

# Tentamen "Structuuropheldering"

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28 augustus 2003, collegezaal 3

14.00 uur - 17.00 uur

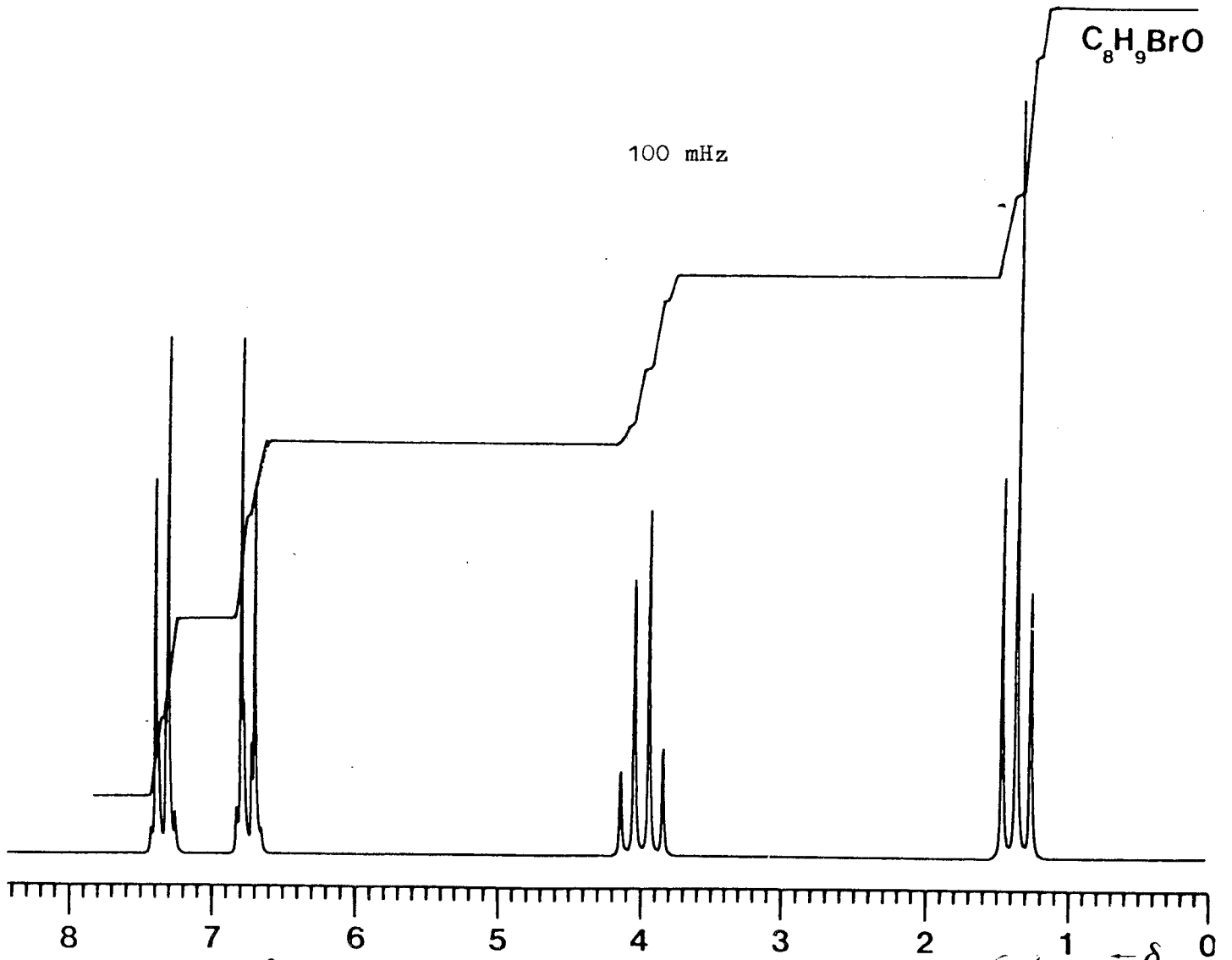
In alle onderstaande vraagstukken wordt gevraagd van een onbekende verbinding de structuur te bepalen. Geef hierbij een zo volledig mogelijke motivering. Ken in de spectra zoveel mogelijk pieken toe, in MS spectra en in IR spectra in elk geval de meest relevante pieken.

**LEES DE VRAAGSTUKKEN VOORAL GOED !**

1. 100 MHz  $^1\text{H}$  NMR spectra van een verbinding met samenstelling  $\text{C}_8\text{H}_9\text{BrO}$
2. De verbinding bevat alleen C, H en N. Molecuul gewicht 103.
3. Van de verbinding  $\text{C}_6\text{H}_7\text{N}$  zijn de volgende spectra gegeven:
  - 3A 600 MHz  $^1\text{H}$  NMR
  - 3B 600 MHz  $^1\text{H}$  NMR aromaat gebied
  - 3C  $^1\text{H}$  -  $^2\text{D}$  cosy
  - 3D  $^{13}\text{C}$  APT
  - 3E CH HM QC
  - 3F  $^{13}\text{C}$ - $^{13}\text{C}$  inadequate spectrum
  - 3G  $^{13}\text{C}$ - $^{13}\text{C}$  aromaat gebied van het inadequate spectrum

Wat is de structuur van  $\text{C}_6\text{H}_7\text{N}$  ? en ken zoveel mogelijk  $^1\text{H}$  en  $^{13}\text{C}$  signalen toe.
4. Fig. 4a en 4b: een combinatie probleem
5. Figuur 5a en 5b: een combinatie probleem

# Fig. 1



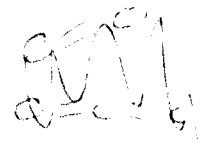
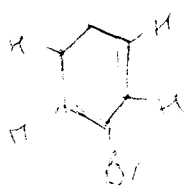
2.5	2.65	4.35
2	2	3
CH <sub>2</sub> CH <sub>2</sub>	CH <sub>2</sub>	CH <sub>3</sub>

CH<sub>3</sub> CH CH  
CH<sub>2</sub> CH<sub>2</sub> CH<sub>2</sub>

CH<sub>2</sub>  
CH  
CH

$$\delta + 1 - \frac{1}{2}(g + 1)$$

$g = 5 \rightarrow 4.0 \text{ ppm} \rightarrow$  benzene?  
 geen c + h  
 kan niet



(a) A compound with a molecular weight of 103.

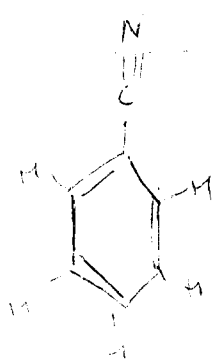
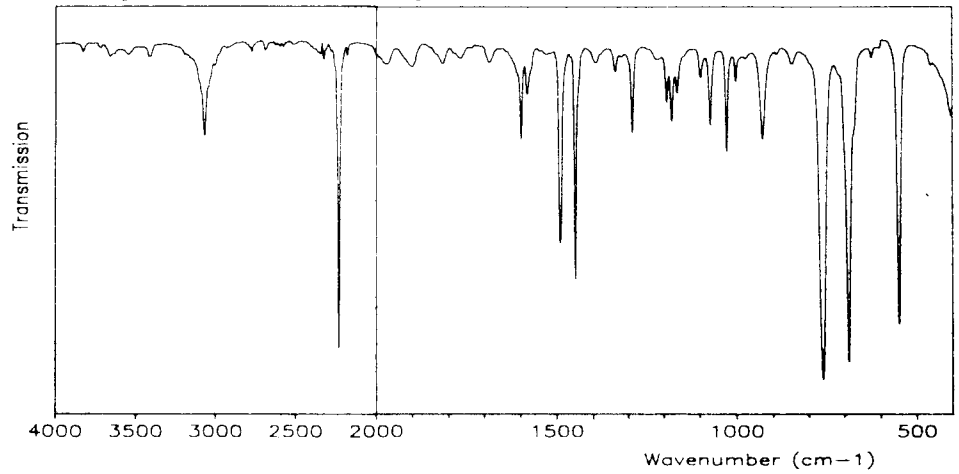


Fig. 2

1x C  
1x N  
5x H

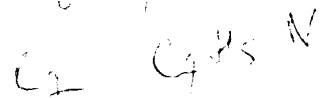
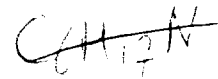
C, H, N

$$2(1) - \frac{1}{2}(5 - 1)$$

6 DBE

$$103 - 14 - 23$$

$$22 = 12$$



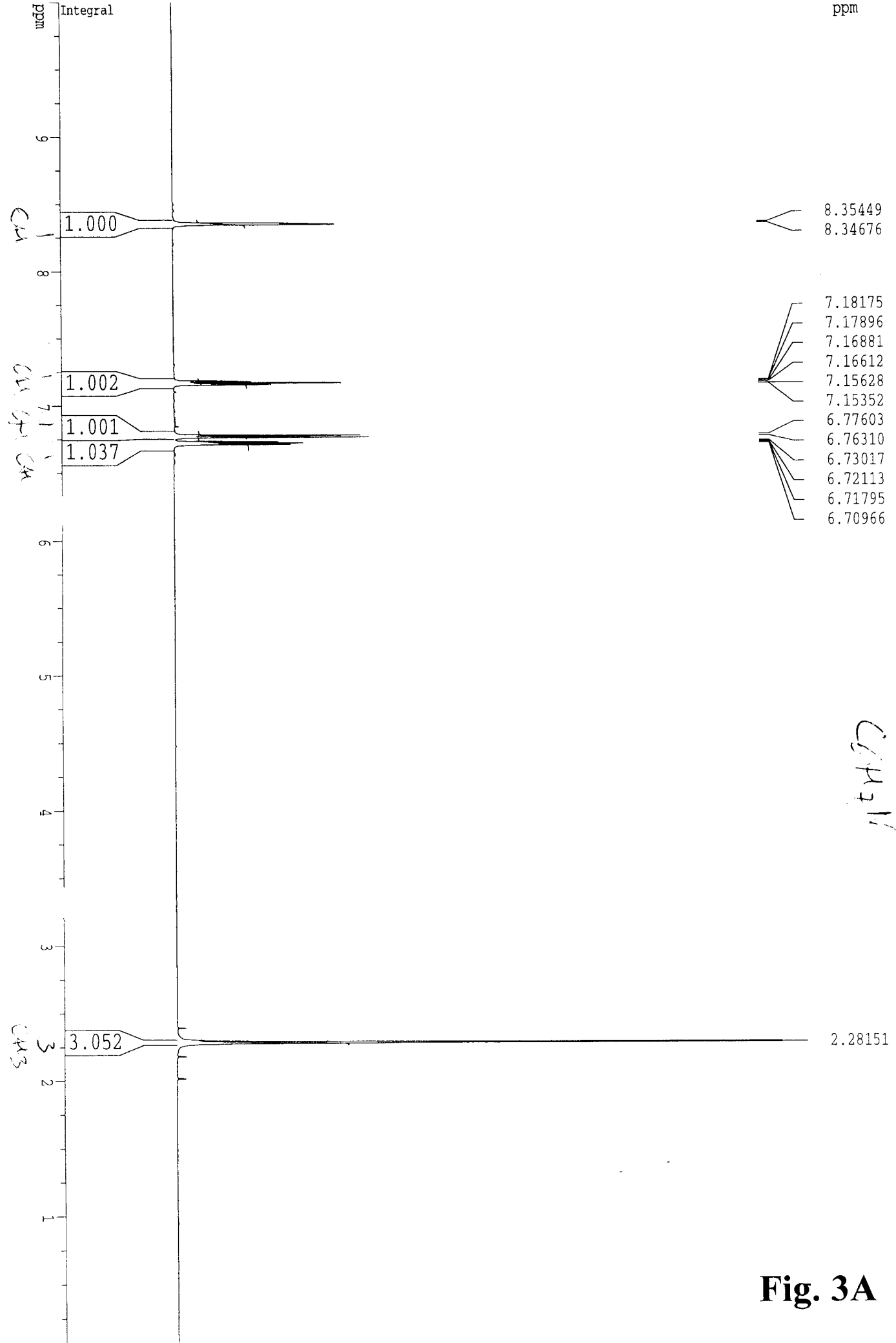
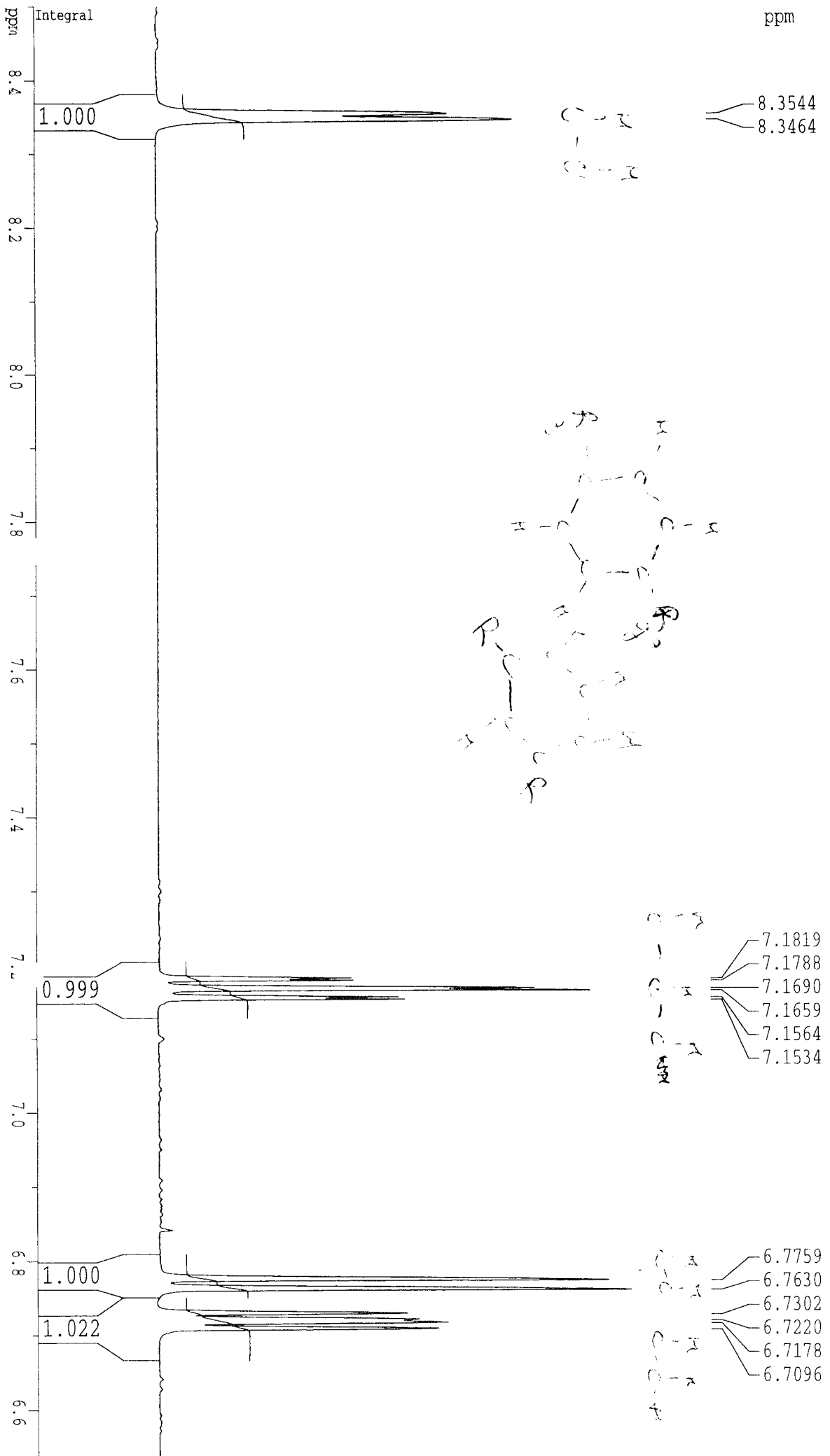


Fig. 3A

3B 600 MHz <sup>1</sup>H-NMR van C<sub>6</sub>H<sub>7</sub>N in C<sub>6</sub>D<sub>6</sub>,  
 aromatisch gebied vergroot, 8.5 - 6.5 ppm



3C  
600 MHz, 1H-2D-COSY van C6H7N  
in C6D6

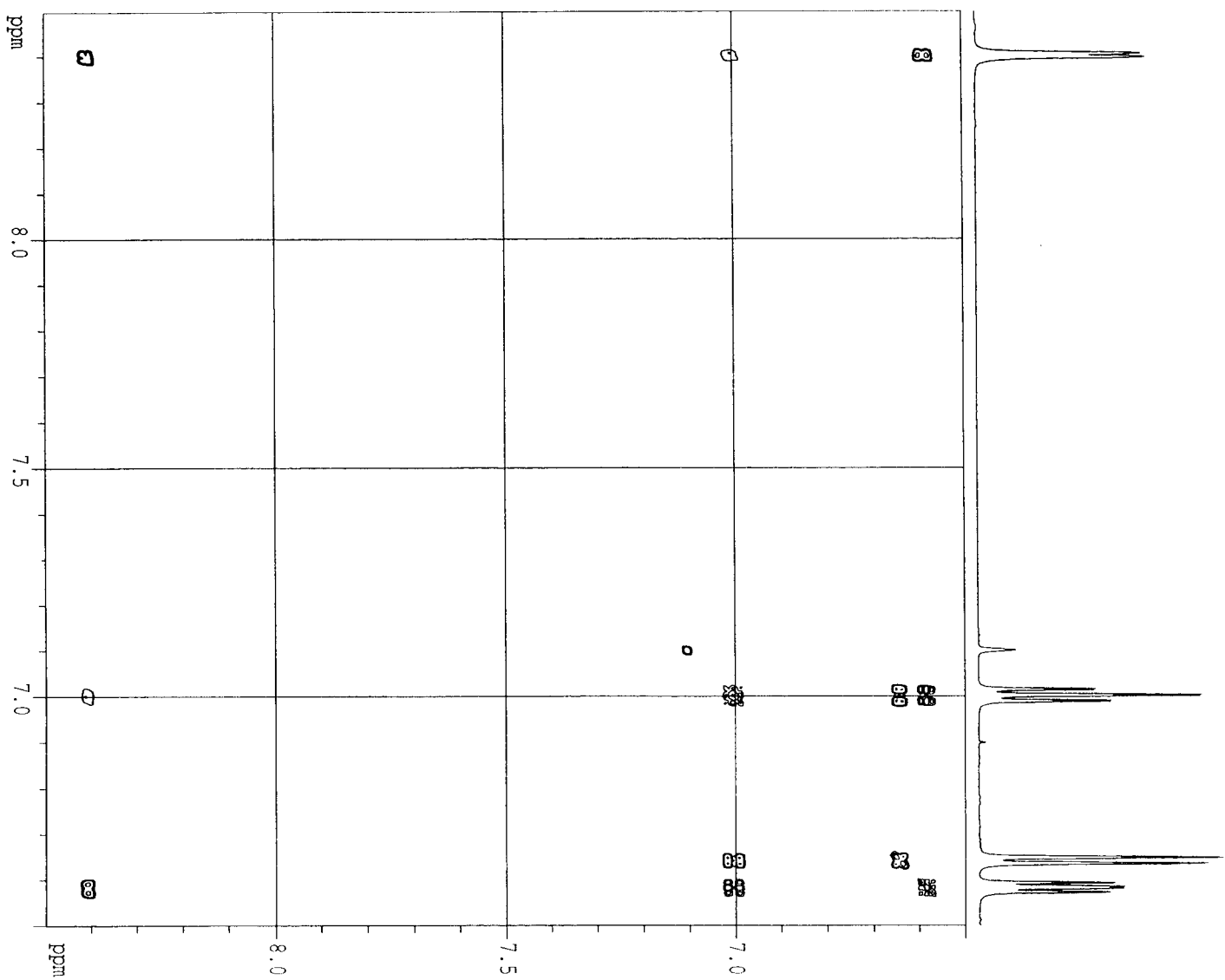
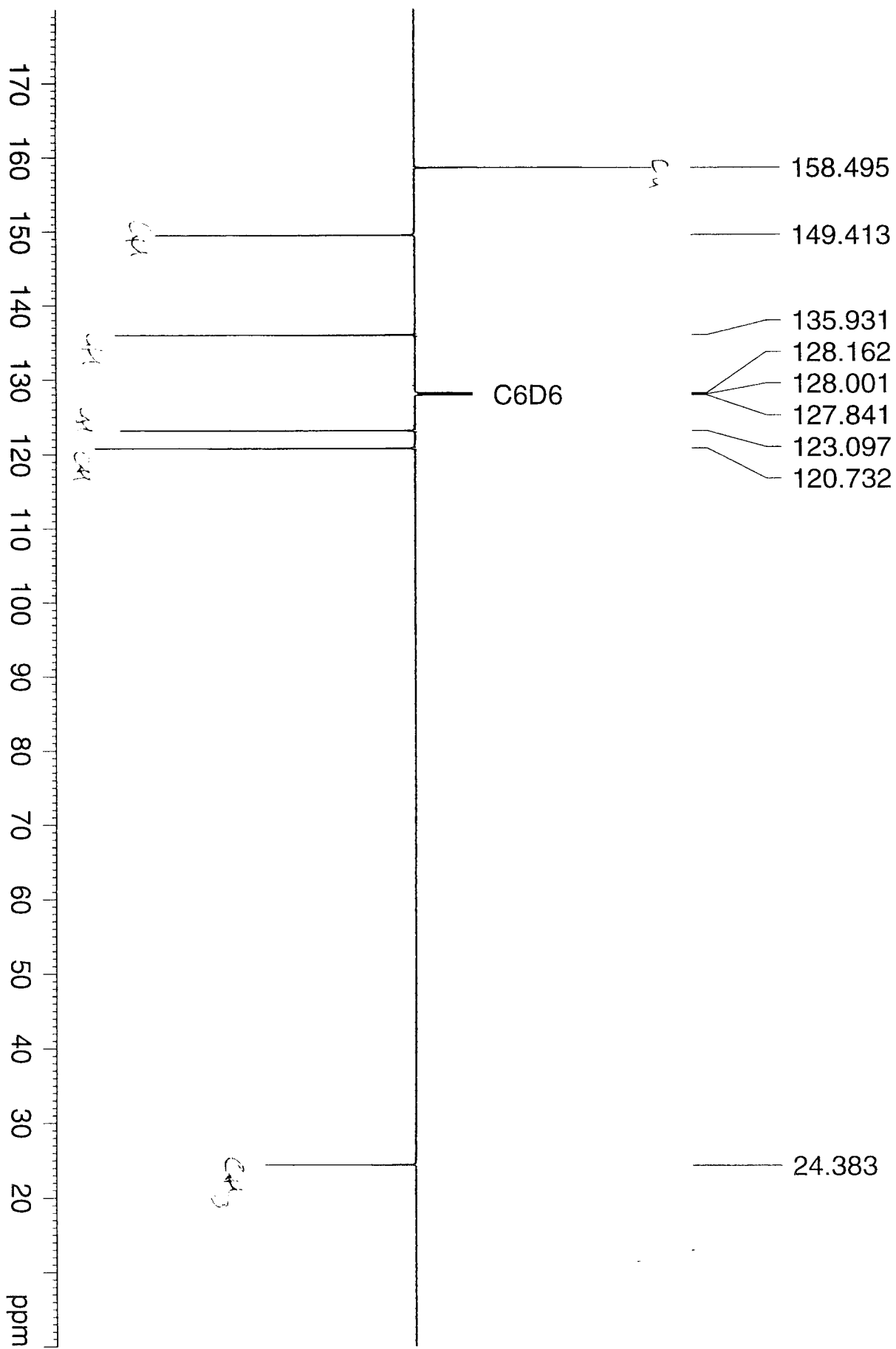
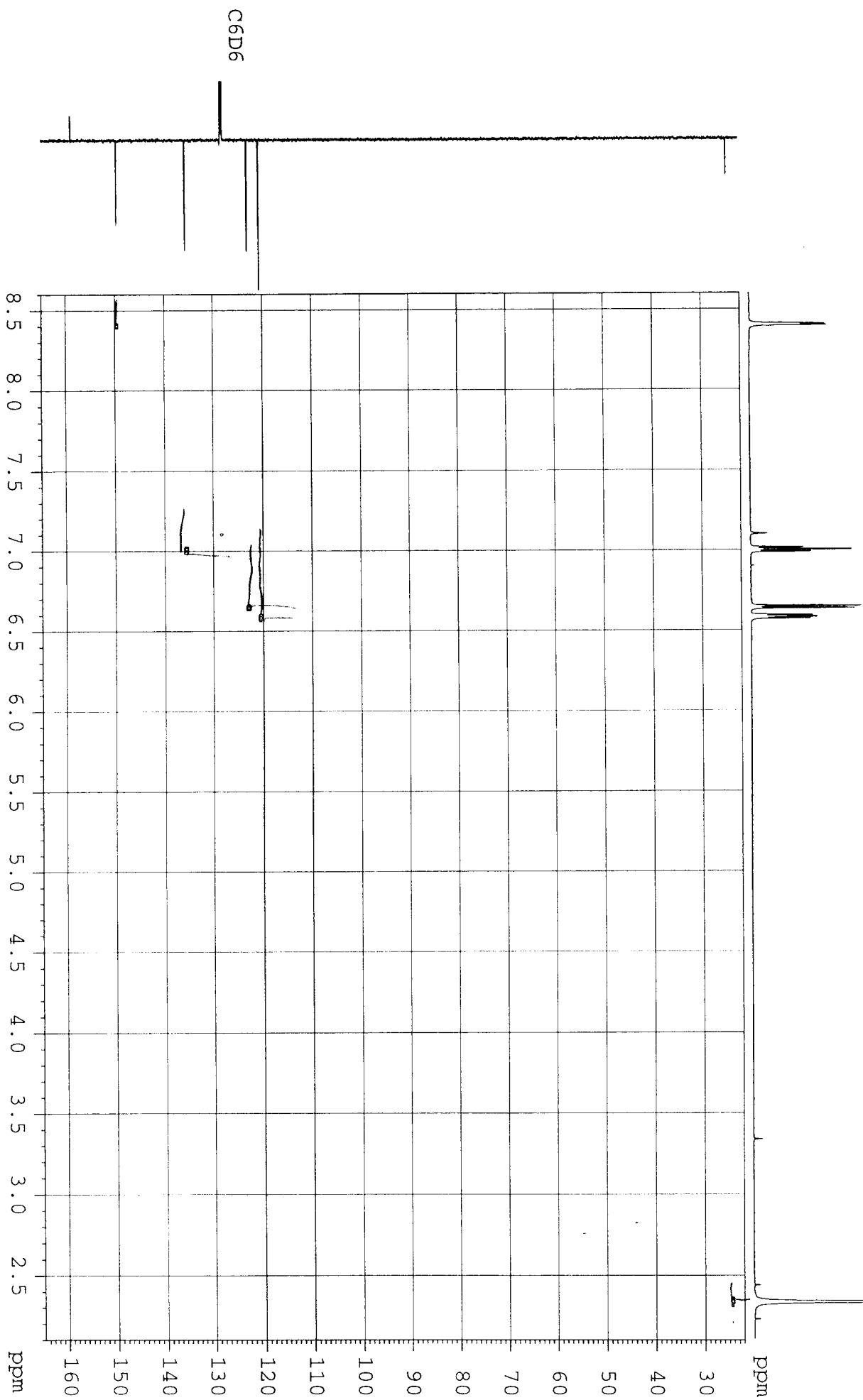


Fig 3C

3D. <sup>13</sup>C-APT NMR van C<sub>6</sub>H<sub>7</sub>N in C<sub>6</sub>D<sub>6</sub>, 150 MHz, dmx-600



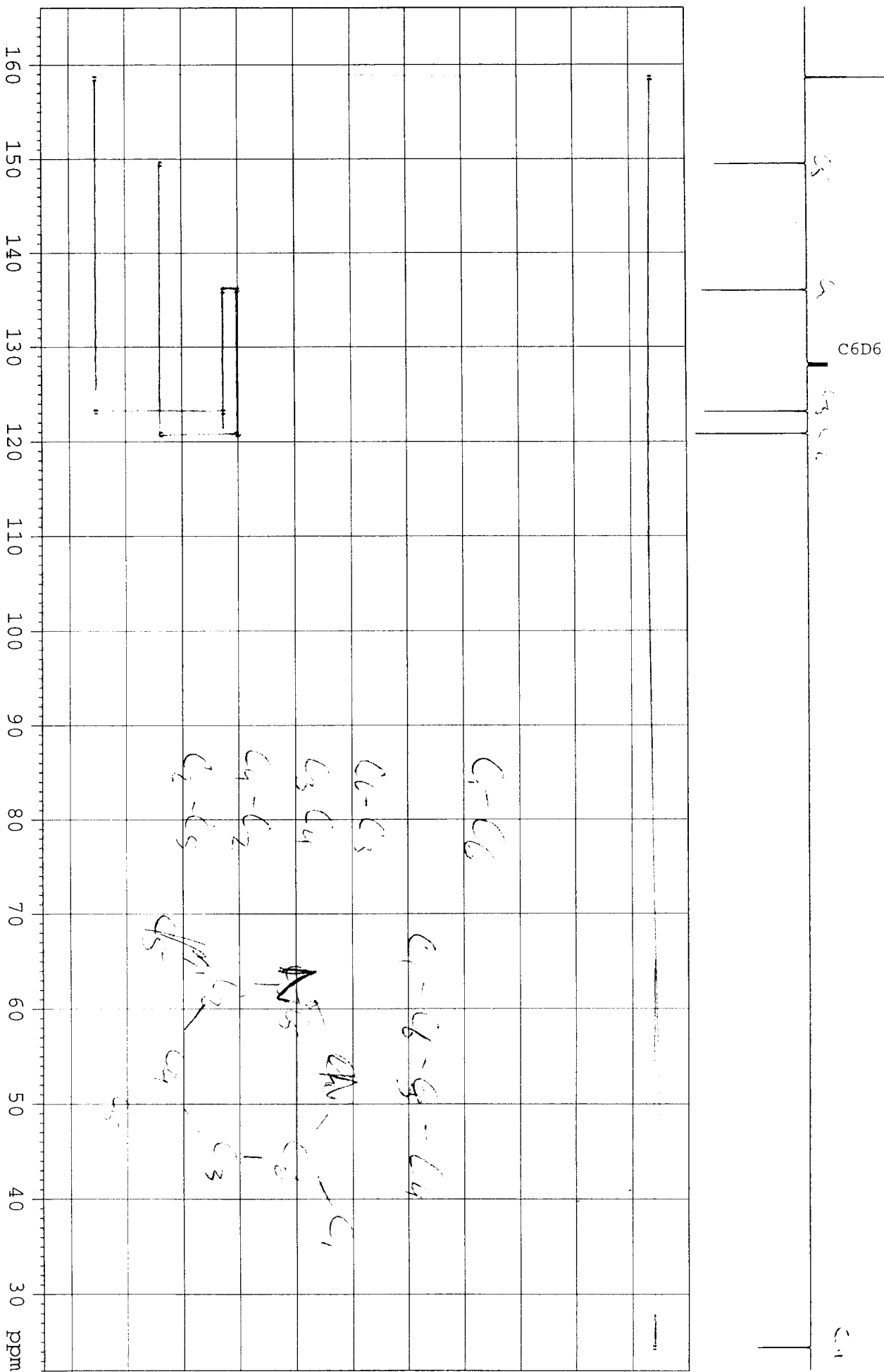
3E CH-HMQC van C<sub>6</sub>H<sub>7</sub>N in C<sub>6</sub>D<sub>6</sub>, dmx-600



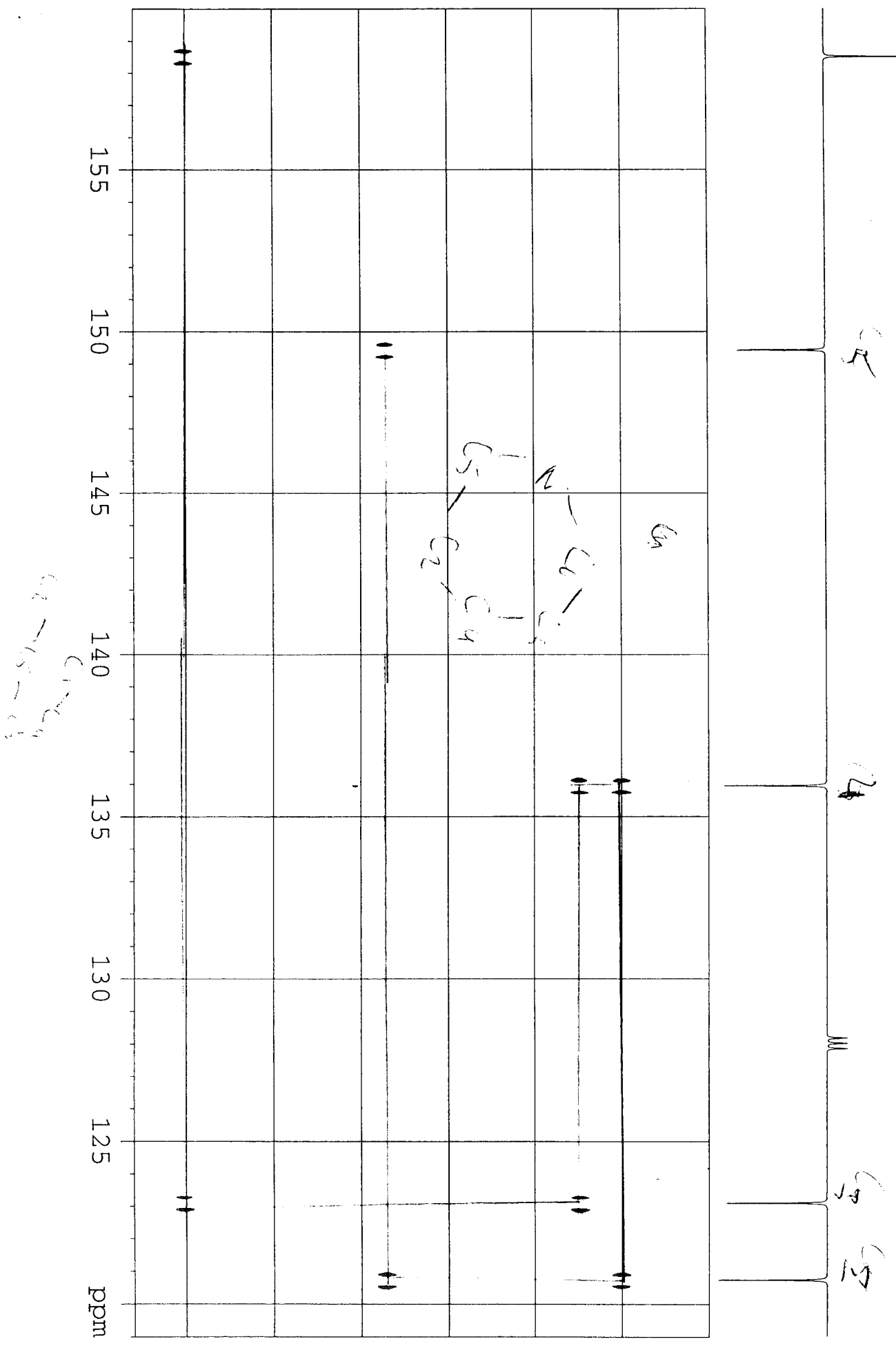


3F. 13C-13C INADEQUATE van C6H7N, in C6D6, dm<sub>x</sub>600

Fig. 2



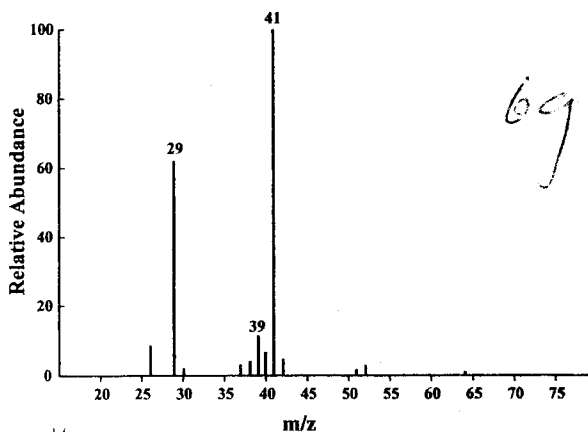
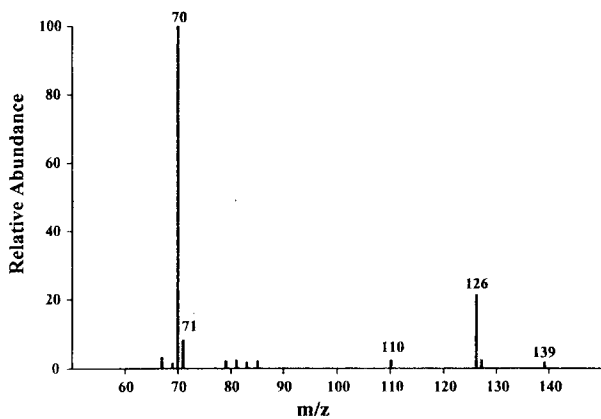
3G. vergroting van aromatisch gebied van  $^{13}\text{C}$ - $^{13}\text{C}$  INADEQUATE van  $\text{C}_6\text{H}_7\text{N}$ , in  $\text{C}_6\text{D}_6$ ,  $\text{dmx600}$



PROBLEM 16-4

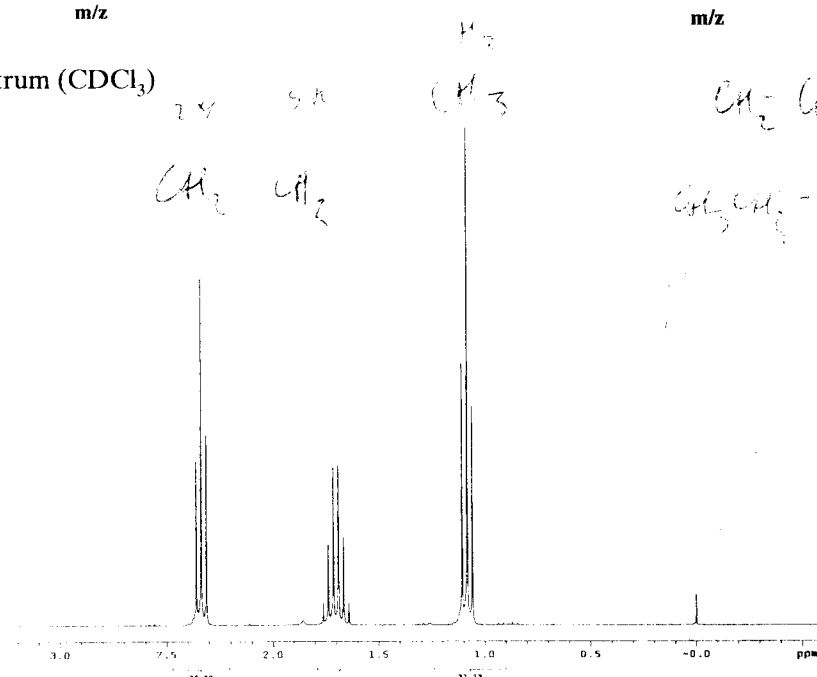
Mass spectrum (EI) [Note: The molecular ion is absent. Because EI is a hard method of ionization, loss of multiple hydrogen atoms sometimes occurs, that is, H loss followed by one or more H<sub>2</sub> losses.]

Mass spectrum (CI)



69

Proton NMR spectrum (CDCl<sub>3</sub>)



2H 5H  
CH<sub>2</sub> CH<sub>2</sub>

H<sub>2</sub>  
CH<sub>3</sub>

CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>3</sub> CH<sub>2</sub> CH<sub>2</sub> CH<sub>3</sub>  
CH<sub>3</sub>CH<sub>2</sub>-C(CH<sub>3</sub>)<sub>2</sub>-CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>

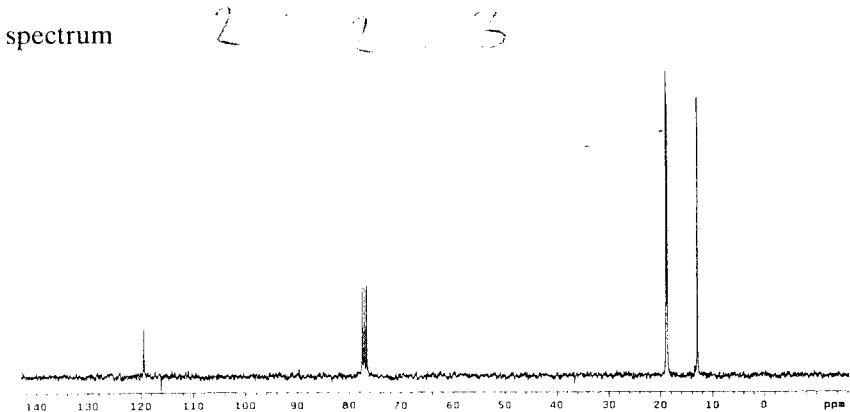
3H C

H<sub>7</sub>

H<sub>3</sub>

1H CH<sub>2</sub> CH<sub>2</sub> CH<sub>3</sub>

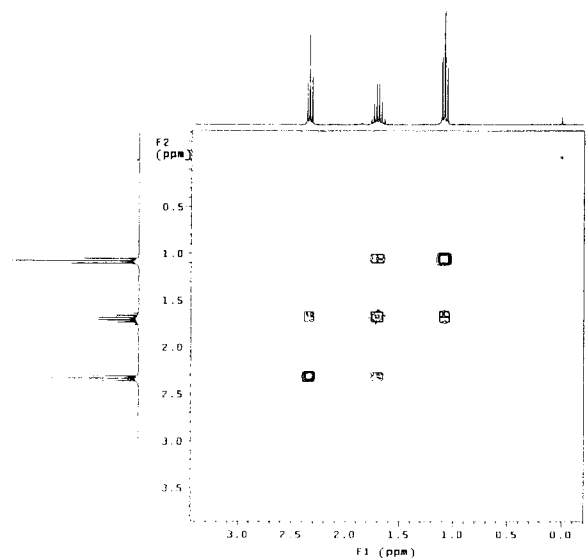
Carbon-13 NMR spectrum



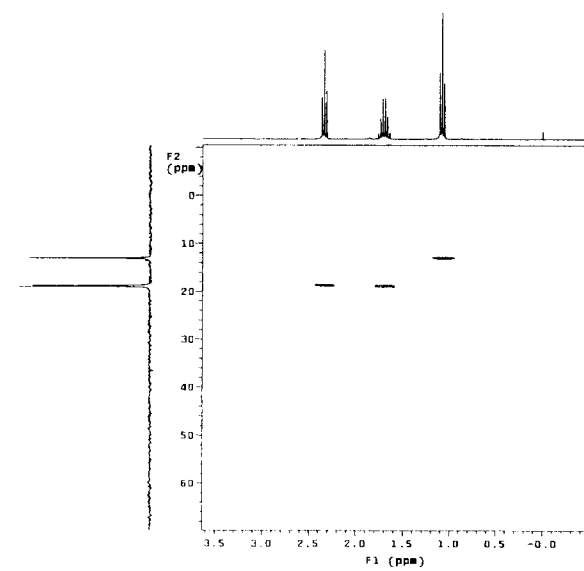
2 2 3

# Fig. 4 B

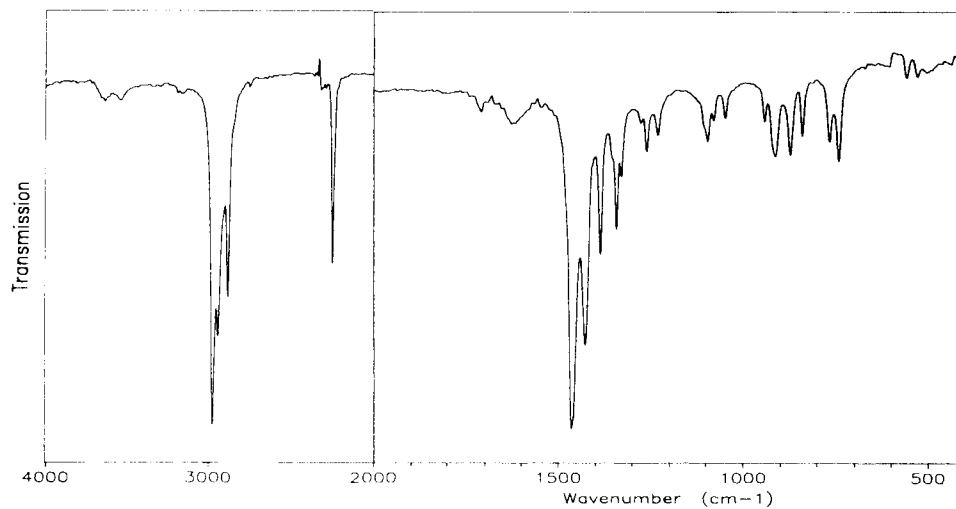
COSY spectrum



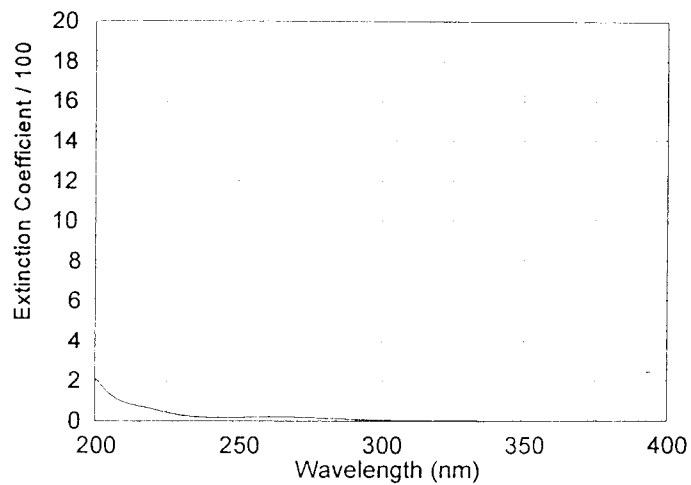
HETCOR spectrum



Infrared spectrum (neat)

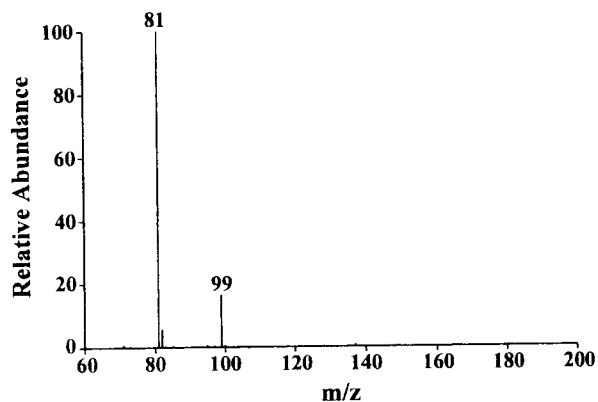


Ultraviolet-visible spectrum (EtOH)

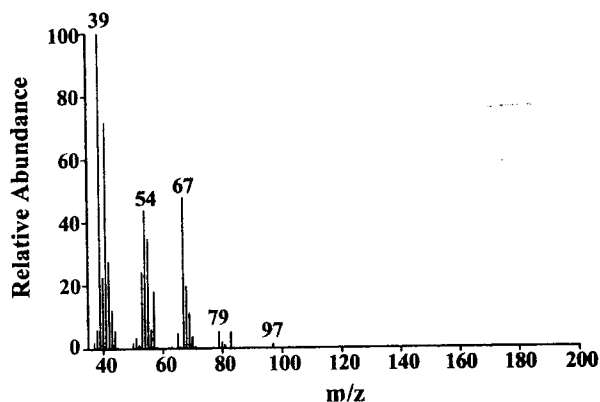


PROBLEM 16-18

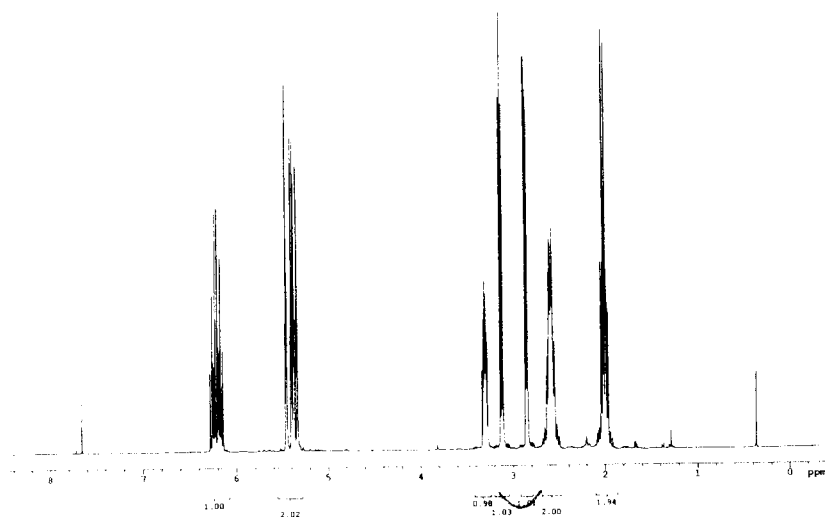
Mass spectrum (CI)



Mass spectrum (EI)



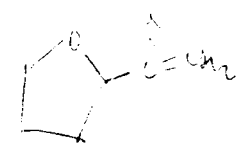
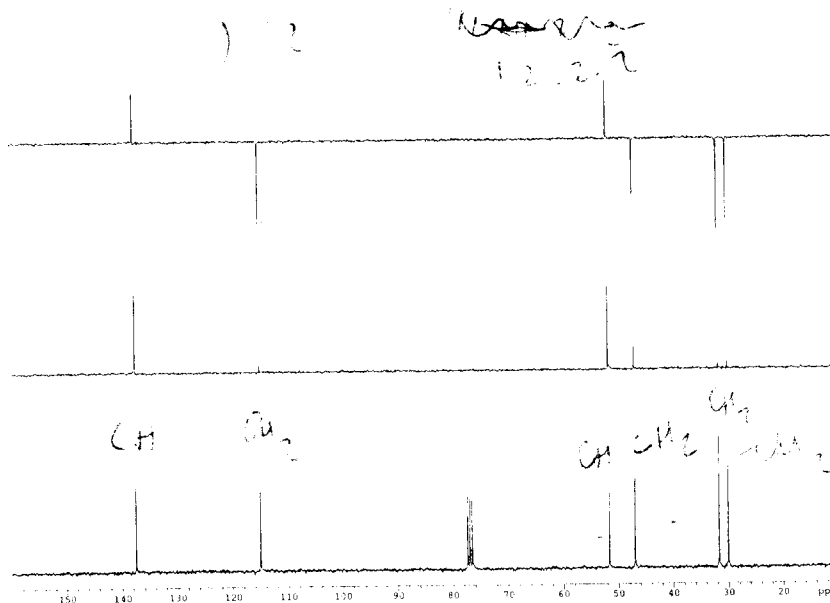
Proton NMR spectrum (CDCl<sub>3</sub>)



79  
C<sub>6</sub>H<sub>5</sub>O

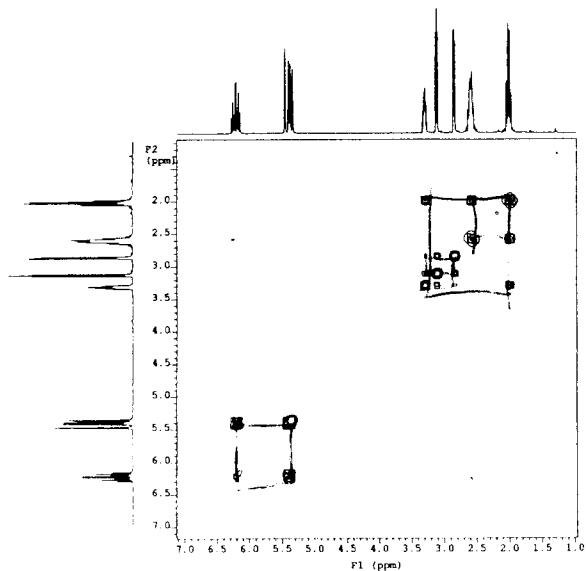


DEPT spectra

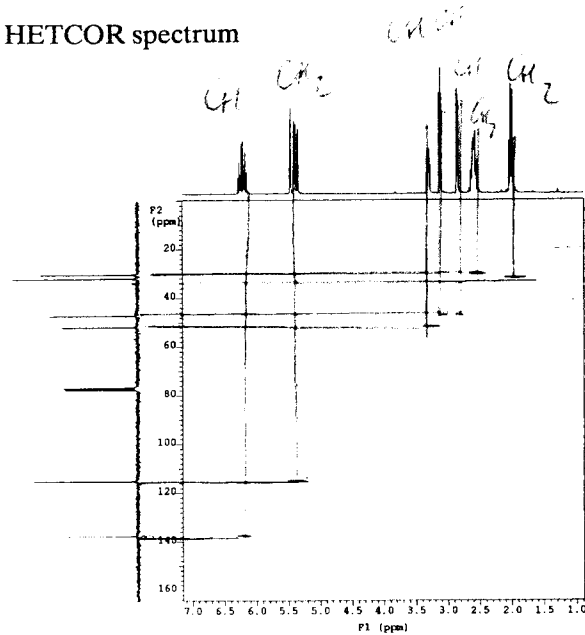


# Fig. 5 B

COSY spectrum

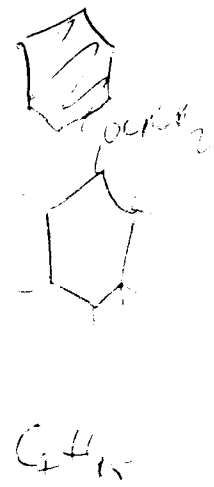
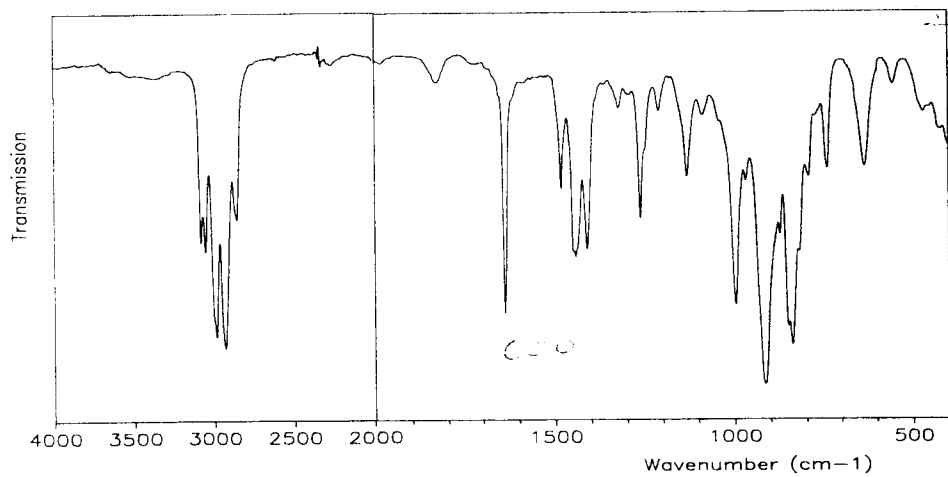


HETCOR spectrum



$C_7H_{10}$   
 79  
 65  
 48  
~~57~~  
 53  
 $C_4H_5ON$

Infrared spectrum (neat)



Ultraviolet-visible spectrum (EtOH,  $\epsilon[202]$  49,  $\epsilon[211]$  13,  $\epsilon[271]$  14)

