Supplementary Materials

Synthesis and Antiradical Activity of Isoquercitrin Esters with Aromatic Acids and Their Homologues

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Table S1. ^{13}C NMR data of the products **2** - **5** (DMSO-d_6, 303.2 K).

Comp.	2		3		4		5	
Atom number	δ	m	δ	m	δ	m	δ	m
2	156.38	s	156.51	s	156.38	s	156.33	s
3	132.94	s	133.17	s	133.10	s	133.11	s
4	177.40	s	177.43	s	177.40	s	177.42	s
4a	103.86	s	103.96	s	103.85	s	103.86	s
5	161.29	s	161.33	s	161.23	s	161.19	s
6	98.79	d	98.76	d	98.74	d	98.74	d
7	164.17	s	164.24	s	164.23	s	164.12	s
8	93.58	d	93.62	d	93.52	d	93.50	d
8a	156.30	s	156.41	s	156.32	s	156.30	s
1′	121.13	s	121.14	s	121.10	s	121.12	s
2′	116.12	d	116.26	d	116.18	d	116.24	d
3′	144.90	s	144.91	s	144.88	s	144.85	s
4′	148.51	s	148.60	s	148.56	s	148.53	s
5′	115.20	d	115.23	d	115.18	d	115.22	d
6′	121.47	d	121.57	d	121.51	d	121.52	d
1″	100.63	d	100.94	d	100.81	d	100.77	d
2″	74.02	d	73.98	d	73.97	d	74.02	d
3″	76.41	d	76.31	d	76.33	d	76.37	d
4″	70.08	d	69.87	d	70.03	d	70.04	d
5″	74.25	d	74.13	d	74.17	d	74.30	d
6″	63.74	t	63.45	t	63.18	t	63.37	t
1‴	165.37	s	170.73	s	171.64	s	165.78	s
2‴	-	-	39.95	t	34.91	t	117.58	d
3‴	-	-	-	-	30.11	t	144.35	d
ipso	129.56	s	134.15	s	140.30	s	133.85	s
ortho	128.82	d	129.23	d	128.05	d	128.21	d
meta	128.48	d	128.21	d	128.21	d	128.88	d
para	133.07	d	126.73	d	125.94	d	130.40	d

m - the multiplicity of ^{13}C NMR signals was resolved using multiplicity-edited gradient-enhanced ^{1}H-^{13}C HSQC

Table S2. ^{1}H NMR data of the products **2** - **3** (DMSO-d_6, 303.2 K).

Comp.	2				3			
Atom number	δ	n	m	*J* [Hz]	δ	n	m	*J* [Hz]
2	-	-	-	-	-	-	-	-
3	-	-	-	-	-	-	-	-
4	-	-	-	-	-	-	-	-
4a	-	-	-	-	-	-	-	-
5	-	-	-	-	-	-	-	-
6	6.185	1	d	2.1	6.207	1	d	2.1
7	-	-	-	-	-			
8	6.345	1	d	2.1	6.424	1	d	2.1
8a	-	-	-	-	-	-	-	-
1′	-	-	-	-	-	-	-	-
2′	7.530	1	d	2.2	7.583	1	d	2.2
3′	-	-	-	-	-	-	-	-
4′	-	-	-	-	-	-	-	-
5′	6.787	1	d	8.0	6.860	1	d	8.3
6′	7.514	1	dd	8.0, 2.2	7.562	1	dd	8.3, 2.2
1″	5.552	1	d	7.5	5.440	1	d	7.5
2″	3.34 ^{H}	1	m	-	3.300	1	dd	7.5, 8.6
3″	3.30 ^{H}	1	m	-	3.255	1	dd	8.6, 8.6
4″	3.26 ^{H}	1	m	-	3.153	1	dd	9.6, 8.6
5″	3.458	1	ddd	9.2, 6.7, 2.3	3.327	1	ddd	9.6, 6.4, 2.1
6″	4.399	1	dd	11.8, 2.3	4.197	1	dd	11.8, 2.1
	4.210	1	dd	11.8, 6.7	3.966	1	dd	11.8, 6.4
5-OH	12.613	1	s	-	12.645	1	s	-
7-OH	10.770	1	br.s.	-	10.835	1	s	-
3′-OH	9.267	1	br.s.	-	9.206	1	s	-
4′-OH	9.612	1	br.s.	-	9.705	1	s	-
2″-OH	5.387	1	br.s.	-	n.a.	-	-	-
3″-OH	5.233	1	br.s.	-	n.a.	-	-	-
4″-OH	5.198	1	br.s.	-	n.a.	-	-	-
1‴	-	-	-	-	-	-	-	-
2‴	-	-	-	-	3.402	1	d	15.5
	-	-	-	-	3.350	1	d	15.5
3‴	-	-	-	-	-	-	-	-
ipso	-	-	-	-	-	-	-	-
ortho	7.687	2	m	-	7.03 ^{H}	2	m	-
meta	7.338	2	m	-	7.20 ^{H}	2	m	-
para	7.551	1	m	-	7.19 ^{H}	1	m	-

^{H} - HSQC readout; n.a. – not asssigned due to signal broadening of the hydroxyl signals

Table S3. ^{1}H NMR data of prepared compounds **4** - **5** (DMSO-d_6, 303.2 K).

	4				**5**			
Atom number	**δ**	**n**	**m**	***J* [Hz]**	**δ**	**n**	**m**	***J* [Hz]**
2	-	-	-	-	-	-	-	-
3	-	-	-	-	-	-	-	-
4	-	-	-	-	-	-	-	-
4a	-	-	-	-	-	-	-	-
5	-	-	-	-	-	-	-	-
6	6.154	1	d	2.1	6.107	1	d	2.0
7	-	-	-	-	-	-	-	-
8	6.342	1	d	2.1	6.331	1	d	2.0
8a	-	-	-	-	-	-	-	-
1′	-	-	-	-	-	-	-	-
2′	7.544	1	d	2.3	7.551	1	d	2.2
3′	-	-	-	-	-	-	-	-
4′	-	-	-	-	-	-	-	-
5′	6.838	1	d	8.0	6.830	1	d	8.4
6′	7.530	1	dd	8.0, 2.3	7.526	1	dd	8.4, 2.2
1″	5.453	1	d	7.5	5.496	1	d	7.4
2″	3.29 ^{H}	1	m	-	3.310	1	m	-
3″	3.250	1	t	8.9	3.281	1	m	-
4″	3.145	1	t	8.9	3.213	1	dd	9.5, 8.9
5″	3.31 ^{H}	1	m	-	3.405	1	ddd	9.5, 6.7, 2.2
6″	4.184	1	dd	11.8, 2.1	4.304	1	dd	11.8, 2.2
	3.919	1	dd	11.8, 6.8	4.090	1	dd	11.8, 6.7
5-OH	12.611	1	s	-	12.608	1	s	-
7-OH	10.781	1	br.s.	-	10.775	1	s	-
3′-OH	9.221	1	br.s.	-	9.207	1	s	-
4′-OH	9.674	1	br.s.	-	9.659	1	s	-
2″-OH	5.354	1	br.s.	-	n.a.	-	-	-
3″-OH	5.156	1	br.s.	-	n.a.	-	-	-
4″-OH	5.156	1	br.s.	-	n.a.	-	-	-
1‴	-	-	-	-	-	-	-	-
2‴	2.340	2	m	-	6.343	1	d	16.0
3‴	2.575	2	m	-	7.413	1	d	16.0
ipso	-	-	-	-	-	-	-	-
ortho	7.017	2	m	-	7.509	2	m	-
meta	7.189	2	m	-	7.416	2	m	-
para	7.131	1	m	-	7.419	1	m	-

^{H} - HSQC readout; n.a. – not asssigned due to signal broadening of the hydroxyl signals

Table S4. ^{13}C NMR data of prepared compounds **6** - **11** (DMSO-d_6, 303.2 K).

	6		**7**		**8**		**9**		**10**		**11**	
Atom number	**δ**	**m**	**δ**	**m**	**δ**	**m**	**δ**	**m**	**δ**	**m**	**δ**	**m**
2	156.22	s	156.17	s	156.47	s	156.43	s	156.39	s	156.39	s
3	132.96	s	132.99	s	133.42	s	133.11	s	133.15	s	133.12	s
4	177.37	s	177.27	s	177.31	s	177.42	s	177.37	s	177.38	s
4a	103.76	s	103.61	s	103.94	s	103.88	s	103.80	s	103.83	s
5	161.26	s	161.21	s	161.22	s	161.25	s	161.24	s	161.23	s
6	98.85	d	98.92	d	98.74	d	98.73	d	98.82	d	98.73	d
7	164.38	s	164.75	s	164.21	s	164.18	s	164.43	s	164.29	s
8	93.54	d	93.57	d	93.56	d	93.53	d	93.59	d	93.52	d
8a	156.31	s	156.32	s	156.34	s	156.34	s	156.37	s	156.34	s
1′	121.06	s	121.03	s	120.96	s	121.11	s	121.09	s	121.08	s
2′	116.04	d	115.98	d	115.81	d	116.19	d	116.16	d	116.17	d
3′	144.91	s	144.92	s	144.84	s	144.88	s	144.88	s	144.88	s
4′	148.53	s	148.59	s	148.54	s	148.54	s	148.57	s	148.56	s
5′	115.19	d	115.18	d	115.32	d	115.18	d	115.17	d	115.16	d
6′	121.49	d	121.52	d	121.89	d	121.51	d	121.51	d	121.51	d
1″	100.65	d	100.79	d	101.44	d	100.80	d	100.92	d	100.88	d
2″	74.01	d	74.03	d	74.09	d	73.97	d	73.99	d	73.98	d
3″	76.43	d	76.42	d	76.35	d	76.34	d	76.34	d	76.33	d
4″	69.98	d	70.08	d	69.56	d	70.02	d	69.99	d	69.97	d
5″	74.43	d	74.44	d	74.31	d	74.19	d	74.18	d	74.20	d
6″	63.29	t	63.50	t	63.22	t	63.07	t	63.03	t	63.05	t
1‴	165.24	s	165.28	s	165.72	s	171.75	s	171.83	s	171.80	s
2‴	-	-	120.40	s	-	-	35.36	t	35.24	t	35.36	t
3‴	-	-	123.12	d	-	-	29.34	t	29.50	t	29.79	t
4‴	-	-	115.10	d	-	-	-	-	131.23	s	131.18	s
5‴	-	-	151.44	s	-	-	-	-	118.66	d	120.17	d
6‴	-	-	147.27	s	-	-	-	-	115.45	d	115.29	d
7‴	-	-	112.34	d	-	-	-	-	143.42	s	144.68	s
8‴	-	-	-	-	-	-	-	-	144.98	s	147.35	s
9‴	-	-	-	-	-	-	-	-	115.58	d	112.29	d
ipso	120.20	s	-	-	119.37	s	130.40	s	-	-	-	-
ortho	131.11	d	-	-	108.63	d	128.93	d	-	-	-	-
meta	115.19	d	-	-	145.48	s	115.05	d	-	-	-	-
para	161.88	s	-	-	138.44	s	155.50	s	-	-	-	-
6‴-MeO	-	-	55.45	q	-	-	-	-	-	-	-	-
8‴-MeO	-	-	-	-	-	-	-	-	-	-	55.51	q

m - the multiplicity of ^{13}C NMR signals was resolved using multiplicity-edited gradient-enhanced ^{1}H-^{13}C HSQC

Table S5. ^{1}H NMR data of prepared compounds **6** - **8** (DMSO-d_6, 303.2 K).

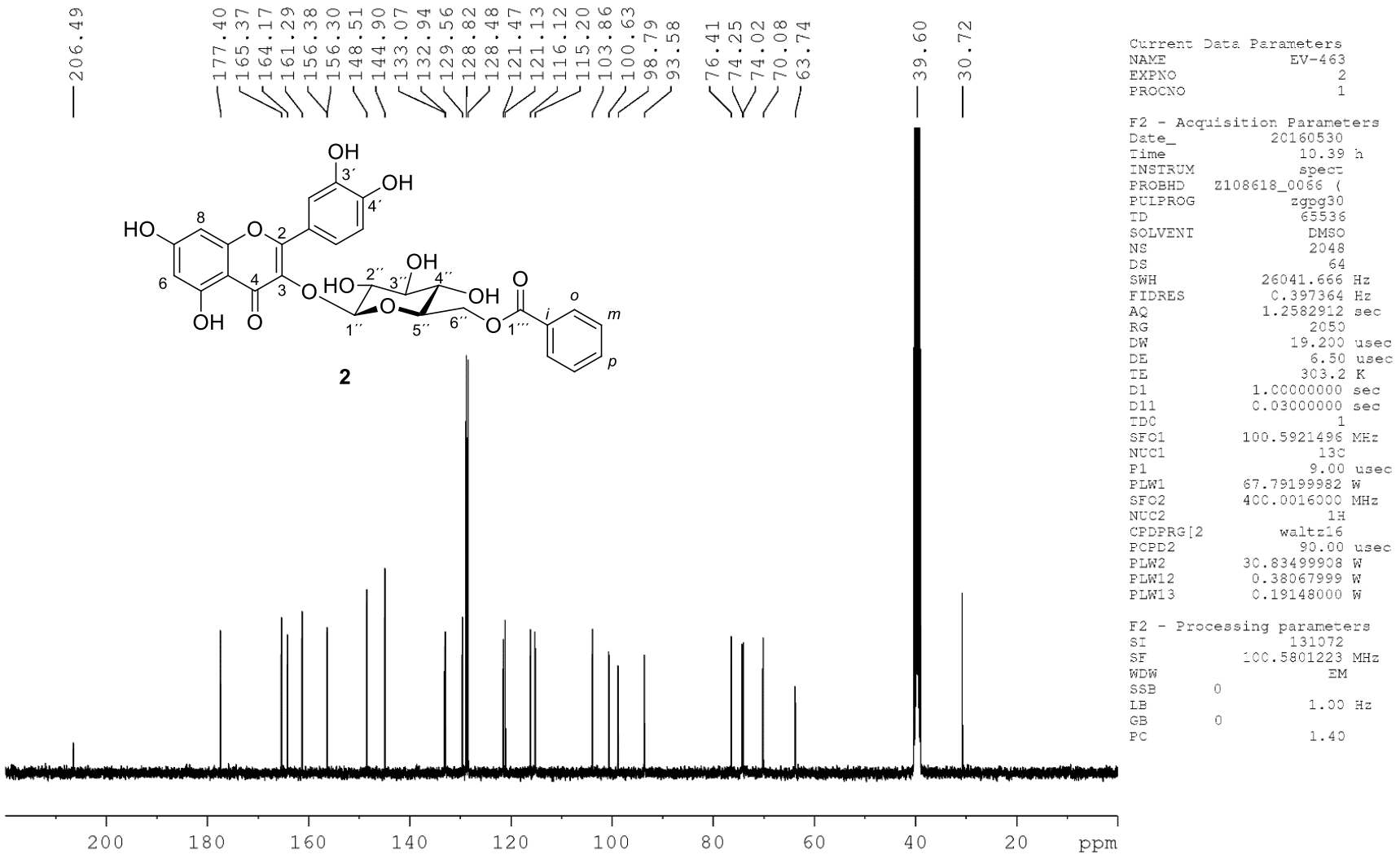
Comp.	6				7				8			
Atom number	δ	n	m	*J* [Hz]	δ	n	m	*J* [Hz]	δ	n	m	*J* [Hz]
2	-	-	-	-	-	-	-	-	-	-	-	-
3	-	-	-	-	-	-	-	-	-	-	-	-
4	-	-	-	-	-	-	-	-	-	-	-	-
4a	-	-	-	-	-	-	-	-	-	-	-	-
5	-	-	-	-	-	-	-	-	-	-	-	-
6	6.198	1	d	2.1	6.163	1	d	2.0	6.176	1	d	2.1
7	-	-	-	-	-	-	-	-	-	-	-	-
8	6.355	1	d	2.1	6.327	1	d	2.0	6.360	1	d	2.1
8a	-	-	-	-	-	-	-	-	-	-	-	-
1′	-	-	-	-	-	-	-	-	-	-	-	-
2′	7.518	1	d	2.2	7.504	1	d	2.2	7.436	1	d	2.2
3′	-	-	-	-	-	-	-	-	-	-	-	-
4′	-	-	-	-	-	-	-	-	-	-	-	-
6′	7.518	1	d	2.2	7.504	1	d	2.2	7.436	1	d	2.2
1′	-	-	-	-	-	-	-	-	-	-	-	-
5′	6.789	1	d	8.2	6.766	1	d	8.4	6.724	1	d	8.4
6′	7.528	1	dd	8.2, 2.2	7.527	1	dd	8.4, 2.2	7.570	1	dd	8.4, 2.2
1″	5.558	1	d	7.6	5.535	1	d	7.6	5.434	1	d	7.5
2″	3.331	1	dd	8.8, 7.6	3.318	1	dd	9.0, 7.6	3.288	1	m	-
3″	3.288	1	dd	8.8, 8.6	3.287	1	dd	9.0, 8.5	3.288	1	m	-
4″	3.237	1	dd	9.6, 8.6	3.232	1	dd	9.6, 8.5	3.360	1	dd	9.7, 8.3
5″	3.419	1	ddd	9.6, 6.6, 2.2	3.438	1	ddd	9.6, 6.7, 2.2	3.428	1	ddd	9.7, 4.5, 2.0
6″	4.358	1	dd	11.8, 2.2	4.386	1	dd	11.8, 2.2	4.257	1	dd	11.9, 2.0
	4.094	1	dd	11.8, 6.6	4.117	1	dd	11.8, 6.7	4.169	1	dd	11.9, 4.5
5-OH	12.626			-	12.582	1	s	-	12.540	1	s	-
7-OH	9.949	3	br.s	-	n.a.	-	-	-	n.a.	-	-	-
4′-OH									n.a.	-	-	-
3′-OH									n.a.	-	-	-
2″-OH	5.189	2	br.s	-					n.a.	-	-	-
3″-OH				-					n.a.	-	-	-
4″-OH	5.351	1	br.s	-					n.a.	-	-	-
3‴	-	-	-	-	7.179	1	dd	8.2, 2.0	-	-	-	-
4‴	-	-	-	-	6.690	1	d	8.2	-	-	-	-
7‴	-	-	-	-	7.262	1	d	2.0	-	-	-	-
ortho	7.559	2	m	-	-	-	-	-	6.889	2	s	-
meta	6.699	2	m	-	-	-	-	-	-	-	-	-
5‴-OH	-	-	-	-	n.a.	-	-	-	-	-	-	-
6‴-MeO	-	-	-	-	3.696	3	s	-	-	-	-	-
meta-OH	-	-	-	-	-	-	-	-	n.a.	-	-	-
para-OH	-	-	-	-	-	-	-	-	n.a.	-	-	-

n.a. - not assigned due to the broadening of the hydroxyl signals

Table S6. ^{1}H NMR data of prepared compounds **9** - **11** (DMSO-d_6, 303.2 K).

Comp.	9				10				11			
Atom number	δ	n	m	*J* [Hz]	δ	n	m	*J* [Hz]	δ H	n	m	*J* [Hz]
2	-	-	-	-	-	-	-	-	-	-	-	-
3	-	-	-	-	-	-	-	-	-	-	-	-
4	-	-	-	-	-	-	-	-	-	-	-	-
4a	-	-	-	-	-	-	-	-	-	-	-	-
5	-	-	-	-	-	-	-	-	-	-	-	-
6	6.167	1	d	2.1	6.168	1	d	2.1	6.154	1	d	2.1
7	-	-	-	-	-	-	-	-	-	-	-	-
8	6.363	1	d	2.1	6.367	1	d	2.1	6.352	1	d	2.1
8a	-	-	-	-	-	-	-	-	-	-	-	-
1′	-	-	-	-	-	-	-	-	-	-	-	-
2′	7.540	1	m	-	7.534	1	m	-	7.537	1	m	-
3′	-	-	-	-	-	-	-	-	-	-	-	-
4′	-	-	-	-	-	-	-	-	-	-	-	-
5′	6.835	1	d	8.0	6.828	1	m	-	6.831	1	m	-
6′	7.530	1	m	-	7.529	1	m	-	7.530	1	m	-
1″	5.444	1	d	7.4	5.415	1	d	7.6	5.436	1	d	7.6
2″	3.28 [H]	1	m	-	3.279	1	dd	9.0, 7.6	3.285	1	m	-
3″	3.26 [H]	1	m	-	3.242	1	dd	9.0, 8.6	3.248	1	t	8.7
4″	3.143	1	m	-	3.143	1	dd	9.8, 8.6	3.154	1	dd	8.7, 9.8
5″	3.30 [H]	1	m	-	3.308	1	ddd	9.8, 6.7, 2.1	3.320	1	ddd	9.8, 6.7, 2.2
6″	4.175	1	dd	11.8, 2.0	4.166	1	dd	11.8, 2.1	4.176	1	dd	11.8, 2.2
	3.913	1	dd	11.8, 6.8	3.924	1	dd	11.8, 6.7	3.937	1	dd	11.8, 6.7
5-OH	12.611	1	s	-	12.599	1	s	-	12.603	1	s	-
7-OH	10.807	1	s	-	n.a.	-	-	-	n.a.	-	-	-
4'-OH	9.678	1	s	-	n.a.	-	-	-				
3′-OH	9.186	1	s	-	n.a.	-	-	-				
2″-OH	5.349	1	d	4.4	5.339	1	br.s.	-				
3″-OH	5.157	1	d	5.9	5.142	2	br.s.	-				
4″-OH	5.143	1	d	4.8								
2‴	2.262	2	m	-	2.242	2	m	-	2.301	2	m	-
3‴	2.460	2	m	-	2.423	2	m	-	2.486	2	m	-
4‴	-	-	-	-	-	-	-	-	-	-	-	-
5‴	-	-	-	-	6.254	1	dd	8.0, 2.1	6.404	1	dd	8.0, 2.0
6‴	-	-	-	-	6.554	1	d	8.0	6.595	1	d	8.0
9‴	-	-	-	-	6.480	1	d	2.1	6.630	1	d	2.0
ipso	-	-	-	-	n.a.	-	-	-	-	-	-	-
ortho	6.807	2	m	-	n.a.	-	-	-	-	-	-	-
meta	6.591	2	m	-	-	-	-	-	-	-	-	-
5‴-OH	-	-	-	-	6.480	1	d	2.1	-	-	-	-
para-OH	9.088	1	s	-	-	-	-	-	-	-	-	-
7‴-OH	-	-	-	-	n.a.	-	-	-	n.a.	-	-	-
8‴-OH	-	-	-	-	n.a.	-	-	-	-	-	-	-
8‴-MeO	-	-	-	-	-	-	-	-	3.703	3	s	-

[H] - HSQC readout; n.a. - not assigned due to the broadening of the hydroxyl signals

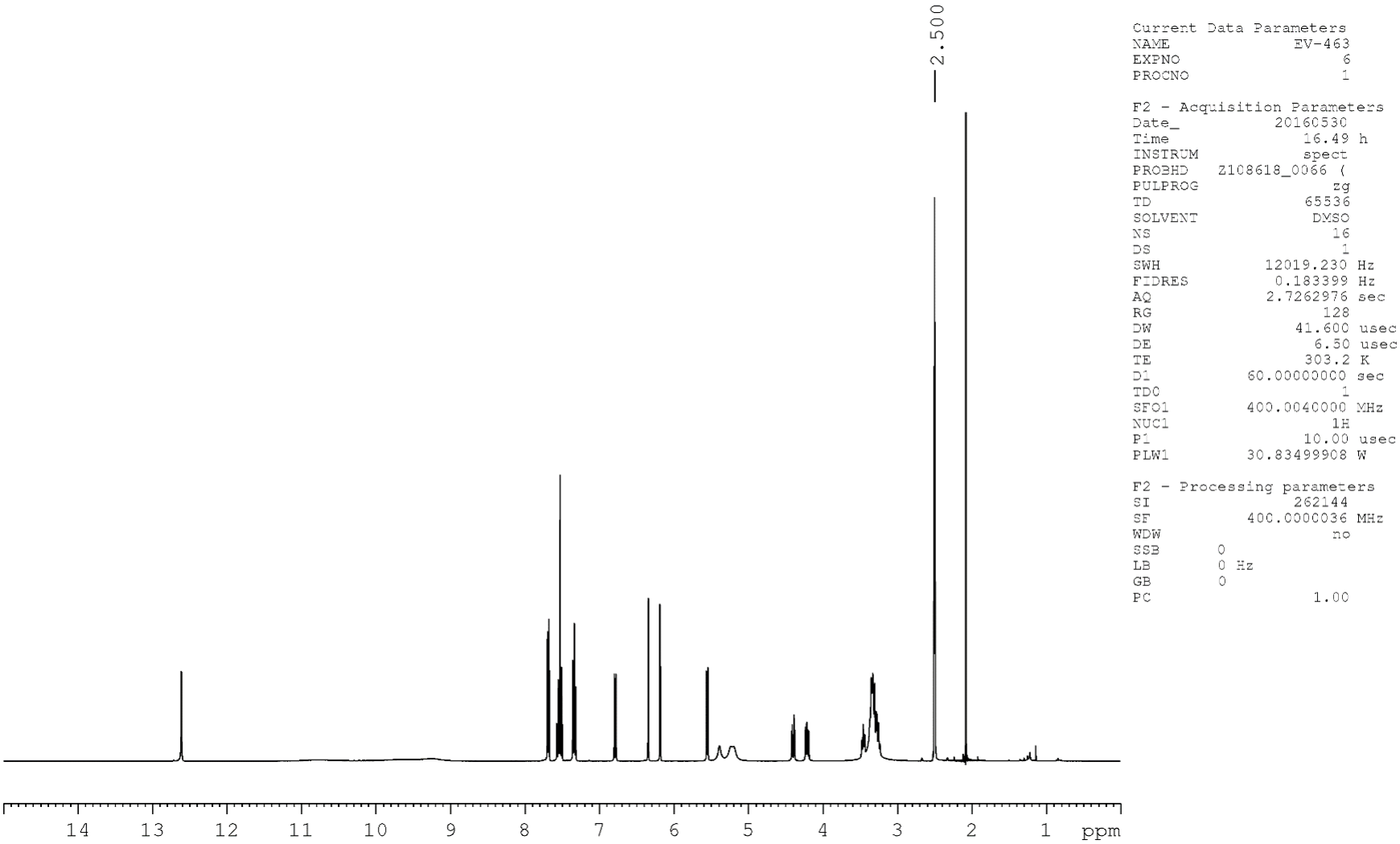



```
Current Data Parameters
NAME                  EV-463
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160530
Time                 10.39 h
INSTRUM                spect
PROBHD    Z108618_0066 (
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
NS                      2048
DS                        64
SWH               26041.666 Hz
FIDRES             0.397364 Hz
AQ                1.2582912 sec
RG                     2050
DW                   19.200 usec
DE                    6.50 usec
TE                   303.2 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                        1
SFO1            100.5921496 MHz
NUC1                     13C
P1                    9.00 usec
PLW1            67.79199982 W
SFO2            400.0016000 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2            30.83499908 W
PLW12            0.38067999 W
PLW13            0.19148000 W

F2 - Processing parameters
SI                     131072
SF              100.5801223 MHz
WDW                        EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

Figure S1. ^{13}C NMR spectrum of compound **2** (DMSO-d_6, 303.2 K).




```
Current Data Parameters
NAME                  EV-463
EXPNO                      6
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160530
Time                  16.49 h
INSTRUM               spect
PROBHD    Z108618_0066 (
PULPROG                   zg
TD                     65536
SOLVENT                DMSO
NS                        16
DS                         1
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                2.7262976 sec
RG                       128
DW                   41.600 usec
DE                    6.50 usec
TE                   303.2 K
D1              60.00000000 sec
TD0                        1
SFO1            400.0040000 MHz
NUC1                      1H
P1                   10.00 usec
PLW1            30.83499908 W

F2 - Processing parameters
SI                    262144
SF              400.0000036 MHz
WDW                       no
SSB         0
LB          0 Hz
GB          0
PC                     1.00
```

Figure S2. ^{1}H NMR spectrum of compound **2** (DMSO-d_6, 303.2 K).

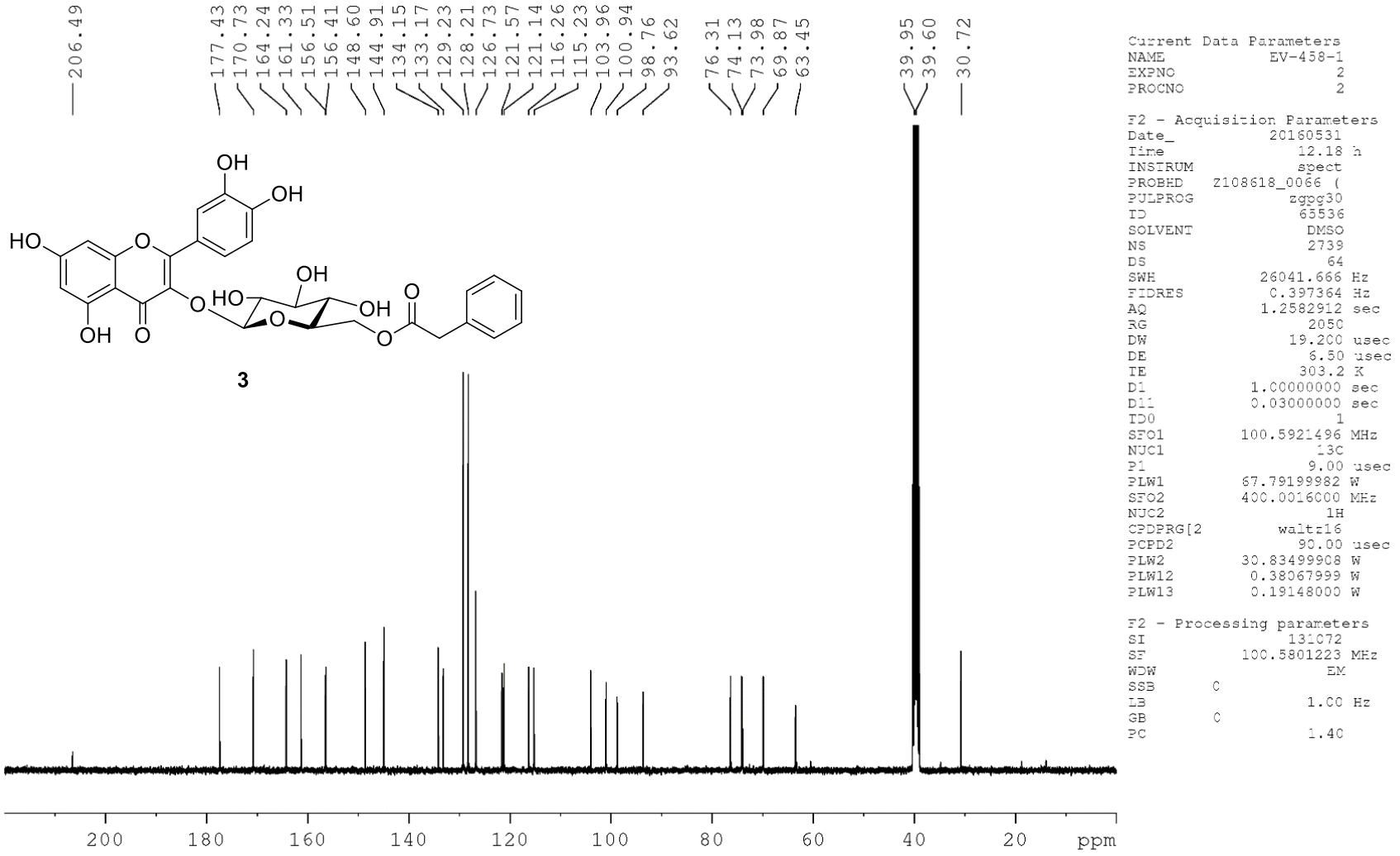
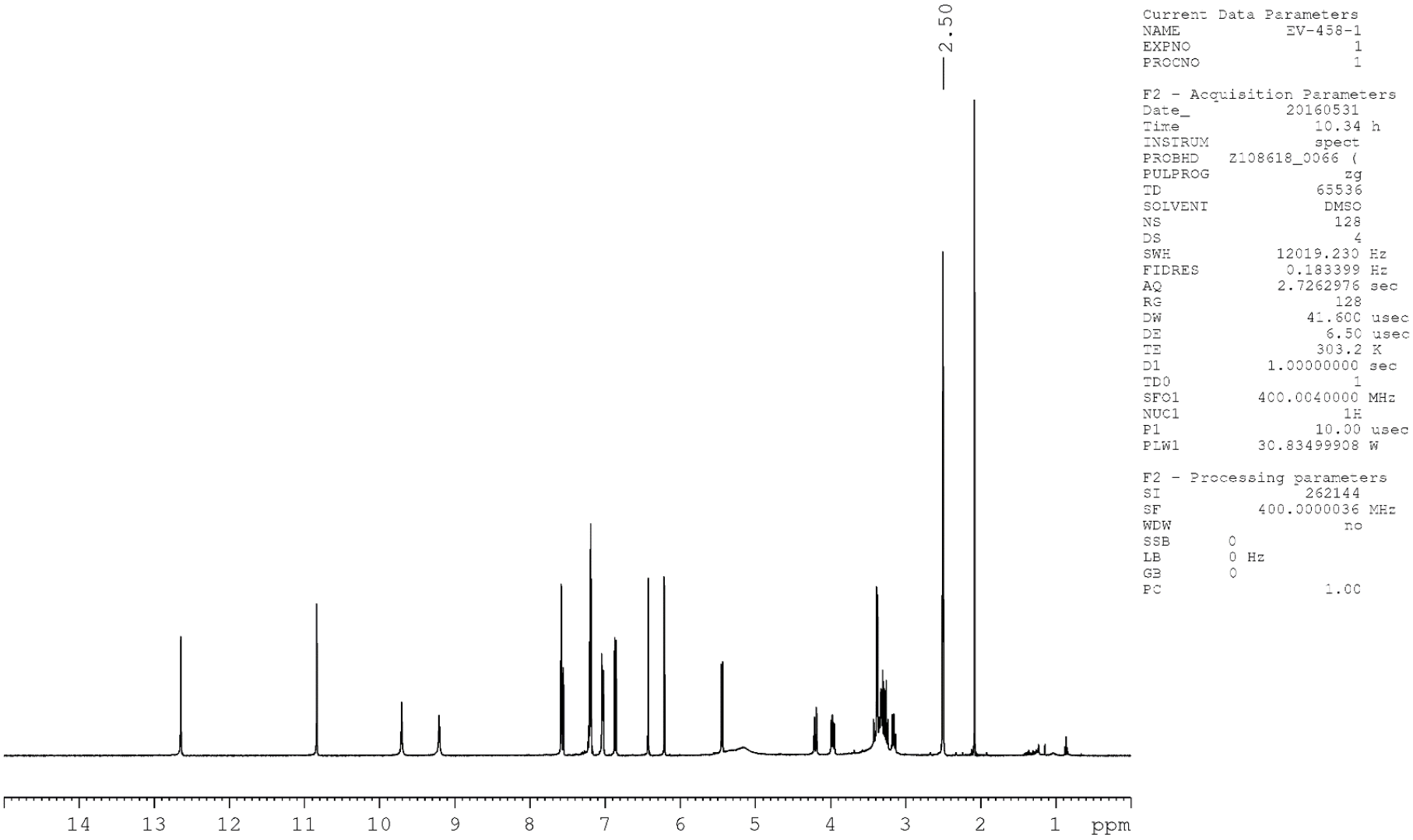


Figure S3. ^{13}C NMR spectrum of compound **3** (DMSO-d_6, 303.2 K).




```
Current Data Parameters
NAME                  EV-458-1
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20160531
Time                     10.34 h
INSTRUM                  spect
PROBHD    Z108618_0066 (
PULPROG                     zg
TD                       65536
SOLVENT                   DMSO
NS                         128
DS                           4
SWH                  12019.230 Hz
FIDRES                0.183399 Hz
AQ                    2.7262976 sec
RG                         128
DW                        41.600 usec
DE                         6.50 usec
TE                        303.2 K
D1                   1.00000000 sec
TD0                          1
SFO1                400.0040000 MHz
NUC1                        1H
P1                        10.00 usec
PLW1                30.83499908 W

F2 - Processing parameters
SI                       262144
SF                  400.0000036 MHz
WDW                         no
SSB          0
LB           0 Hz
GB           0
PC                        1.00
```

Figure S4. ^{1}H NMR spectrum of compound **3** (DMSO-d_6, 303.2 K).

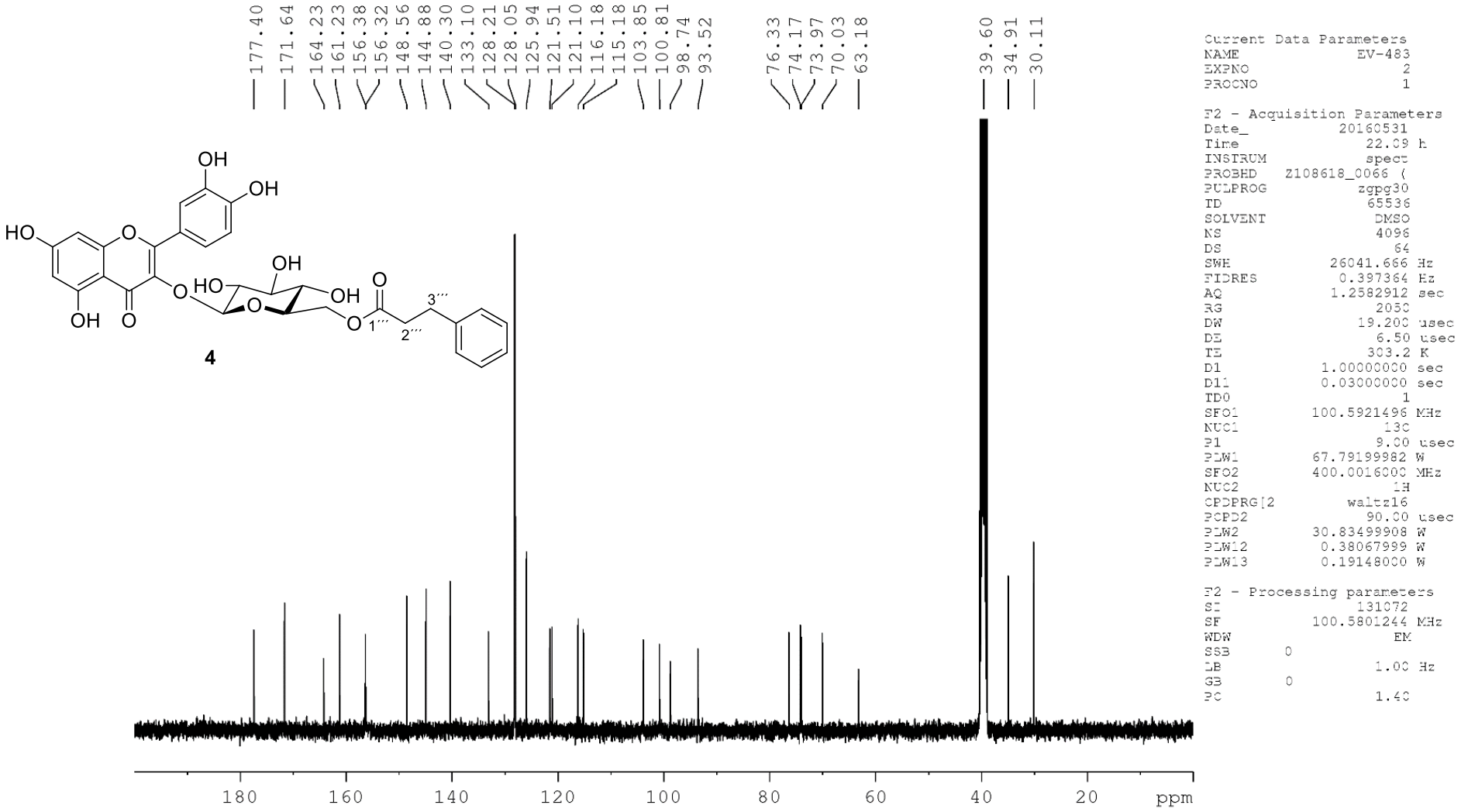


Figure S5. ^{13}C NMR spectrum of compound **4** (DMSO-d_6, 303.2 K).

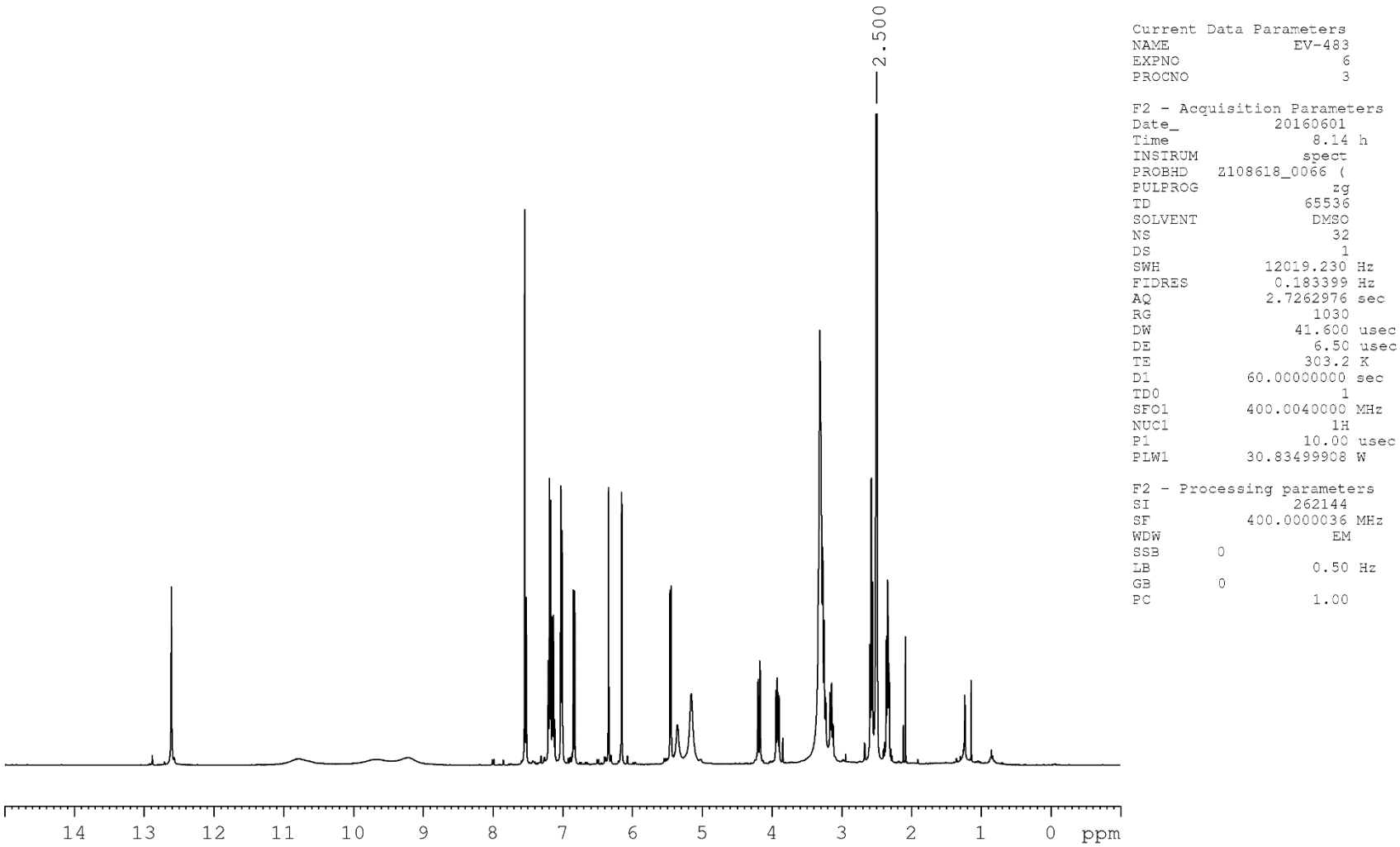


Figure S6. ^{1}H NMR spectrum of compound **4** (DMSO-d_6, 303.2 K).

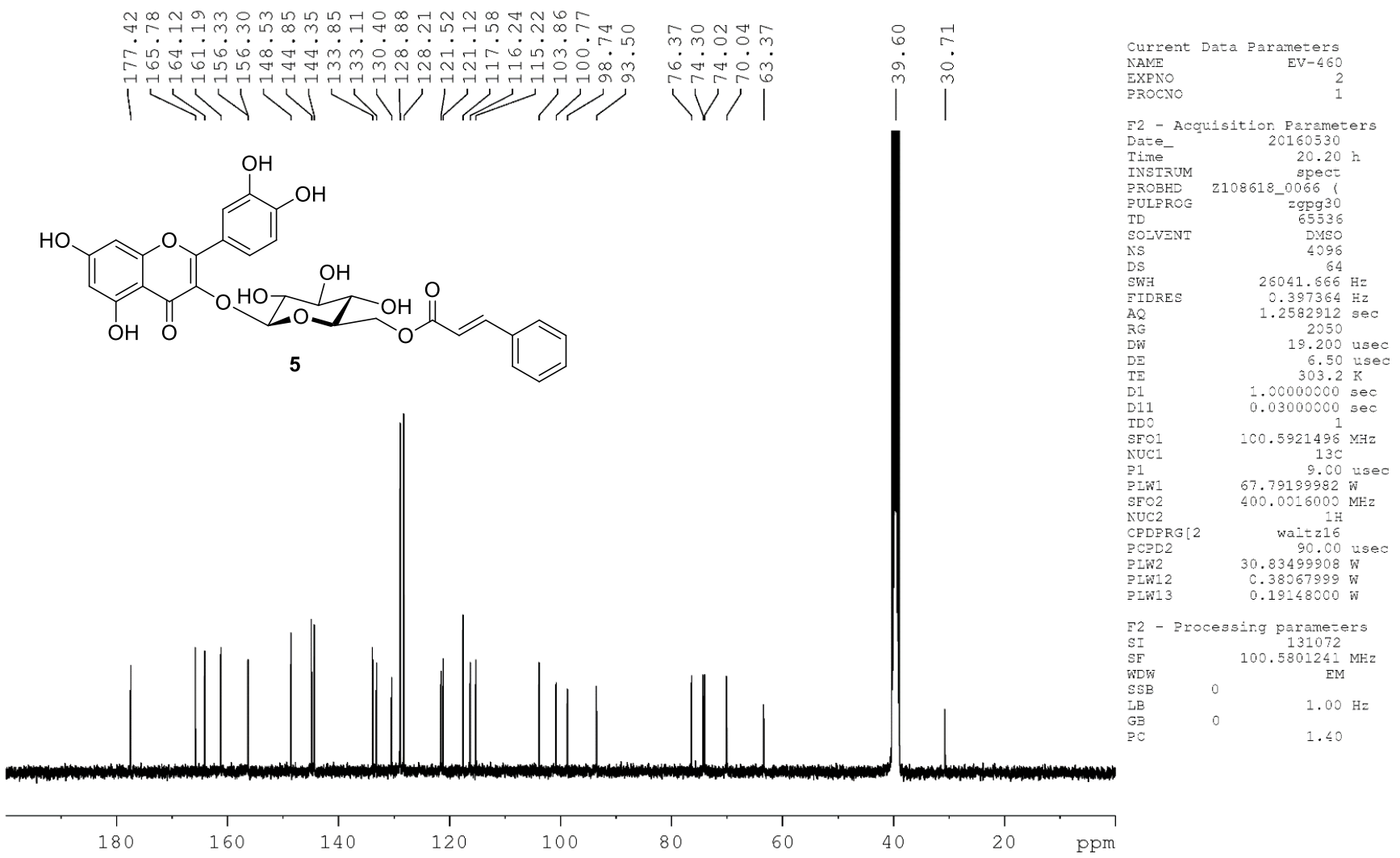
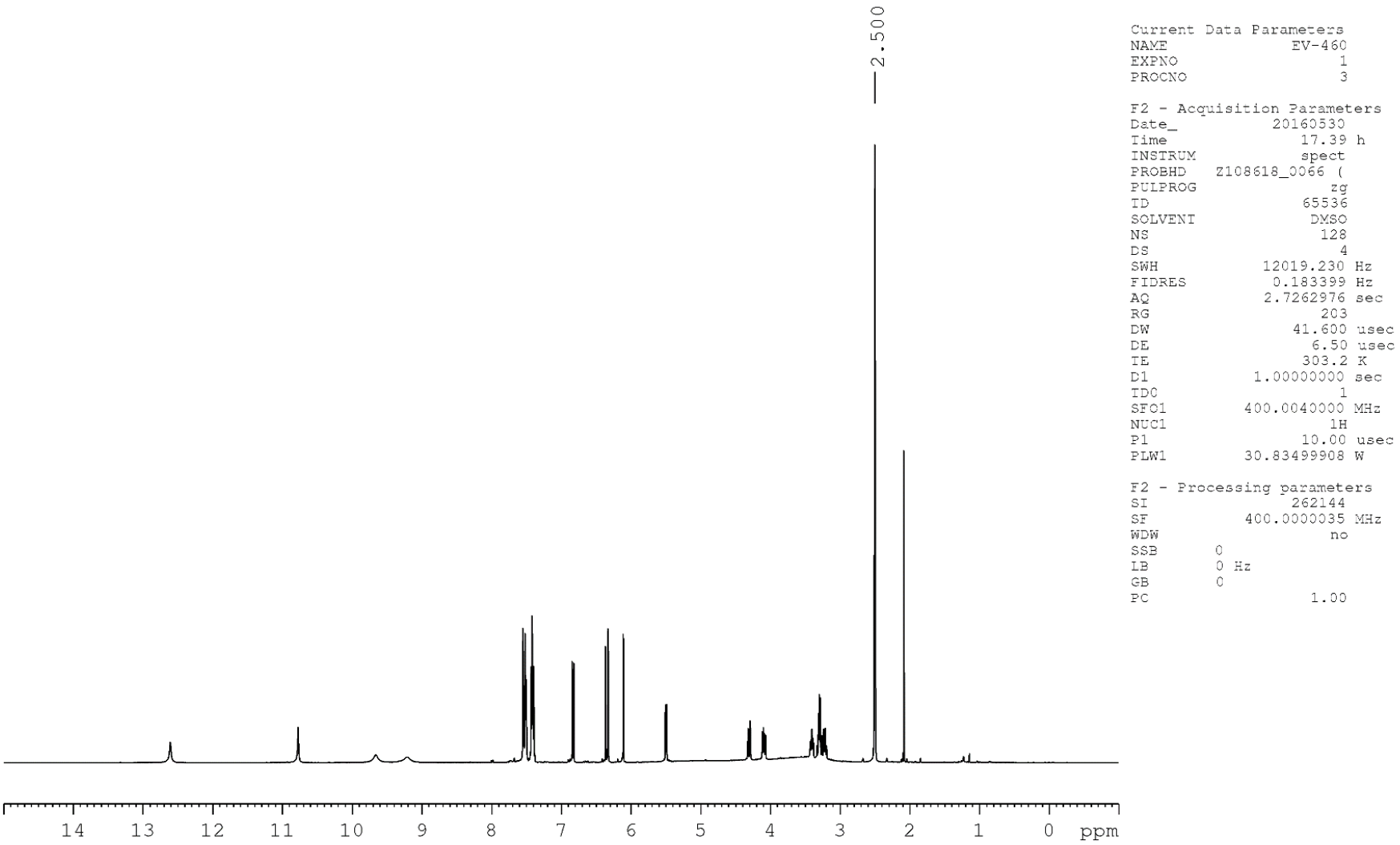


Figure S7. ^{13}C NMR spectrum of compound **5** (DMSO-d_6, 303.2 K).




```
Current Data Parameters
NAME                 EV-460
EXPNO                     1
PROCNO                    3

F2 - Acquisition Parameters
Date_              20160530
Time                 17.39 h
INSTRUM                spect
PROBHD    Z108618_0066 (
PULPROG                  zg
TD                    65536
SOLVENT                DMSO
NS                      128
DS                        4
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                2.7262976 sec
RG                      203
DW                   41.600 usec
DE                    6.50 usec
TE                   303.2 K
D1                1.00000000 sec
TD0                       1
SFO1            400.0040000 MHz
NUC1                     1H
P1                   10.00 usec
PLW1            30.83499908 W

F2 - Processing parameters
SI                     262144
SF              400.0000035 MHz
WDW                       no
SSB       0
LB        0 Hz
GB        0
PC                     1.00
```

Figure S8. ^{1}H NMR spectrum of compound **5** (DMSO-d_6, 303.2 K).

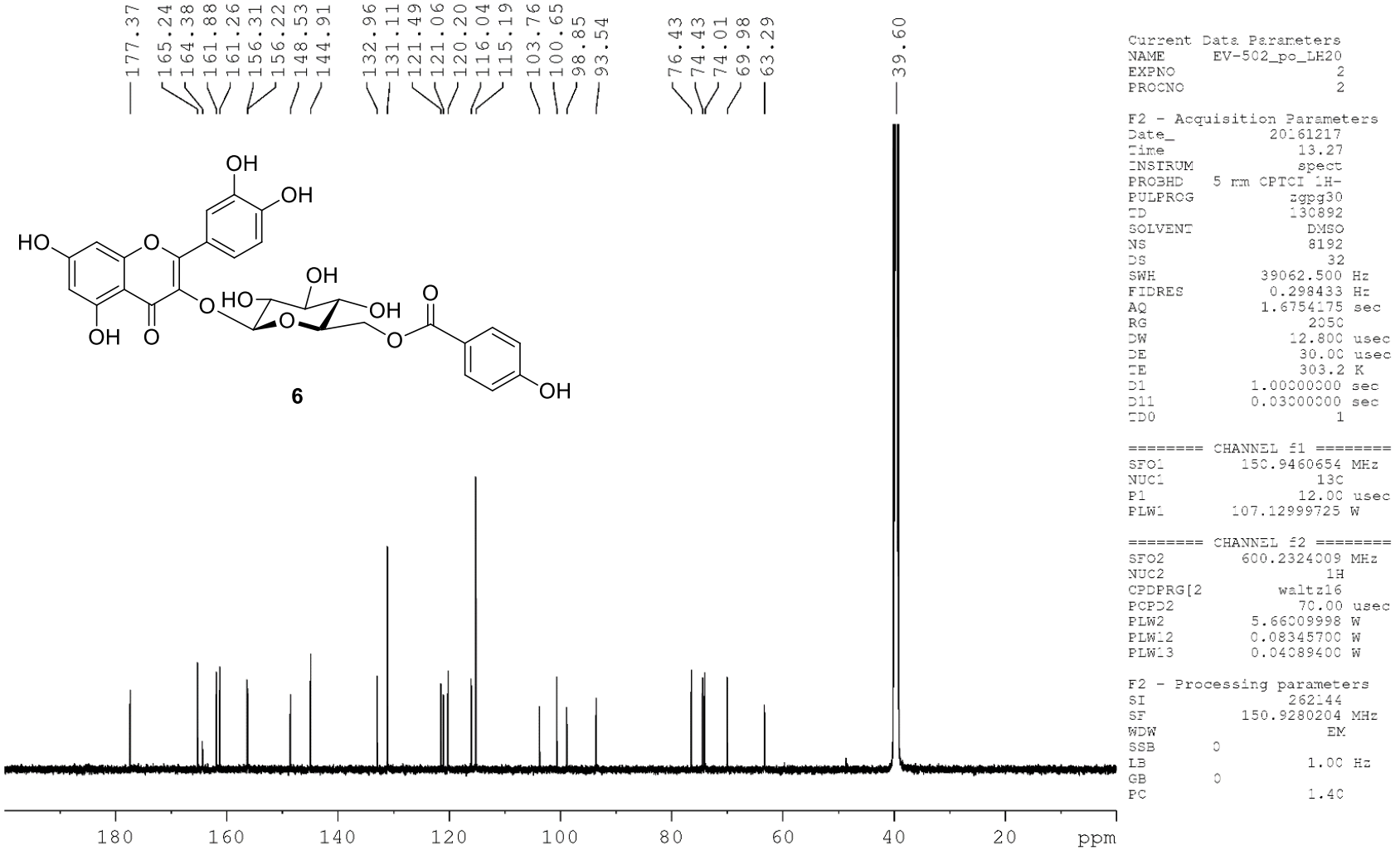


Figure S9. ^{13}C NMR spectrum of compound **6** (DMSO-d_6, 303.2 K).

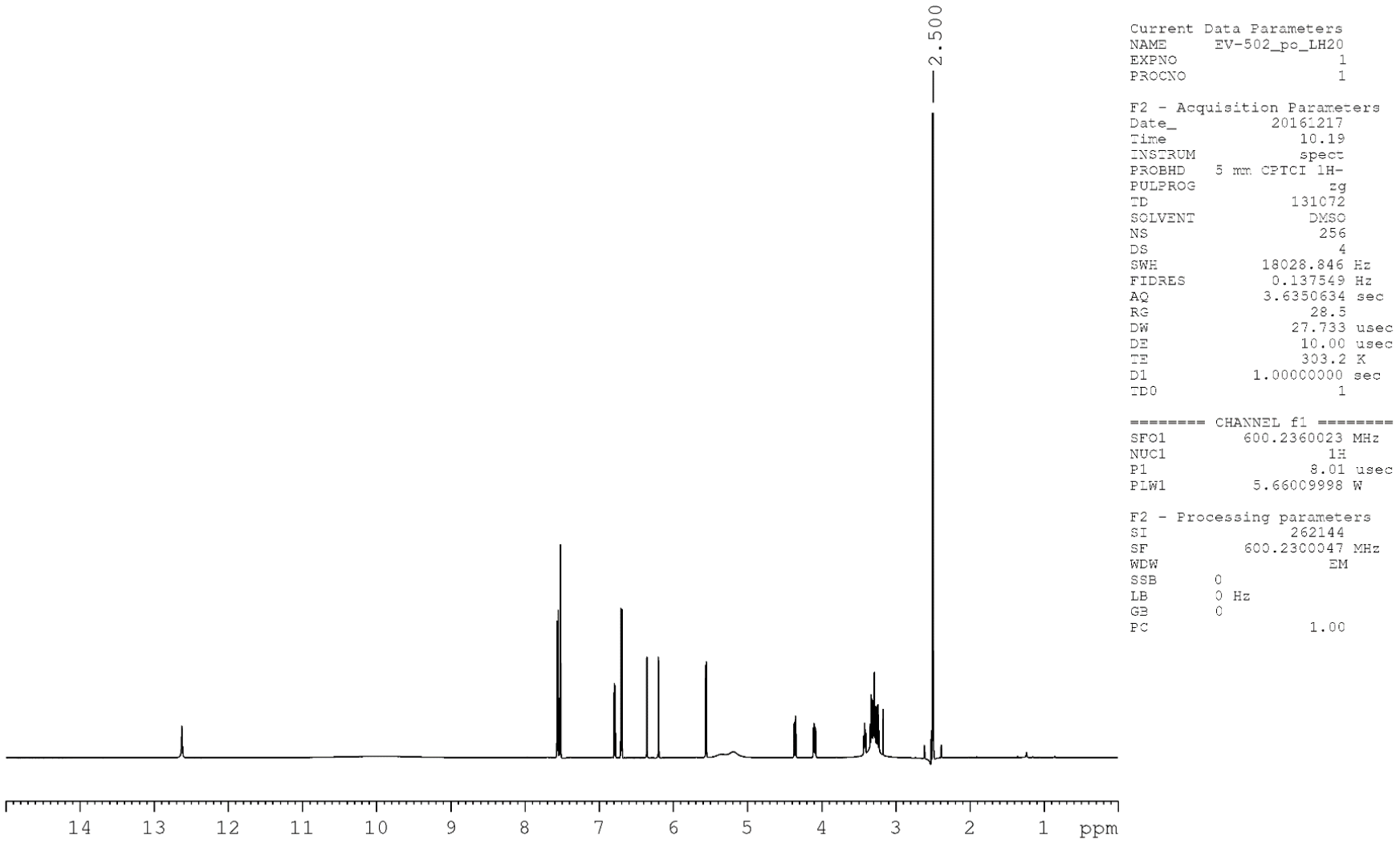


Figure S10. ^{1}H NMR spectrum of compound **6** (DMSO-d_6, 303.2 K).

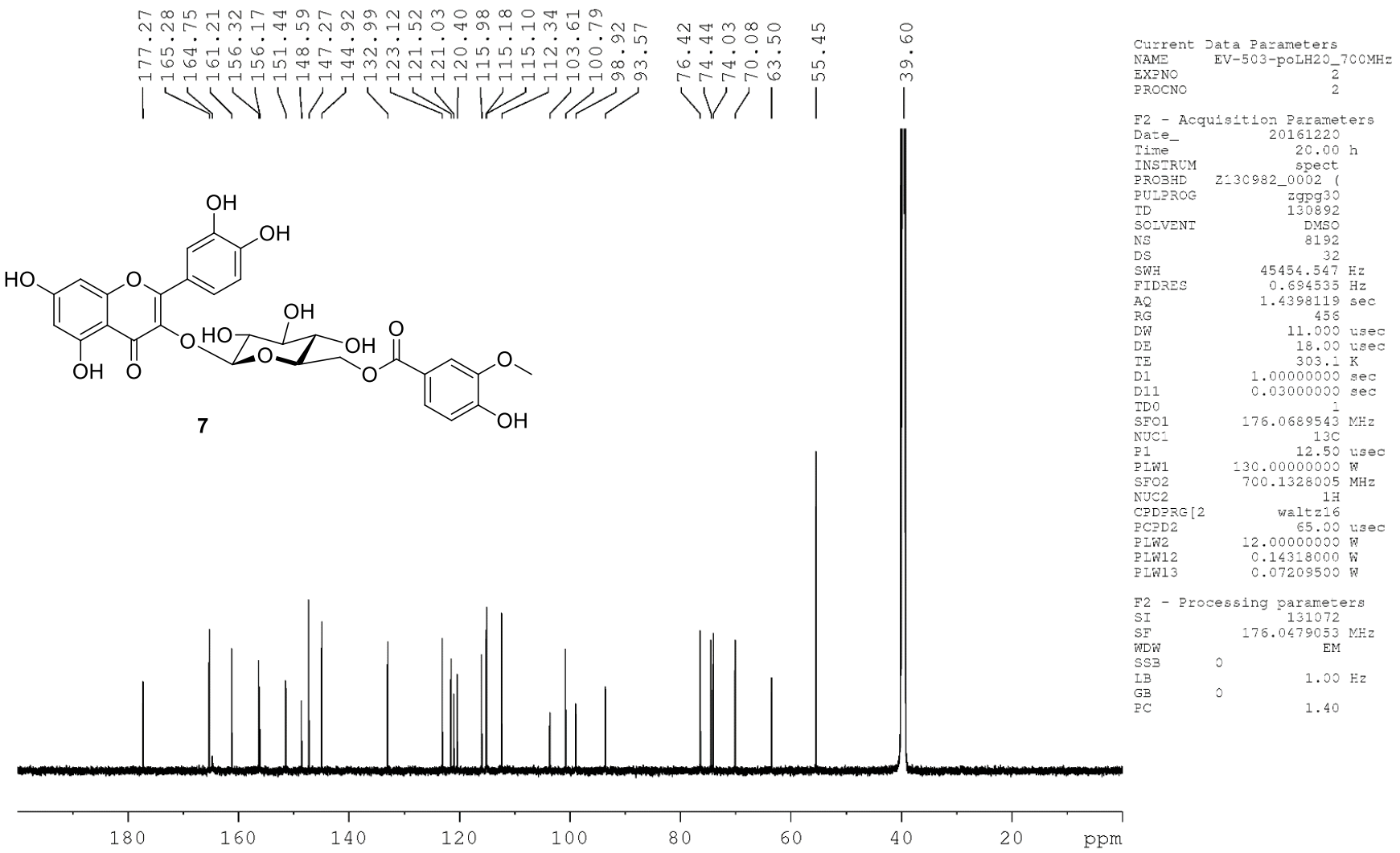


Figure S11. ^{13}C NMR spectrum of compound **7** (DMSO-d_6, 303.2 K).

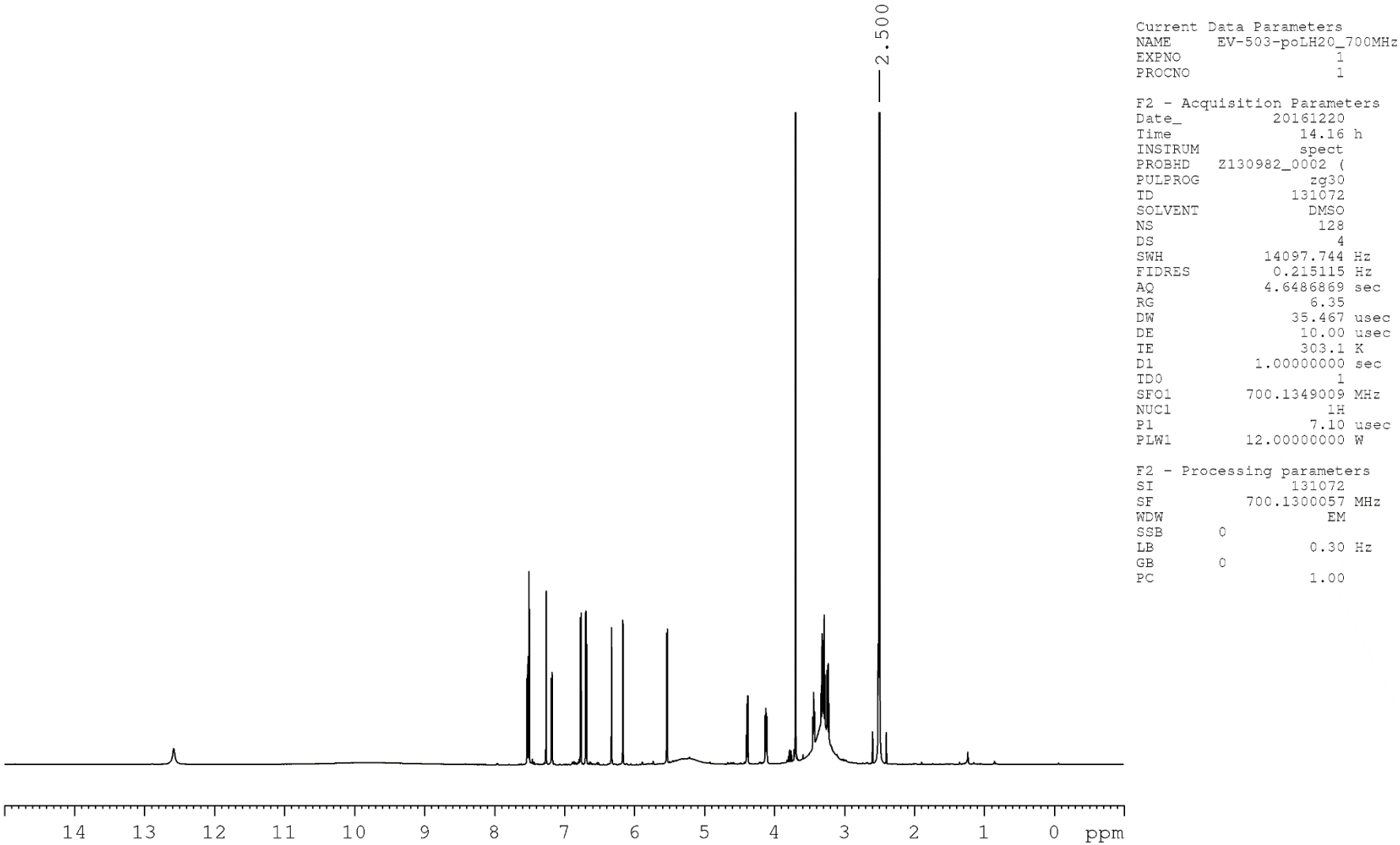


Figure S12. ^{1}H NMR spectrum of compound **7** (DMSO-d_6, 303.2 K).

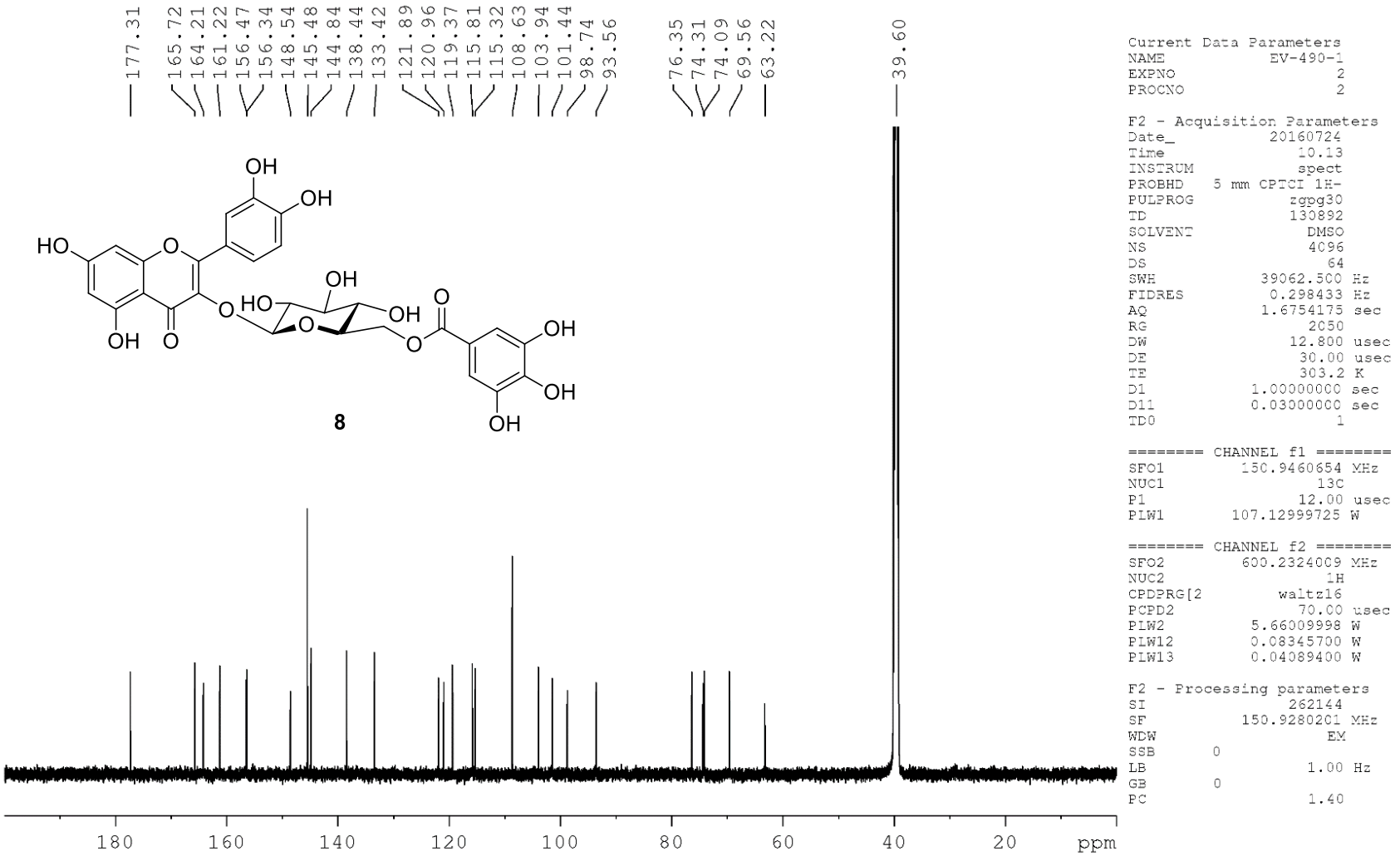


Figure S13. ^{13}C NMR spectrum of compound **8** (DMSO-d_6, 303.2 K).

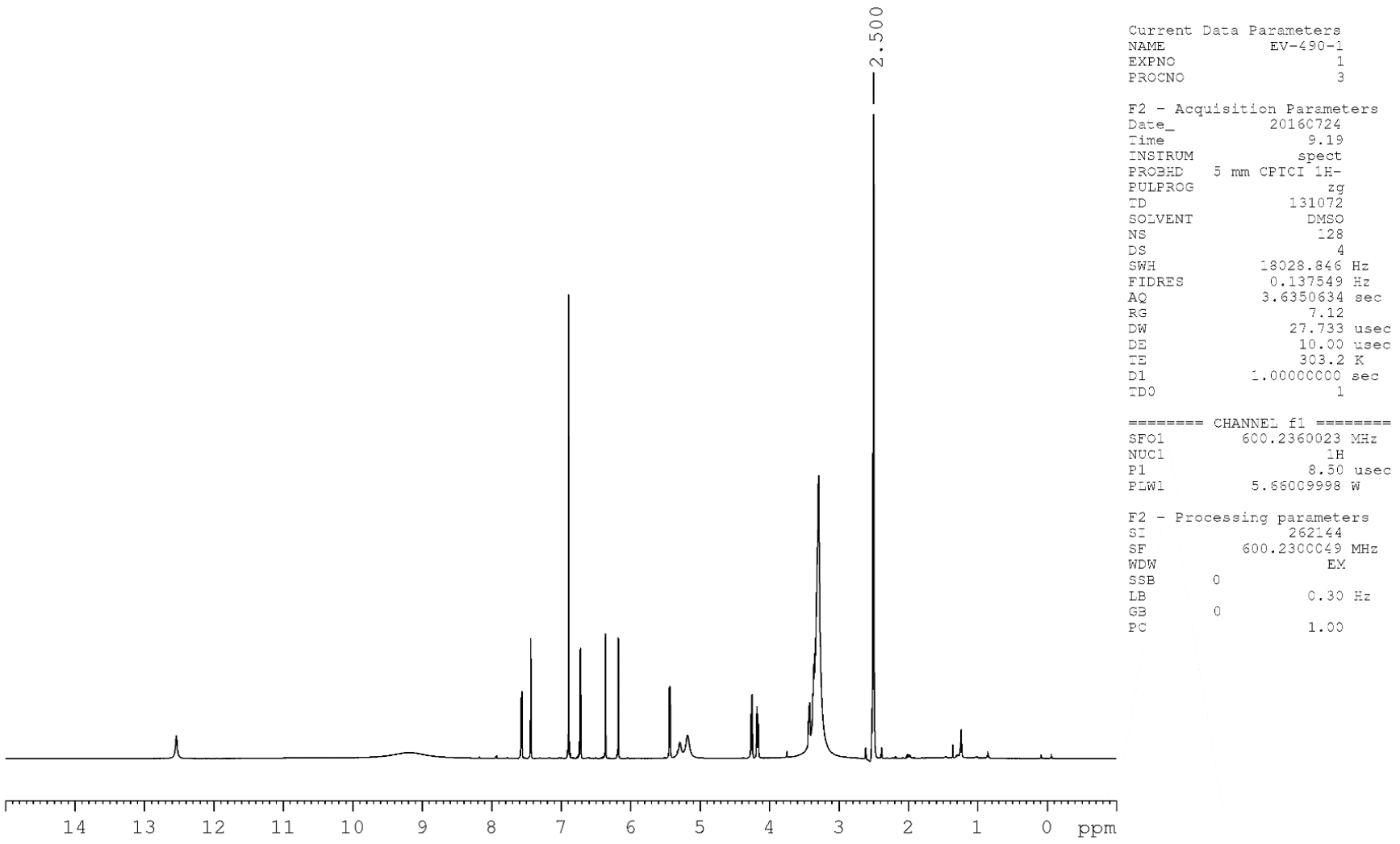


Figure S14. ^{1}H NMR spectrum of compound **8** (DMSO-d_6, 303.2 K).

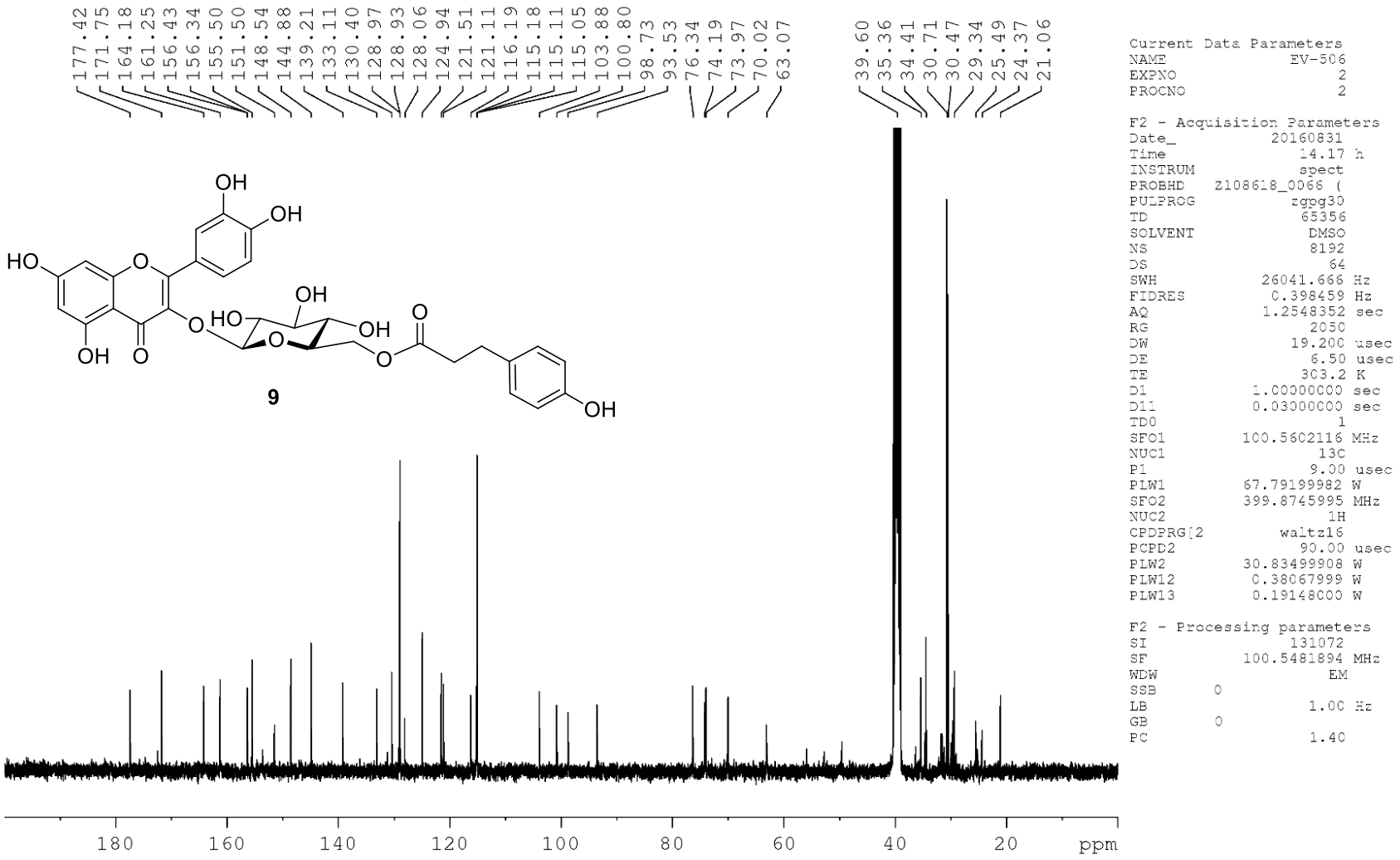
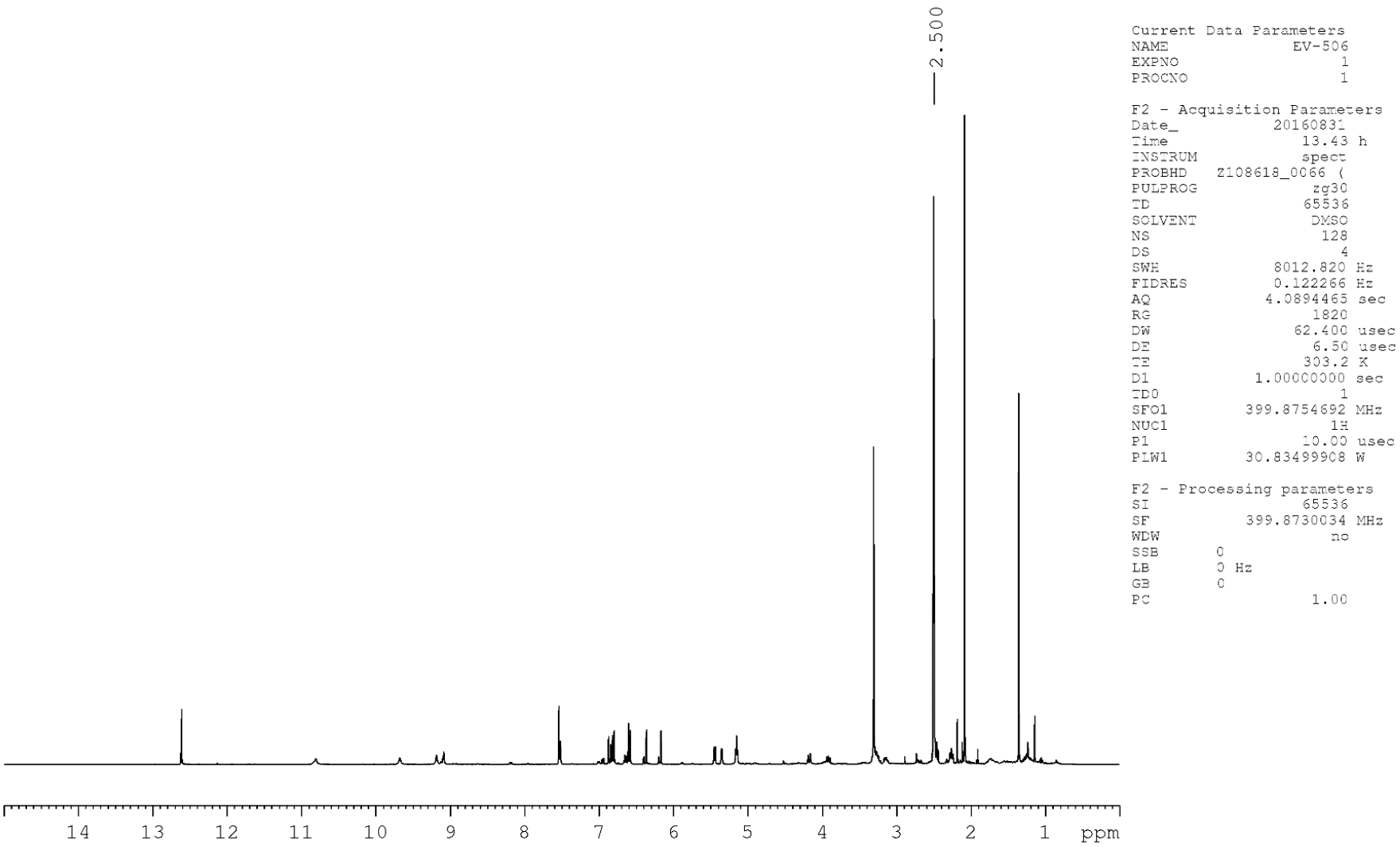


Figure S15. ^{13}C NMR spectrum of compound **9** (DMSO-d_6, 303.2 K).




```
Current Data Parameters
NAME                  EV-506
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160831
Time                  13.43 h
INSTRUM               spect
PROBHD     Z108618_0066 (
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                      128
DS                        4
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894465 sec
RG                     1820
DW                   62.400 usec
DE                    6.50 usec
TE                   303.2 K
D1              1.00000000 sec
TD0                        1
SFO1           399.8754692 MHz
NUC1                    1H
P1                    10.00 usec
PLW1           30.83499908 W

F2 - Processing parameters
SI                    65536
SF             399.8730034 MHz
WDW                      no
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

Figure S16. ^{1}H NMR spectrum of compound **9** (DMSO-d_6, 303.2 K).

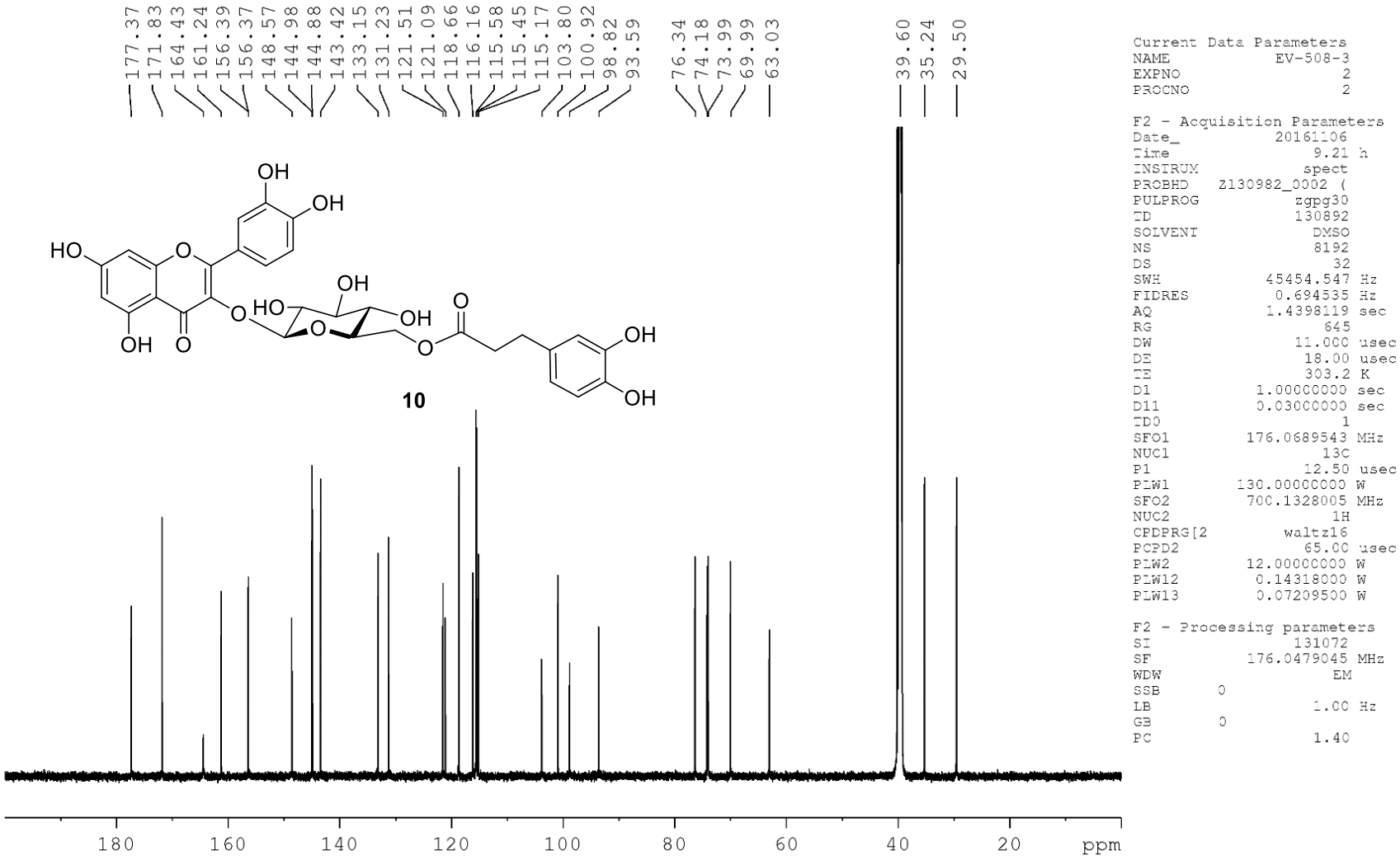
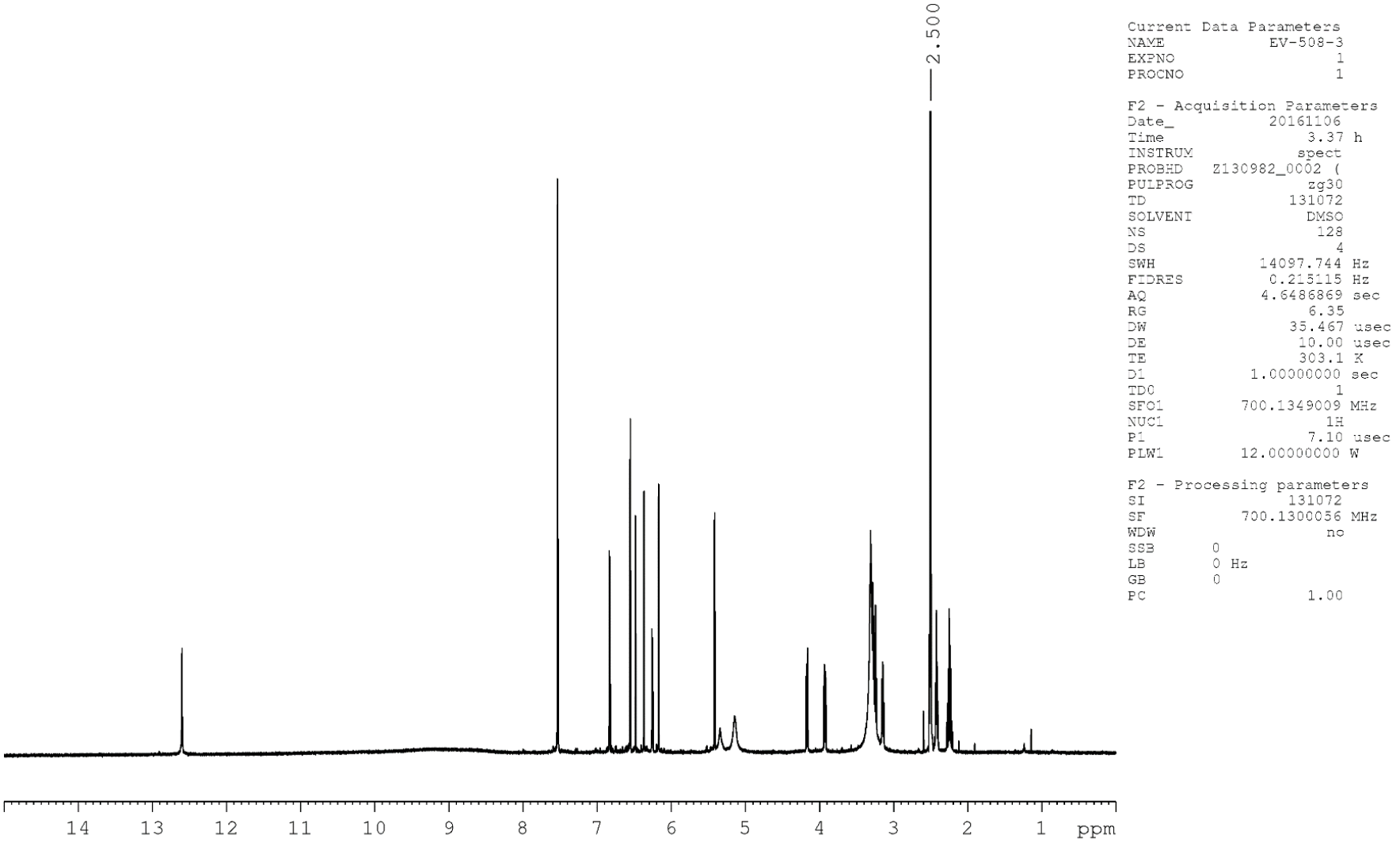


Figure S17. ^{13}C NMR spectrum of compound **10** (DMSO-d_6, 303.2 K)**.**




```
Current Data Parameters
NAME            EV-508-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161106
Time                3.37 h
INSTRUM            spect
PROBHD   Z130982_0002 (
PULPROG             zg30
TD                131072
SOLVENT             DMSO
NS                    128
DS                      4
SWH           14097.744 Hz
FIDRES         0.215115 Hz
AQ            4.6486869 sec
RG                   6.35
DW                35.467 usec
DE                10.00 usec
TE                303.1 K
D1           1.00000000 sec
TD0                    1
SFO1         700.1349009 MHz
NUC1                  1H
P1                 7.10 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                131072
SF           700.1300056 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

Figure S18. ^{1}H NMR spectrum of compound **10** (DMSO-d_6, 303.2 K).

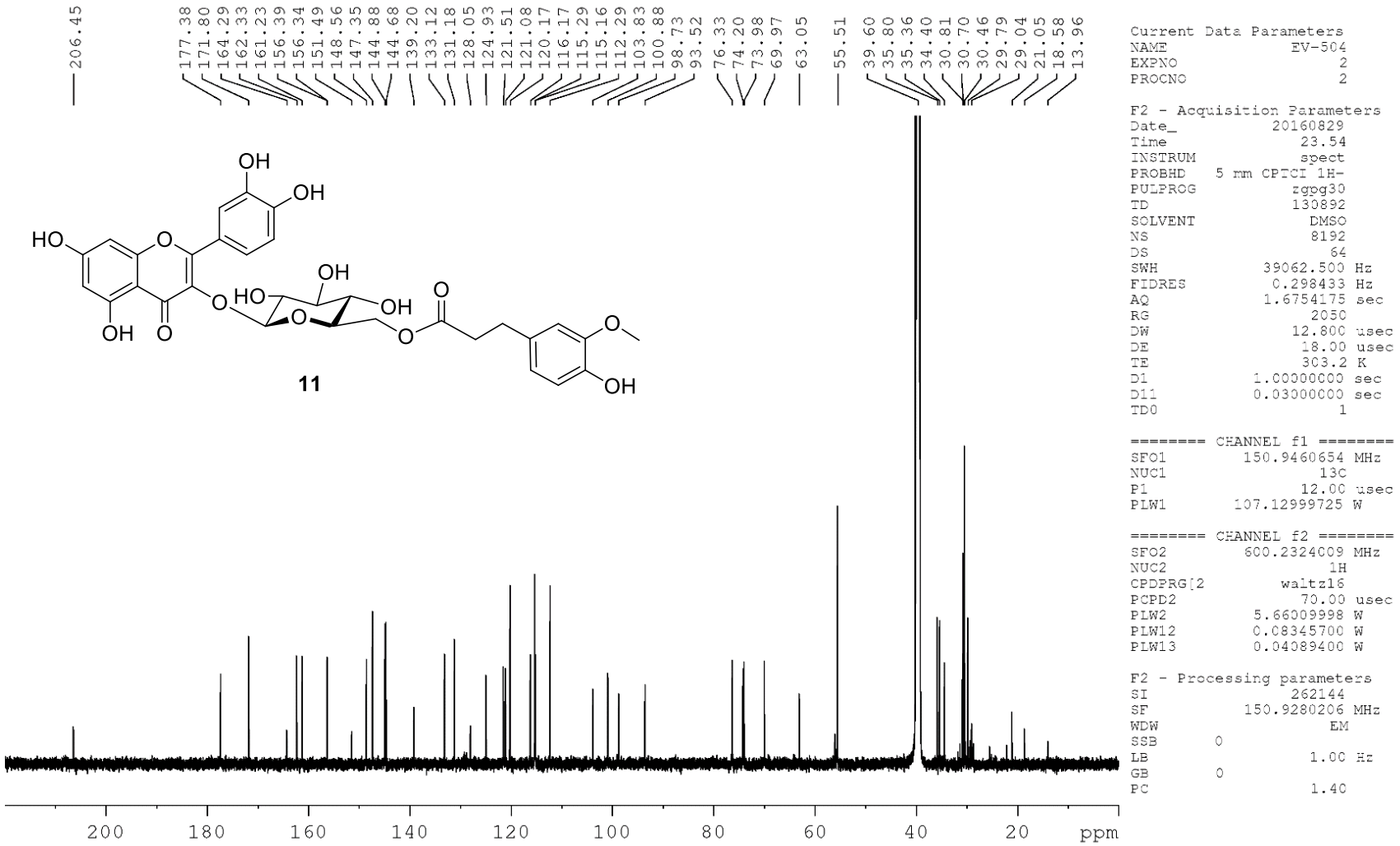
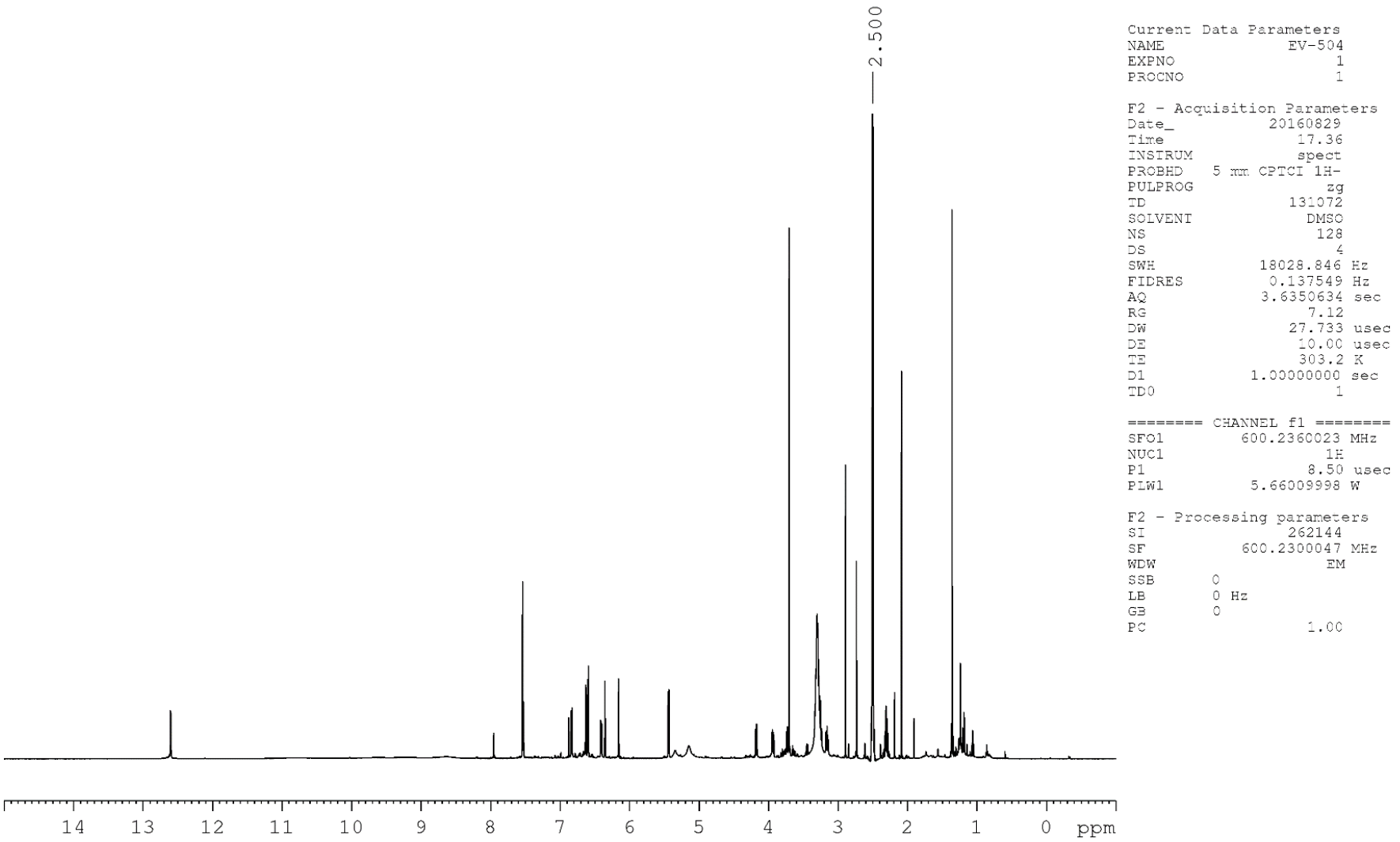


Figure S19. ^{13}C NMR spectrum of compound **11** (DMSO-d_6, 303.2 K).




```
Current Data Parameters
NAME                  EV-504
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160829
Time                  17.36
INSTRUM               spect
PROBHD    5 mm CPTCI 1H-
PULPROG                  zg
TD                   131072
SOLVENT                DMSO
NS                      128
DS                        4
SWH               18028.846 Hz
FIDRES             0.137549 Hz
AQ                3.6350634 sec
RG                     7.12
DW                   27.733 usec
DE                   10.00 usec
TE                   303.2 K
D1               1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            600.2360023 MHz
NUC1                    1H
P1                    8.50 usec
PLW1             5.66009998 W

F2 - Processing parameters
SI                    262144
SF              600.2300047 MHz
WDW                     EM
SSB          0
LB           0 Hz
GB           0
PC                    1.00
```

Figure S20. ^{1}H NMR spectrum of compound **11** (DMSO-d_6, 303.2 K).

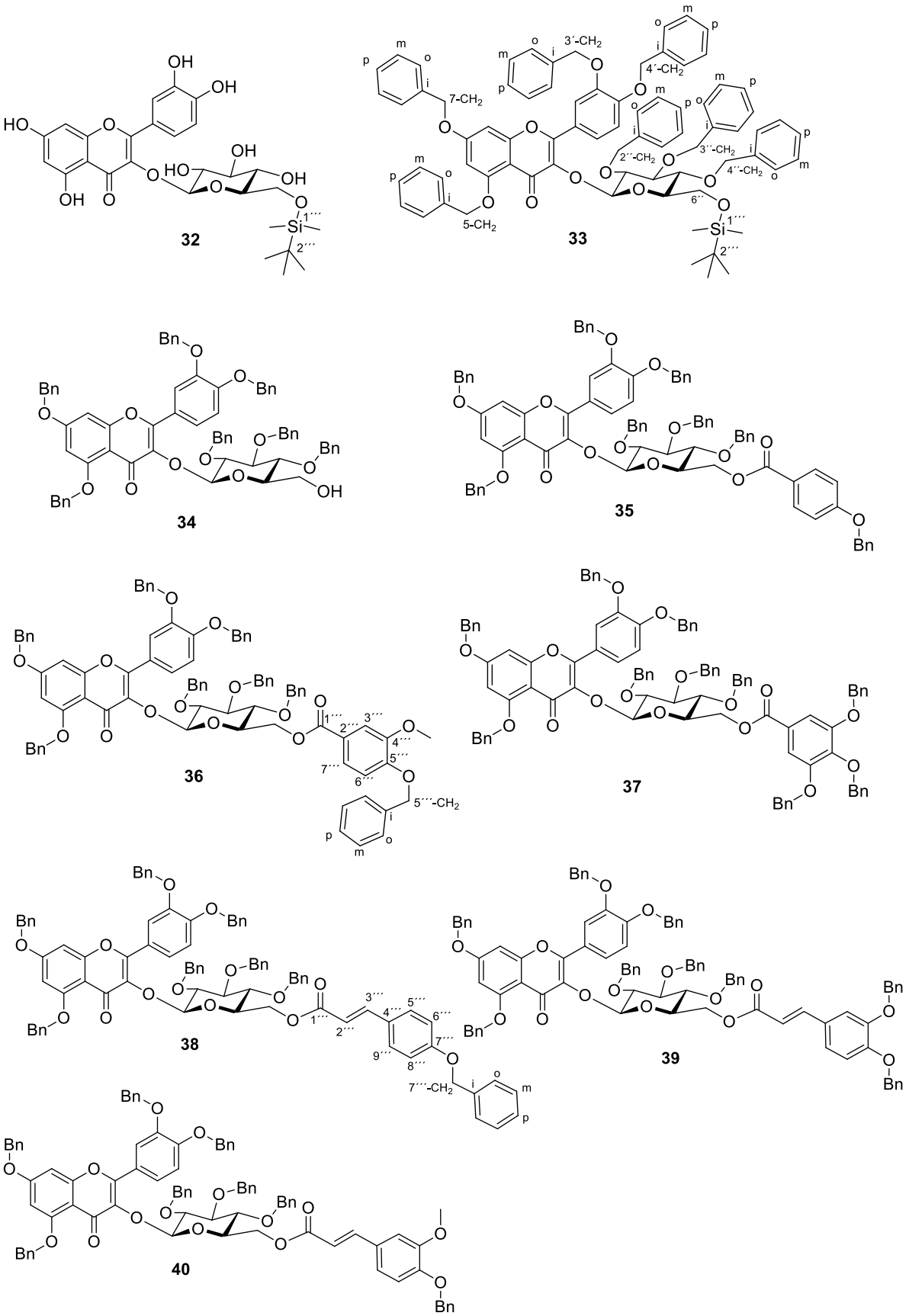


Figure S21. Structures of the intermediates **32** - **40**

Table S7. ^{13}C NMR data of the intermediates **32** - **34** (DMSO-d_6, 303.2 K), **33** (acetone, 293.2 K).

Comp.	32		33		34	
Atom number	δ	m	δ	m	δ	m
2	155.97	s	154.36	s	152.90	s
3	132.99	s	137.57	s	135.42	s
4	177.36	s	173.60	s	171.80	s
4a	103.80	s	111.04	s	108.86	s
5	161.34	s	161.33	s	159.24	s
6	98.78	d	99.26	d	97.99	d
7	164.89	s	164.63	s	162.75	s
8	93.42	d	95.53	d	94.21	d
8a	156.46	s	160.23	s	158.18	s
1′	121.15	s	125.43	s	123.16	s
2′	116.05	d	116.96	d	115.01	d
3′	144.95	s	149.76	s	147.65	s
4′	148.60	s	152.25	s	150.00	s
5′	115.21	d	115.06	d	113.58	d
6′	121.57	d	123.91	d	121.76	d
1″	100.17	d	102.21	d	100.09	d
2″	74.03	d	84.22	d	82.03	d
3″	76.62	d	86.00	d	83.40	d
4″	69.96	d	79.18	d	77.35	d
5″	77.72	d	77.29	d	75.34	d
6″	62.95	t	63.60	t	59.70	t
1‴	-	-	-	-	-	-
2‴	17.65	s	19.39	s	-	-
1‴-CH_3a	-5.84	q	-4.86	q	-	-
1‴-CH_3b	-5.72	q	-4.59	q	-	-
2‴-CH_3	25.43	q	26.84	q	-	-
5-CH_2	-	-	71.85	t	70.13	t
***i*-**	-	-	138.70	s	136.77	s
***o*-**	-	-	128.38*	d	127.05	d
***m*-**	-	-	129.86*	d	128.39	d
***p*-**	-	-	128.96	d	127.62	d
7-CH_2	-	-	71.81	t	70.12	t
***i*-**	-	-	137.98	s	136.12	s
***o*-**	-	-	129.43*	d	128.03	d
***m*-**	-	-	130.08*	d	128.57	d
***p*-**	-	-	129.69	d	128.22	d
3′-CH_2	-	-	72.43	t	70.29	t
***i*-**	-	-	138.99	s	137.11	s
***o*-**	-	-	128.76*	d	127.43	d
***m*-**	-	-	129.91*	d	128.39	d
***p*-**	-	-	129.22	d	127.83	d
4′-CH_2	-	-	71.87	t	69.94	t
***i*-**	-	-	138.90	s	137.01	s
***o*-**	-	-	128.91*	s	127.47	d
***m*-**	-	-	129.91*	d	128.47	d
***p*-**	-	-	129.33	d	127.88	d
2″-CH_2	-	-	75.09	t	73.06	t
***i*-**	-	-	140.47	s	138.42	s
***o*-**	-	-	129.83*	d	127.95	d

Comp.	32		33		34	
Atom number	δ	m	δ	m	δ	m
***m*-**	-	-	129.56*	d	127.99	d
***p*-**	-	-	128.83	d	127.36	d
3″-CH_2	-	-	76.66	t	74.29	t
***i*-**	-	-	140.81	s	138.78	s
***o*-**	-	-	129.14*	d	127.40	d
***m*-**	-	-	129.67*	d	128.16	d
***p*-**	-	-	128.78	d	127.34	d
4″-CH_2	-	-	75.94	t	73.84	t
***i*-**	-	-	140.35	s	138.35	s
***o*-**	-	-	128.91*	d	127.75	d
***m*-**	-	-	129.70*	d	128.21	d
***p*-**	-	-	128.95	d	127.54	d

m - the multiplicity of ^{13}C NMR signals was resolved using multiplicity-edited gradient-enhanced ^{1}H-^{13}C HSQC

* - 2C

Table S8. ^{1}H NMR data of the intermediates **32** - **34** (DMSO-d_6, 303.2 K), **33** (acetone, 293.2 K).

Comp.	32				33				34			
Atom number	δ	n	m	*J* [Hz]	δ	n	m	*J* [Hz]	δ H	n	m	*J* [Hz]
2	-	-	-	-	-	-	-	-	-	-	-	-
3	-	-	-	-	-	-	-	-	-	-	-	-
4	-	-	-	-	-	-	-	-	-	-	-	-
4a	-	-	-	-	-	-	-	-	-	-	-	-
5	-	-	-	-	-	-	-	-	-	-	-	-
6	6.150	1	d	2.0	6.701	1	d	2.3	6.736	1	d	2.3
7	-	-	-	-	-	-	-	-	-	-	-	-
8	6.356	1	d	2.0	6.869	1	d	2.3	6.931	1	d	2.3
8a	-	-	-	-	-	-	-	-	-	-	-	-
1′	-	-	-	-	-	-	-	-	-	-	-	-
2′	7.526	1	d	2.1	8.138	1	d	2.1	8.137	1	d	2.1
3′	-	-	-	-	-	-	-	-	-	-	-	-
4′	-	-	-	-	-	-	-	-	-	-	-	-
5′	6.821	1	d	8.3	7.217	1	d	8.6	7.247	1	d	8.6
6′	7.546	1	dd	8.3, 2.1	7.818	1	dd	8.6, 2.1	7.658	1	dd	8.6, 2.1
1″	5.520	1	d	7.6	6.010	1	d	7.7	5.861	1	d	7.6
2″	3.239	1	m	-	3.731	1	dd	8.9, 7.7	3.54 ^{H}	1	m	-
3″	3.239	1	m	-	3.831	1	dd	9.0, 8.9	3.787	1	dd	8.8, 8.8
4″	3.015	1	m	-	3.602	1	dd	9.8, 9.0	3.54 ^{H}	1	m	-
5″	3.091	1	ddd	9.8, 6.6, 1.5	3.386	1	ddd	9.8, 4.0, 2.2	3.379	1	ddd	9.8, 4.0, 2.1
6″	3.771	1	dd	11.4, 1.5	3.747	1	dd	11.5, 2.2	3.578	1	ddd	11.9, 4.6, 2.1
	3.435	1	dd	11.4, 6.6	3.721	1	dd	11.5, 4.0	3.510	1	ddd	11.9, 5.2, 4.0
5-OH	12.642	1	s	-	-	-	-	-	-	-	-	-
7-OH	n.a.	-	-	-	-	-	-	-	-	-	-	-
4'-OH	n.a.	-	-	-	-	-	-	-				
3′-OH	n.a.	-	-	-	-	-	-	-				
2″-OH	n.a.	-	-	-	-	-	-	-				
3″-OH	n.a.	-	-	-	-	-	-	-				
4″-OH	n.a.	-	-	-								
1‴	-	-	-	-	-	-	-	-	-	-	-	-
2‴	-	-	-	-	-	-	-	-	-	-	-	-
1‴-CH_3a	-0.326	3	s	-	-0.244	3	s	-	-	-	-	-
1‴-CH_3b	-0.252	3	s	-	-0.233	3	s	-	-	-	-	-
2‴-CH_3	0.592	9	s	-	0.677	9	s	-	-	-	-	-
5-CH_2	-	-	-	-	5.317	1	d	12.6	5.278	1	d	12.8
	-	-	-	-	5.284	1	d	12.6	5.243	1	d	12.8
***i*-**	-	-	-	-	-	-	-	-	-	-	-	-
***o*-**	-	-	-	-	7.761	2	m	-	7.630	2	m	-
***m*-**	-	-	-	-	7.42 ^{H}	2	m	-	7.419	2	m	-
***p*-**	-	-	-	-	7.33 ^{H}	1	m	-	7.335	1	m	-
7-CH_2	-	-	-	-	5.262	2	s	-	5.255	2	s	-
***i*-**	-	-	-	-	-	-	-	-	-	-	-	-
***o*-**	-	-	-	-	7.53 ^{H}	2	m	-	7.50 ^{H}	2	m	-
***m*-**	-	-	-	-	7.43 ^{H}	2	m	-	7.430	2	m	-
***p*-**	-	-	-	-	7.38 ^{H}	1	m	-	7.374	1	m	-
3′-CH_2	-	-	-	-	5.262	1	d	11.8	5.300	1	d	11.9
	-	-	-	-	5.157	1	d	11.8	5.115	1	d	11.9
***i*-**	-	-	-	-	-	-	-	-	-	-	-	-
***o*-**	-	-	-	-	7.55 ^{H}	2	m	-	7.50 ^{H}	2	m	-

Comp.	32				33				34			
Atom number	δ	n	m	*J* [Hz]	δ	n	m	*J* [Hz]	δ H	n	m	*J* [Hz]
m-	-	-	-	-	7.36 [H]	2	m	-	7.362	2	m	-
p-	-	-	-	-	7.31 [H]	1	m	-	7.322	1	m	-
4'-CH_2	-	-	-	-	5.288	2	s	-	5.264	2	s	-
i-	-	-	-	-	-	-	-	-	-	-	-	-
o-	-	-	-	-	7.54 [H]	2	m	-	7.489	2	m	-
m-	-	-	-	-	7.40 [H]	2	m	-	7.402	2	m	-
p-	-	-	-	-	7.34 [H]	1	m	-	7.333	1	m	-
2″-CH_2	-	-	-	-	5.130	1	d	11.4	4.897	1	d	11.5
	-	-	-	-	4.809	1	d	11.4	4.643	1	d	11.5
i-	-	-	-	-	-	-	-	-	-	-	-	-
o-	-	-	-	-	7.45 [H]	2	m	-	7.311	2	m	-
m-	-	-	-	-	7.22 [H]	2	m	-	7.21 [H]	2	m	-
p-	-	-	-	-	7.21 [H]	1	m	-	7.21 [H]	1	m	-
3″-CH_2	-	-	-	-	5.014	1	d	11.0	4.838	1	d	11.3
	-	-	-	-	4.858	1	d	11.0	4.745	1	d	11.3
i-	-	-	-	-	-	-	-	-	-	-	-	-
o-	-	-	-	-	7.37 [H]	2	m	-	7.26 [H]	2	m	-
m-	-	-	-	-	7.33 [H]	2	m	-	7.292	2	m	-
p-	-	-	-	-	7.29 [H]	1	m	-	7.274	1	m	-
4″-CH_2	-	-	-	-	4.828	1	d	11.2	4.638	1	d	11.0
	-	-	-	-	4.645	1	d	11.2	4.561	1	d	11.0
i-	-	-	-	-	-	-	-	-	-	-	-	-
o-	-	-	-	-	7.26 [H]	2	m	-	7.21 [H]	2	m	-
m-	-	-	-	-	7.27 [H]	2	m	-	7.271	2	m	-
p-	-	-	-	-	7.24 [H]	1	m	-	7.255	1	m	-
6''-OH	-	-	-	-	-	-	-	-	4.722	1	dd	5.2, 4.6

n.a. - not assigned due to the broadening of the hydroxyl signals

[H] - HSQC readout

Table S9. ^{13}C NMR data of the intermediates **35** - **38** (DMSO-d_6, 303.2 K).

Comp.	35		36		37		38	
Atom number	δ	m	δ	m	δ	m	δ	m
2	153.15	s	153.16	s	153.24	s	153.18	s
3	135.35	s	135.29	s	135.33	s	135.35	s
4	171.65	s	171.65	s	171.58	s	171.68	s
4a	108.77	s	108.73	s	108.64	s	108.76	s
5	159.22	s	159.17	s	159.05	s	159.16	s
6	97.93	d	97.89	d	97.80	d	97.93	d
7	162.75	s	162.70	s	162.65	s	162.73	s
8	94.22	d	94.09	d	93.94	d	94.14	d
8a	158.14	s	158.10	s	158.00	s	158.12	s
1'	123.07	s	123.11	s	123.11	s	123.05	s
2'	114.78	d	114.80	d	114.81	d	115.16	d
3'	147.45	s	147.47	s	147.48	s	147.43	s
4'	150.19	s	150.15	s	150.11	s	150.21	s
5'	113.23	d	113.27	d	113.05	d	113.36	d
6'	122.51	d	122.44	d	122.47	d	122.32	d
1''	100.24	d	100.16	d	100.31	d	100.27	d
2''	82.08	d	82.01	d	81.88	d	82.00	d
3''	83.45	d	83.43	d	83.44	d	83.32	d
4''	77.42	d	77.76	d	77.98	d	77.22	d
5''	72.05	d	72.19	d	71.91	d	71.94	d
6''	62.37	t	62.57	t	62.82	t	61.93	t
5-CH_2	70.12	t	70.07	t	70.03	t	70.10	t
***i*-**	136.79	s	136.82	s	136.78	s	136.79	s
***o*-**	126.99	d	126.96	d	126.90	d	127.01	d
***m*-**	128.37	d	128.37	d	128.34	d	128.38	d
***p*-**	127.60	d	127.59	d	127.55	d	127.61	d
7-CH_2	70.20	t	70.17	t	70.02	t	70.10	t
***i*-**	136.00	s	136.00	s	135.97	s	136.06	s
***o*-**	128.04	d	128.06	d	127.98	d	128.04	d
***m*-**	128.52	d	128.51	d	128.45	d	128.55	d
***p*-**	128.20	d	128.19	d	128.12	d	128.22	d
3'-CH_2	70.51	t	70.49	t	70.47	t	70.51	t
***i*-**	136.99	s	136.99	s	137.00	s	137.01	s
***o*-**	127.24	d	127.27	d	127.27	d	127.15	d
***m*-**	128.37	d	128.39	d	128.37	d	128.43	d
***p*-**	127.82	d	127.83	d	127.80	d	127.84	d
4'-CH_2	69.91	t	69.91	t	69.88	t	69.86	t
***i*-**	136.79	s	136.84	s	136.75	s	136.84	s
***o*-**	127.51	d	127.50	d	127.48	d	127.41	d
***m*-**	128.43	d	128.43	d	128.37	d	128.38	d
***p*-**	127.89	d	127.89	d	127.84	d	127.82	d
2''-CH_2	73.13	t	73.10	t	73.09	t	73.11	t
***i*-**	138.40	s	138.37	s	138.38	s	138.40	s
***o*-**	128.05	d	128.04	d	128.05	d	128.06	d
***m*-**	128.07	d	128.06	d	128.05	d	128.05	d
***p*-**	127.45	d	127.45	d	127.44	d	127.44	d
3''-CH_2	74.62	t	74.59	t	74.63	t	74.52	t
***i*-**	138.63	s	138.63	s	138.63	s	138.67	s
***o*-**	127.54	d	127.53	d	127.57	d	127.49	d

Comp.	35		36		37		38	
Atom number	δ	m	δ	m	δ	m	δ	m
***m*-**	128.23	d	128.23	d	128.23	d	128.23	d
***p*-**	127.47	d	127.46	d	127.48	d	127.45	d
4'''-CH_2	73.88	t	73.90	t	73.94	t	73.92	t
***i*-**	137.84	s	137.89	s	137.96	s	137.89	s
***o*-**	127.94	d	127.92	d	127.89	d	127.97	d
***m*-**	128.23	d	128.26	d	128.25	d	128.24	d
***p*-**	127.69	d	127.71	d	127.69	d	127.71	d
1'''	164.78	s	164.85	s	164.53	s	165.85	s
***2*'''**	121.79	s	121.79	s	124.46	s	114.72	d
***3*'''**	131.00	d	111.73	d	107.86	d	144.27	d
***4*'''**	114.78	d	148.52	s	151.84	s	126.66	s
***5*'''**	162.13	s	151.87	s	141.43	s	129.99	d
6'''	-	d	111.94	d	-	-	115.03	d
7'''	-	d	122.66	d	-	-	160.18	s
4'''-MeO	-	-	55.34	q	-	-	-	-
***4*'''-CH_2**	-	-	-	-	70.07	t	-	-
***i*-**	-	-	-	-	136.63	s	-	-
***o*-**	-	-	-	-	127.35	d	-	-
***m*-**	-	-	-	-	128.41	d	-	-
***p*-**	-	-	-	-	127.86	d	-	-
***5*'''-CH_2**	69.50	t	69.89	t	74.31	t	-	-
***i*-**	136.25	s	136.26	s	137.34	s	-	-
***o*-**	127.77	d	127.90	d	128.07	d	-	-
***m*-**	128.43	d	128.40	d	128.06	d	-	-
***p*-**	128.01	d	128.02	d	127.84	d	-	-
7'''-CH_2	-	-	-	-	-	-	69.32	t
***i*-**	-	-	-	-	-	-	136.66	s
***o*-**	-	-	-	-	-	-	127.68	d
***m*-**	-	-	-	-	-	-	128.47	d
***p*-**	-	-	-	-	-	-	127.94	d

m - the multiplicity of ^{13}C NMR signals was resolved using multiplicity-edited gradient-enhanced ^{1}H-^{13}C HSQC

Table S10. ^{1}H NMR data of the intermediates **35** - **36** (DMSO-d_6, 303.2 K).

	35				**36**			
Atom number	**δ**	**n**	**m**	***J* [Hz]**	**δ**	**n**	**m**	***J* [Hz]**
2	-	-	-	-	-	-	-	-
3	-	-	-	-	-	-	-	-
4	-	-	-	-	-	-	-	-
4a	-	-	-	-	-	-	-	-
5	-	-	-	-	-	-	-	-
6	6.723	1	d	2.3	6.696	1	d	2.2
7	-	-	-	-	-	-	-	-
8	6.912	1	d	2.3	6.862	1	d	2.2
8a	-	-	-	-	-	-	-	-
1'	-	-	-	-	-	-	-	-
2'	7.857	1	d	2.1	7.844	1	d	2.1
3'	-	-	-	-	-	-	-	-
4'	-	-	-	-	-	-	-	-
5'	7.086	1	d	8.7	7.088	1	d	8.7
6'	7.747	1	dd	8.7, 2.1	7.700	1	dd	8.7, 2.1
1''	5.791	1	d	7.7	5.804	1	d	7.7
2''	3.625	1	dd	8.9, 7.7	3.610	1	dd	8.8, 7.7
3''	3.886	1	dd	8.9, 8.9	3.888	1	dd	8.9, 8.8
4''	3.531	1	dd	9.8, 8.9	3.506	1	dd	9.7, 8.9
5''	3.735	1	ddd	9.8, 5.3, 2.5	3.755	1	ddd	9.7, 6.0, 2.5
6''	4.298	1	dd	11.9, 2.5	4.335	1	dd	11.9, 2.5
	4.201	1	dd	11.9, 5.3	4.223	1	dd	11.9, 6.0
5-CH$_2$	5.260	1	d	12.6	5.253	1	d	12.8
	5.218	1	d	12.6	5.209	1	d	12.8
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.632	2	m	-	7.629	2	m	-
***m*-**	7.41 ^{H}	2	m	-	7.41 ^{H}	2	m	-
***p*-**	7.34 ^{H}	1	m	-	7.33 ^{H}	1	m	-
7-CH$_2$	5.233	1	d	11.4	5.214	2	s	-
	5.209	1	d	11.4				
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.45 ^{H}	2	m	-	7.44 ^{H}	2	m	-
***m*-**	7.39 ^{H}	2	m	-	7.39 ^{H}	2	m	-
***p*-**	7.36 ^{H}	1	m	-	7.35 ^{H}	1	m	-
3'-CH$_2$	5.111	1	d	12.0	5.110	1	d	11.8
	5.073	1	d	12.0	5.070	1	d	11.8
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.42 ^{H}	2	m	-	7.42 ^{H}	2	m	-
***m*-**	7.33 ^{H}	2	m	-	7.34 ^{H}	2	m	-
***p*-**	7.31 ^{H}	1	m	-	7.30 ^{H}	1	m	-
4'-CH$_2$	5.132	1	d	11.9	5.144	1	d	12.0
	5.088	1	d	11.9	5.106	1	d	12.0
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.45 ^{H}	2	m	-	7.45 ^{H}	2	m	-
***m*-**	7.36 ^{H}	2	m	-	7.37 ^{H}	2	m	-
***p*-**	7.31 ^{H}	1	m	-	7.31 ^{H}	1	m	-
2''-CH$_2$	5.024	1	d	11.5	5.004	1	d	11.5
	4.726	1	d	11.5	4.714	1	d	11.5
***i*-**	-	-	-	-	-	-	-	-

	35				36			
Atom number	**δ**	**n**	**m**	***J* [Hz]**	**δ**	**n**	**m**	***J* [Hz]**
***o*-**	7.39 [H]	2	m	-	7.37 [H]	2	m	-
***m*-**	7.24 [H]	2	m	-	7.23 [H]	2	m	-
***p*-**	7.24 [H]	1	m	-	7.23 [H]	1	m	-
3''-CH₂	4.923	1	d	11.1	4.917	1	d	11.2
	4.788	1	d	11.1	4.784	1	d	11.2
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.33 [H]	2	m	-	7.33 [H]	2	m	-
***m*-**	7.34 [H]	2	m	-	7.34 [H]	2	m	-
***p*-**	7.30 [H]	1	m	-	7.31 [H]	1	m	-
4''-CH₂	4.718	1	d	11.1	4.726	1	d	11.1
	4.502	1	d	11.1	4.530	1	d	11.1
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.18 [H]	2	m	-	7.20 [H]	2	m	-
***m*-**	7.21 [H]	2	m	-	7.24 [H]	2	m	-
***p*-**	7.20 [H]	1	m	-	7.22 [H]	1	m	-
1'''					-	-	-	-
2'''	-	-	-	-	-	-	-	-
3'''	7.624	2	m	-	7.218	1	m	-
4'''	6.872	2	m	-	-	-	-	-
5'''	-	-	-	-	-	-	-	-
6'''	-	-	-	-	6.768	1	m	-
7'''	-	-	-	-	7.213	1	m	-
4'''-MeO	-	-	-	-	3.620	3	s	-
***5'''*-CH₂**	5.001	1	d	11.9	4.961	1	d	11.6
	4.982	1	d	11.9	4.934	1	d	11.6
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.33 [H]	2	m	-	7.33 [H]	2	m	-
***m*-**	7.34 [H]	2	m	-	7.33 [H]	2	m	-
***p*-**	7.33 [H]	1	m	-	7.32 [H]	1	m	-

[H] - HSQC readout

Table S11. ^{1}H NMR data of the intermediates **37** - **38** (DMSO-d_6, 30 °C).

	37				**38**			
Atom number	**δ**	**n**	**m**	***J* [Hz]**	**δ**	**n**	**m**	***J* [Hz]**
2	-	-	-	-	-	-	-	-
3	-	-	-	-	-	-	-	-
4	-	-	-	-	-	-	-	-
4a	-	-	-	-	-	-	-	-
5	-	-	-	-	-	-	-	-
6	6.611	1	d	2.2	6.660	1	d	2.2
7	-	-	-	-	-	-	-	-
8	6.712	1	d	2.2	6.839	1	d	2.2
8a	-	-	-	-	-	-	-	-
1'	-	-	-	-	-	-	-	-
2'	7.803	1	d	2.1	7.977	1	d	2.1
3'	-	-	-	-	-	-	-	-
4'	-	-	-	-	-	-	-	-
5'	6.933	1	d	8.6	7.171	1	d	8.6
6'	7.624	1	dd	8.6, 2.1	7.712	1	dd	8.6, 2.1
1''	5.787	1	d	7.6	5.779	1	d	7.6
2''	3.602	1	dd	8.8, 7.6	3.608	1	dd	8.8, 7.6
3''	3.888	1	dd	8.8, 8.8	3.877	1	dd	8.9, 8.8
4''	3.495	1	dd	9.7, 8.8	3.553	1	dd	9.8, 8.9
5''	3.784	1	ddd	9.7, 5.9, 2.7	3.702	1	ddd	9.8, 4.0, 3.2
6''	4.393	1	dd	11.9, 2.7	4.163	2	m	-
	4.260	1	dd	11.9, 5.9				
5-CH$_2$	5.187	1	d	12.6	5.238	1	d	12.7
	5.109	1	d	12.6	5.189	1	d	12.7
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.594	2	m	-	7.635	2	m	-
***m*-**	7.40 ^{H}	2	m	-	7.42 ^{H}	2	m	-
***p*-**	7.32 ^{H}	1	m	-	7.34 ^{H}	1	m	-
7-CH$_2$	5.119	1	d	11.4	5.206	1	d	11.6
	5.095	1	d	11.4	5.165	1	d	11.6
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.35 H	2	m	-	7.495	2	m	-
***m*-**	7.36 H	2	m	-	7.41 ^{H}	2	m	-
***p*-**	7.35 H	1	m	-	7.36 ^{H}	1	m	-
3'-CH$_2$	5.102	1	d	12.0	5.216	1	d	12.0
	5.067	1	d	12.0	5.145	1	d	12.0
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.40 ^{H}	2	m	-	7.461	2	m	-
***m*-**	7.31 ^{H}	2	m	-	7.33 ^{H}	2	m	-
***p*-**	7.28 ^{H}	1	m	-	7.30 ^{H}	1	m	-
4'-CH$_2$	5.102	1	d	12.0	5.114	1	d	12.2
	5.067	1	d	12.0	5.078	1	d	12.2
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.41 ^{H}	2	m	-	7.39 ^{H}	2	m	-
***m*-**	7.32 ^{H}	2	m	-	7.34 ^{H}	2	m	-
***p*-**	7.28 ^{H}	1	m	-	7.30 ^{H}	1	m	-
2''-CH$_2$	5.001	1	d	11.5	5.003	1	d	11.4
	4.704	1	d	11.5	4.695	1	d	11.4
***i*-**	-	-	-	-	-	-	-	-

	37				38			
Atom number	**δ**	**n**	**m**	***J* [Hz]**	**δ**	**n**	**m**	***J* [Hz]**
***o*-**	7.37 [H]	2	m	-	7.37 [H]	2	m	-
***m*-**	7.22 [H]	2	m	-	7.23 [H]	2	m	-
***p*-**	7.23 [H]	1	m	-	7.23 [H]	1	m	-
3''-CH₂	4.933	1	d	11.0	4.919	1	d	11.1
	4.796	1	d	11.0	4.787	1	d	11.1
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.34 [H]	2	m	-	7.33 [H]	2	m	-
***m*-**	7.34 [H]	2	m	-	7.34 [H]	2	m	-
***p*-**	7.31 [H]	1	m	-	7.30 [H]	1	m	-
4''-CH₂	4.739	1	d	11.1	4.722	1	d	10.9
	4.542	1	d	11.1	4.504	1	d	10.9
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.20 [H]	2	m	-	7,185	2	m	-
***m*-**	7.23 [H]	2	m	-	7.23 [H]	2	m	-
***p*-**	7.22 [H]	1	m	-	7.22 [H]	1	m	-
1'''	-	-	-	-	-	-	-	-
2'''	-	-	-	-	6.171	1	d	16.0
3'''	7.064	2	s	-	7.355	1	d	16.0
4'''	-	-	-	-	-	-	-	-
5'''	-	-	-	-	7.412	2	m	-
6'''	-	-	-	-	6.910	2	m	-
7'''	-	-	-	-	-	-	-	-
***4'''*-CH₂**	4.923	2	d	11.8	-	-	-	-
	4.853	2	d	11.8	-	-	-	-
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.33 [H]	4	m	-	-	-	-	-
***m*-**	7.32 [H]	4	m	-	-	-	-	-
***p*-**	7.28 [H]	2	m	-	-	-	-	-
***5'''*-CH₂**	4.951	2	s	-	-	-	-	-
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.30 [H]	2	m	-	-	-	-	-
***m*-**	7.21 [H]	2	m	-	-	-	-	-
***p*-**	7.26 [H]	1	m	-	-	-	-	-
7'''-CH₂	-	-	-	-	5.031	1	d	11.9
	-	-	-	-	5.011	1	d	11.9
***i*-**	-	-	-	-	-	-	-	-
***o*-**	-	-	-	-	7.39 [H]	2	m	-
***m*-**	-	-	-	-	7.39 [H]	2	m	-
***p*-**	-	-	-	-	7.34 [H]	1	m	-

[H] - HSQC readout

Table S12. ^{13}C NMR data of the intermediates **39** - **40** (DMSO-d_6, 30 °C).

Comp.	39		40	
Atom number	δ	m	δ	m
2	153.24	s	153.22	s
3	135.39	s	135.37	s
4	171.68	s	171.68	s
4a	108.77	s	108.77	s
5	159.15	s	159.16	s
6	97.90	d	97.88	d
7	162.72	s	162.72	s
8	94.10	d	94.11	d
8a	158.12	s	158.12	s
1'	123.02	s	123.01	s
2'	115.08	d	115.14	d
3'	147.42	s	147.44	s
4'	150.15	s	150.17	s
5'	113.28	d	113.29	d
6'	122.34	d	122.31	d
1''	100.34	d	100.33	d
2''	81.99	d	81.97	d
3''	83.29	d	83.28	d
4''	77.26	d	77.24	d
5''	71.94	d	71.93	d
6''	61.98	t	61.96	t
5-CH_2	70.10	t	70.10	t
***i*-**	136.78	s	136.78	s
***o*-**	126.99	d	127.00	d
***m*-**	128.37	d	128.37	d
***p*-**	127.60	d	127.61	d
7-CH_2	70.09	t	70.09	t
***i*-**	136.03	s	136.06	s
***o*-**	128.05	d	128.06	d
***m*-**	128.52	d	128.55	d
***p*-**	128.19	d	128.22	d
3'-CH_2	70.54	t	70.60	t
***i*-**	136.98	s	136.97	s
***o*-**	127.32	d	127.36	d
***m*-**	128.44	d	128.45	d
***p*-**	127.87	d	127.91	d
4'-CH_2	69.78	t	69.81	t
***i*-**	136.78	s	136.78	s
***o*-**	127.38	d	127.40	d
***m*-**	128.35	d	128.36	d
***p*-**	127.80	d	127.82	d
2''-CH_2	73.11	t	73.10	t
***i*-**	138.40	s	138.40	s
***o*-**	128.06	d	128.06	d
***m*-**	128.05	d	128.04	d
***p*-**	127.43	d	127.44	d
3''-CH_2	74.49	t	74.47	t
***i*-**	138.65	s	138.65	s
***o*-**	127.49	d	127.48	d

Comp.	39		40	
Atom number	δ	m	δ	m
***m*-**	128.22	d	128.22	d
***p*-**	127.46	d	127.44	d
4'''-CH_2	73.94	t	73.93	t
***i*-**	137.90	s	137.90	s
***o*-**	127.98	d	127.97	d
***m*-**	128.25	d	128.25	d
***p*-**	127.72	d	127.72	d
1'''	165.93	s	165.98	s
2'''	115.04	d	114.84	d
3'''	144.64	d	144.80	d
4'''	126.96	s	127.01	s
5'''	112.90	d	110.51	d
6'''	148.33	s	149.20	s
7'''	150.41	s	150.01	s
8'''	113.68	d	112.91	d
9'''	123.13	d	122.79	d
6'''-MeO	-	-	55.53	q
6'''-CH_2	70.10	t	-	-
***i*-**	137.09	s	-	-
***o*-**	127.56	d	-	-
***m*-**	128.35	d	-	-
***p*-**	127.79	d	-	-
7'''-CH_2	69.86	t	69.81	t
***i*-**	136.89	s	136.71	s
***o*-**	127.40	d	127.78	d
***m*-**	128.40	d	128.43	d
***p*-**	127.84	d	127.94	d

m - the multiplicity of ^{13}C NMR signals was resolved using multiplicity-edited gradient-enhanced ^{1}H-^{13}C HSQC

Table S13. ^{1}H NMR data of the intermediates **39** - **40** (DMSO-d_6, 30 °C).

	39				**40**			
Atom number	**δ**	**n**	**m**	***J* [Hz]**	**δ**	**n**	**m**	***J* [Hz]**
2	-	-	-	-	-	-	-	-
3	-	-	-	-	-	-	-	-
4	-	-	-	-	-	-	-	-
4a	-	-	-	-	-	-	-	-
5	-	-	-	-	-	-	-	-
6	6.647	1	d	2.3	6.654	1	d	2.2
7	-	-	-	-	-	-	-	-
8	6.799	1	d	2.3	6.807	1	d	2.2
8a	-	-	-	-	-	-	-	-
1'	-	-	-	-	-	-	-	-
2'	7.947	1	d	2.2	7.970	1	d	2.1
3'	-	-	-	-	-	-	-	-
4'	-	-	-	-	-	-	-	-
5'	7.135	1	d	8.7	7.152	1	d	8.6
6'	7.701	1	dd	8.7, 2.2	7.699	1	dd	8.6, 2.1
1''	5.756	1	d	7.6	5.766	1	d	7.6
2''	3.584	1	dd	8.8, 7.6	3.591	1	dd	8.6, 7.6
3''	3.871	1	dd	8.8, 8.8	3.872	1	dd	9.2, 8.6
4''	3.550	1	dd	9.8, 8.8	3.554	1	dd	9.8, 9.2
5''	3.699	1	ddd	9.8, 3.7, 3.5	3.702	1	ddd	9.8, 4.5, 2.5
6''	4.180	2	m	-	4.202	1	dd	12.1, 4.5
					4.169	1	dd	12.1, 2.5
5-CH$_2$	5.231	1	d	12.6	5.234	1	d	12.7
	5.176	1	d	12.6	5.180	1	d	12.7
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.627	2	m	-	7.636	2	m	-
***m*-**	7.41 ^{H}	2	m	-	7.42 ^{H}	2	m	-
***p*-**	7.33 ^{H}	1	m	-	7.34 ^{H}	1	m	-
7-CH$_2$	5.182	1	d	11.6	5.192	1	d	11.5
	5.143	1	d	11.6	5.155	1	d	11.5
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.475	2	m	-	7.494	2	m	-
***m*-**	7.39 ^{H}	2	m	-	7.41 ^{H}	2	m	-
***p*-**	7.35 ^{H}	2	m	-	7.36 ^{H}	1	m	-
3'-CH$_2$	5.222	1	d	11.9	5.228	1	d	11.8
	5.153	1	d	11.9	5.148	1	d	11.8
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.455	2	m	-	7.464	2	m	-
***m*-**	7.32 ^{H}	2	m	-	7.34 ^{H}	2	m	-
***p*-**	7.29 ^{H}	1	m	-	7.31 ^{H}	1	m	-
4'-CH$_2$	5.070	1	d	12.1	5.087	1	d	12.2
	5.031	1	d	12.1	5.055	1	d	12.2
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.34 ^{H}	2	m	-	7.35 ^{H}	2	m	-
***m*-**	7.31 ^{H}	2	m	-	7.33 ^{H}	2	m	-
***p*-**	7.27 ^{H}	1	m	-	7.29 ^{H}	1	m	-
2''-CH$_2$	4.999	1	d	11.4	5.002	1	d	11.4
	4.677	1	d	11.4	4.682	1	d	11.4
***i*-**	-	-	-	-	-	-	-	-

	39				40			
Atom number	**δ**	**n**	**m**	***J* [Hz]**	**δ**	**n**	**m**	***J* [Hz]**
***o*-**	7.37 [H]	2	m	-	7.37 [H]	2	m	-
***m*-**	7.23 [H]	2	m	-	7.23 [H]	2	m	-
***p*-**	7.23 [H]	1	m	-	7.23 [H]	1	m	-
3''-CH₂	4.906	1	d	11.1	4.906	1	d	11.2
	4.778	1	d	11.1	4.778	1	d	11.2
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.31 [H]	2	m	-	7.31 [H]	2	m	-
***m*-**	7.32 [H]	2	m	-	7.32 [H]	2	m	-
***p*-**	7.29 [H]	1	m	-	7.29 [H]	1	m	-
4''-CH₂	4.726	1	d	11.0	4.725	1	d	11.1
	4.520	1	d	11.0	4.519	1	d	11.1
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.196	2	m	-	7.20 [H]	2	m	-
***m*-**	7.25 [H]	2	m	-	7.25 [H]	2	m	-
***p*-**	7.22 [H]	1	m	-	7.23 [H]	1	m	-
1'''	-	-	-	-	-	-	-	-
***2*'''**	6.293	1	d	16.0	6.294	1	d	16.0
***3*'''**	7.346	1	d	16.0	7.369	1	d	16.0
***4*'''**	-	-	-	-	-	-	-	-
***5*'''**	7.335	1	d	1.9	7.172	1	d	2.0
6'''	-	-	-	-	-	-	-	-
7'''	-	-	-	-	-	-	-	-
8'''	6.945	1	d	8.5	6.925	1	d	8,4
9'''	7.012	1	dd	8.5, 1.9	6.992	1	dd	8.4, 2.0
6'''-MeO	-	-	-	-	3.670	3	s	-
6'''-CH₂	5.087	1	d	11.9	-	-	-	-
	5.001	1	d	11.9	-	-	-	-
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.39 [H]	2	m	-	-	-	-	-
***m*-**	7.34 [H]	2	m	-	-	-	-	-
***p*-**	7.30 [H]	1	m	-	-	-	-	-
7'''-CH₂	5.053	2	m	-	5.014	1	d	12.0
					4.995	1	d	12.0
***i*-**	-	-	-	-	-	-	-	-
***o*-**	7.38 [H]	2	m	-	7.38 [H]	2	m	-
***m*-**	7.36 [H]	2	m	-	7.38 [H]	2	m	-
***p*-**	7.31 [H]	1	m	-	7.33 [H]	1	m	-

[H] - HSQC readout

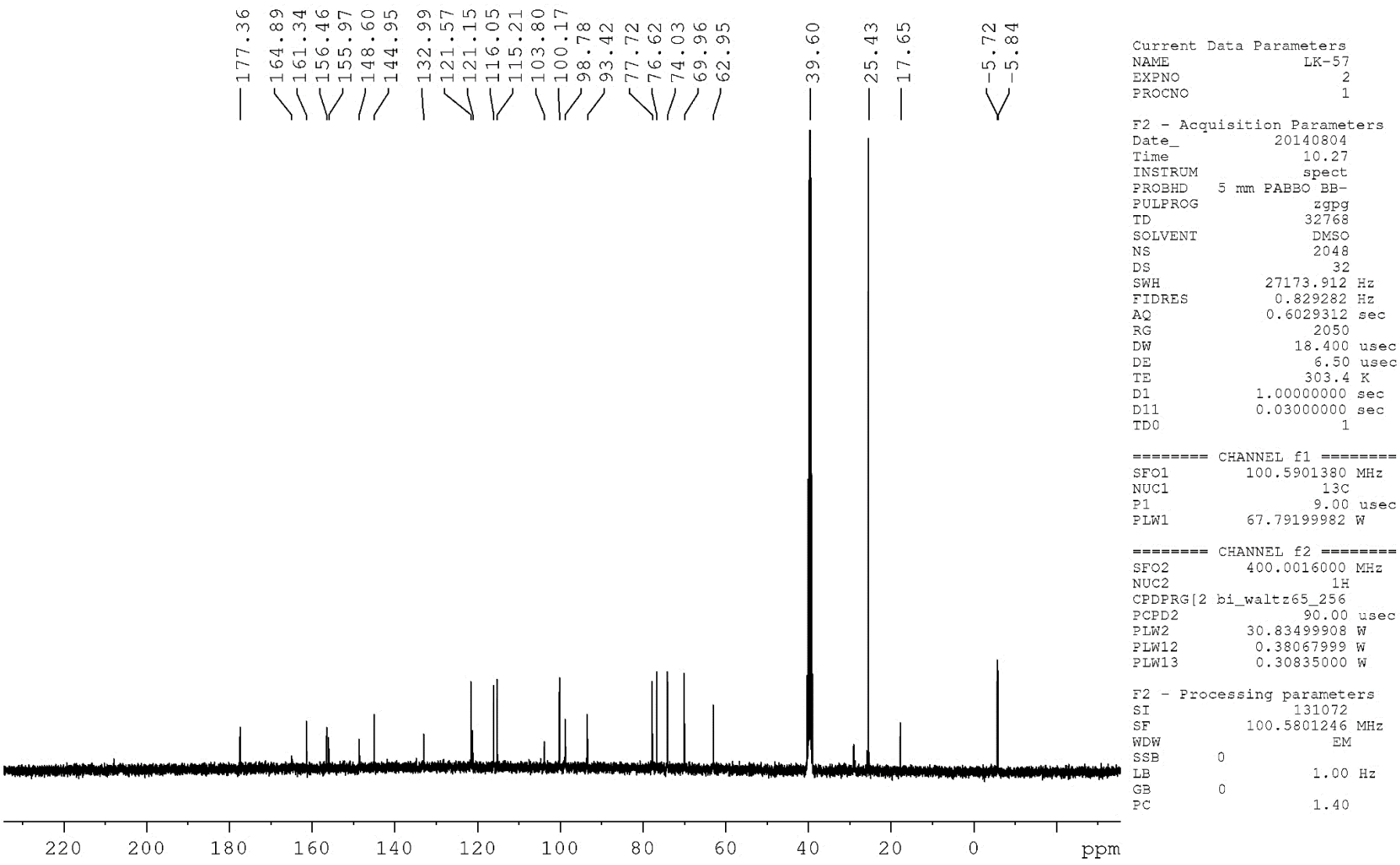


Figure S22. ^{13}C NMR spectrum of compound **32** (DMSO-d_6, 303.2 K).

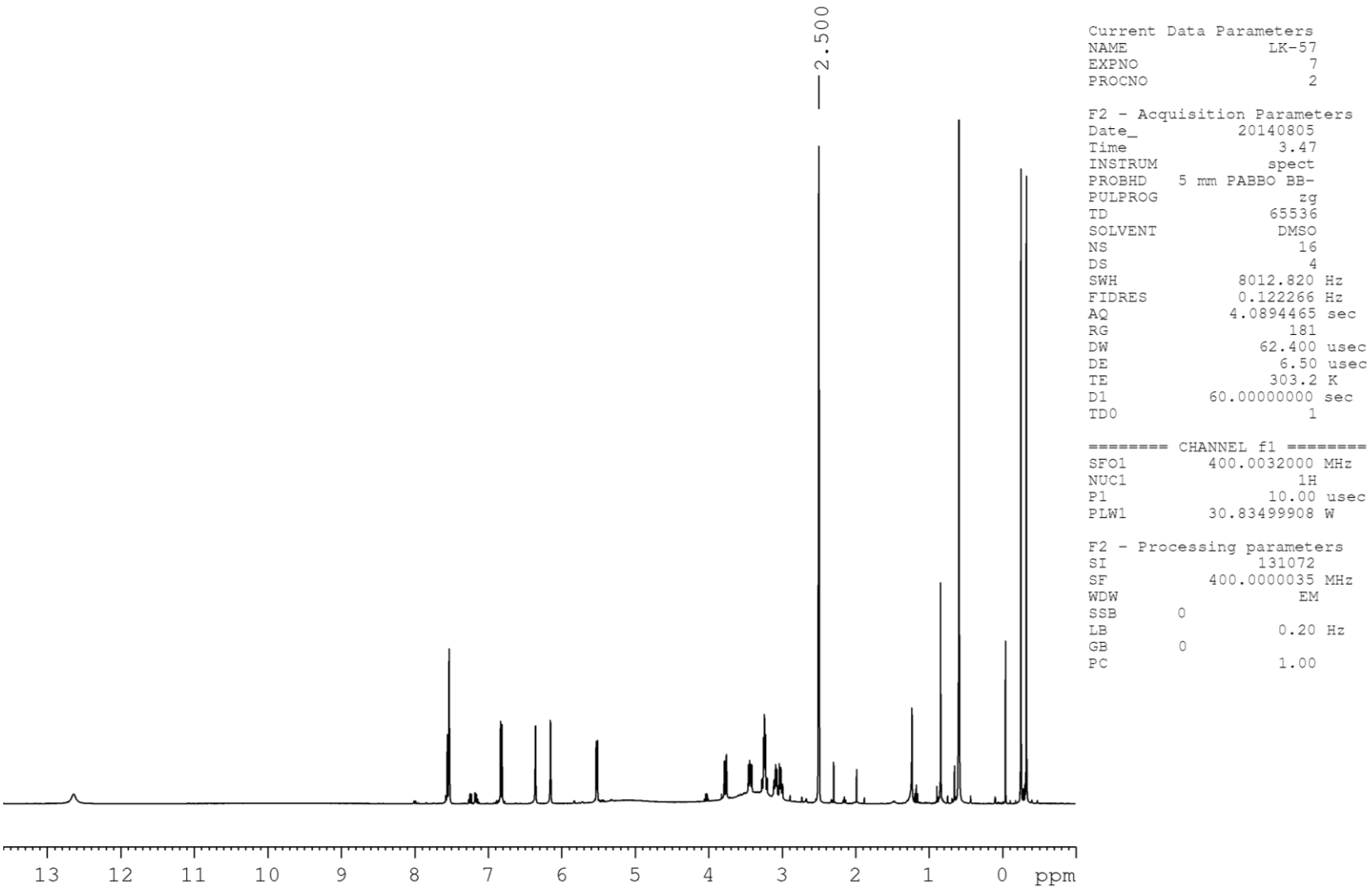


Figure S23. ^{1}H NMR spectrum of compound **32** (DMSO-d_6, 303.2 K).

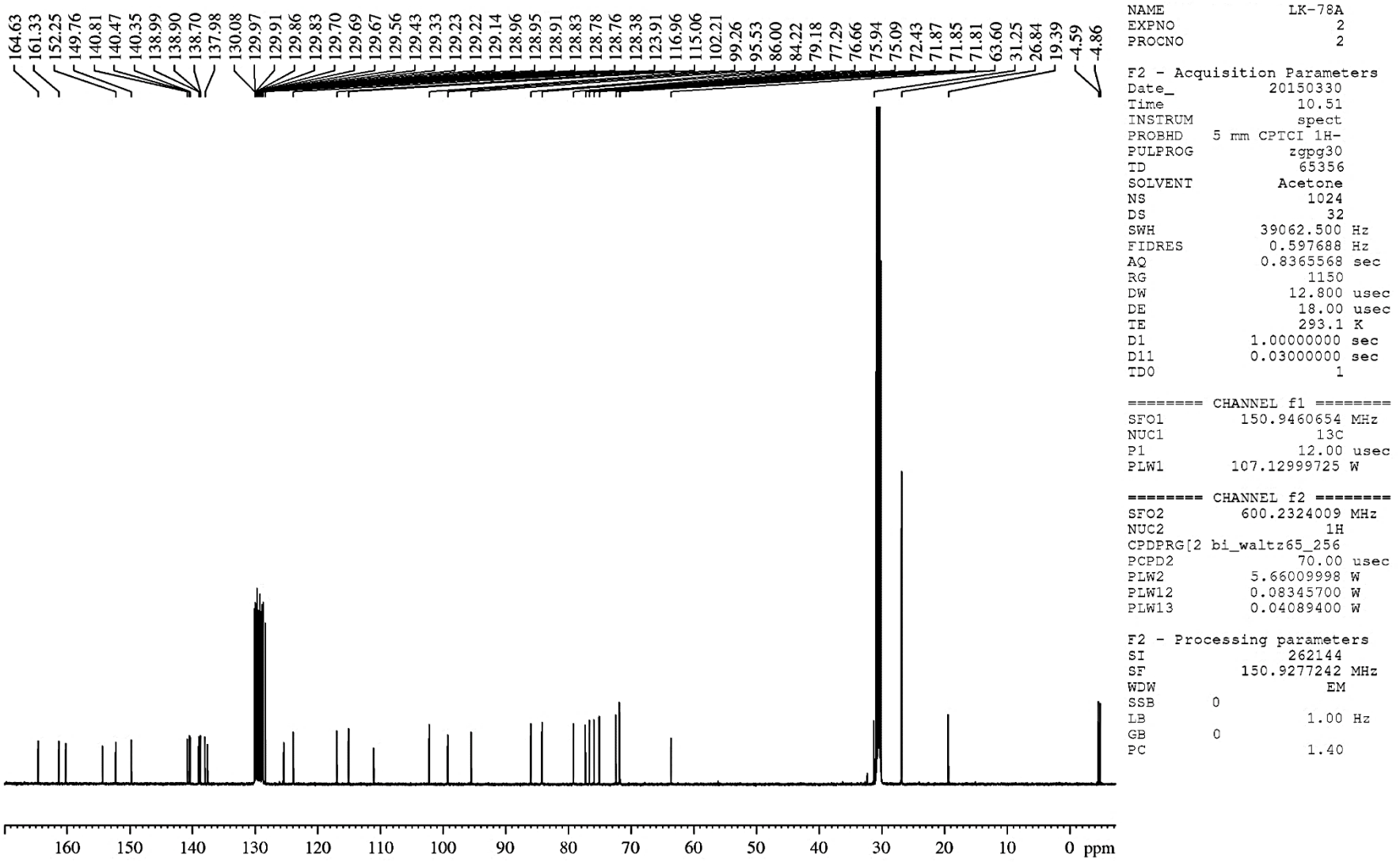


Figure S24. ^{13}C NMR spectrum of compound **33** (acetone, 293.2 K).

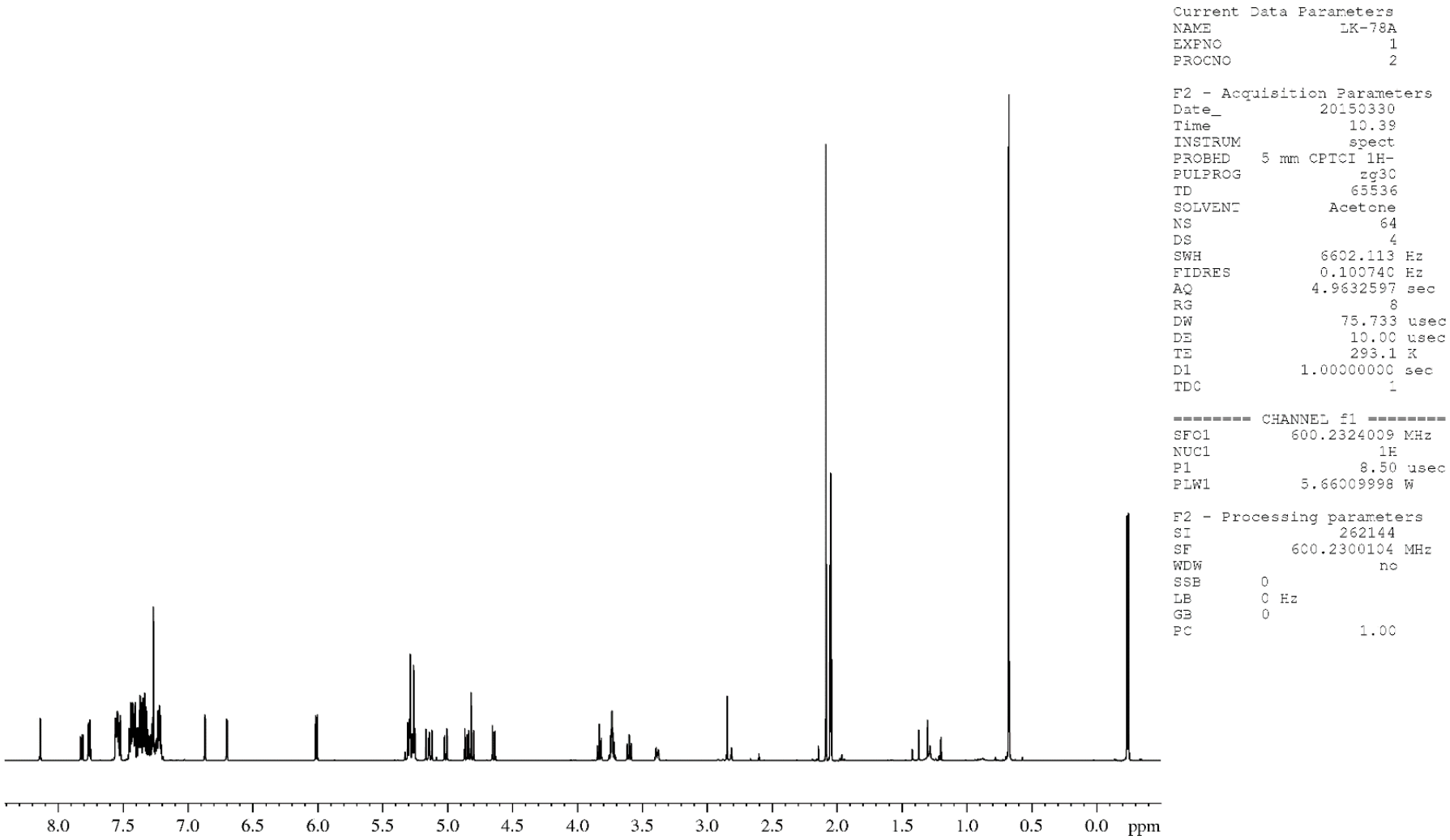


Figure S25. ^{1}H NMR spectrum of compound **33** (acetone, 293.2 K).

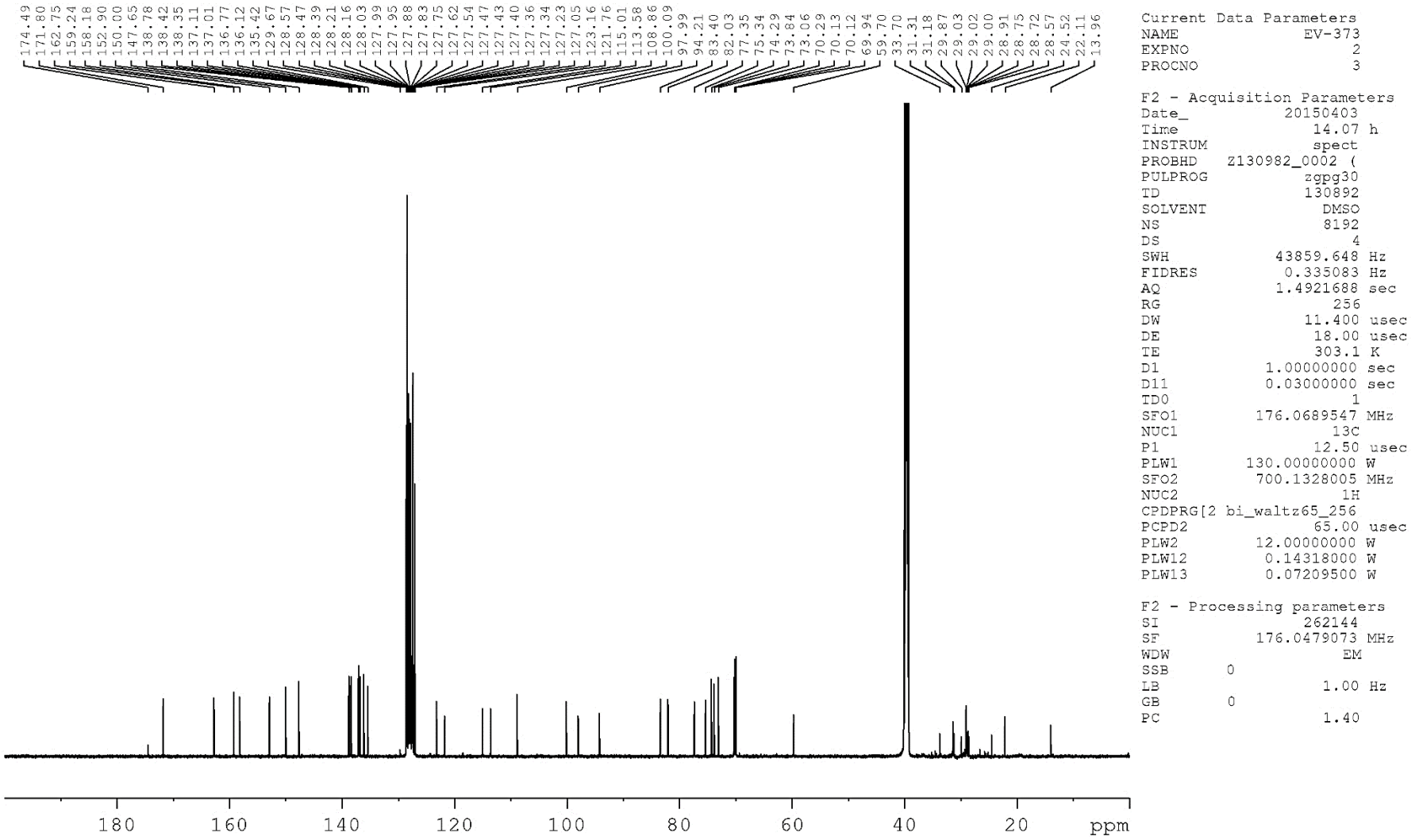


Figure S26. ^{13}C NMR spectrum of compound **34** (DMSO-d_6, 303.2 K).

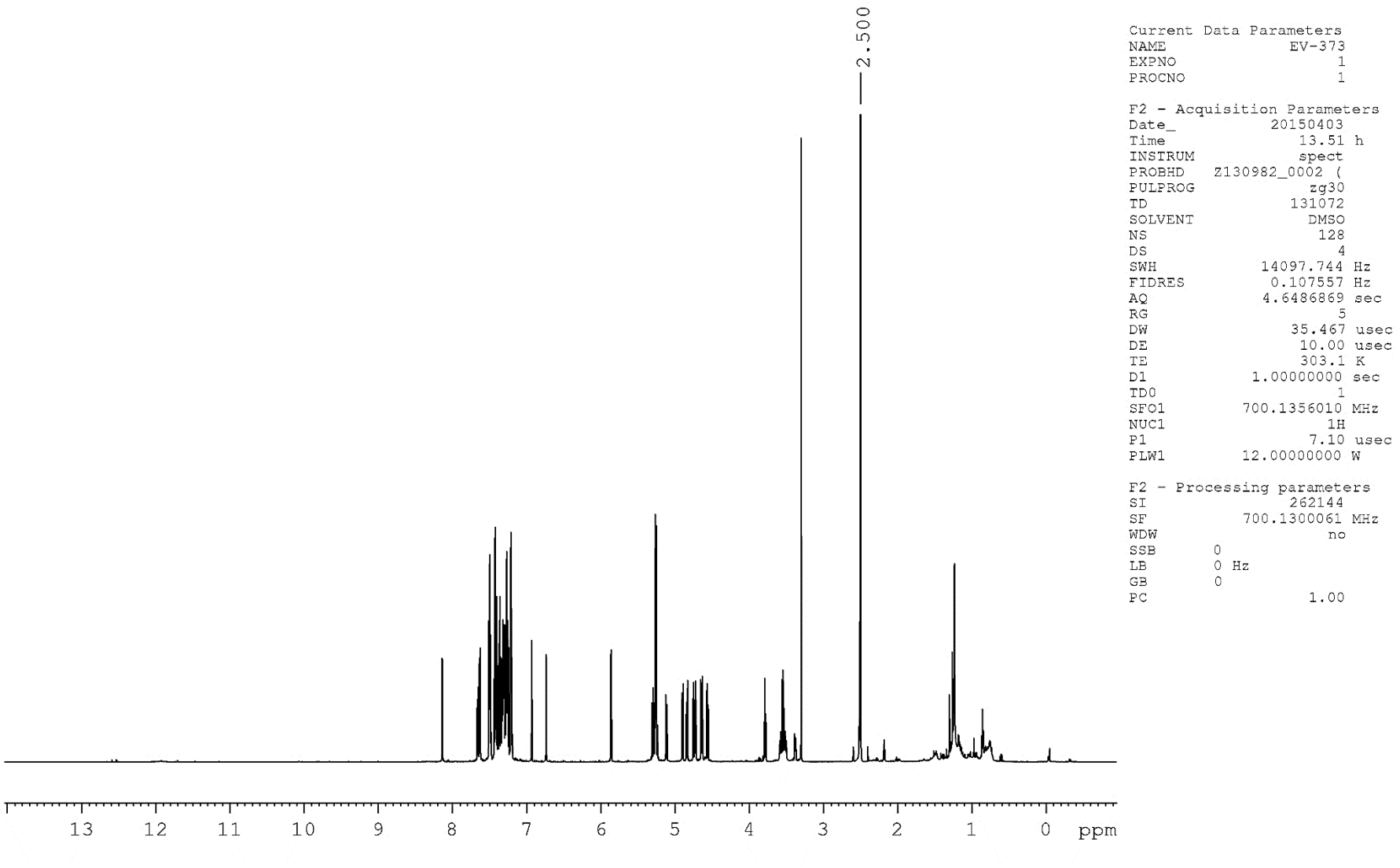


Figure S27. ^{1}H NMR spectrum of compound **34** (DMSO-d_6, 303.2 K).

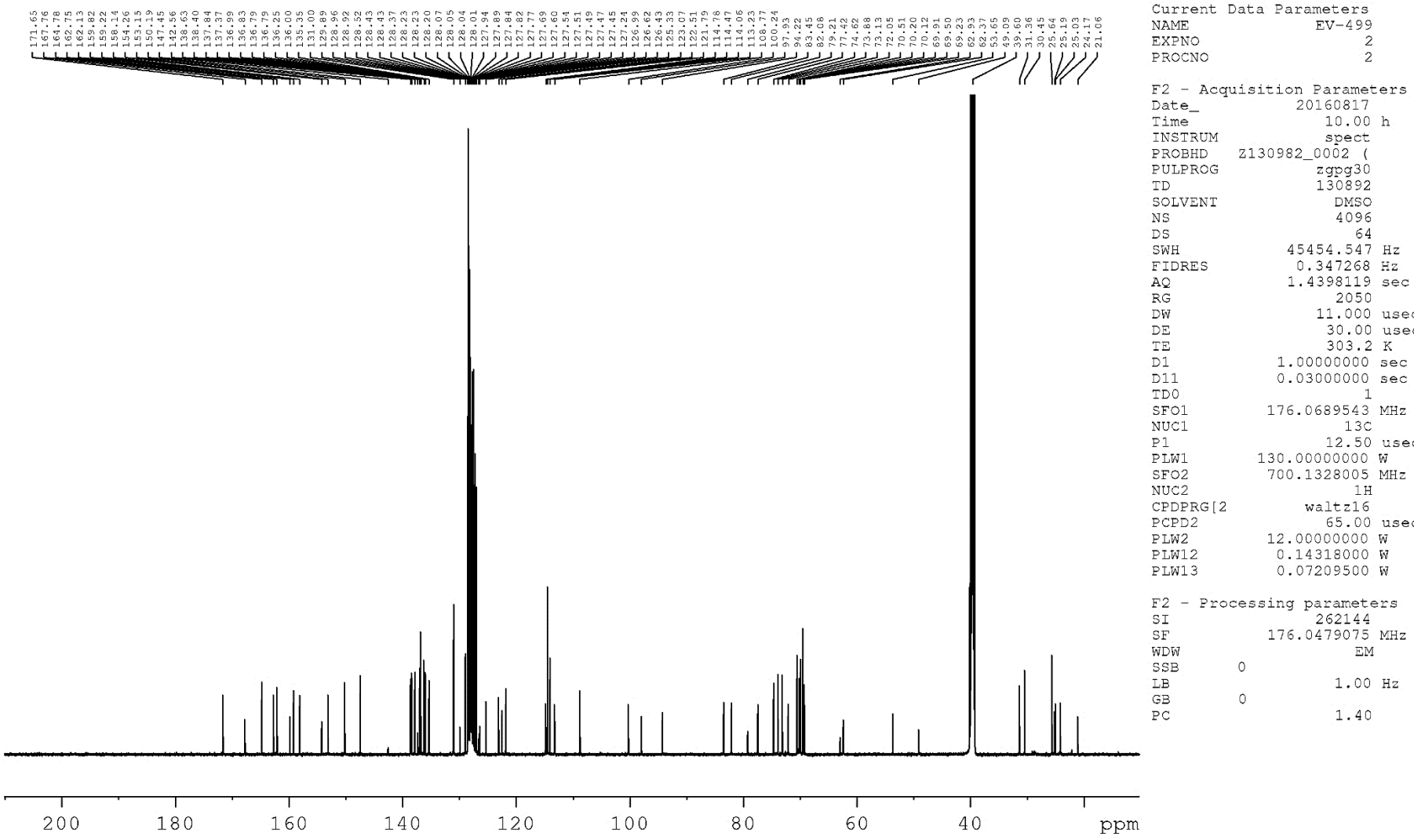
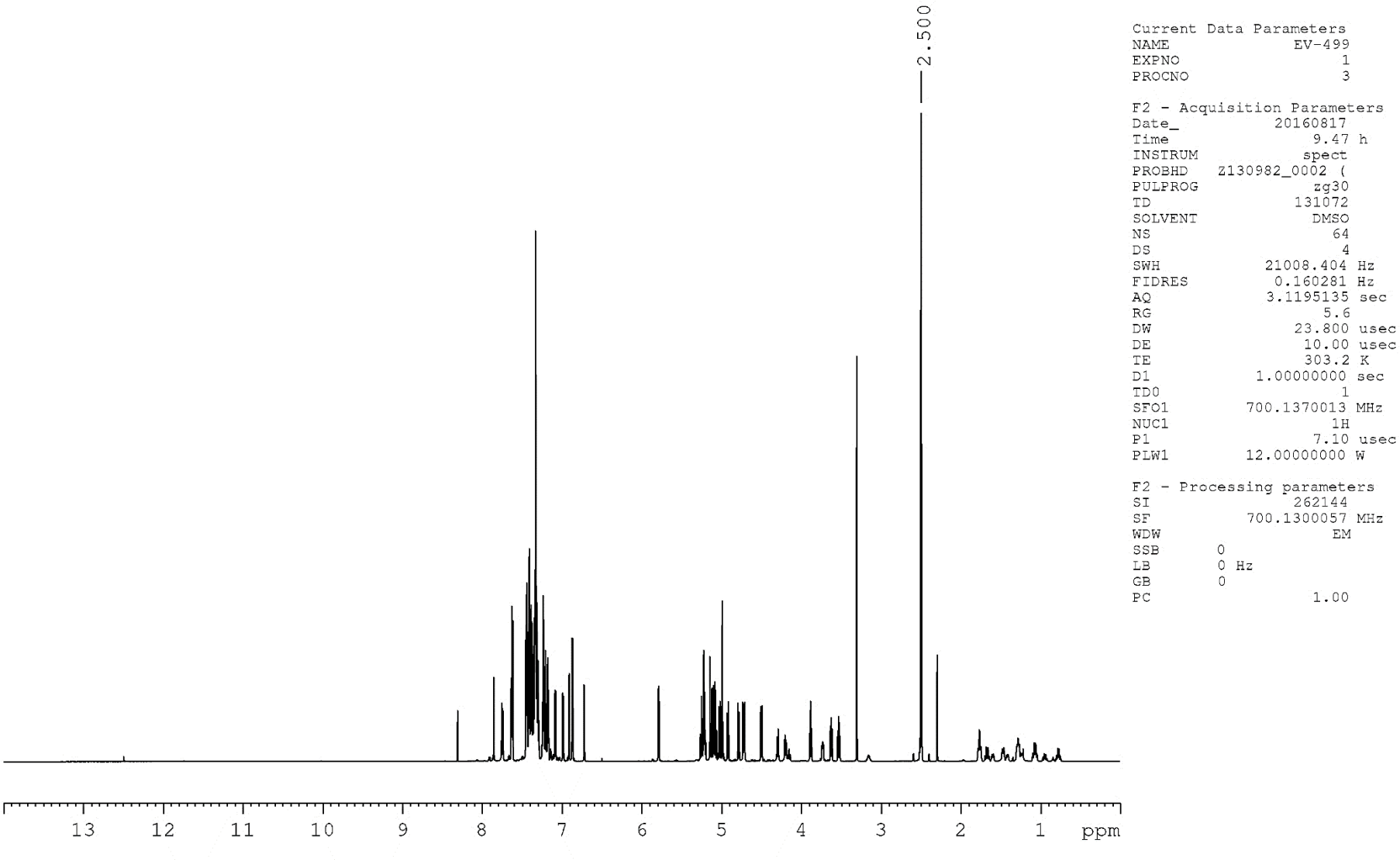


Figure S28. ^{13}C NMR spectrum of compound **35** (DMSO-d_6, 303.2 K).

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PROCNO                     3

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TD                   131072
SOLVENT                DMSO
NS                       64
DS                        4
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AQ               3.1195135 sec
RG                      5.6
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DE                 10.00 usec
TE                  303.2 K
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NUC1                    1H
P1                  7.10 usec
PLW1           12.00000000 W

F2 - Processing parameters
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GB        0
PC                      1.00
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Figure S29. ^{1}H NMR spectrum of compound **35** (DMSO-d_6, 303.2 K).

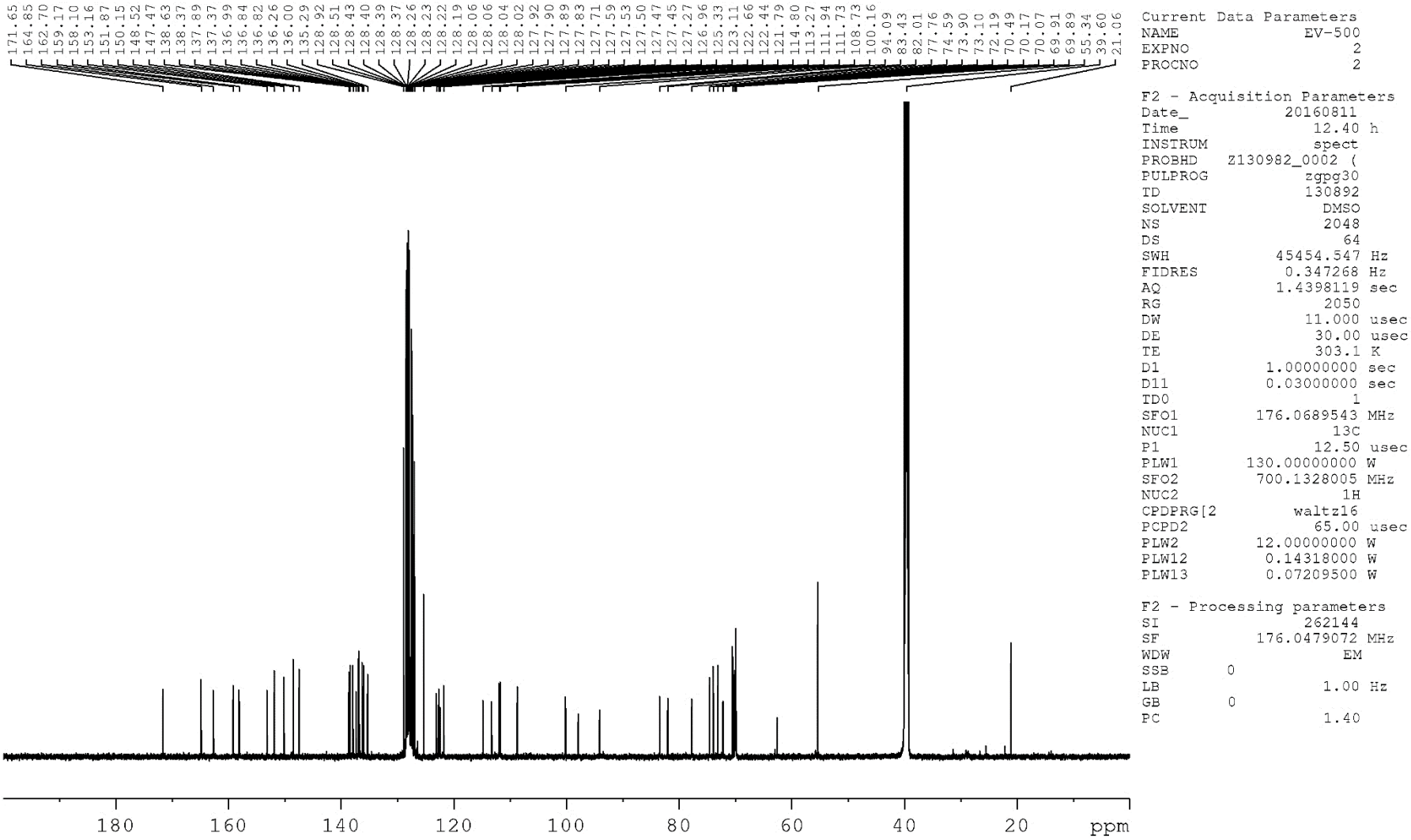


Figure S30. ^{13}C NMR spectrum of compound **36** (DMSO-d_6, 303.2 K).

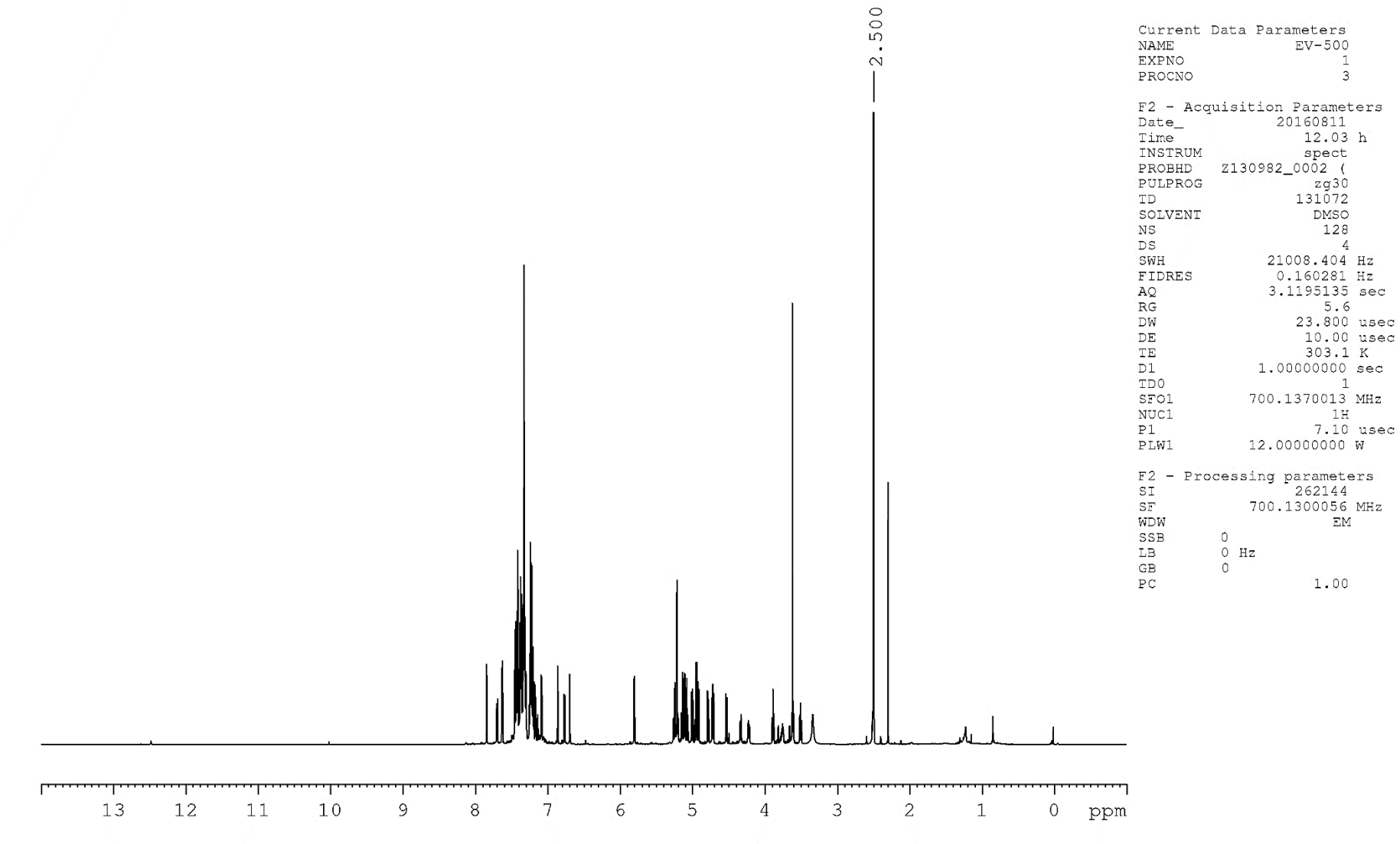


Figure S31. ^{1}H NMR spectrum of compound **36** (DMSO-d_6, 303.2 K).

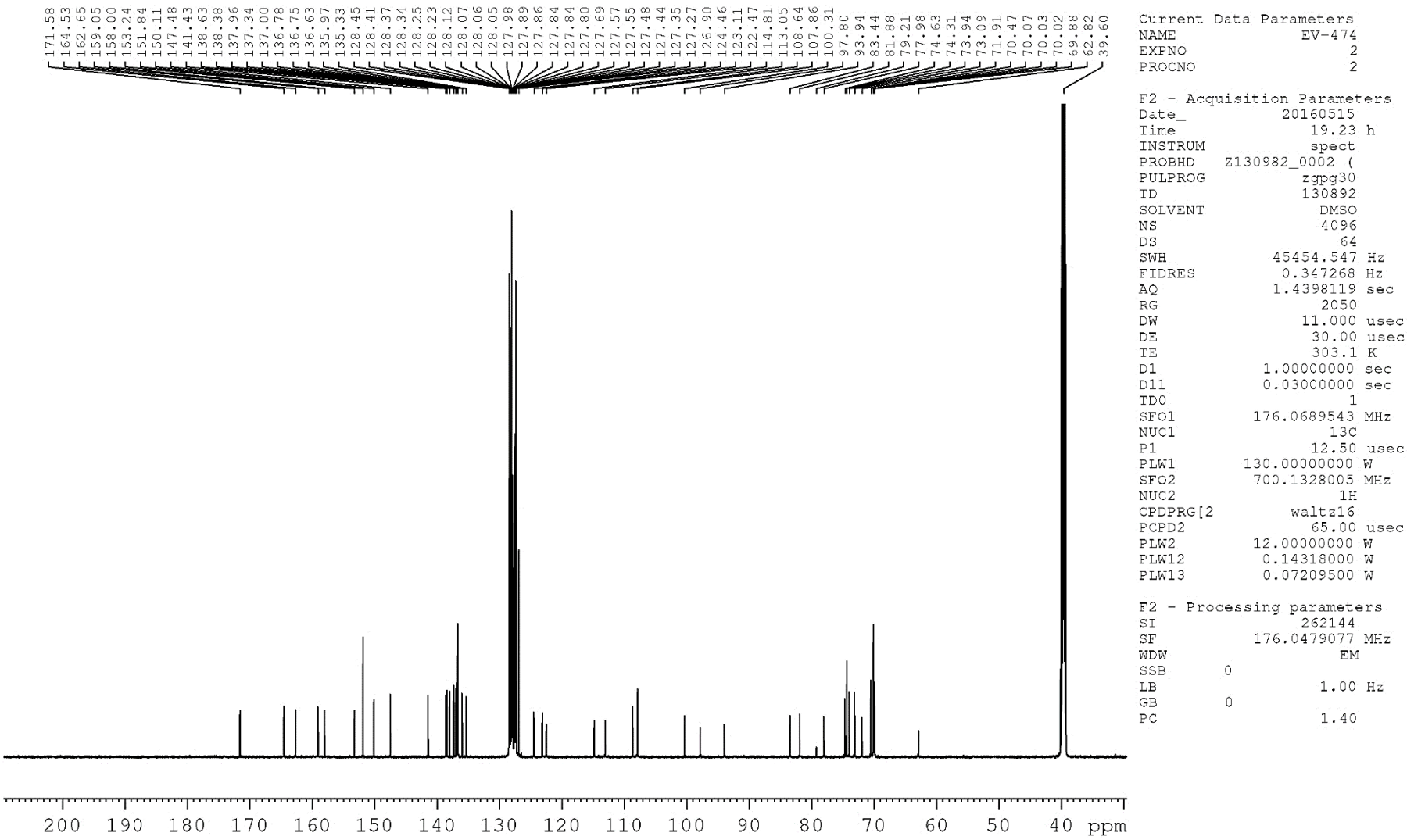


Figure S32. ^{13}C NMR spectrum of compound **37** (DMSO-d_6, 303.2 K).

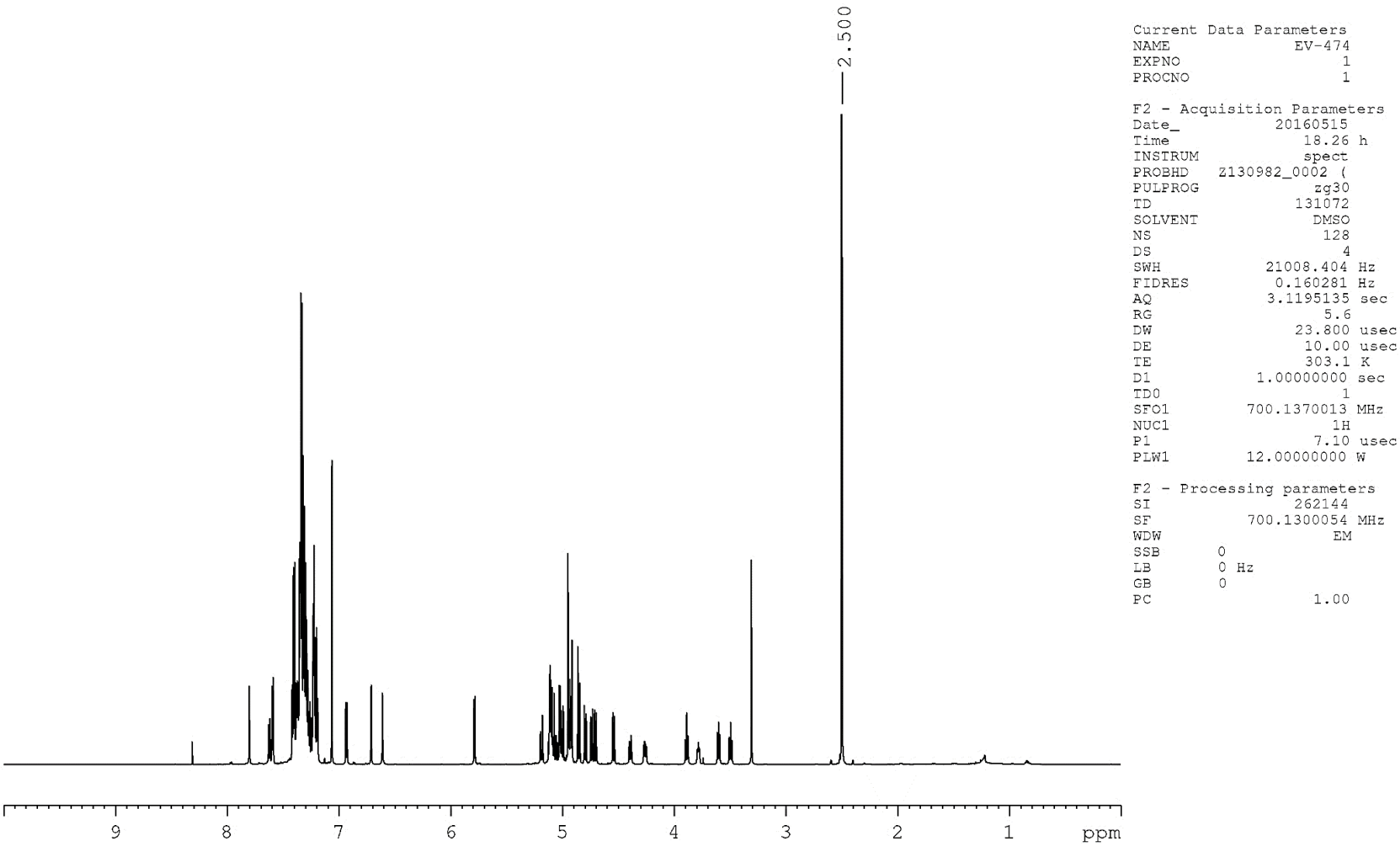


Figure S33. ^{1}H NMR spectrum of compound **37** (DMSO-d_6, 303.2 K).

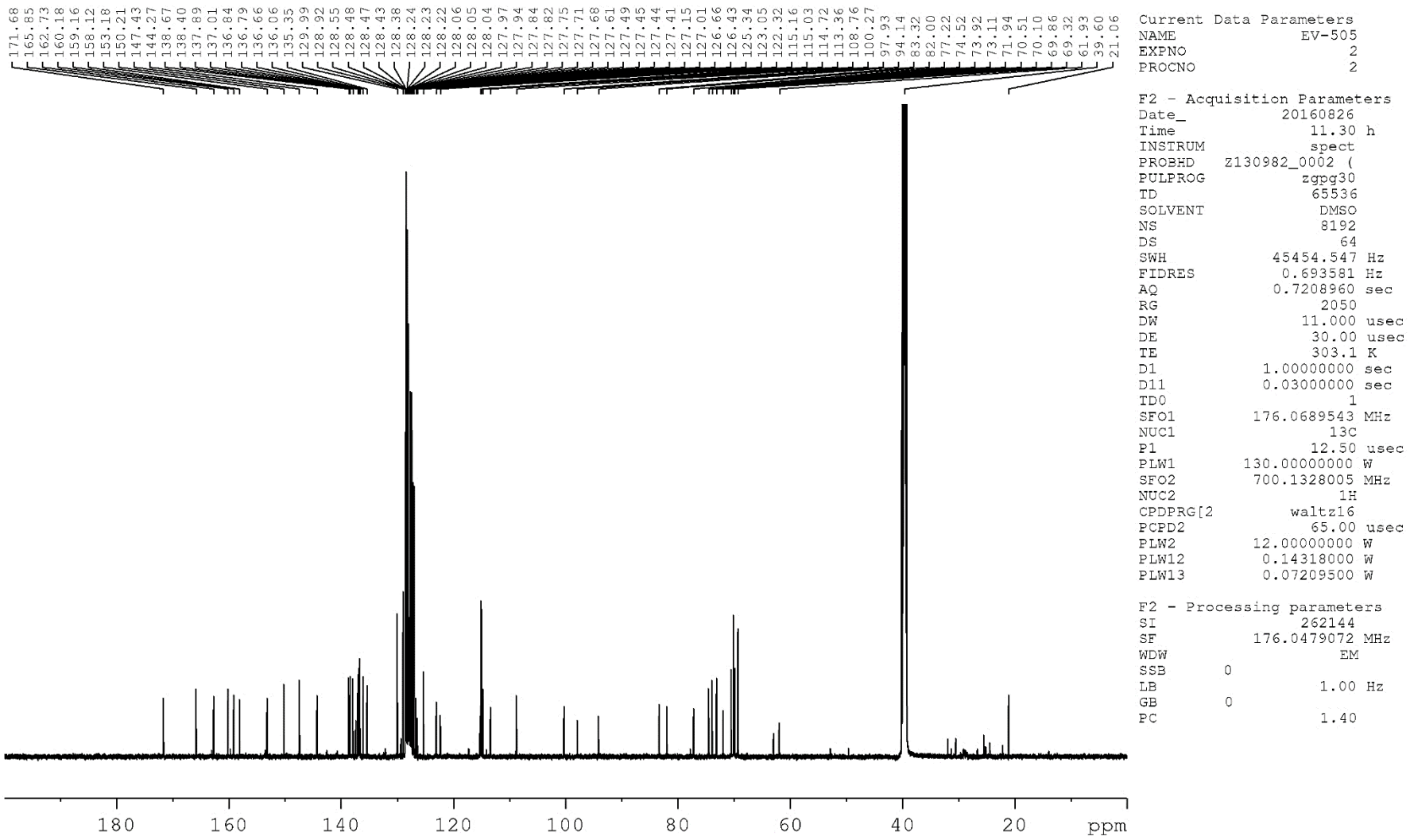
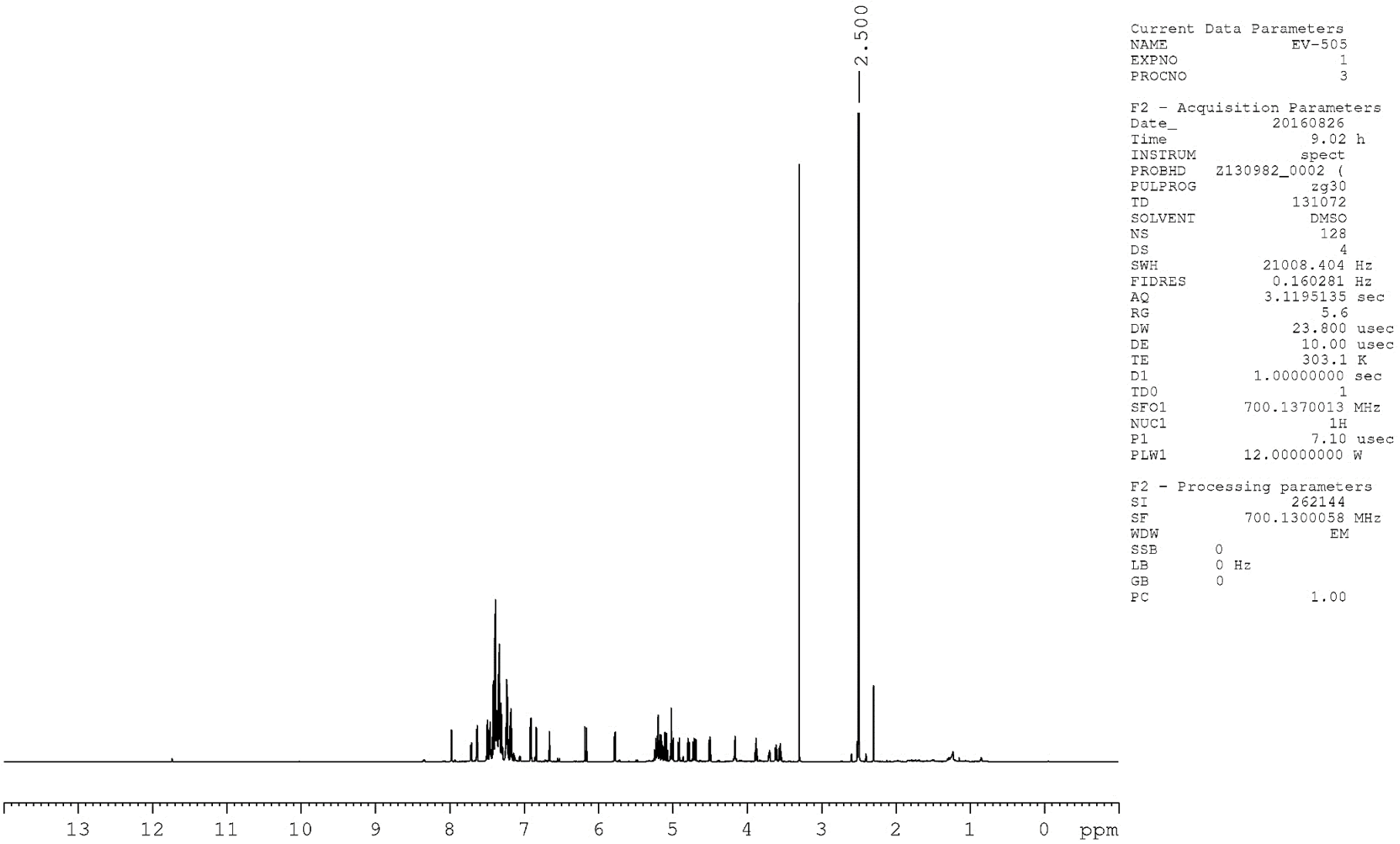


Figure S34. ^{13}C NMR spectrum of compound **38** (DMSO-d_6, 303.2 K).

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Current Data Parameters
NAME                  EV-505
EXPNO                      1
PROCNO                     3

F2 - Acquisition Parameters
Date_              20160826
Time                  9.02 h
INSTRUM               spect
PROBHD    Z130982_0002 (
PULPROG                zg30
TD                   131072
SOLVENT                DMSO
NS                      128
DS                        4
SWH               21008.404 Hz
FIDRES             0.160281 Hz
AQ                3.1195135 sec
RG                      5.6
DW                   23.800 usec
DE                   10.00 usec
TE                   303.1 K
D1               1.00000000 sec
TD0                       1
SFO1           700.1370013 MHz
NUC1                    1H
P1                    7.10 usec
PLW1           12.00000000 W

F2 - Processing parameters
SI                     262144
SF             700.1300058 MHz
WDW                       EM
SSB       0
LB        0 Hz
GB        0
PC                     1.00
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Figure S35. ^{1}H NMR spectrum of compound **38** (DMSO-d_6, 303.2 K).

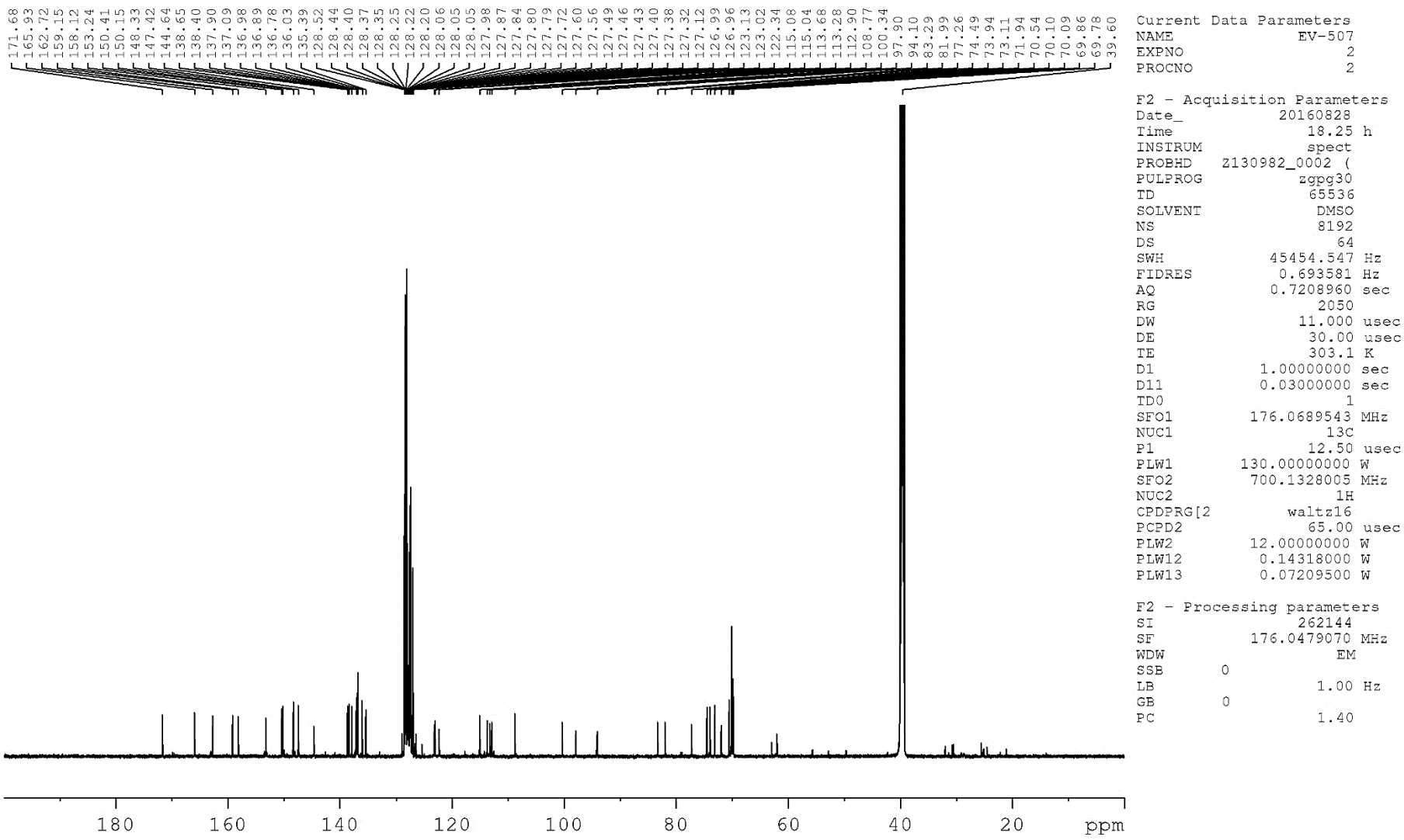
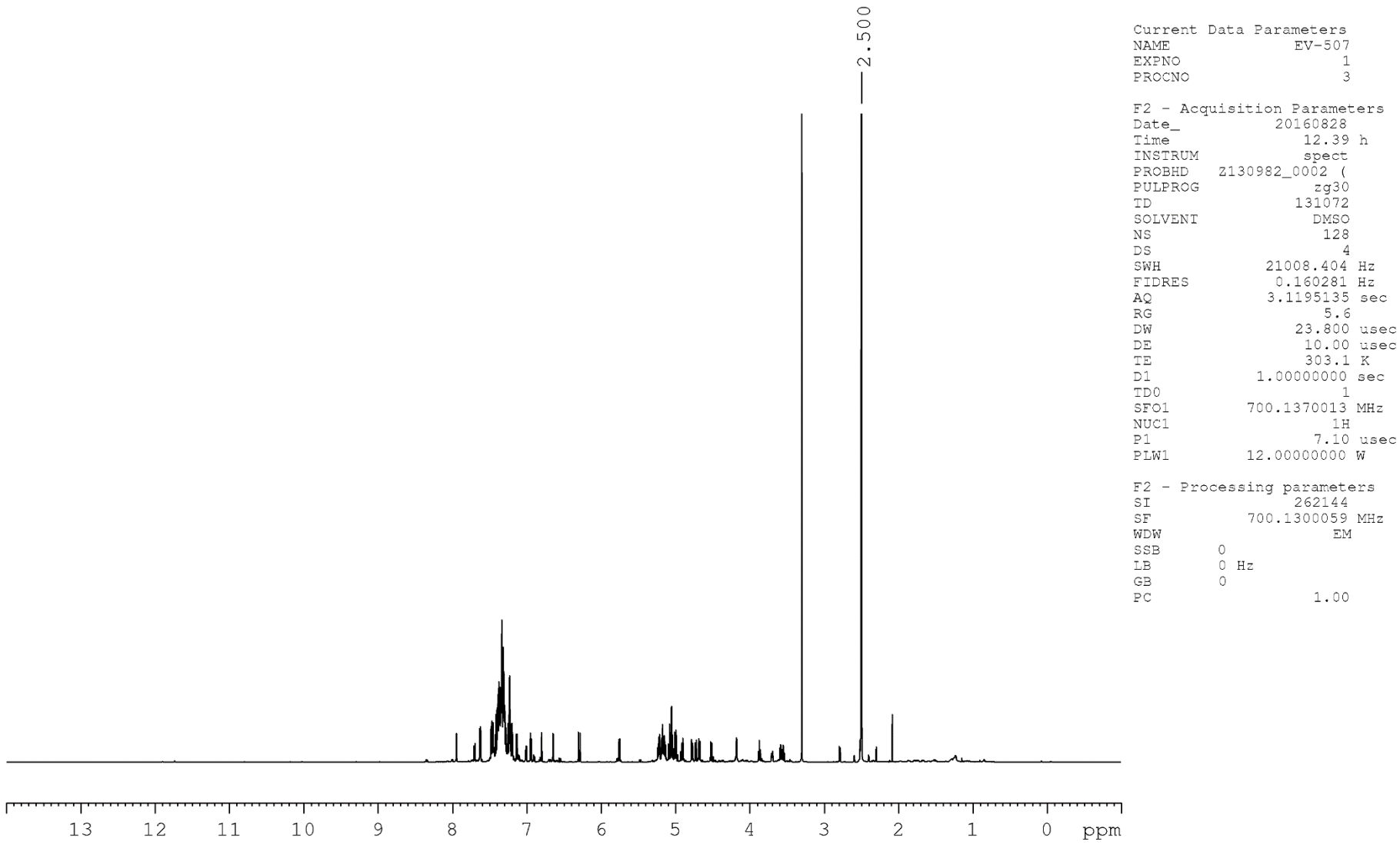


Figure S36. ^{13}C NMR spectrum of compound **39** (DMSO-d_6, 303.2 K).

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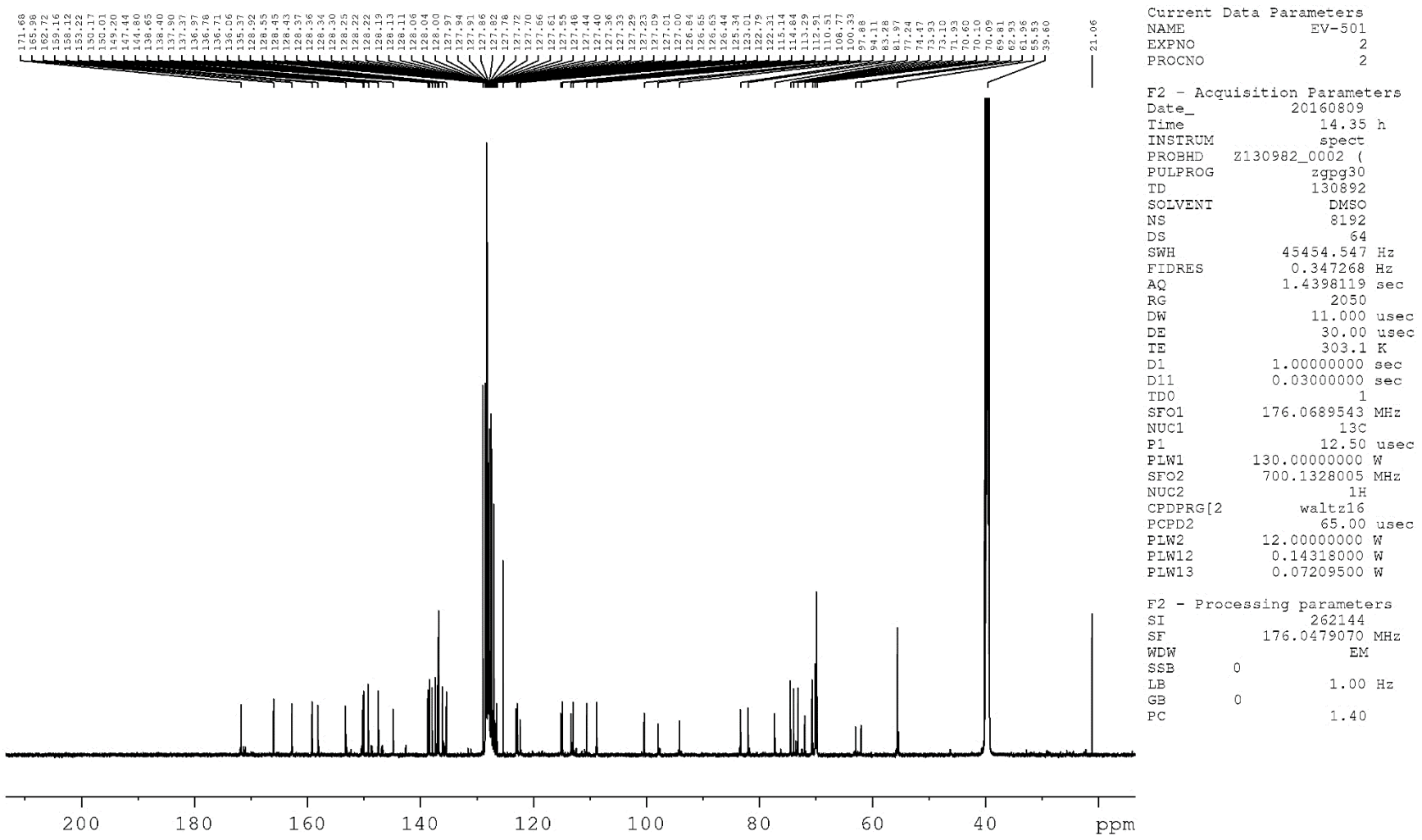
Current Data Parameters
NAME                  EV-507
EXPNO                      1
PROCNO                     3

F2 - Acquisition Parameters
Date_              20160828
Time                 12.39 h
INSTRUM              spect
PROBHD    Z130982_0002 (
PULPROG                zg30
TD                   131072
SOLVENT                DMSO
NS                       128
DS                         4
SWH              21008.404 Hz
FIDRES            0.160281 Hz
AQ               3.1195135 sec
RG                       5.6
DW                  23.800 usec
DE                  10.00 usec
TE                  303.1 K
D1              1.00000000 sec
TD0                        1
SFO1           700.1370013 MHz
NUC1                     1H
P1                    7.10 usec
PLW1           12.00000000 W

F2 - Processing parameters
SI                    262144
SF             700.1300059 MHz
WDW                      EM
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

Figure S37. ^{1}H NMR spectrum of compound **39** (DMSO-d_6, 303.2 K).




```
Current Data Parameters
NAME                  EV-501
EXPNO                      2
PROCNO                     2

F2 - Acquisition Parameters
Date_              20160809
Time                 14.35 h
INSTRUM                spect
PROBHD    Z130982_0002 (
PULPROG              zgpg30
TD                   130892
SOLVENT                DMSO
NS                      8192
DS                        64
SWH               45454.547 Hz
FIDRES             0.347268 Hz
AQ                1.4398119 sec
RG                      2050
DW                  11.000 usec
DE                  30.00 usec
TE                  303.1 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                        1
SFO1           176.0689543 MHz
NUC1                    13C
P1                  12.50 usec
PLW1          130.00000000 W
SFO2           700.1328005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2               65.00 usec
PLW2           12.00000000 W
PLW12           0.14318000 W
PLW13           0.07209500 W

F2 - Processing parameters
SI                    262144
SF             176.0479070 MHz
WDW                       EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

Figure S38. ^{13}C NMR spectrum of compound **40** (DMSO-d_6, 303.2 K).

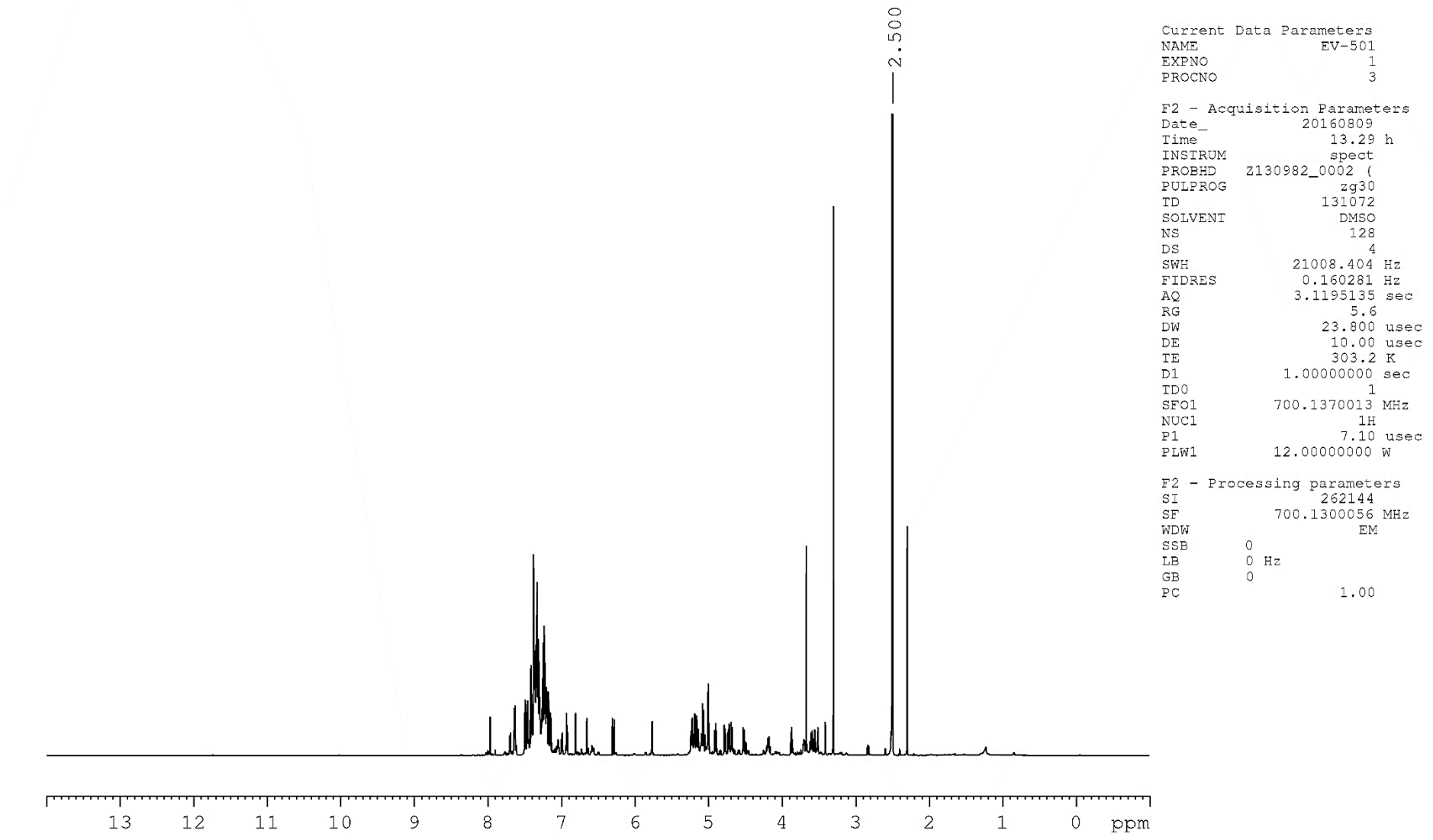


Figure S39. ^{1}H NMR spectrum of compound **40** (DMSO-d_6, 303.2 K).