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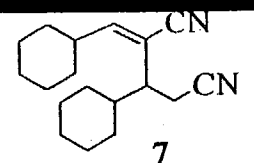


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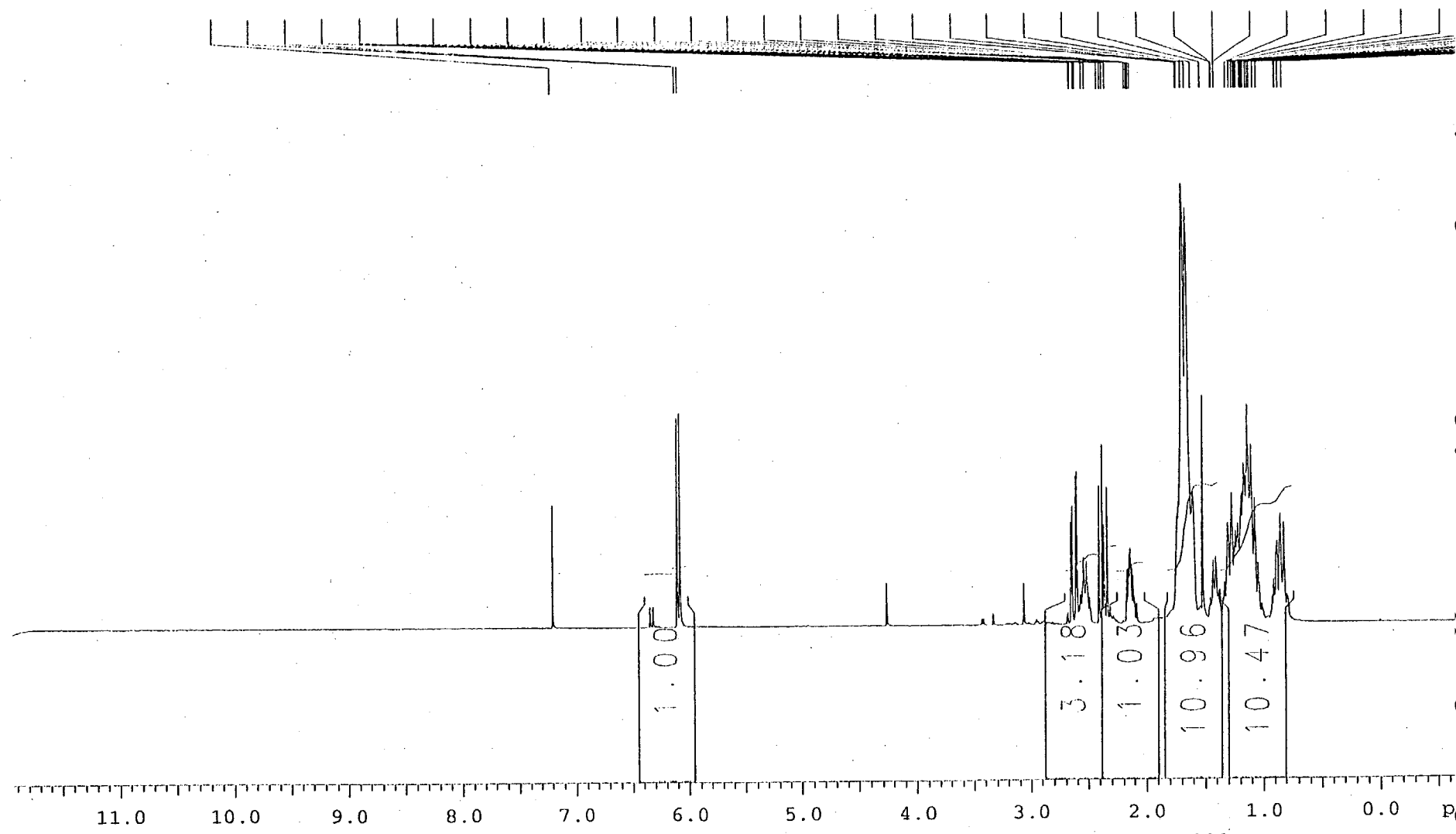
**<sup>1</sup>H NMR and <sup>13</sup>C NMR Data**

7. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 0.80-1.30 overlapping region (10H), 1.52-1.80 overlapping region (11H), 2.1-2.2 (m, 1H), 2.34-2.65 (m, 3H), 6.09 (d, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>): 20.23, 25.27, 25.31, 25.98, 26.09, 30.58, 30.77, 32.20, 32.25, 38.28, 39.61, 41.08, 47.59, 112.28, 116.01, 118.03, 156.77, 157.16 ppm. Anal. Calcd for C<sub>18</sub>H<sub>26</sub>N<sub>2</sub>: C, 79.95; H, 9.69; N, 10.36. Found C, 80.15; H, 9.91; N, 10.08. HR MS (EI) *m/e* (M<sup>+</sup>) calcd for C<sub>18</sub>H<sub>26</sub>N<sub>2</sub> 270.20960, obsd 270.20896.
8. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 0.88-0.97 overlapping region (6H), 1.03-1.12 overlapping region (6H), 1.65-1.83 (m, 1H), 2.08-2.16 (m, 1H), 2.36-2.45 (m, 1H) 2.57-2.65 (m, 1H) 2.80-2.88 (m, 1H), 6.09 (d, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>): 20.29, 20.43, 20.66, 22.22, 22.24, 30.72, 31.73, 48.59, 112.18, 115.83, 117.91, 158.58 ppm. Anal. Calcd for C<sub>12</sub>H<sub>18</sub>N<sub>2</sub> C, 75.74; H, 9.53; N, 14.72. Found C, 76.65; H, 9.67; N, 13.68. HR MS (EI) *m/e* (M<sup>+</sup>) calcd for C<sub>12</sub>H<sub>18</sub>N<sub>2</sub> 190.14700, obsd 190.14685.
9. Obtained as a mixture of diastereomers. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 0.88-0.98 overlapping region (9H), 1.10 (t, 3H), 1.32-1.67 overlapping region (4H), 2.3-2.55 (m, 2H), 2.64-2.71 (m, 2H), 6.12 (d, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>): 10.85, 10.88, 10.95, 11.93, 12.05, 12.09, 16.06, 16.10, 16.18, 16.29, 20.13, 20.21, 20.25, 20.33, 20.57, 20.63, 26.48, 26.50, 26.60, 26.66, 29.57, 29.58, 29.60, 36.63, 36.75, 36.80, 36.85, 38.77, 38.83, 46.48, 46.63, 47.02, 47.21, 113.12, 113.62, 116.08, 116.10, 116.22, 116.26, 117.93, 117.97, 118.06, 118.10, 157.33, 157.42, 157.62, 157.71 ppm. HR MS (EI) *m/e* (M<sup>+</sup>) calcd for C<sub>14</sub>H<sub>22</sub>N<sub>2</sub> 218.17830, obsd 218.17812.
10. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 0.84-0.98 overlapping region (13H), 1.27-1.40 overlapping region (4H), 1.44-1.61 (m, 4H), 2.44-2.52 (m, 3H), 2.60-2.72 (dd, 1H), 6.07 (d, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>): 10.31, 10.47, 11.92, 12.05, 20.49, 21.44, 22.19, 27.85, 27.88, 42.28, 44.57, 46.31, 115.13, 116.47, 118.21, 156.42 ppm. HR MS (EI) *m/e* (M<sup>+</sup>) calcd for C<sub>16</sub>H<sub>26</sub>N<sub>2</sub> 246.20960, obsd 246.20958.



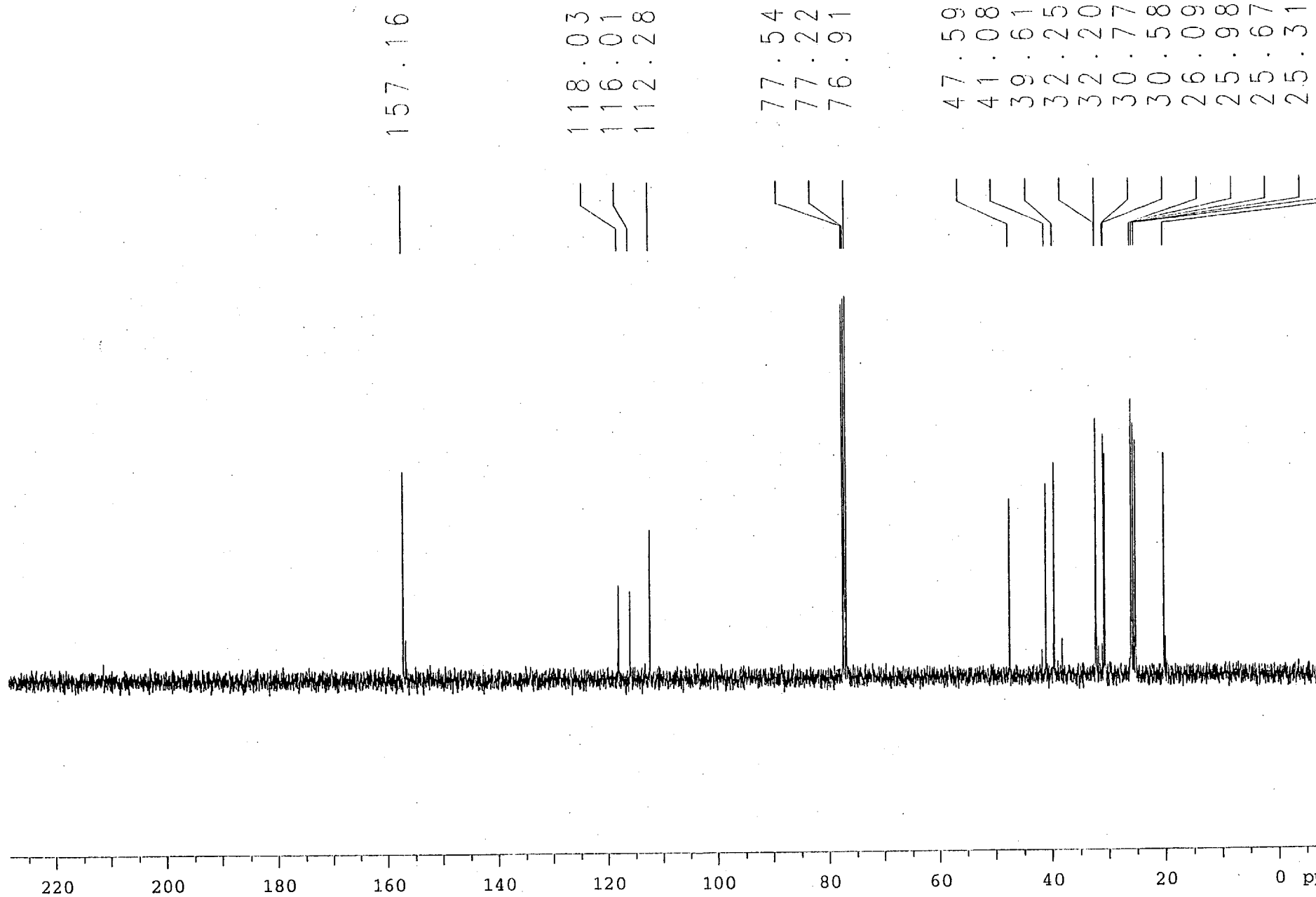
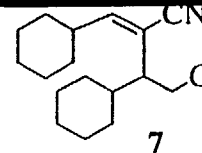
cyclohexanecarboxaldehyde + CH<sub>3</sub>CN / iPr<sub>3</sub>Phan

20	21	08	54	10	44	20	38	36	34	17	16	15	14	14	12	73	72	69	66	61	52	43	43	41	40	30	30	27	25	23	
7	6	6	2	2	2	2	2	2	2	2	2	2	2	2	2	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1



/drxl/data/baiwo/nmr/pk172a11/1/pdata/1 unknown Fri Jan 2 12:50:37 1998

cyclohexanecarboxaldehyde + CH<sub>3</sub>CN/iPr<sub>3</sub>Phan



/drxl/data/baiwo/nmr/pk172af/2/pdata/1 unknown Mon Jan 5 13:33:50 1998

## CHNS SAMPLE SUBMISSION FORM

12/12/96 swv

NAME: P. KISANGA Group: Verkade  
 CIA#: 485-21-3209 E-mail: baiwo  
 Lab #: 2306 Phone: 6986  
 Date: 2/6/98

Sample ID: bki729 Formula: C<sub>10</sub>H<sub>26</sub>N<sub>2</sub>

Crystallization solvent(s): oil

Theoretical weight percents (we can calculate these for you if you wish):

%C = 79.95 %H = 9.69 %N = 10.36 %S = \_\_\_\_\_

Number of runs requested 2. You are charged for each run. If you wish, save money and sample by just requesting one. For critical work you might want to request two or more runs.

Check here  if you absolutely require %S. This is not the normal configuration of the instrument, and you may have to wait a couple of weeks for these results. Check here  if you would like a CHN analysis prior to the CHNS analysis. You will be charged for each run.

Special combustion conditions (if you have information that would help us get better results on your sample, use this space, or attach the information to this form): \_\_\_\_\_

\*\*\*\*\*

RATES: CHN = \$11.00 per run CHNS = \$15.50 per run

\*\*\*\*\*

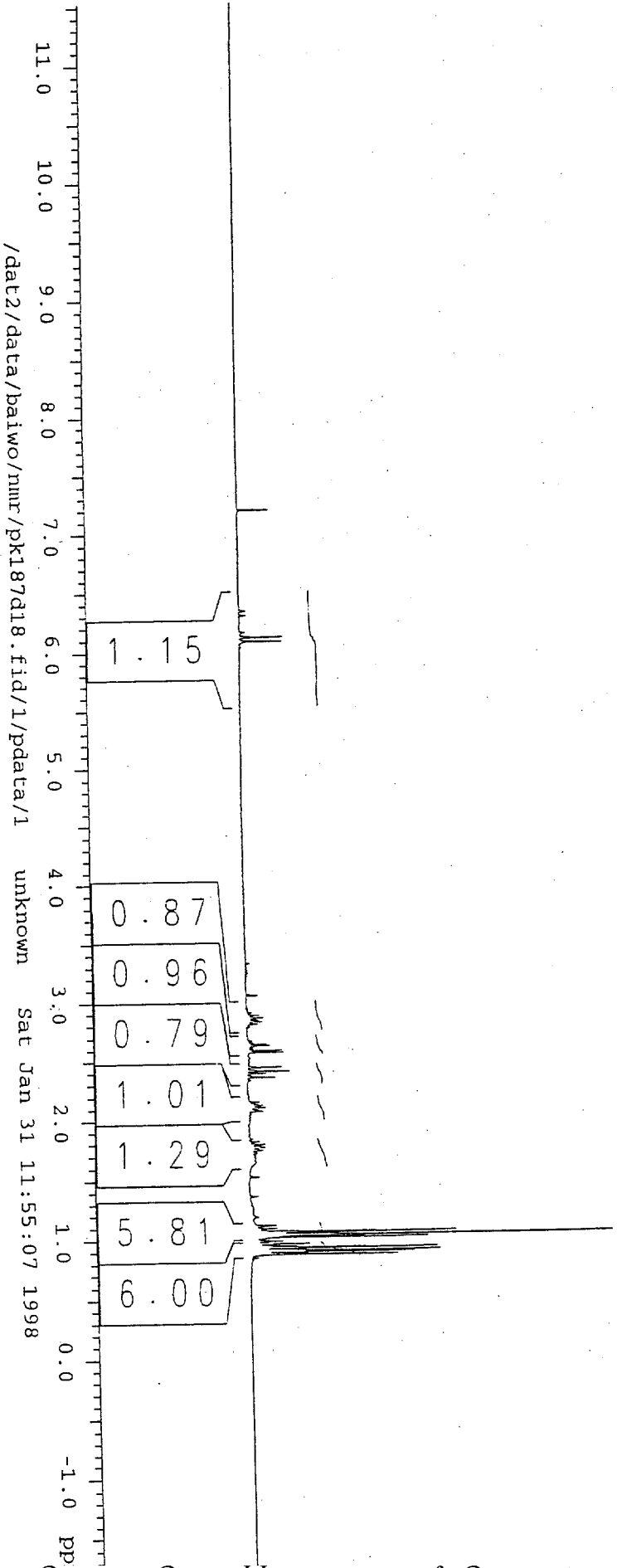
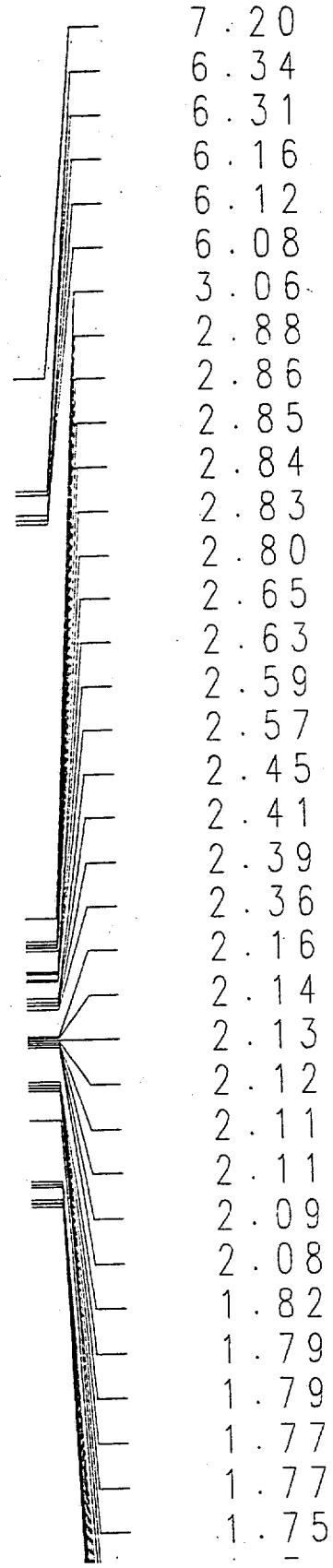
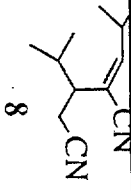
### RESULTS

	#1	#2	#3	AVG
%C	<u>80.40</u>	<u>79.89</u>		<u>80.15</u>
%H	<u>9.92</u>	<u>9.91</u>		<u>9.91</u>
%N	<u>10.11</u>	<u>10.06</u>		<u>10.08</u>
%S				
	<u>1.648mg</u>	<u>1.640mg</u>		<u>2/11/98</u>

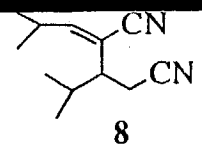


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269.20226	2.4	1.80	18	25	2
247.98996	2.0	No matches found			
241.17105	2.6	2.40	16	21	2
231.19383	16.1	No matches found			
230.19085	100.0	-0.10	16	24	1
229.18206	7.4	-4.31	16	23	1
190.14158	3.7	No matches found			
189.13882	40.4	-1.86	12	17	2
188.13124	27.8	-0.58	12	16	2
187.12372	25.0	1.06	12	15	2
162.12828	2.2	0.01	11	16	1
161.12131	2.2	5.37	11	15	1
161.11952	4.0	-5.79	11	15	1
159.09206	2.1	-1.01	10	11	2
148.11236	8.6	-1.79	10	14	1
146.09687	2.0	-0.68	10	12	1
135.10476	6.9	-0.27	9	13	1
134.09641	2.1	-4.18	9	12	1
118.06566	2.0	-0.11	8	8	1
107.05976	4.4	No matches found			
106.05347	42.9	3.48	6	6	2
104.04984	6.7	-1.75	7	6	1
91.05497	6.8	2.16	7	7	0
84.09055	2.7	No matches found			
83.08734	68.8	No matches found			
83.08481	6.8	No matches found			
82.07968	43.4	No matches found			
81.07218	8.8	No matches found			
79.05670	6.0	No matches found			
77.04086	8.1	No matches found			
69.99891	8.7	No matches found			
67.05545	27.1	No matches found			
65.04092	3.2	No matches found			
55.05662	59.9	No matches found			
54.04862	5.4	No matches found			
53.04002	5.2	No matches found			
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40.26443	18.7	No matches found			

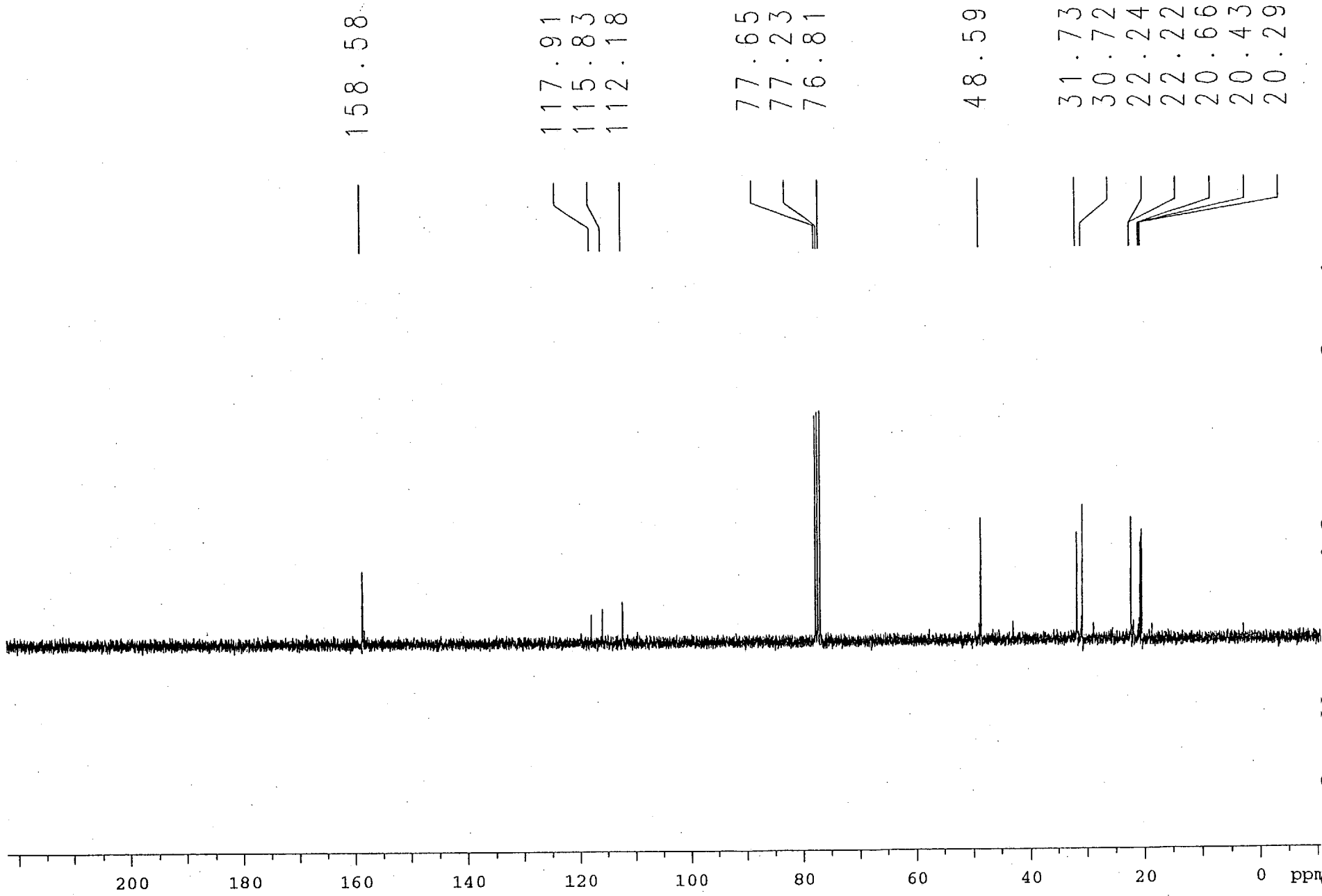
isobutyraldehyde + CH<sub>3</sub>CN / iPr<sub>3</sub>P<sub>h</sub>an







isobutyraldehyde + CH<sub>3</sub>CN / iPr<sub>3</sub>Phan



/dat2/data/baiwo/nmr/pk187d18.c13.fi/1/pdata/1 unknown Sat Jan 31 11:50:16 1998

### CHNS SAMPLE SUBMISSION FORM

12/12/96 swv



NAME: P-KISANGA Group: Verkade  
 CIA#: 485-21-3209 E-mail: baiwo  
 Lab #: 2306 GILMAN Phone: 4-6986  
 Date: 2/6/98

Sample ID: DK187d Formula: C<sub>12</sub>H<sub>18</sub>N<sub>2</sub>

Crystallization solvent(s): oil

Theoretical weight percents (we can calculate these for you if you wish):

%C = 75.74 %H = 9.53 %N = 14.72 %S = \_\_\_\_\_

Number of runs requested 2. You are charged for each run. If you wish, save money and sample by just requesting one. For critical work you might want to request two or more runs.

Check here \_\_\_ if you absolutely require %S. This is not the normal configuration of the instrument, and you may have to wait a couple of weeks for these results. Check here \_\_\_ if you would like a CHN analysis prior to the CHNS analysis. You will be charged for each run.

Special combustion conditions (if you have information that would help us get better results on your sample, use this space, or attach the information to this form): \_\_\_\_\_

\*\*\*\*\*

RATES: CHN = \$11.00 per run CHNS = \$15.50 per run

\*\*\*\*\*

#### RESULTS

	#1	#2	#3	AVG
%C	76.67	76.62		76.65
%H	9.67	9.67		9.67
%N	13.61	13.46		13.68
%S				

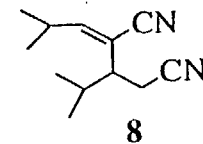
1.508mg

1.702mg

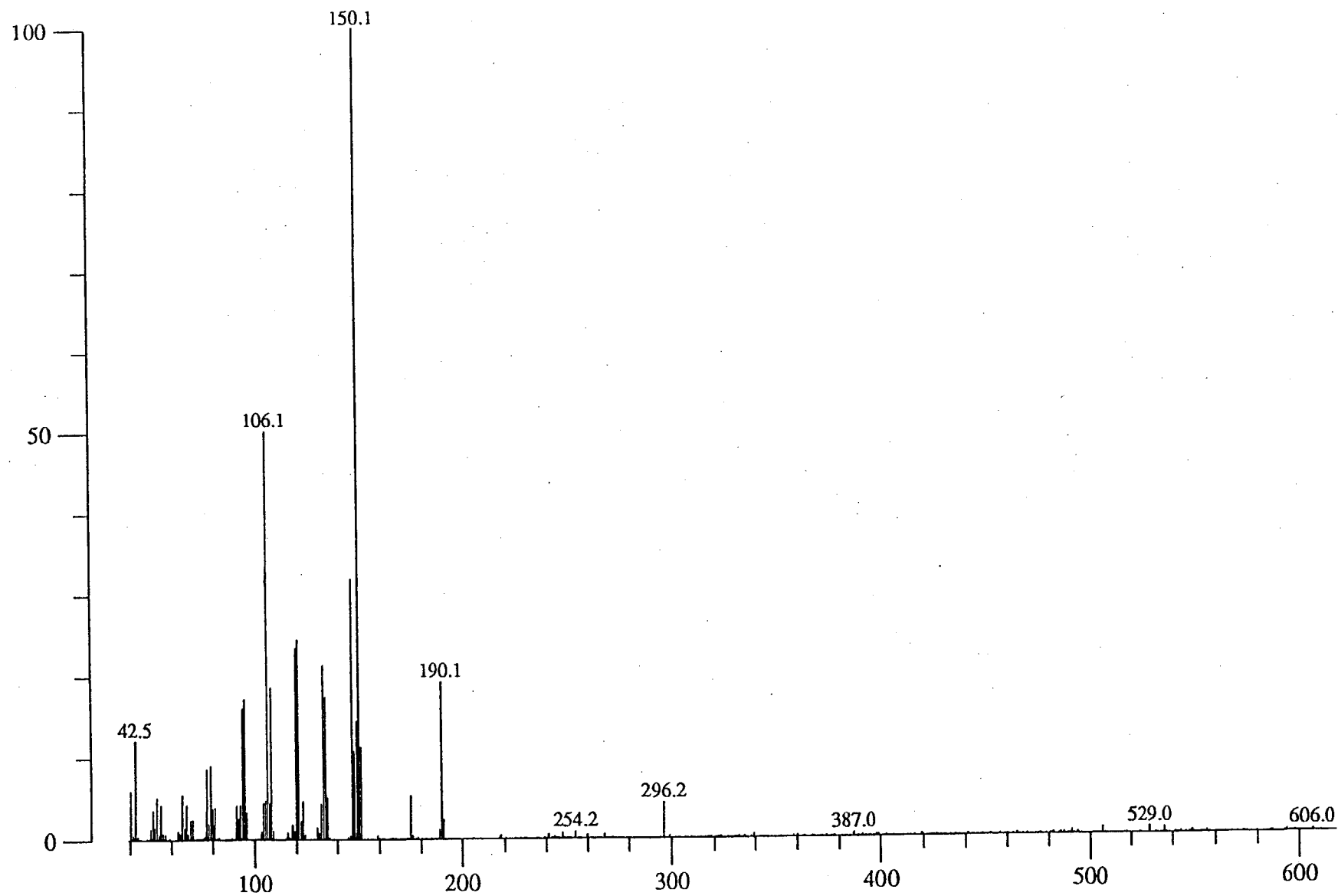
2/11/98

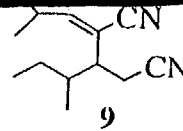
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190.14685	19.3	-0.76	12	18	2
175.12352	5.3	-0.05	11	15	2
151.13131	11.3	No matches found			
150.12792	100.0	-2.39	10	16	1
149.11925	14.4	-8.03	10	15	1
148.10194	10.8	No matches found			
148.09832	3.2	No matches found			
147.09195	32.0	-1.87	9	11	2
135.10262	5.2	No matches found			
134.09668	17.4	-2.16	9	12	1
134.07923	2.4	No matches found			
133.10146	2.2	-2.00	10	13	0
133.08011	2.3	No matches found			
133.07626	21.4	-2.35	8	9	2
132.06927	4.4	3.94	8	8	2
123.11728	4.7	-0.74	9	15	0
122.09375	2.4	No matches found			
121.08897	24.5	-1.48	8	11	1
120.08117	23.5	-1.31	8	10	1
108.08081	18.6	-4.74	7	10	1
107.07098	2.6	No matches found			
107.06388	4.5	No matches found			
107.05592	2.2	No matches found			
106.06537	18.5	-2.91	7	8	1
106.05756	50.2	No matches found			
106.05265	27.9	-4.23	6	6	2
105.07061	2.1	1.76	8	9	0
105.04495	4.8	-3.07	6	5	2
104.04972	4.6	-2.94	7	6	1
96.08042	3.5	-9.45	6	10	1
95.07348	17.2	-0.15	6	9	1
94.07752	2.0	-7.74	7	10	0
94.06564	16.1	-0.33	6	8	1
93.07017	4.3	-2.77	7	9	0
93.05742	2.8	-4.64	6	7	1
92.05043	2.6	4.46	6	6	1
91.05518	4.3	4.47	7	7	0
91.04772	2.4	No matches found			
81.07213	4.0	No matches found			
80.05222	3.9	No matches found			
79.05680	9.1	No matches found			
78.99293	3.9	No matches found			
78.04869	2.0	No matches found			
77.04103	8.7	No matches found			
76.98269	3.6	No matches found			
69.99891	2.6	No matches found			
69.07001	2.5	-6.08	5	9	0
67.05536	4.3	8.73	5	7	0
65.04816	2.2	No matches found			
65.04038	5.5	No matches found			
55.05665	4.3	No matches found			
53.04486	2.6	No matches found			
53.04022	5.2	No matches found			
51.02402	2.1	No matches found			
51.02315	3.7	-6.41	4	3	0



MS70840001 Scan 1 (Av 6-28 Acq) 100%=7800 mv 23 Feb 98 11:18  
HRP PK187d P.Kisanga 10K Resolution 70eV EI Solids Probe



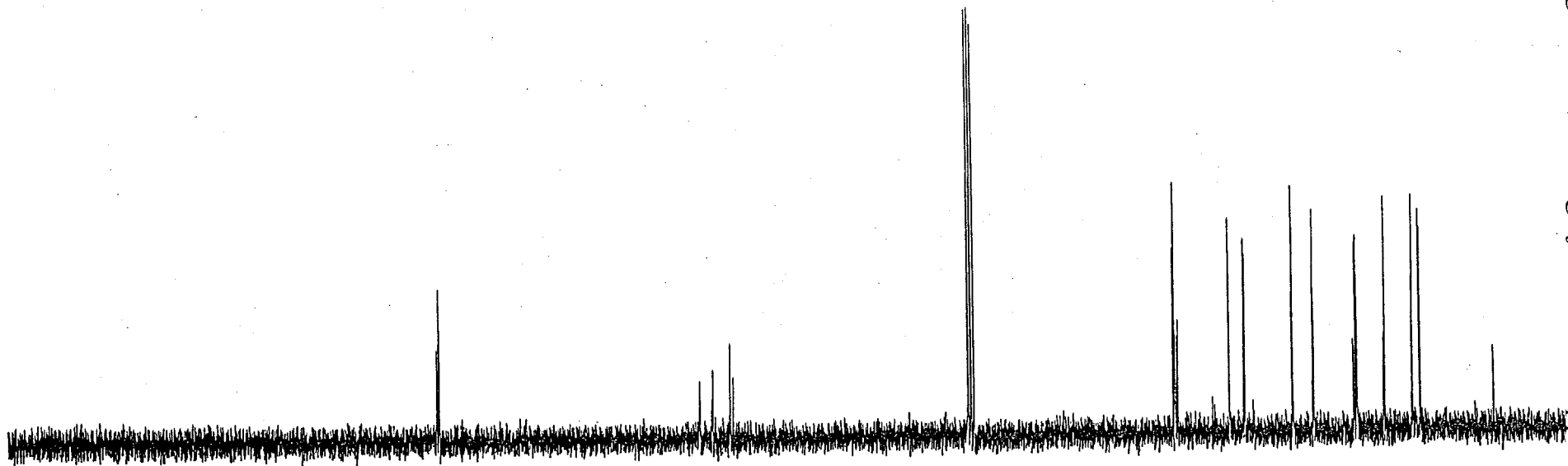
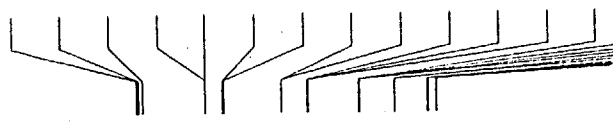
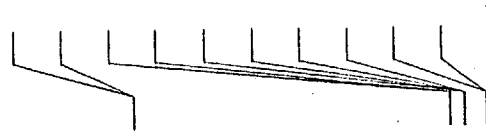


2-methylbutyraldehyde + CH<sub>3</sub>CN / iPr<sub>3</sub>Phan

157.42  
157.33  
118.10  
117.97  
116.23  
116.22  
116.10  
116.07  
113.61  
113.60  
113.12

77.65  
77.23  
76.80

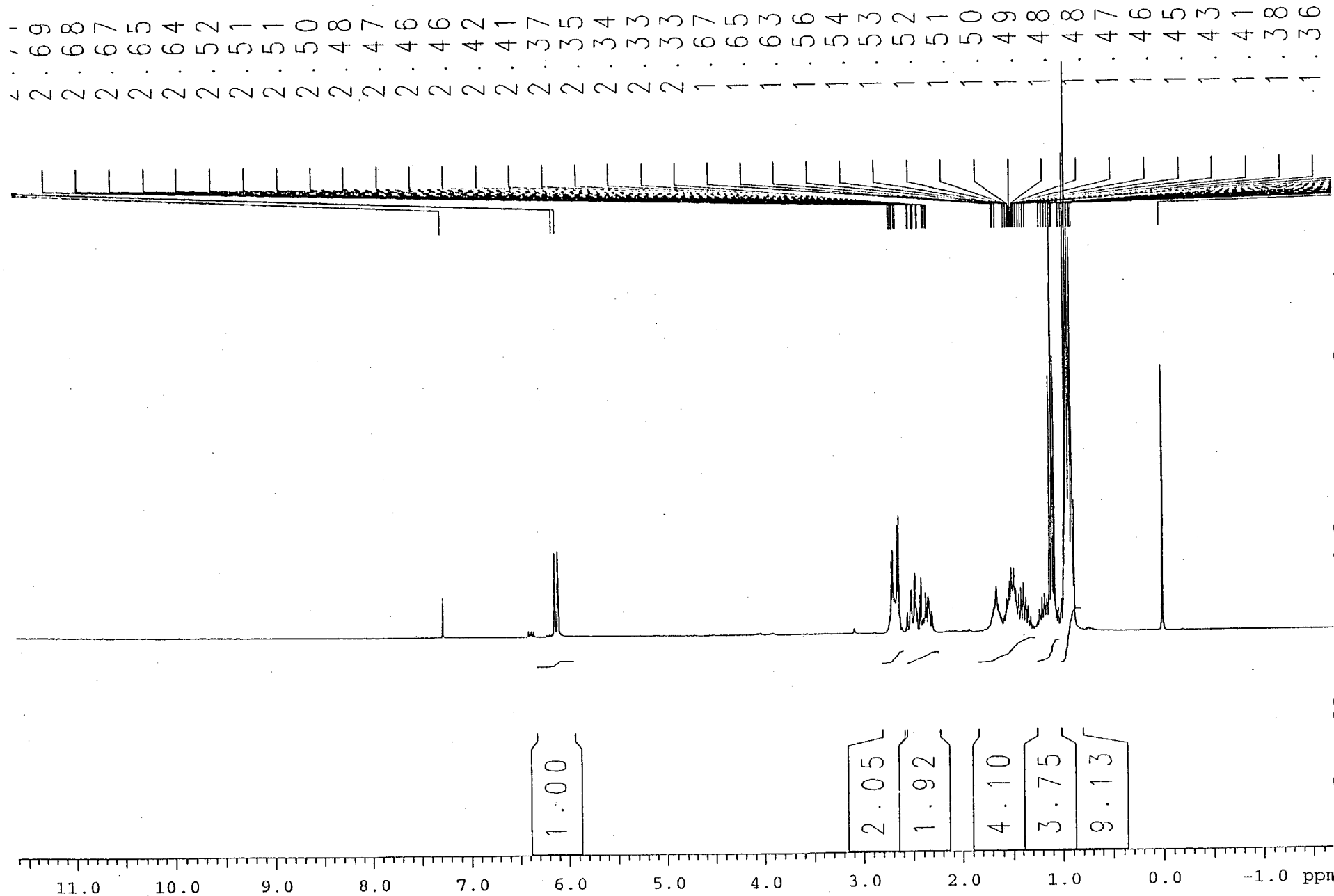
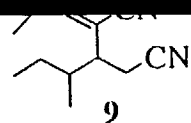
47.20  
47.01  
46.47  
38.82  
38.77  
36.80  
36.62  
29.60  
29.58  
26.65  
26.59  
26.49  
20.32



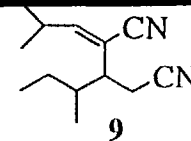
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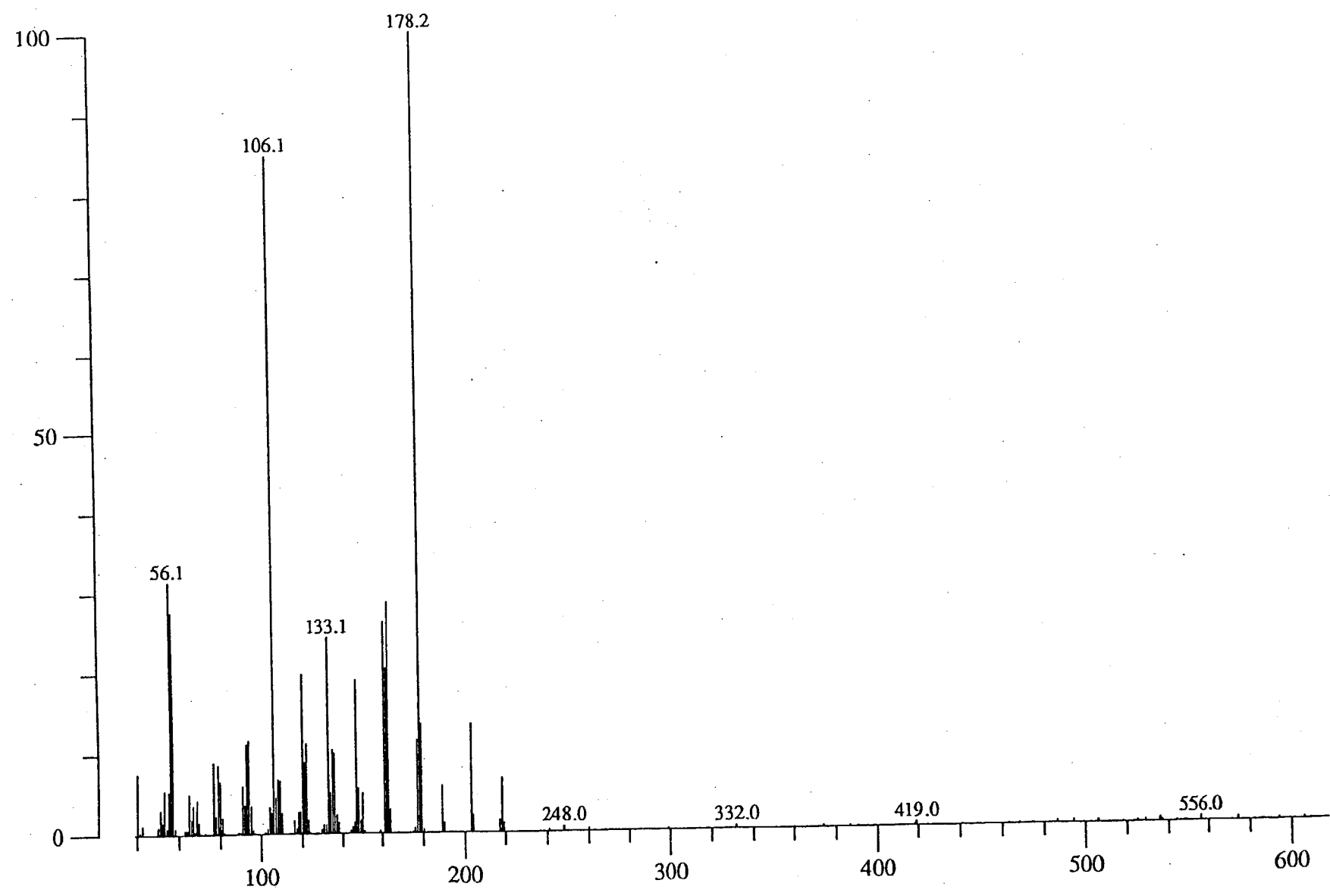
2-methylbutyraldehyde + CH<sub>3</sub>CN / iPr<sub>3</sub>Phan



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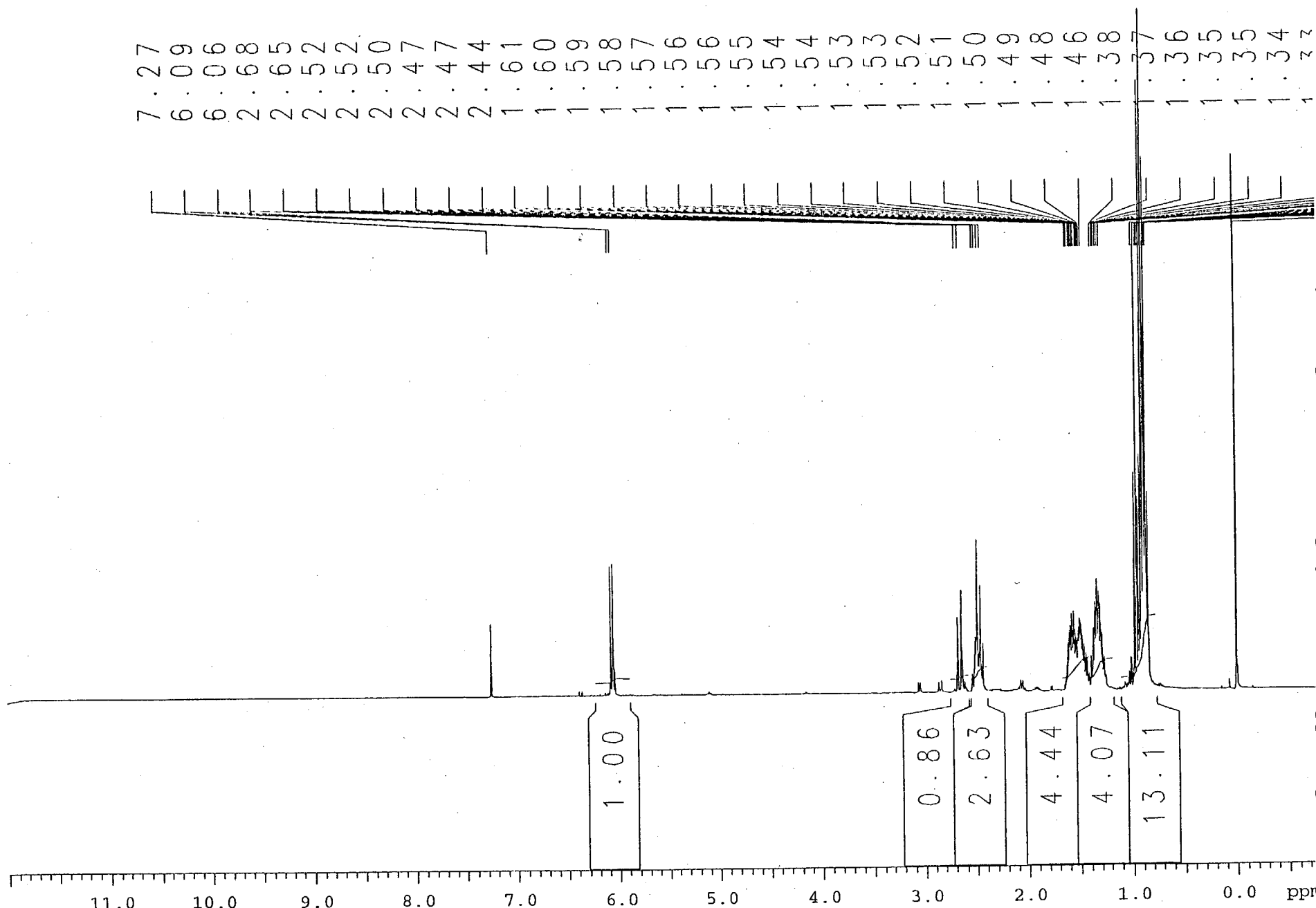
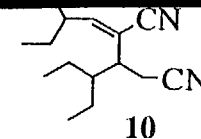
MS70850001 Scan 1 (Av 2-51 Acq) 100%=13814 mv 23 Feb 98 11:41  
HRP PK172c P.Kisanga 10K Resolution 70eV EI Solids Probe



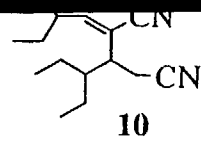
Mass	Int%	Dev ppm	C	H	N
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203.15478	13.5	-0.22	13	19	2
189.13906	5.9	-0.58	12	17	2
179.16262	13.6	No matches found			
178.15931	100.0	-1.46	12	20	1
177.14381	11.7	No matches found			
164.12736	3.1	No matches found			
163.12341	28.8	-0.67	10	15	2
162.11834	20.6	No matches found			
161.10750	26.3	-2.35	10	13	2
150.12788	5.1	-2.65	10	16	1
148.11210	5.7	-3.54	10	14	1
147.09223	19.1	0.02	9	11	2
137.11862	2.4	No matches found			
136.11209	10.1	-3.91	9	14	1
135.10429	10.5	-3.78	9	13	1
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119.06128	2.8	2.96	7	7	2
118.06553	2.7	-1.21	8	8	1
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91.04784	6.1	No matches found			
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79.98759	6.6	No matches found			
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78.98036	2.3	No matches found			
77.98659	2.3	No matches found			
76.98279	9.0	No matches found			
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69.05698	3.0	No matches found			
67.05955	3.7	No matches found			
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57.08453	6.5	No matches found			
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57.07317	2.8	No matches found			



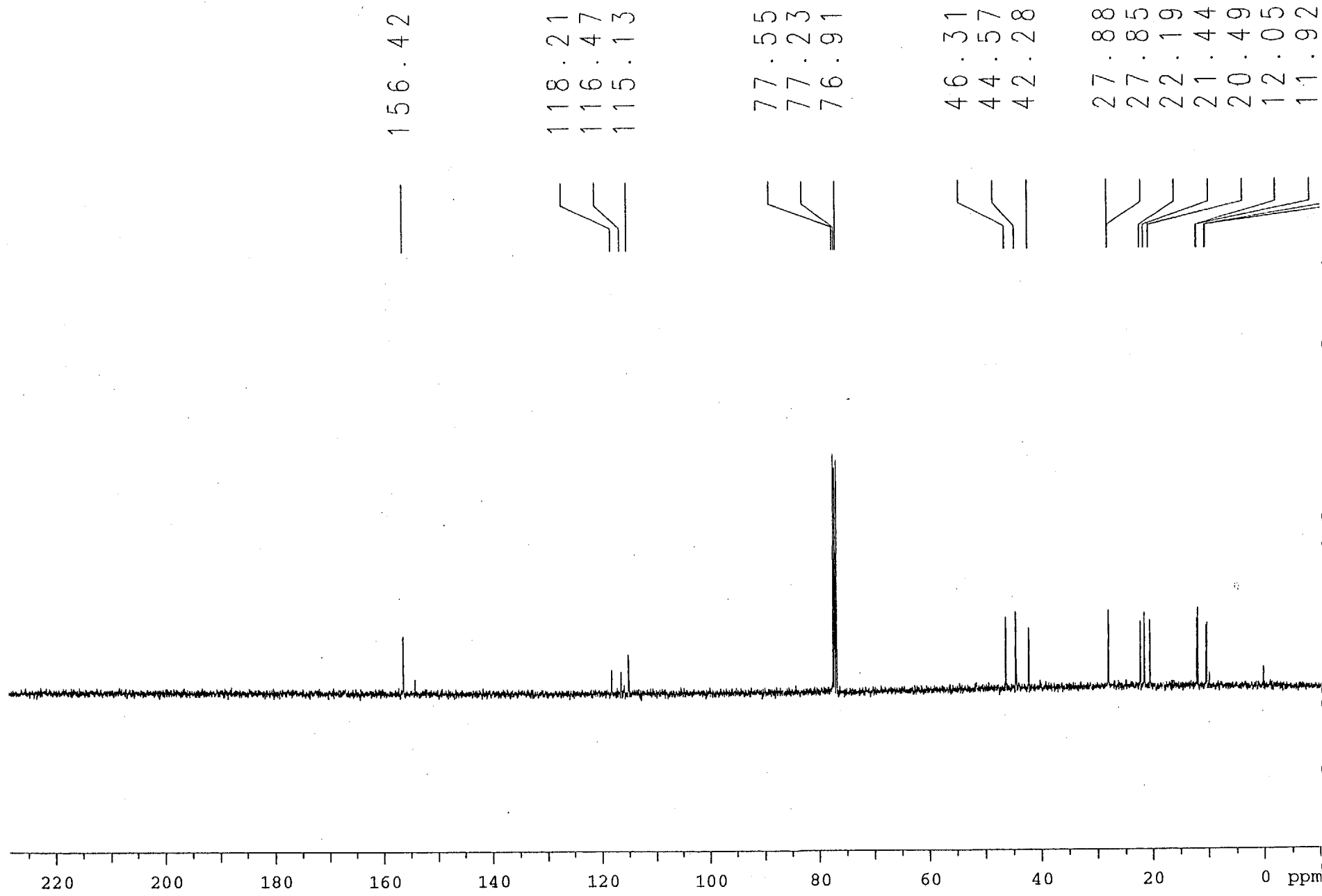
2-ethylbutyraldehyde + CH<sub>3</sub>CN /iPr<sub>3</sub>Phan



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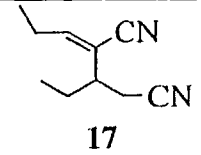
2-ethylbutyraldehyde + MeCN / iPr3Phan



/drx1/data/baiwo/nmr/pk198c4/2/pdata/1 unknown Sat Feb 21 14:20:02 1998

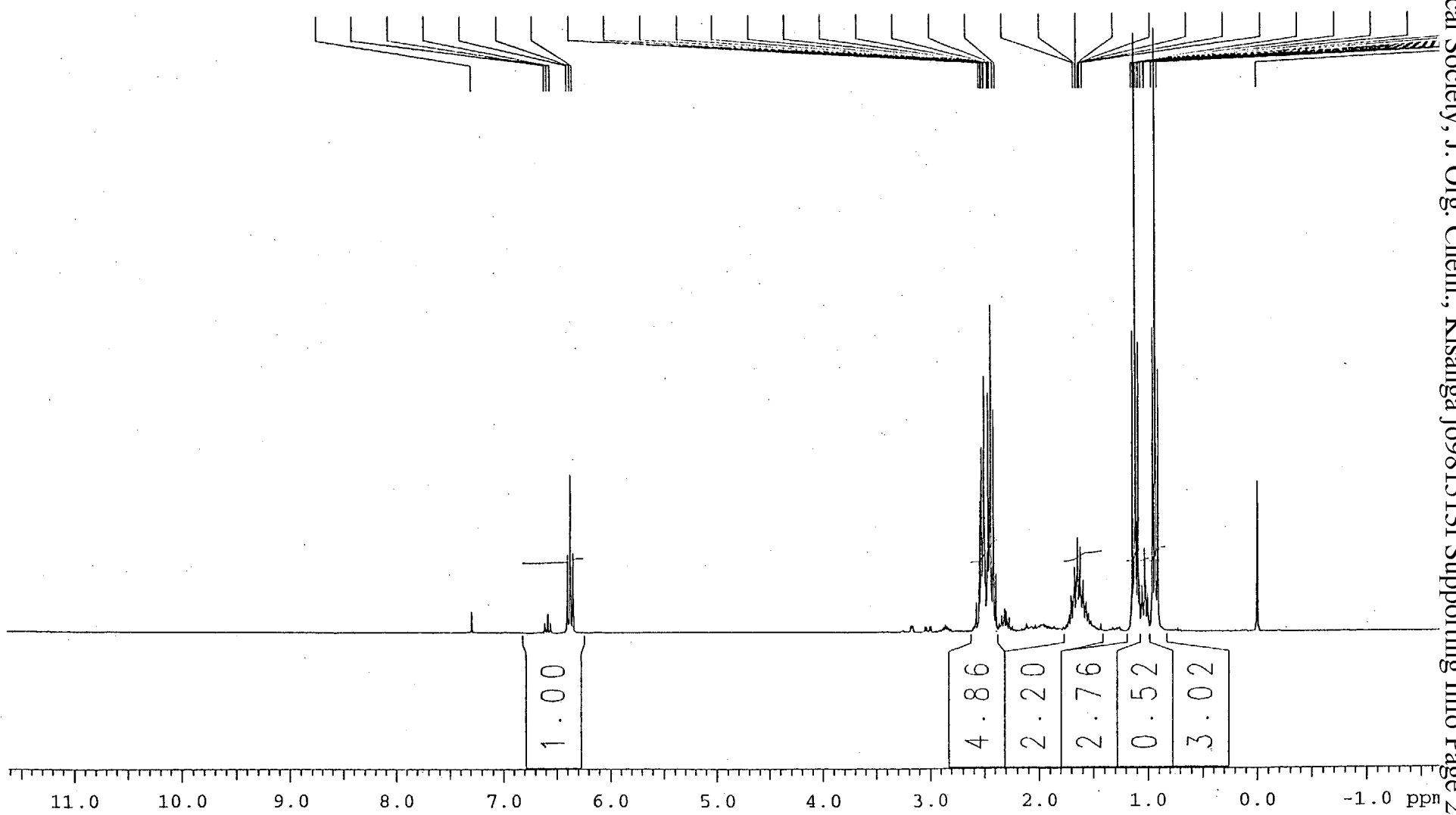


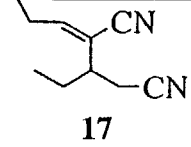
Mass	Int%	Dev ppm	C	H	N
258.21002	9.9	1.63	17	26	2
257.20100	3.2	-3.02	17	25	2
246.20958	5.5	-0.09	16	26	2
231.18593	11.6	-0.85	15	23	2
230.17542	2.2	No matches found			
229.17057	15.6	0.43	15	21	2
218.17448	4.1	No matches found			
217.17073	24.6	1.17	14	21	2
207.19429	14.8	No matches found			
206.19113	97.6	1.24	14	24	1
205.17278	11.8	No matches found			
188.13133	10.6	-0.09	12	16	2
187.12303	3.1	-2.65	12	15	2
178.15192	4.2	No matches found			
177.13918	34.4	0.02	11	17	2
176.13318	31.8	No matches found			
175.12360	22.2	0.42	11	15	2
164.14356	6.7	-2.20	11	18	1
161.10835	6.2	2.98	10	13	2
159.09237	3.1	0.91	10	11	2
150.12763	5.3	-4.32	10	16	1
149.12092	20.5	3.18	10	15	1
148.11301	2.0	2.64	10	14	1
148.10004	10.8	-0.05	9	12	2
147.09269	53.8	3.15	9	11	2
145.07704	3.4	3.22	9	9	2
136.11222	8.9	-2.94	9	14	1
135.10295	4.2	No matches found			
134.09509	14.3	No matches found			
133.07805	3.8	No matches found			
132.07045	2.1	No matches found			
124.11067	2.1	No matches found			
123.10448	6.4	-2.62	8	13	1
122.09670	7.1	-2.27	8	12	1
121.08733	4.4	No matches found			
120.07966	26.2	No matches found			
119.07376	3.6	2.17	8	9	1
119.06076	3.2	-1.35	7	7	2
118.06486	5.7	-6.93	8	8	1
116.04963	2.4	-3.44	8	6	1
108.08096	14.3	-3.33	7	10	1
107.07299	8.5	-4.74	7	9	1
107.06608	2.1	No matches found			
107.05683	7.6	No matches found			
106.05420	100.0	No matches found			
105.05396	2.2	No matches found			
104.05007	8.5	0.49	7	6	1
103.05418	2.3	-5.80	8	7	0
94.06525	6.5	-4.50	6	8	1
93.07004	6.9	-4.11	7	9	0
93.05701	5.1	-9.04	6	7	1
92.04994	7.0	-0.88	6	6	1
91.05438	9.2	-4.39	7	7	0
80.05167	6.0	No matches found			
79.05671	13.3	No matches found			
77.04123	13.4	No matches found			
72.09098	2.0	No matches found			



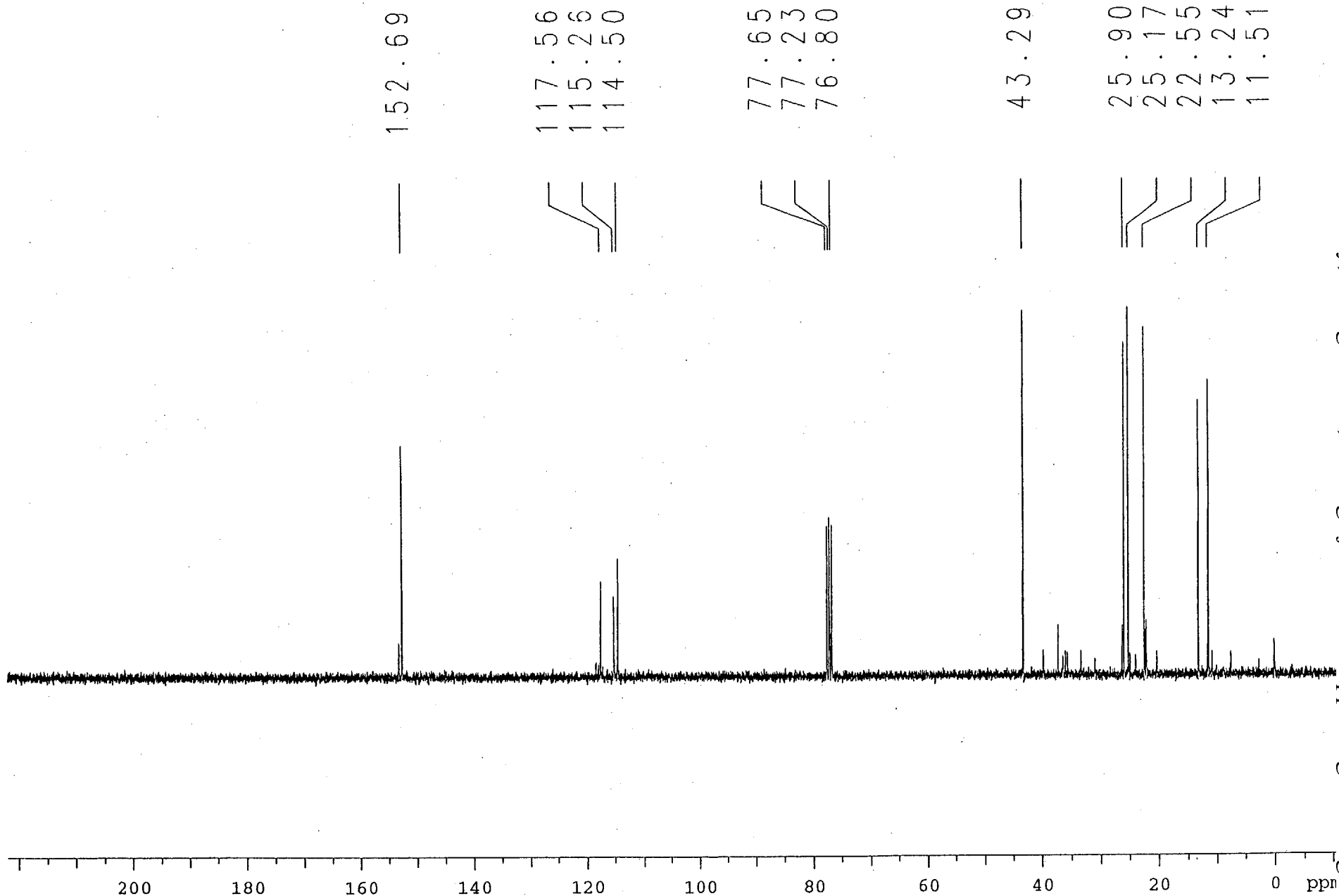
E-3-pentenenitrile / iPr3Phan

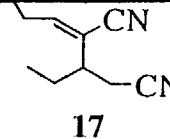
7.29	6.61	6.58	6.56	6.40	6.37	6.35	6.54	6.53	6.52	6.51	6.50	6.49	6.47	6.46	6.45	6.44	6.42	6.39	6.67	6.66	6.65	6.63	6.62	6.62	6.60	6.59	6.14	6.13	6.11	6.10
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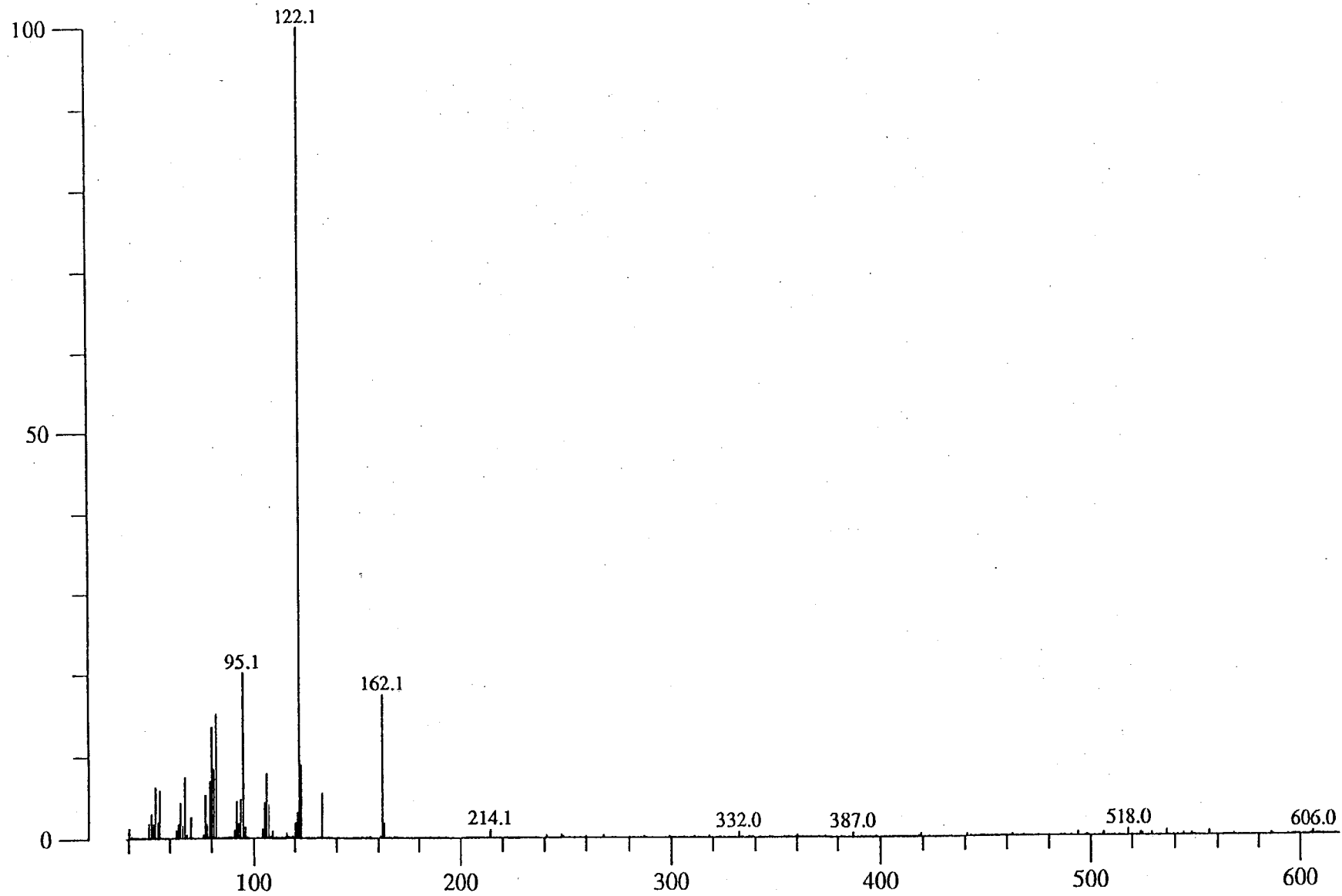


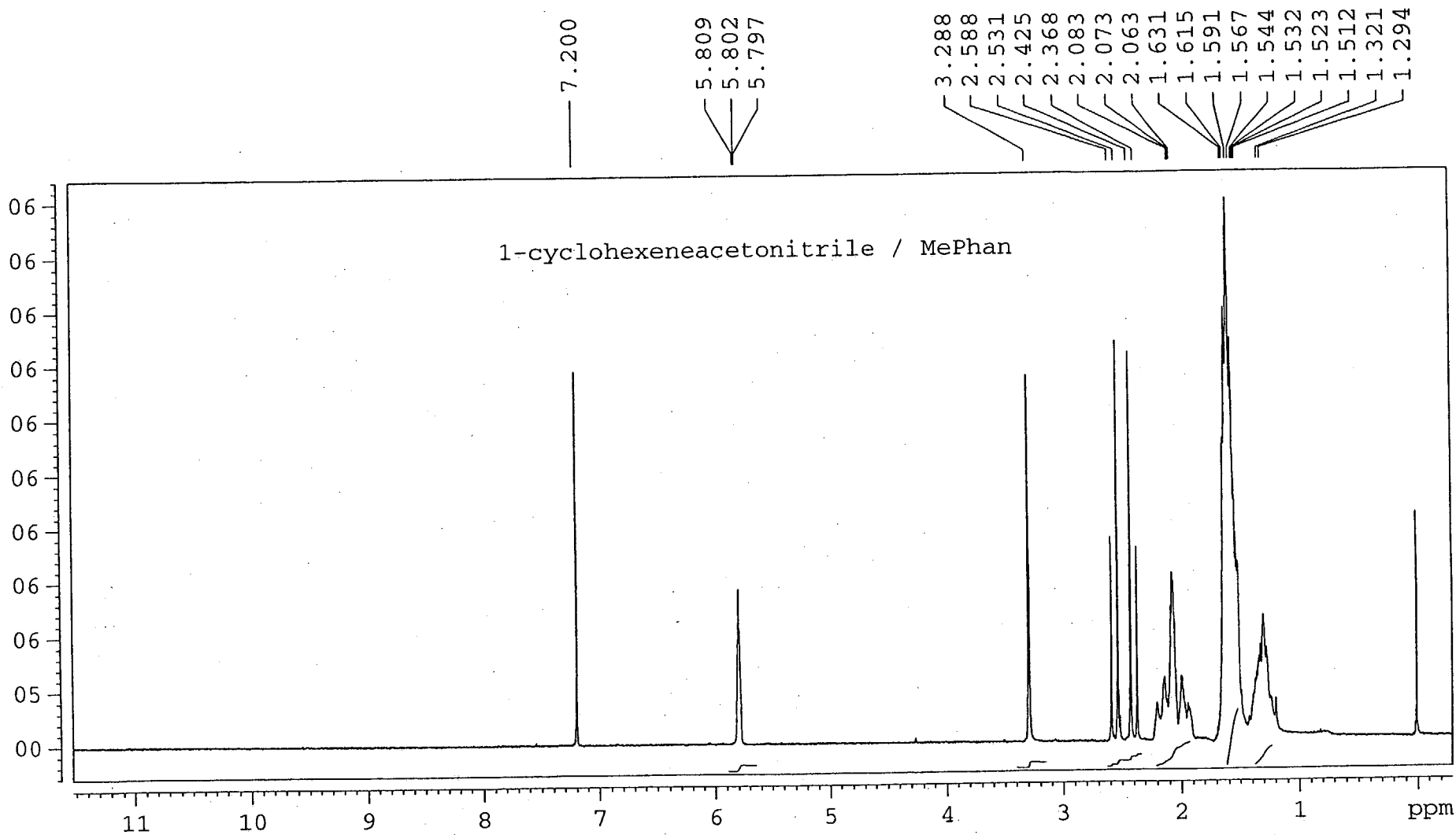
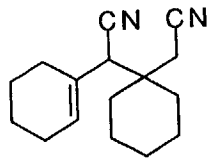
trans-3-pentenenitrile / iPr3Phan/C6H6





MS70820001 Scan 1 (Av 2-45 Acq) 100%=6789 mv 20 Feb 98 16:44  
HRP PK196a12 P.Kisanga 10K Resolution 70eV EI Solids Probe





dat2 pk88d.fid 1

1.8000

0.940

2.202

4.456

10.467

3.563