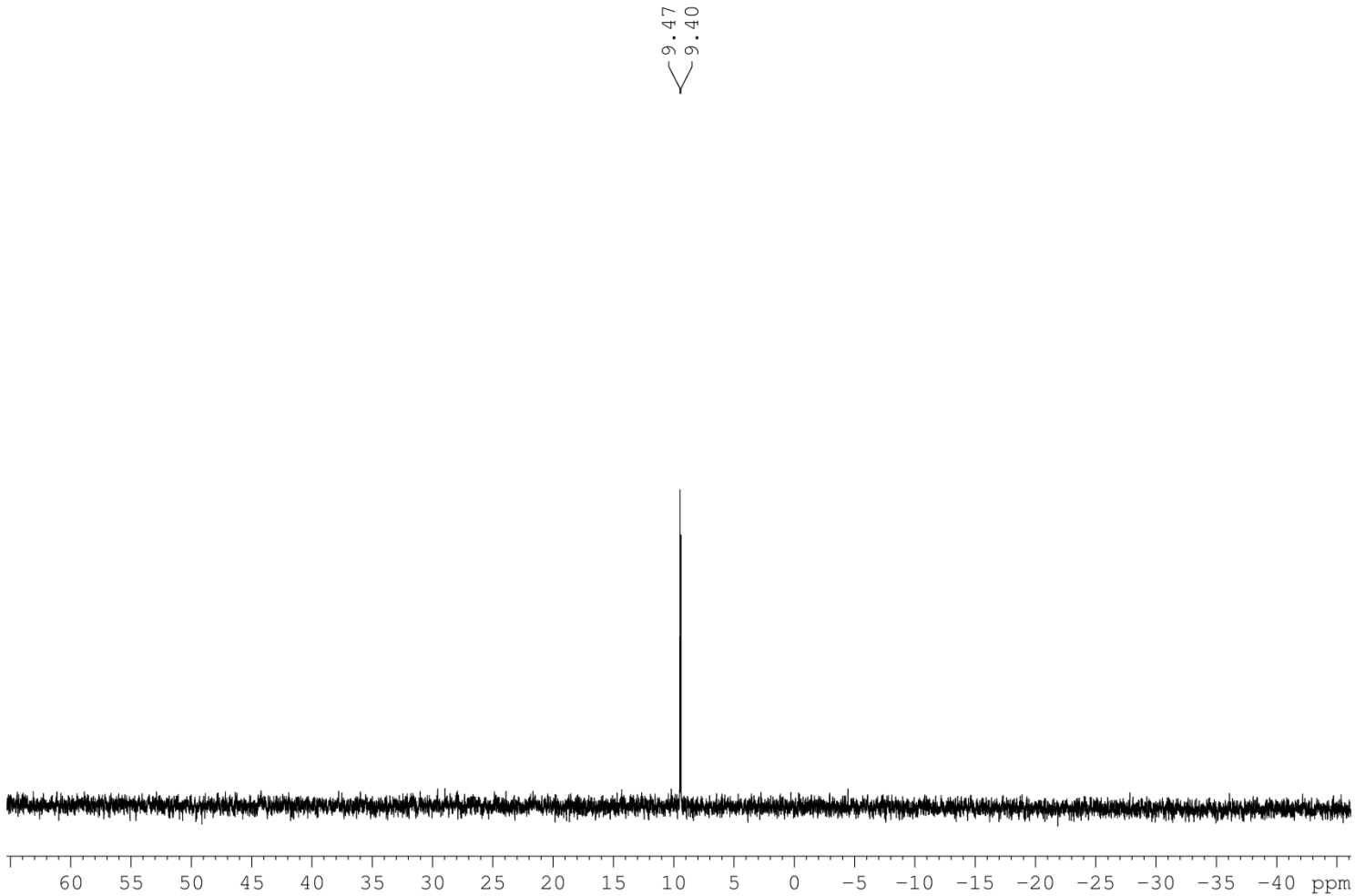

Figure S38 ^{29}Si NMR spectrum of compound **16**.

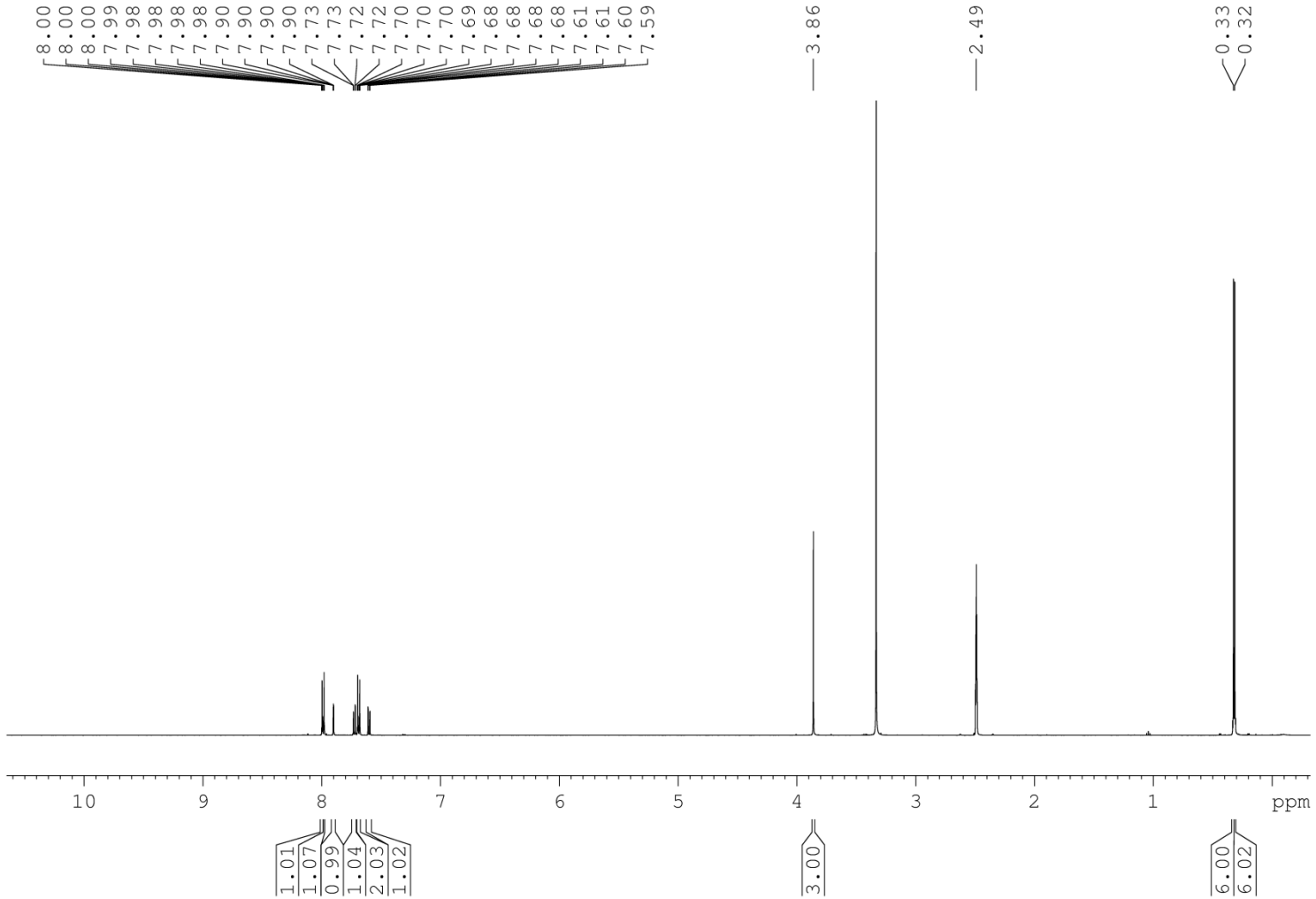

Figure S39 ^{1}H NMR spectrum of compound **17**.

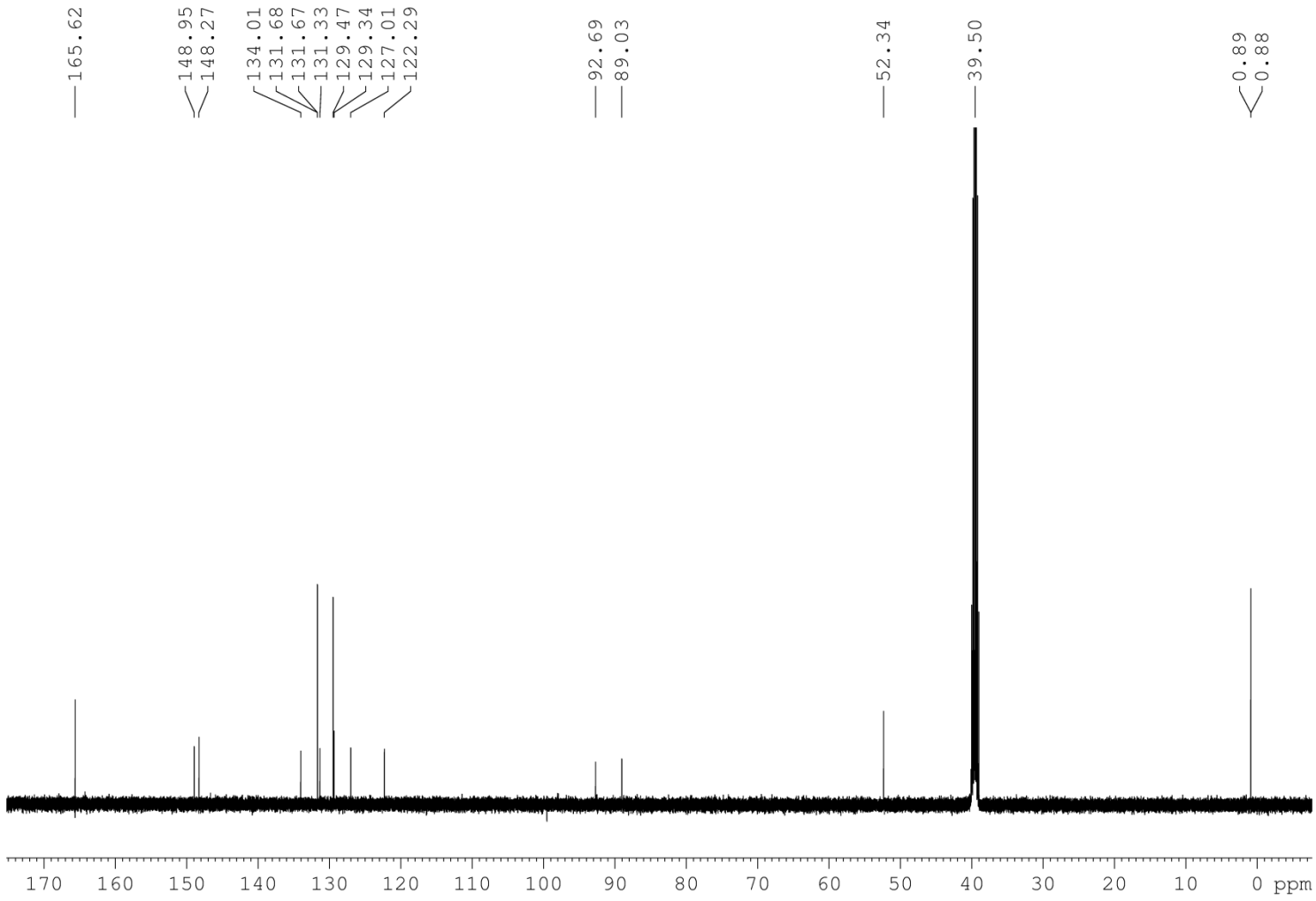

Figure S40 ^{13}C NMR spectrum of compound **17**.

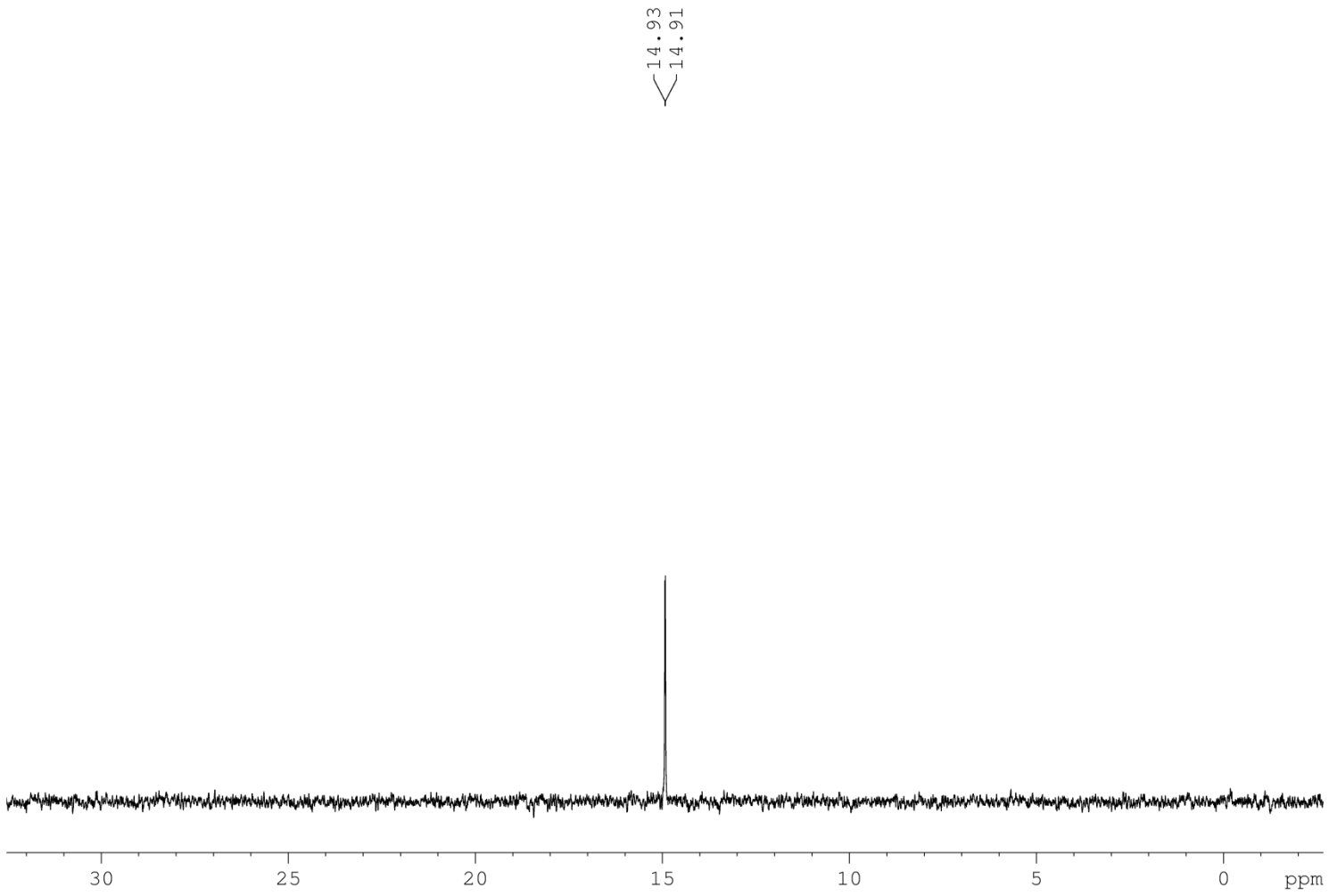

Figure S41 ^{29}Si NMR spectrum of compound **17**.

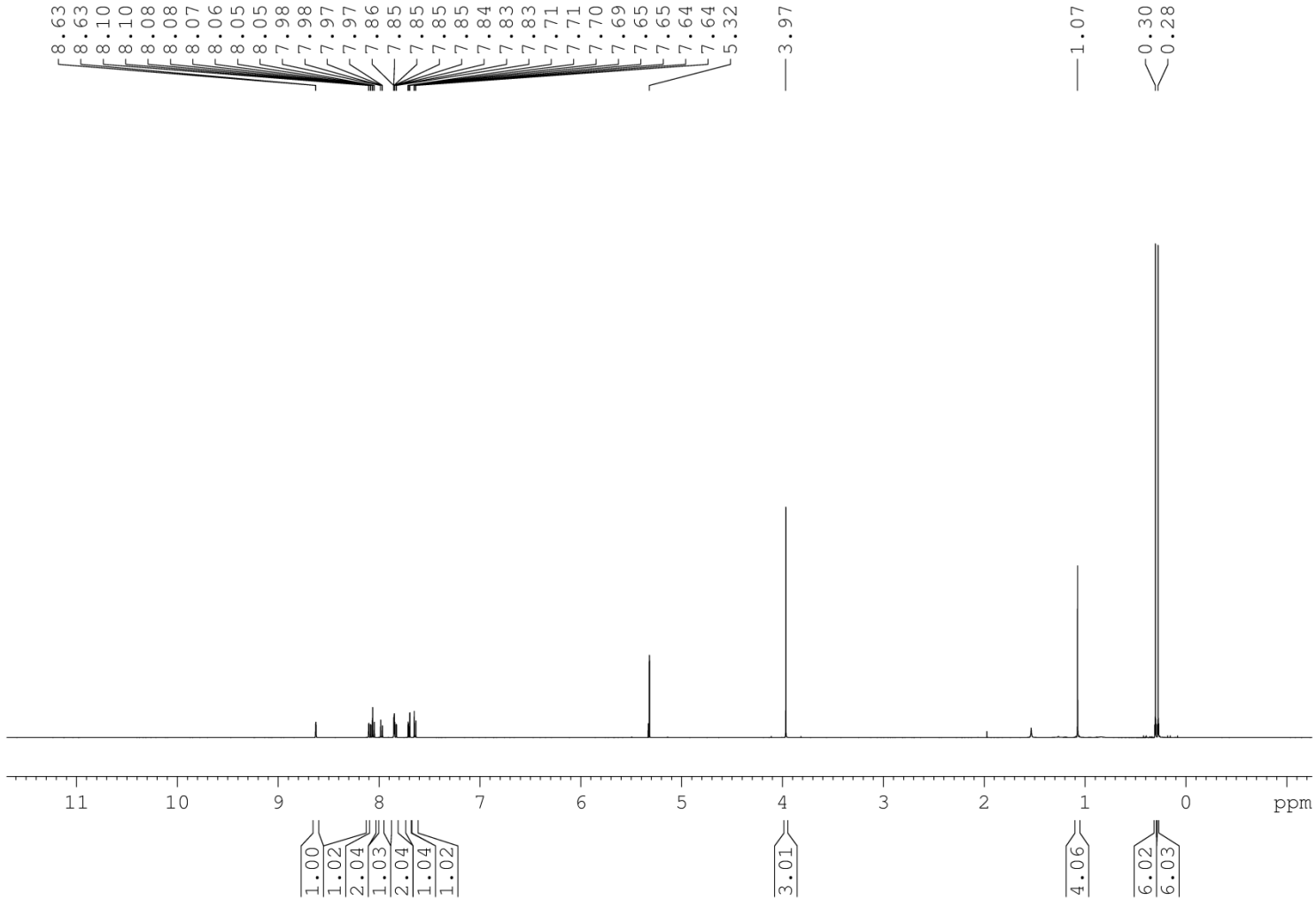

Figure S42 ^{1}H NMR spectrum of compound **20**.

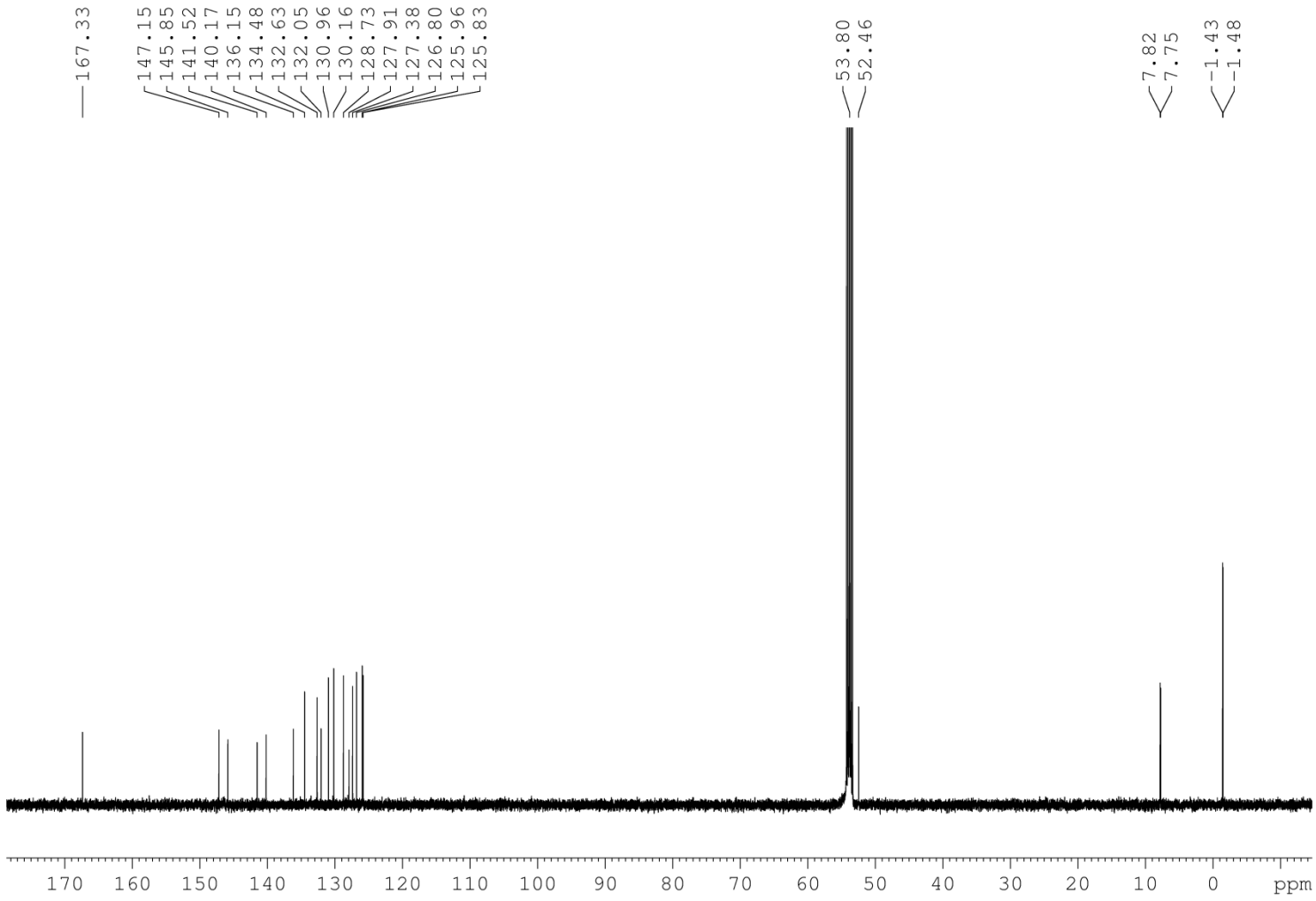

Figure S43 ^{13}C NMR spectrum of compound **20**.

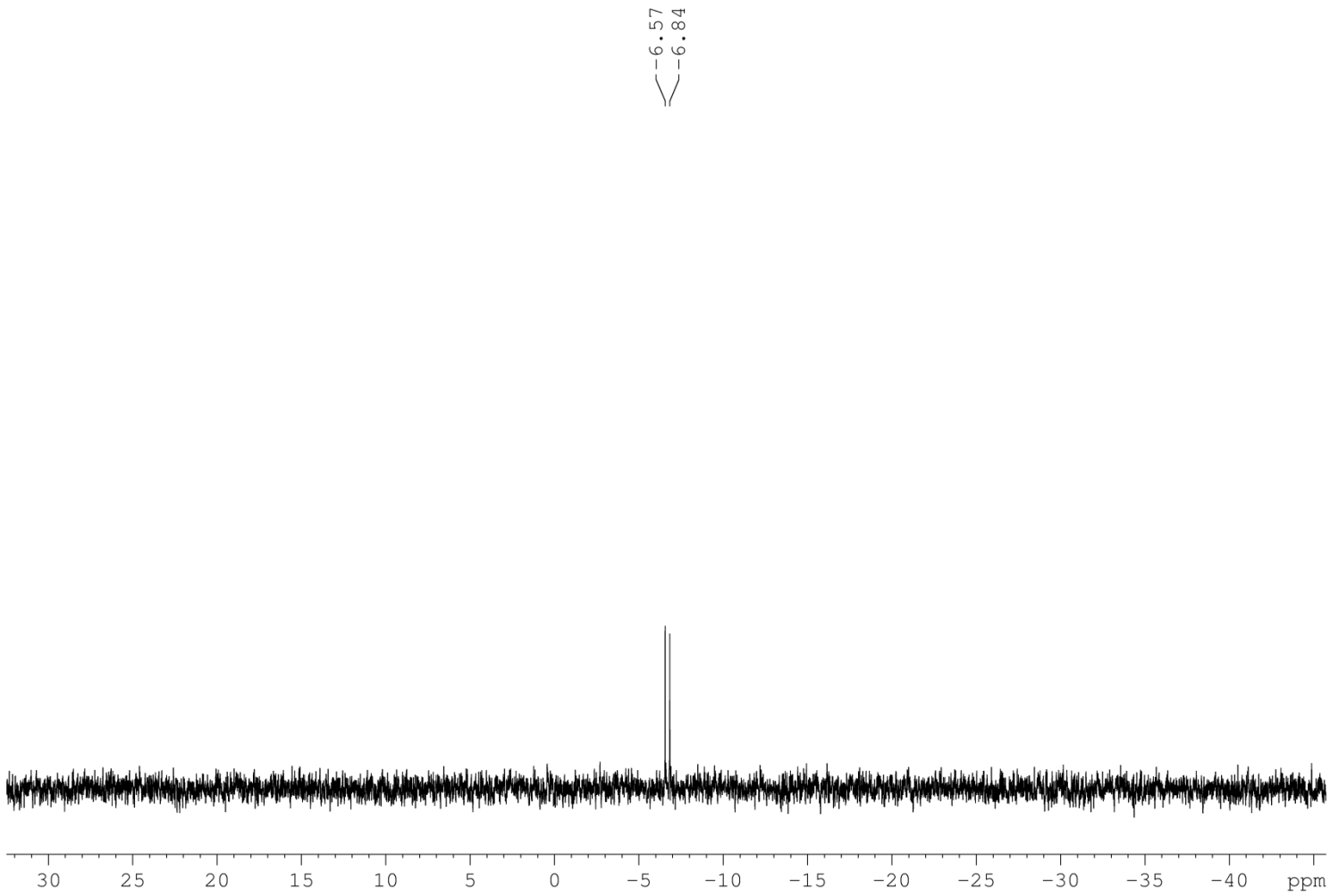

Figure S44 ^{29}Si NMR spectrum of compound **20**.

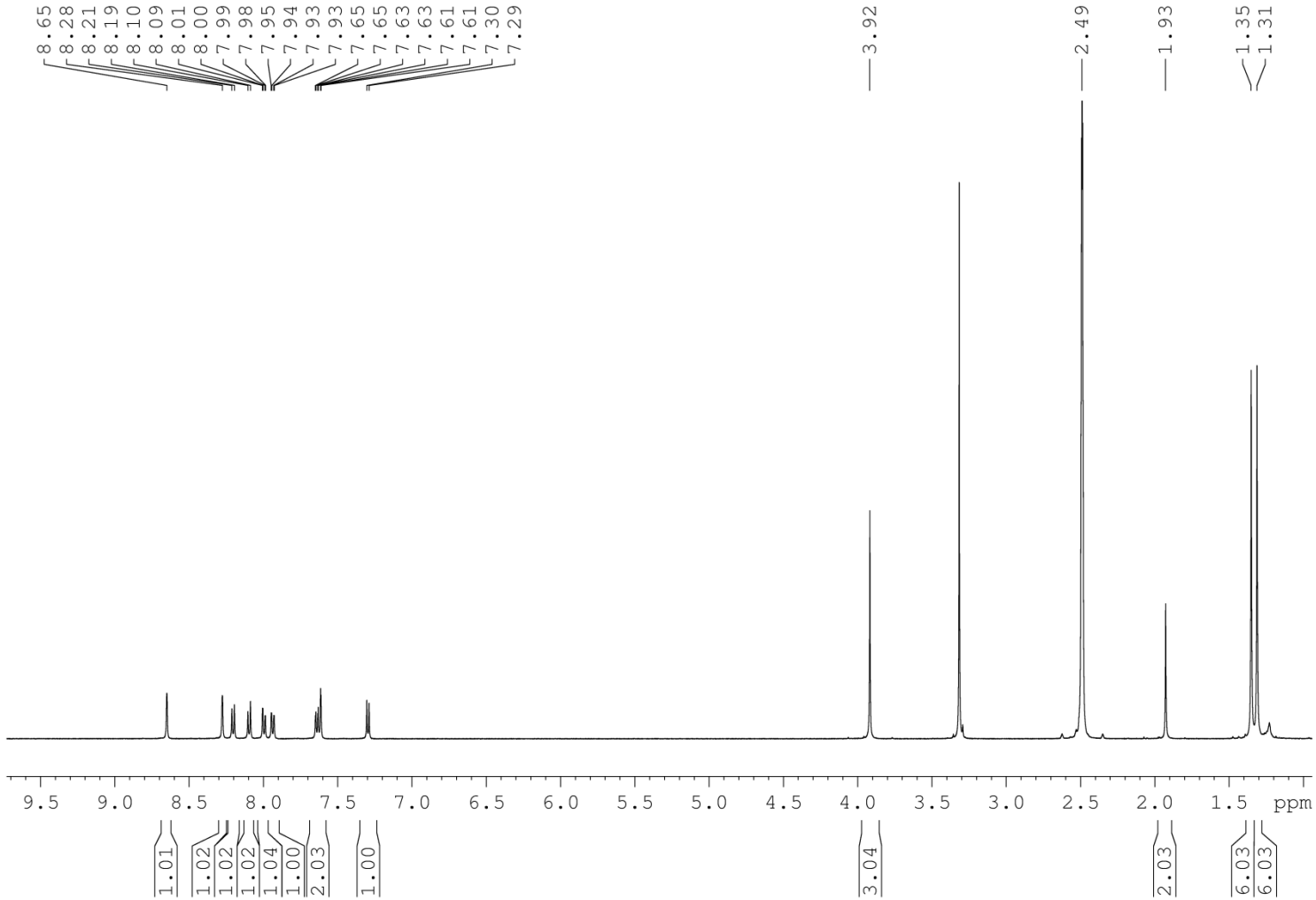

Figure S45 ^{1}H NMR spectrum of compound **21**.

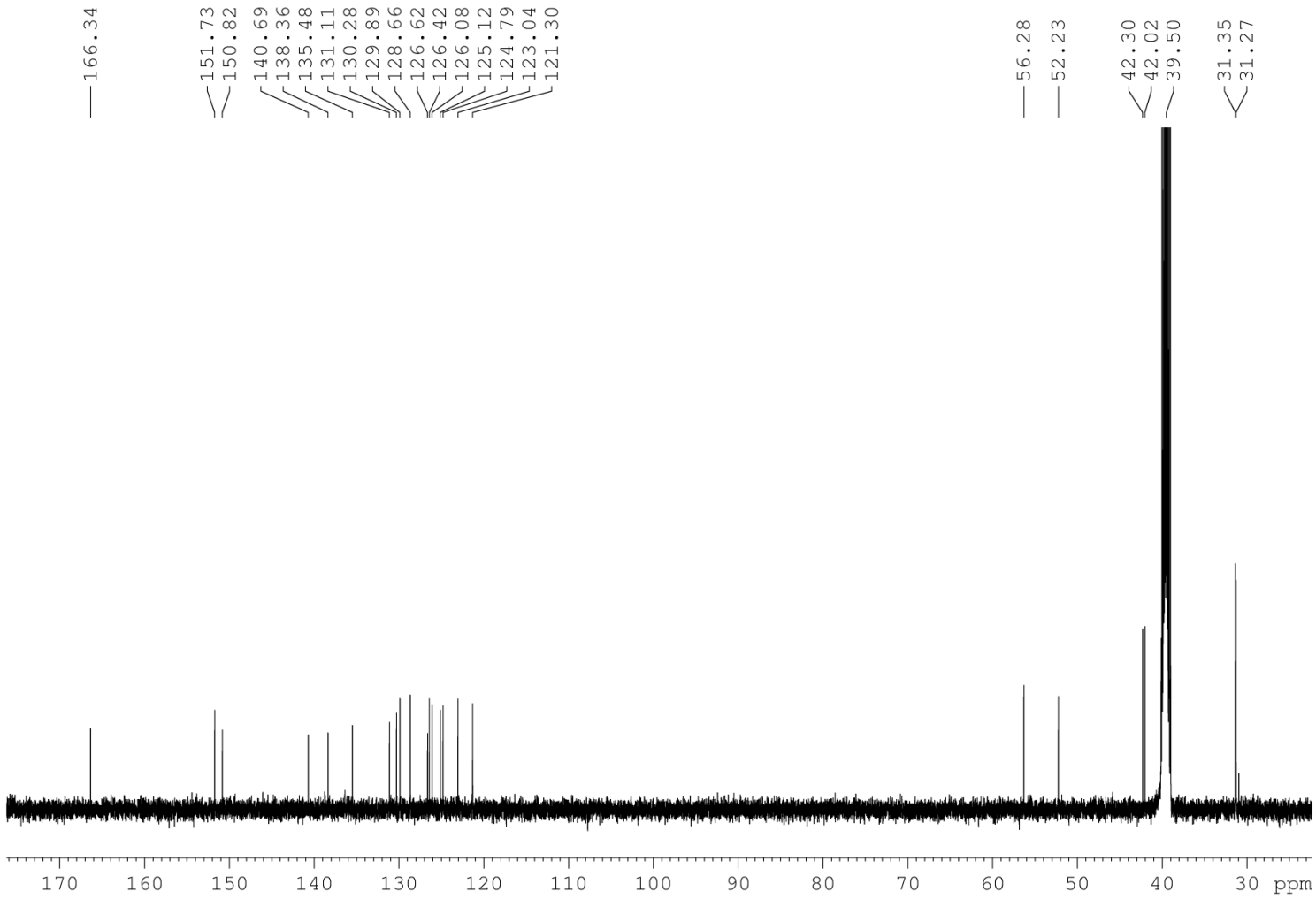

Figure S46 ^{13}C NMR spectrum of compound **21**.

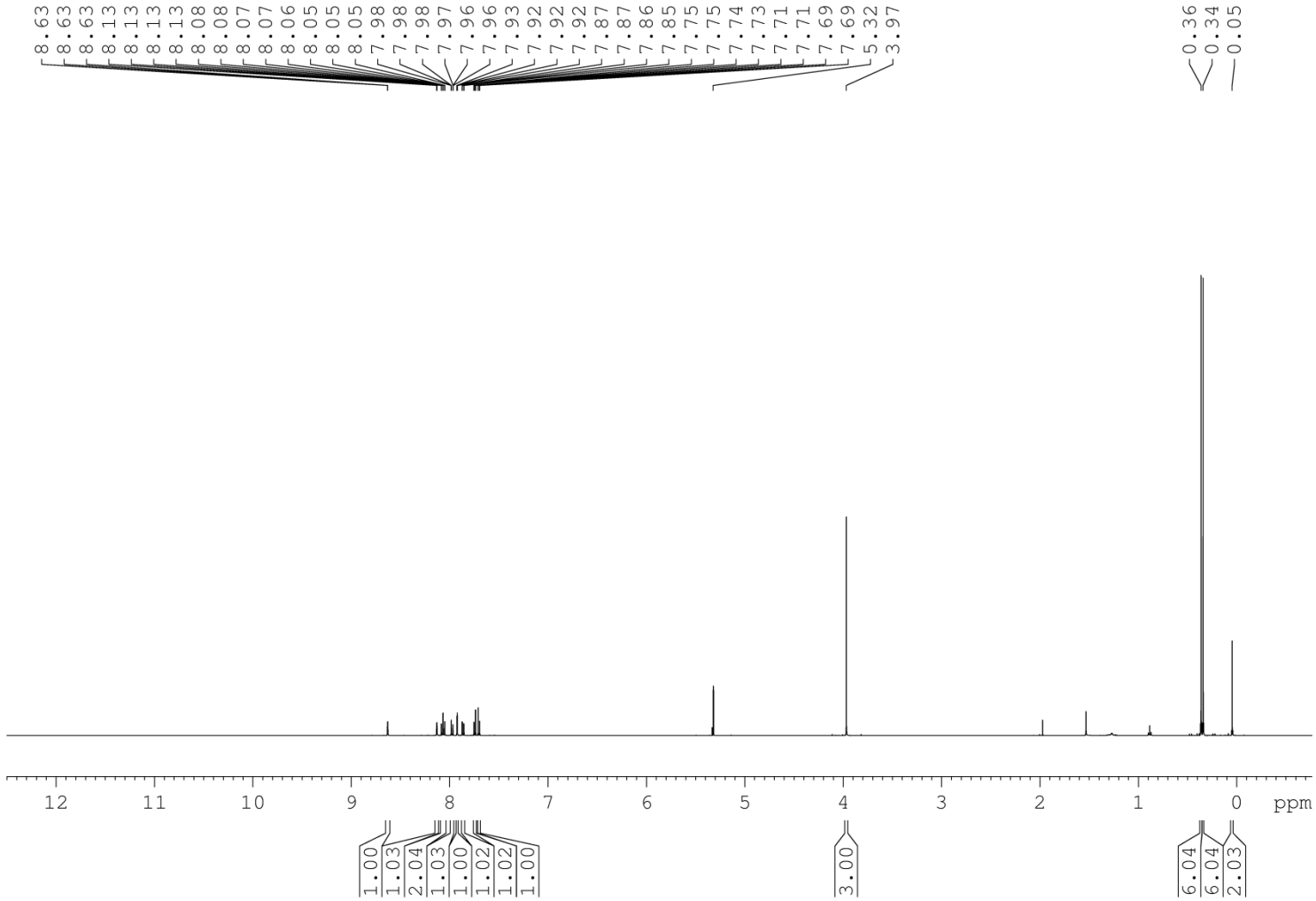

Figure S47 ^{1}H NMR spectrum of compound **22**.

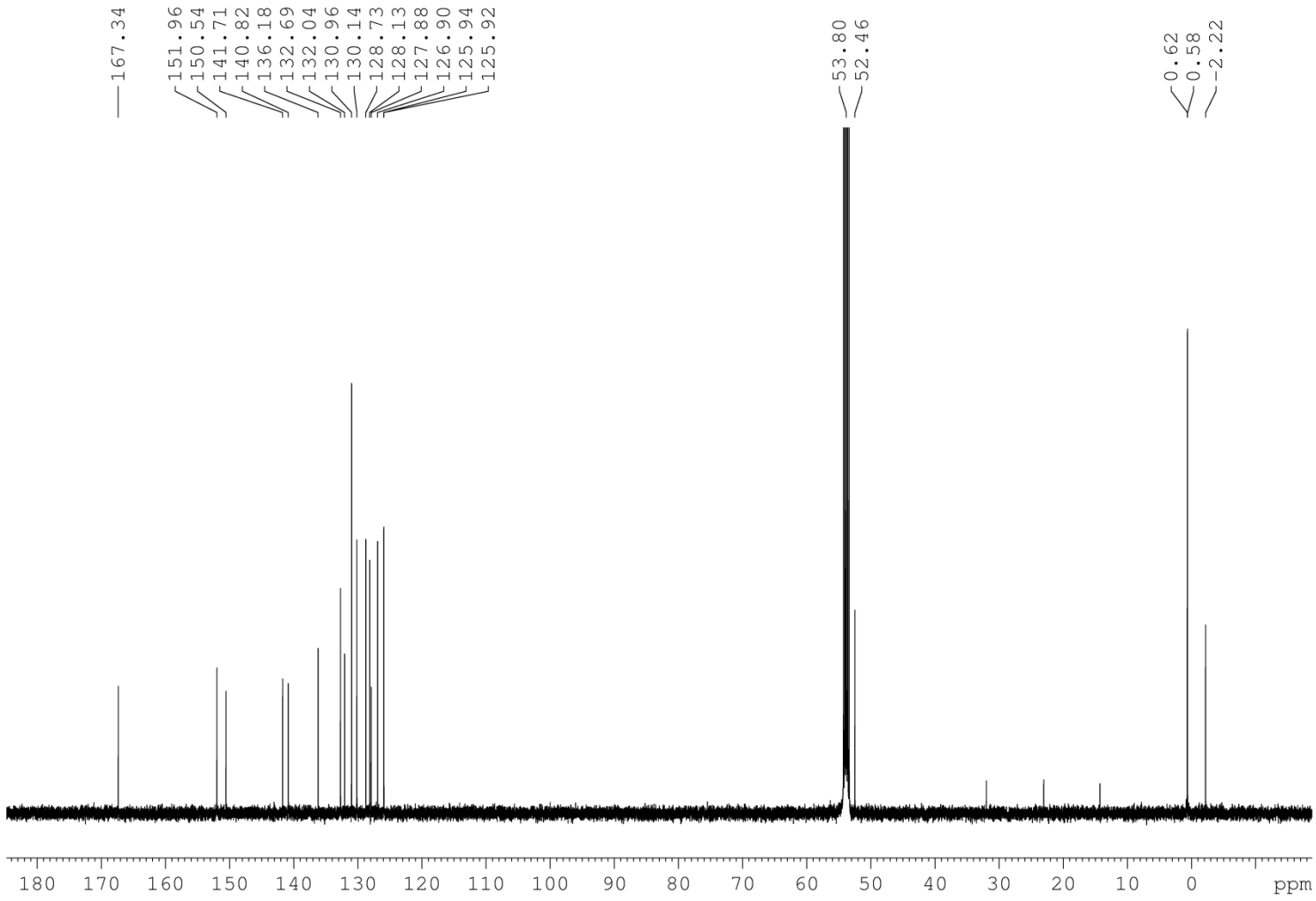

Figure S48 ^{13}C NMR spectrum of compound **22**.

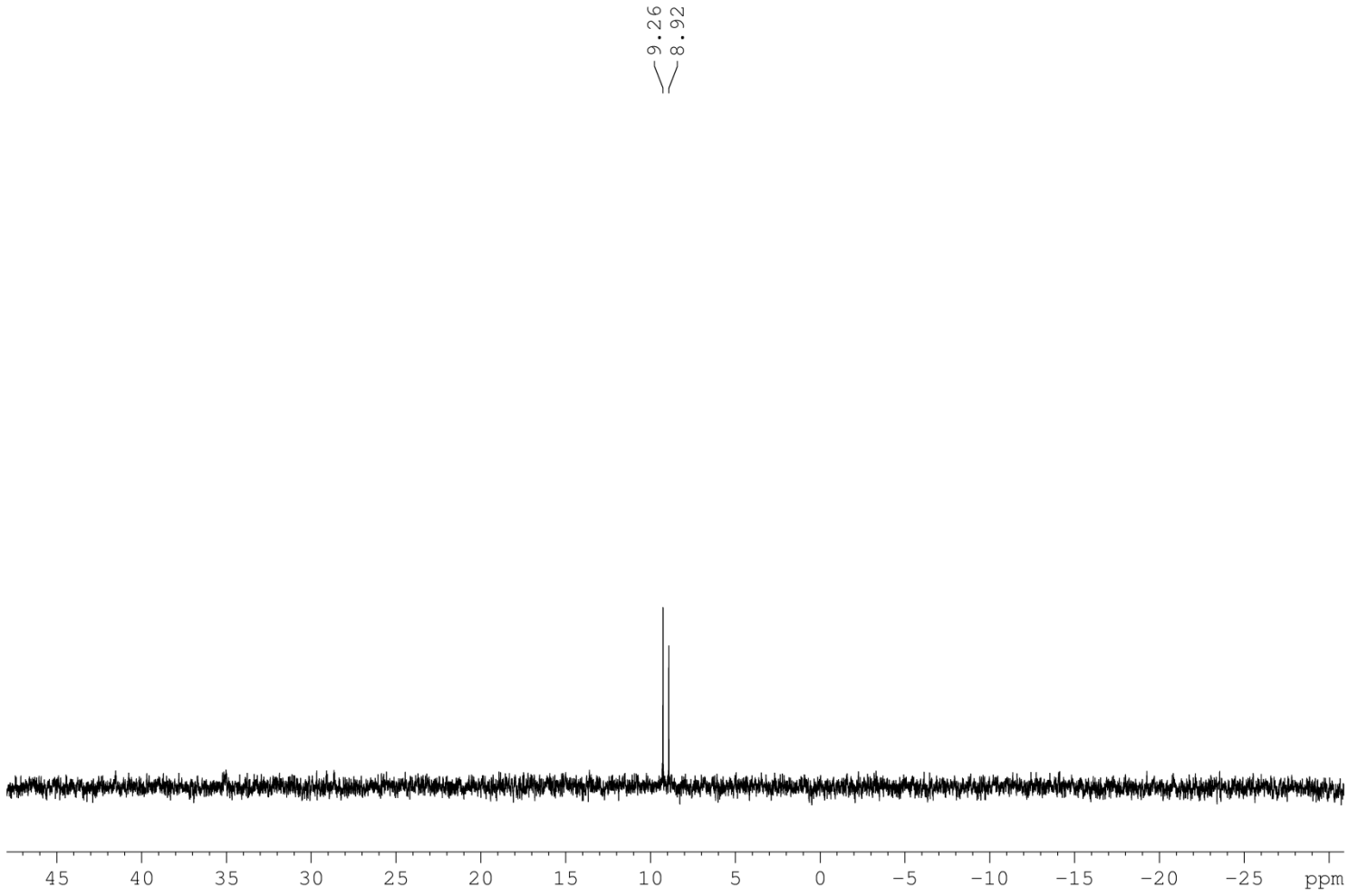

Figure S49 ^{29}Si NMR spectrum of compound **22**.

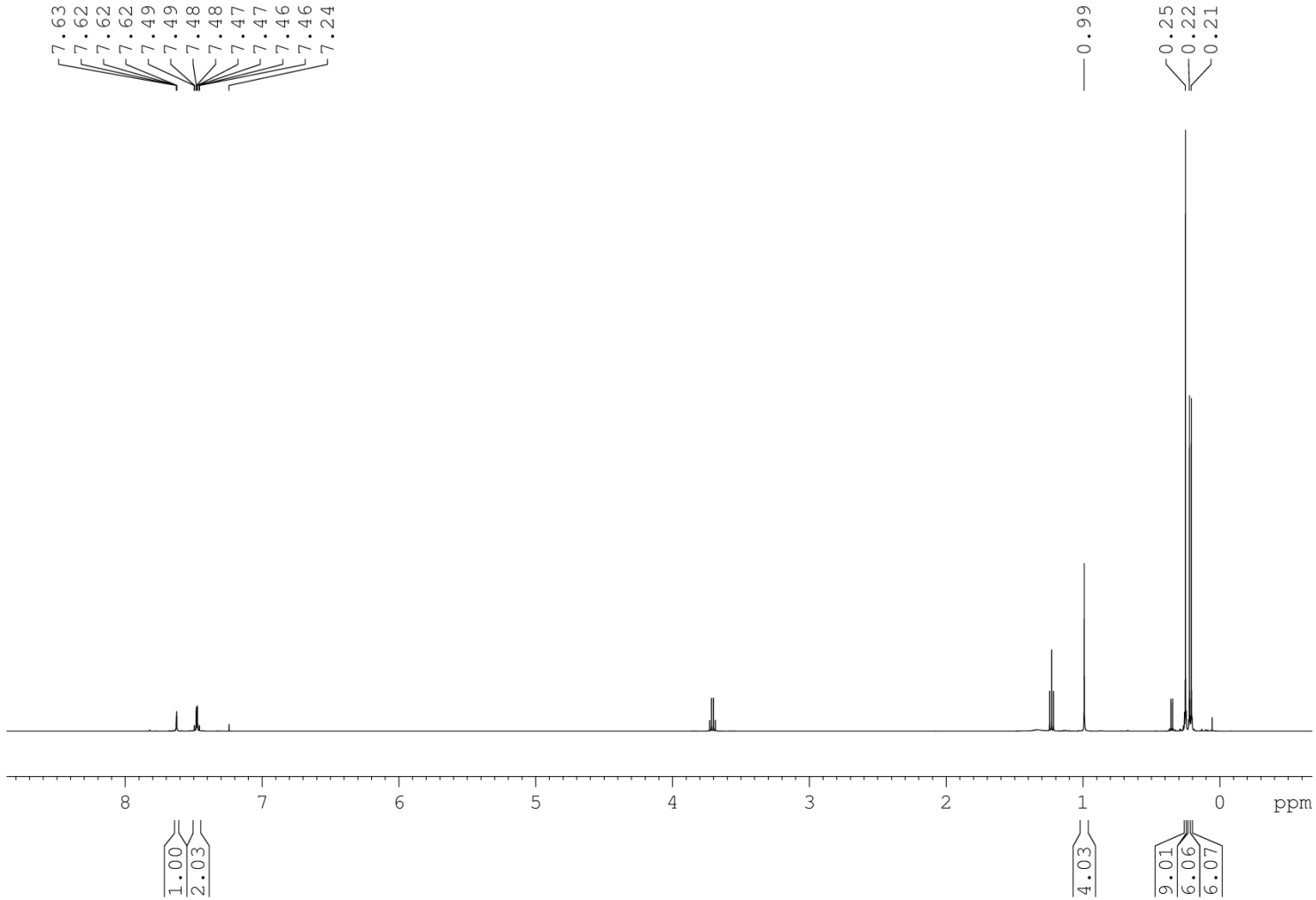

Figure S50 ^{1}H NMR spectrum of compound **27**.

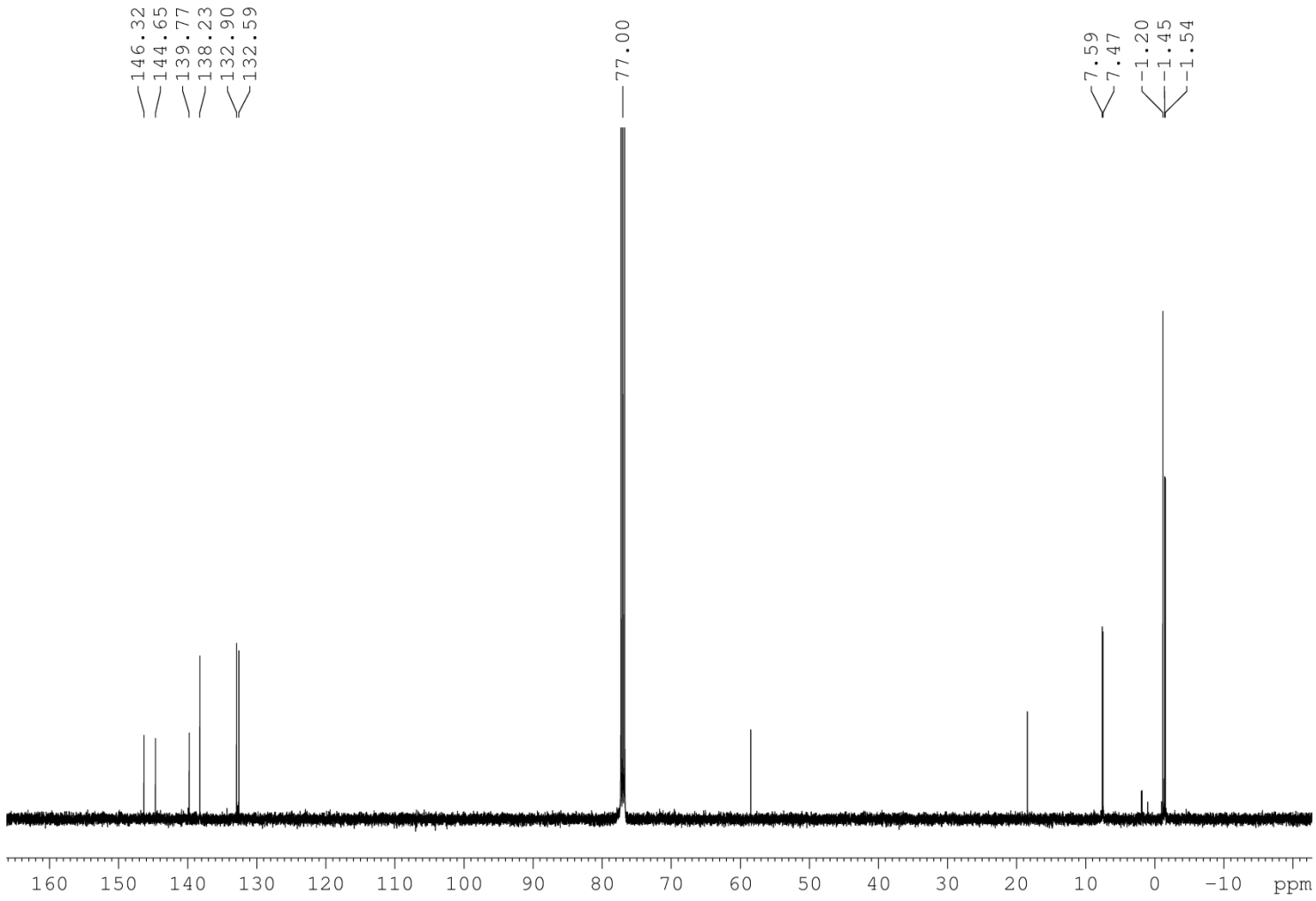

Figure S51 ^{13}C NMR spectrum of compound **27**.

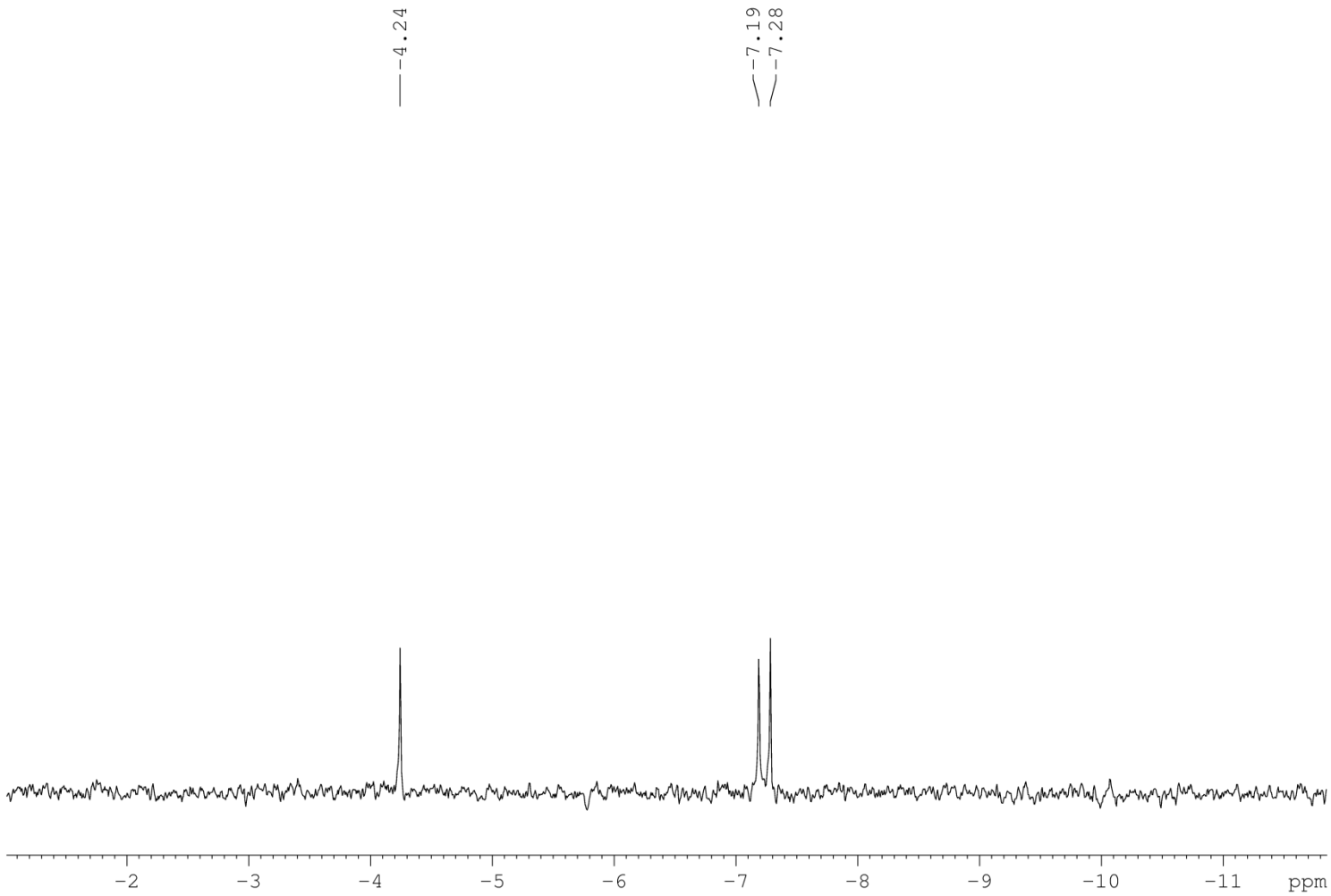

Figure S52 ^{29}Si NMR spectrum of compound **27**.

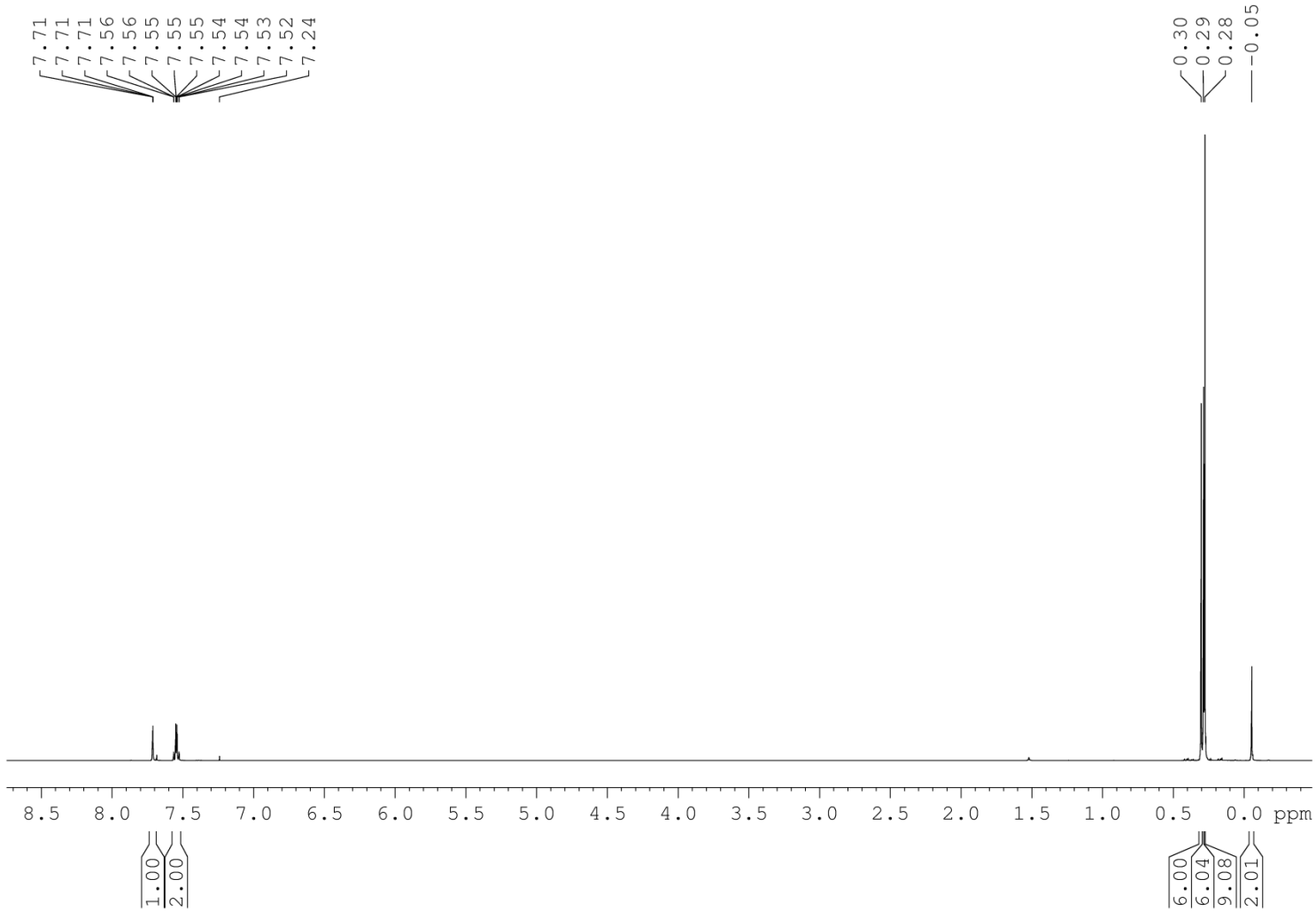

Figure S53 ^{1}H NMR spectrum of compound **28**.

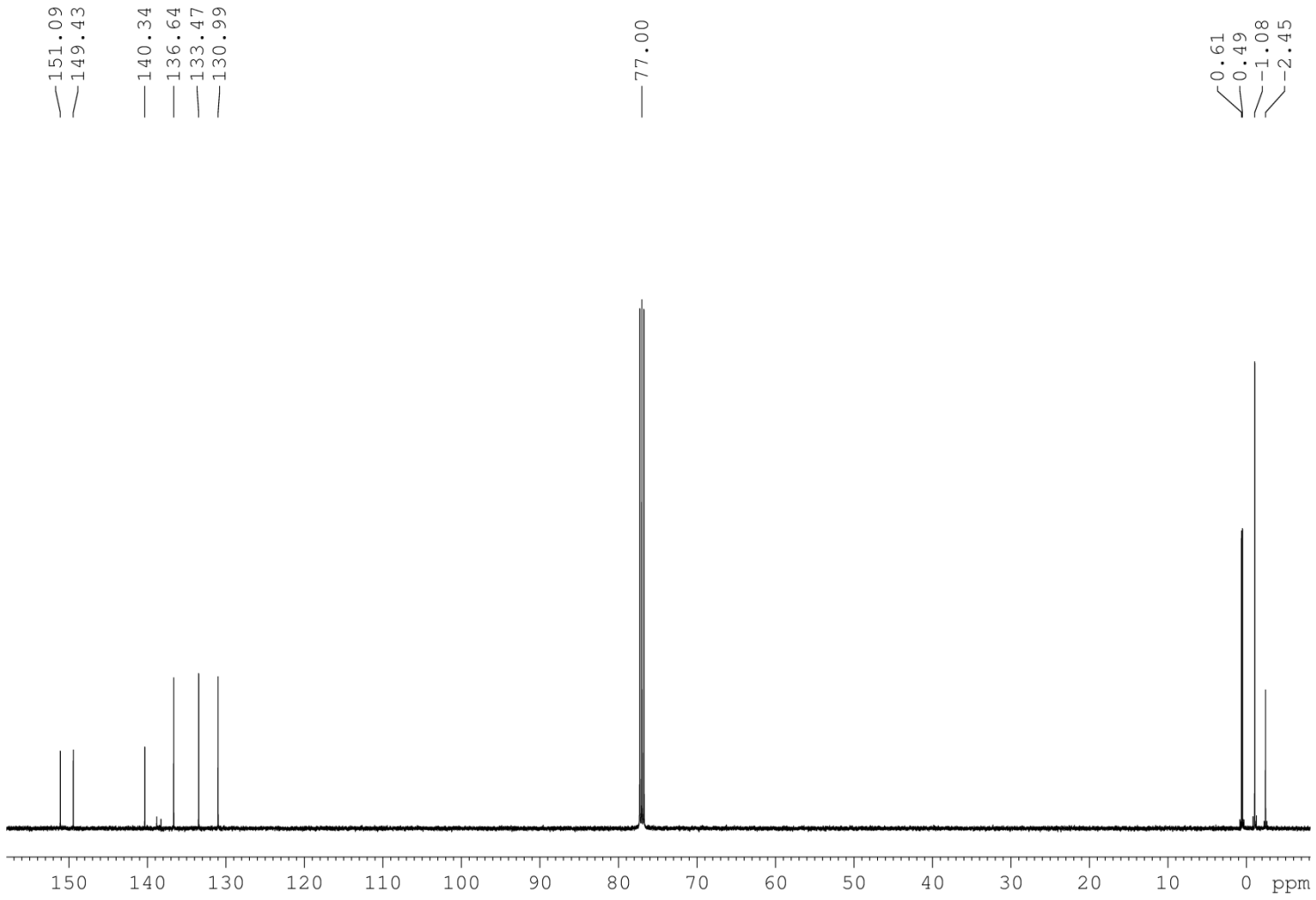

Figure S54 ^{13}C NMR spectrum of compound **28**.

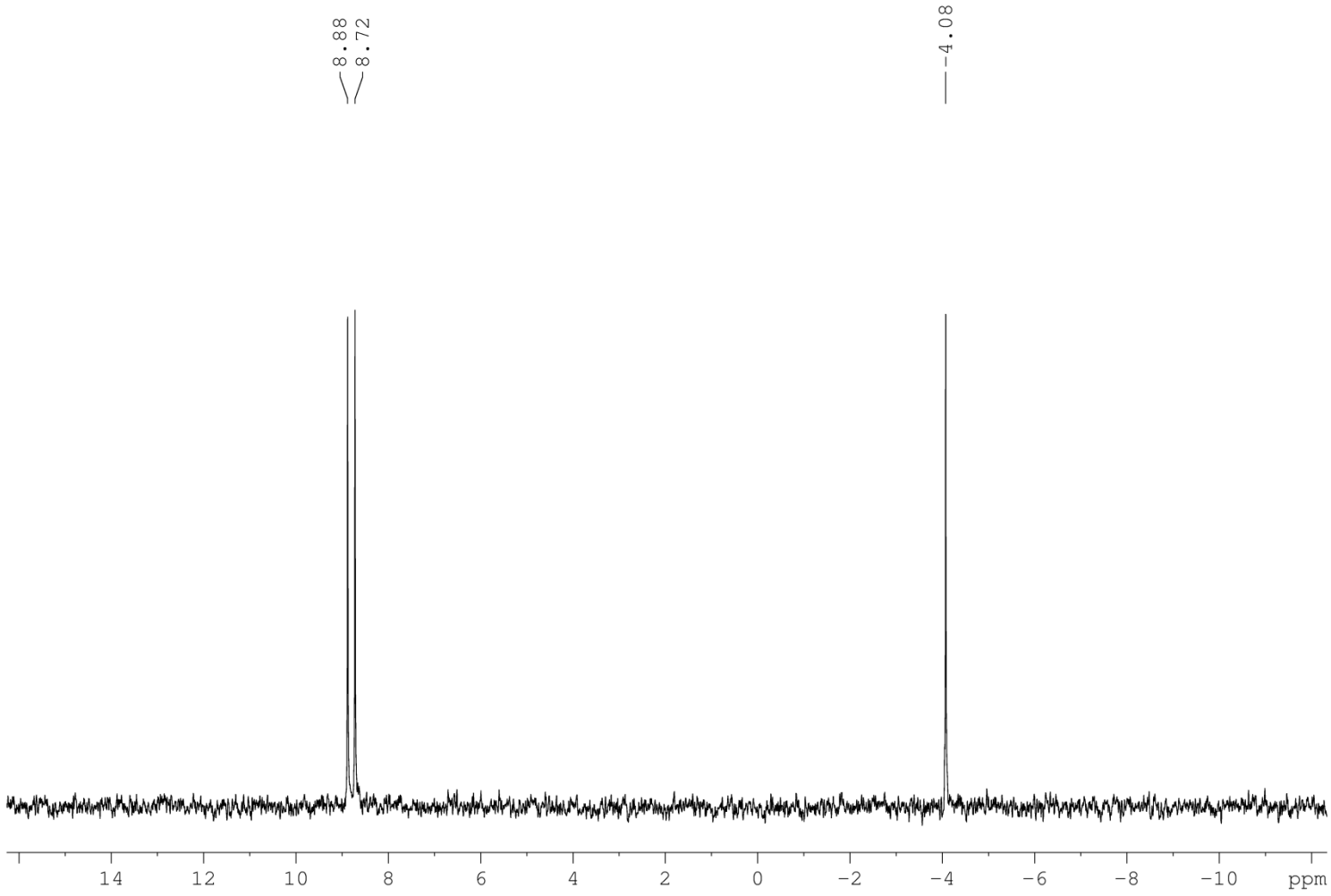

Figure S55 ^{29}Si NMR spectrum of compound **28**.

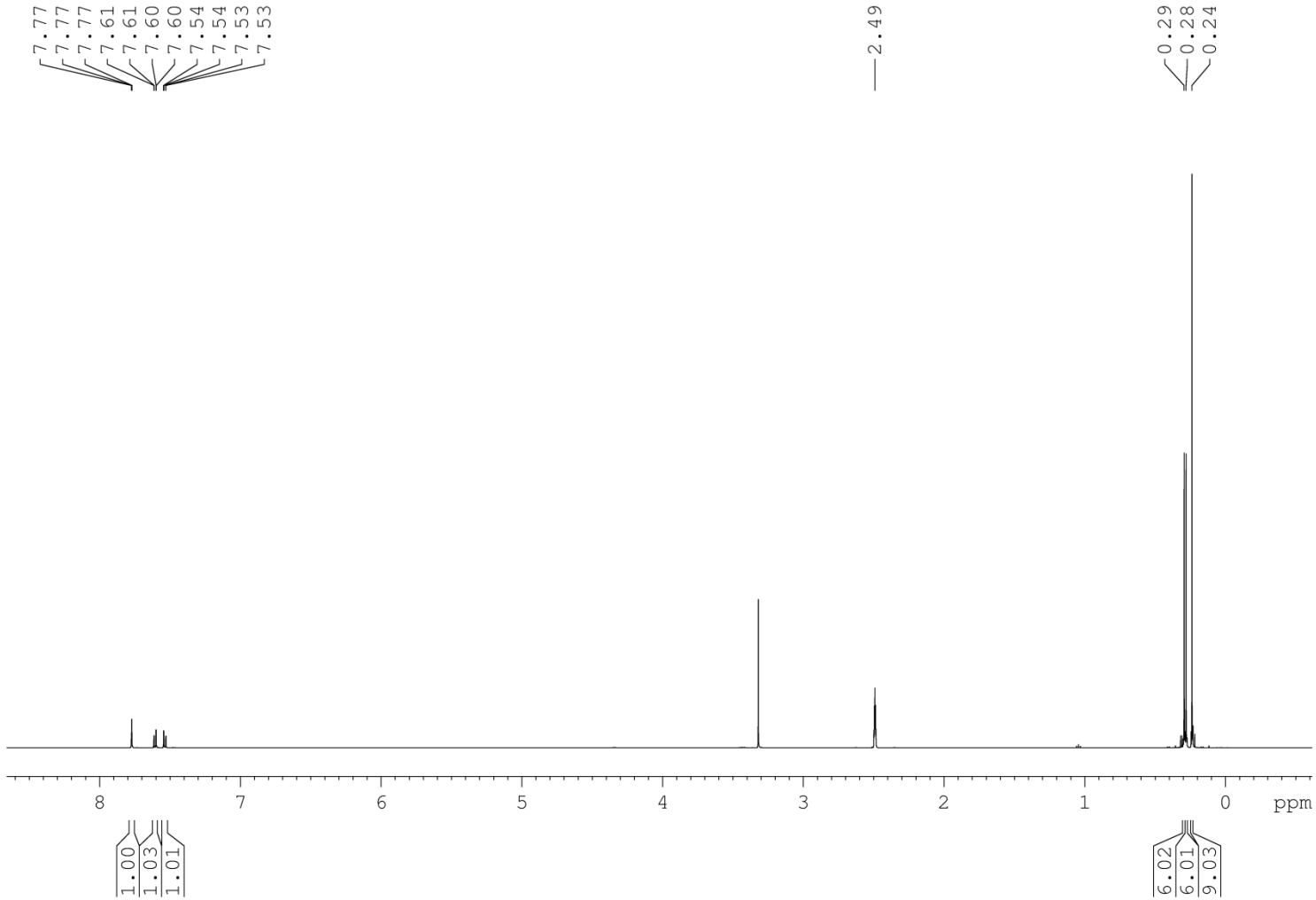

Figure S56 ^{1}H NMR spectrum of compound **29**.

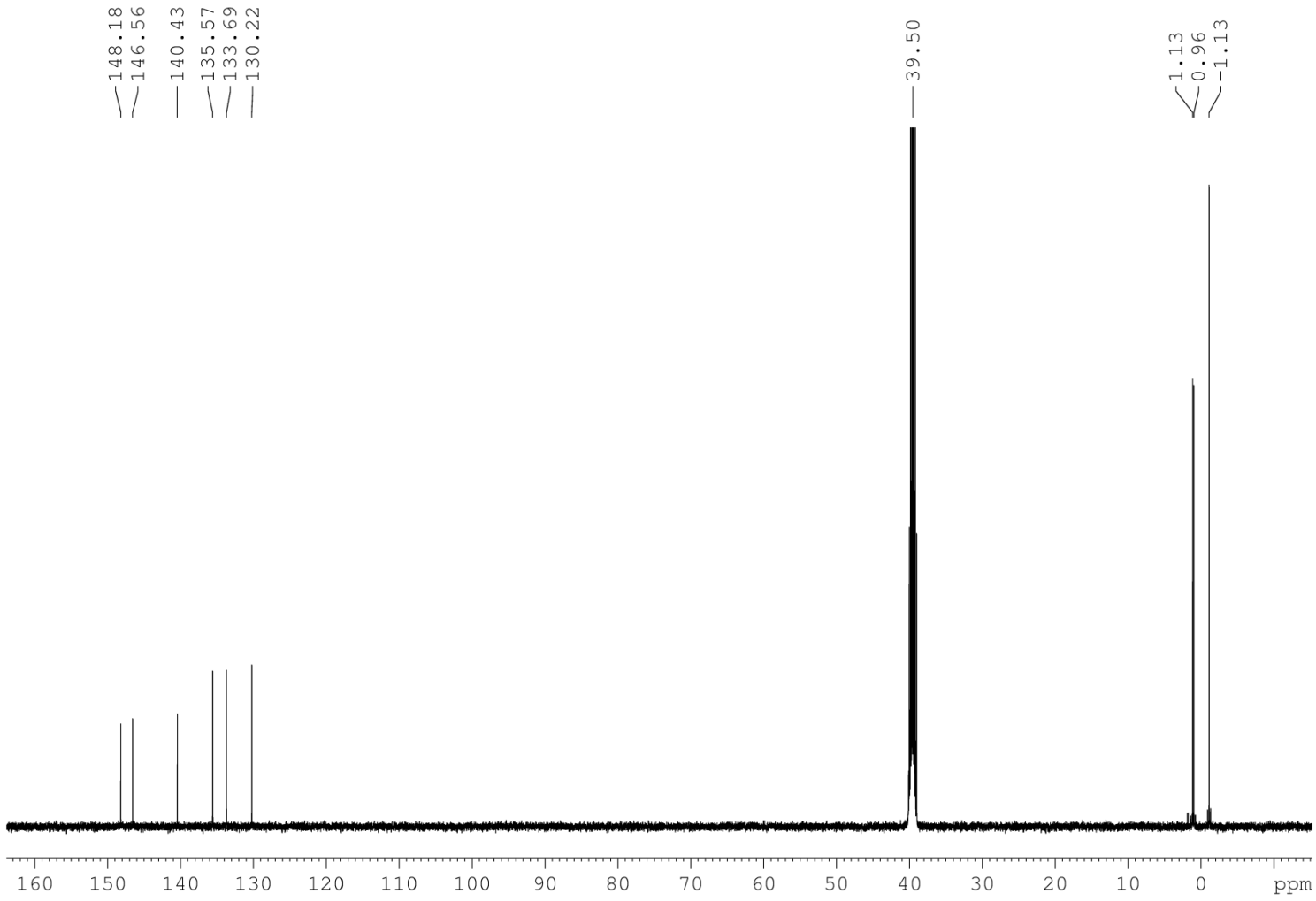

Figure S57 ^{13}C NMR spectrum of compound **29**.

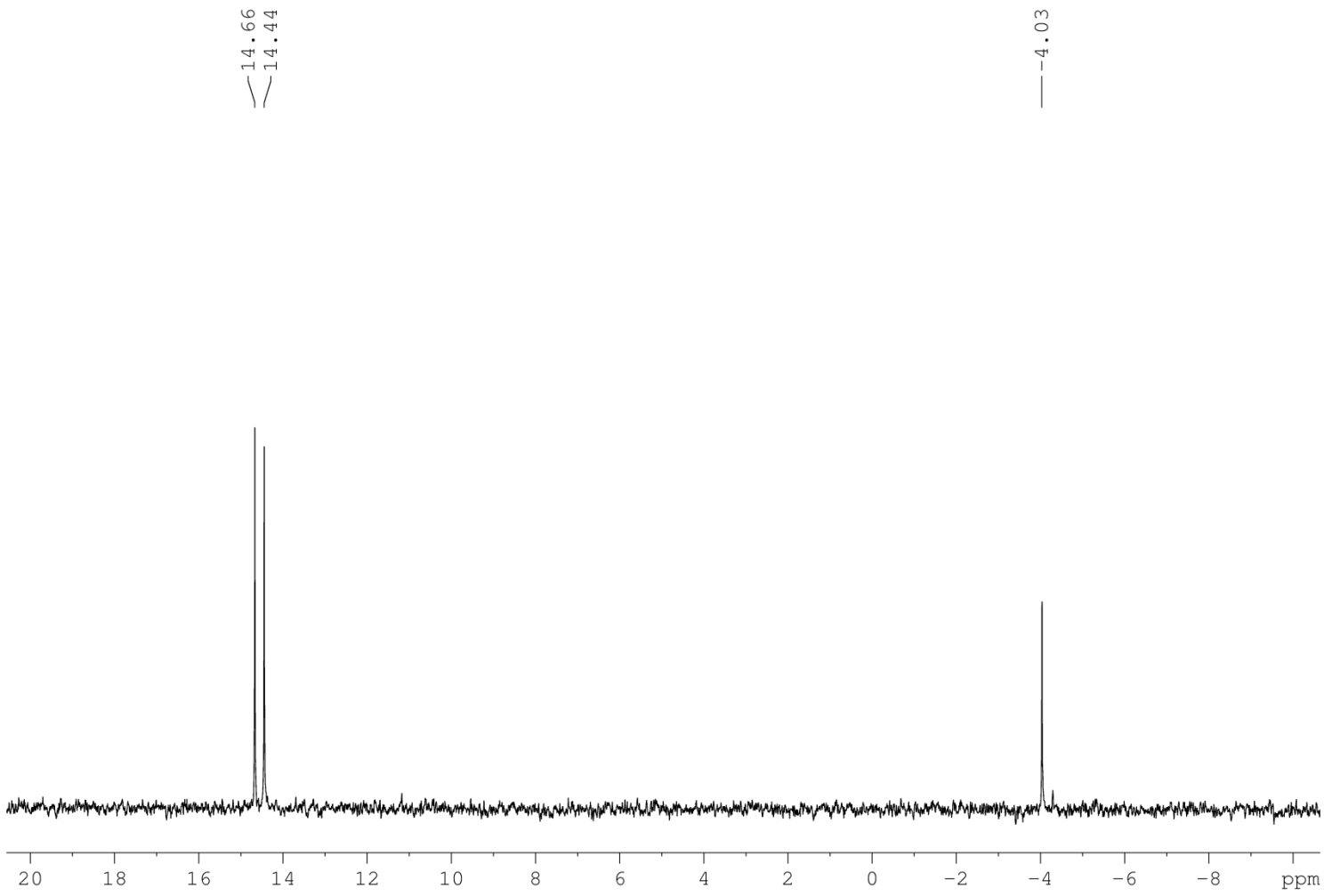

Figure S58 ^{29}Si NMR spectrum of compound **29**.

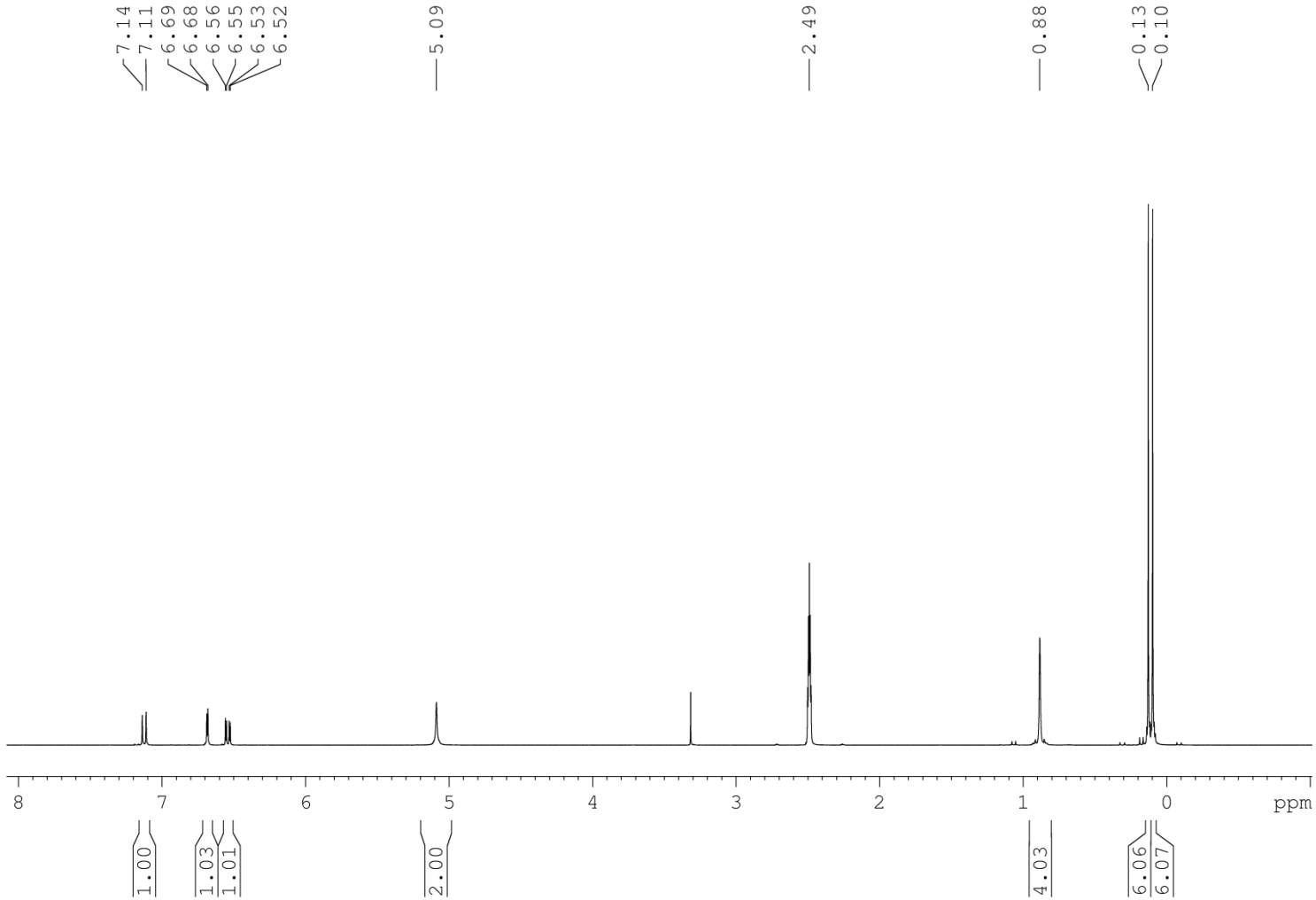

Figure S59 ^{1}H NMR spectrum of compound **30**.

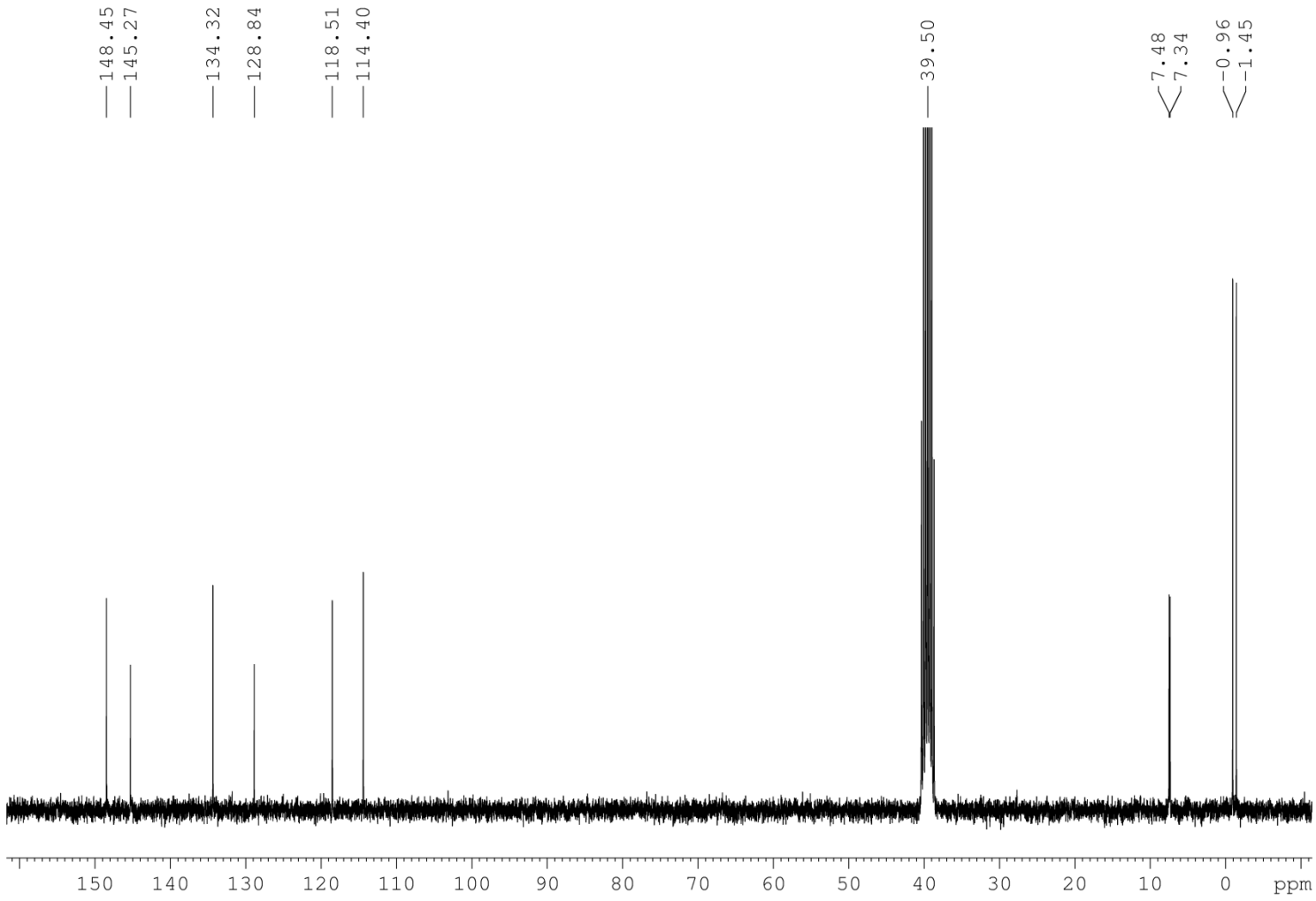

Figure S60 ^{13}C NMR spectrum of compound **30**.

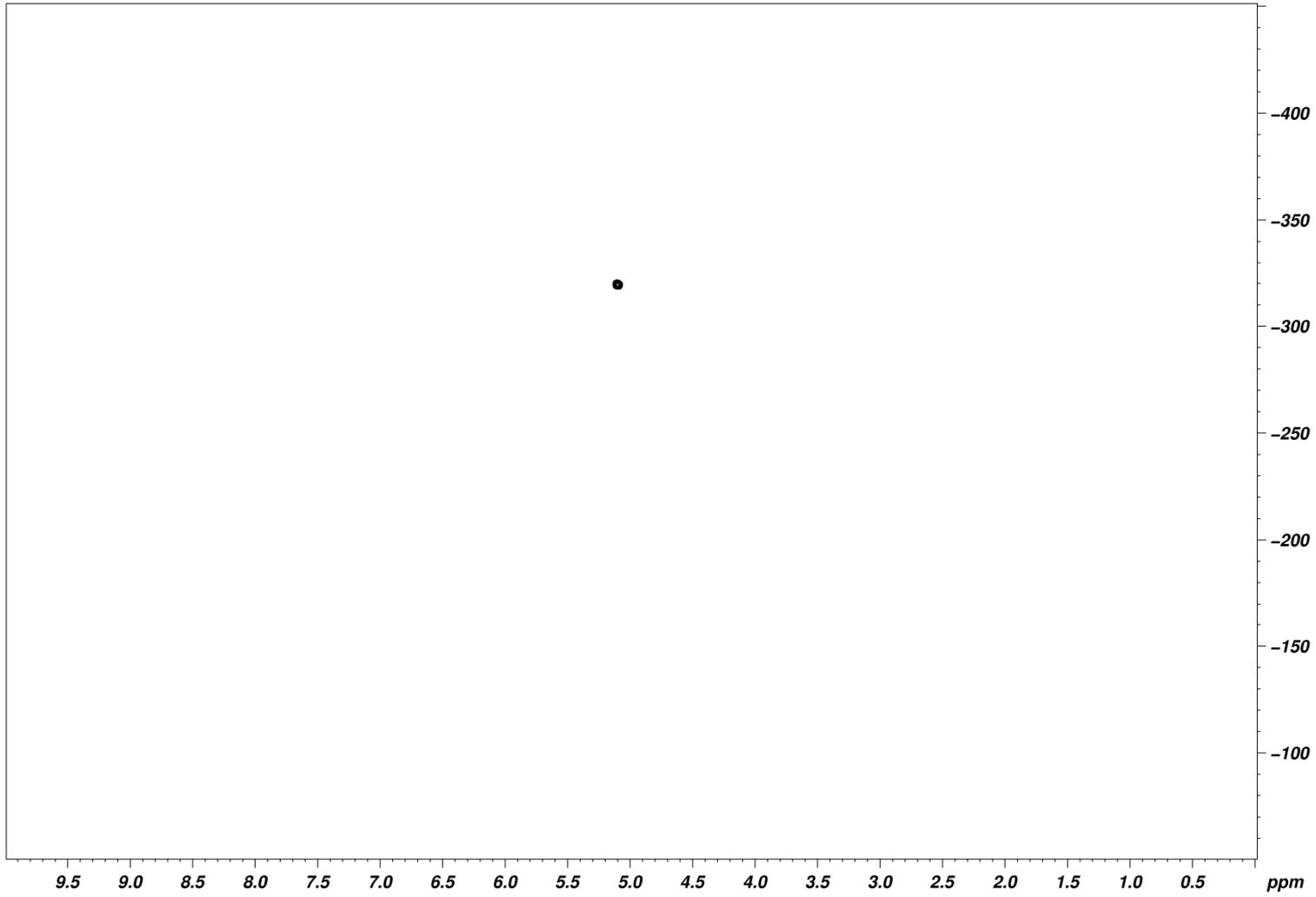

Figure S61 ^{15}N NMR spectrum of compound **30**.

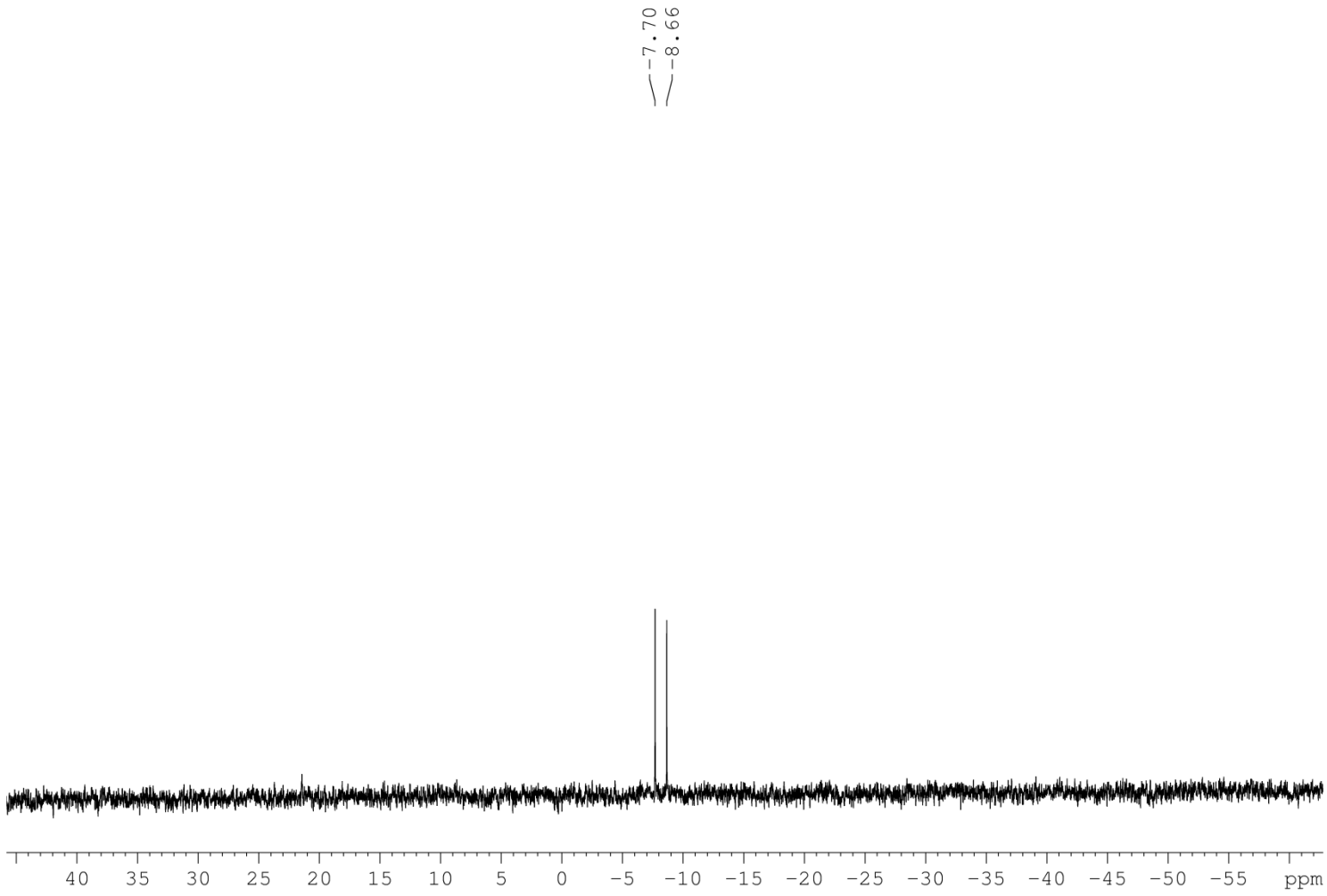

Figure S62 ^{29}Si NMR spectrum of compound **30**.

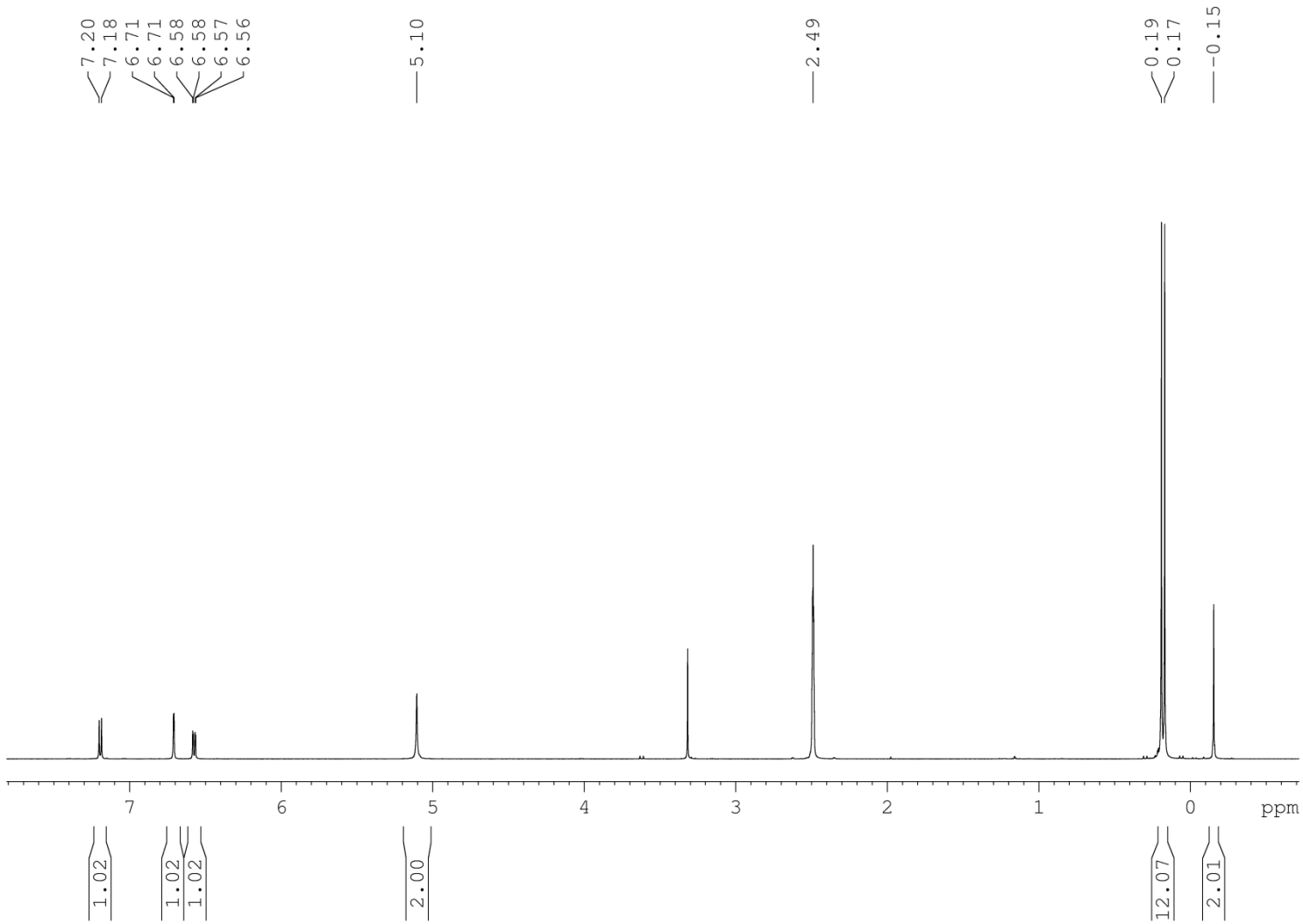

Figure S63 ^{1}H NMR spectrum of compound **31**.

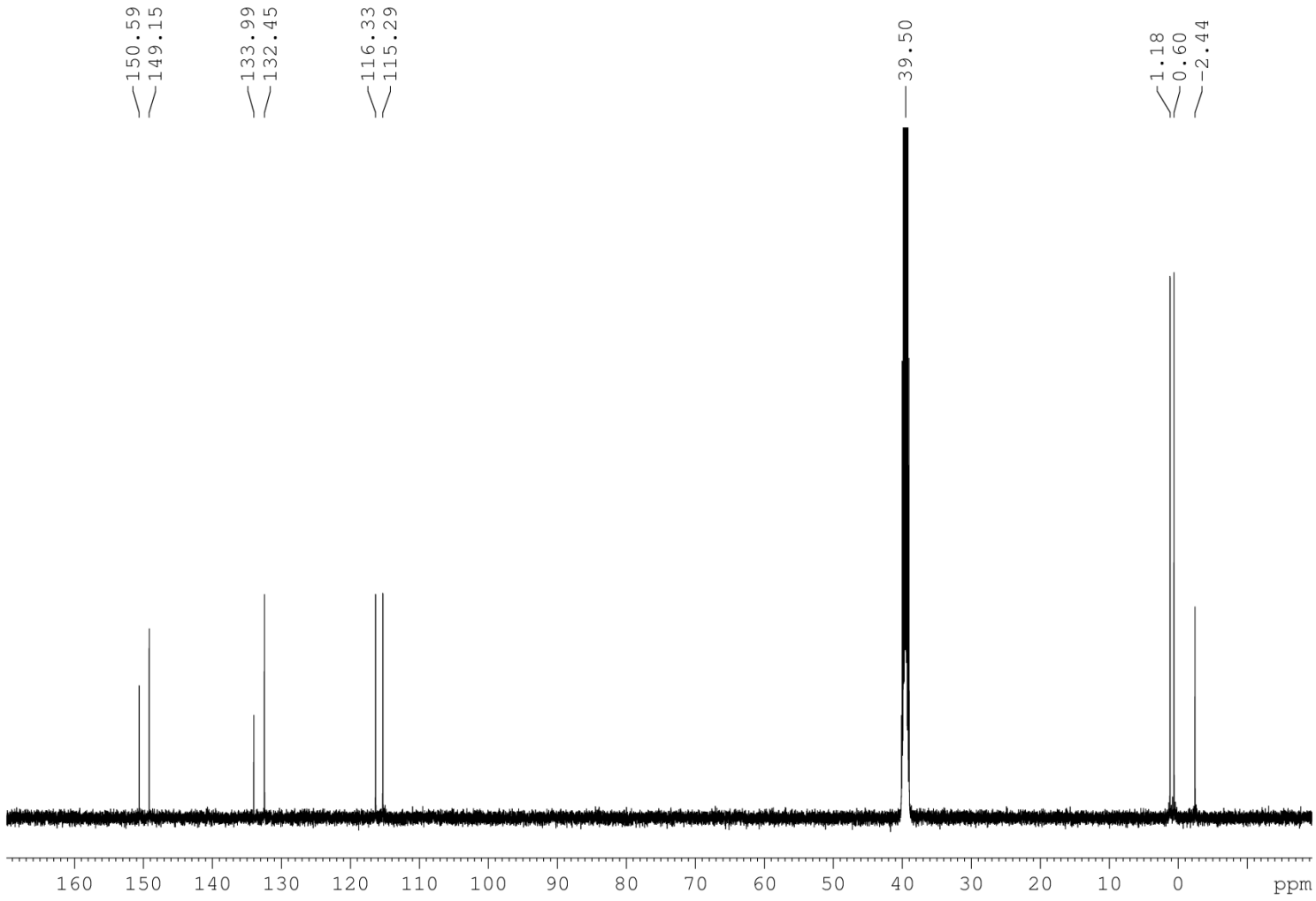

Figure S64 ^{13}C NMR spectrum of compound **31**.

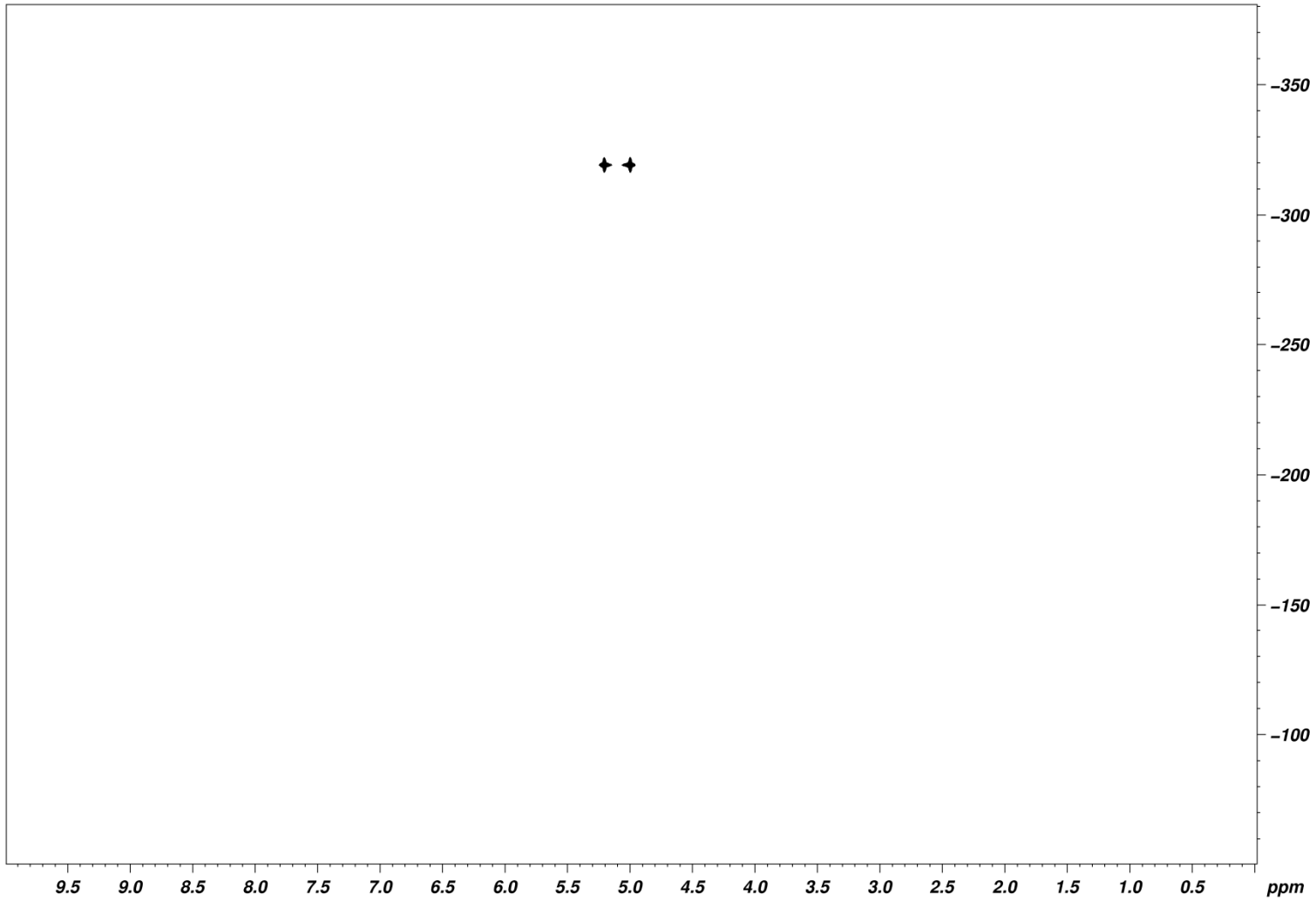

Figure S65 ^{15}N NMR spectrum of compound **31**.

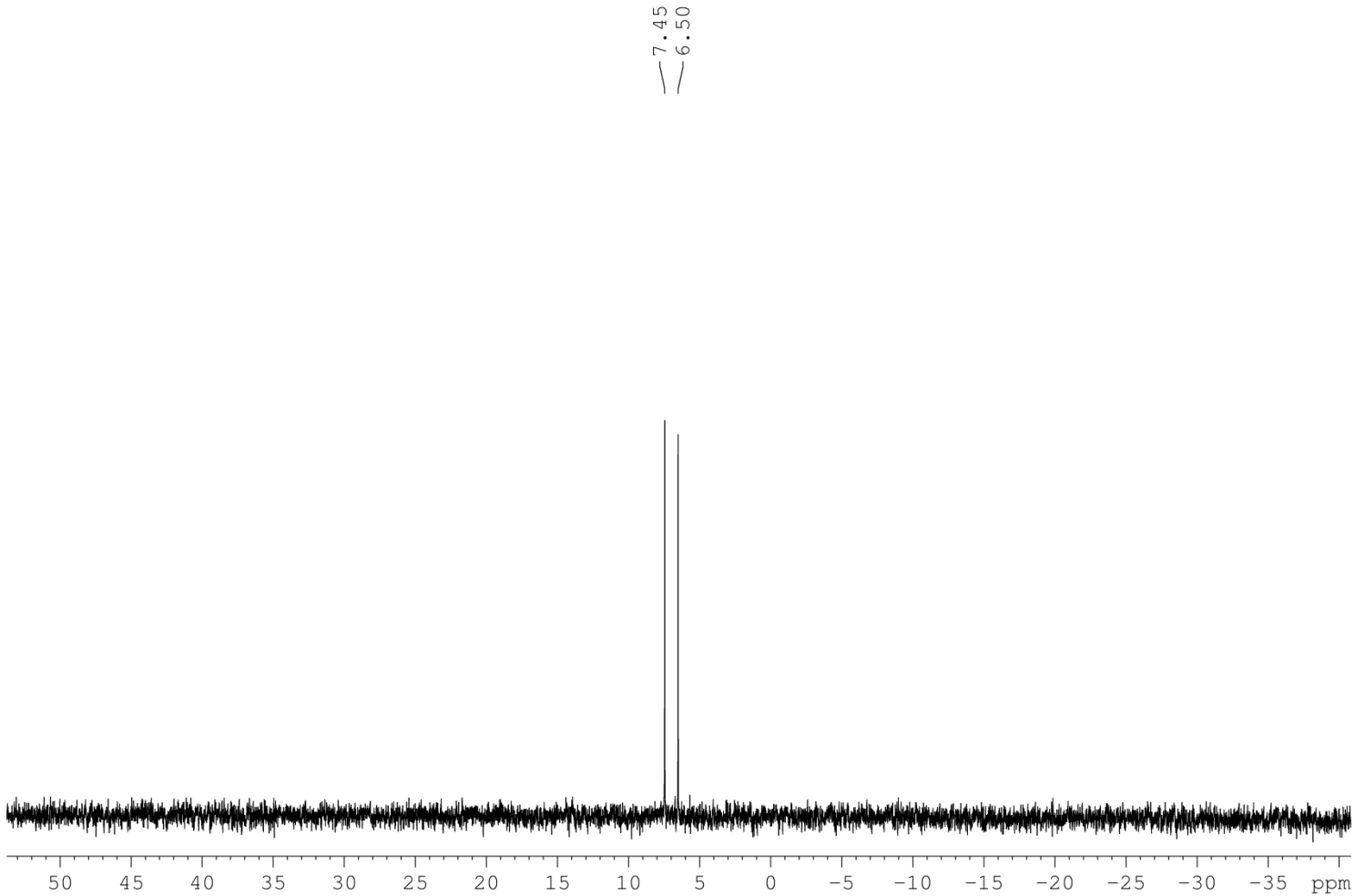

Figure S66 ^{29}Si NMR spectrum of compound **31**.

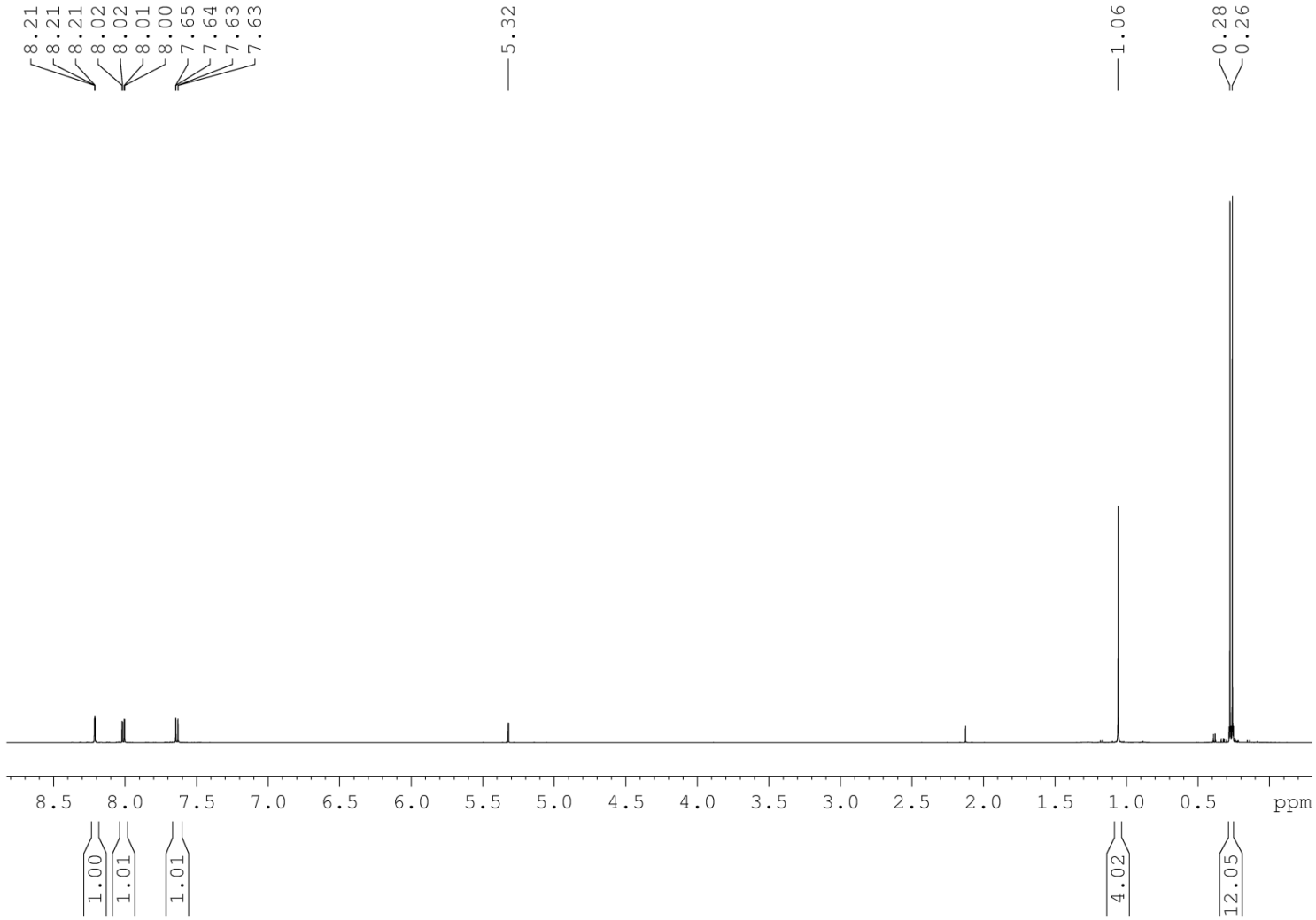

Figure S67 ^{1}H NMR spectrum of compound **33**.

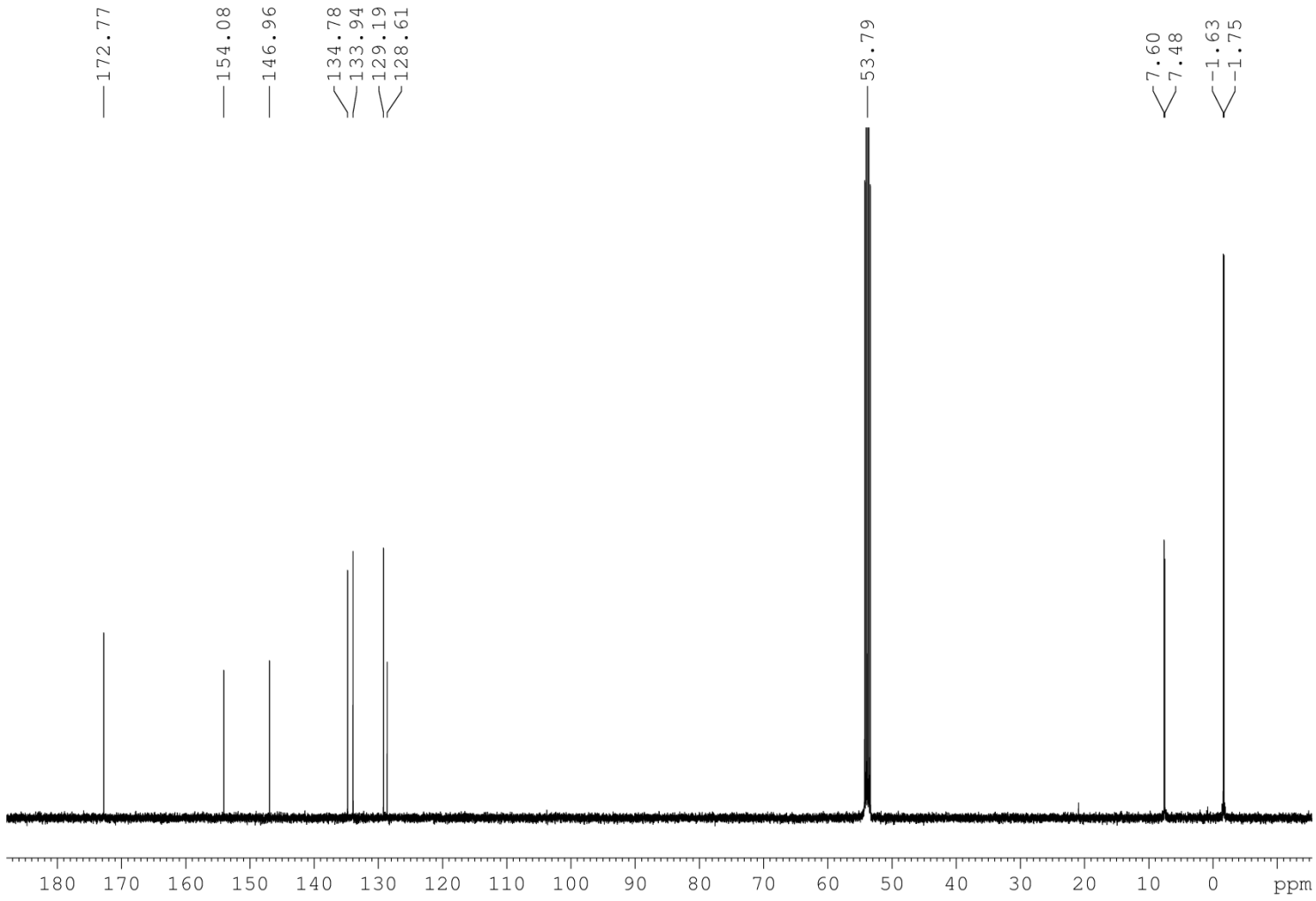

Figure S68 ^{13}C NMR spectrum of compound **33**.

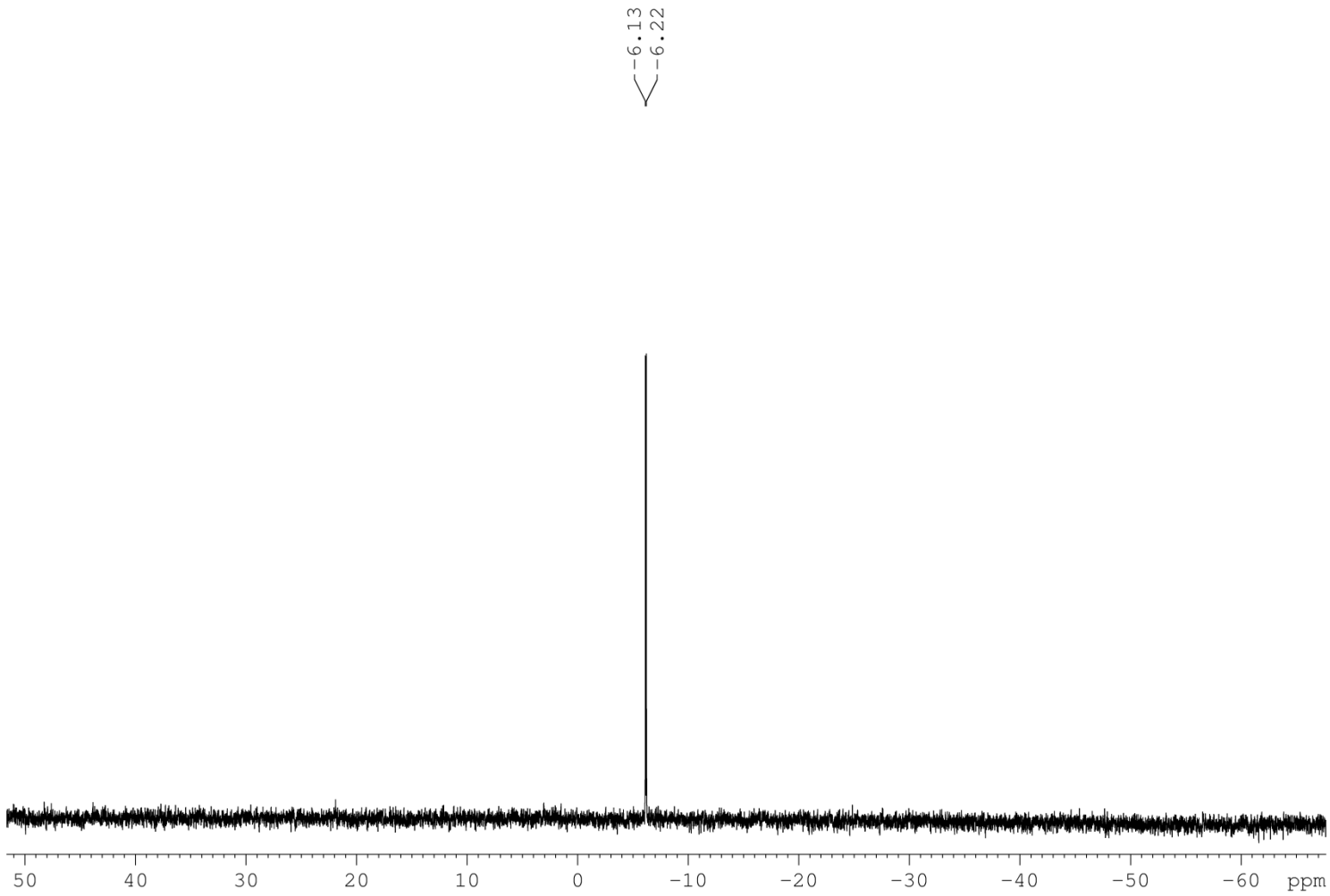

Figure S69 ^{29}Si NMR spectrum of compound **33**.

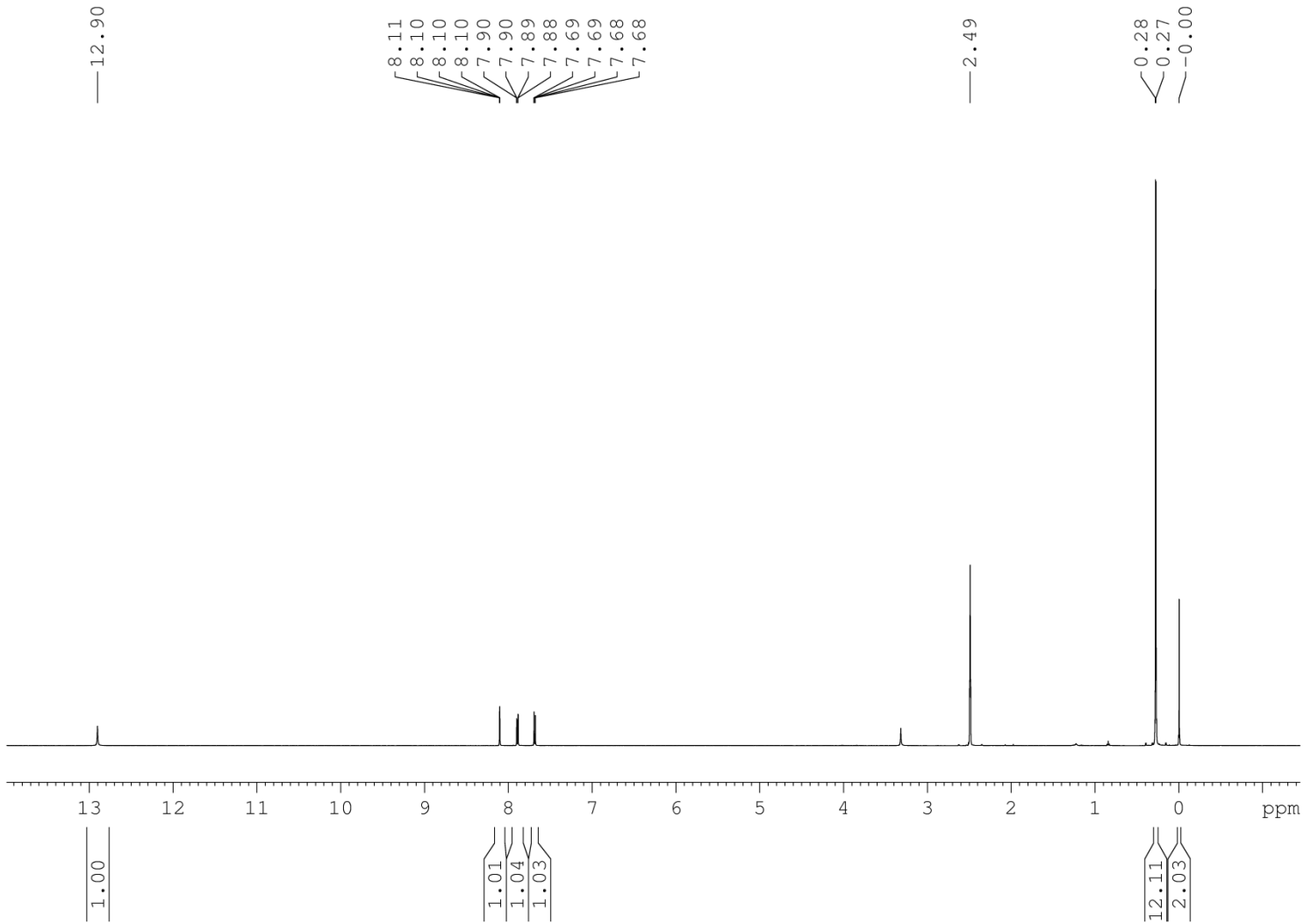

Figure S70 ^{1}H NMR spectrum of compound **34**.

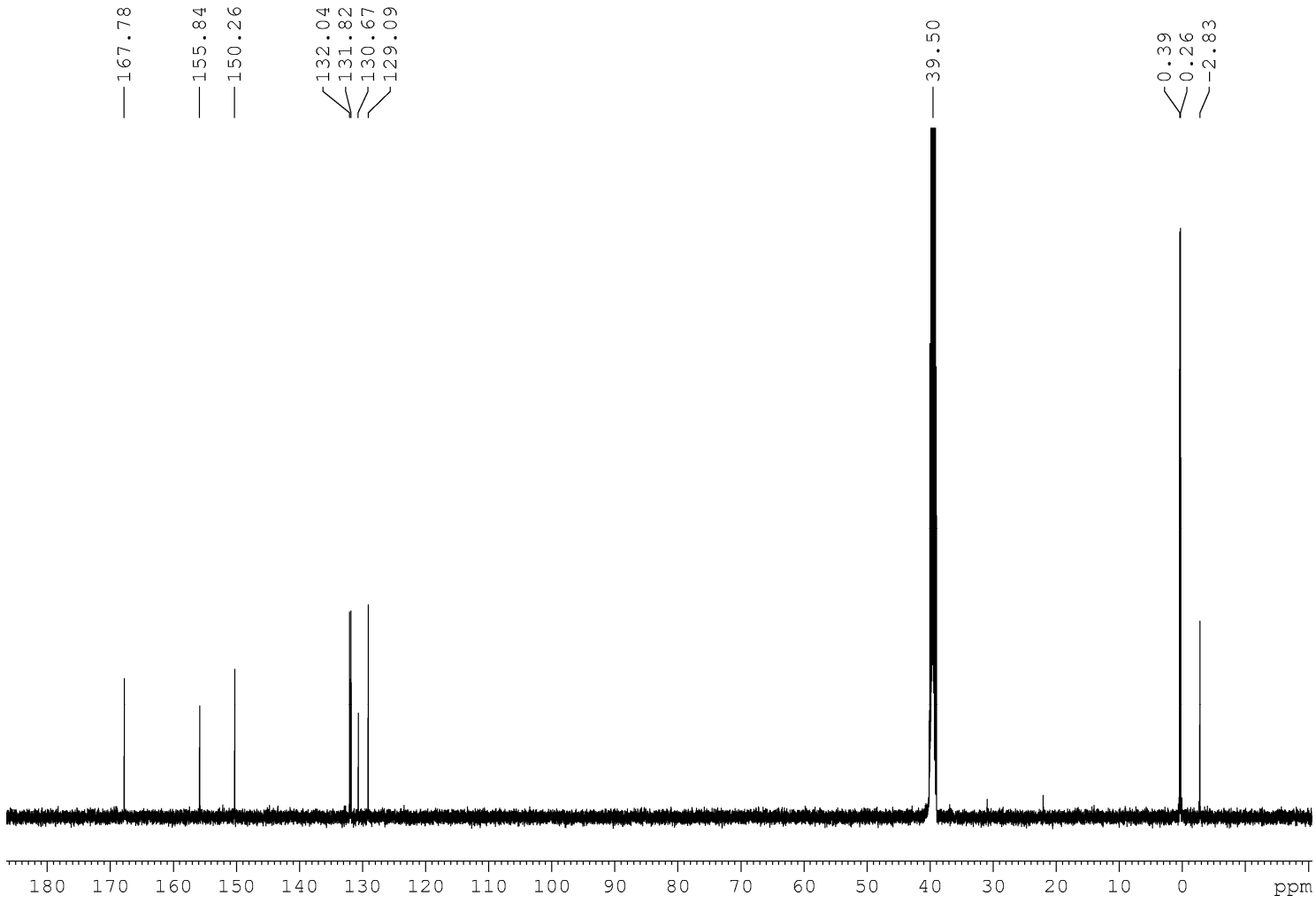

Figure S71 ^{13}C NMR spectrum of compound **34**.

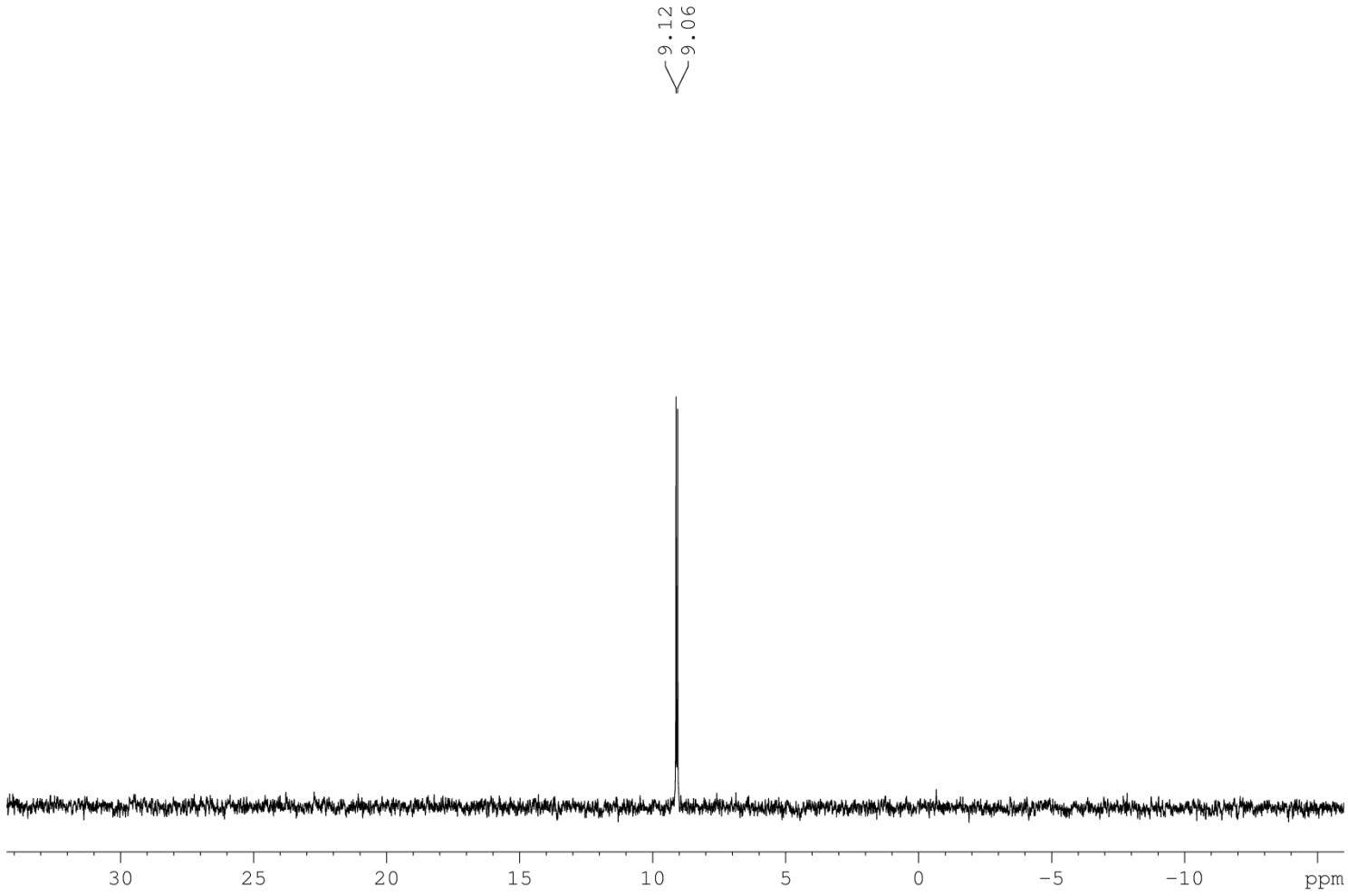

Figure S72 ^{29}Si NMR spectrum of compound **34**.