

Supporting Information for
Highly Efficient Preparation of Aryl β -Diketo Acids Using
tert-Butyl Methyl Oxalate

Xiao-Hua Jiang, Lai-Dong Song and Ya-Qiu Long*

State Key Laboratory of Drug Research, Shanghai Institute of Materia Medica, Shanghai

Institutes for Biological Sciences, CAS, 555 Zuchongzhi Road, Shanghai 201203, China

yqlong@mail.shcnc.ac.cn

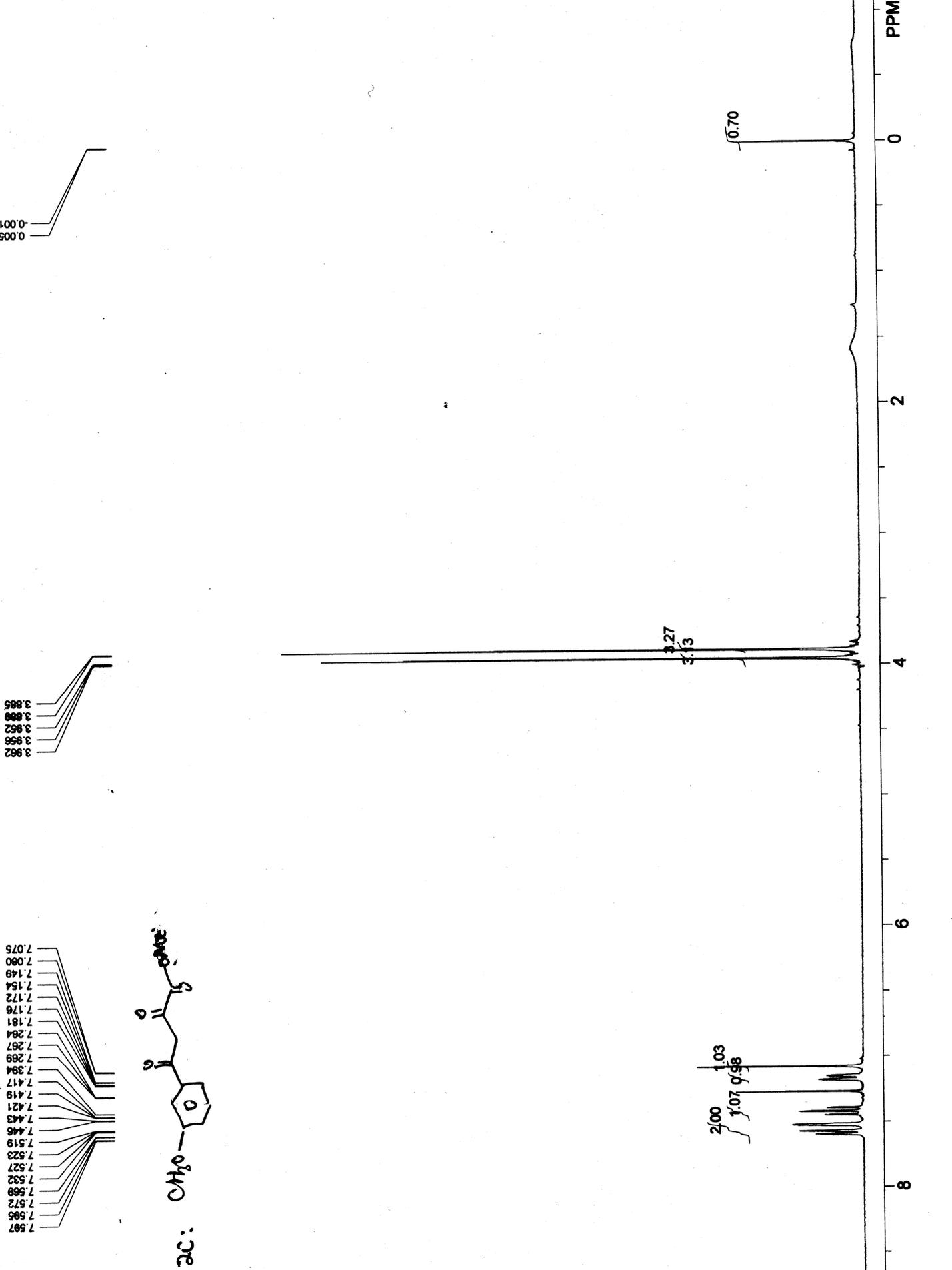
Table of Contents

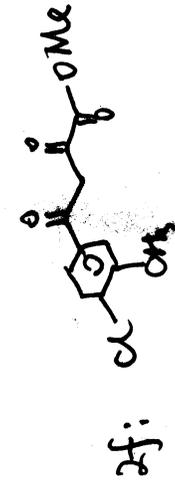
S1	Title Page
S2	General Experimental Section
S3-S9	¹ H NMR Spectra for Selected Compounds, i.e. 2c , 2f , 2h , 2i , 2k , 2l , 4b
S10-S17	¹³ C NMR Spectra for Selected Compounds, i.e. 2a , 2c , 2e , 2f , 2h , 2i , 2k , 2l

* To whom correspondence should be addressed. Tel: 86-21-50806876; Fax: 86-21-50807088.

Experimental Section

¹H-NMR spectra were recorded on a Varian 300-MHz or 400-MHz spectrometer. ¹³C-NMR spectra were recorded on a Varian Mercury VX 400-MHz spectrometer. Elemental analyses were obtained from Vario EL spectrometer. Melting points (uncorrected) were determined on a Buchi-510 capillary apparatus. IR spectra were recorded on Bio-Rad FTS-185 spectrometer. HRMS (EI) spectra were obtained on a Finnigan MAT 95 mass spectrometer. Substituted aryl acetophenones **1a-f** were purchased from commercial sources, **1g-l** were synthesized according to the literature procedure. The solvents (toluene, THF and DME) were dried over sodium wire and distilled prior to use. Flash column chromatography was performed on silica gel H (10-40 μm) with petroleum ether-ethyl acetate system as eluent.

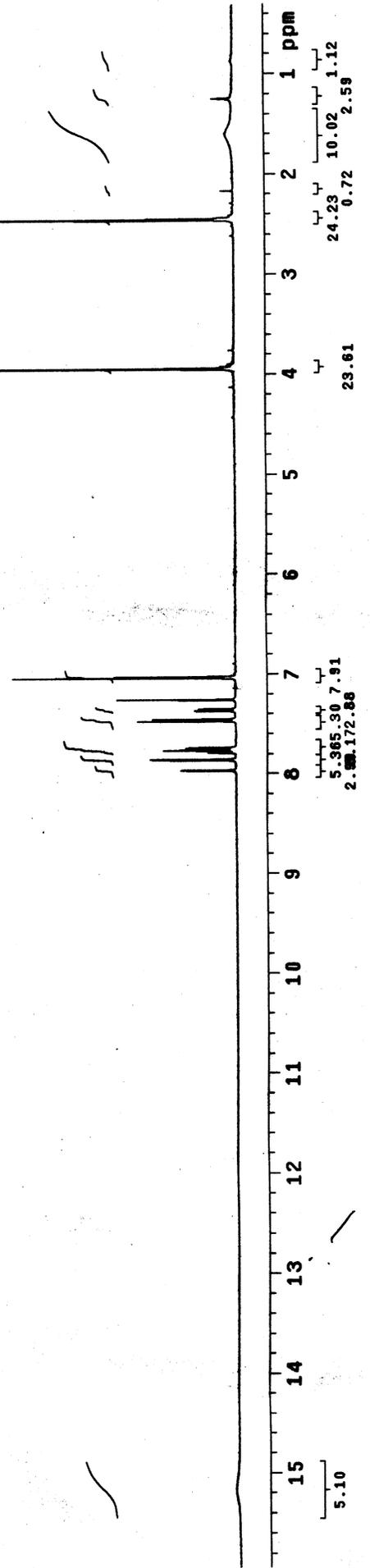




mp. 99-101°C.

- 3189.39
- 3187.74
- 3146.81
- 3144.89
- 3107.53
- 3099.29
- 3087.09
- 2990.79
- 2982.55
- 2949.86
- 2941.89
- 2904.81
- 2810.88

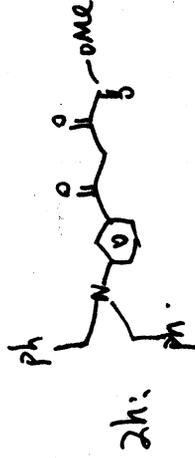
1578.41
981.13



15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm

5.10
2.98
7.91
5.36
29.61
10.02
1.12
2.59

4419.39
4398.56
4391.23
4383.60
4355.73
4352.21
4348.99
4341.07
4332.27
4156.26
4152.45

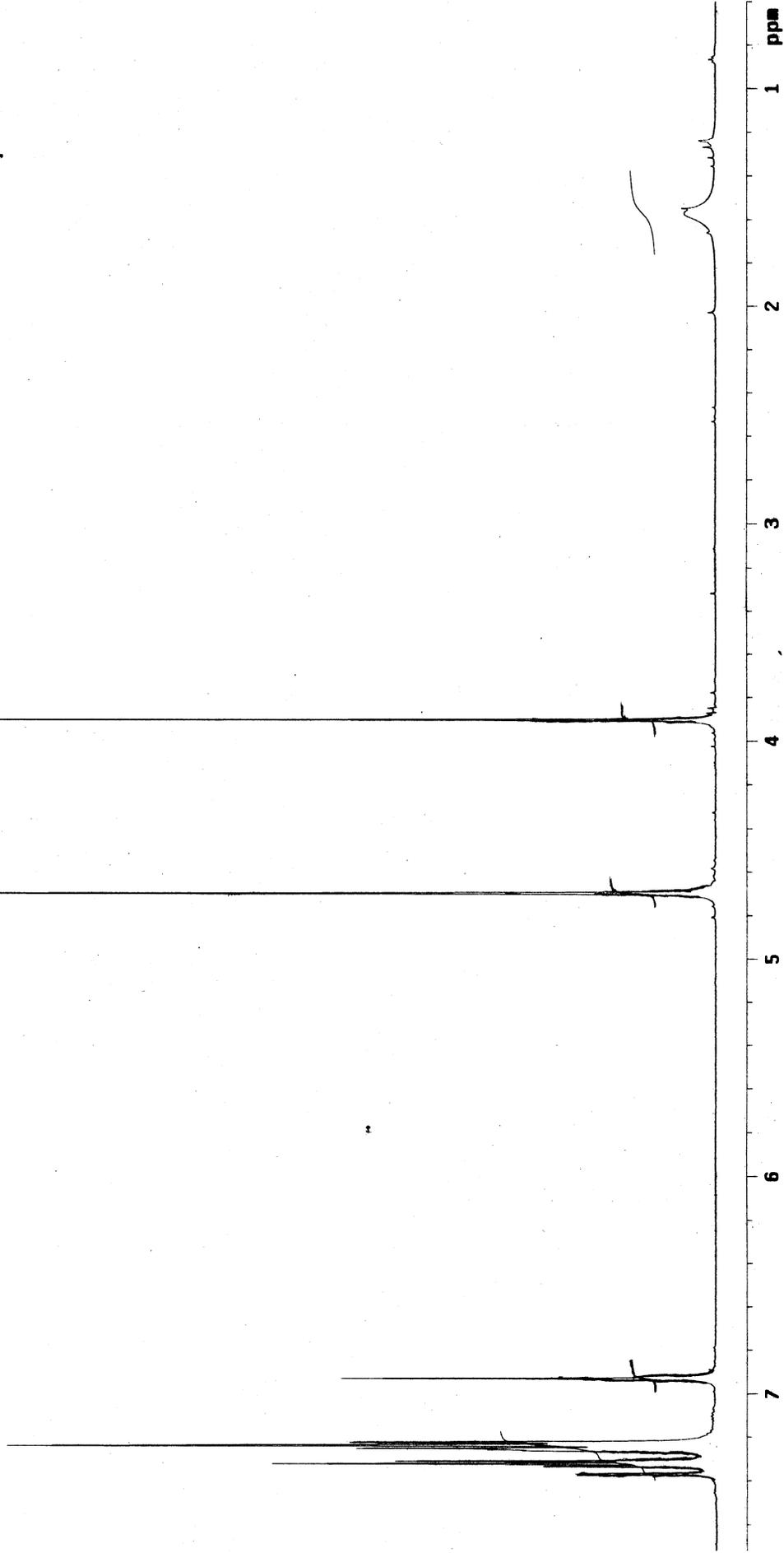


mp. 101~102°C

929.21

2340.48

2616.28



1 ppm

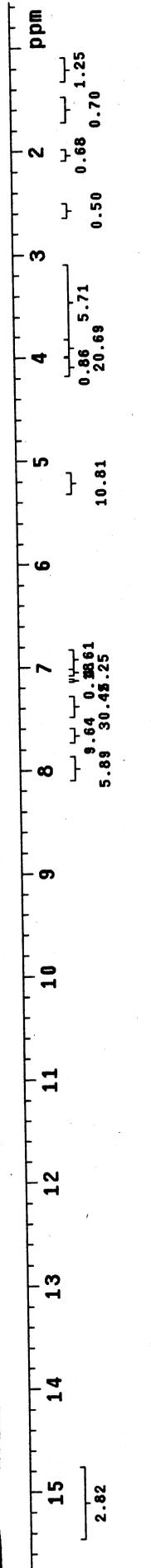
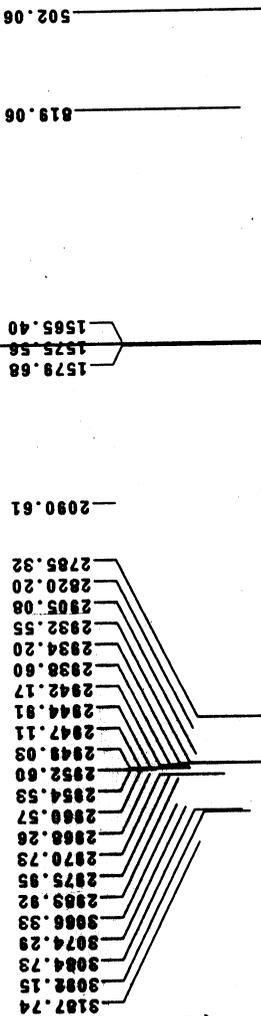
8.63

12.02

16.04

8.22

55.09

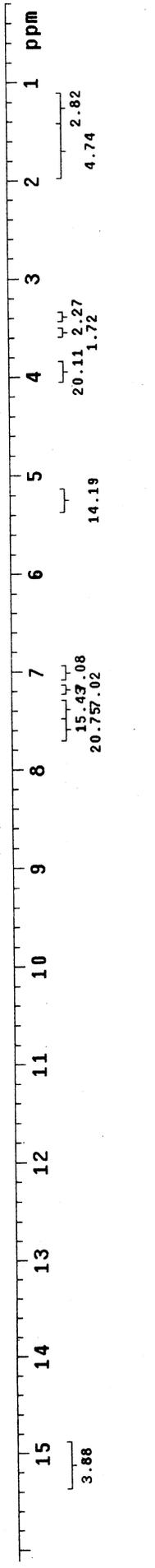


Pulse Sequence: s2pul



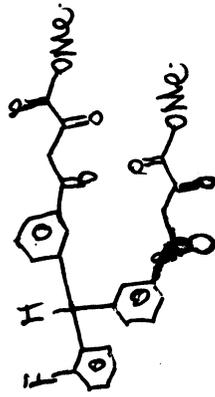
- 3030.49
- 3023.07
- 3020.92
- 3016.75
- 3014.69
- 3011.26
- 3007.55
- 2974.73
- 2970.74
- 2967.31
- 2964.97
- 2963.33
- 2961.82
- 2955.50
- 2947.39
- 2904.95
- 2882.16
- 2881.06
- 2880.23
- 2879.00
- 2873.37
- 2871.44
- 2810.46
- 2103.00

- 1575.46
- 1417.78
- 1357.76
- 502.79



JXH-0032 CDCl3 1H 02-4-15

Pulse Sequence: s2pu1



3160.69
3159.31
3154.10
3152.73
314.30
312.66
2994.64
2986.69
2979.00
2938.66
2930.70
2919.17
2911.49
2909.29
2905.72
2904.08
2848.09
2846.72
2844.52
2843.42
2839.31
2838.29
2834.37
2833.00
2826.13
2825.04
2805.28
2765.48
2763.83
2757.52
2756.15
2750.11
2748.37
2988.64
2117.51
1574.11
1568.99

27.05
-3.14



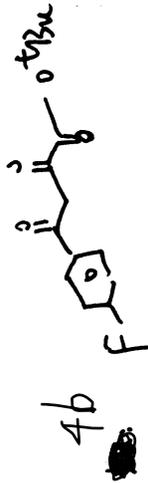
4.48

18.34 21.74 22.63

5.03

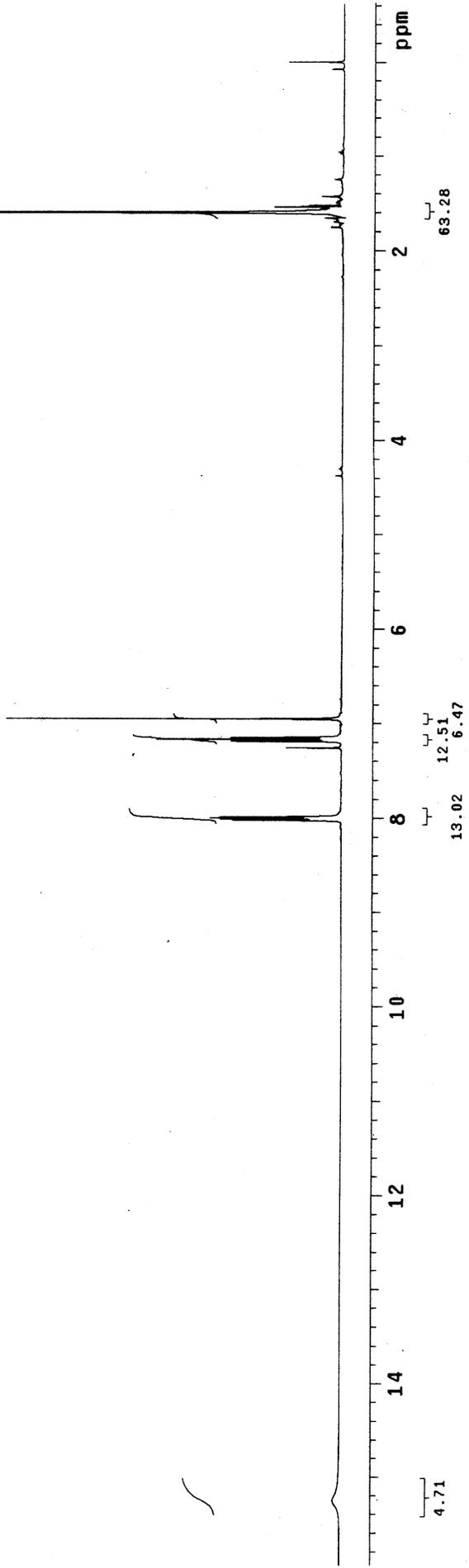
1.98

25.81

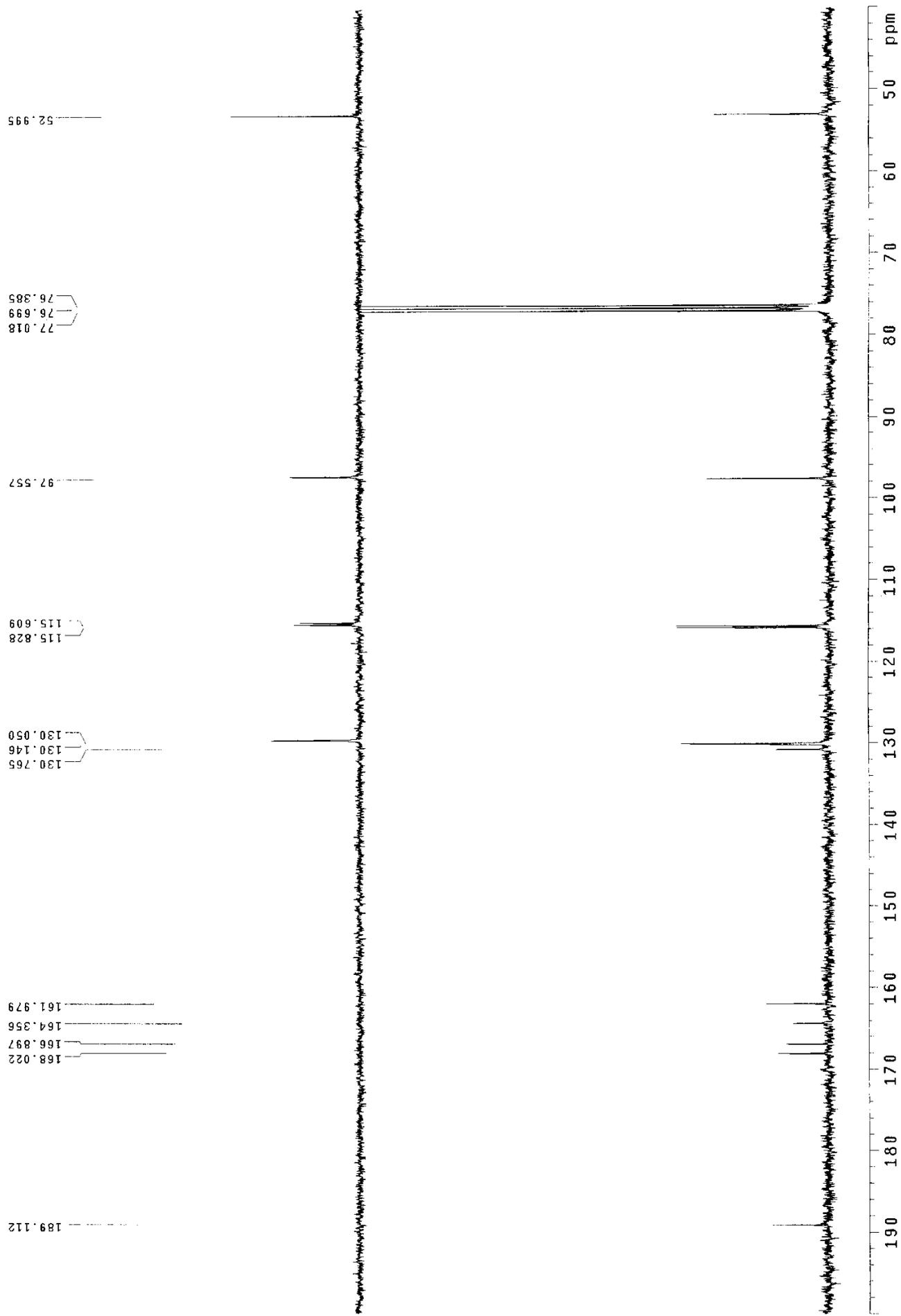


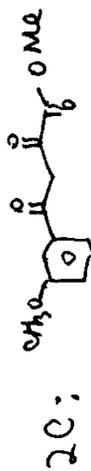
- 3209.14
- 3207.13
- 3208.77
- 3201.92
- 3200.25
- 3197.06
- 3194.87
- 2903.79
- 2876.94
- 2874.92
- 2868.21
- 2861.66
- 2859.65
- 2781.42

- 697.27
- 695.11
- 609.90
- 1.97



2a:





77.032
76.713
76.399
55.213
52.955

97.958

111.775

119.813
120.022

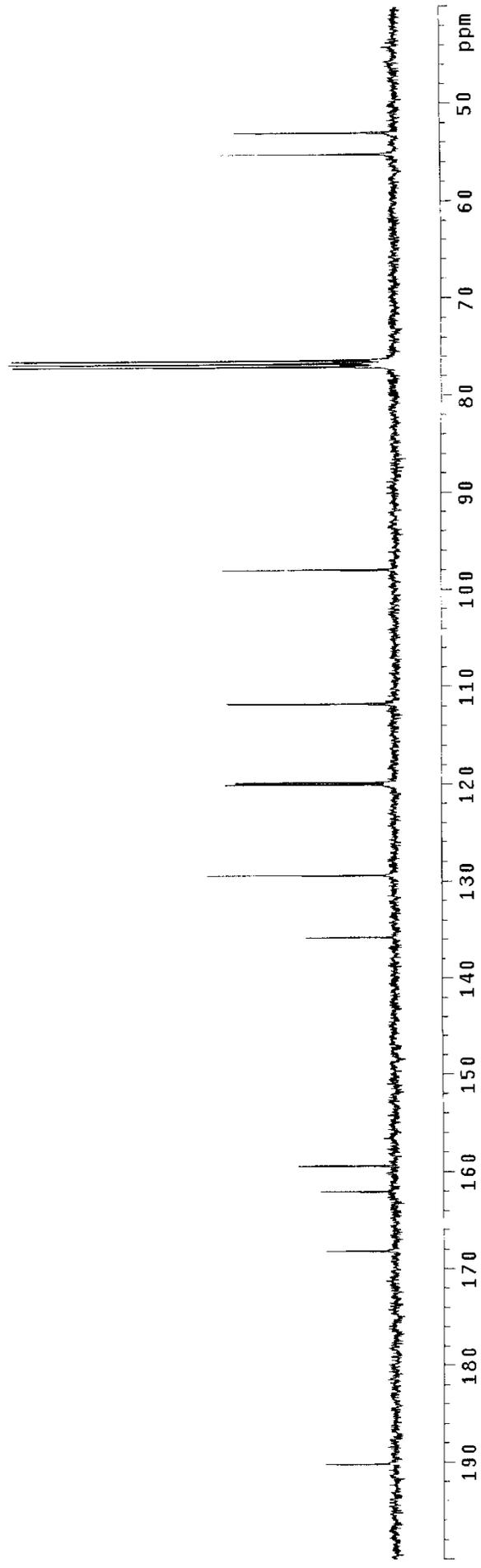
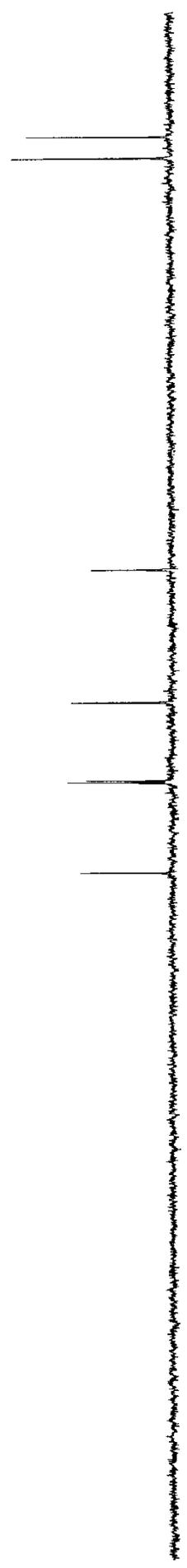
129.390

135.766

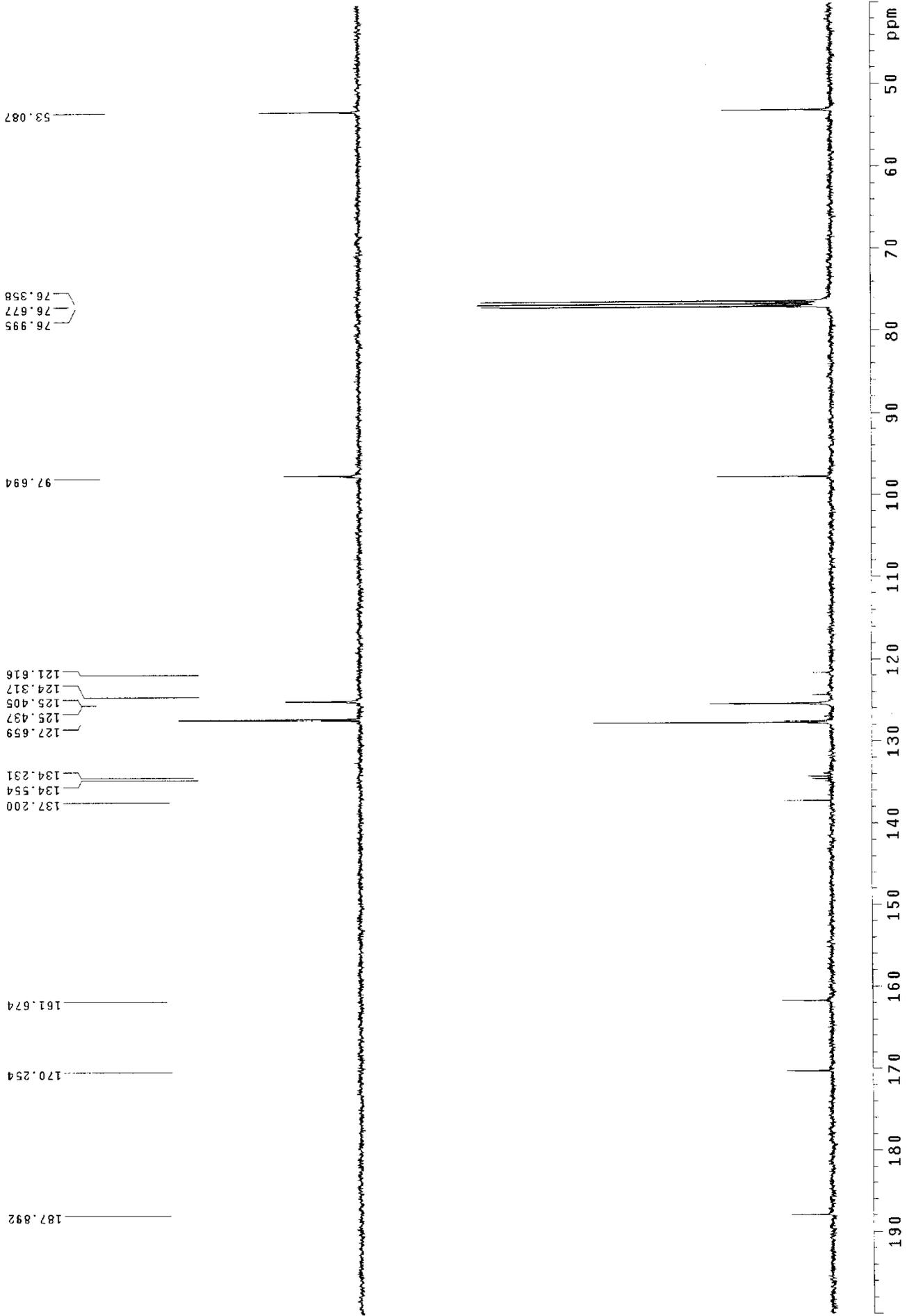
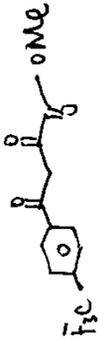
159.420
162.052

168.168

190.219

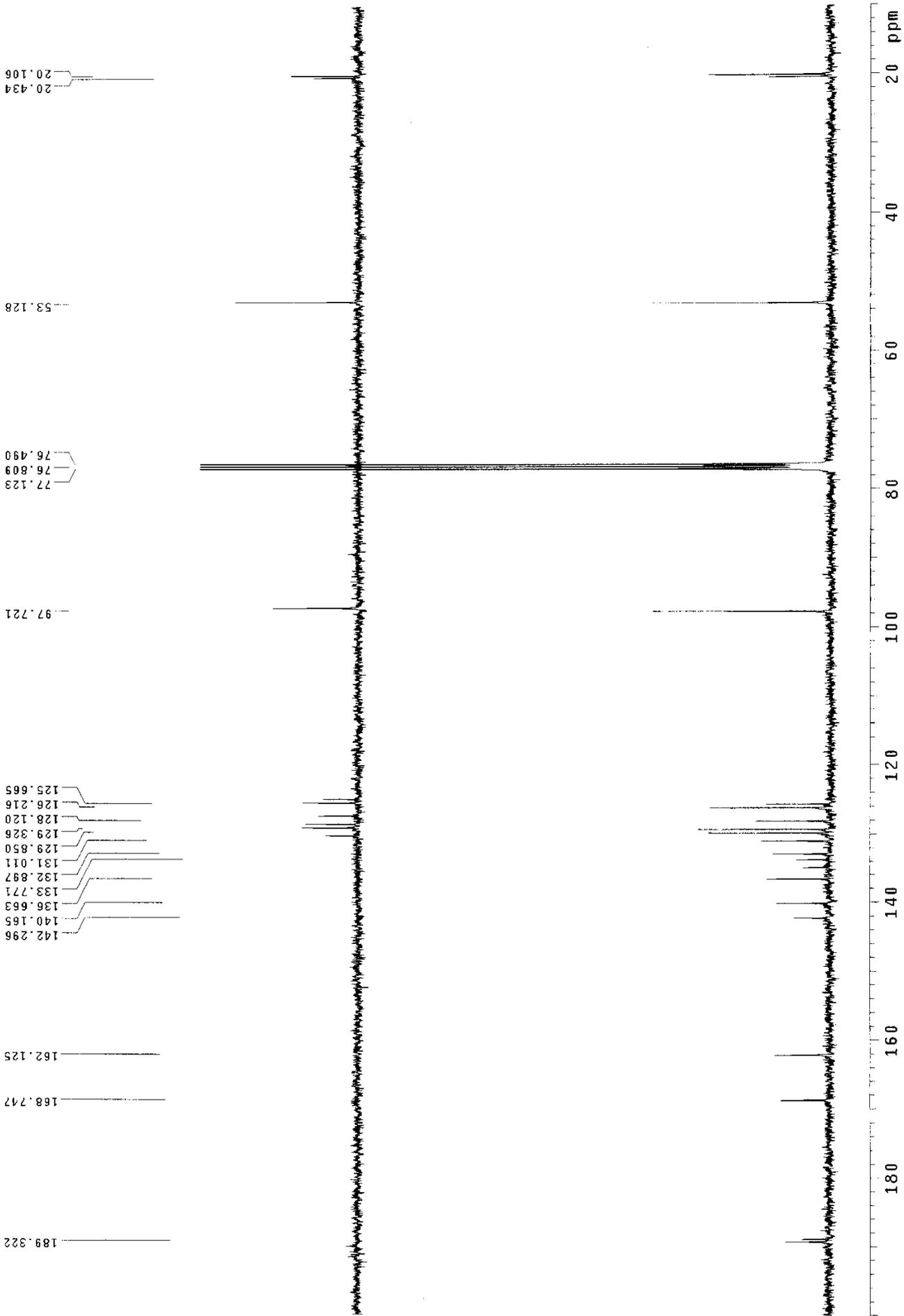


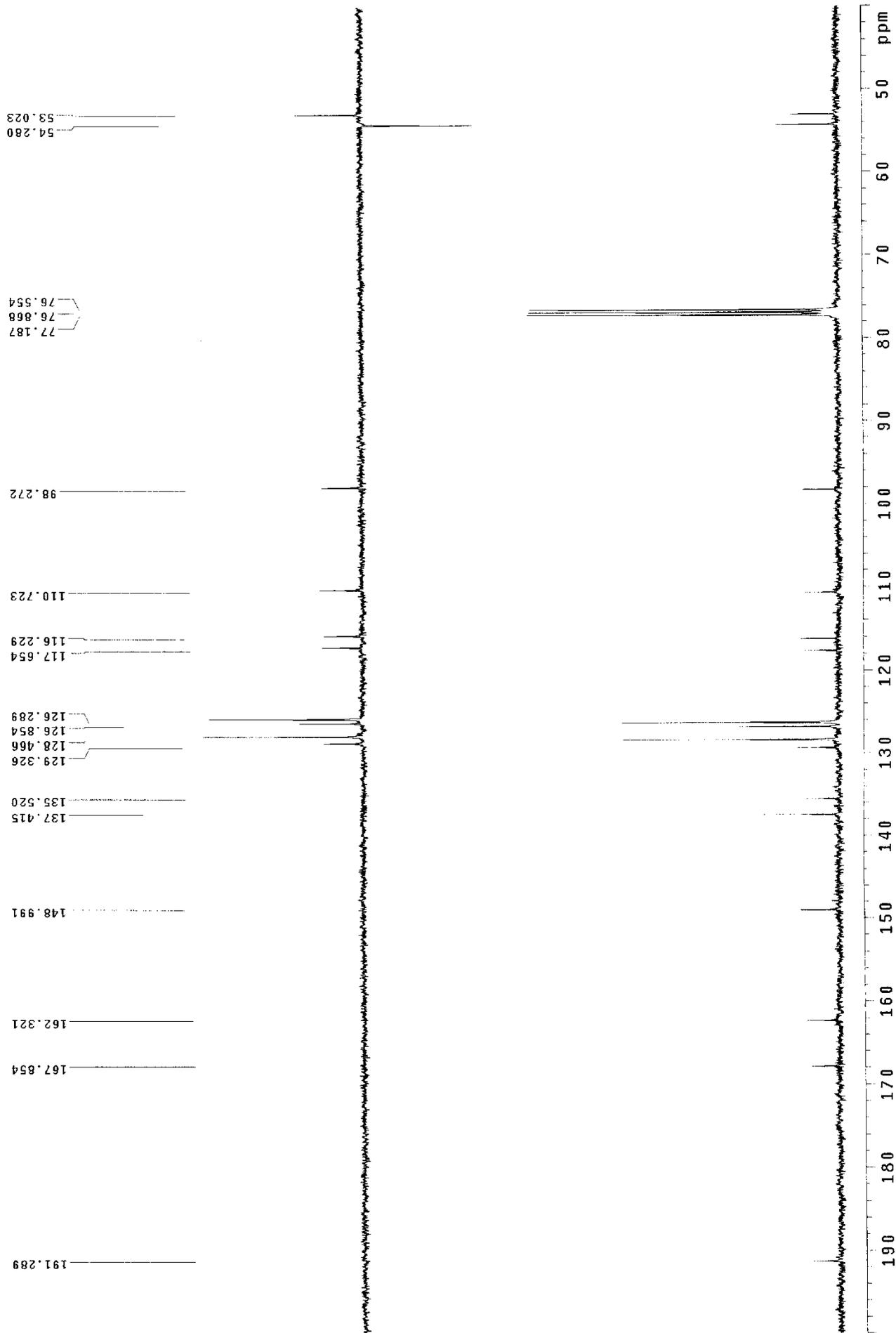
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 ppm





2-f:

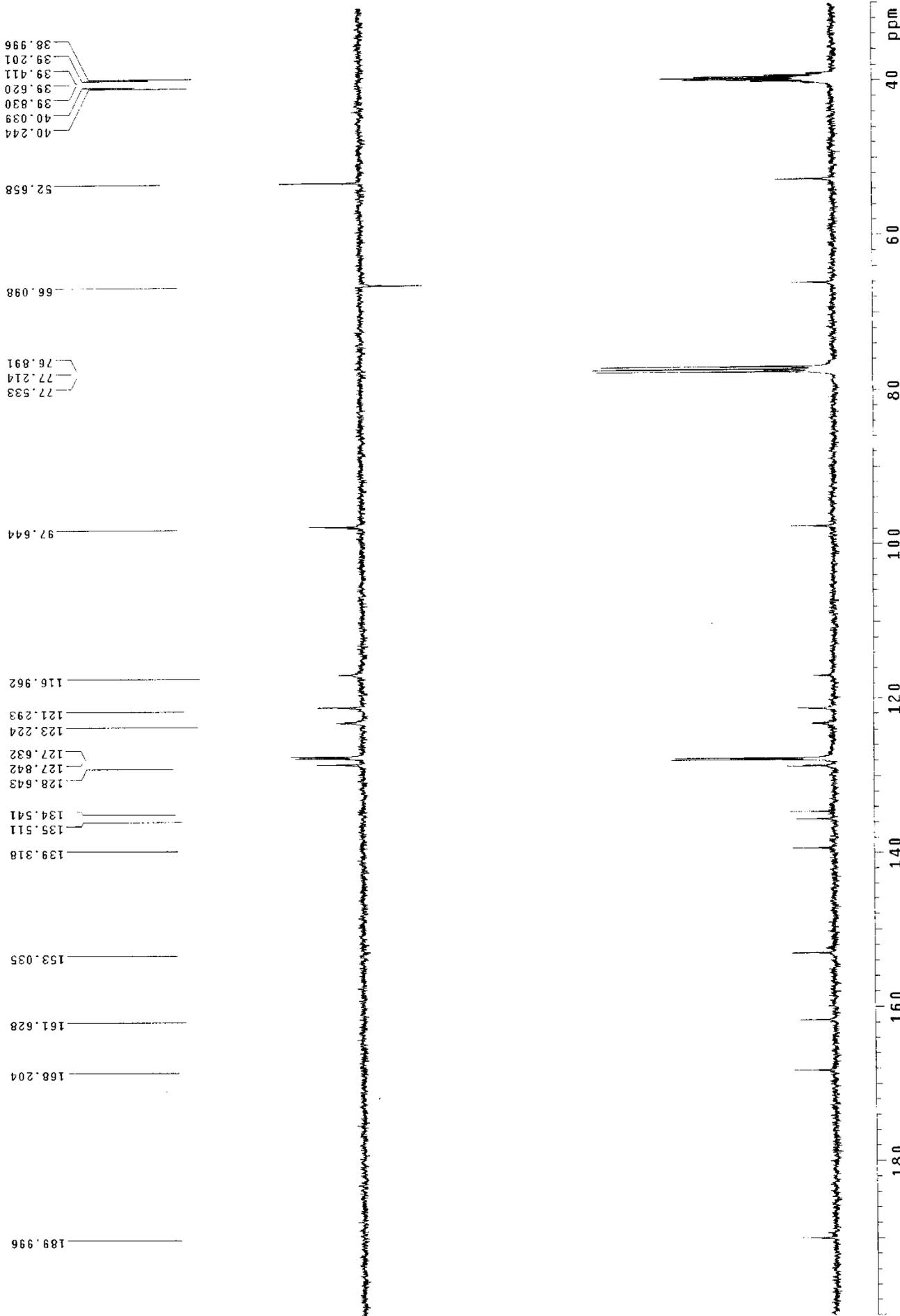


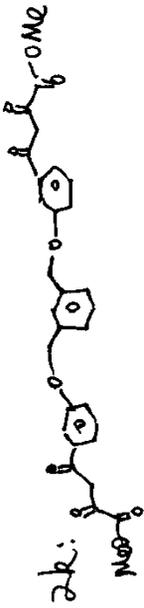


+ DMSO (d6)



2.4:





40.124
39.919
39.709
39.500
39.291
39.081
38.876

58.071

67.666

98.360

113.452

120.816

128.184

128.981

130.311

135.024

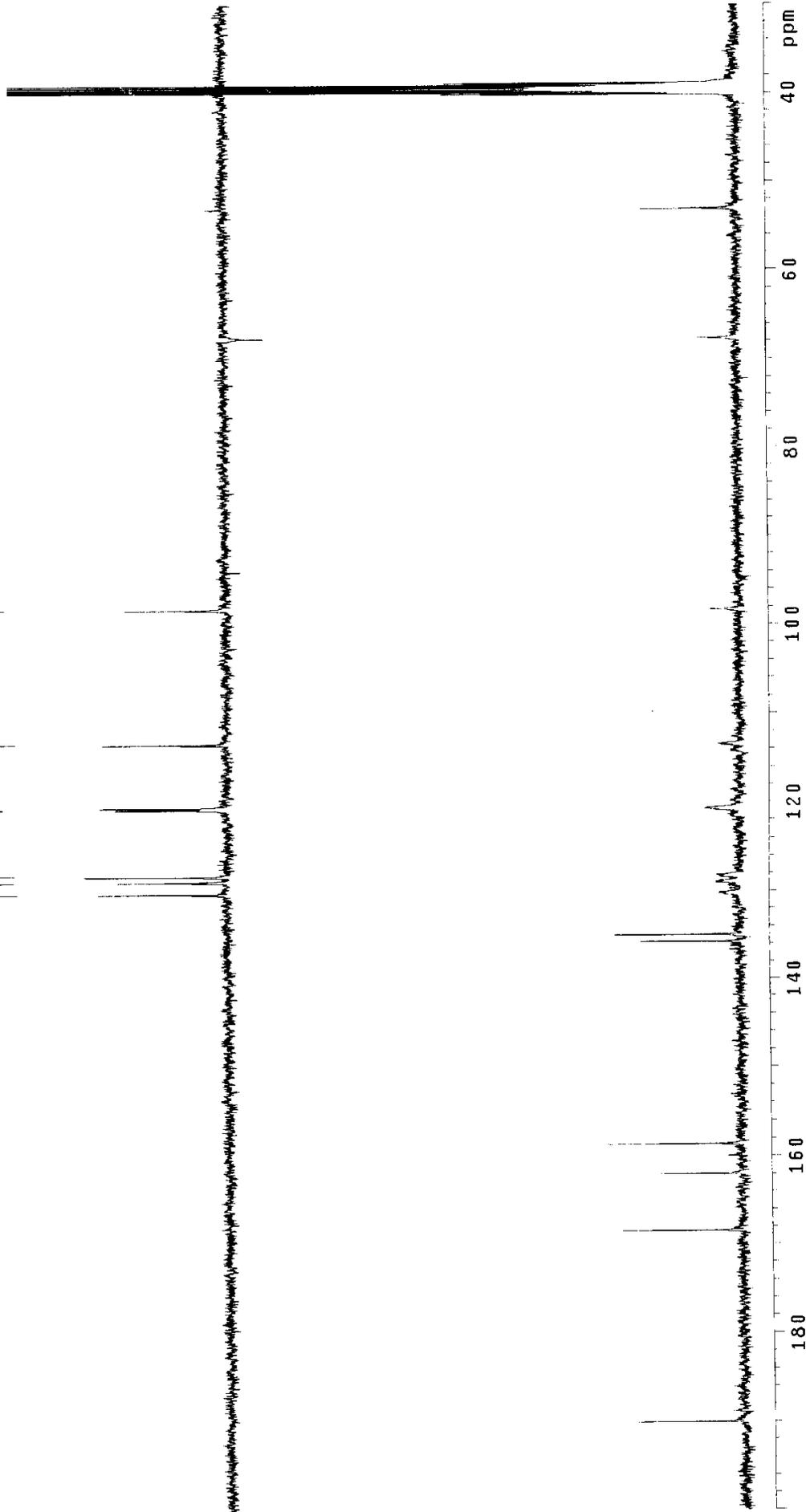
135.725

158.677

162.015

168.473

190.086



2d:

