

Tentamen "Structuuropheldering"

Docent: Prof.dr. J. Lugtenburg

26 augustus 2002, collegezaal 4-5

13.30 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 ^1H NMR spectra van verbinding met samenstelling $\text{C}_7\text{H}_6\text{N}_2\text{O}_5$
2. In Figuur 2A, 2B en 2C zijn de ^1H NMR, ^{13}C NMR en off resonance ^1H decoupled ^{13}C NMR spectra gegeven van een verbinding die uitsluitend C, H en O bevat en waarvan het moleculair gewicht 126 bedraagt.
3. Van de verbinding $\text{C}_6\text{H}_7\text{N}$ zijn de volgende spectra gegeven:
 - 3A 600 MHz ^1H NMR
 - 3B 600 MHz ^1H NMR aromaats gebied
 - 3C ^1H - ^2D cosy
 - 3D ^{13}C APT
 - 3E CH HM QC
 - 3F ^{13}C - ^{13}C inadequate spectrum
 - 3G ^{13}C - ^{13}C aromaats gebied van het inadequate spectrumWat is de structuur van $\text{C}_6\text{H}_7\text{N}$? en ken zoveel mogelijk ^1H en ^{13}C signalen toe.
4. Fig. 4a en 4b: een combinatie probleem
5. Figuur 5a en 5b: een combinatie probleem



1H - NMR, 100 MHz

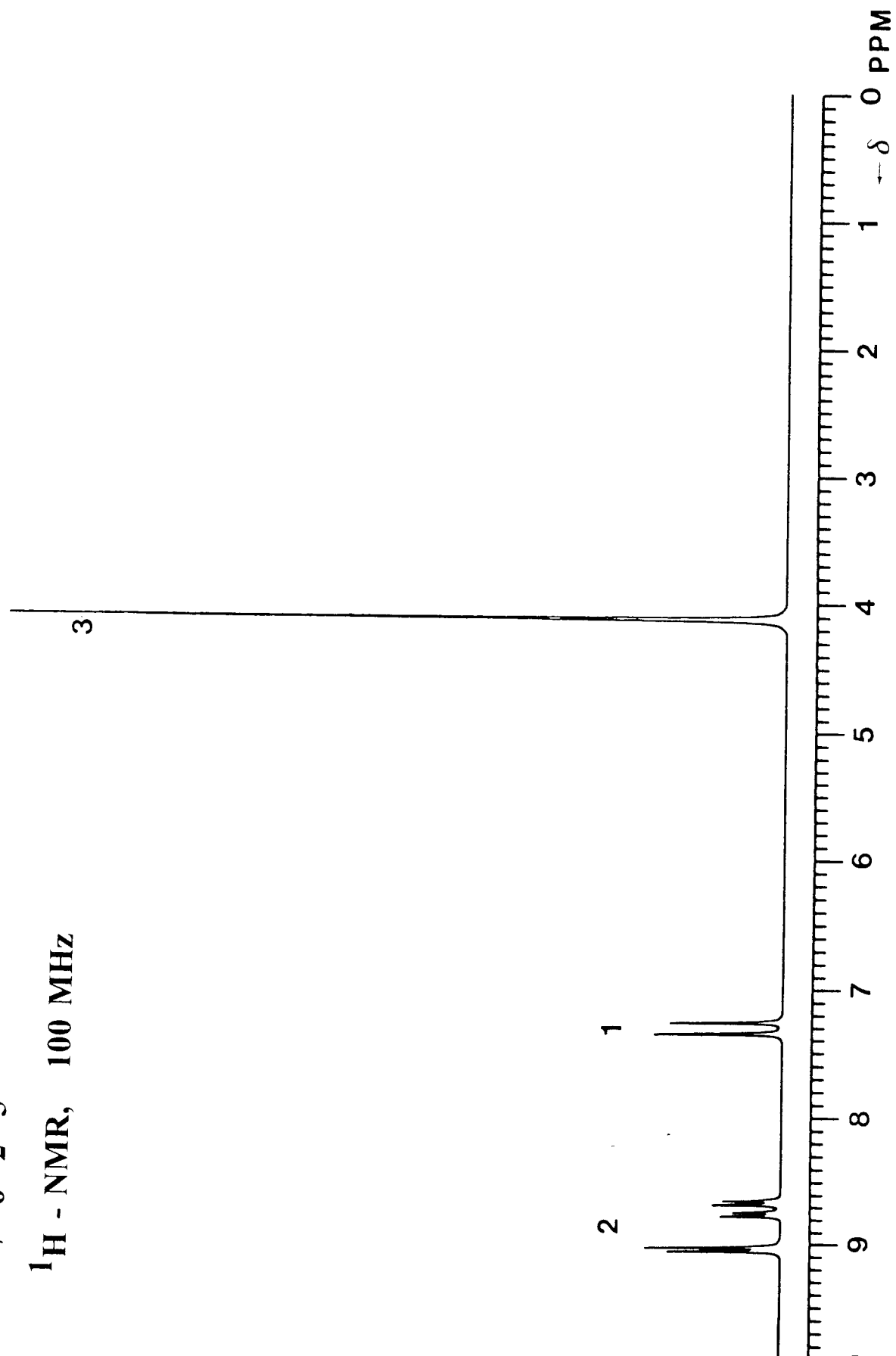


Fig. 1

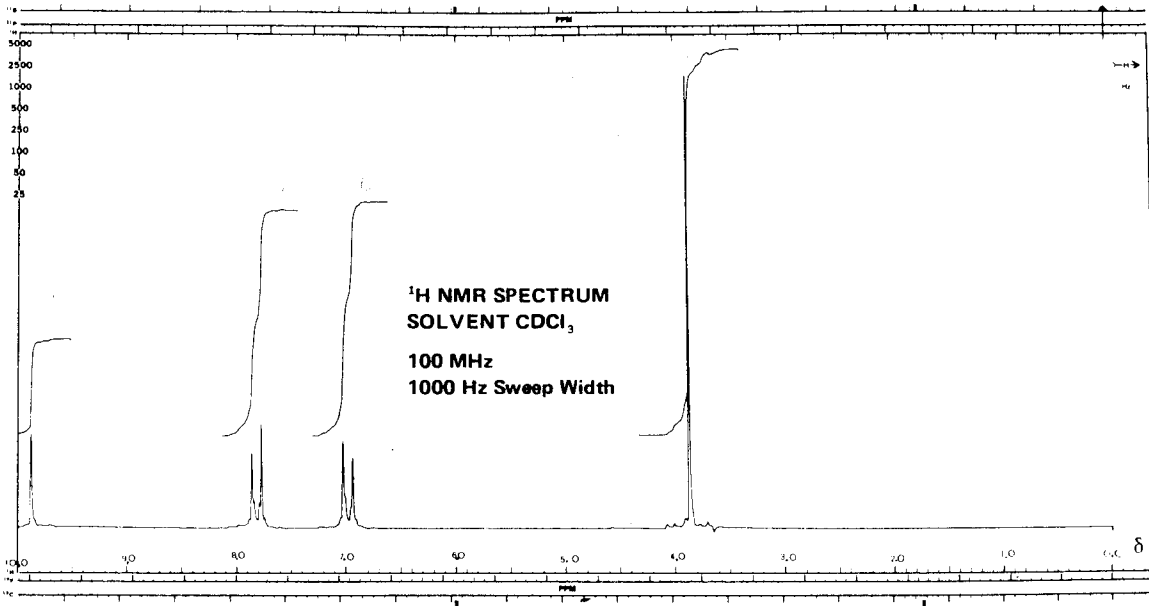


Fig. 2A

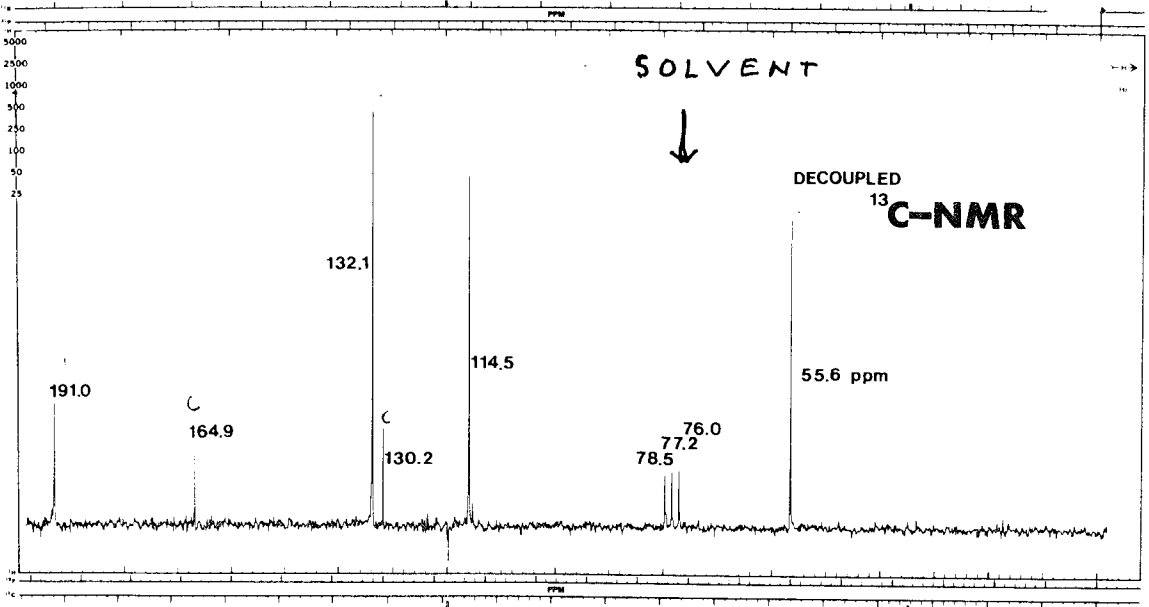


Fig. 2B

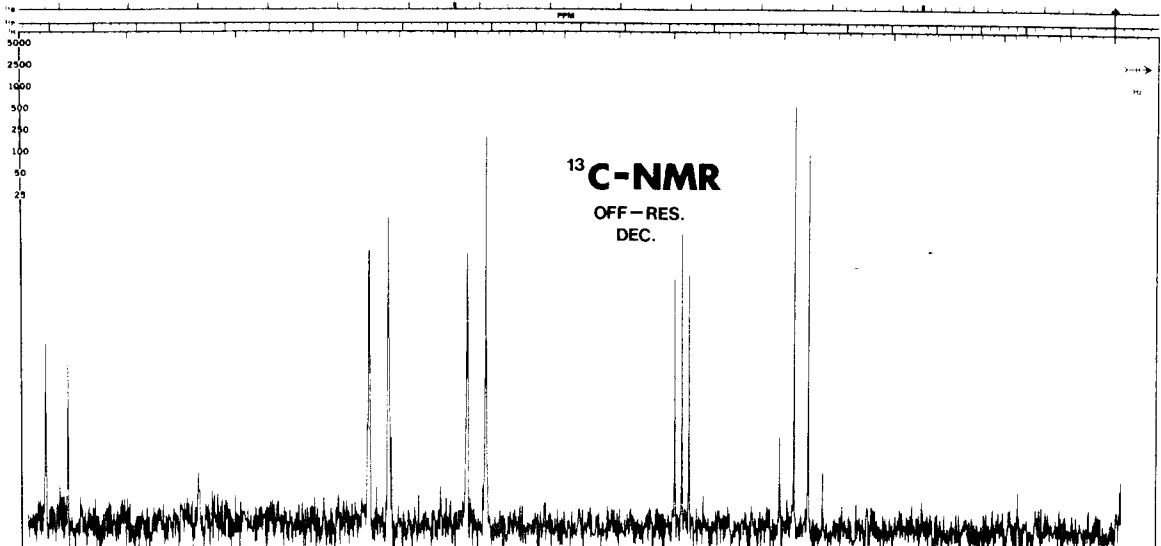
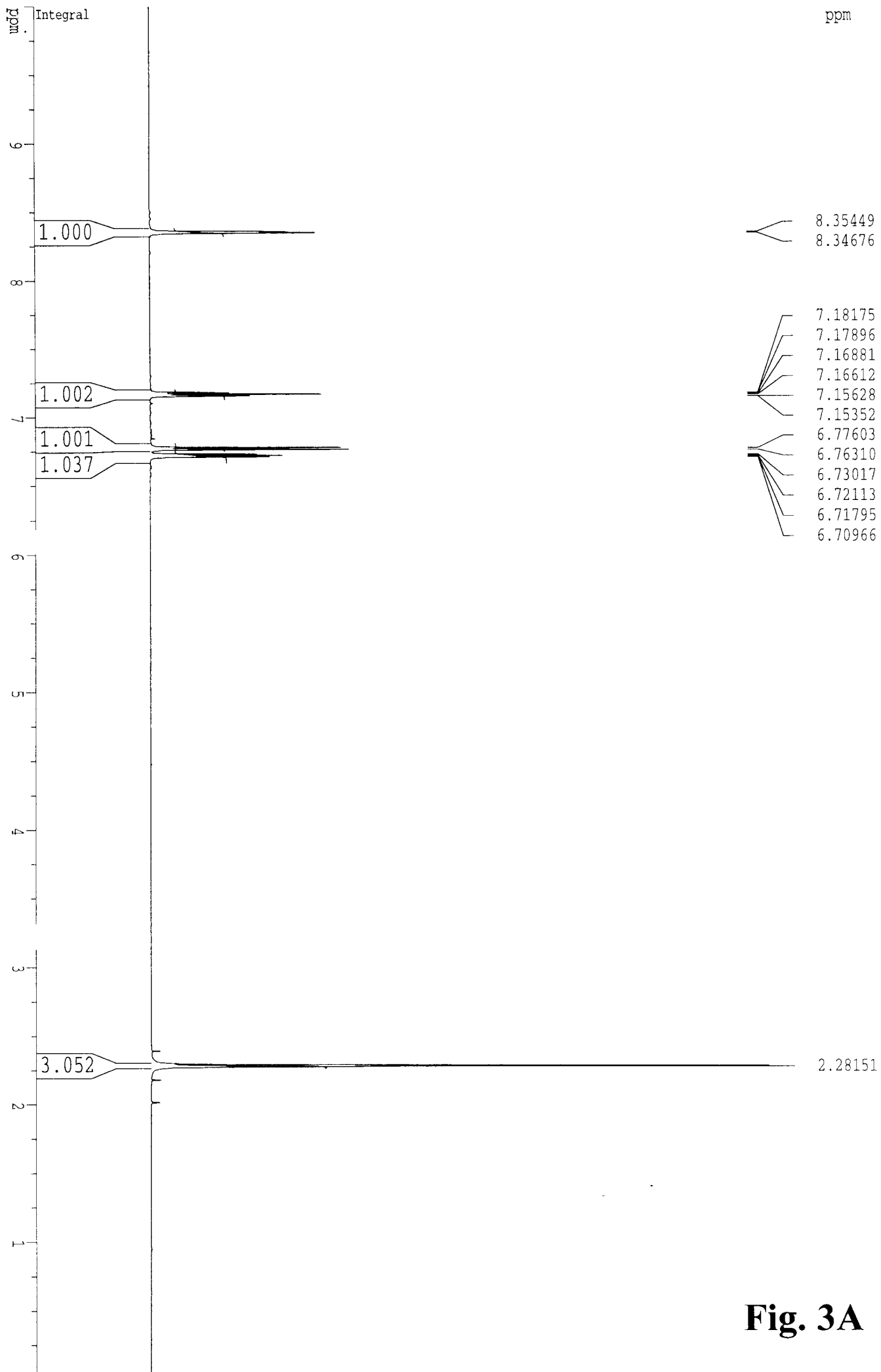


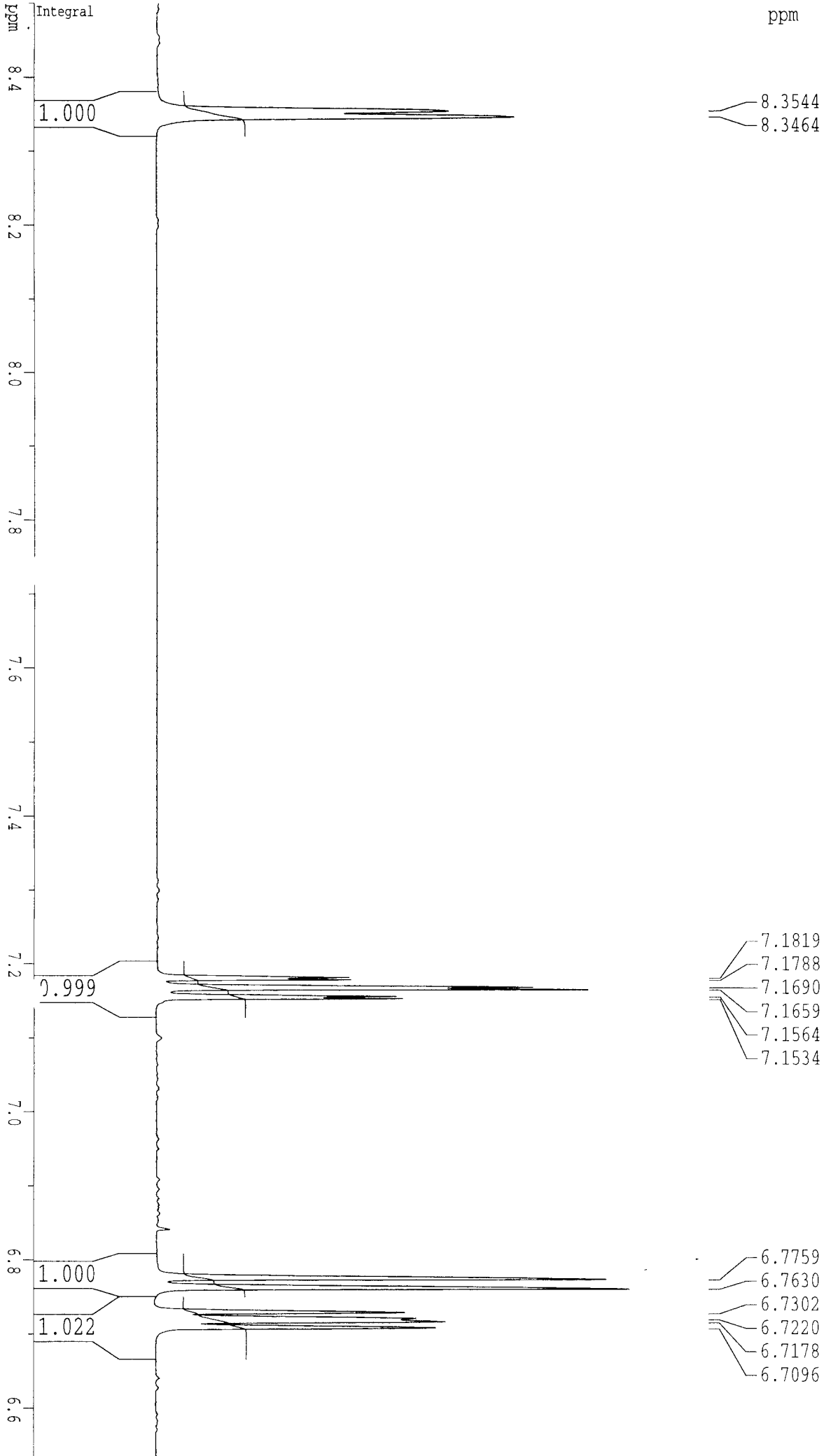
Fig. 2C



3A 600 MHz ¹H-NMR van C₆H₇N in C₆D₆

Fig. 3A

3B 600 MHz ¹H-NMR van C₆H₇N in C₆D₆,
aromatisch gebied vergroot, 8.5 - 6.5 ppm



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

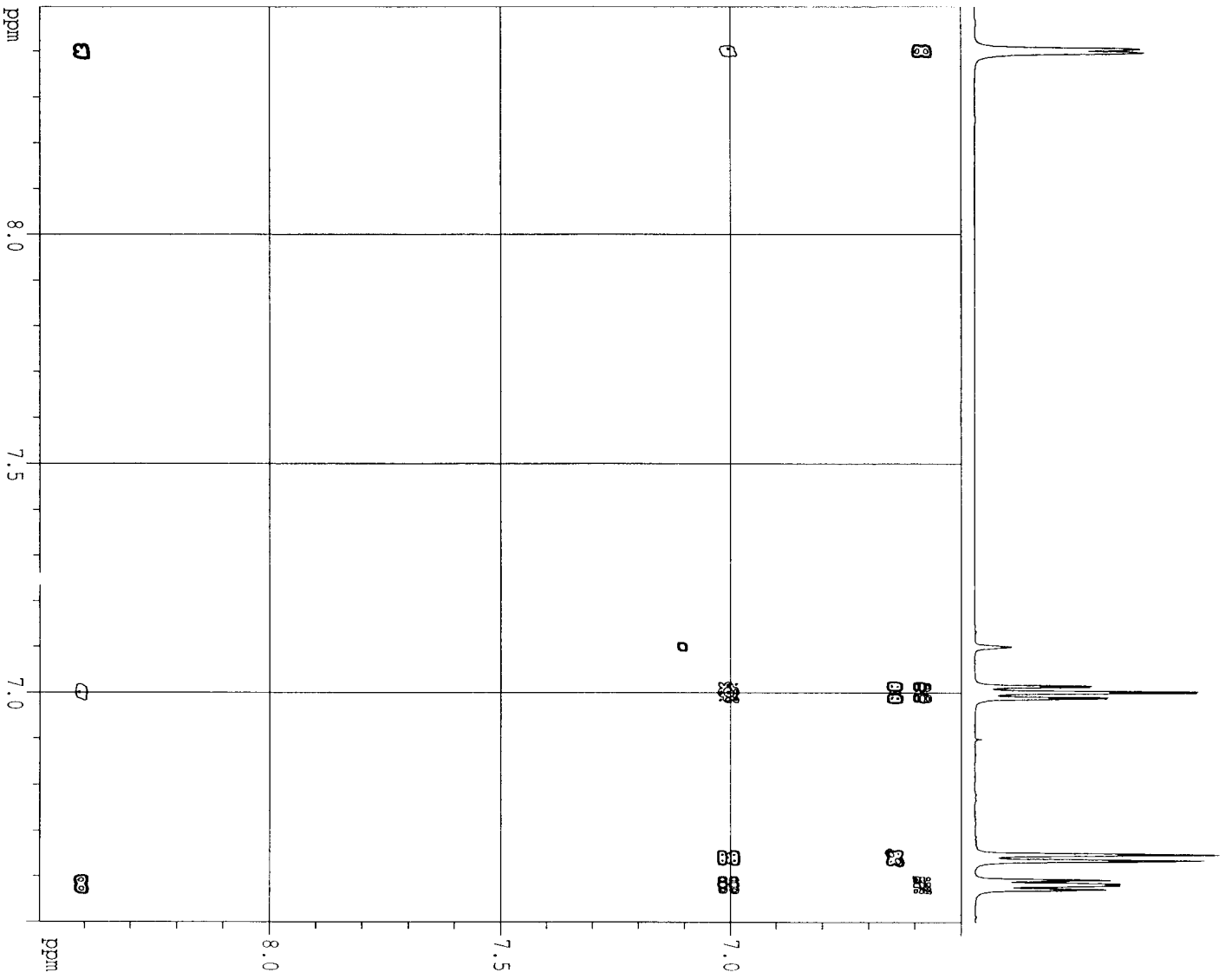
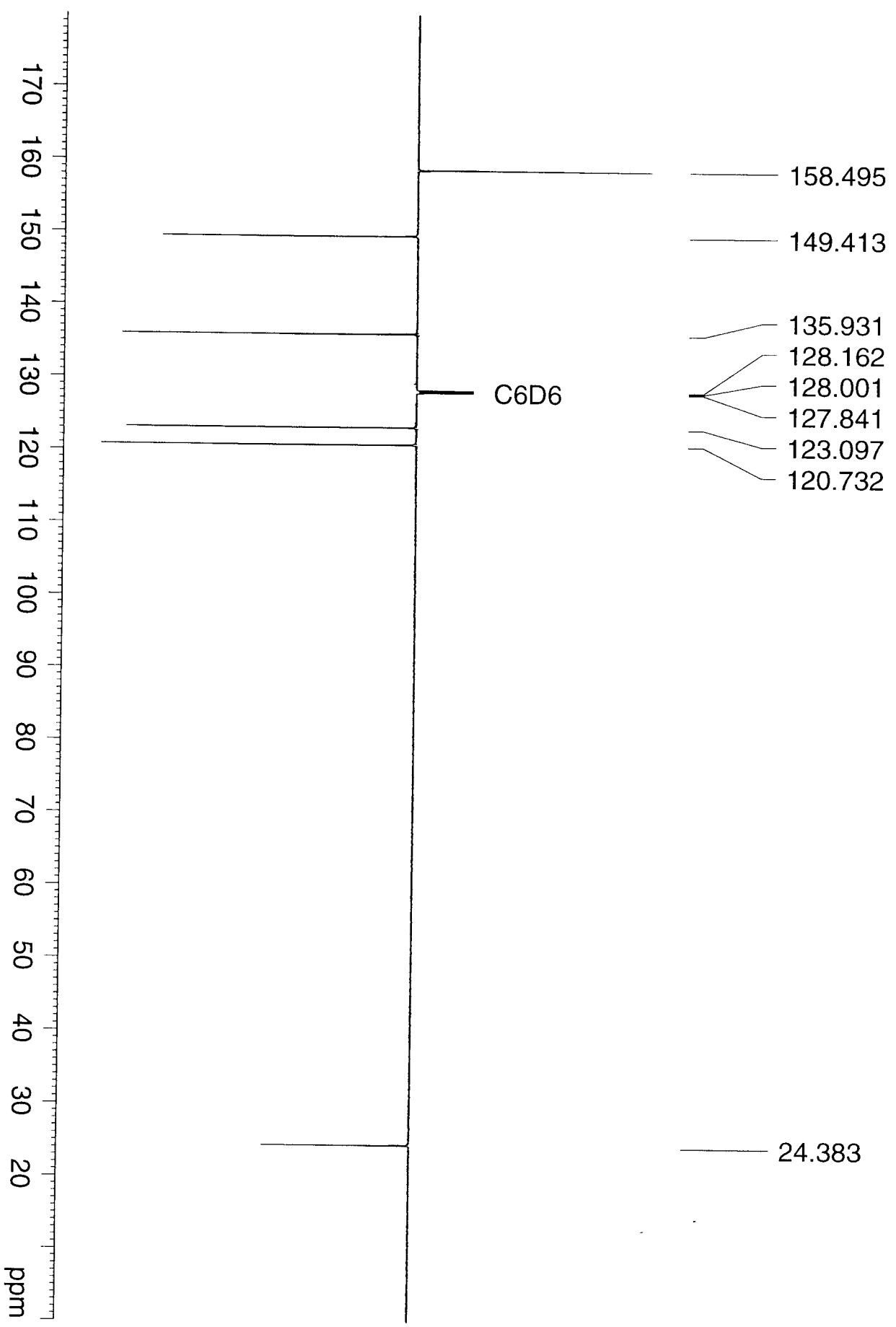


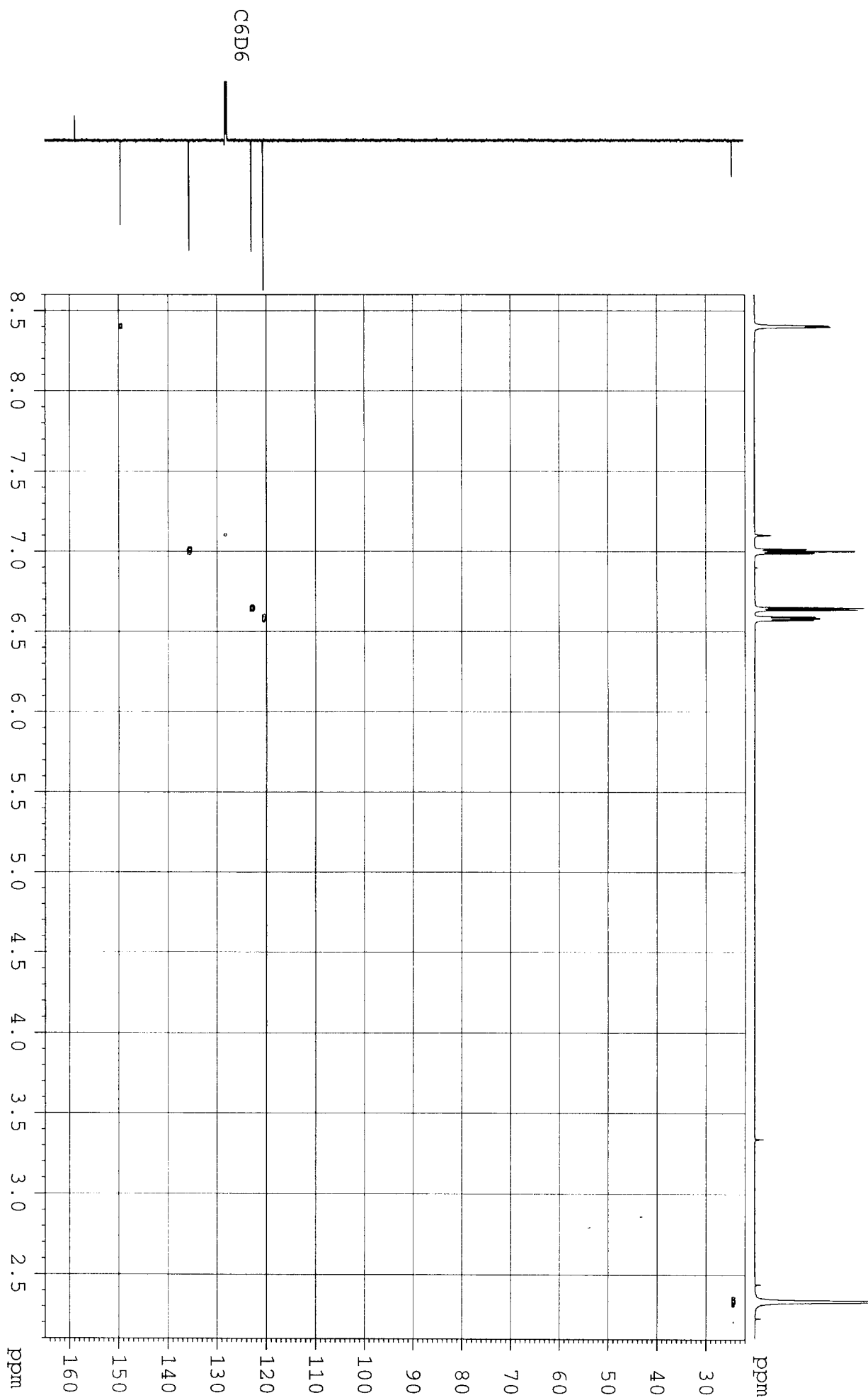
Fig 3C

3D. ¹³C-APT NMR van C₆H₇N in C₆D₆, 150 MHz, δ_{max}-600



3E CH-HMQC van C₆H₇N in C₆D₆, dmx-600

Fig. 3E



3F. 13C-13C INADEQUATE van C6H7N, in C6D6, $\delta_{\text{max}} 600$

C6D6

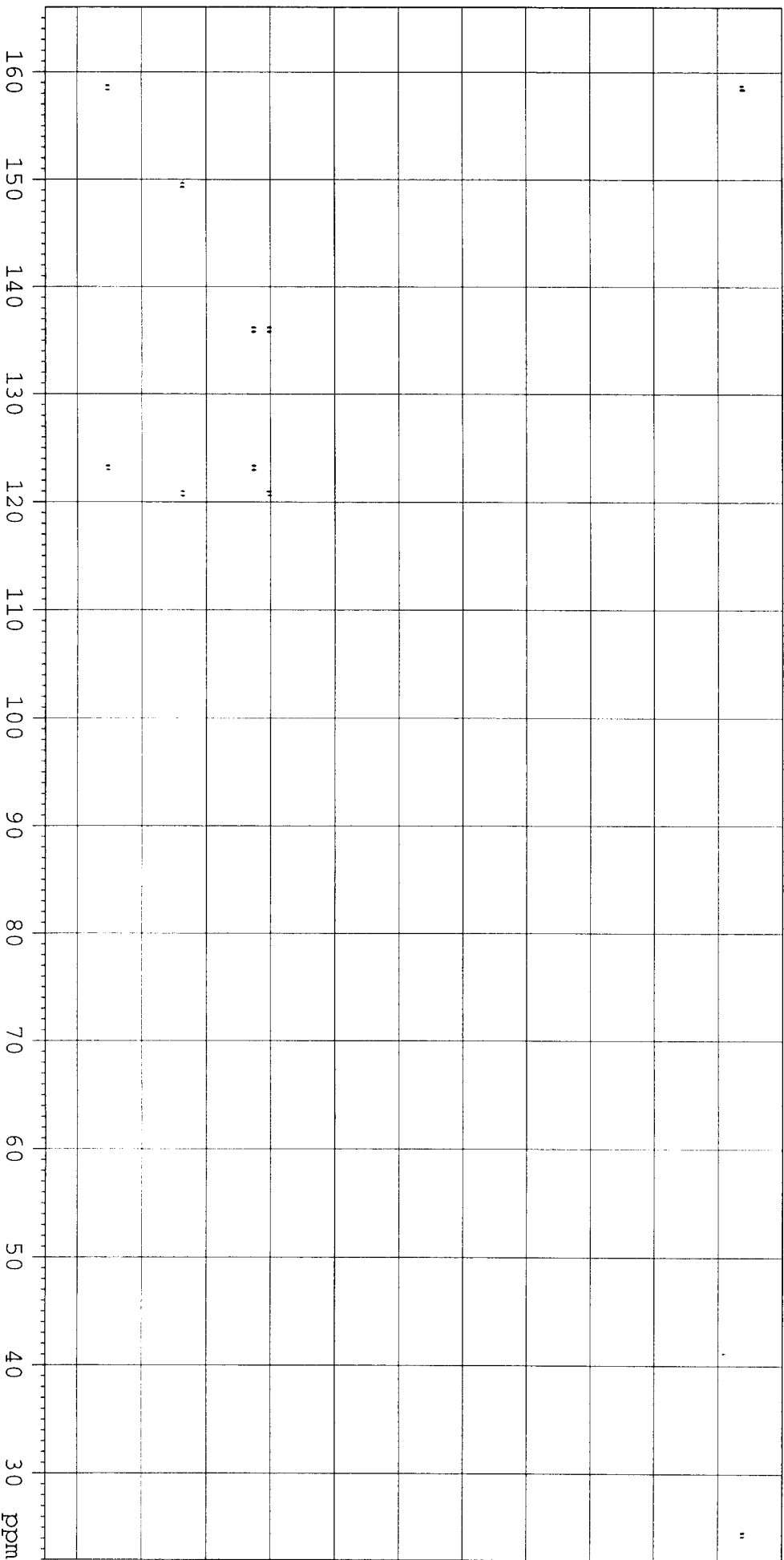
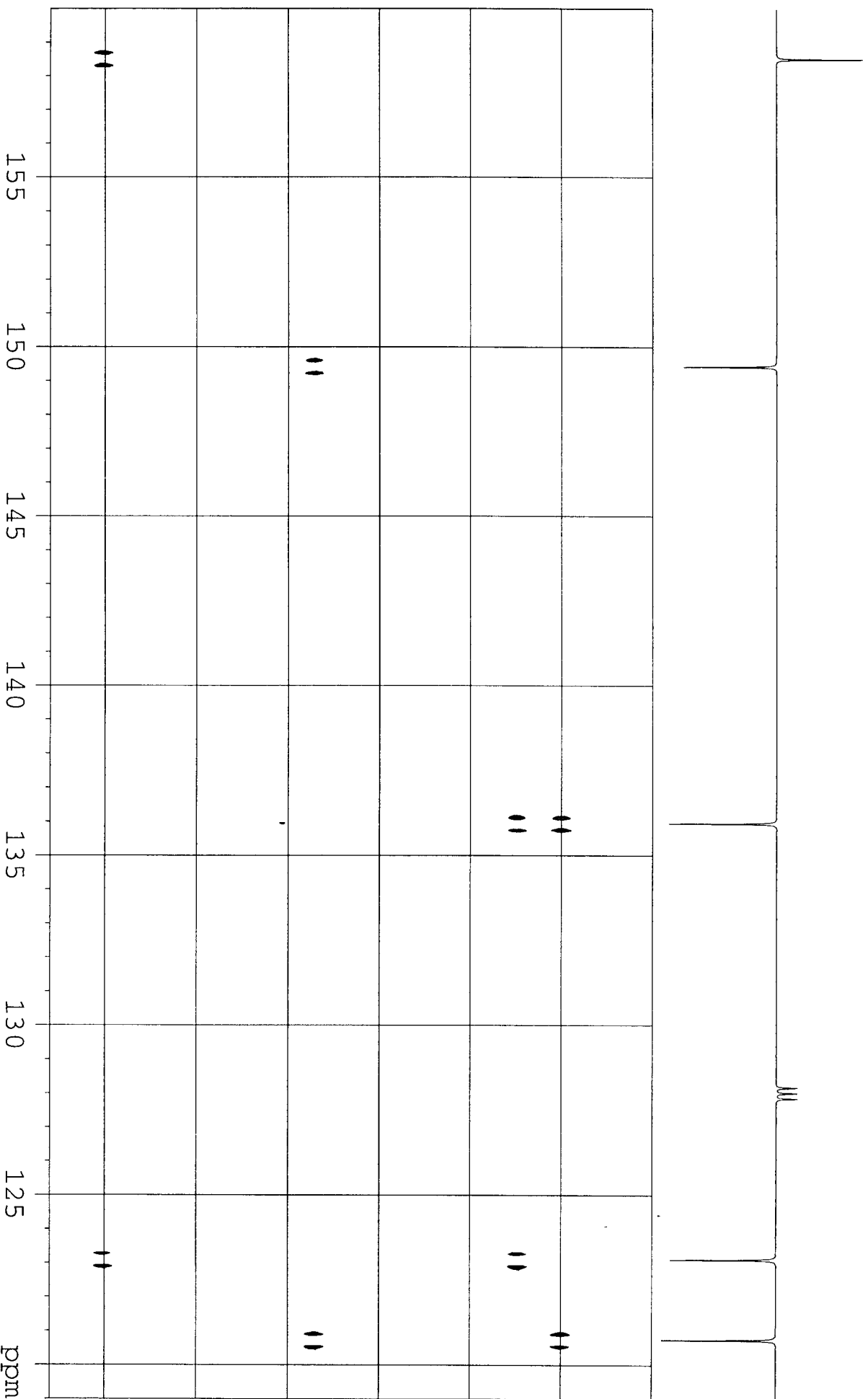


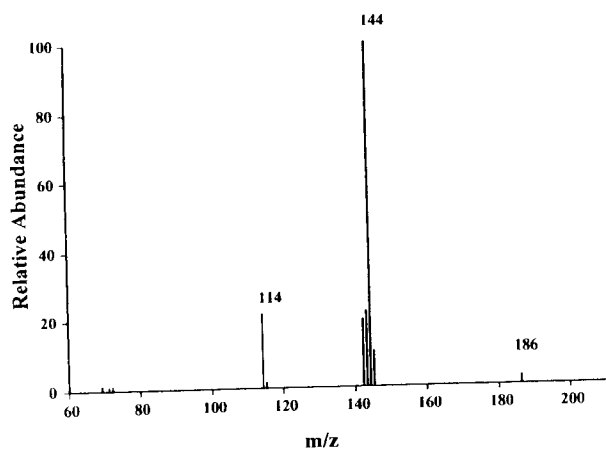
Fig. 1

3G. Vergroting van aromatisch gebied van ^{13}C - ^{13}C INADEQUATE van $\text{C}_6\text{H}_7\text{N}$, in C_6D_6 , dmx600

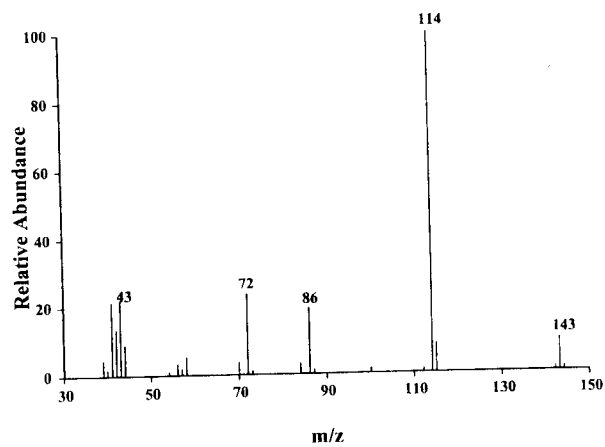
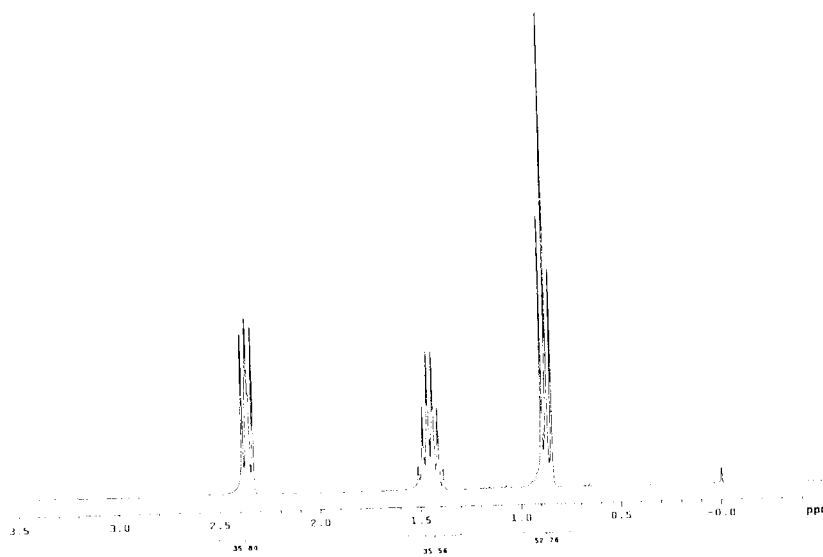


PROBLEM 16-3

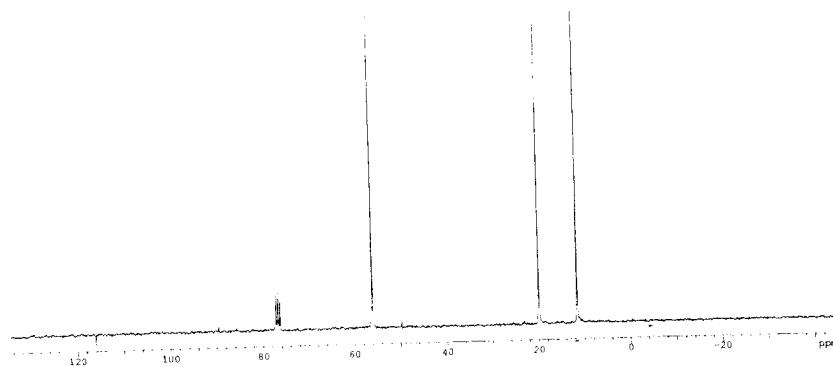
Mass spectrum (CI)



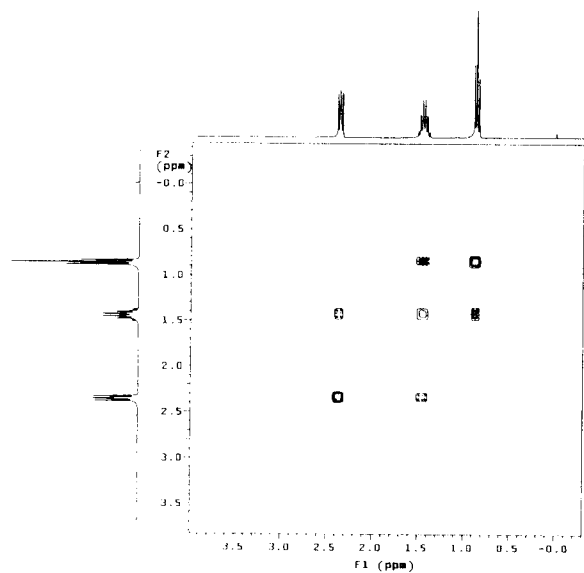
Mass spectrum (EI)

Proton NMR spectrum (CDCl₃)

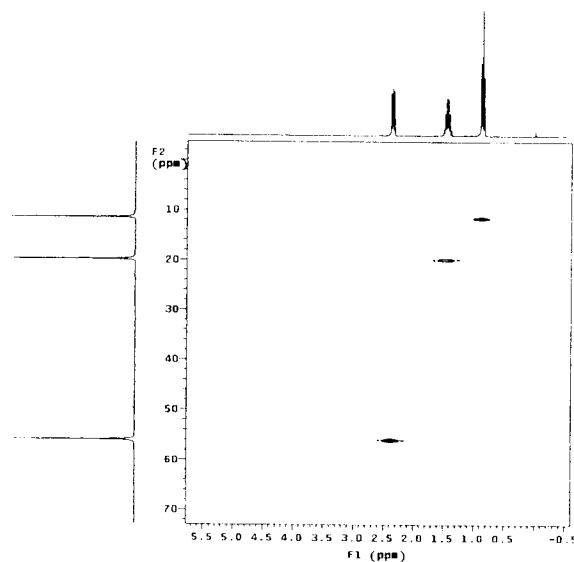
Carbon-13 NMR spectrum



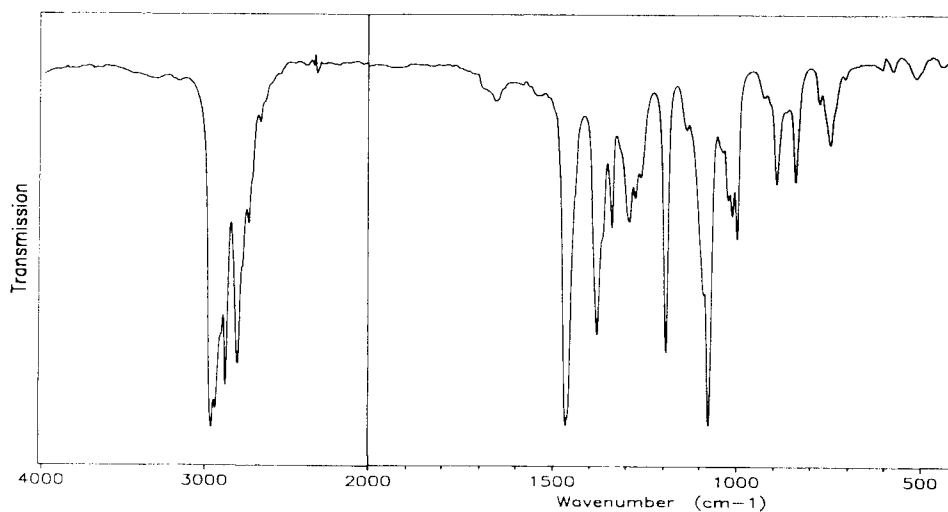
COSY spectrum



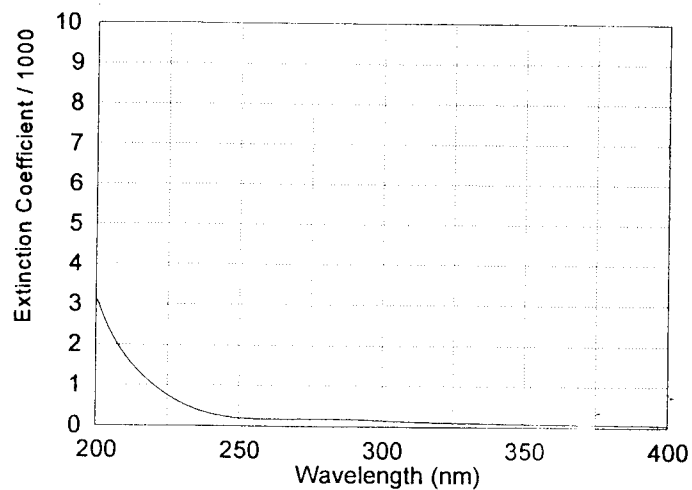
HETCOR spectrum



Infrared spectrum (neat)

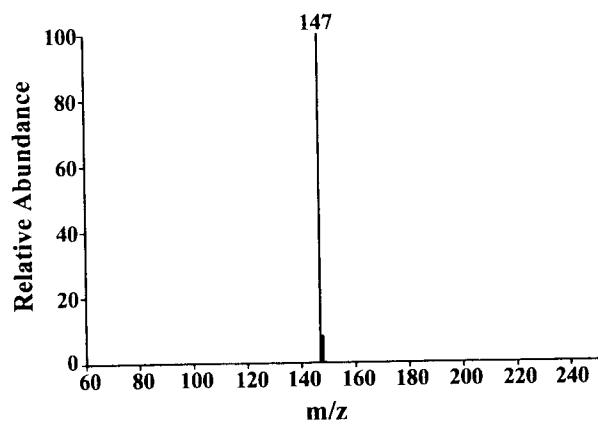


Ultraviolet-visible spectrum (EtOH)

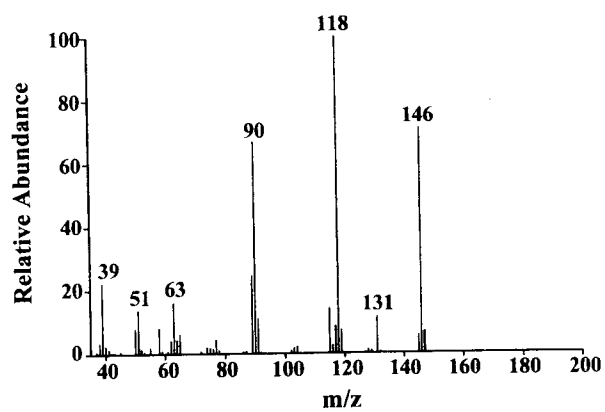
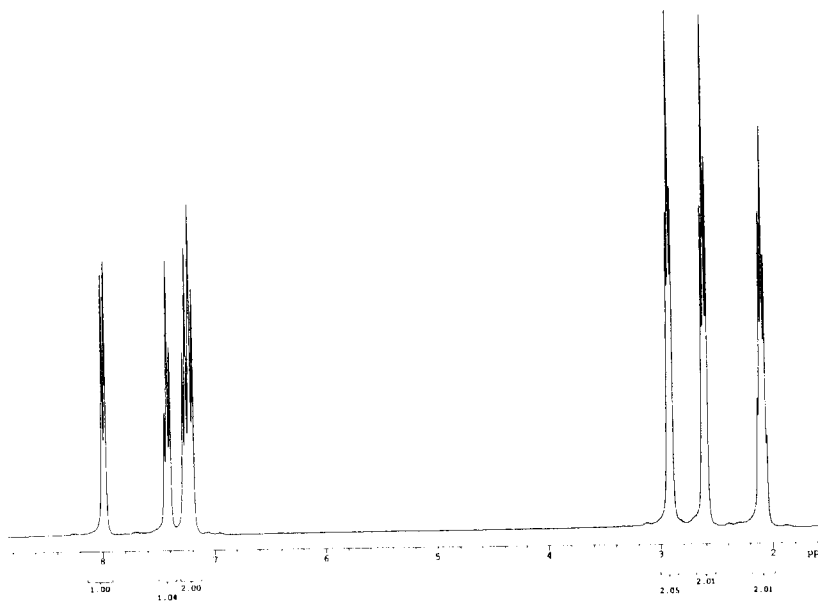


PROBLEM 16-28

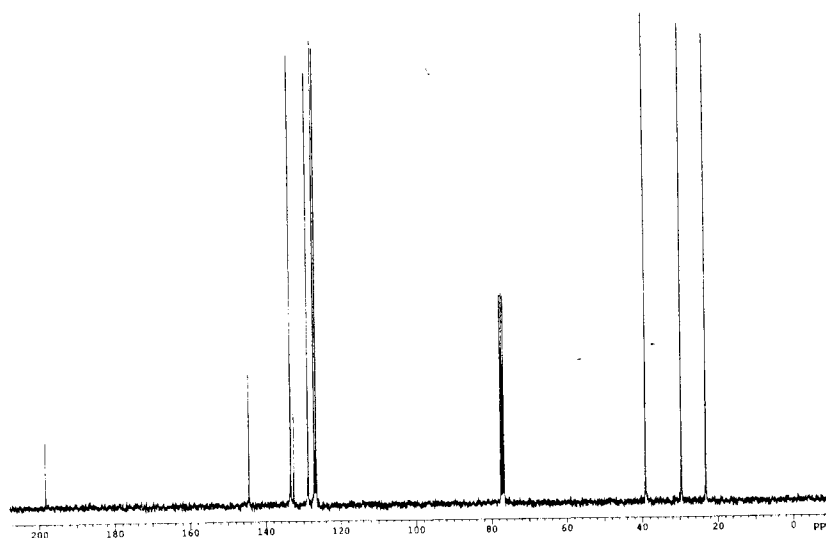
Mass spectrum (CI)



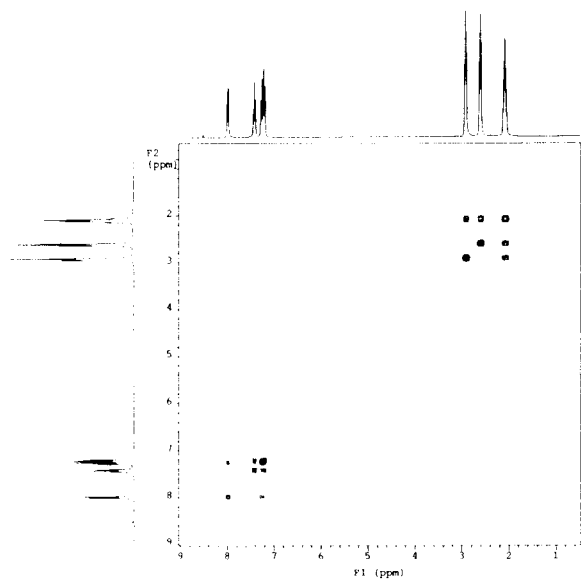
Mass spectrum (EI)

Proton NMR spectrum (CDCl_3)

Carbon-13 NMR spectrum



COSY spectrum



HETCOR spectrum

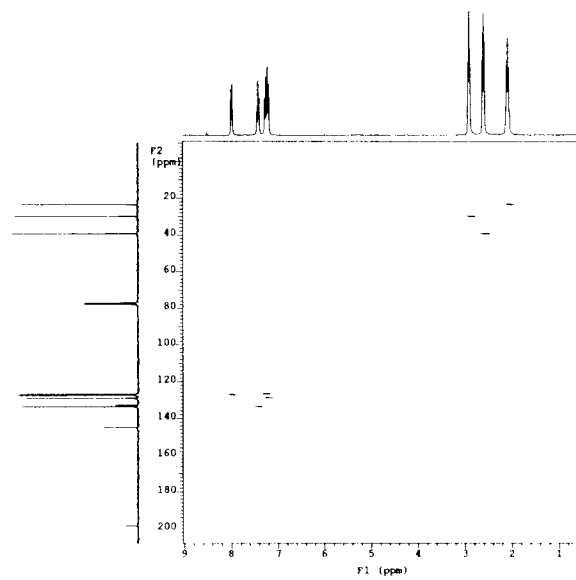
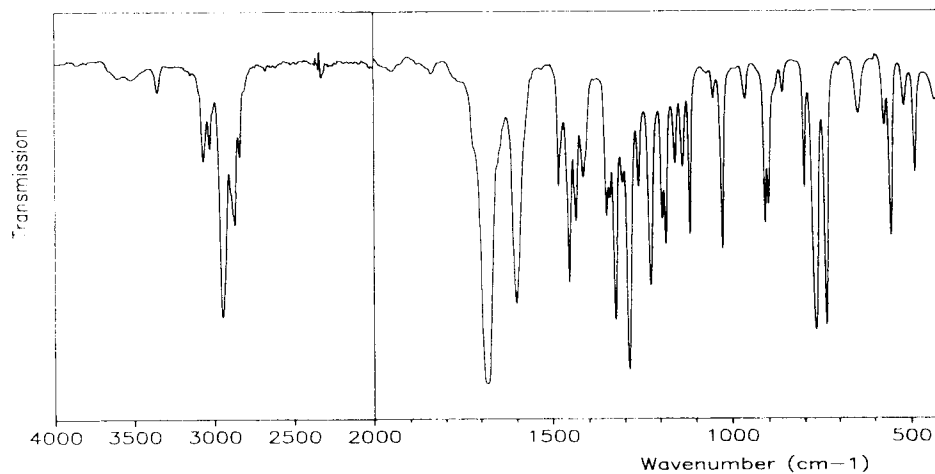


Fig. 5b

Infrared spectrum (neat)



Ultraviolet-visible spectrum (EtOH, ε[206] 24,500, ε[248] 12,200, ε[291] 2200)

