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Rearrangement of 5',6',7',8'-tetrahydro-1'*H*-spiro(cyclohexane-1,2'-quinazolin)-4'(3'*H*)-one during the Vilsmeier reaction

Victor I. Markov,* Oleg K. Farat, Svetlana A. Varenichenko and Ekaterina V. Velikaya

NMR spectra were recorded on a Varian VXR–300 spectrometer at 299.9 (^{1}H) and 100.6 (^{13}C) MHz as a solution in DMSO-d_6. ^{1}H NMR chemical shifts are referenced to the residual proton signal for DMSO-d_6 (2.50 ppm). ^{13}C NMR chemical shifts are referred to the carbon signal of DMSO-d_6 (39.50 ppm). IR spectra (4000–600 cm^{-1}) were recorded on a Bruker ALPHA FT-IR with a prefix ATR. Mass spectra are fixed on the device Kratos MS 890 device, with direct introduction of the sample into a source of ions at chambers temperature 180-250°C, energy of ionizing ions 70 eV.

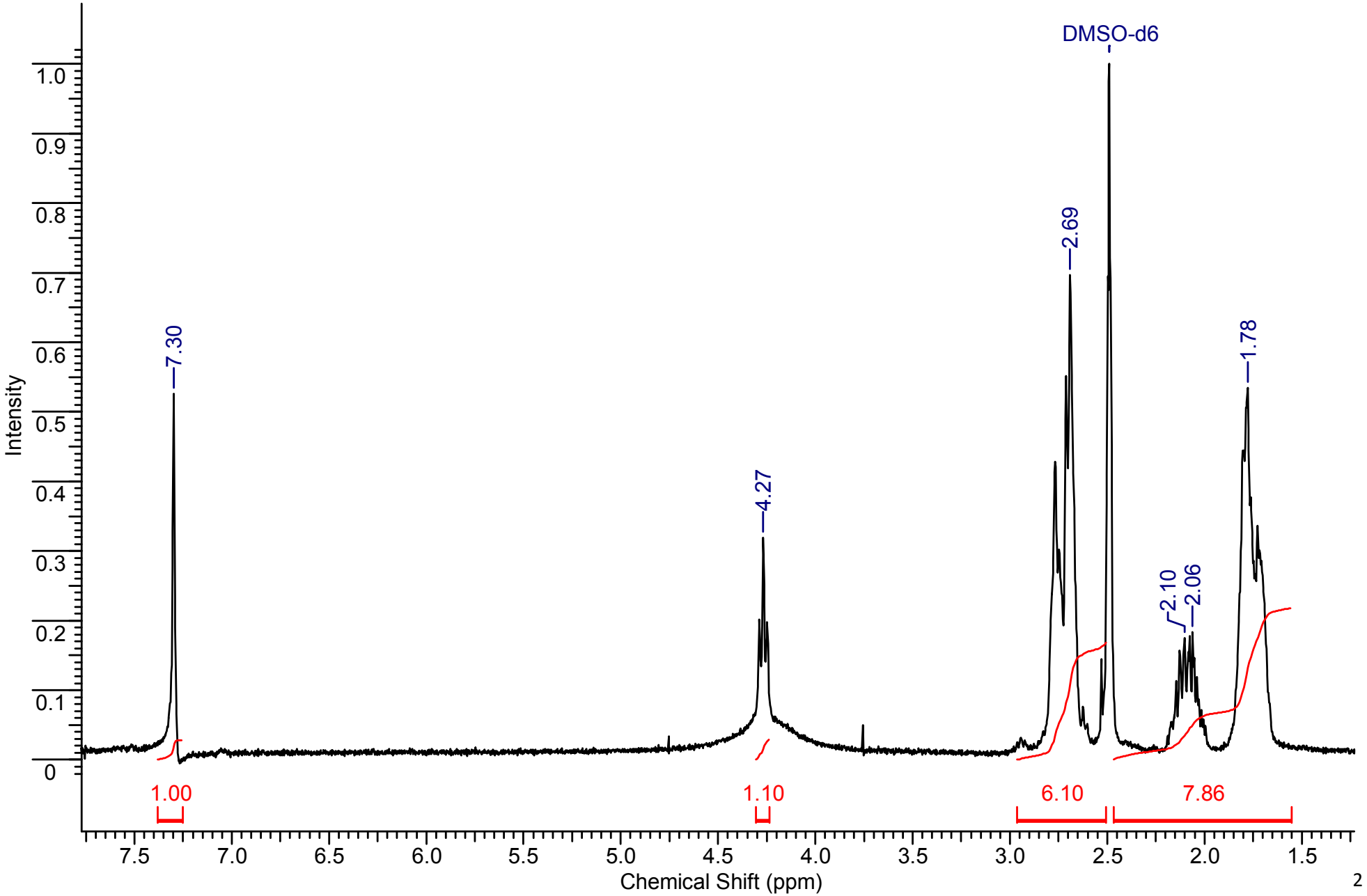
X-ray diffraction study of compound 7.

The structure of compound **7** was established by X-ray diffraction analysis.

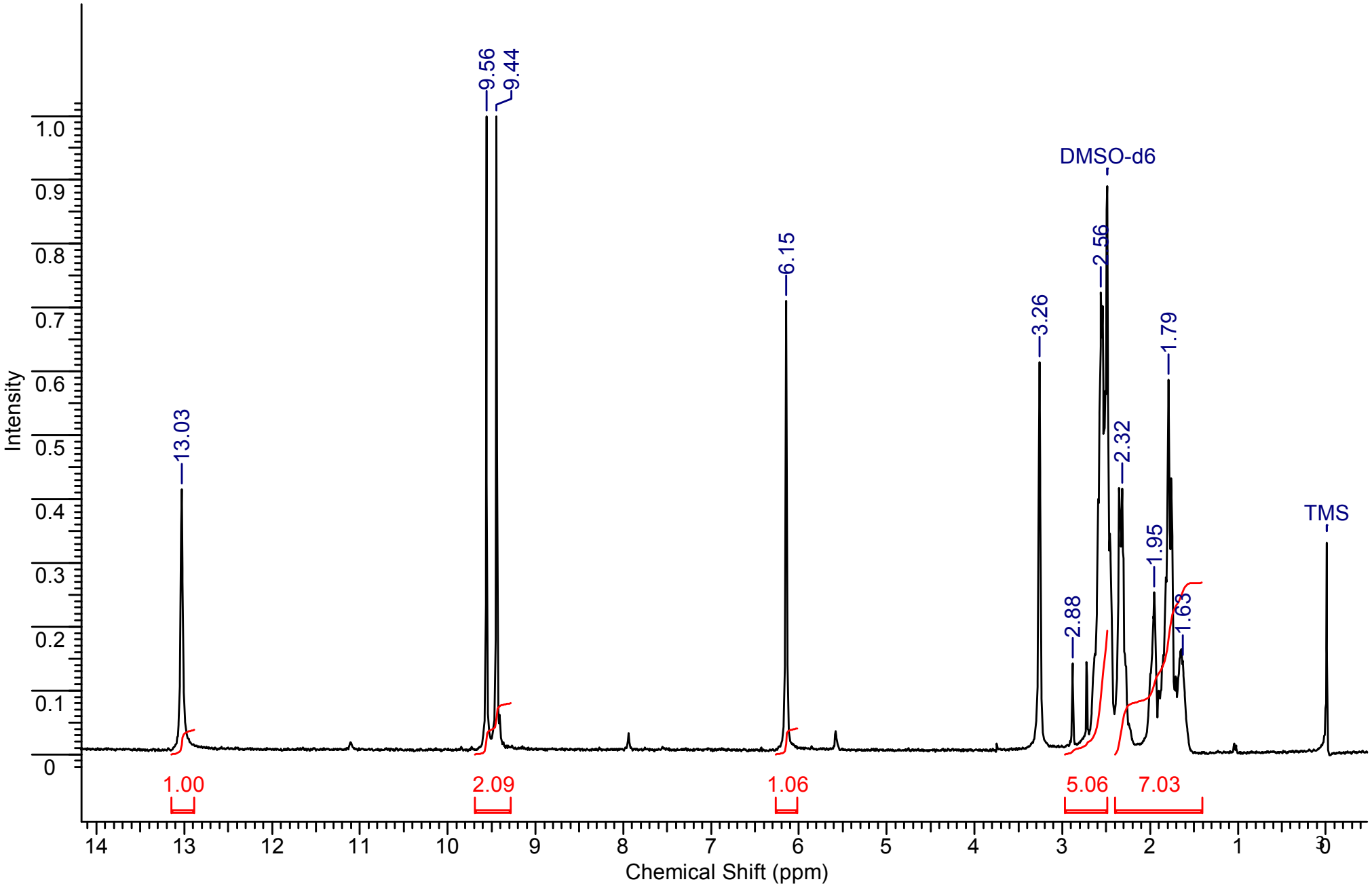
All three rings in compound **7** are non-planar. The C7-C12 ring is disordered over two conformations A and B with occupation ratio 0.68:0.32. In A the ring has a distorted half-chair conformation with deviations of the C10 and C11 atoms from the plane of the C7=C8 double bond of 0.438(15) Å and -0.201(11) Å, respectively. Conformation B may be described as a twist-boat. The deviations of the C10 and C11 atoms from the mean plane of the remaining ring atoms are -0.61(2) Å and -0.201(11) Å, respectively. Another cyclohexene ring adopts a sofa conformation. The C2 atom deviates from the plane of the remaining ring atoms by 0.644(8) Å. Tetrahydropyridine ring has a twist-boat conformation. The deviations of the C1 and C6 atoms from the plane of the C12=C13 double bond are -0.207(8) Å and -0.416(8) Å, respectively.

Carbonyl groups are almost coplanar to the C=C double bonds of the cyclohexene rings and have s-cis conformation with respect to these double bonds (the C6-C5-C15-O1 and C7-C8-C16-O2 torsion angles are 11.5(9)° and 0.0(9)°). Such conformation of acrolein fragments is stabilized by formation of bifurcation intramolecular hydrogen bonds in which hydrogen atom at the N1 atom is involved: H1…O1 2.08 Å, N-H…O 130° and H1…O2 2.02 Å, N-H…O 134°.

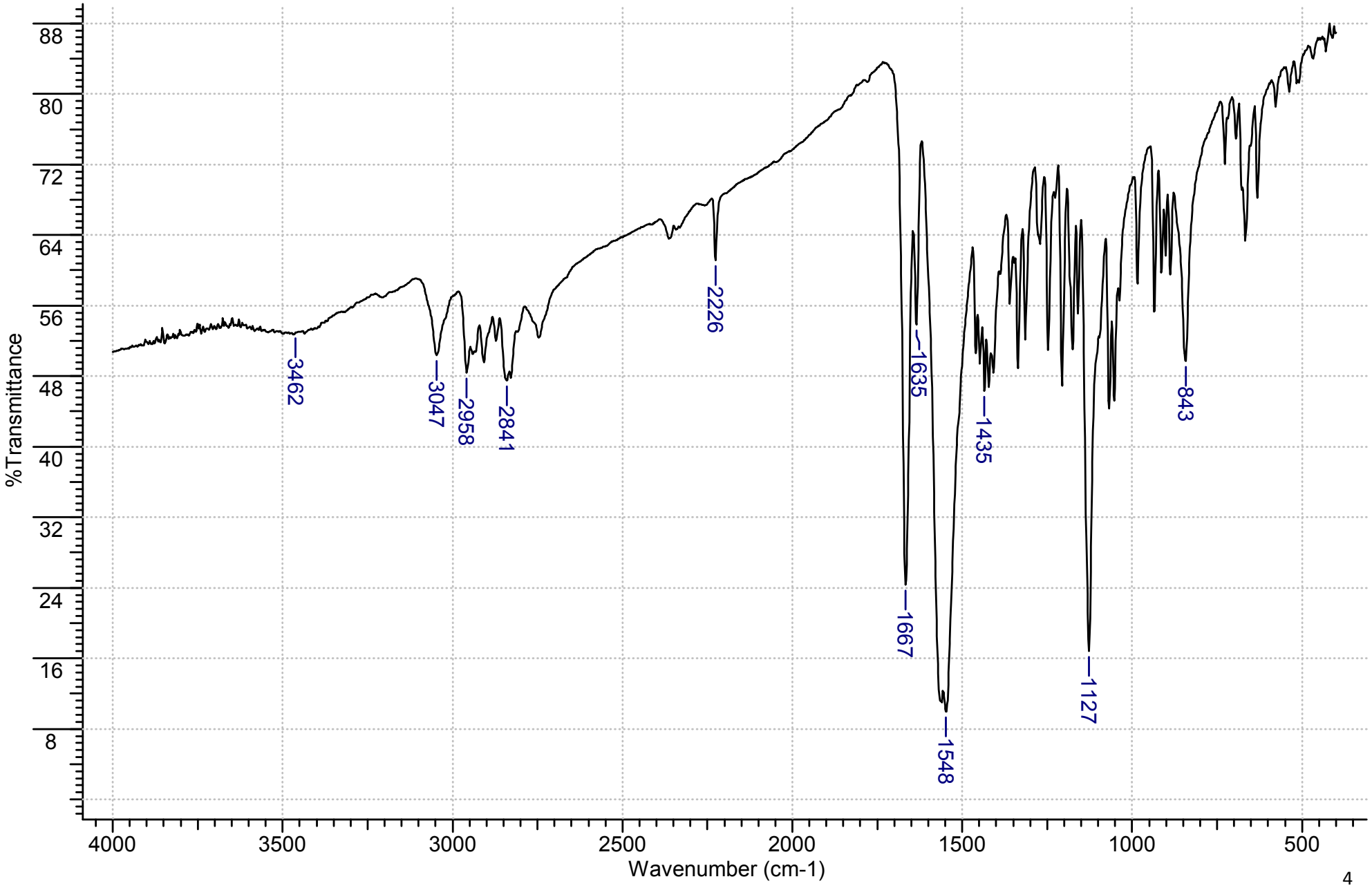
^{1}H NMR spectrum of **6** in DMSO-d_6, Varian VXR – 300

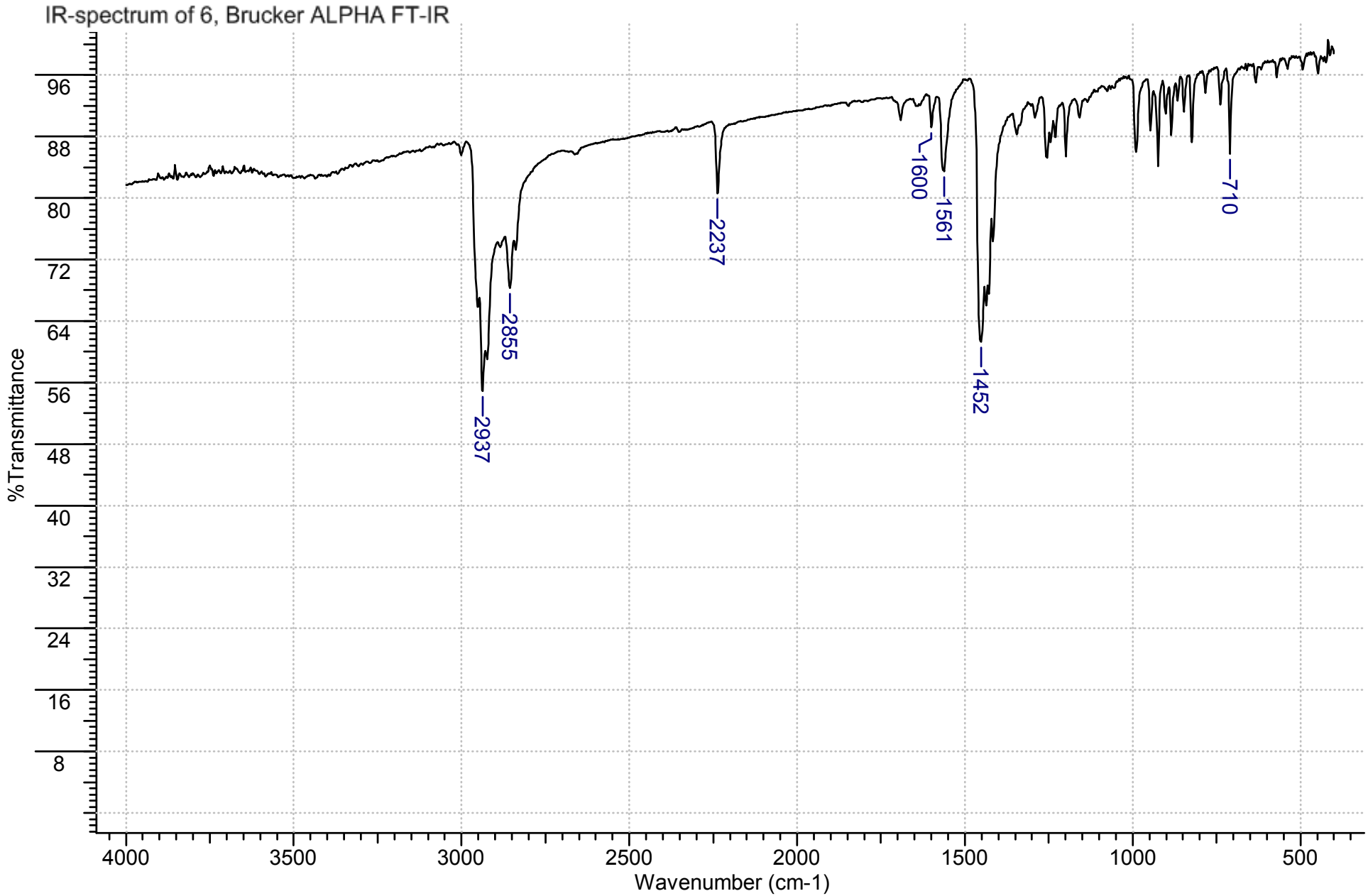


^{1}H NMR spectrum of **7** in DMSO-d_6, Varian VXR – 300

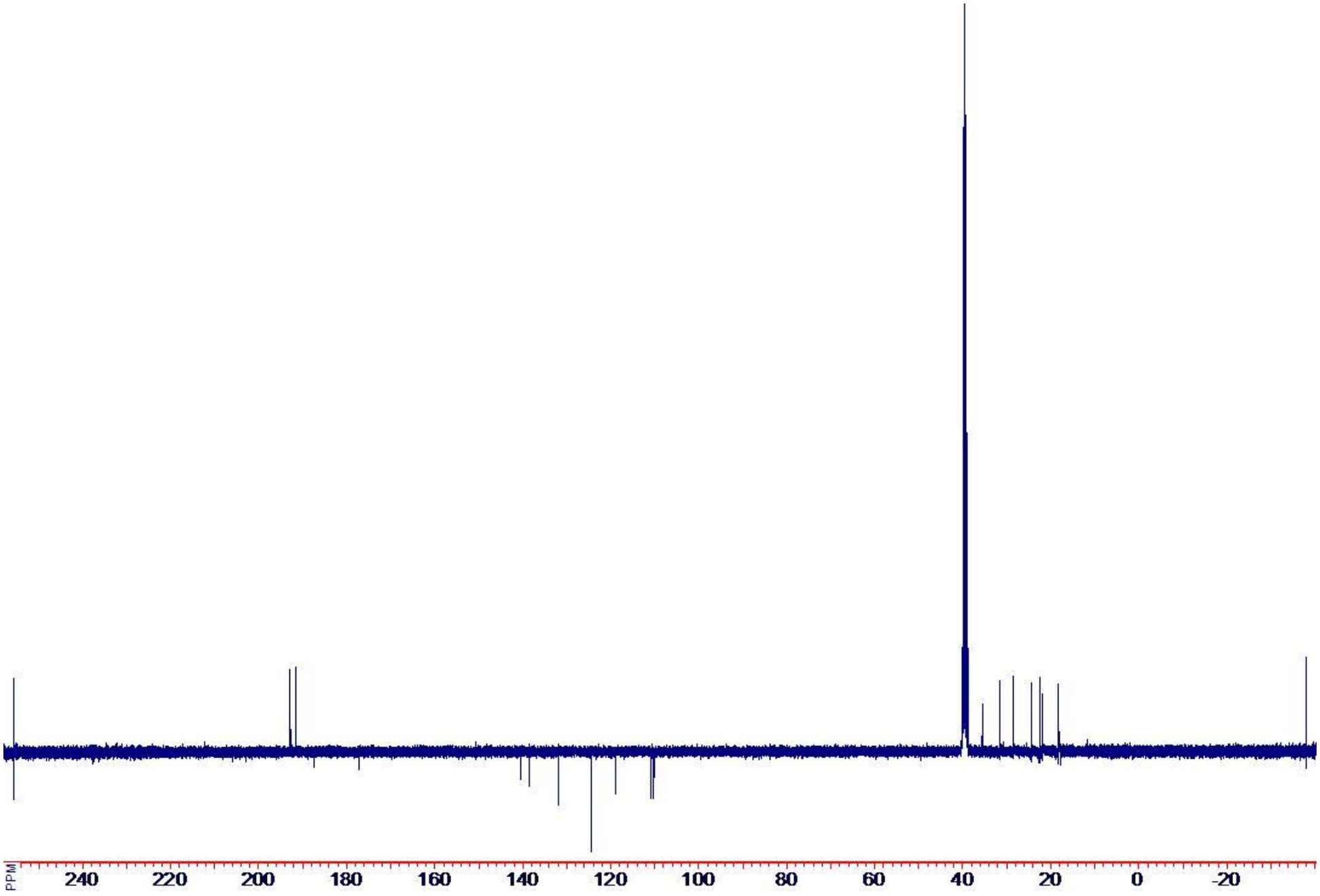


IR-spectrum of 7, Bruker ALPHA FT-IR

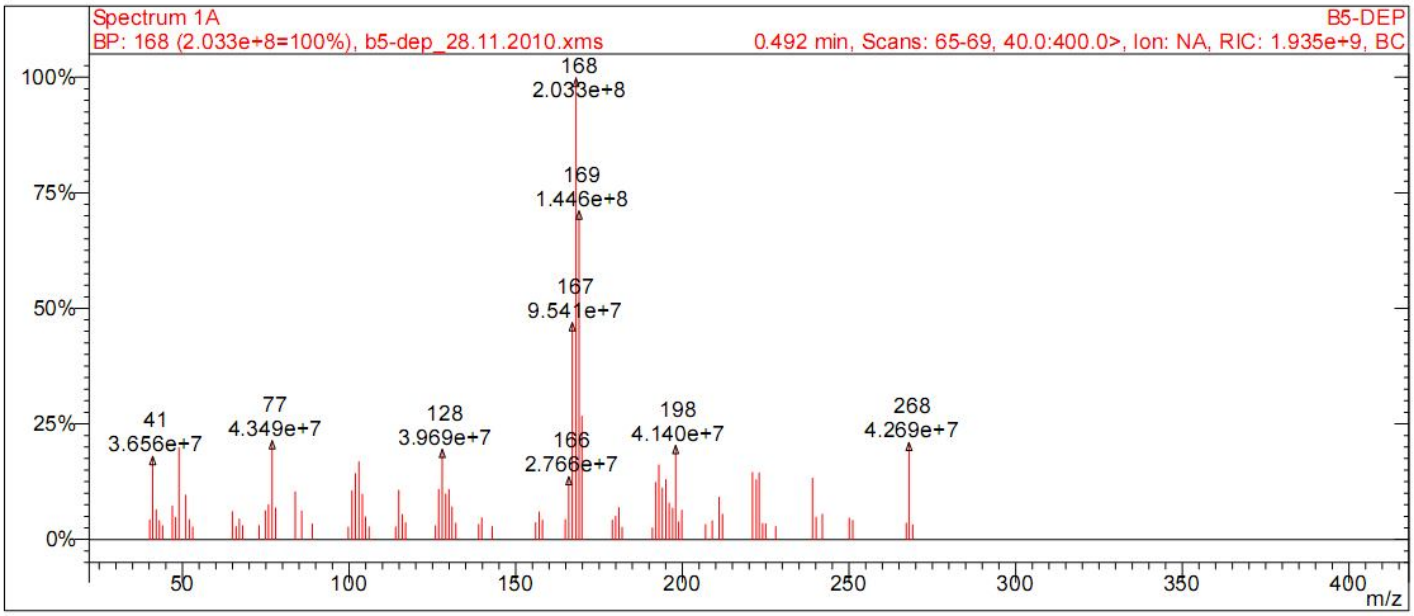



IR-spectrum of 6, Brucker ALPHA FT-IR
%Transmittance
96
88
80
72
64
56
48
40
32
24
16
8
2937
2855
2237
1600
1561
1452
710
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)

^{13}C NMR spectrum of **7** in DMSO-d_6, Varian VXR – 300

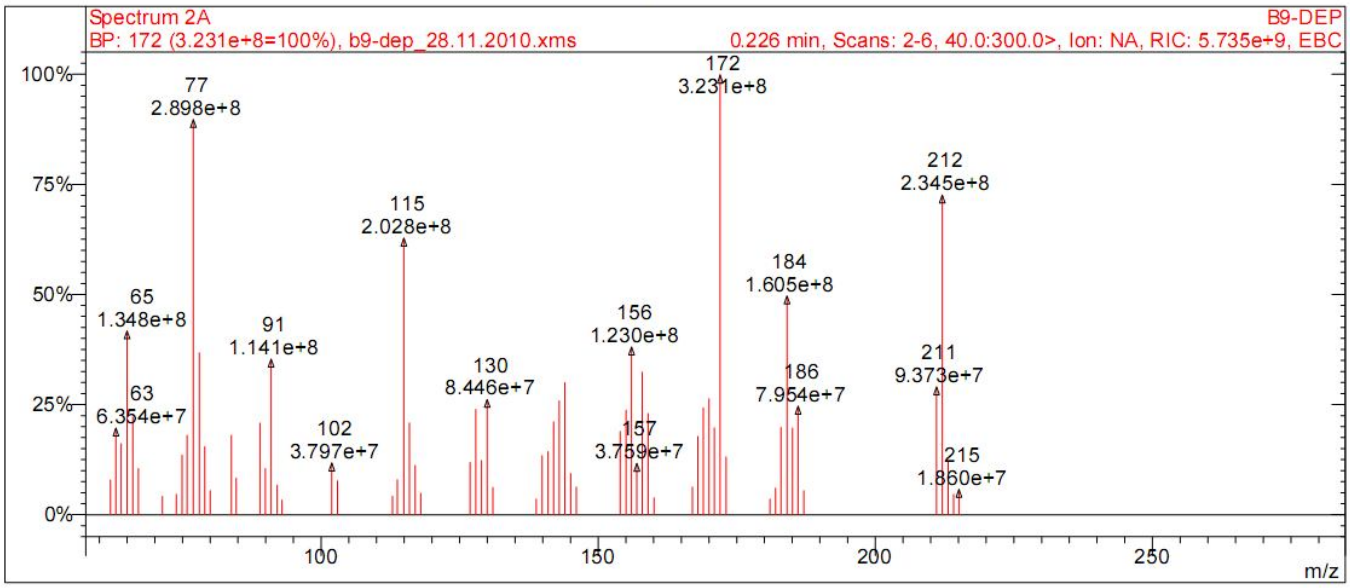


Mass spectrum of **7**, Kratos MS 890



Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
40	8.646e+6	42	106	5.528e+6	27	191	5.106e+6	25
41	3.656e+7	180	114	5.540e+6	27	192	2.511e+7	123
42	1.308e+7	64	115	2.169e+7	107	193	3.280e+7	161
43	8.296e+6	41	116	1.098e+7	54	194	2.262e+7	111
44	6.091e+6	30	117	7.357e+6	36	195	2.635e+7	129
47	1.464e+7	72	126	6.013e+6	30	196	1.600e+7	79
48	9.740e+6	48	127	2.200e+7	108	197	1.381e+7	68
49	4.036e+7	198	128	3.969e+7	195	198	4.140e+7	203
51	1.955e+7	96	129	1.999e+7	98	199	7.710e+6	38
52	8.752e+6	43	130	2.203e+7	108	200	1.286e+7	63
53	5.436e+6	27	131	1.432e+7	70	207	6.648e+6	33
65	1.219e+7	60	132	7.165e+6	35	209	8.206e+6	40
66	5.655e+6	28	139	6.612e+6	32	211	1.858e+7	91
67	8.929e+6	44	140	9.508e+6	47	212	1.112e+7	55
68	6.090e+6	30	143	5.619e+6	28	221	2.951e+7	145
73	6.107e+6	30	156	7.411e+6	36	222	2.629e+7	129
75	1.265e+7	62	157	1.206e+7	59	223	2.934e+7	144
76	1.524e+7	75	158	8.512e+6	42	224	7.053e+6	35
77	4.349e+7	214	165	8.720e+6	43	225	6.860e+6	34
78	1.391e+7	68	166	2.766e+7	136	228	5.727e+6	28
84	2.097e+7	103	167	9.541e+7	469	239	2.692e+7	132
86	1.256e+7	62	168	2.033e+8	999	240	9.766e+6	48
89	6.789e+6	33	169	1.446e+8	710	242	1.114e+7	55
100	5.430e+6	27	170	5.428e+7	267	250	9.364e+6	46
101	2.144e+7	105	179	8.524e+6	42	251	8.422e+6	41
102	2.899e+7	142	180	1.026e+7	50	267	7.124e+6	35
103	3.420e+7	168	181	1.405e+7	69	268	4.269e+7	210
104	1.995e+7	98	182	5.350e+6	26	269	6.367e+6	31
105	9.931e+6	49						

Mass spectrum of **6**, Kratos MS 890



Ion	Int	Norm	Ion	Int	Norm	Ion	Int	Norm
...	...		103	2.482e+7	77	157	3.759e+7	116
62	2.550e+7	79	113	1.368e+7	42	158	1.046e+8	323
63	6.354e+7	196	114	2.571e+7	79	159	7.431e+7	230
64	5.212e+7	161	115	2.028e+8	627	160	1.255e+7	39
65	1.348e+8	417	116	6.733e+7	208	167	2.031e+7	63
66	7.164e+7	222	117	3.617e+7	112	168	5.736e+7	177
67	3.393e+7	105	118	1.568e+7	48	169	7.837e+7	242
71	1.352e+7	42	127	3.842e+7	119	170	8.509e+7	263
74	1.503e+7	46	128	7.725e+7	239	171	6.376e+7	197
75	4.371e+7	135	129	3.982e+7	123	172	3.231e+8	999
76	5.818e+7	180	130	8.446e+7	261	173	4.251e+7	131
77	2.898e+8	896	131	1.999e+7	62	181	1.148e+7	36
78	1.188e+8	367	139	1.153e+7	36	182	1.944e+7	60
79	4.992e+7	154	140	4.339e+7	134	183	6.400e+7	198
80	1.770e+7	55	141	4.634e+7	143	184	1.605e+8	496
84	5.818e+7	180	142	6.823e+7	211	185	6.360e+7	197
85	2.687e+7	83	143	8.366e+7	259	186	7.954e+7	246
89	6.722e+7	208	144	9.692e+7	300	187	1.773e+7	55
90	3.393e+7	105	145	3.026e+7	94	211	9.373e+7	290
91	1.141e+8	353	146	2.028e+7	63	212	2.345e+8	725
92	2.172e+7	67	154	6.103e+7	189	213	3.876e+7	120
93	1.067e+7	33	155	7.673e+7	237	214	1.497e+7	46
102	3.797e+7	117	156	1.230e+8	380	215	1.860e+7	58