

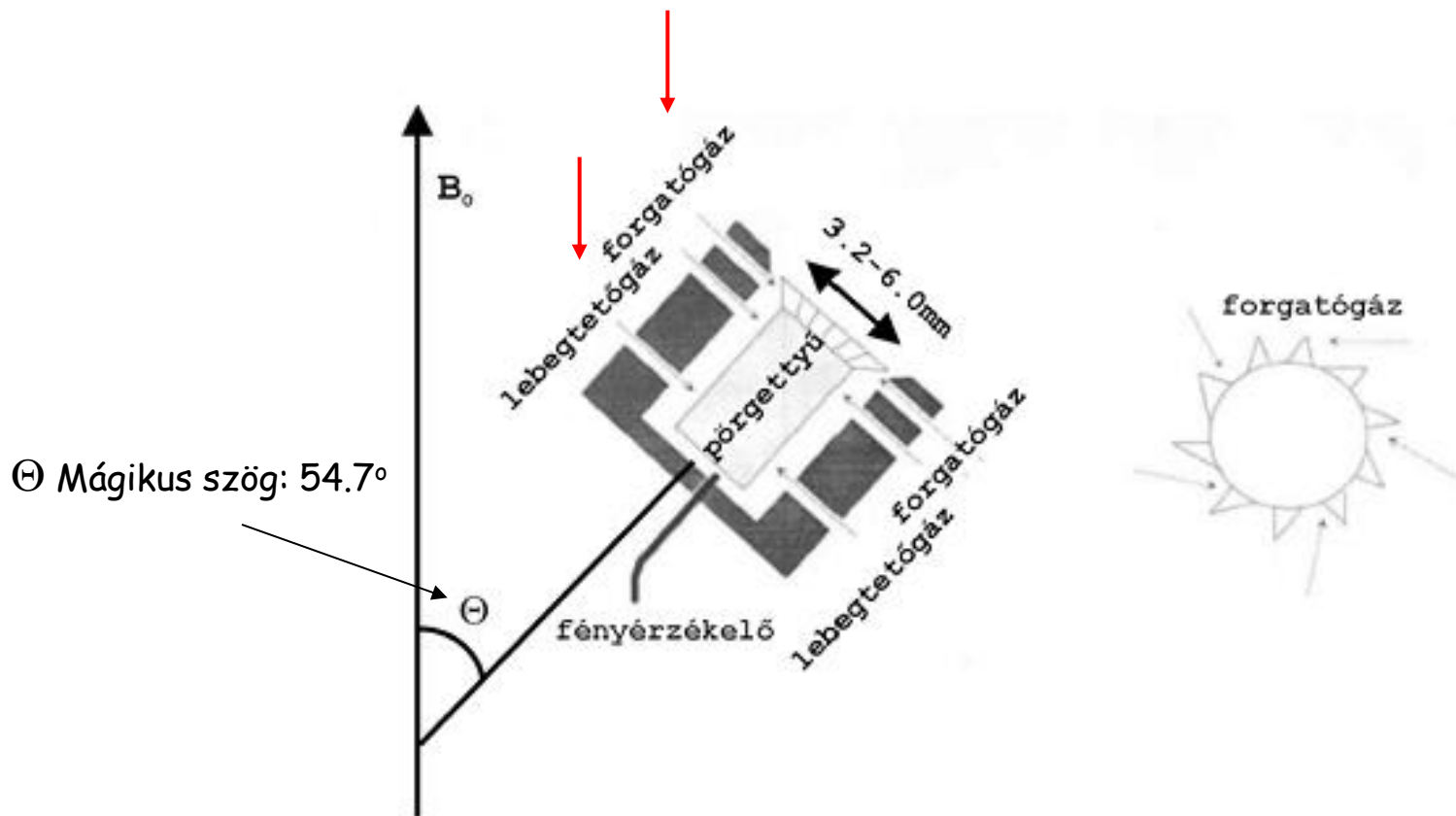
NMR szilárd fázisban

Igen széles jelek, mert a meghatározó kölcsönhatások a „solid state” NMR spektrumokban:

- **dipole-dipole** (homo-, heteronukleáris) csatolódás (direct, through space)
- kémiai eltolódás **anizotrópiája**
- **quadrupolar** kölcsönhatások (csak quadrupolar magoknál)

A kémiai eltolódások irányfüggésének ($\Delta\sigma$ anizotrópia) valamint a téren keresztüli homo- és heteronukleáris dipoláris csatolások jelkiszélesítő hatásának elnyomása

McConnell egyenlet: $\Delta\sigma \propto (\chi_{\parallel} - \chi_{\perp}) (1 - 3\cos^2 \Theta) / R^3$; χ : mágneses szuszceptibilitás



A cirkónia mintatartó (rotor) elhelyezkedése a szilárdfázisú mérőfejben

Minta bevétel/kivétel MAS mérőfejbe

Mintatartó (rotor)

átmérő max. forgási sebesség

1.3 mm: 67 kHz

2.5 mm: 35 kHz

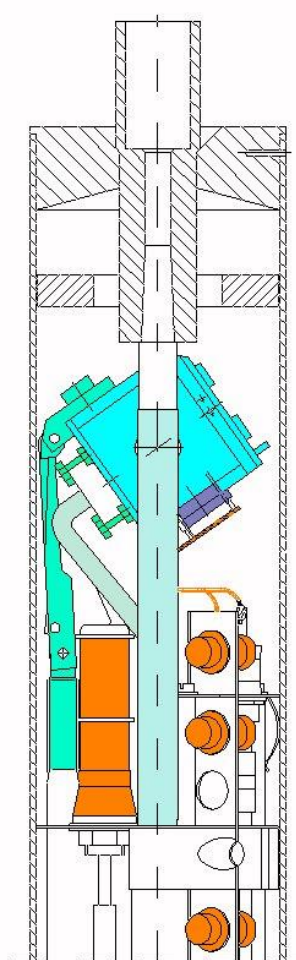
3.2 mm: 24 kHz

4.0 mm: 15 kHz

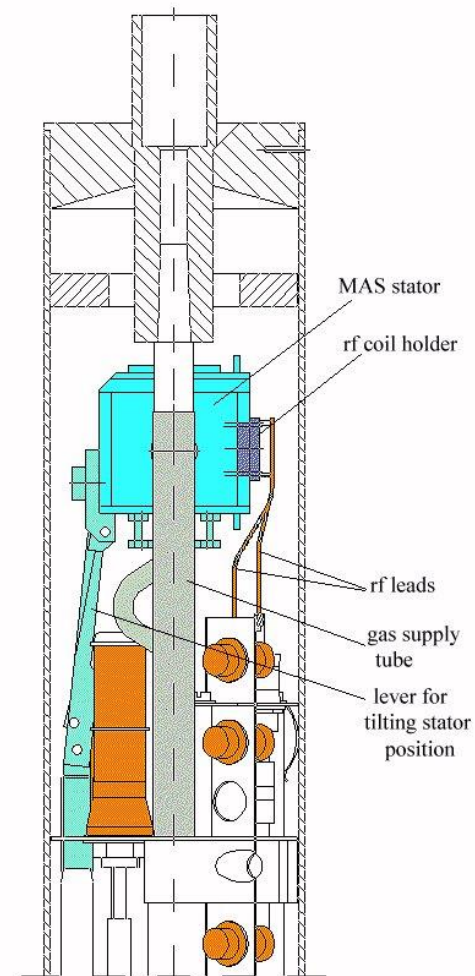
7.0 mm: 7 kHz



HR MAS probes with sample insert/eject capability

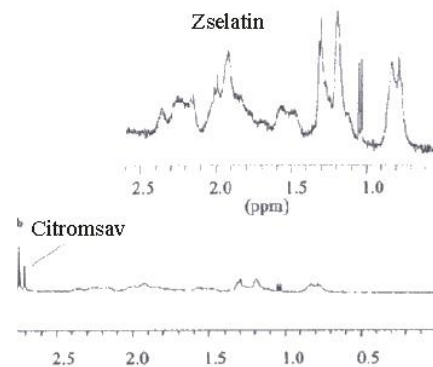
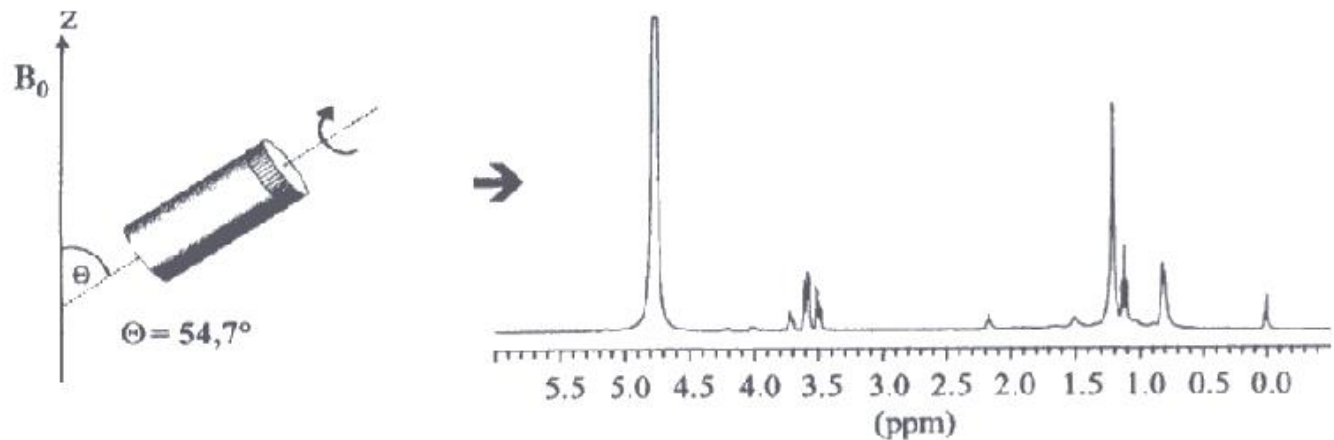
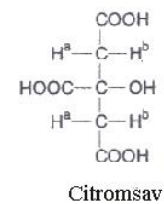
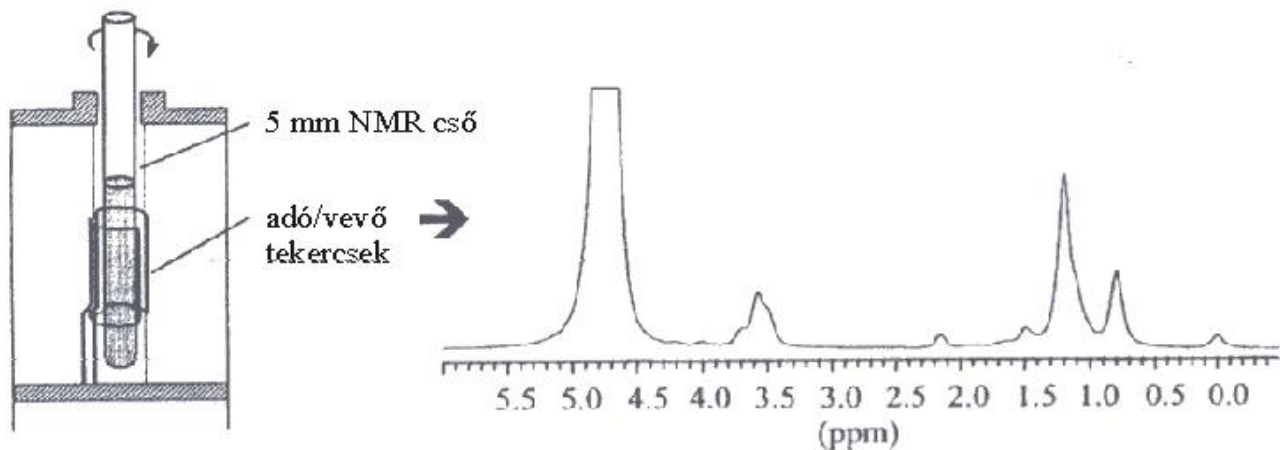


MAS stator in magic angle position



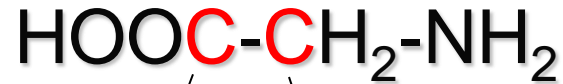
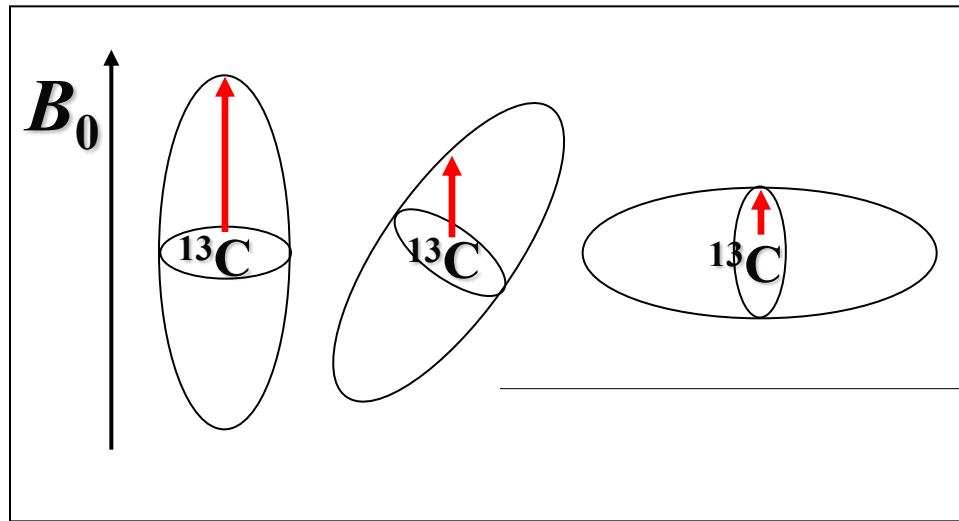
MAS stator in vertical position

HR-MAS-NMR Spektroszkópia (High-Resolution-Magic-Angle-Spinning)

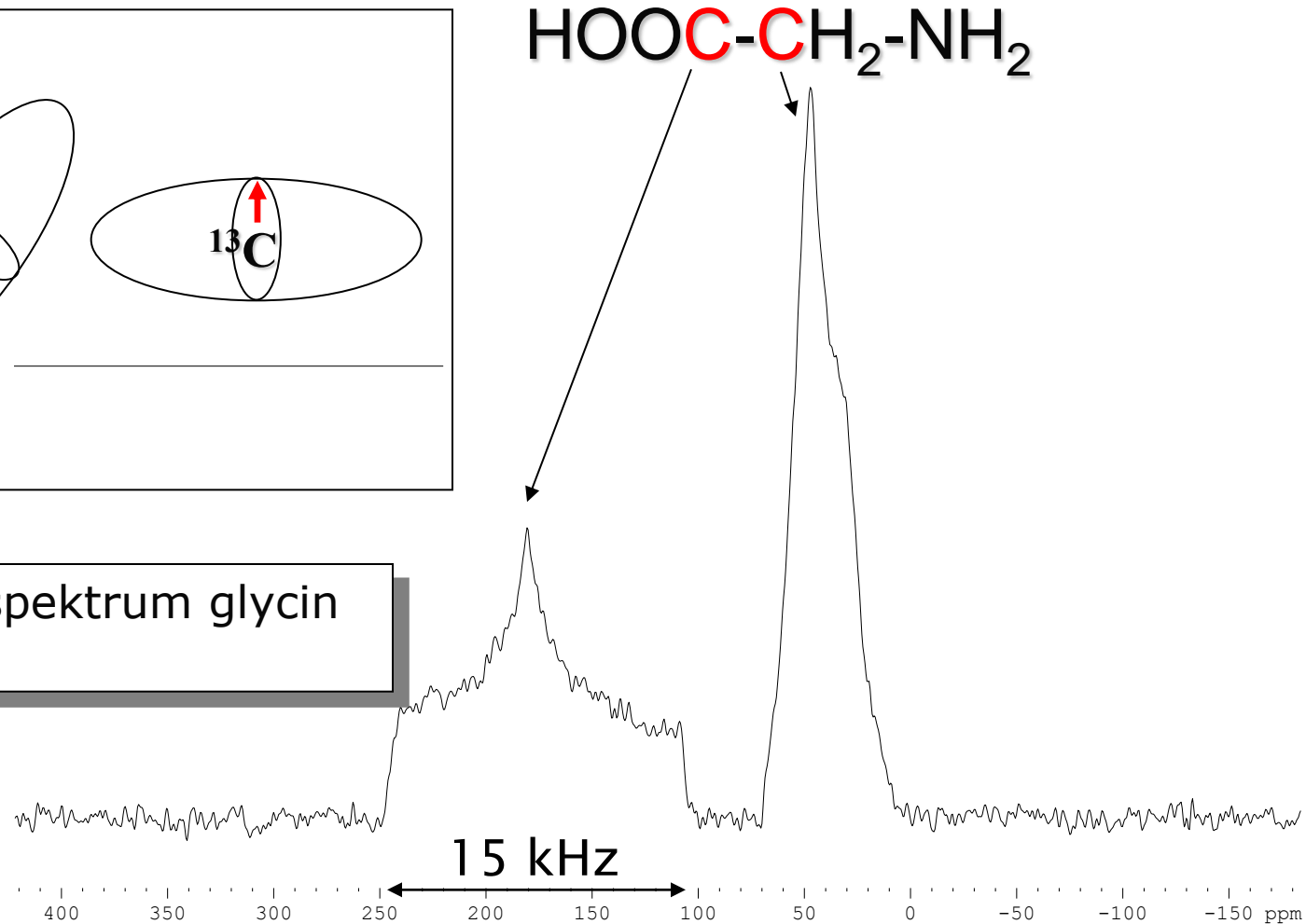


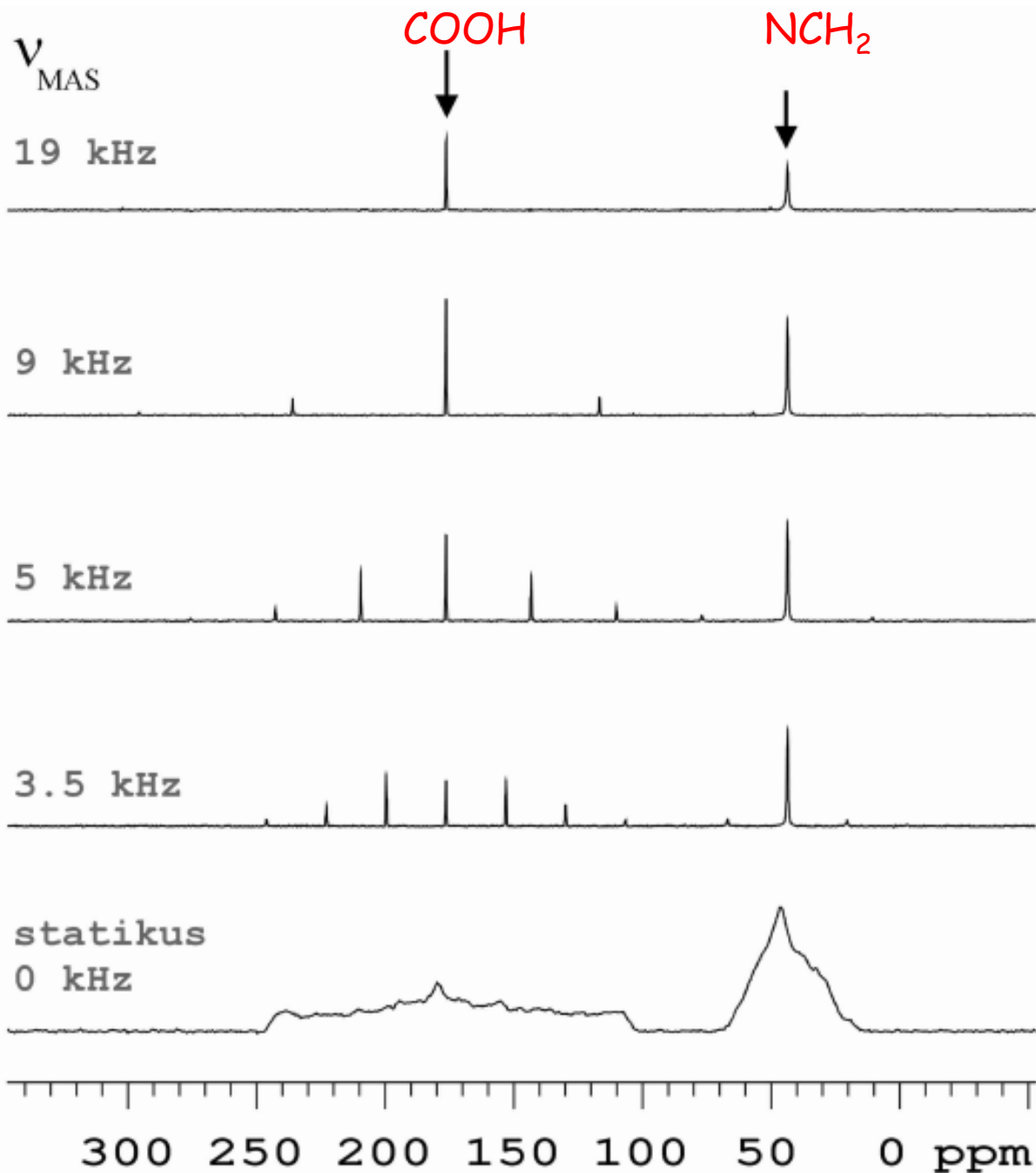
Chemical Shift Anisotropy

Nagy kémiai eltolódás tartományú magok, spin 1/2



static 400 MHz ^{13}C spektrum glycin
(por, ^1H lecsatolt)





A **MAS** forgatás hatása a glicin ($\text{H}_2\text{N}-\text{CH}_2-\text{COOH}$) keresztpolarizációval (CP) és protonlecsatolással felvett szilárdfázisú ^{13}C NMR spektrumára

A forgatási sebesség nagyobb legyen mint az átlagolni kívánt kölcsönhatás.

Álló minta.

$^1\text{H} \rightarrow ^{13}\text{C}$ mágnesezettség átvitel keresztpolarizációval (Cross Polarisation)

(Boltzmann)

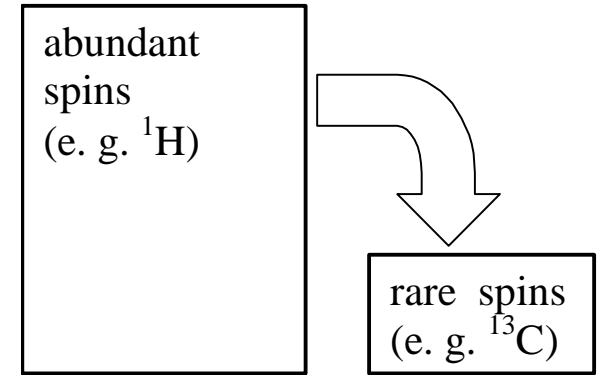
$$\frac{N(\text{felső})}{N(\text{alsó})} = e^{\frac{-\Delta E}{kT}} \approx 1 - \frac{\Delta E}{kT} = 1 - \frac{h}{2\pi} \cdot \frac{\gamma B}{kT}$$

Azonos ^1H és ^{13}C betöltöttséghez eltérő (spin)-hőmérséklet tartozik: pl.:

$$e^{\frac{-\gamma_H B_0}{kT_H}} = \frac{p_2}{p_1} = e^{\frac{-\gamma_C B_0}{kT_C}}$$

$$T_C = \frac{\gamma_C}{\gamma_H} T_H \quad T_C = \frac{1}{4} 300\text{K} = 75\text{K}$$

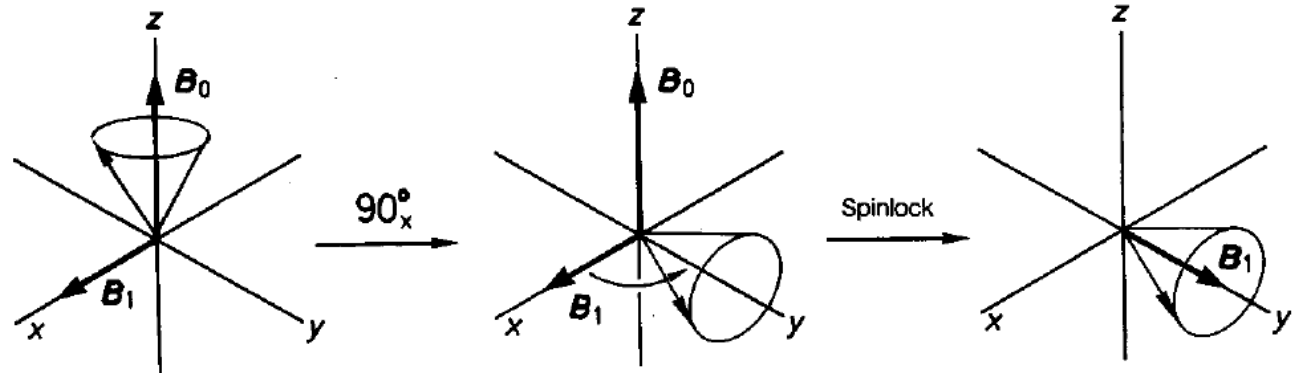
γ : giromágneses hányados, $\gamma_H/\gamma_C = 4/1$
 $\omega = \gamma \cdot B$ $\gamma_H \cdot B \neq \gamma_C \cdot B$



Meleg tartály \rightarrow hideg tartály

Hartmann-Hahn rezonancia feltétel:

$$\gamma_H \cdot B_1(^1\text{H}) = \gamma_C \cdot B_1(^{13}\text{C})$$



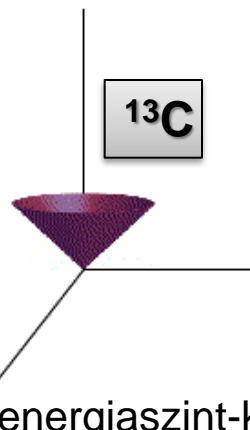
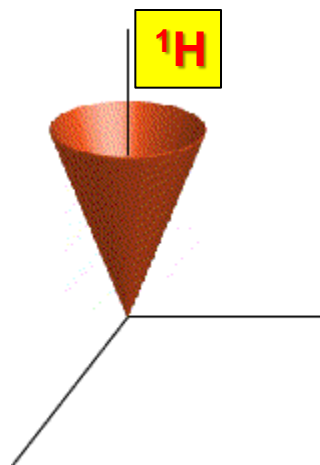
Hartmann-Hahn Spinlock-Experiments

Keresztpolarizáció (CP)

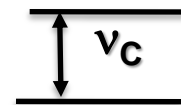
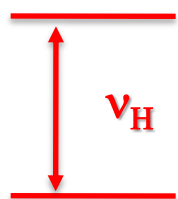
Laboratóriumi koordinátarendszerben

$$\omega = \gamma B_0$$

$$\gamma_H \sim 4 \gamma_C$$



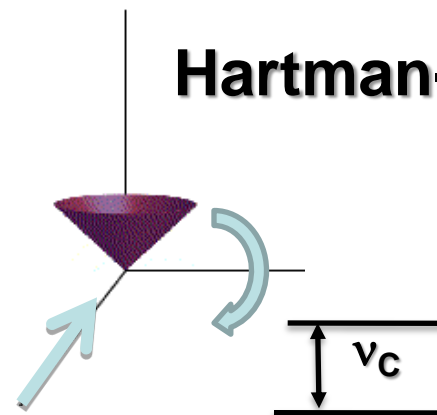
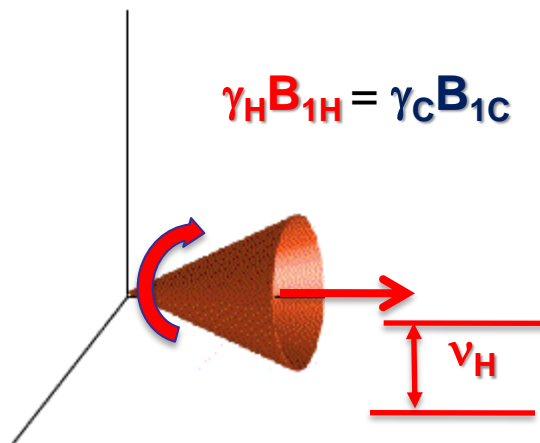
Frekvenciák (energiaszint-különbségek) eltérőek



Forgó koordinátarendszerben

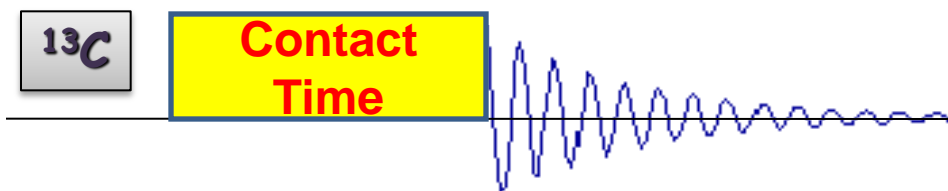
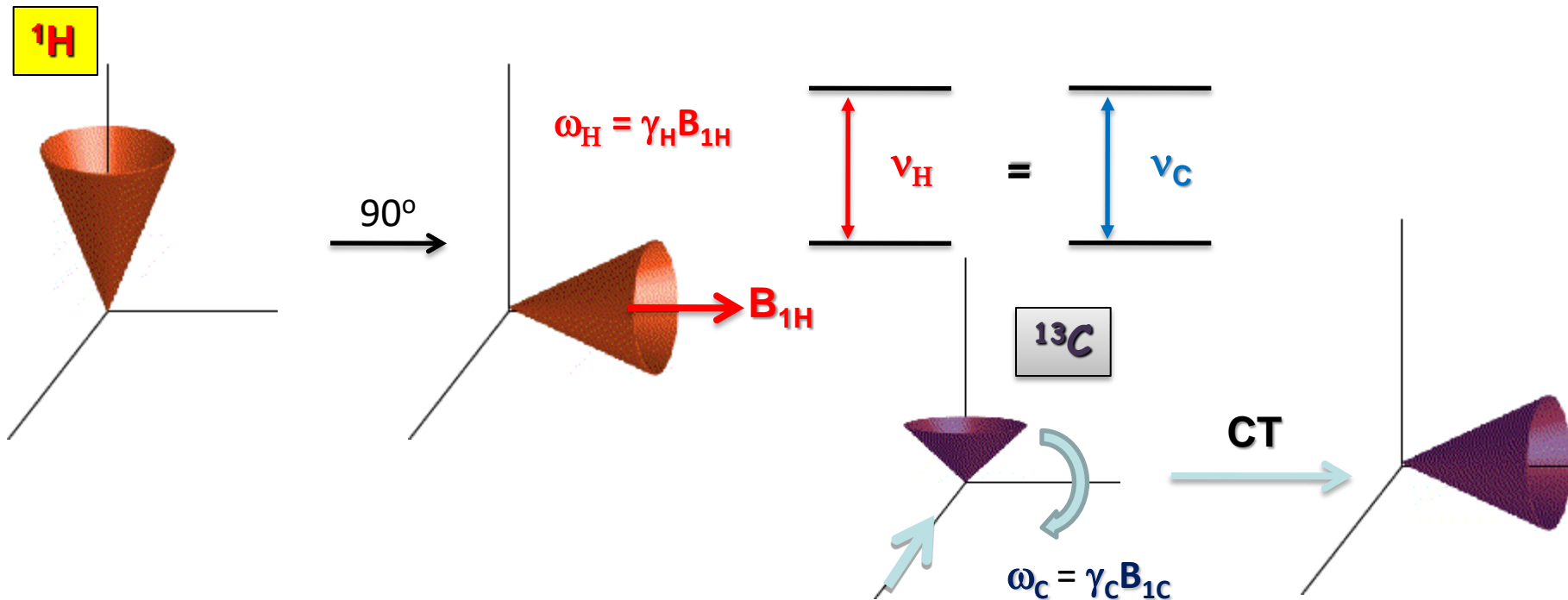
Az energia állításával a ^{13}C rezonanciafrekvenciája az ^1H rezonanciafrekvenciájával egyenlővé tehető

$$\gamma_H B_{1H} = \gamma_C B_{1C}$$



Hartman-Hahn

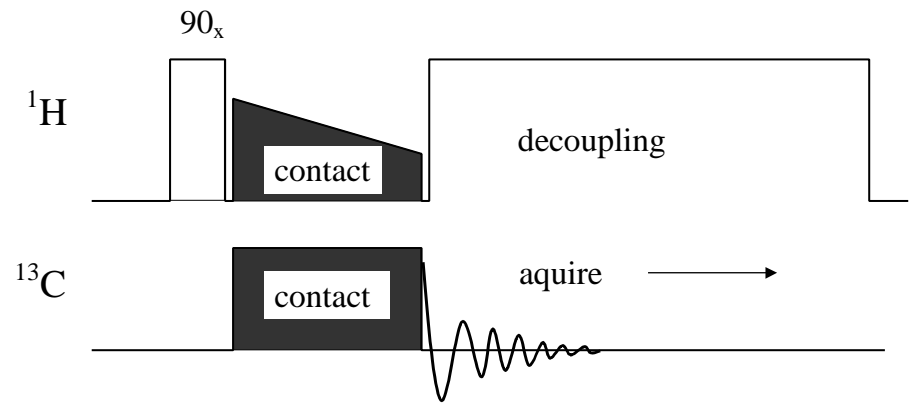
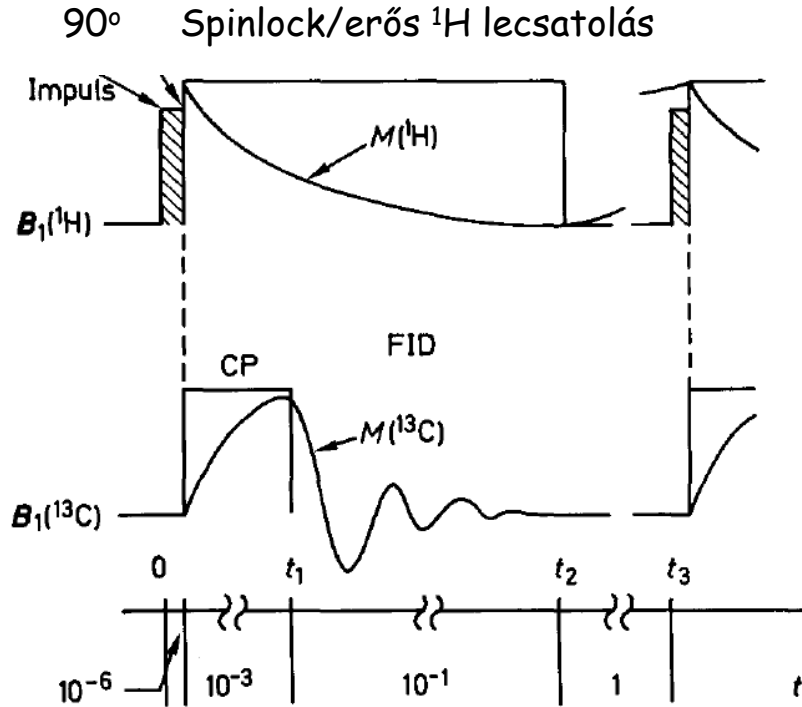
Keresztpolarizáció (CP)



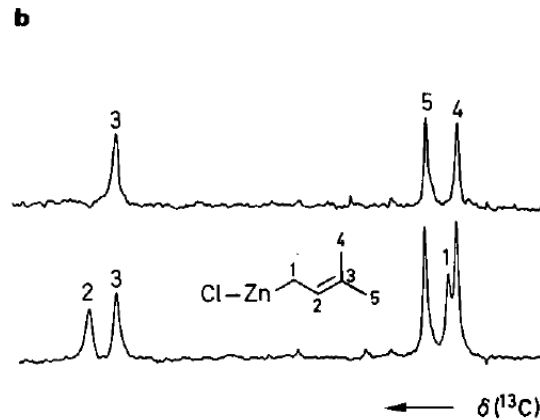
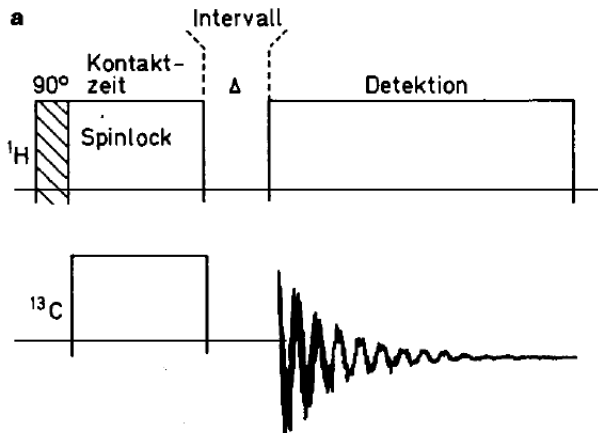
Hartman-Hahn

A keverési idő alatti kölcsönhatás (CT) folytán a ^{13}C mágnesszéttség növekszik

Szilárdfázisú ^{13}C mérés CPMAS technikával

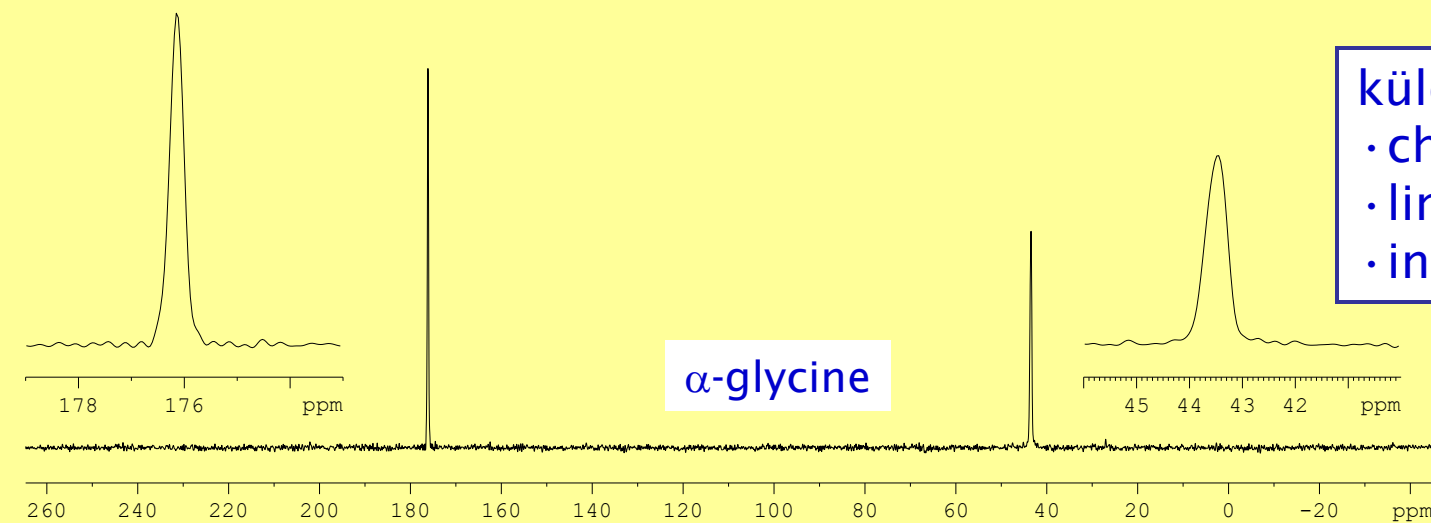
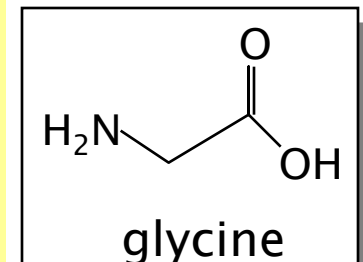


A ^{13}C CPMAS mérés optimalizálása a $\text{CH}_2 + \text{CH} / \text{CH}_3 + \text{C}$ jelek megkülönböztetésére



$\Delta \sim 30-100 \mu\text{s}$

Glycin polimorfok ^{13}C CP/MAS spektruma



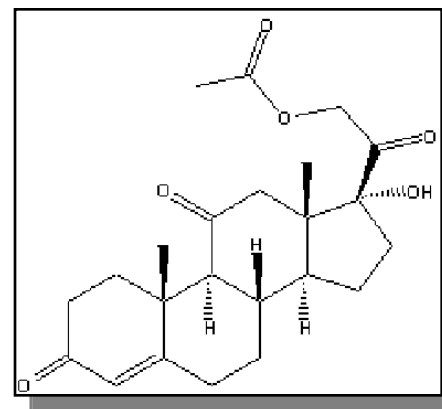
- különbségek:
- chemical shift
 - linewidth
 - interaction with ^{14}N

(spektrumok nem méretarányosak)

400MHz spectrometer, spinning speed 15 kHz
 α - and γ -glycine crystallised from water or 5% aqueous acetic acid, resp.

High Resolution NMR in the Solid State

^{13}C CPMAS spectra of Cortisone acetate,
500 MHz; $\text{C}_{23}\text{H}_{30}\text{O}_6$



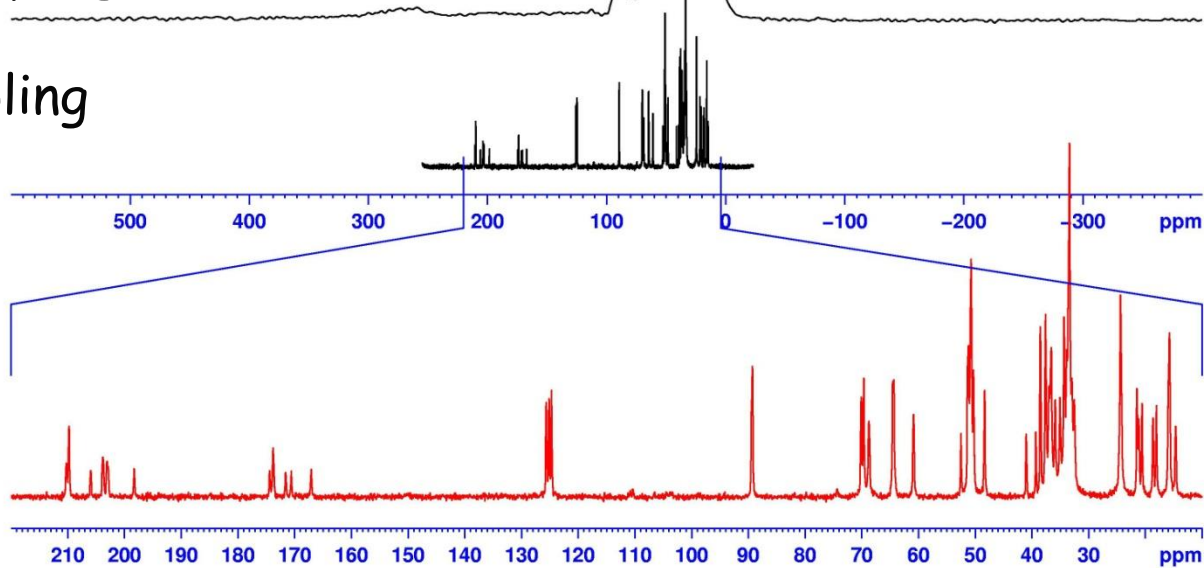
Static no ^1H decoupling



Static with ^1H decoupling

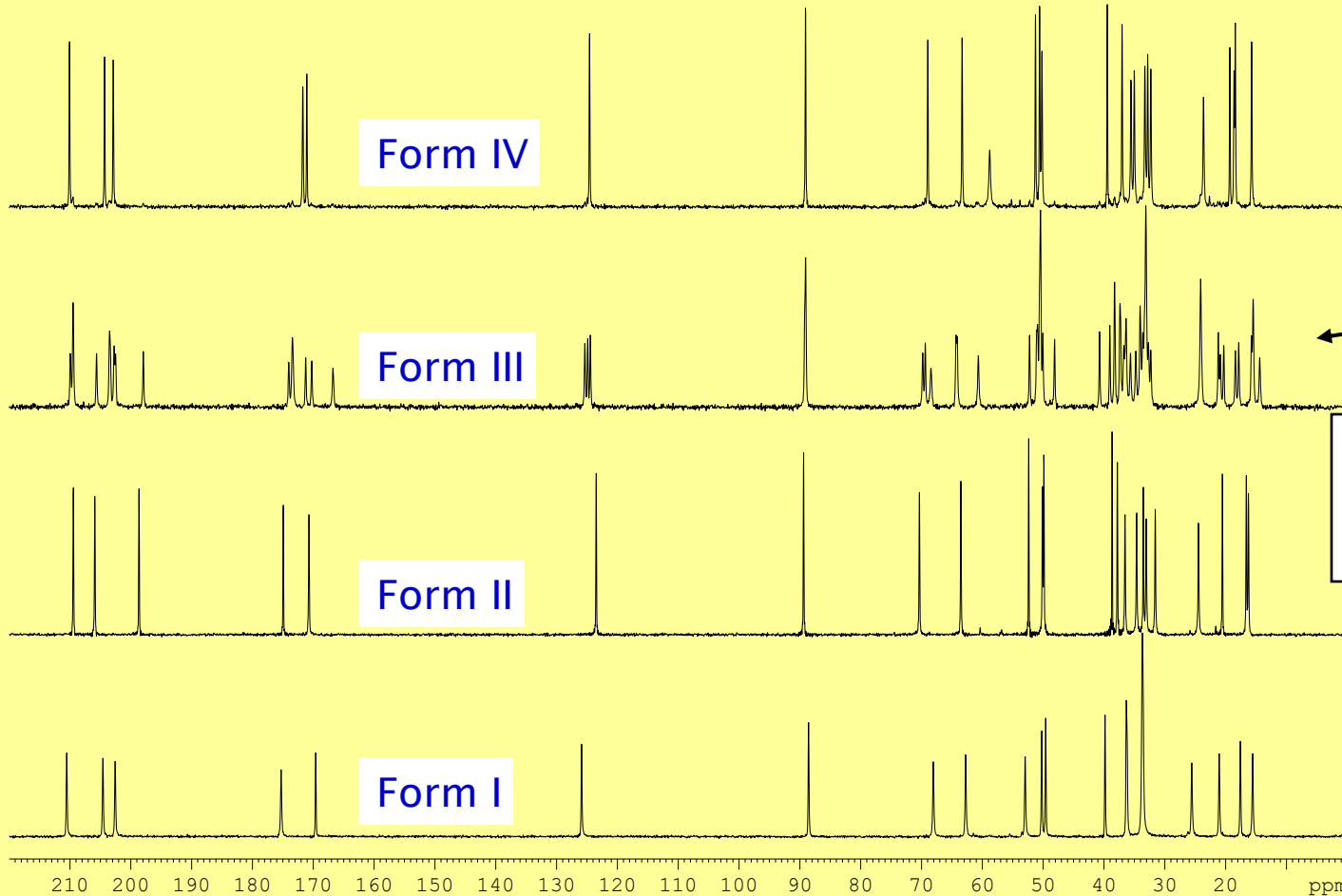


MAS with ^1H decoupling



Cortisone Acetate: Discrimination of Polymorphs

^{13}C CP/MAS spectra of cortisone acetate

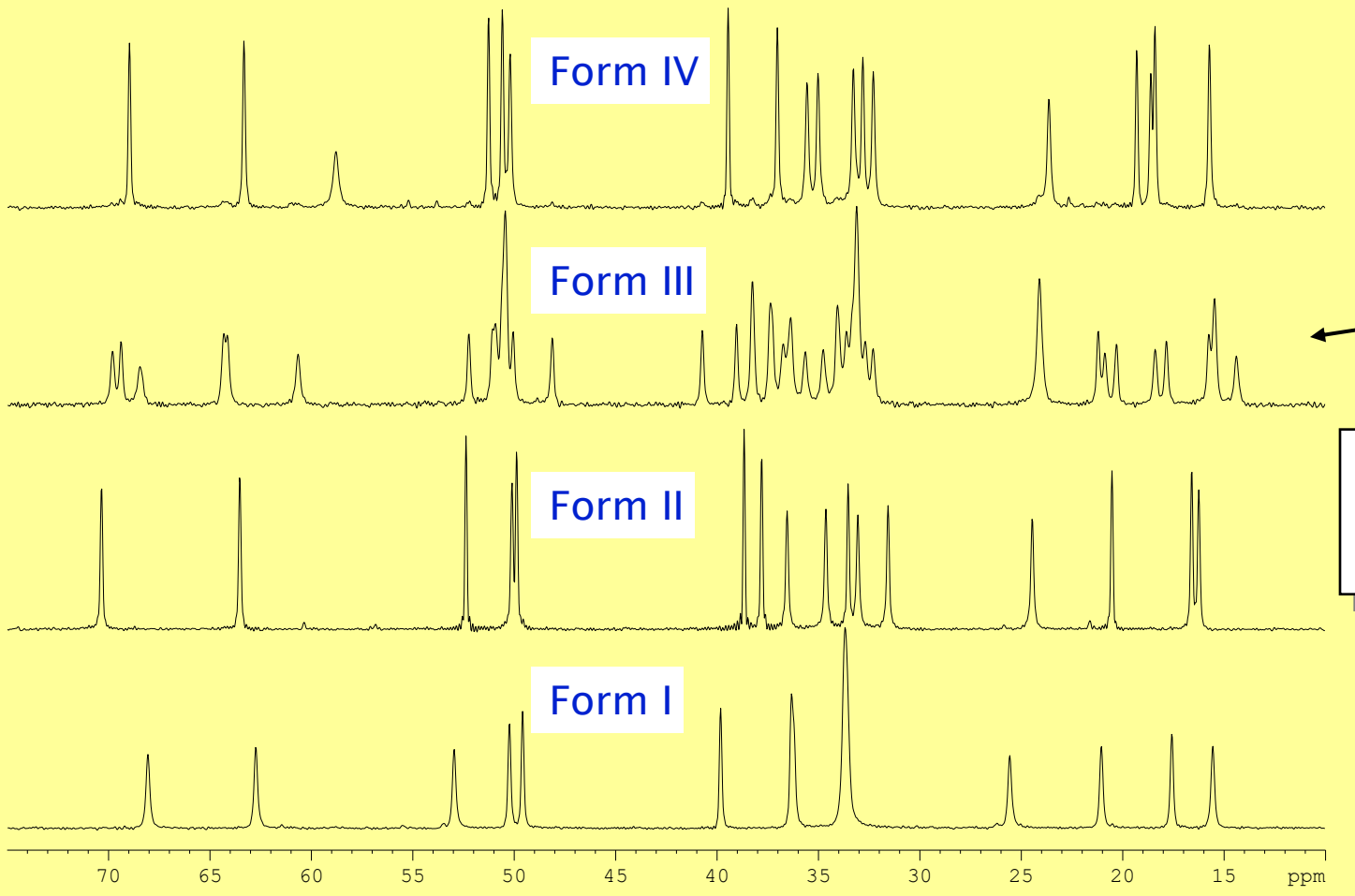


doubling of lines:
2 non-equivalent
lattice sites

400 MHz spectrometer, spinning speed 15 kHz, measurement time \approx 40 min

Cortisone Acetate: Discrimination of Polymorphs

chemical shifts differ for all 4 forms

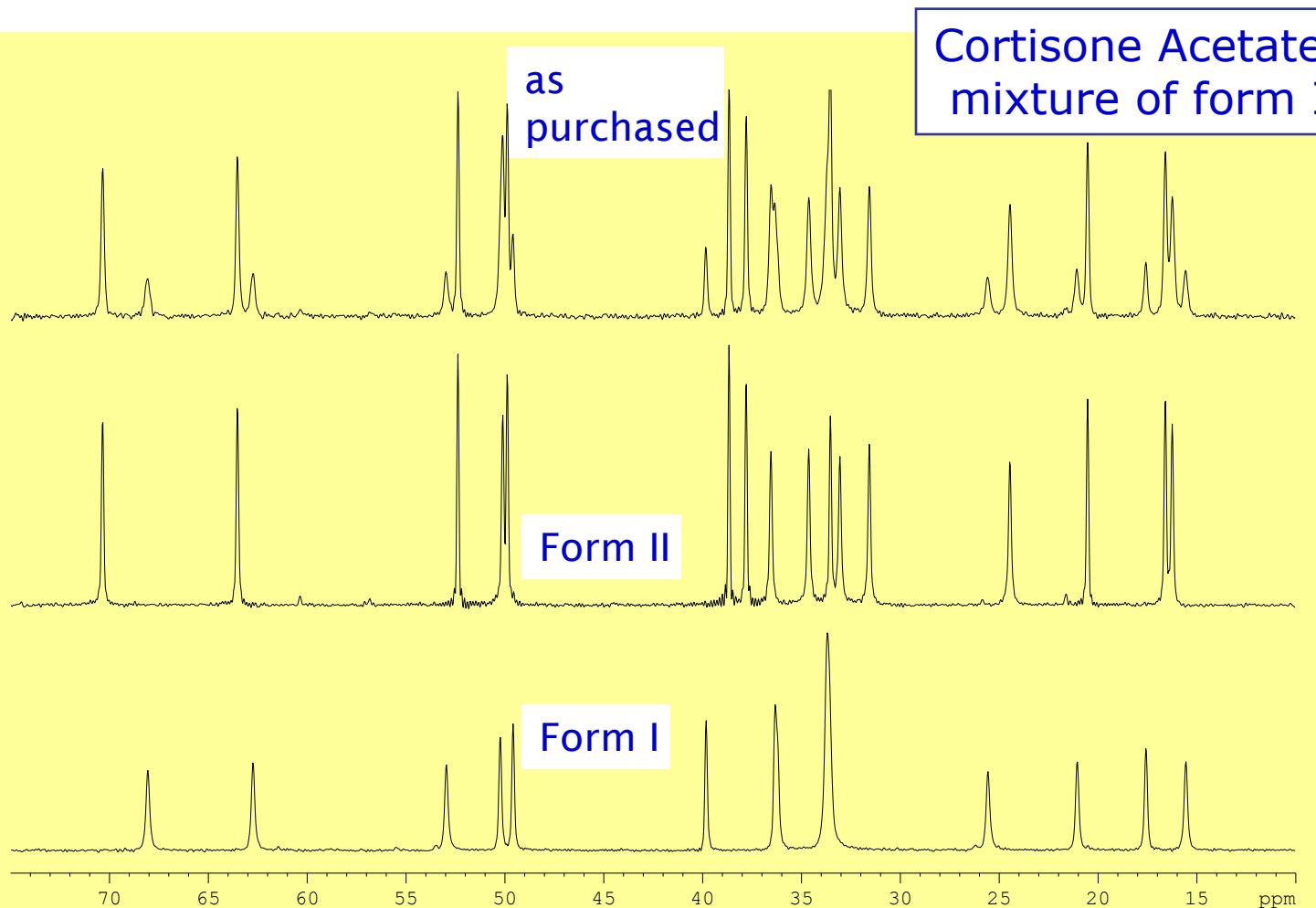


doubling of lines:
2 non-equivalent
lattice sites

(spectra not
drawn to scale)

¹³C CP/MAS spectra, expanded view on 75 - 10 ppm region

Cortisone Acetate: Identification of Polymorphs



Cortisone Acetate as purchased:
mixture of form I and II

as
purchased

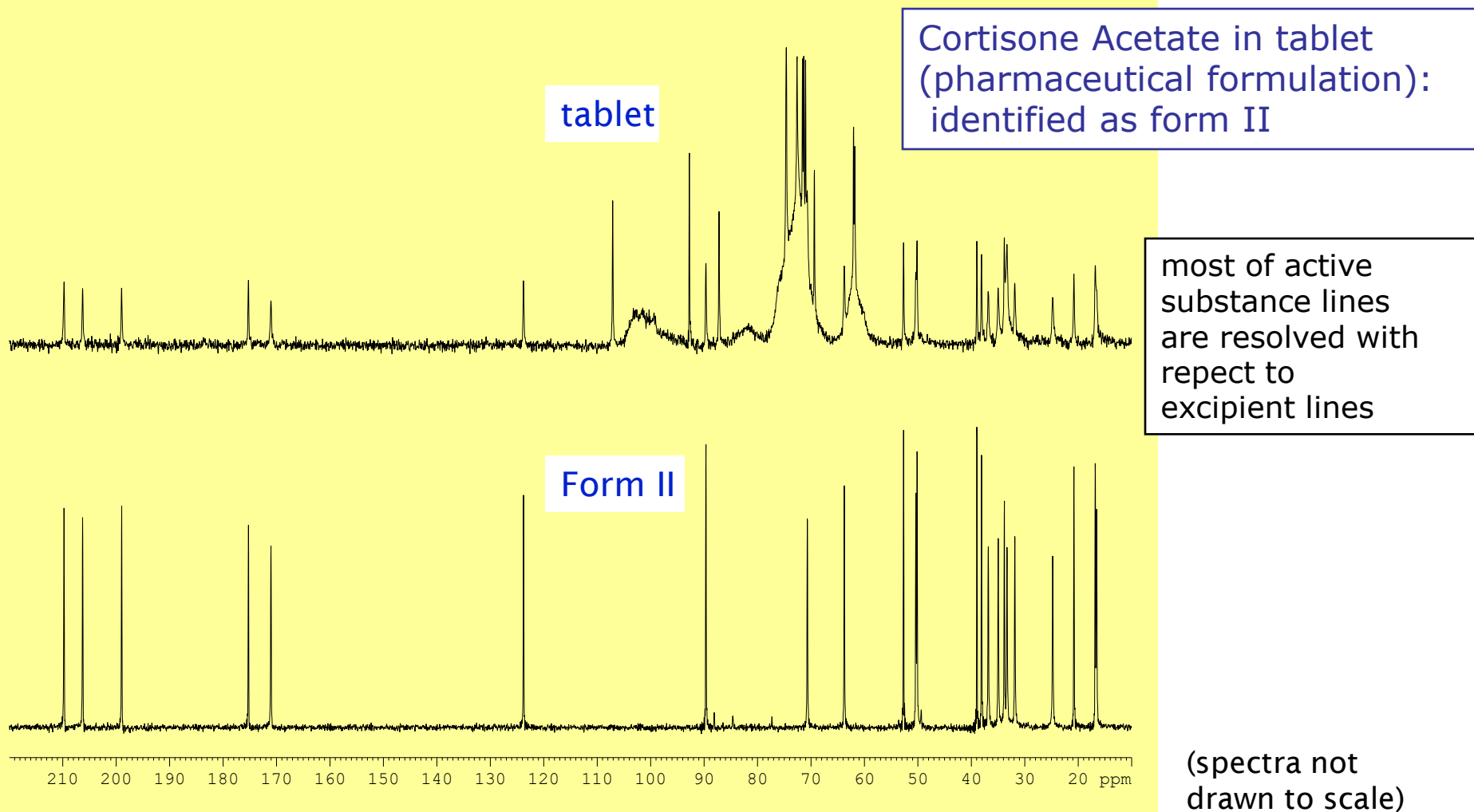
Form II

Form I

(spectra not
drawn to scale)

^{13}C CP/MAS spectra, expanded view of 75 - 10 ppm

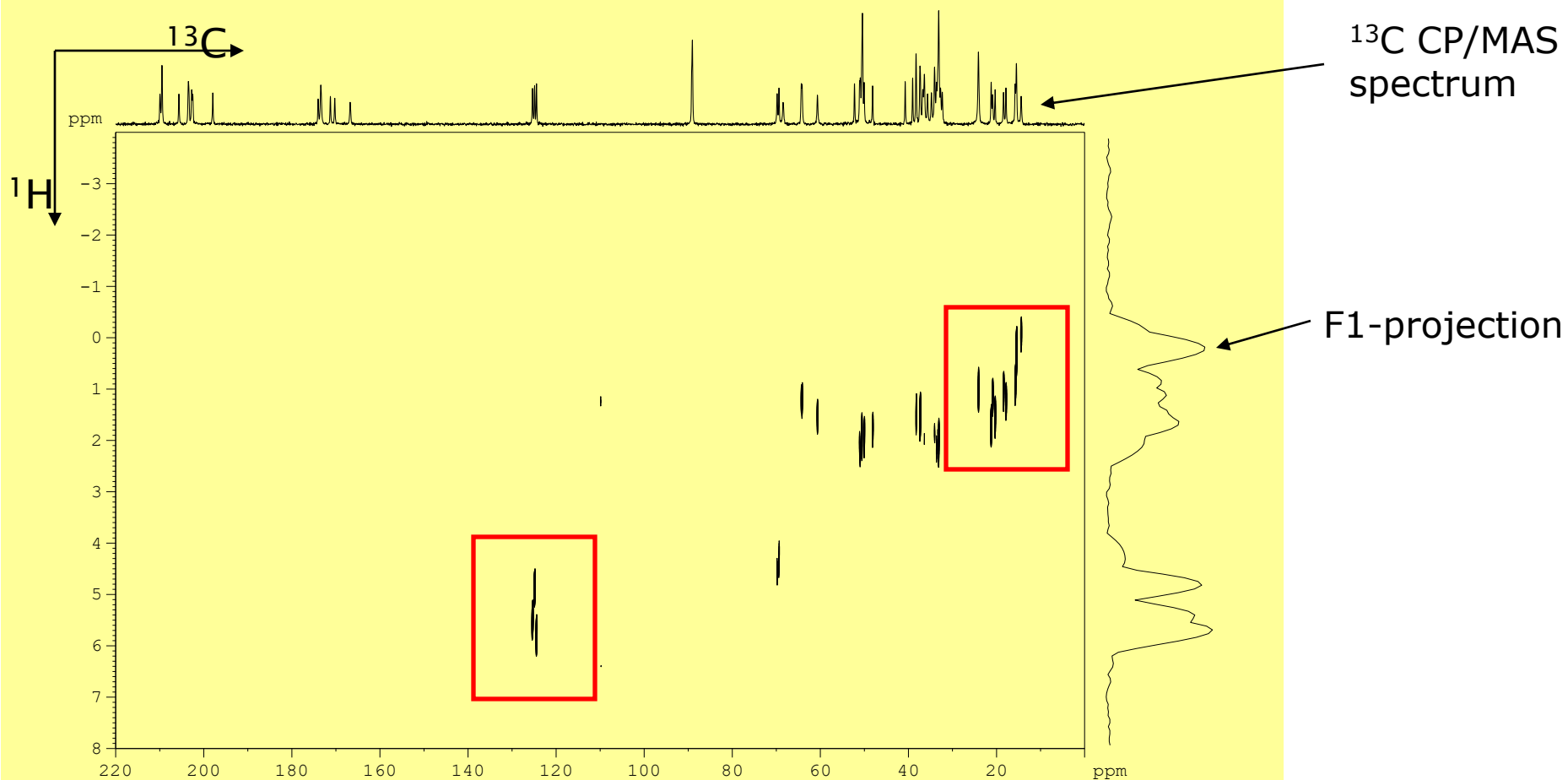
Cortisone Acetate: Identification of Polymorphs



(spectra not
drawn to scale)

Cortisone Acetate: Characterisation of Polymorphs

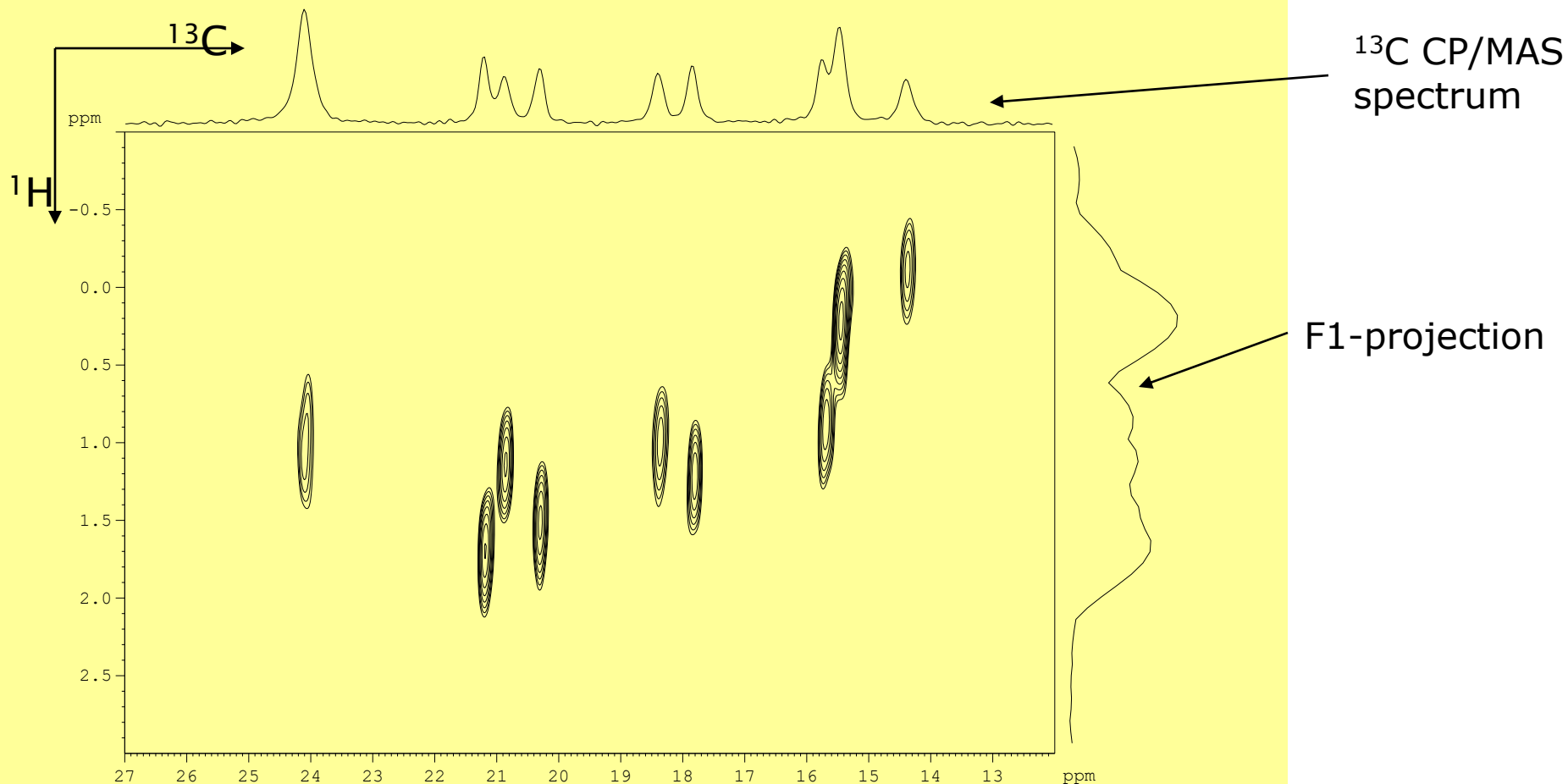
^{13}C - ^1H MAS-J-HMQC spectrum of cortisone acetate, form III



400 MHz spectrometer, spinning speed 13.717 kHz, contact time 400 μs

Cortisone Acetate: Characterisation of Polymorphs

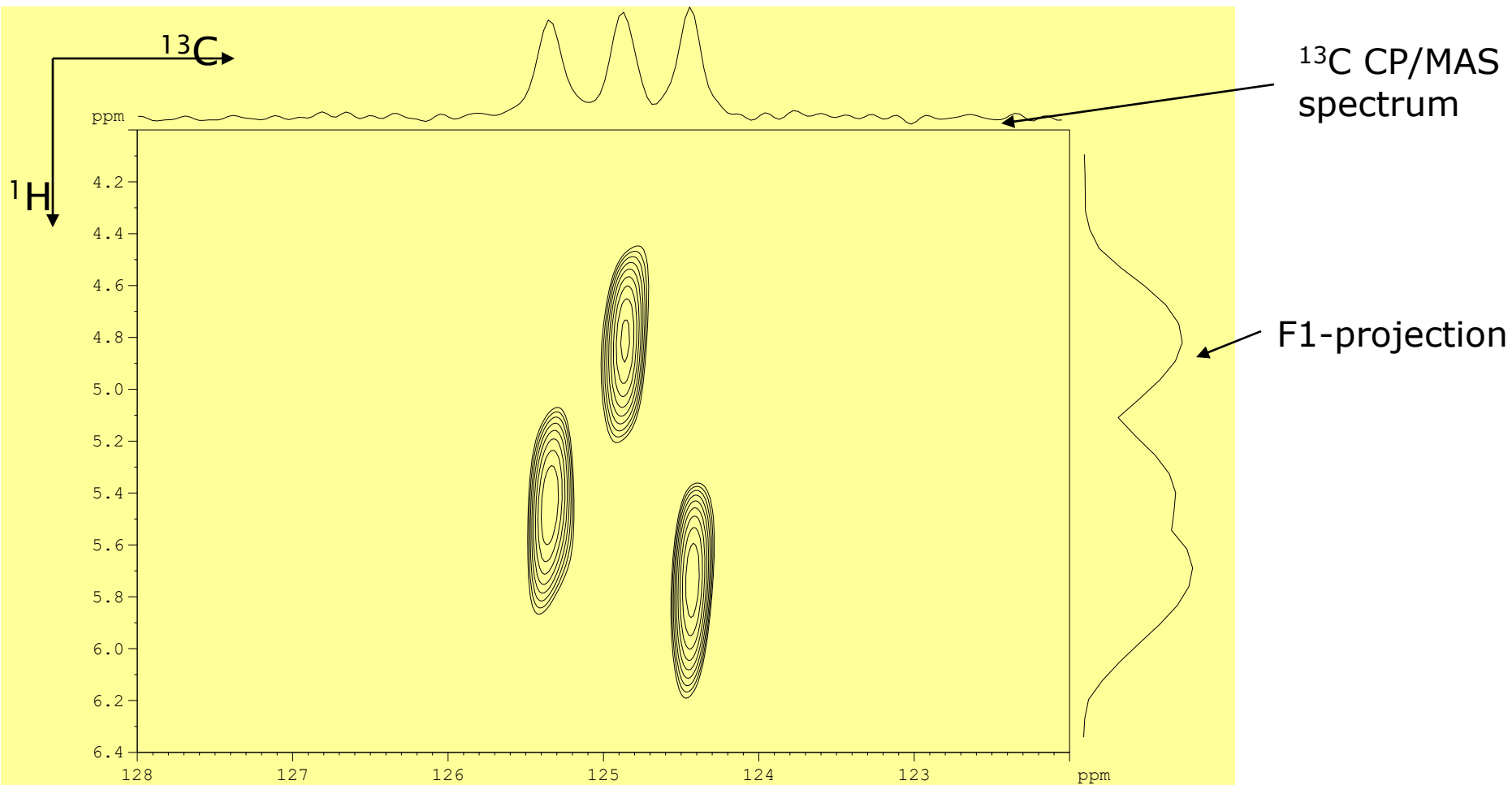
^{13}C - ^1H MAS-J-HMQC spectrum of cortisone acetate, form III



expanded view of ^{13}C 27 ppm - 12 ppm region

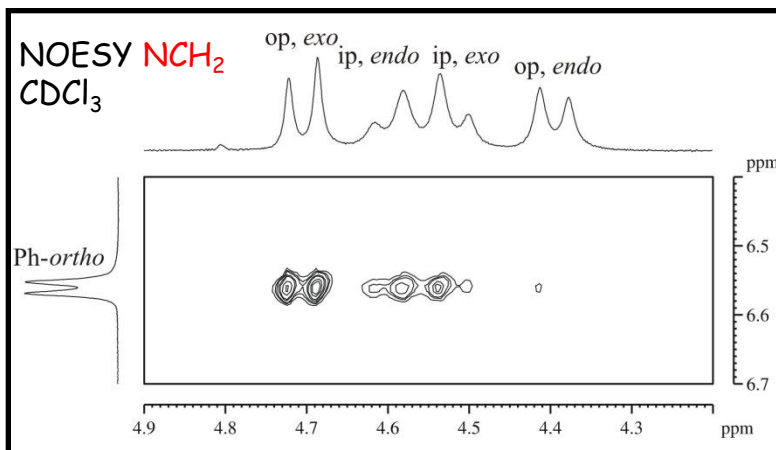
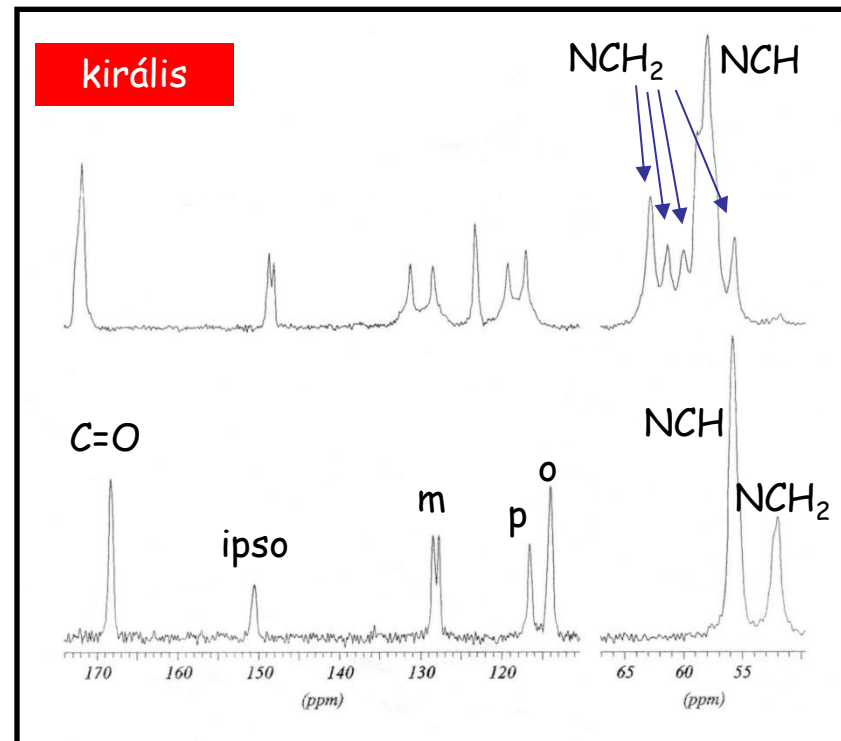
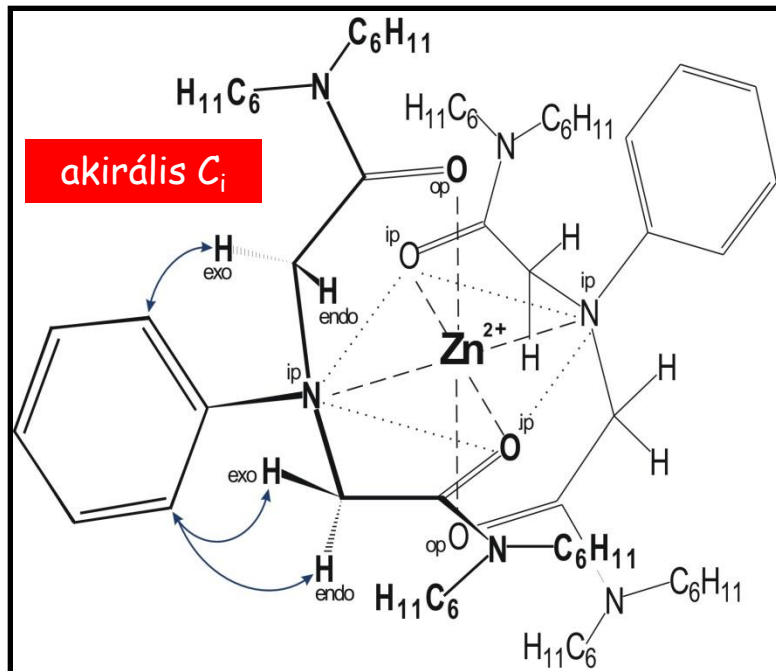
Cortisone Acetate: Characterisation of Polymorphs

^{13}C - ^1H MAS-J-HMQC spectrum of Cortisone Acetate, form III



expanded view of ^{13}C 128 ppm - 122 ppm region

Iminodiacetamid $\text{Ph-N}[\text{CH}_2\text{CON}(\text{C}_6\text{H}_{11})_2]_2$ ionofór cink komplexe



Komplex és szabad iminodiacetamid ionofór
CP MAS ^{13}C NMR spektruma

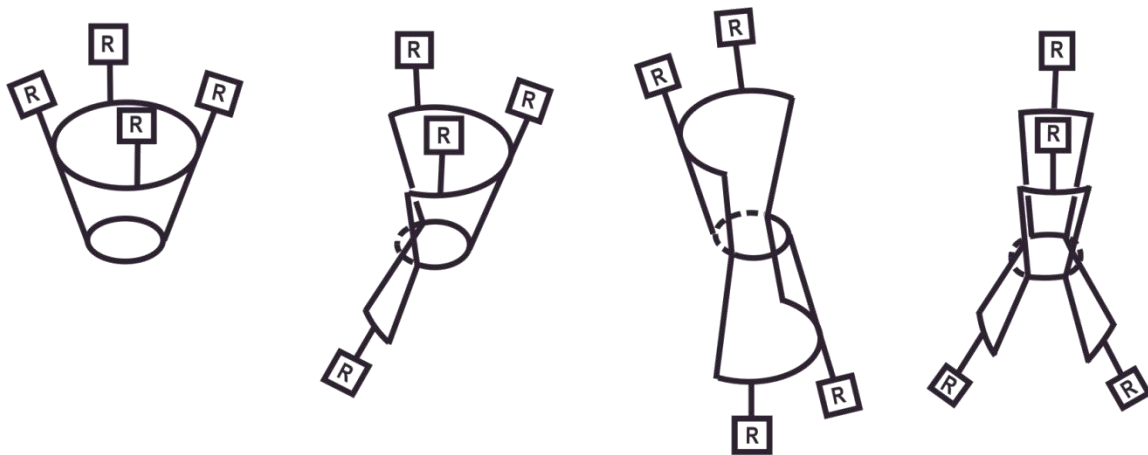
G. Tóth, B. Balázs, Gy. Horváth, D. Magiera, H. Duddeck,
I. Bitter : J. Incl. Phen. Macr. Chem. 43, 145 (2002)

**Kristályban deformáció folytán
megszűnik a centrális szimmetria**

Kalix[4]arének sztereokémiája



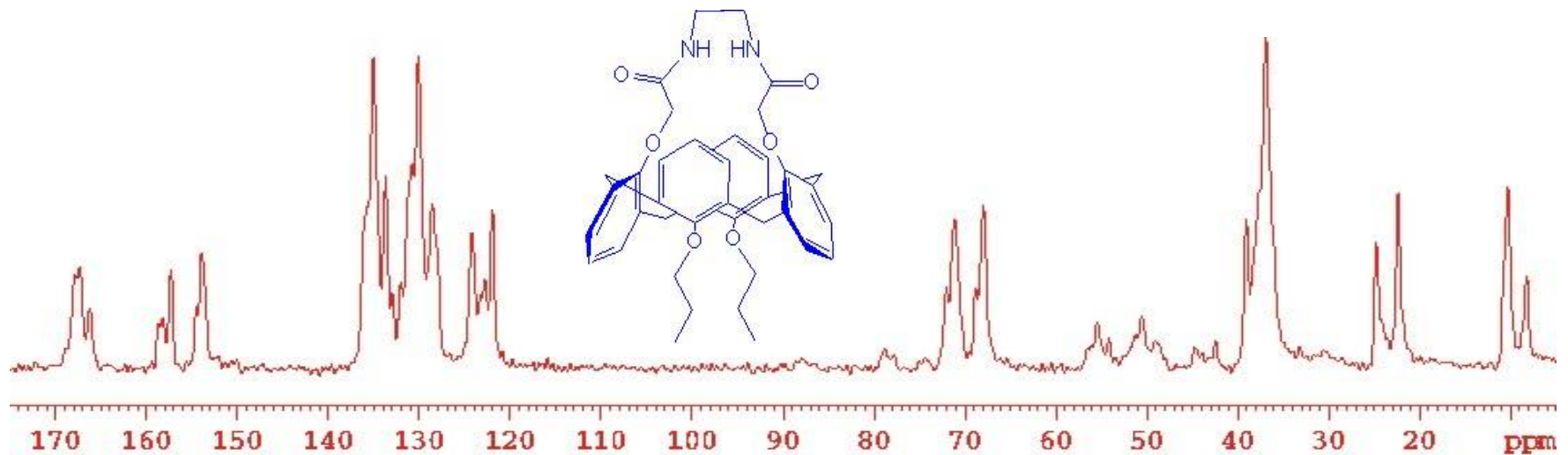
Stabil konformerek



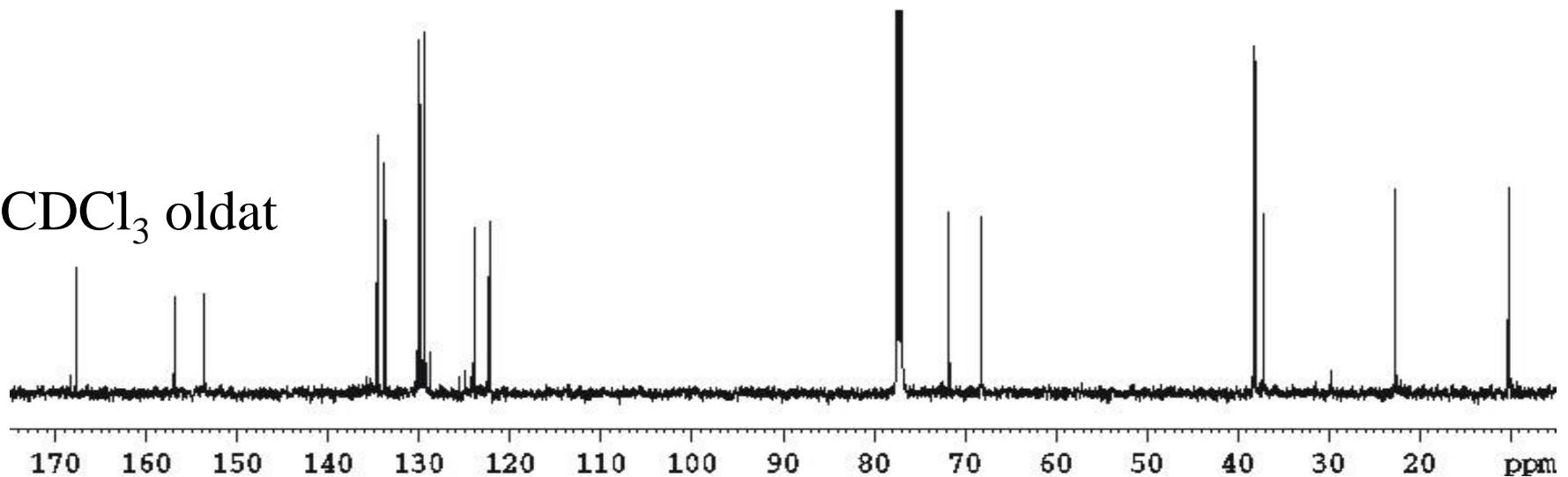
Kónikus Részlegesen kónikus 1,2-Alternáló 1,3-Alternáló

Bitter I., Grün A., Csokai V.,
Tóth G., Balázs B., Horváth Gy.

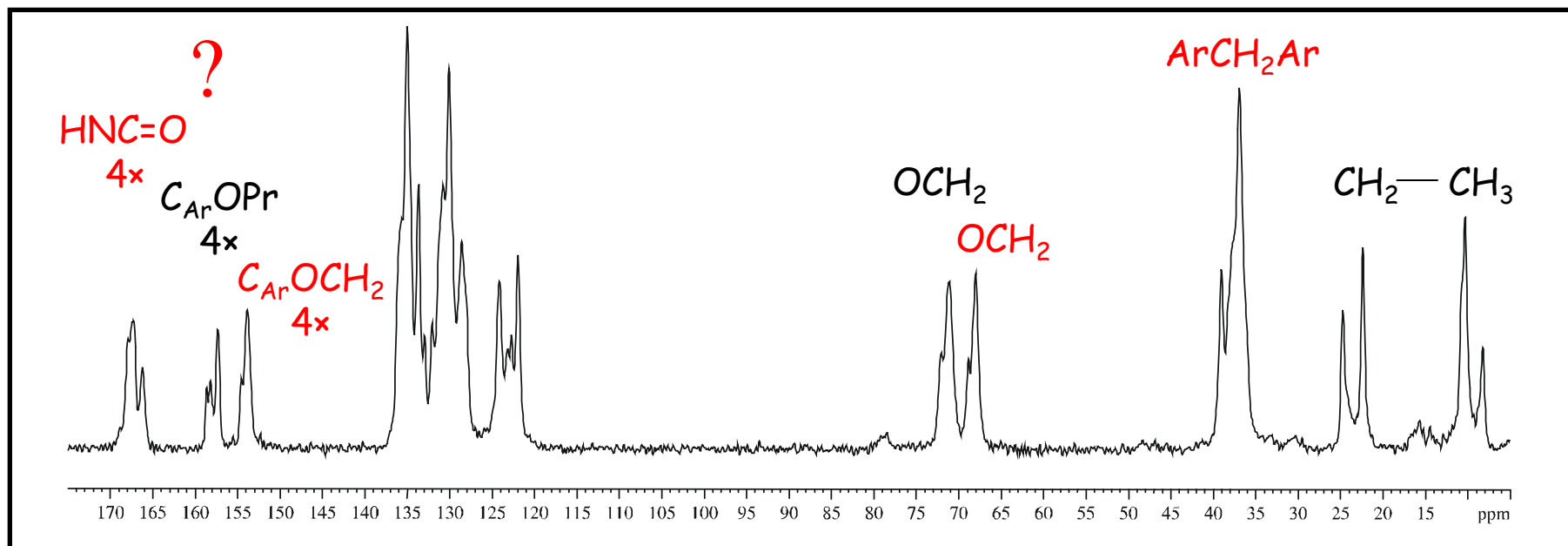
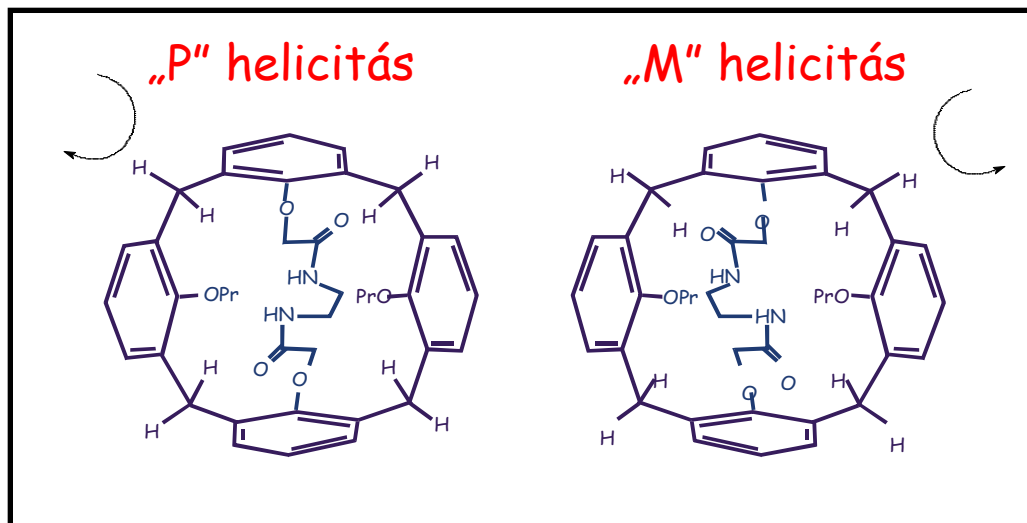
CP-MAS NMR Spektroszkópia (Cross-Polarisation-Magic-Angle-Spinning)



CDCl₃ oldat



5 vegyület 1,3-alternáló konformerének ^{13}C CP MAS spektruma



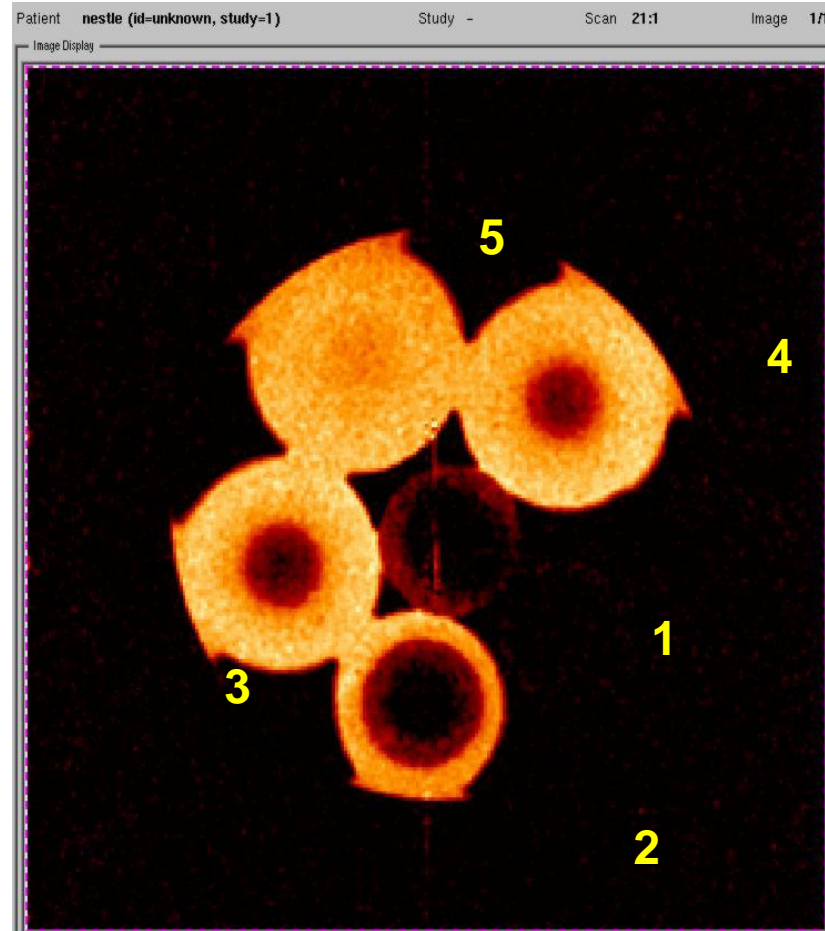
Control of Spagetti cooking by NMR

Art of Cuisine

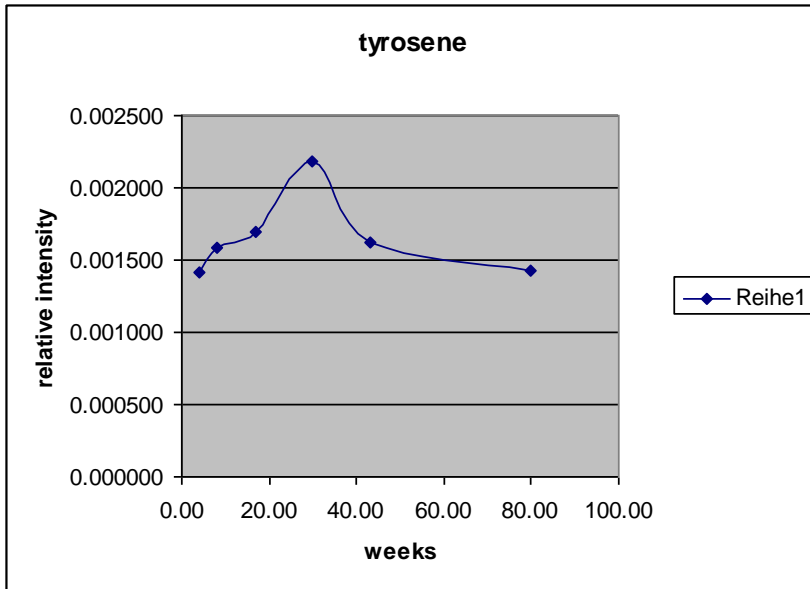
Effect of cooking time (Was ist al dente?)

- 1) dry
- 2) 1 min.
- 3) 3 min.
- 4) 5 min.
- 5) 10 min.

300 MHz 7 T
Method: 3D Spin Echo MRI
Data matrix: 128 x 128 x 32
FOV: 5 x 5 x 15 mm
Resolution: 39 x 39 x 468 μ m
TR: 200 ms
TE: 2 ms
SWH: 100 kHz
Averages: 1
Total time: 12 min



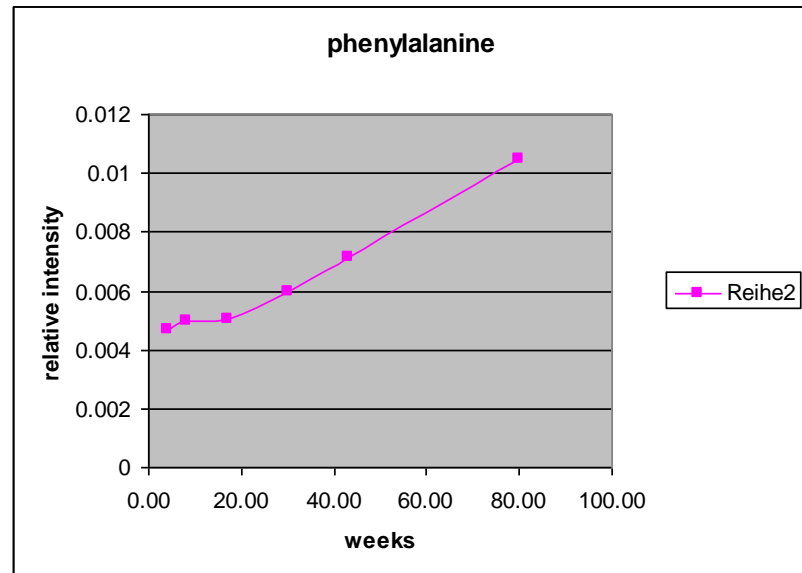
Control of Ripening process of cheeses by NMR



Beemster Gouda cheese
Different ripening stages

HR-MAS spectra NOESY-presat
400MHz spin rate 4.5 KHz
≈ 5 minutes per sample

Tyrosine is converted to phenylic acids determining the taste of the cheese.
Phenylalanine is not converted, Concentration increases with aging.



DNP-NMR a Biomolekuláris NMR spektroszkópiában

A Dinamikus Mag-Polarizáció (DNP) alkalmazása során a páratlan elektron (gyök) extrém nagy spin-polarizációját visszük át egy NMR aktív magra, leggyakrabban az ^1H atomra. (Fig. 1)

Az elektronról kiinduló polarizáció transzfer az NMR aktív magra úgy indul el, hogy egy megfelelő polarizációs reagenst (PA polarizing agent) alkalmazunk és ezt megfelelő ESR frekvencián (electron paramagnetic resonance, EPR) besugározva gerjesztjük. A minél nagyobb hatékonyság elérése céljából a megfelelő polarizációs reagenst kell kiválasztani, miközben figyelembe vesszük a DNP mechanizmusát és az NMR műszer térerejét (frekvenciáját) is (Lásd Fig. 2).

Az ^1H atomra átvitt polarizáció pl. a kereszt-polarizáció (cross-polarization CP) révén tovább vihető az alacsony giromágneses tényezővel (γ) rendelkező ^{13}C vagy ^{15}N magokra is. Ennek eredményeként a rutin ^1H kereszt-polarizáció alkalmazásával elért érzékenység akár 658-ra is megnövekedhet.

A rutin, komerciális szilárdfázisú NMR berendezéseknél a fenti technika a 9.4 Tesla térerejű 400 MHz-es NMR műszerektől kezdődően egészen a 18.8 Teslaig (800 MHz) terjedő tartományban használható.

DNP-enhanced solid-state NMR spektroszkópia a gyógyszerkutatásban

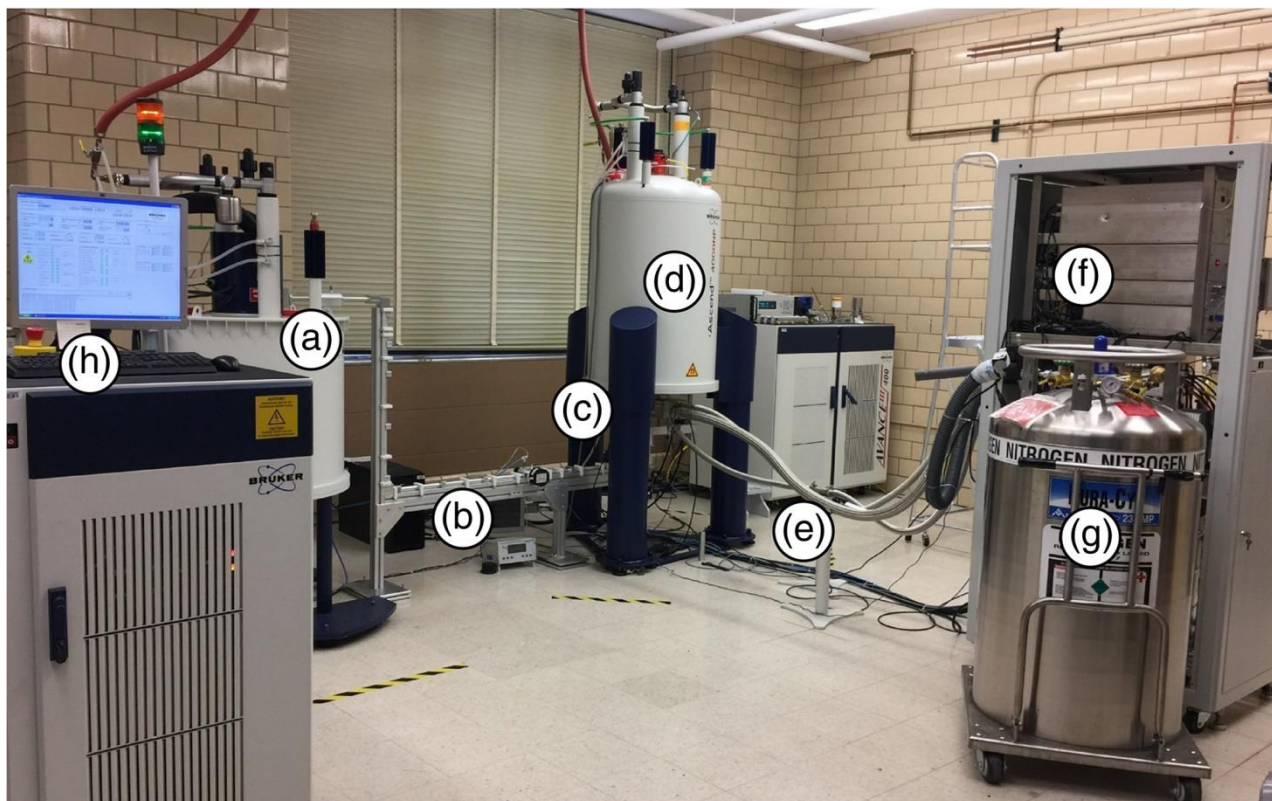


Fig. 1. The commercial Bruker 9.4-T (400 MHz) or 263-GHz dynamic nuclear polarization solid-state NMR spectrometer setup at the Ames Lab.. Continuous-wave 263-GHz microwaves are generated by a gyrotron **(a)** and transmitted via a waveguide **(b)** to the magic angle spinning probe **(c)** housed in the 9.4-T wide-bore NMR magnet **(d)**. Sample temperatures of ~ 100 K are achieved by using cold nitrogen gas for sample spinning and sample cooling. The cold nitrogen gas is delivered to the magic angle spinning probe in an insulated transfer line **(e)**. The nitrogen gas is cooled inside of a pressurized heat exchanger **(f)**, which is fed with liquid nitrogen **(g)**. The gyrotron control computer **(h)** can be used to turn the continuous-wave 263-GHz microwaves on or off and adjust the microwave power

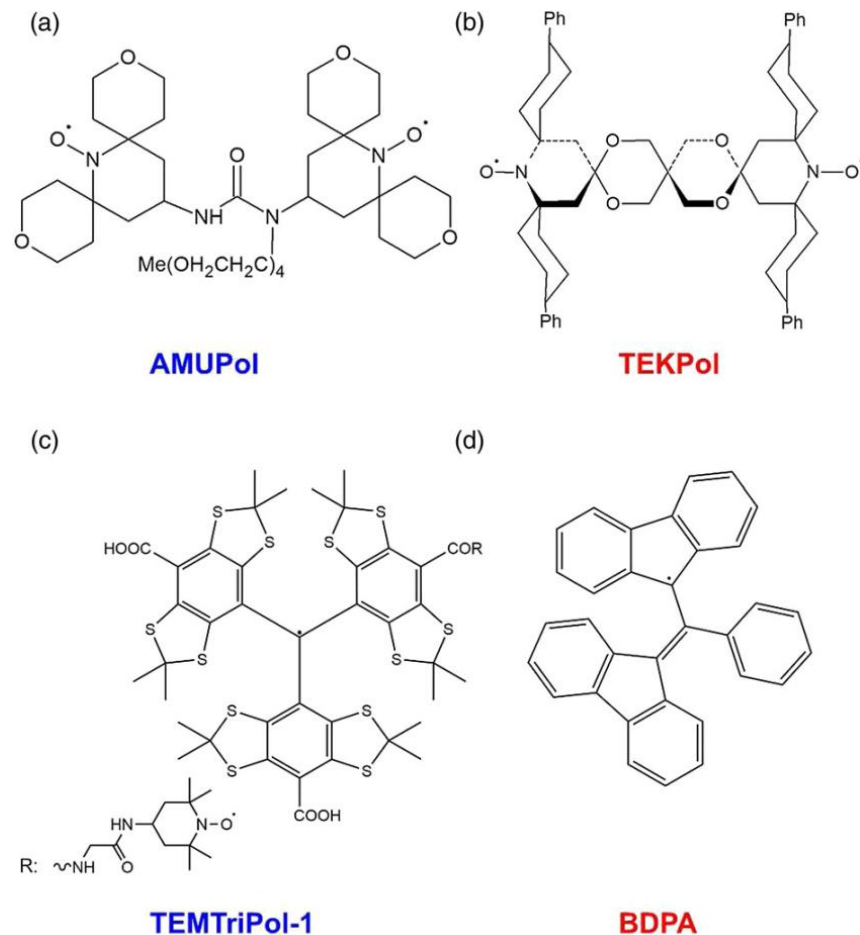


FIGURE 2

The molecular structures of exogenous dynamic nuclear polarization (DNP) polarizing agents.

(a) AMUPol, (b) TEKPol, (c) TEMTriPol-1, (d) BDPA.

The cross-effect polarizing agents AMUPol and TEKPol typically provide the largest ¹H DNP signal enhancements at fields of 9.4 T or lower. AMUPol is used with aqueous solvent mixtures, whereas TEKPol is used with organic solvents.

TEMTriPol-1 and BDPA have provided the largest ¹H DNP enhancements at magnetic fields of 16.4 T and higher

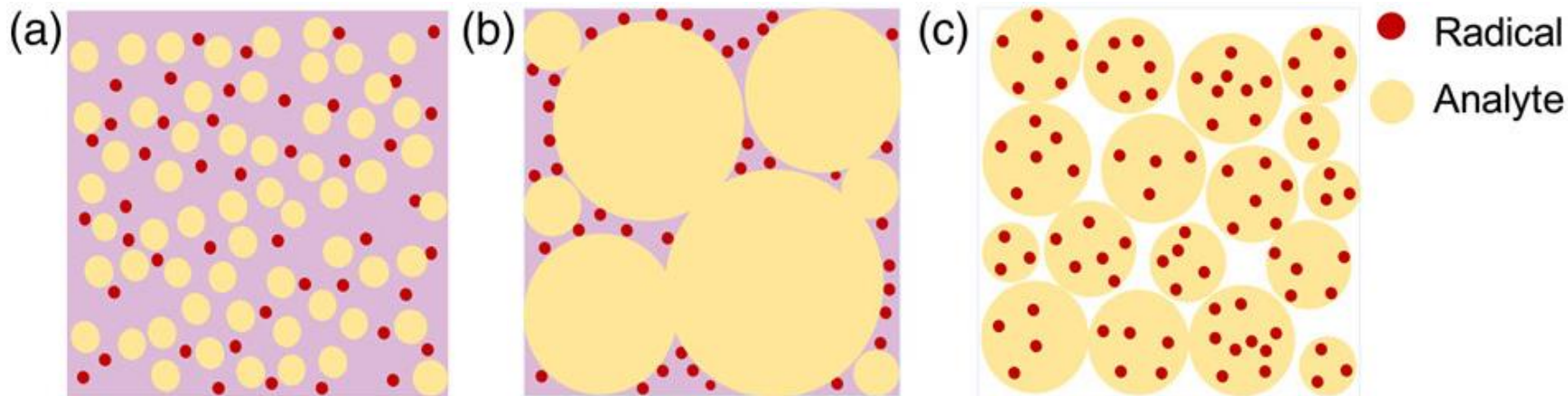


FIGURE 3

Cartoons of dynamic nuclear polarization sample preparations illustrating the distribution of analyte (yellow spheres) and radical polarizing agent (PA; red spheres).

- (a) The analyte is dissolved and homogeneously distributed in the radical-containing solution;
- (b) an inhomogeneous nanoparticulate or microparticulate analyte is impregnated with a radical solution and the PA is restricted to the surface of the analyte domains;
- (c) direct doping of the PA into the analyte particles

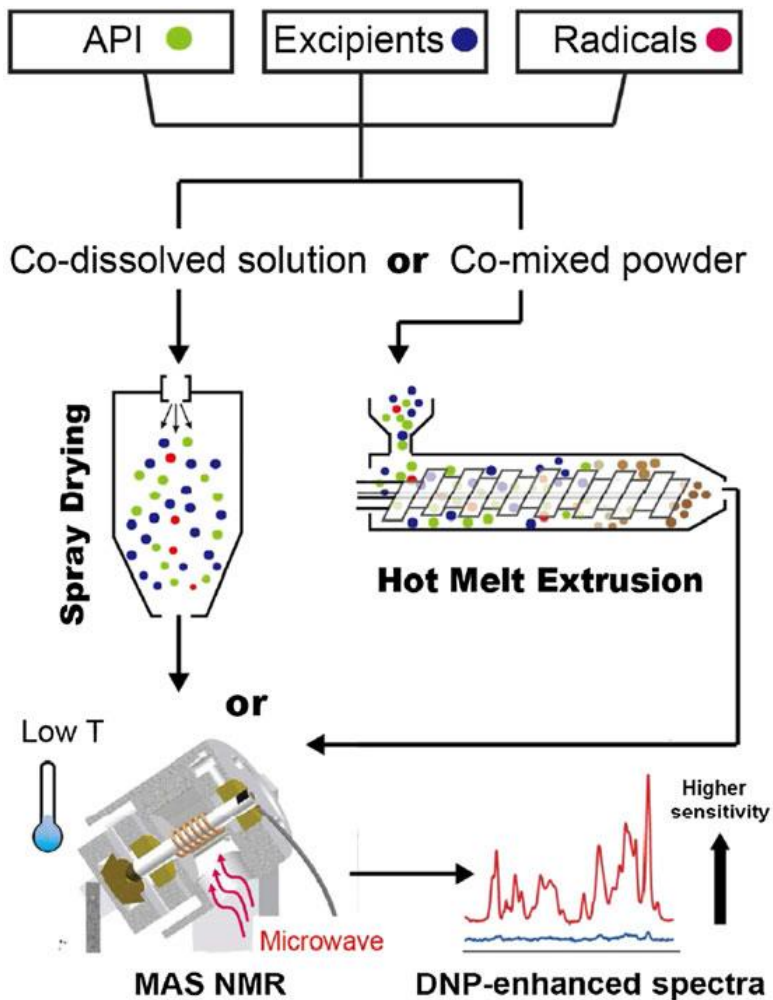


FIGURE 6. Minta előkészítési protokoll.

Sample preparation protocols used to directly dope amorphous solid dispersions prepared by spray drying or hot-melt extrusion.

API = active pharmaceutical ingredient;
 DNP = dynamic nuclear polarization;
 MAS = magic angle spinning.

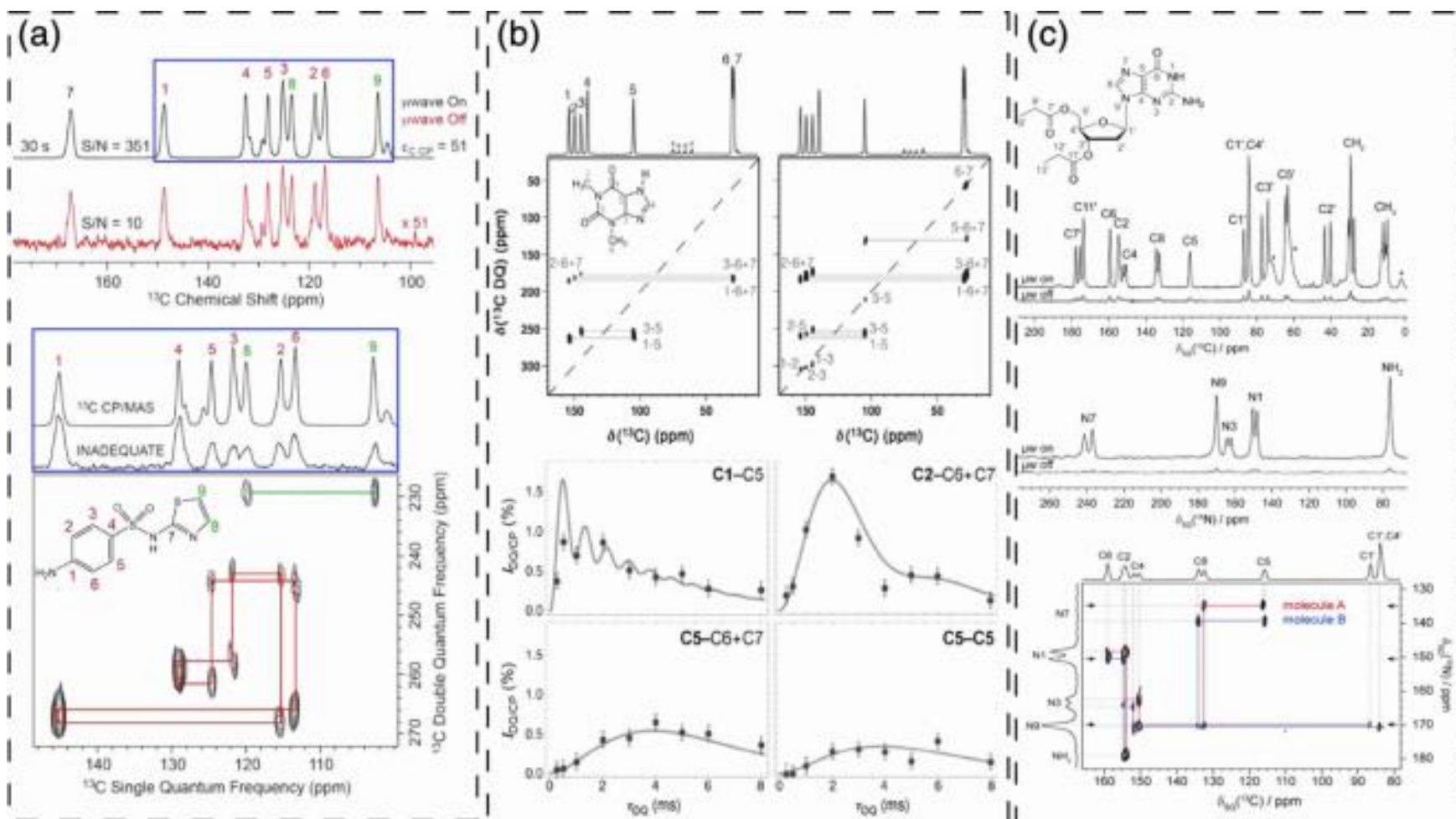
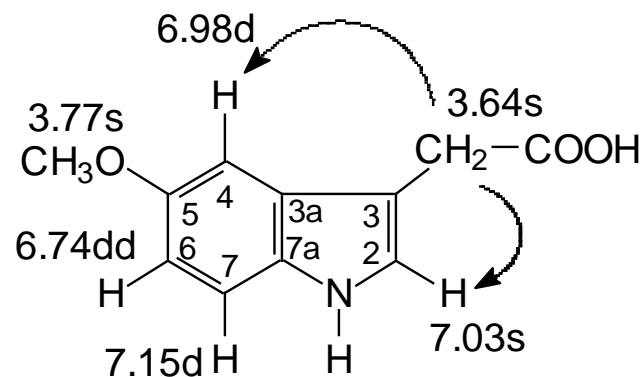
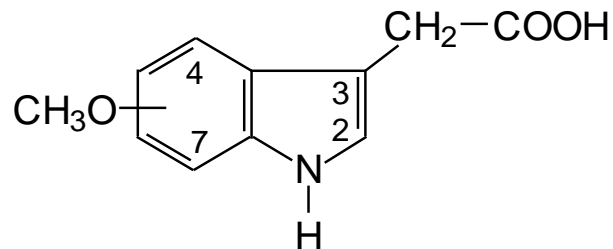
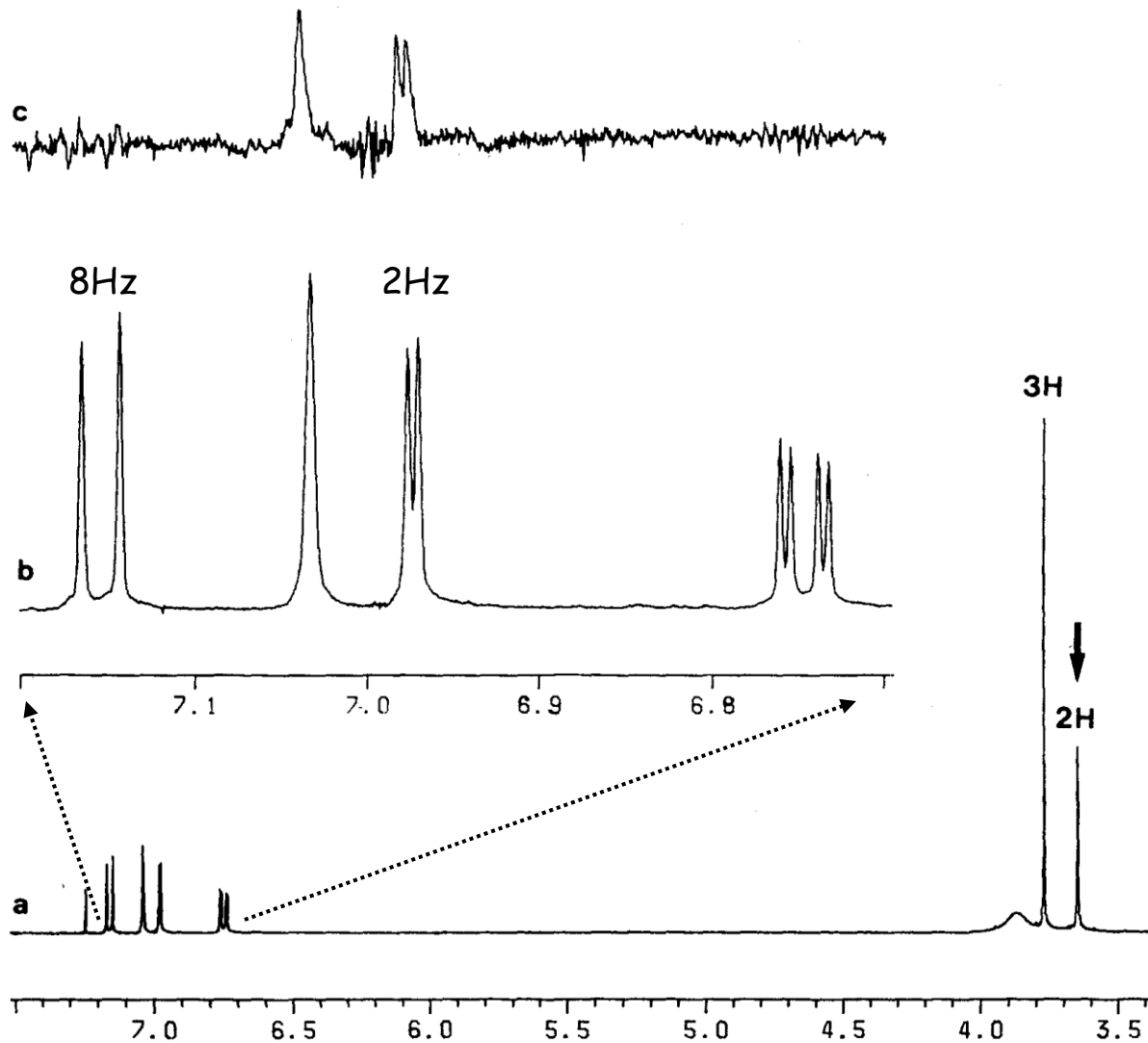


FIGURE 9 Examples of challenging multidimensional solid-state NMR experiments enabled by relayed dynamic nuclear polarization (DNP). (a) DNP-enhanced two-dimensional (2D) scalar ^{13}C - ^{13}C double-quantum single-quantum (DQ-SQ) homonuclear correlation spectrum of sulfathiazole obtained with the refocused INADEQUATE pulse sequence in a total experiment time of 16 hr. MAS = magic angle spinning. Reprinted with permission from Rossini et al.^[122] Copyright 2012 American Chemical Society. (b) DNP-enhanced 2D dipolar ^{13}C - ^{13}C DQ-SQ homonuclear correlation spectra of theophylline Form II and the DQ signal buildup curves extracted from 2D spectra obtained with different mixing times. The DQ signal buildup curves are fit to analytical functions corresponding to ideal buildup curves generated from the carbon-carbon distances and dipolar couplings observed in the crystal structure of theophylline. Reprinted with permission from Mollica et al.^[137] Copyright 2015 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. (c) DNP-enhanced 2D ^{13}C - ^{15}N cross-polarization (CP)-heteronuclear correlation spectrum of a self-assembled 2'-deoxyguanosine derivative. Reprinted with permission from Märker et al.^[157]

Határozza meg a CH_3O csoport helyét a 3.64 ppm-nél besugárzott NOE differencia spektrum (c) alapján!



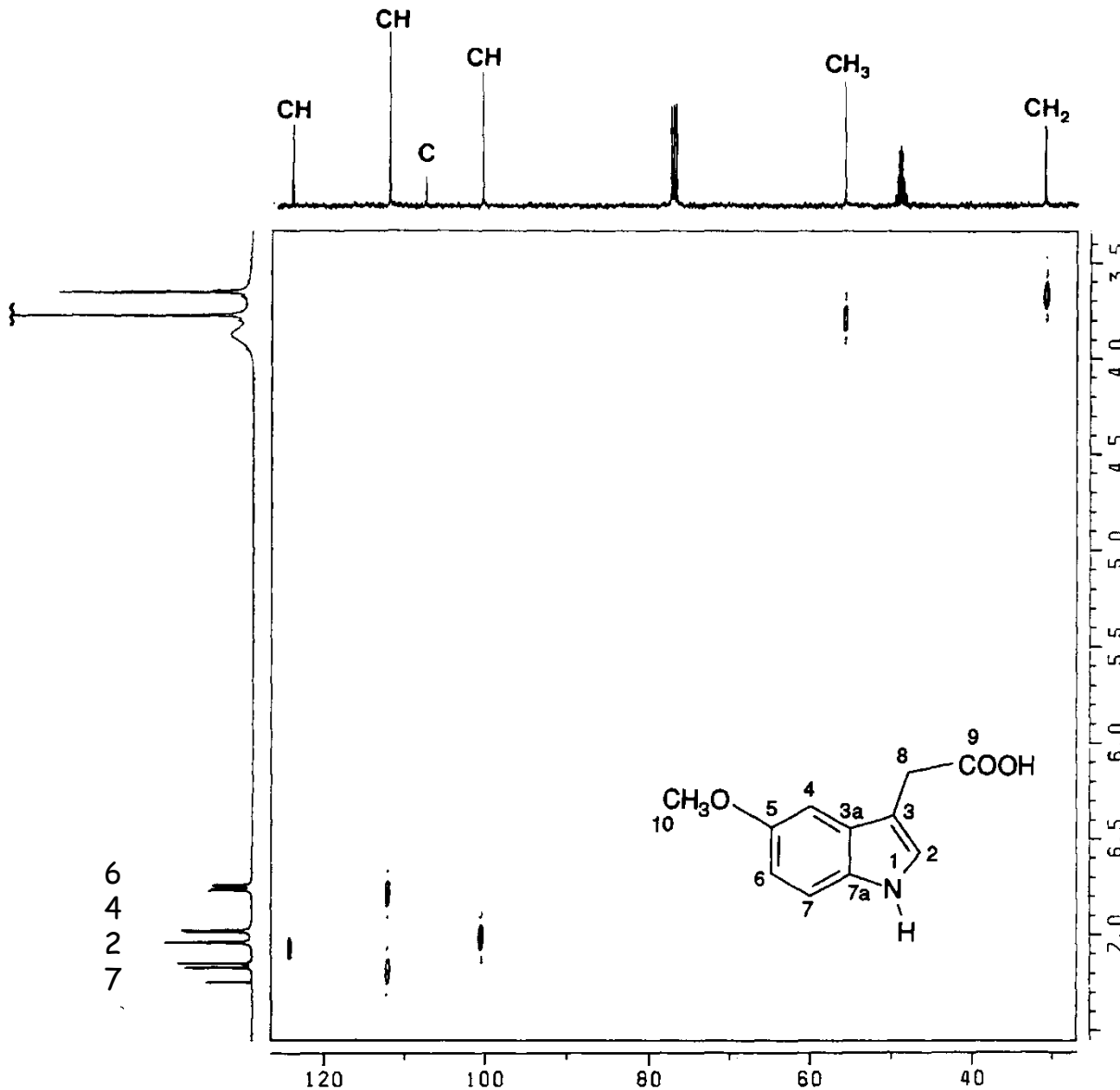
5-metoxi-3-indolilecetsav

Oldószer: $\text{CDCl}_3 + \text{CD}_3\text{OD}$

Végezze el a ^{13}C jelhozzárendelést a C,H-COSY spektrum alapján!

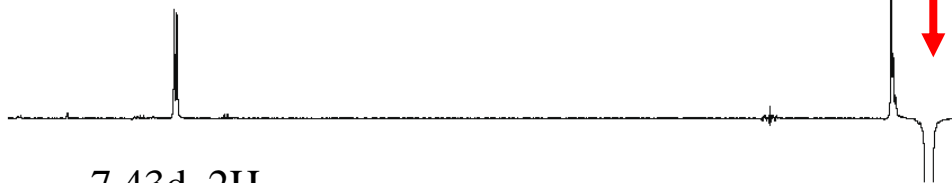
124.0 111.9 100.4 CDCl_3 55.8 CD_3OD 31.0

további kvaterner jelek:
174.5; 153.6; 131.2; 127.3; 107.5

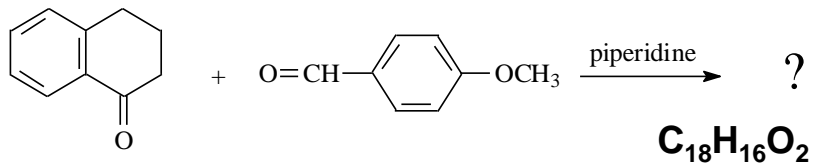


	δC ppm
C-2	124.0
C-4	100.4
C-6	111.9
C-7	111.9
C-8	31.0
CH_3O	55.8
C-3	107.5
C-3a	127.3
C-5	153.6
C-7a	131.2
C-9	174.5

7.24d, 1H

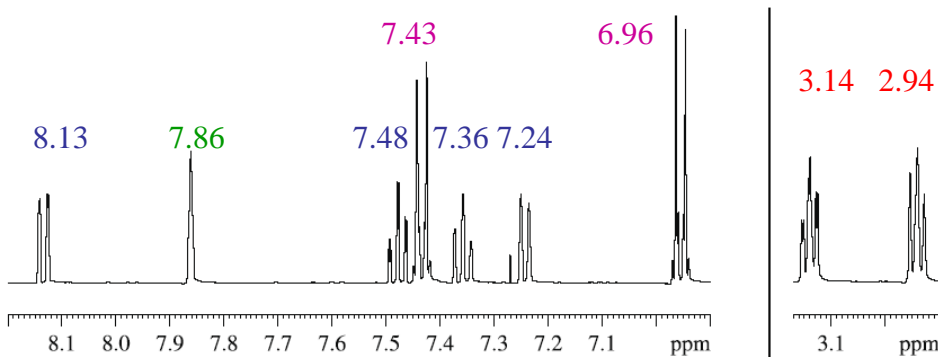
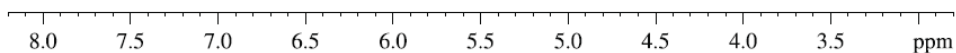


7.43d, 2H



Sel. 1D NOESY

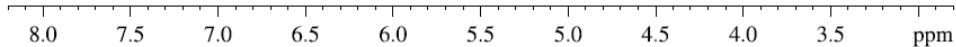
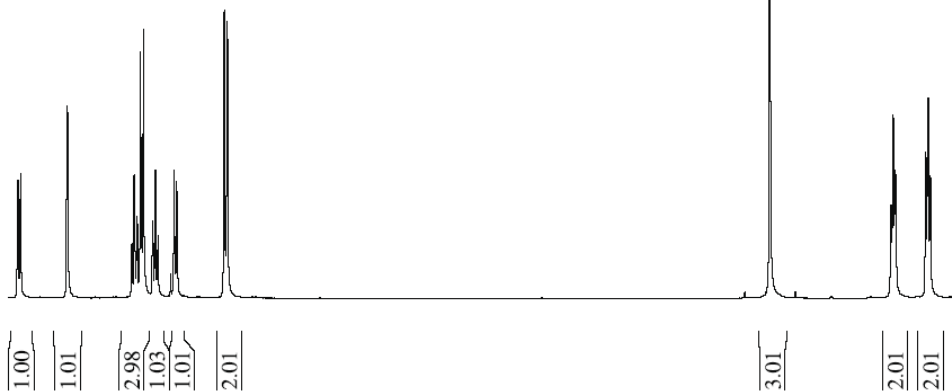
Sel. 1D NOESY



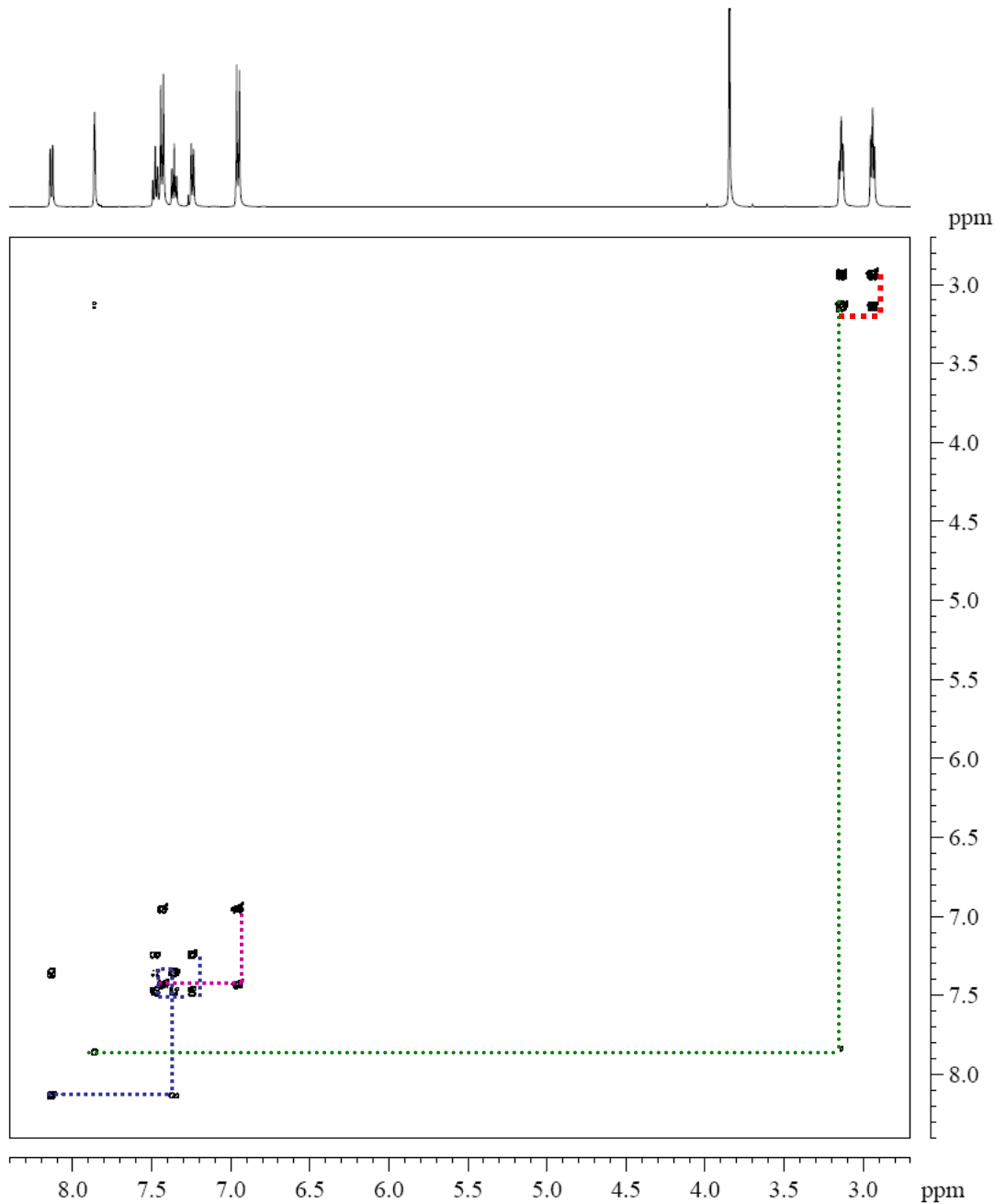
Sections of ^1H NMR

CH_2CH_2 ; OCH_3 ;

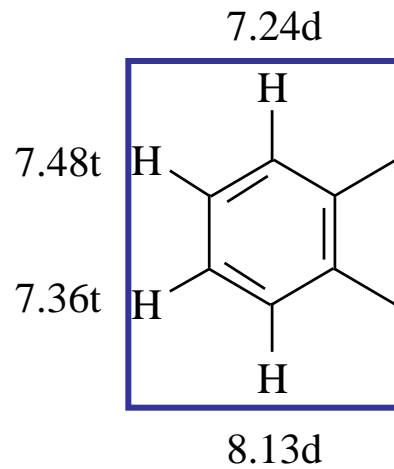
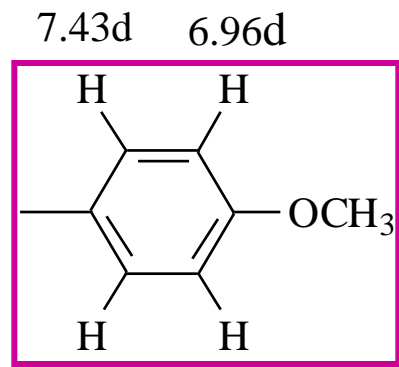
aromatic/olefinic range: 9H



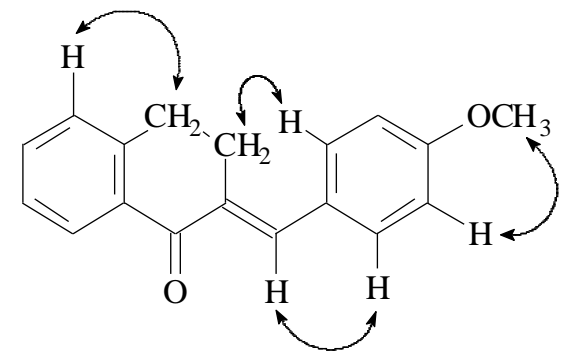
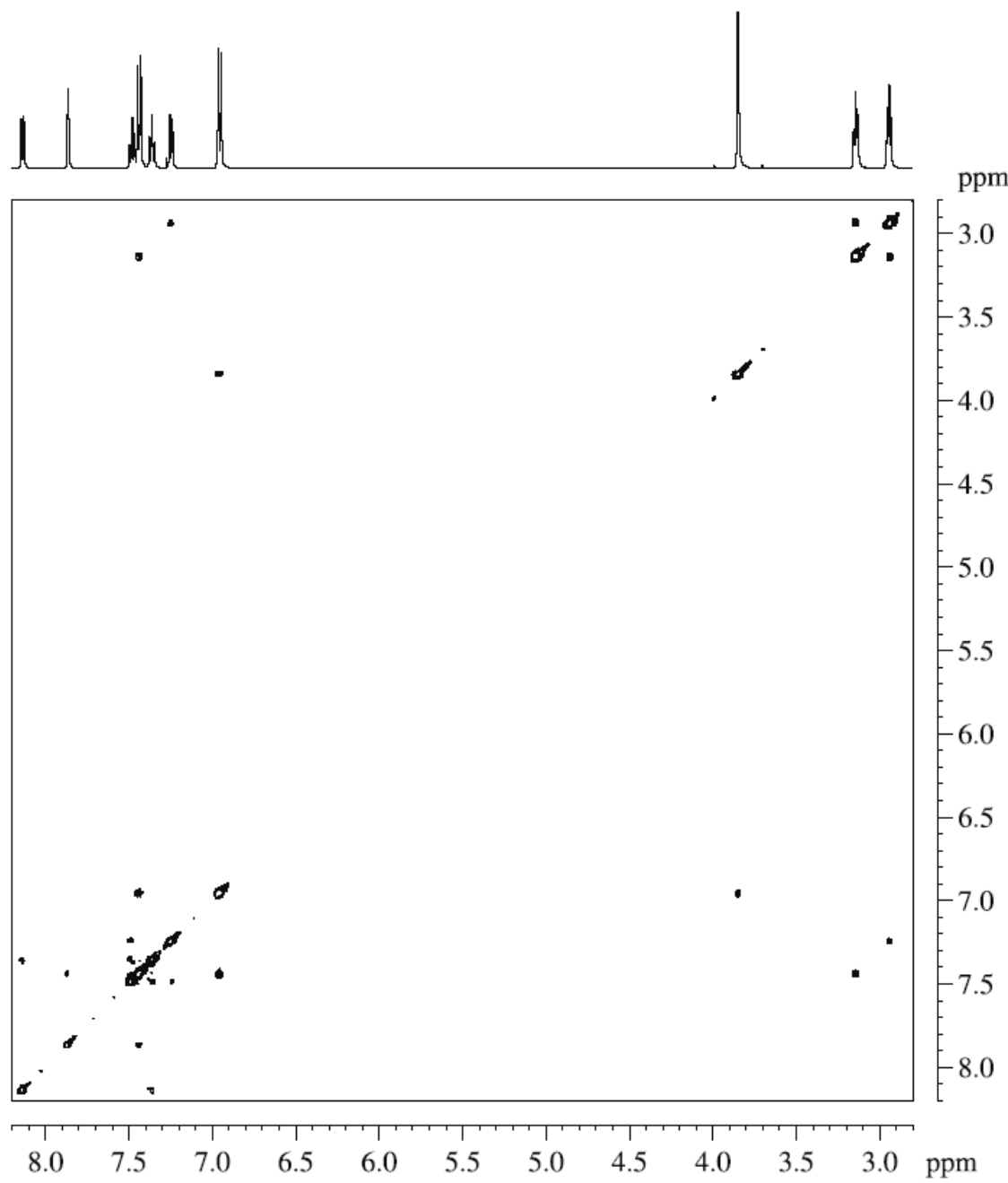
$^1\text{H}, ^1\text{H}$ - COSY



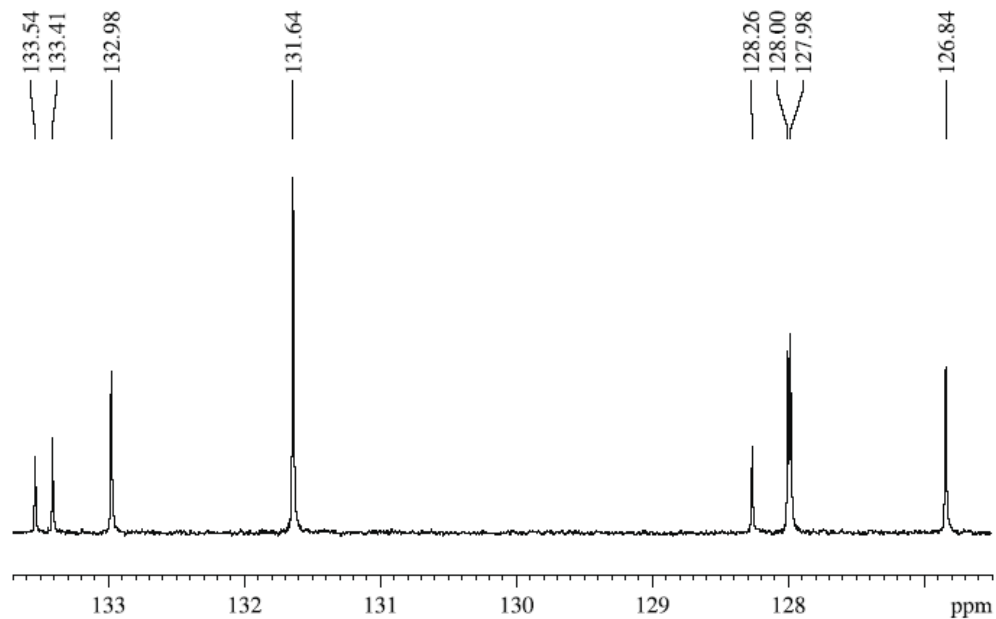
2.94t 3.14t 7.86s
- $\text{CH}_2 - \text{CH}_2 - \text{X} = \text{CH}$



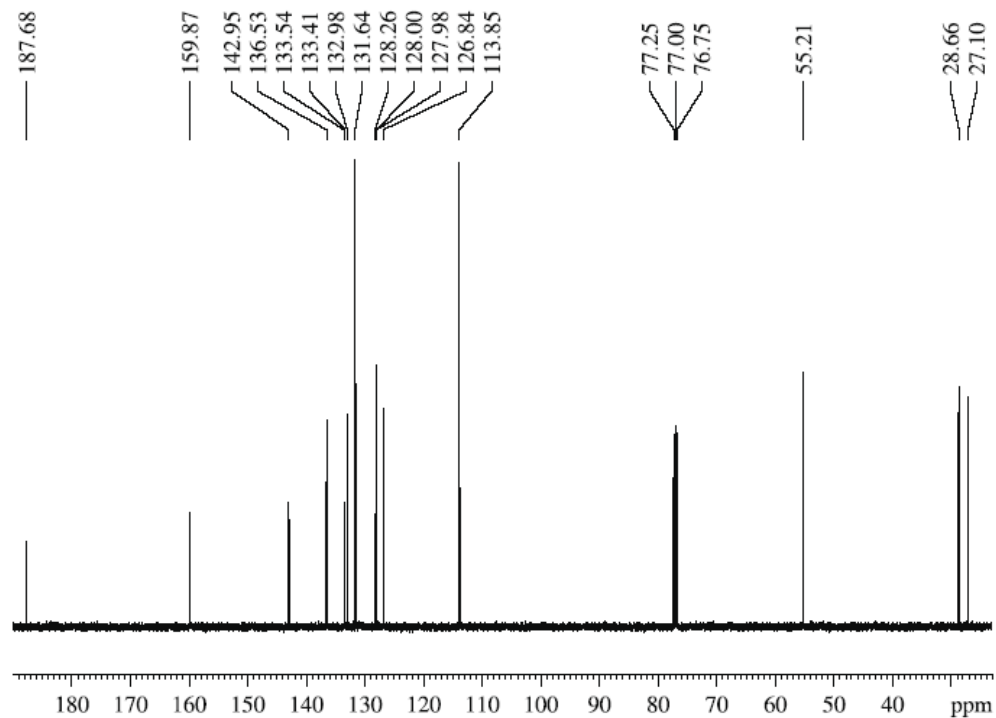
$^1\text{H}, ^1\text{H}$ - NOESY



arrows indicate steric proximities



Section of ^{13}C NMR spectrum

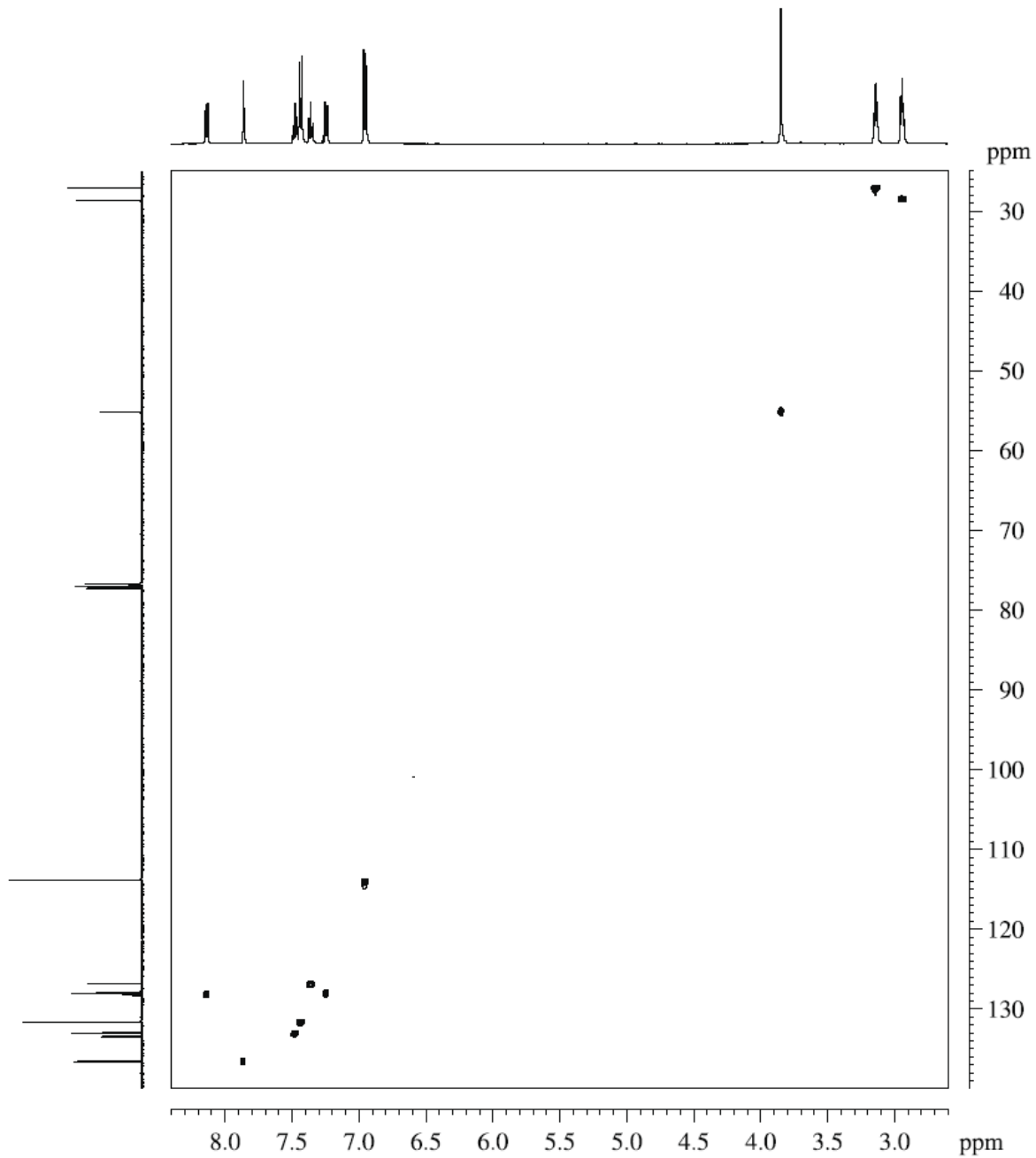


16 Signals corresponding to C_{18} atoms

3 (sp^3C); 1 $\text{C}=\text{O}$; 7 $=\text{CH}$; 5 $=\text{C}<$

^{13}C NMR spectrum

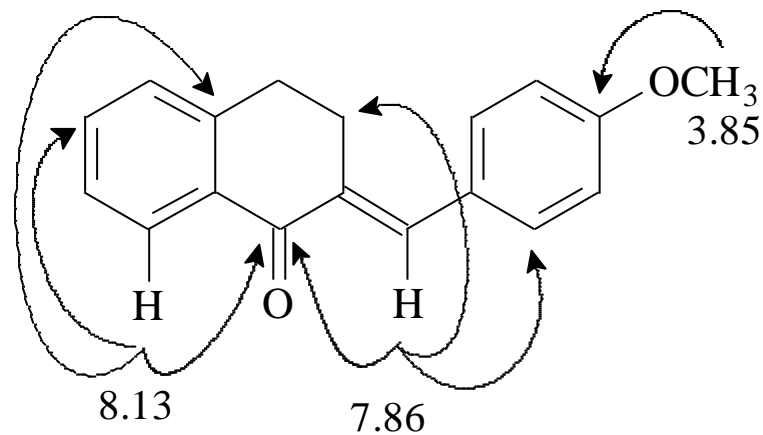
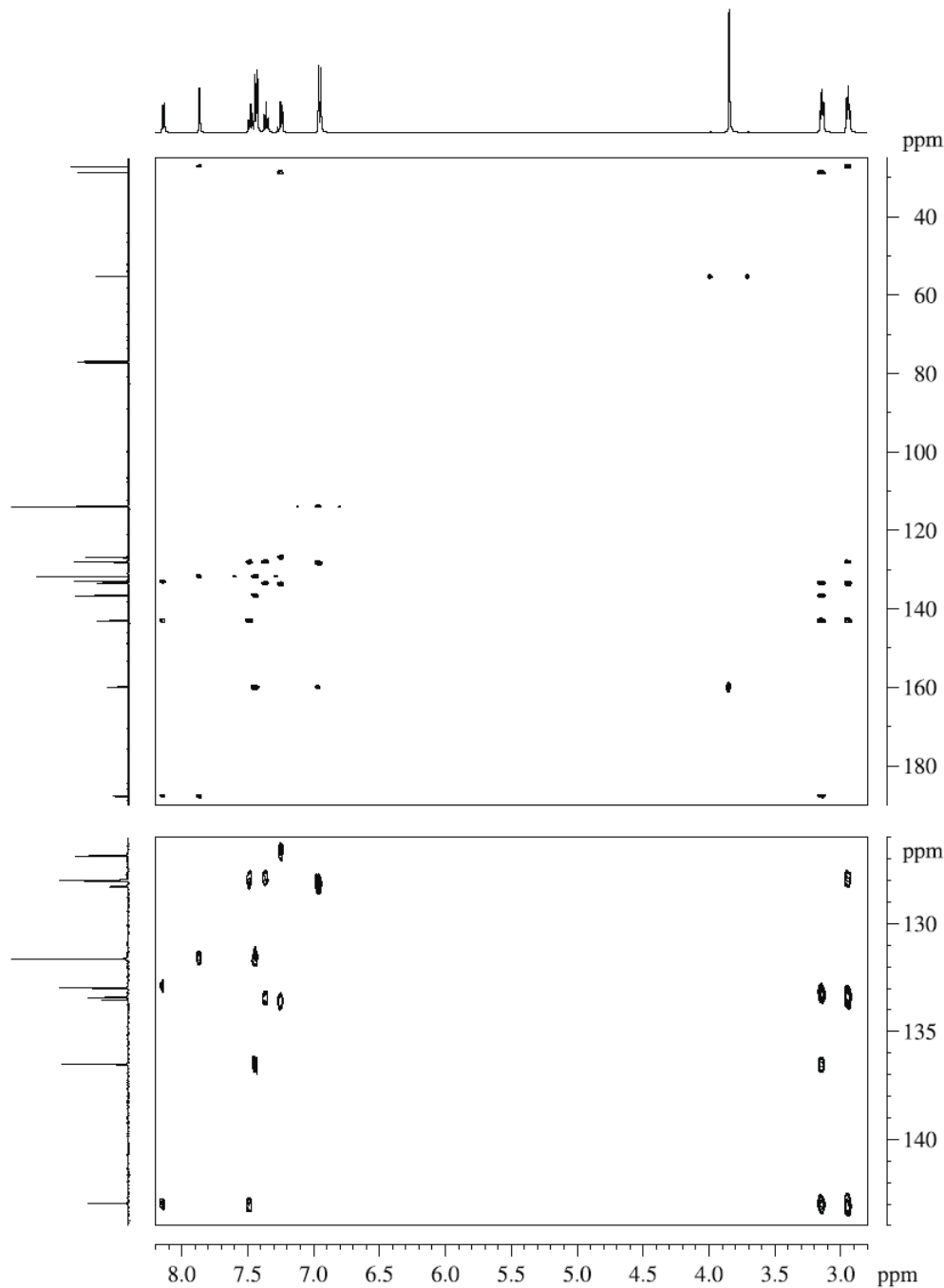
$^1\text{H}, ^{13}\text{C}$ - HSQC



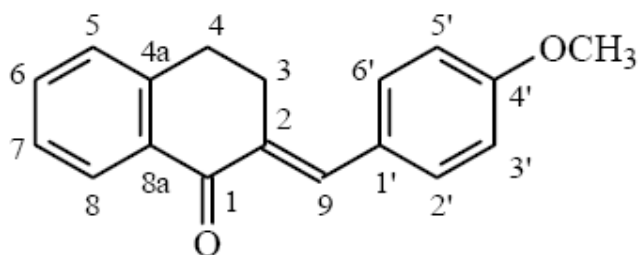
with section of $\text{sp}^2 =\text{CH}$ range

$^1\text{H}, ^{13}\text{C}$ - HMBC

Heteronuclear Multi Bond Correlation
via $^2J_{\text{CH}}$ and $^3J_{\text{CH}}$ couplings



Presentation of NMR Data (500 resp. 125 MHz, CDCl₃, δ)

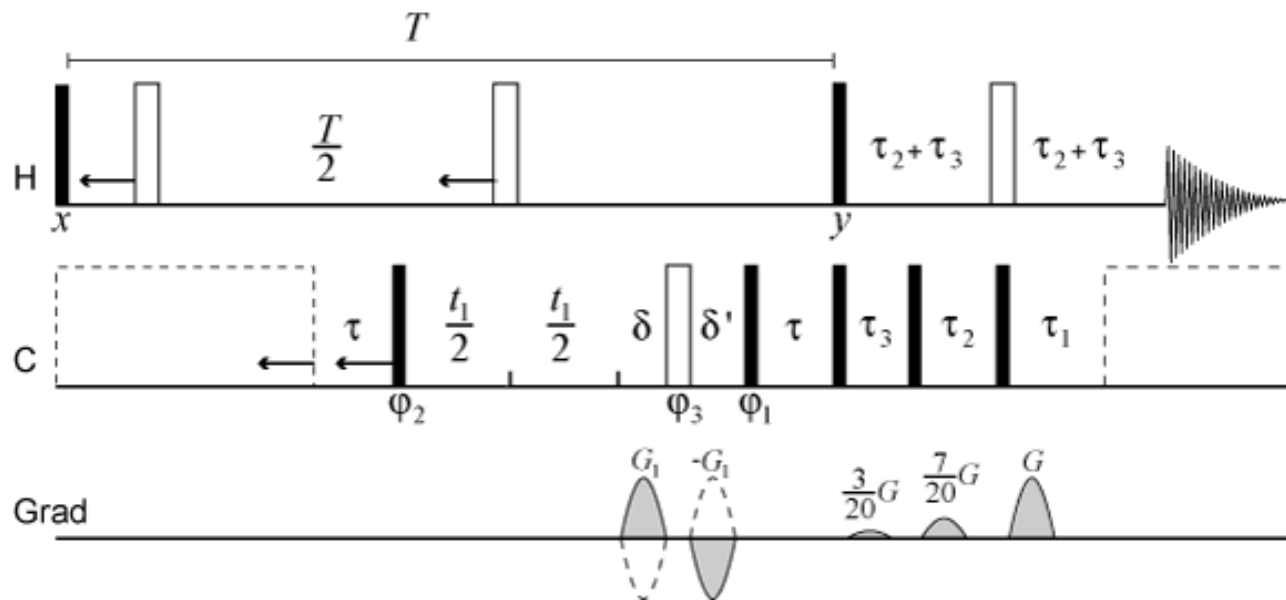


Assignment	¹ H (J)	¹³ C	HMBC responses (¹³ C partners)	NOE responses (¹ H)
1	–	187.7	–	–
2	–	133.4	–	–
3	3.14, m	27.1	C-1, C-2, C-4, C-4a, C-9	4, 2'/6'
4	2.94, m	28.7	C-3, C-4a, C-5, C-8a	3, 5
4a	–	143.0	–	–
5	7.24, d (8.5 Hz)	128.0	C-4, C-7, C-8a	4, 6
6	7.48, t (8.5 Hz)	133.0	C-4a, C-8	5, 7
7	7.36, t (8.5 Hz)	126.8	C-5, C-8a	6, 8
8	8.13, d (8.5 Hz)	128.0	C-1, C-4a, C-6	7
8a	–	133.5	–	–
9	7.86, s	136.5	C-1, C-3, C-2'/6'	2'/6'
1'	–	128.3	–	–
2', 6'	7.43, m	131.6	C-9, C-2'/6'	3, 9, 3'/5'
3', 5'	6.96, m	113.9	C-1', C-3', C-4', C-5'	2'/6', CH ₃ O
4'	–	159.9	–	–
CH ₃ O	3.85, s	55.2	C-4'	3'/5'

A nice experiment: H2BC

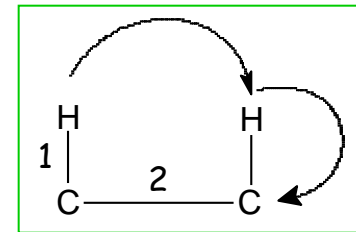
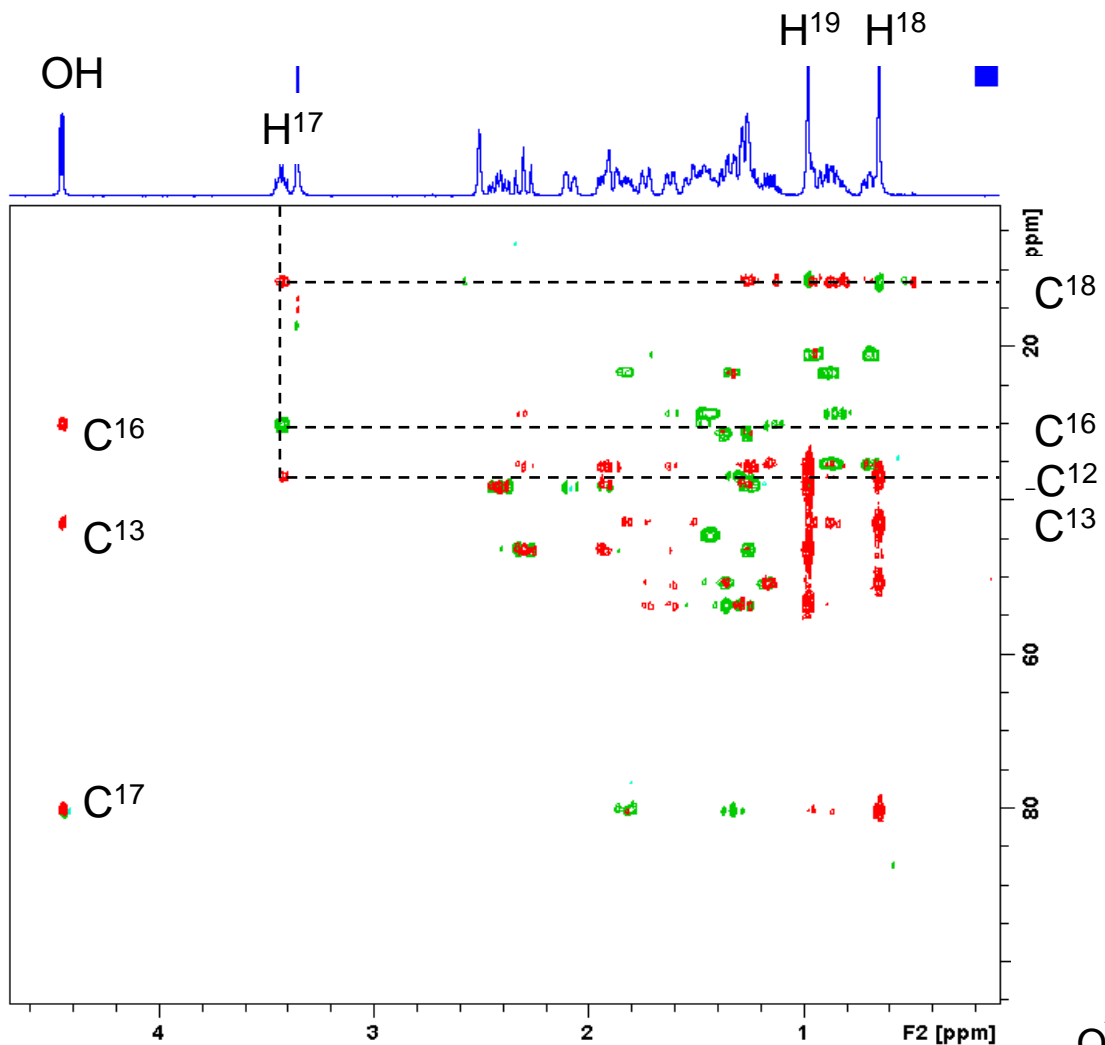
Heteronuclear 2-Bond C Correlations

- felerősíti a 2 kötésen keresztüli korrelációt
- elnyomja a többkötésen keresztüli korrelációt

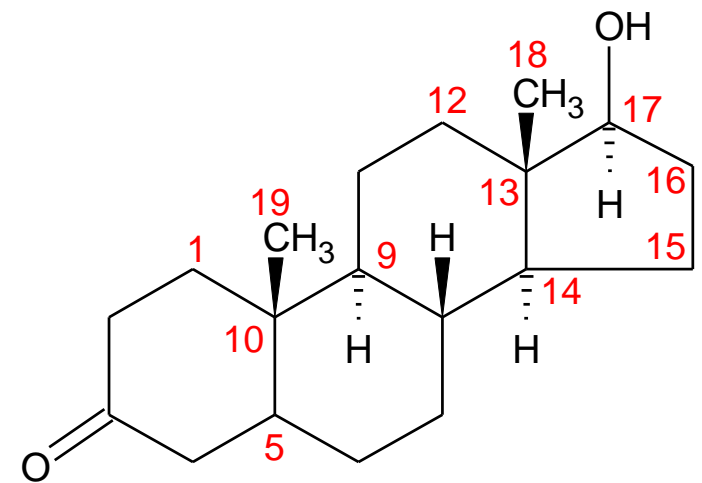


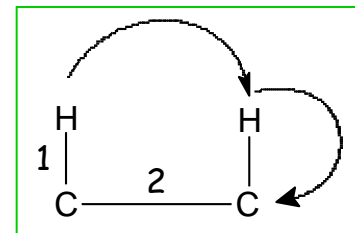
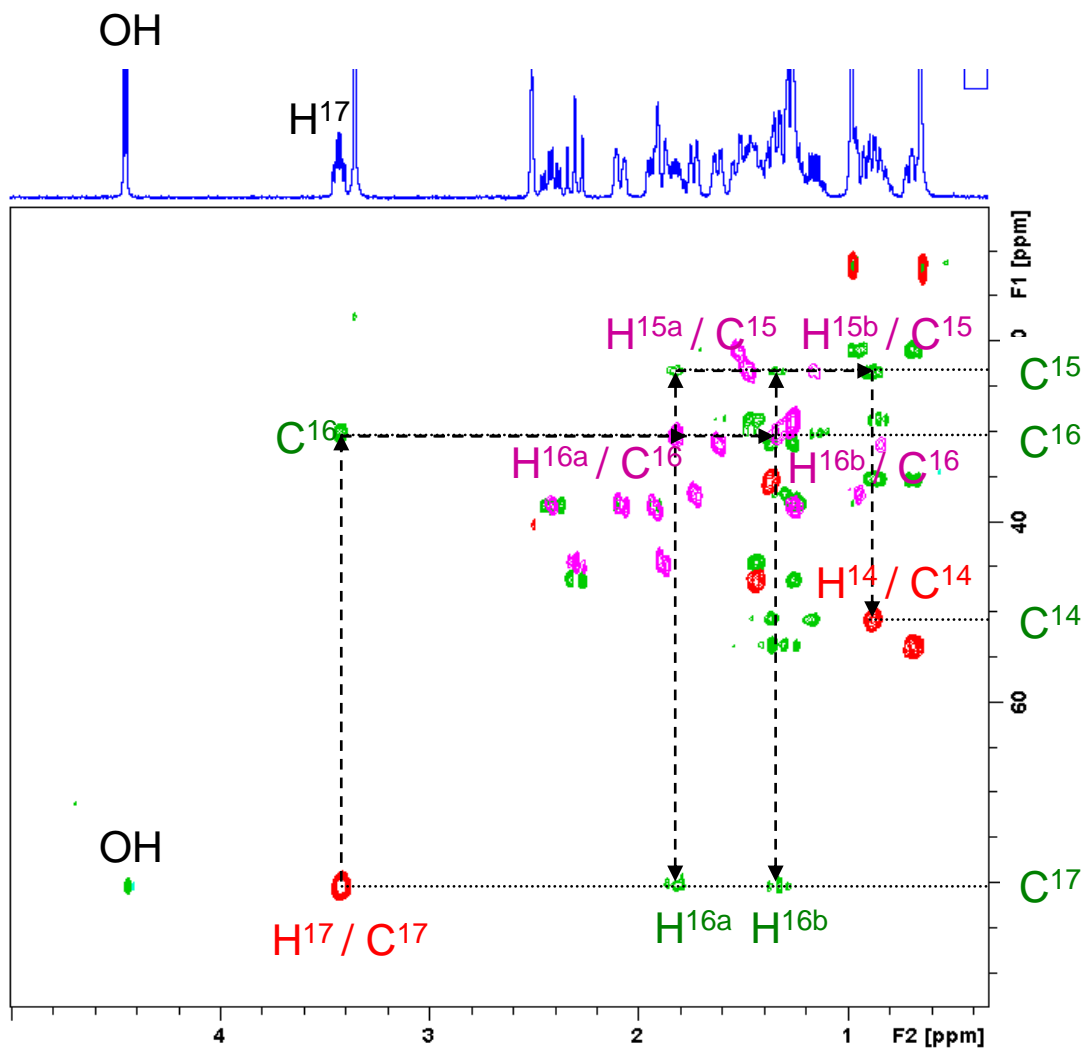
for more information:
www.crc.dk/nmr

3rd order low-pass J filter for correlating ^1H and ^{13}C nuclei
via $^1J_{\text{CH}}$ and $^1\text{H}-^1\text{H}$ couplings. **No $^2J_{\text{CH}}$ involved!**

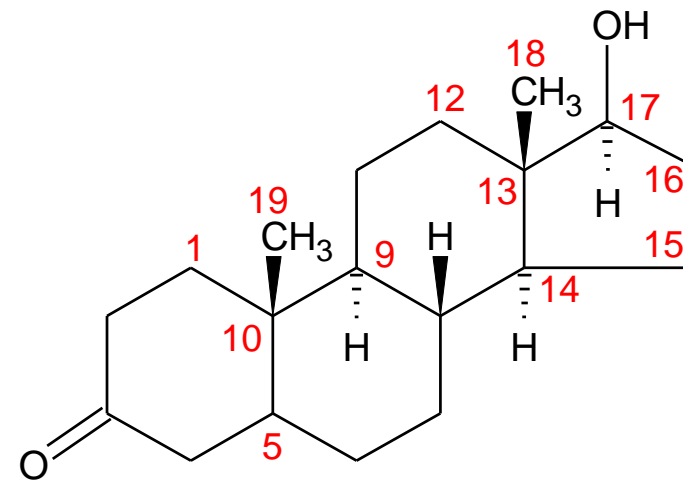


- Overlay **HMBC** / **H2BC**
- **3 bond** és **2 bond CP**
- megkülönböztethetők
- Két kötésen keresztüli **CP** akkor is fellép ha ${}^2J_{CH} \sim 0$ Hz

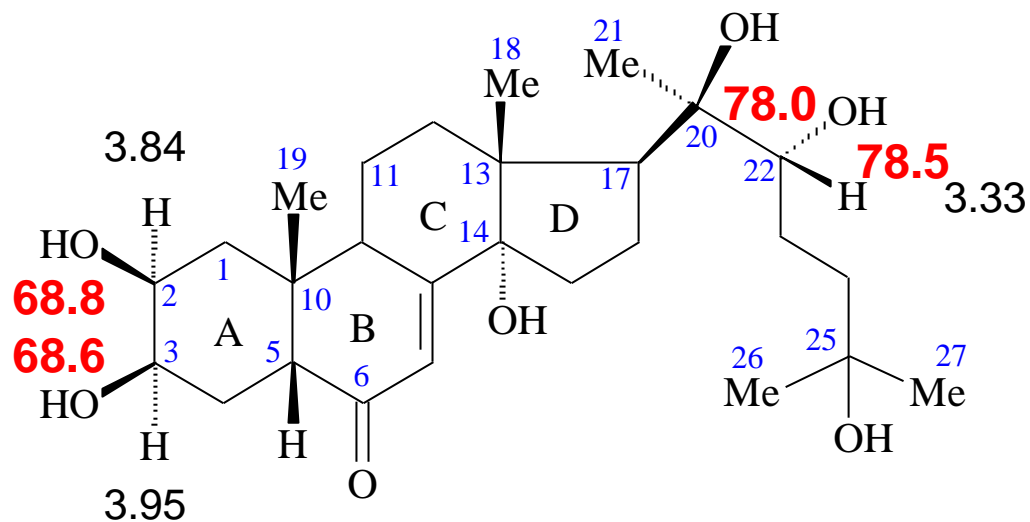




- Fektessük a **HSQC** a **H2BC**-re és így H,H COSY információt nyerünk
- Kiindulva pl. a **H¹⁷/C¹⁷** keresztcsúcsból a **H2BC** alapján H¹⁶ kémiai eltolódások adódnak
- Korreláció kvaterner C atomhoz nem lehetséges!

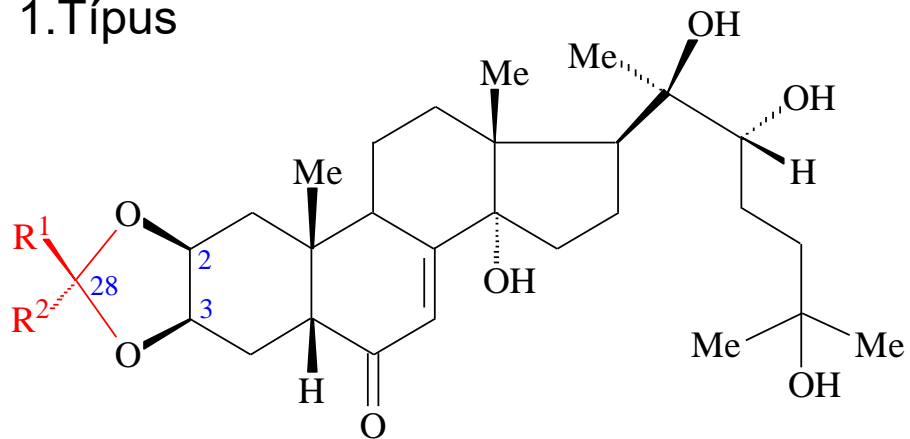


20-Hidroxiiekdizon dioxolánok NMR vizsgálata



20-Hidroxiiekdizon (δ^{1H} , δ^{13C})

1. Típus



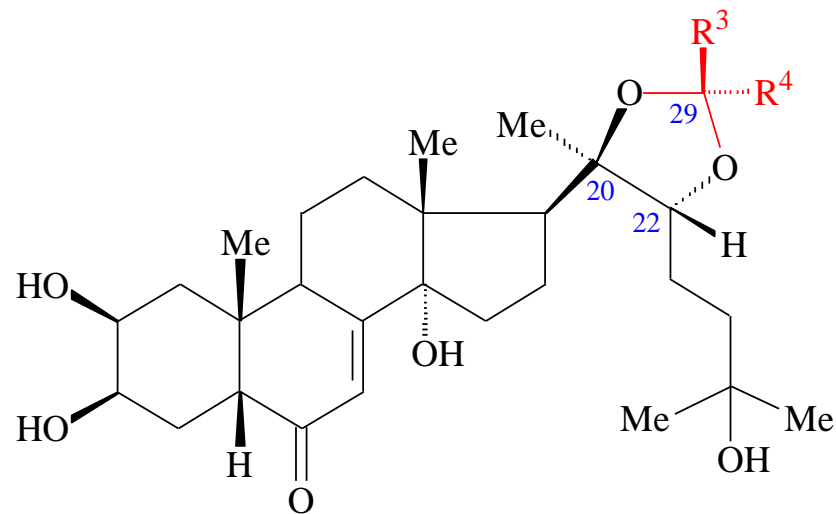
73-76

4.10-4.50

83-86

3.60-3.90

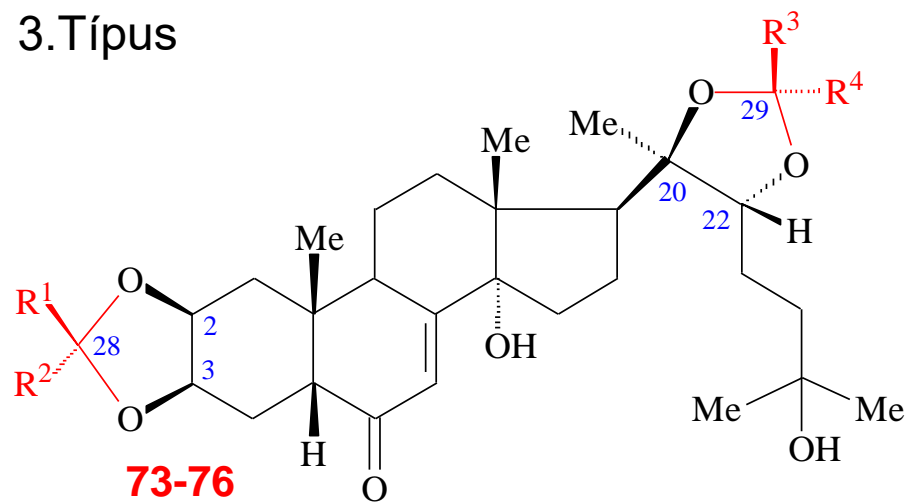
2. Típus



83-86

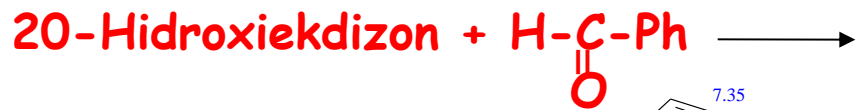
3.60-3.90

3. Típus



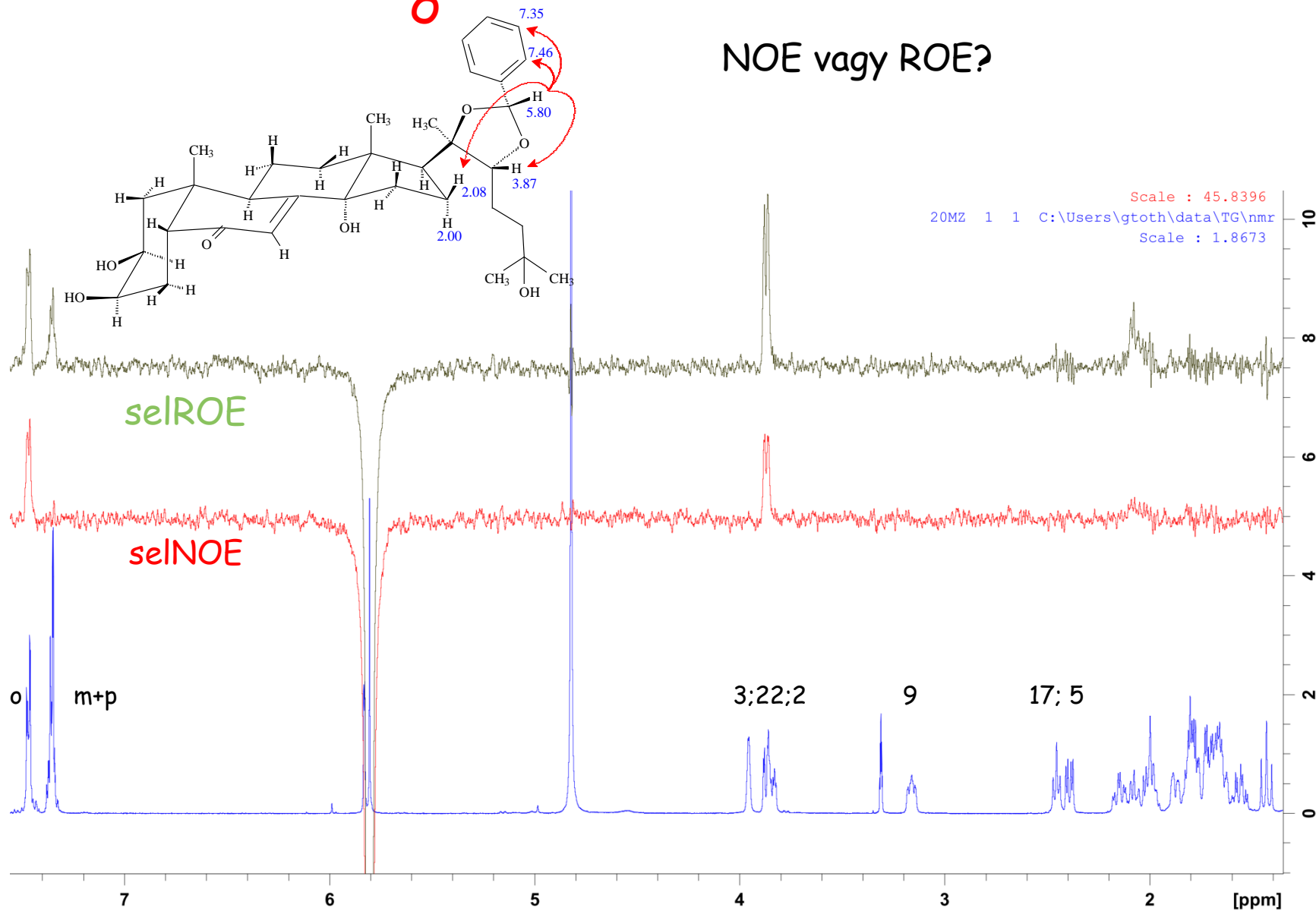
73-76

4.10-4.50

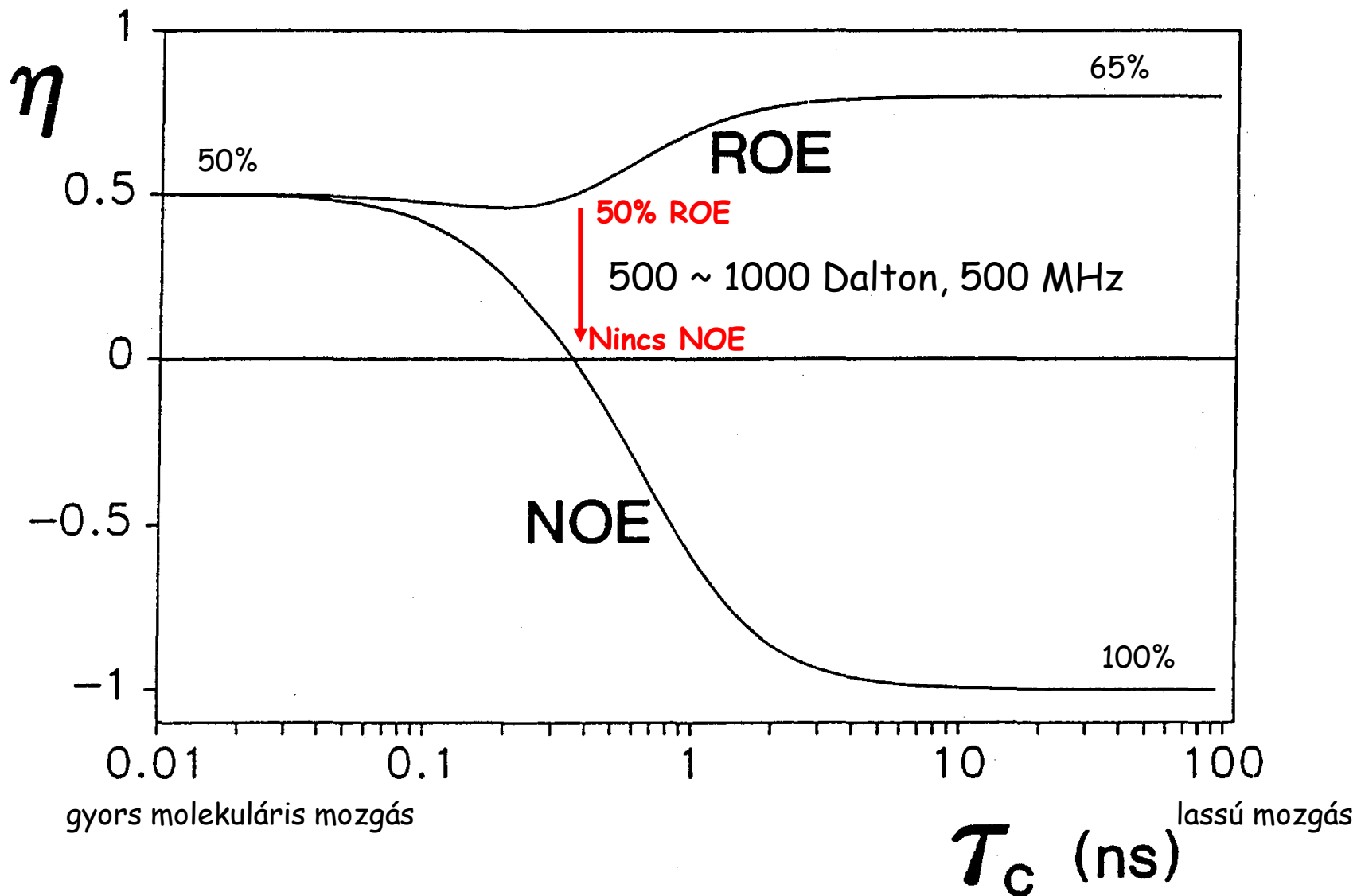


$\text{C}_{34}\text{H}_{48}\text{O}_7$; $M_s = 568$; DBE = 11

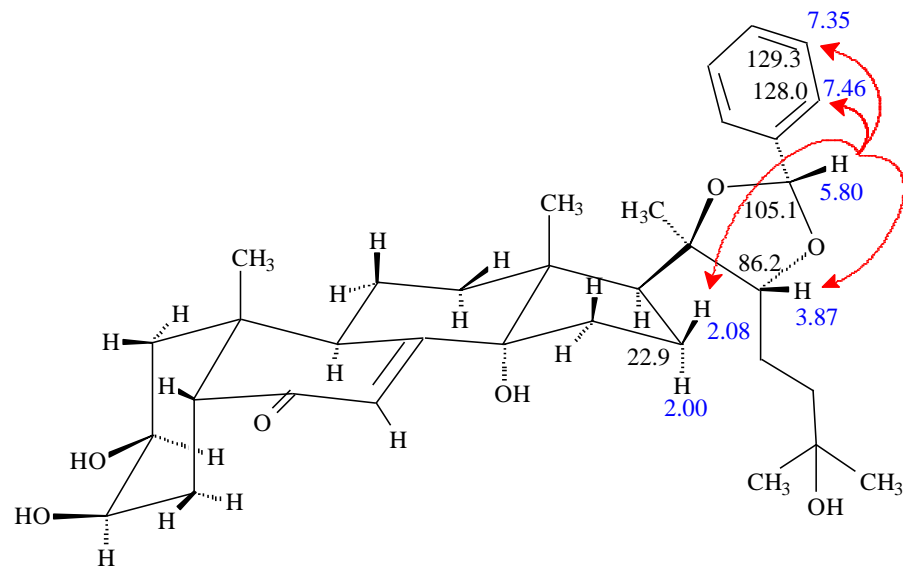
NOE vagy ROE?



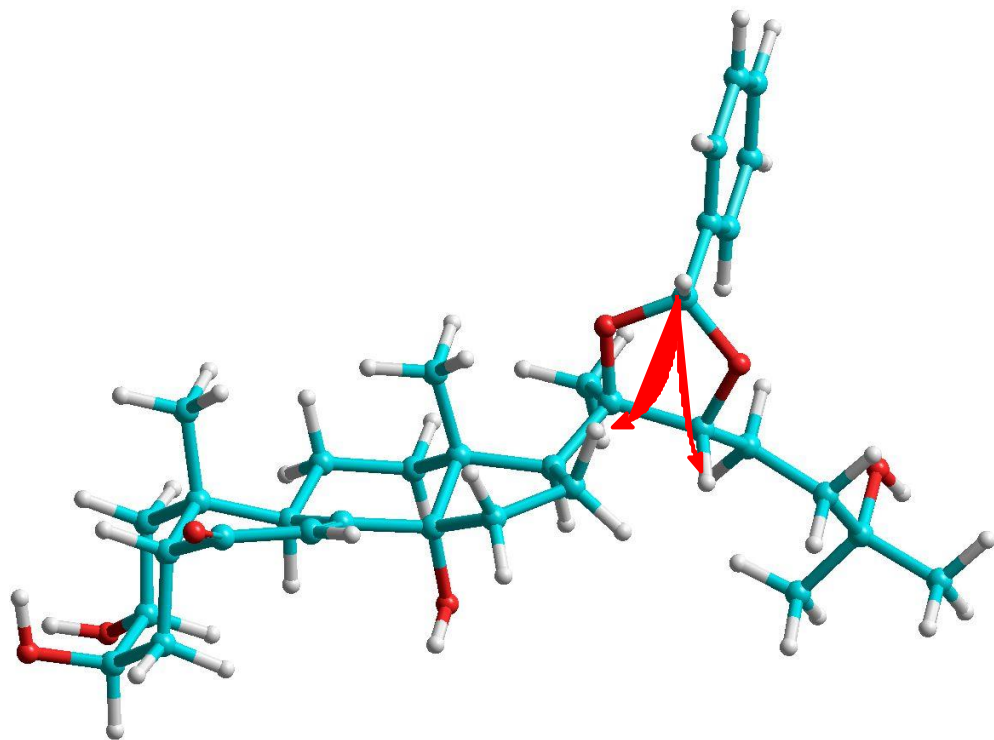
NOE/ROE intenzitás növekedés a korrelációs idő függvényében

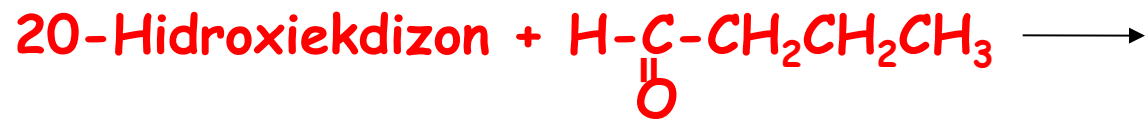


Sematikus térszerkezet
 ^1H kémiai eltolódások
H-29 selROE térközelségei

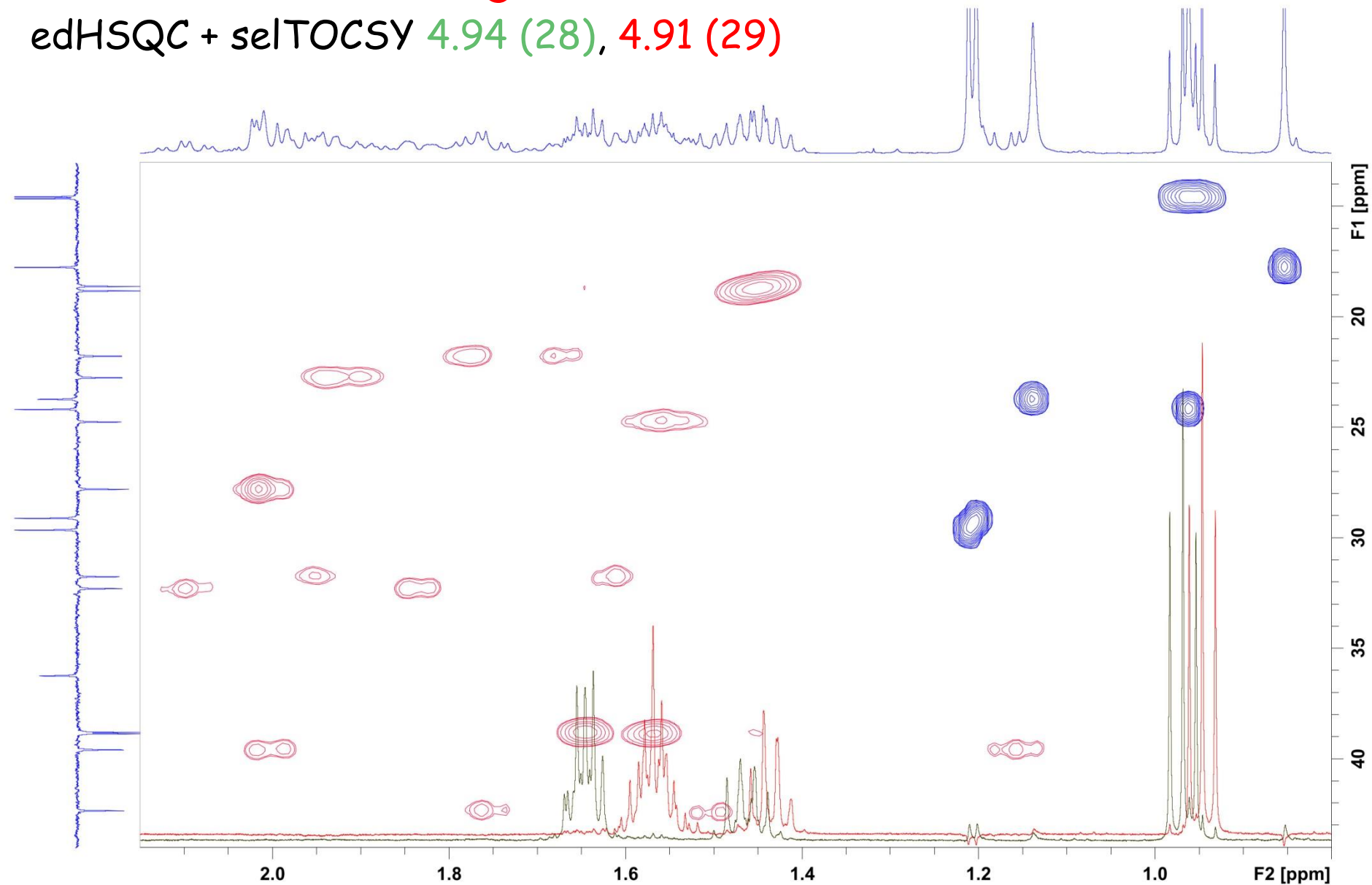


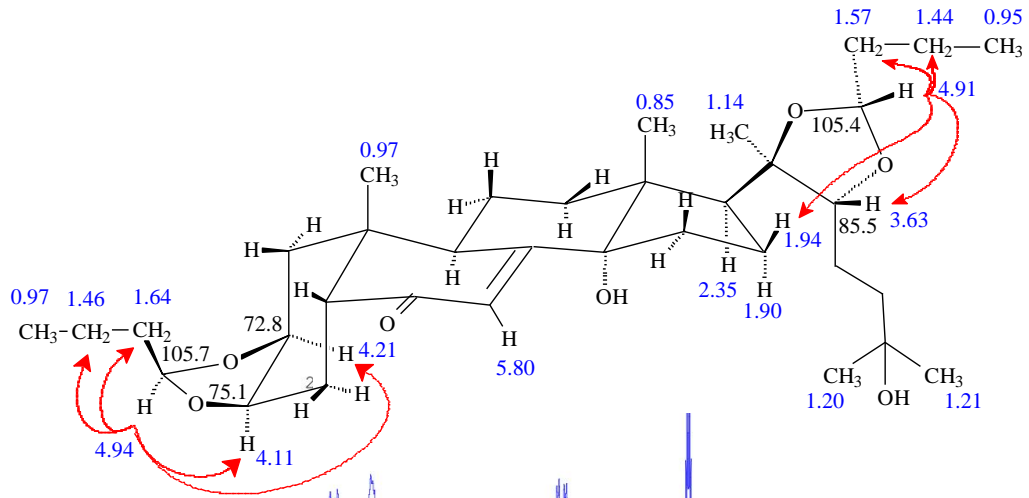
Hyperchem 8.0
PM3 szemiempirikus
finomított térszerkezet



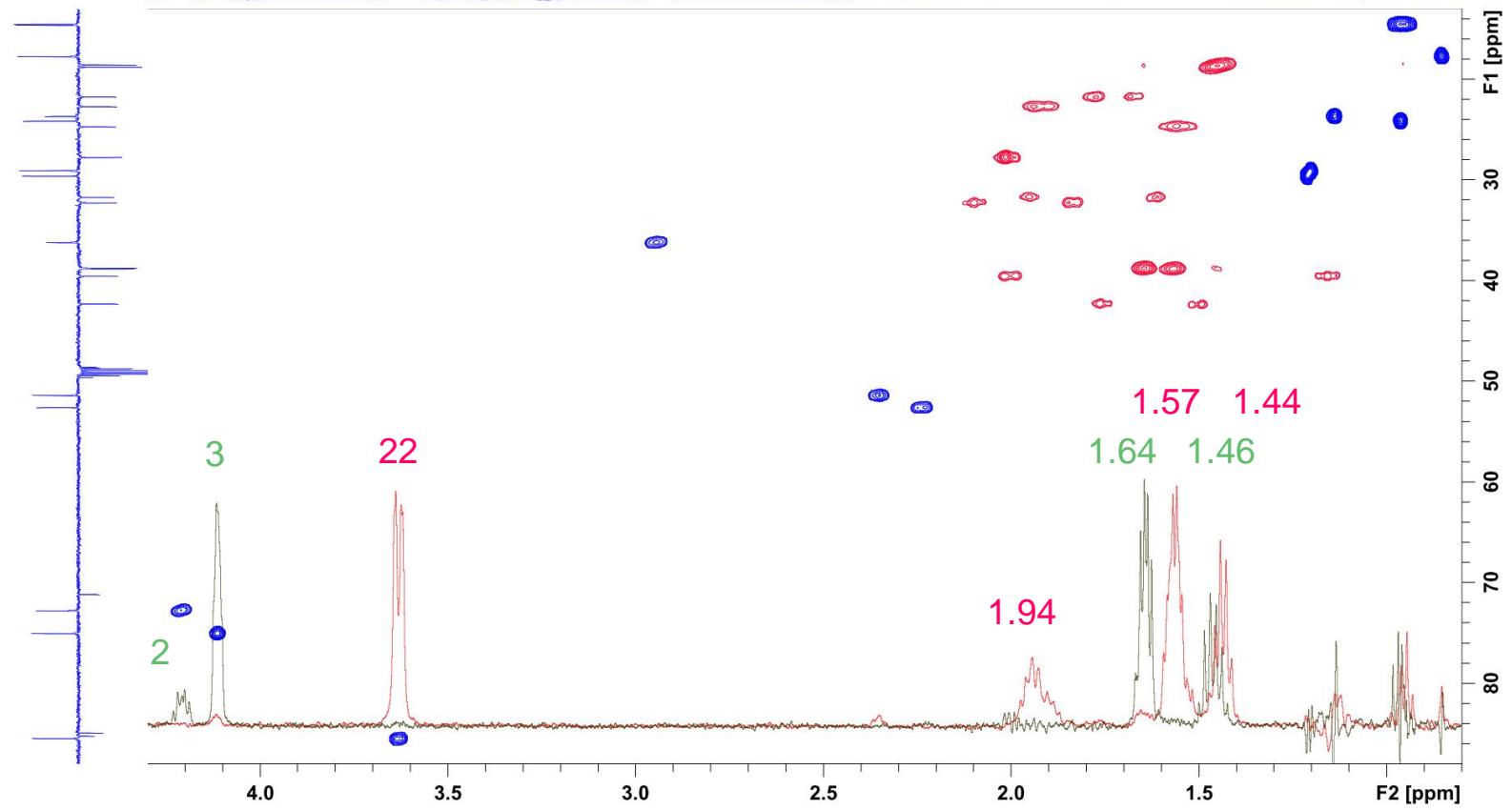
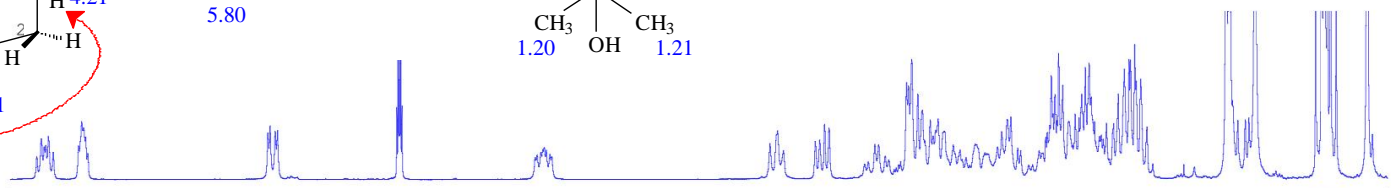


edHSQC + selTOCSY 4.94 (28), 4.91 (29)

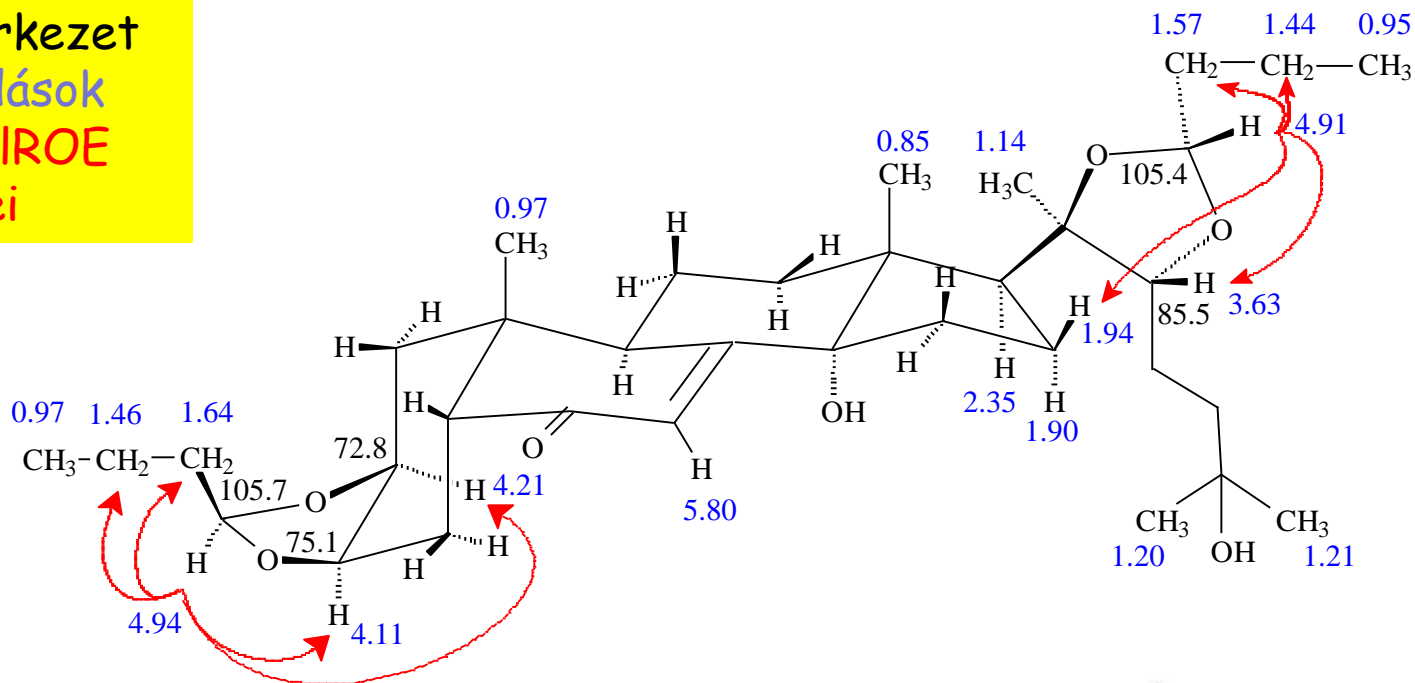




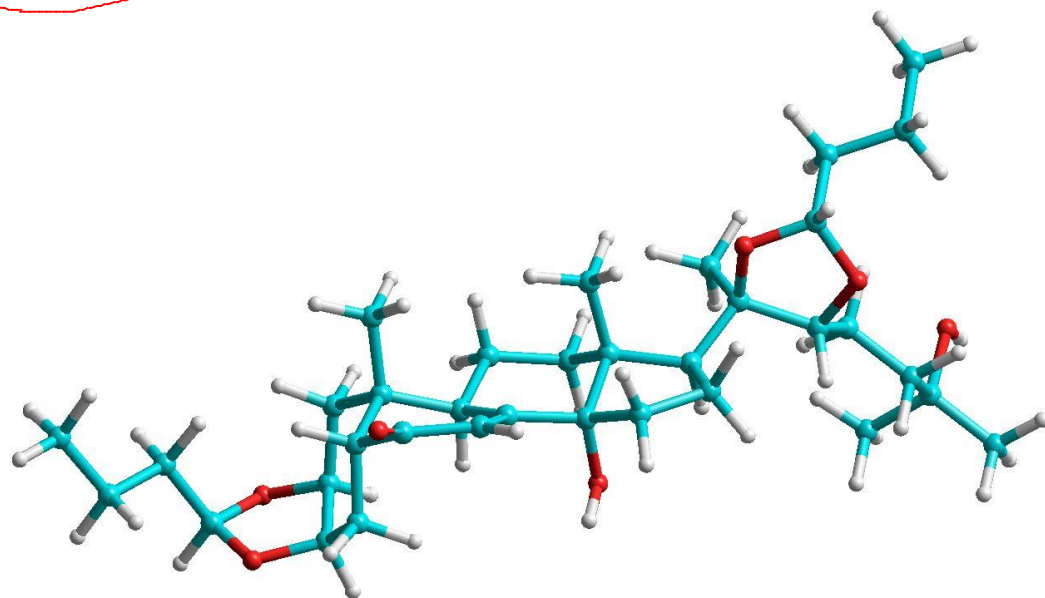
edHSQC + selROE: 4.94 (28),
4.91 (29),



Sematikus térszerkezet
 ^1H kémiai eltolódások
 H-28 és H-29 selROE
 térközelségei



Hyperchem 8.0
 PM3 szemempirikus
 finomított térszerkezet



Bruker Avance 500 (2002); 5mm BBO (direkt) mérőfej; S/N : $^1\text{H} = 350$, $^{13}\text{C} = 230$

MATCH tubes (mérőcsővek)

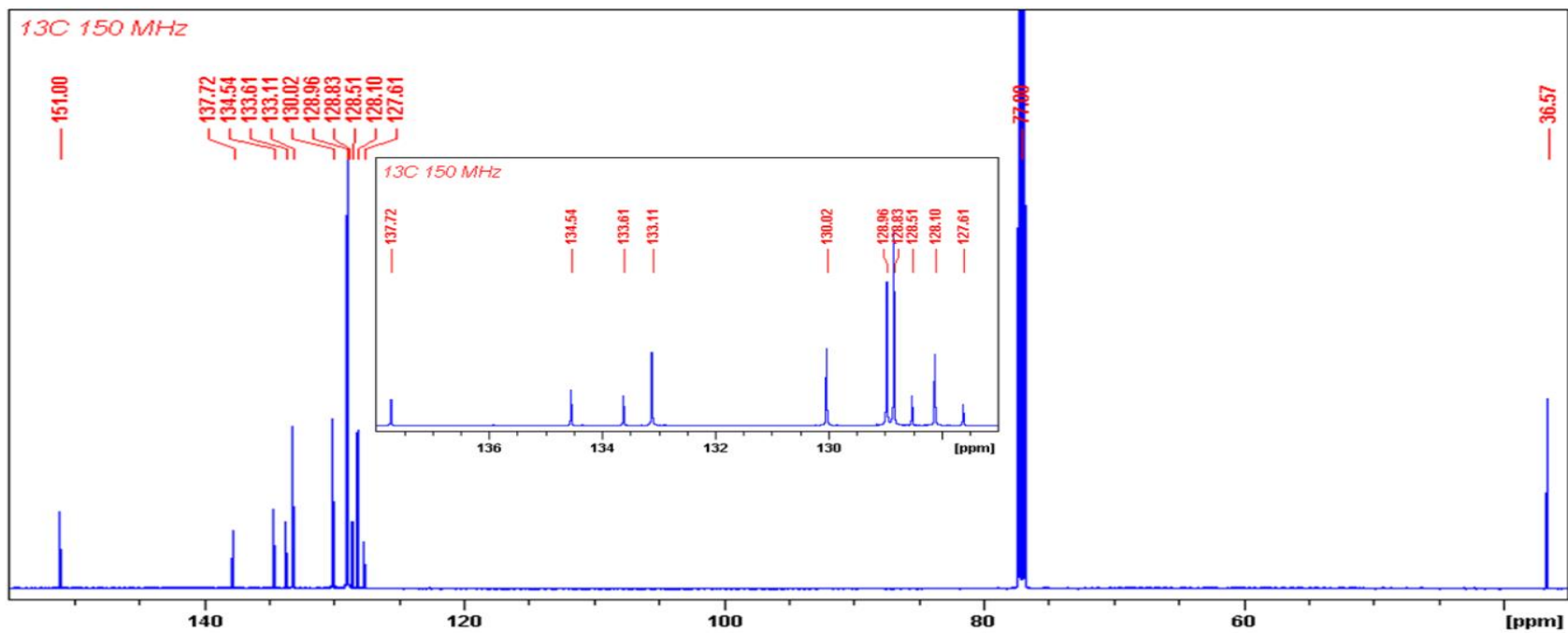
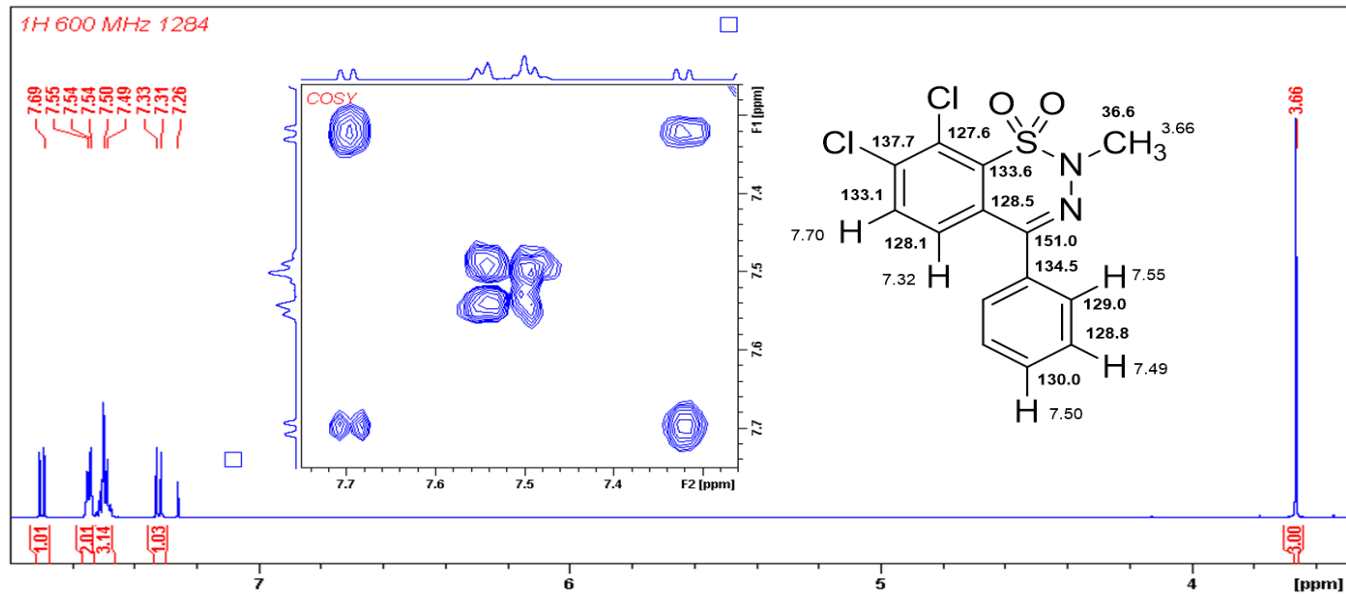
Átmérő: 1, 1.7, 2.0, **2.5**, 3.0, 4.25 and 5mm

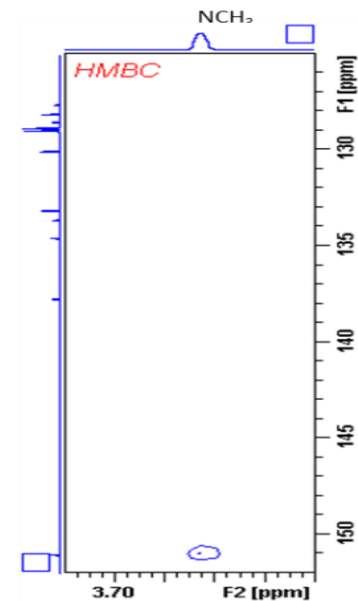
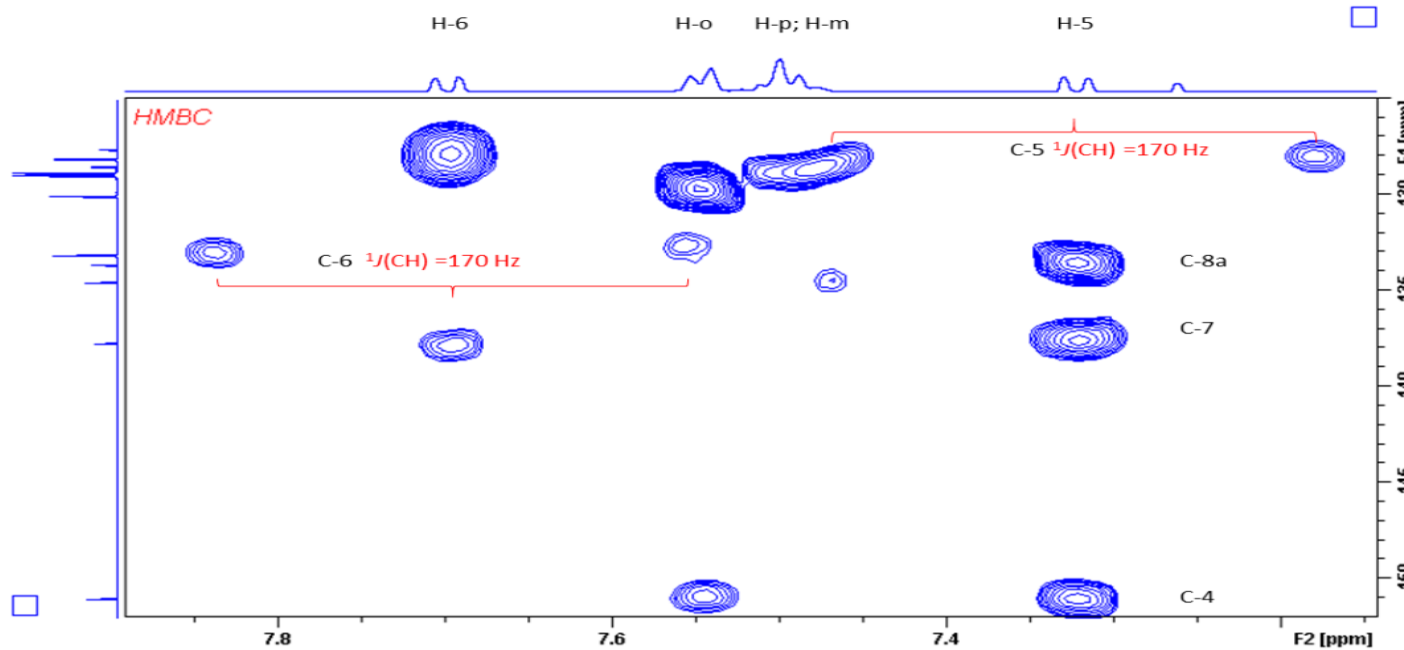
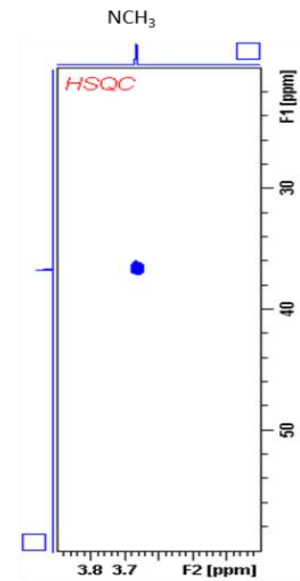
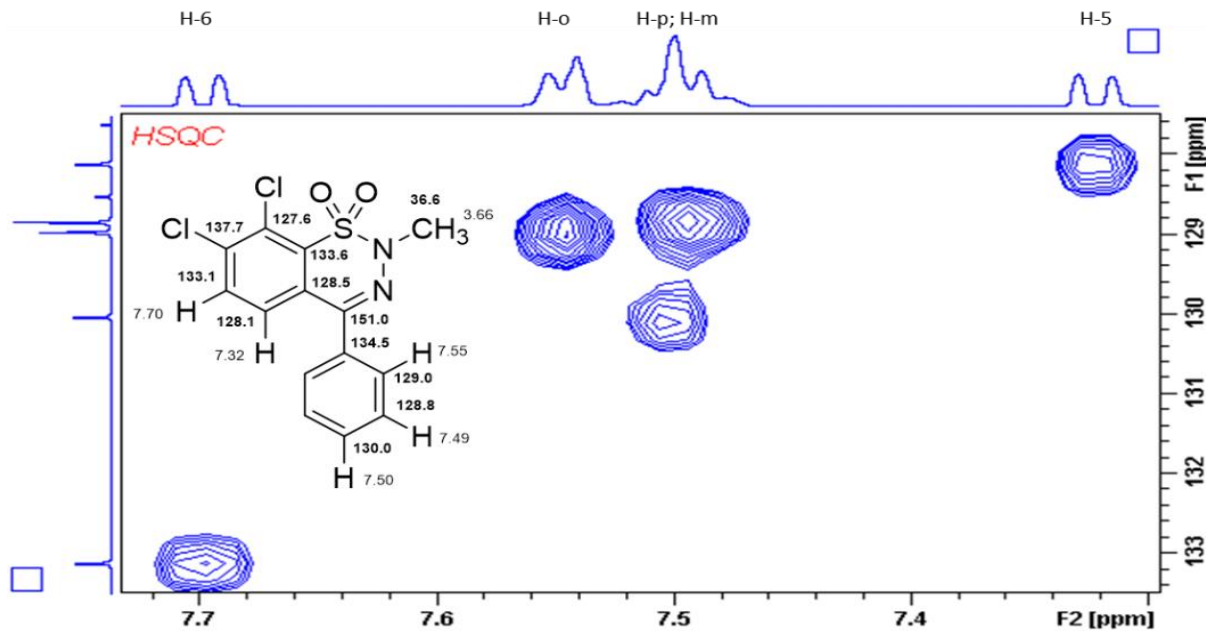
térfogat \longrightarrow >0.1 ml

0.6 ml

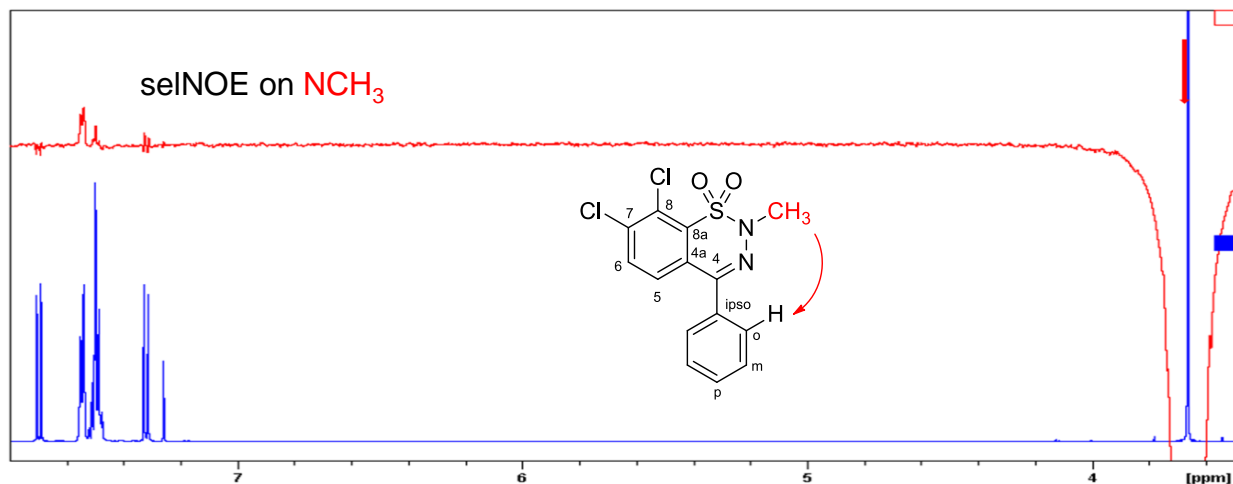
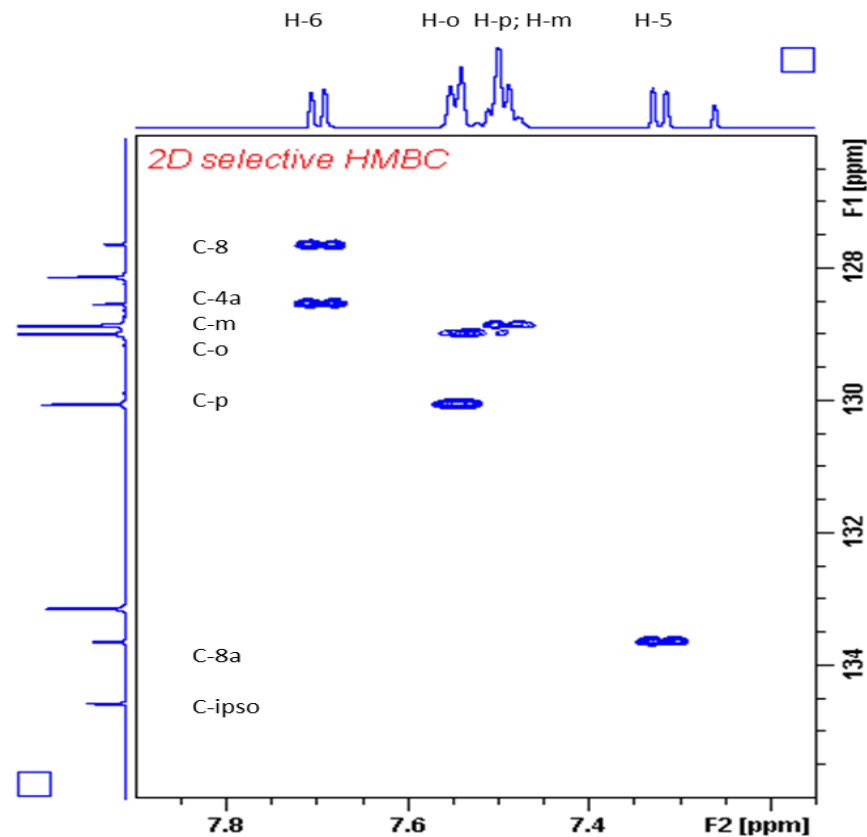
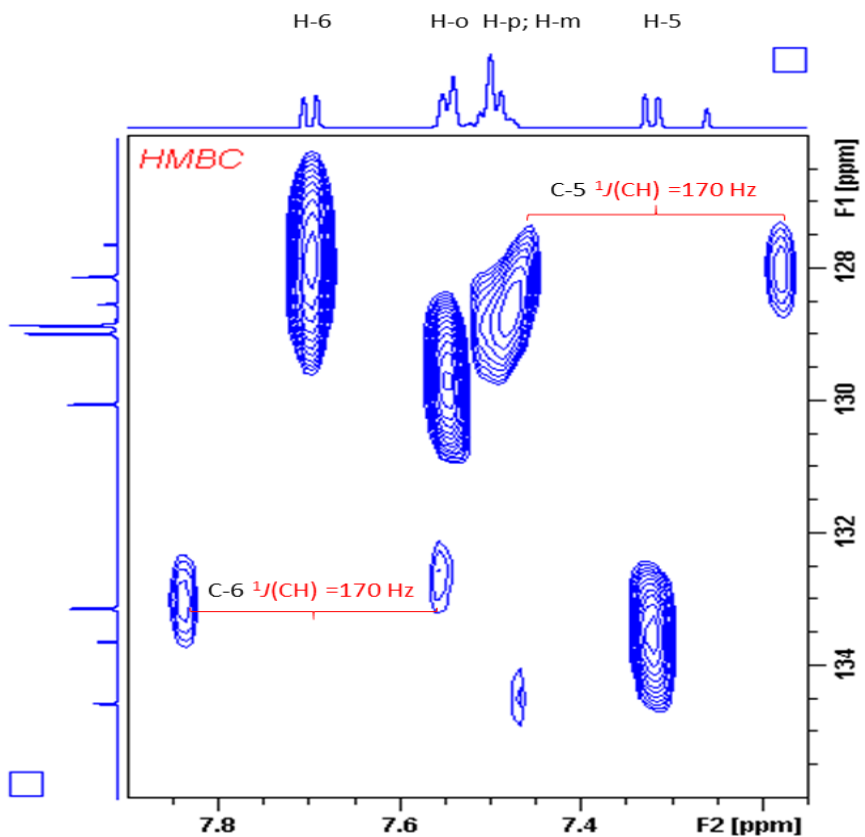
edHSQC, HMBC mérésidő : **6 perc**, selROE ns=32 **>3 perc**



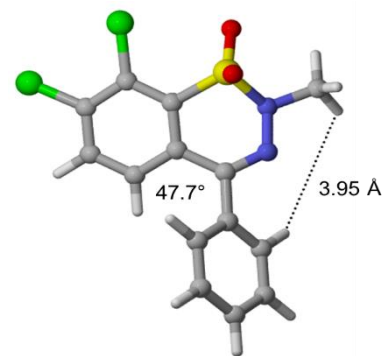




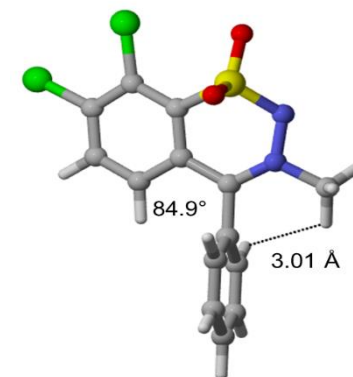
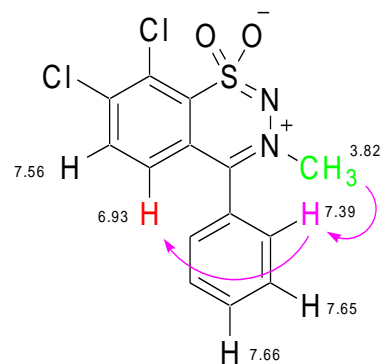
Extrém nagy felbontás az F1 (^{13}C) tartományban a sávszelektív HMBC módszerrel



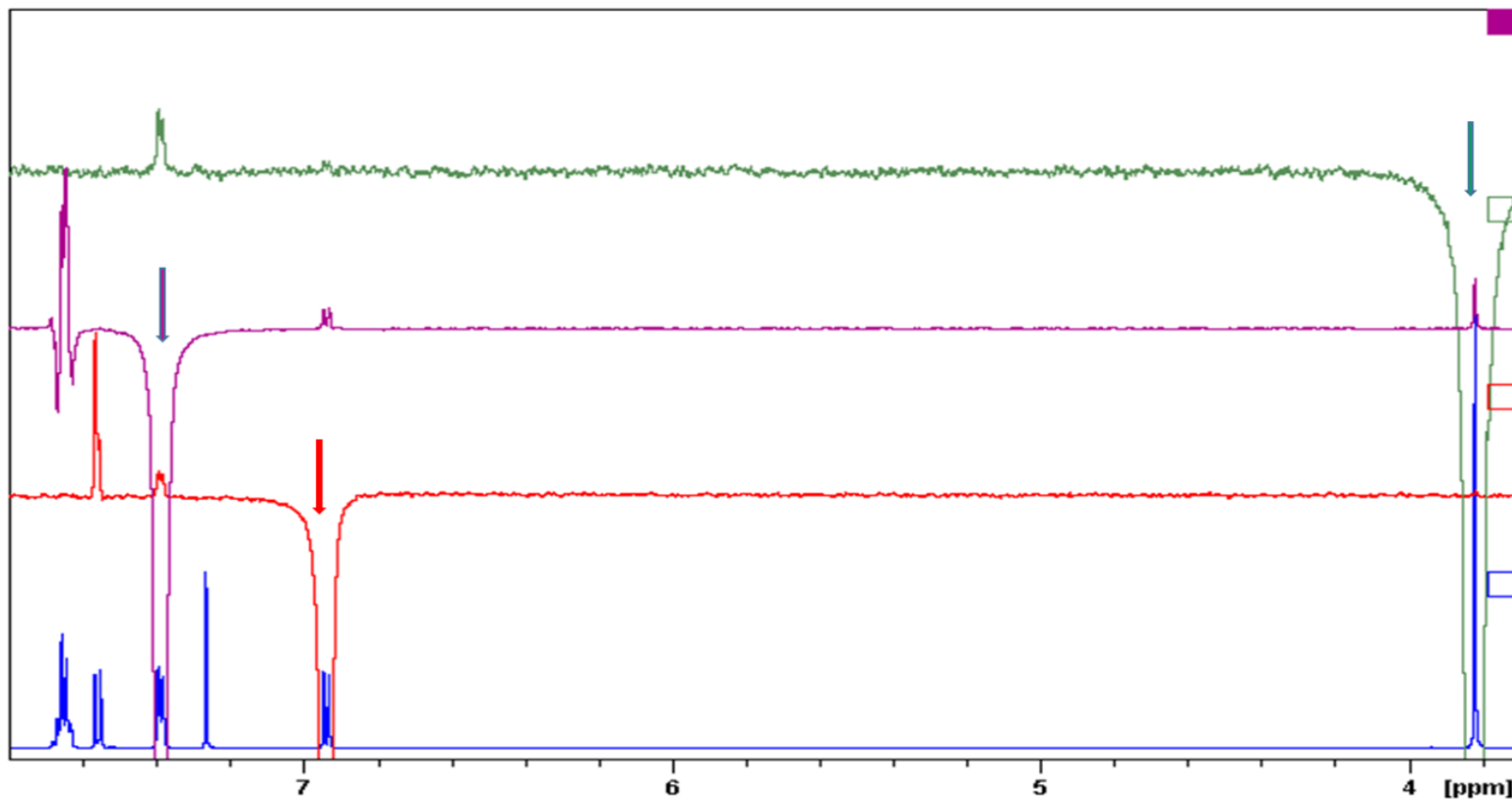
DFT geometria

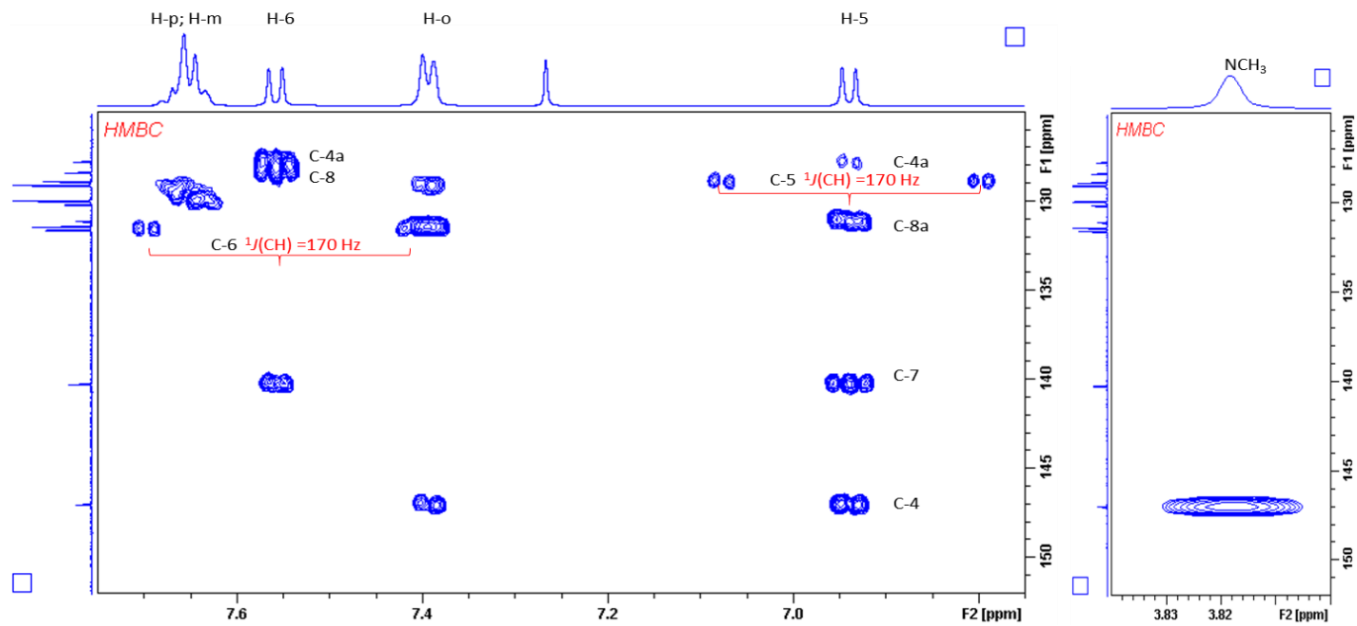
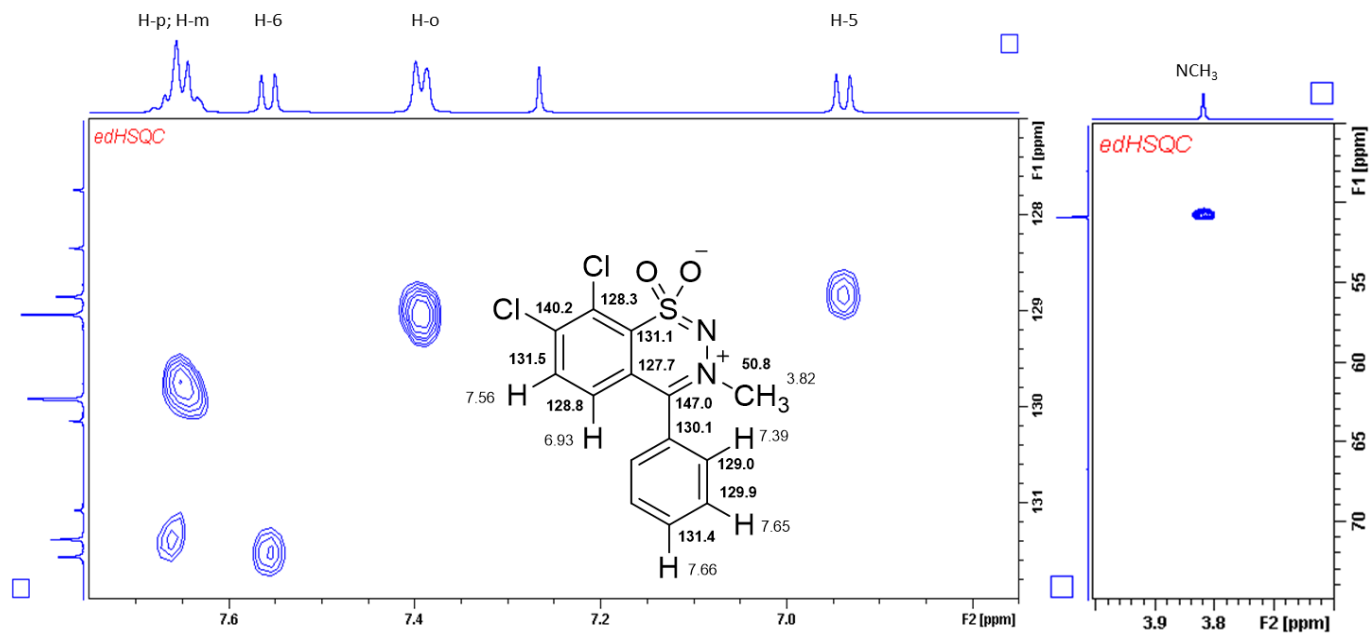


DFT geometria

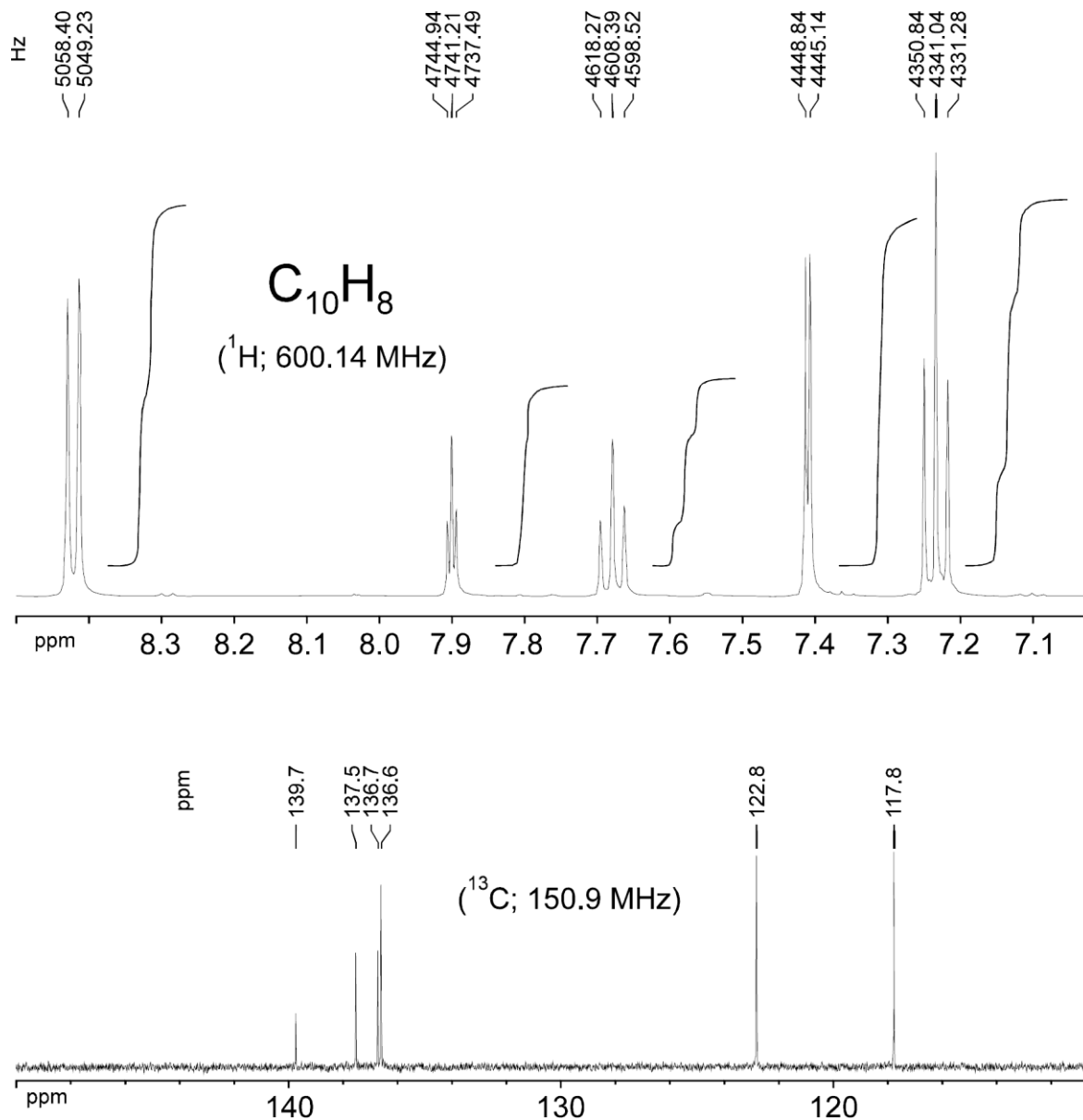


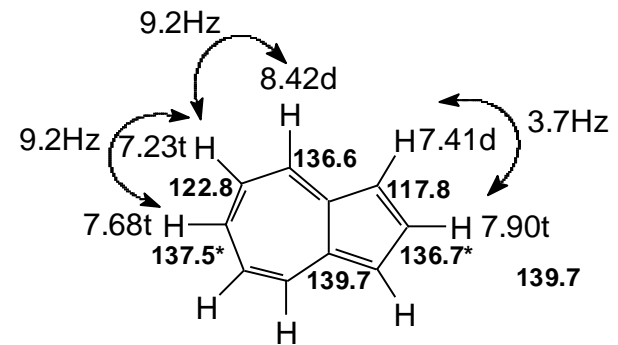
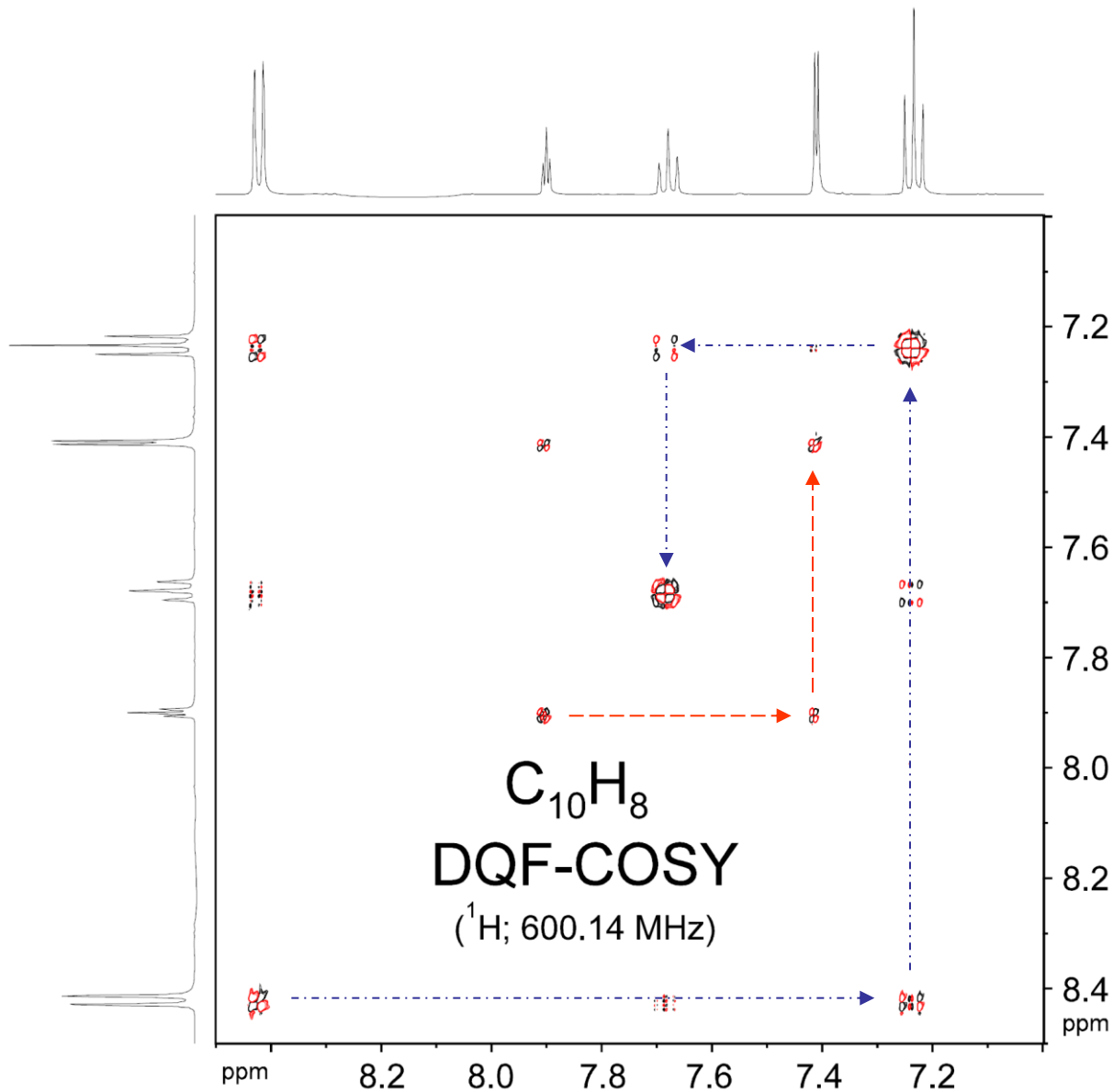
¹H + seINOE on NCH₃, H-ortho and H-5

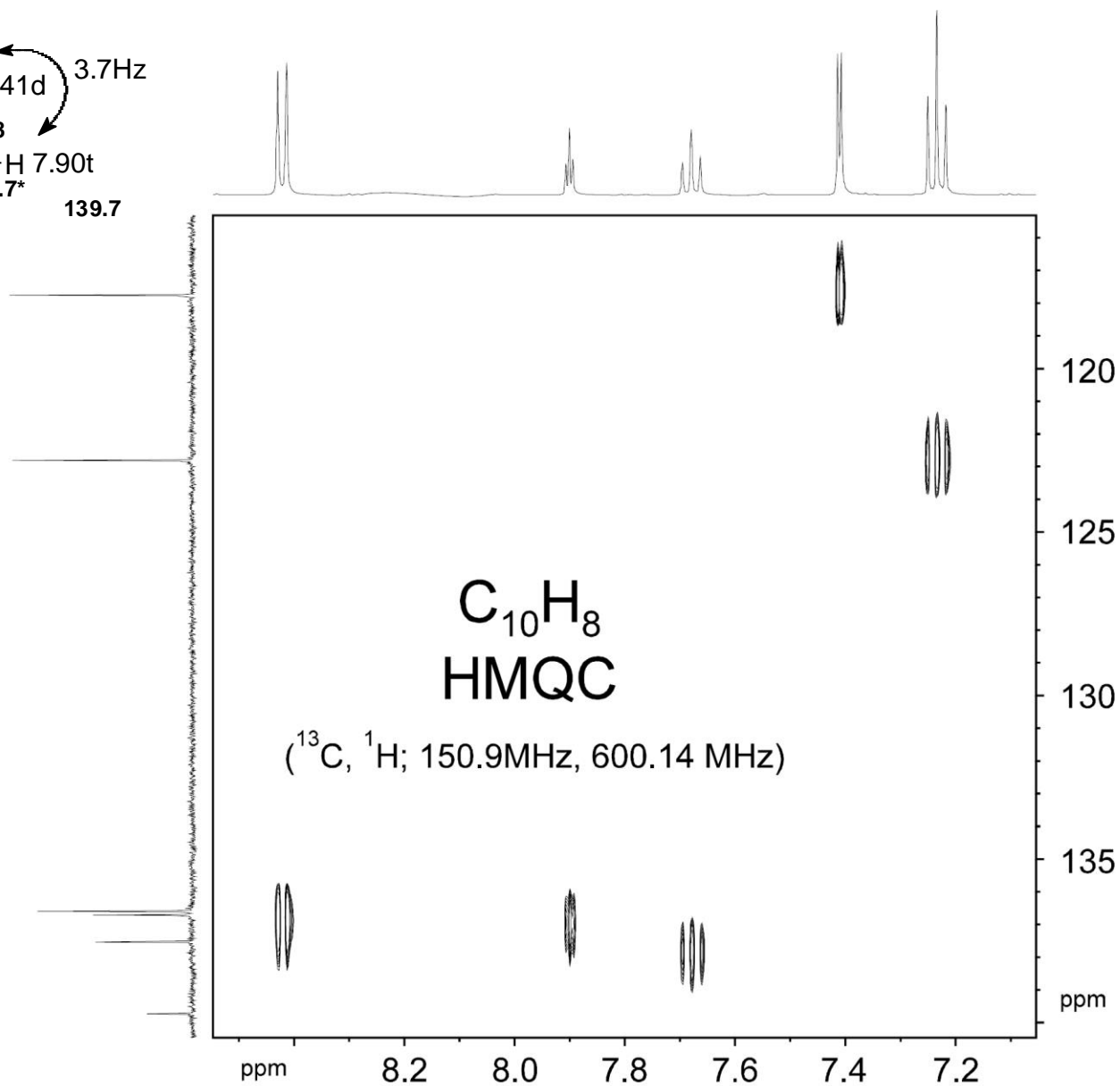
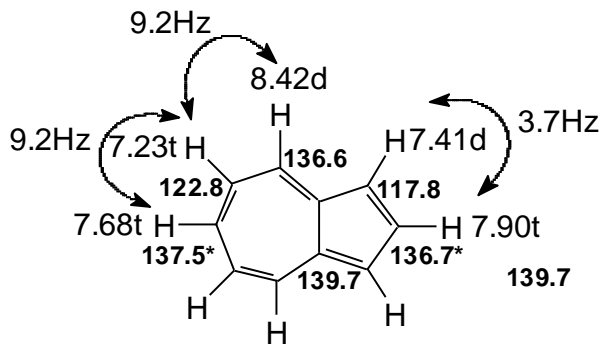




Írja fel a szerkezetet és adja meg az ^1H (és ^{13}C) kémiai eltolódásokat, valamint a $J(\text{H},\text{H})$ csatolási állandókat!







Compound **34** has been isolated from the phenolic fraction of the plant *Polygala arvensis*. Its molecular formula – determined by high-resolution mass spectrometry – is $C_{21}H_{20}O_5$. What is its structure?

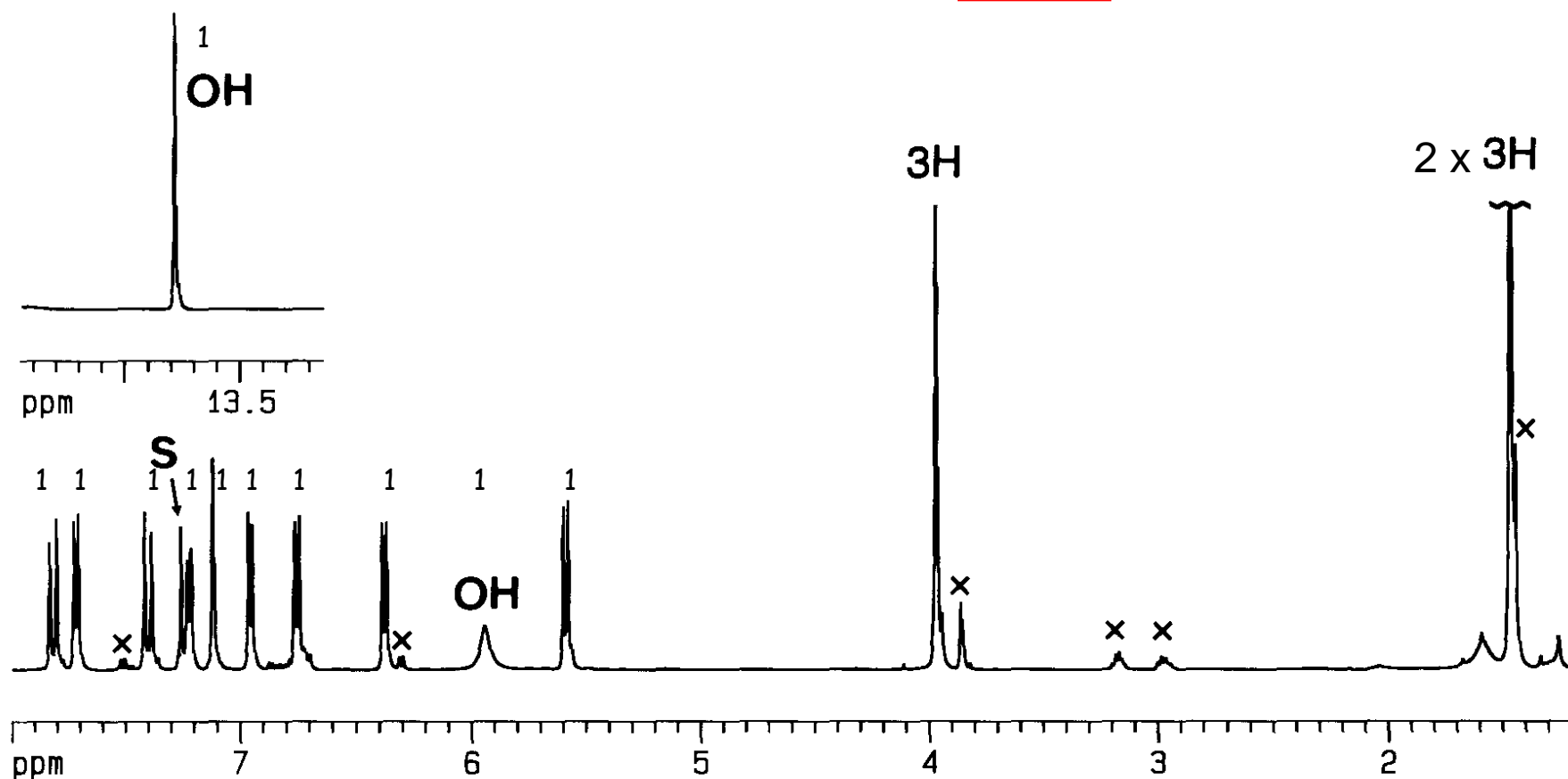


Fig. 4.25.1. 500 MHz ^1H NMR spectrum of **34**, in CDCl_3 ; impurity signals are marked by "x"; S: solvent. The following signal splittings (doublets) have been determined: $\delta = 7.82$, 15.3 Hz; $\delta = 7.72$, 8.8 Hz; $\delta = 7.40$, 15.3 Hz; $\delta = 7.23$, 8.2 Hz; $\delta = 6.96$, 8.2 Hz; $\delta = 6.76$, 10.0 Hz; $\delta = 6.38$, 8.8 Hz; $\delta = 5.59$, 10.0 Hz. For an expansion see 1D spectra in Fig. 4.25.3.

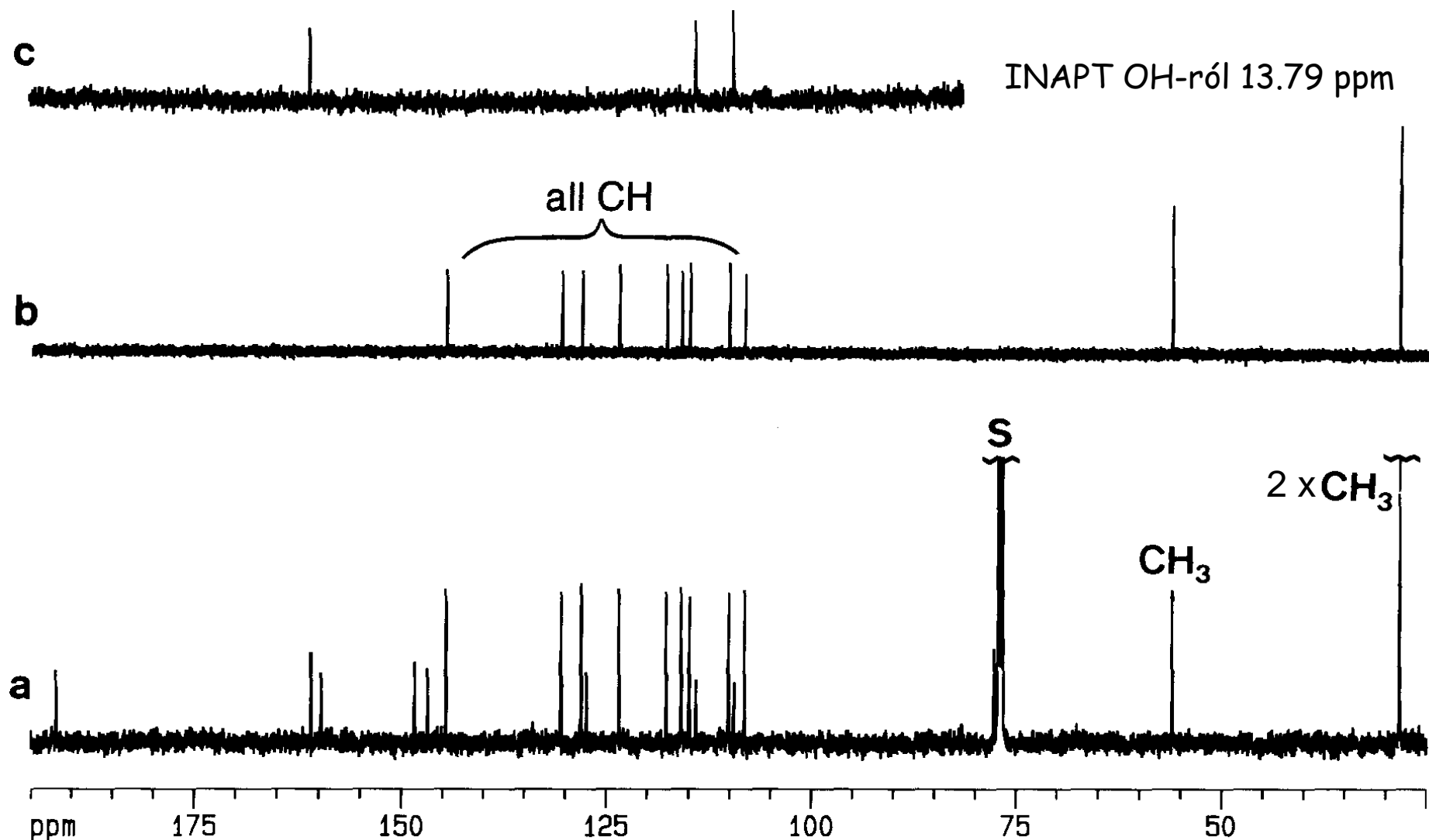


Fig. 4.25.2.a 125.7 MHz ^{13}C NMR spectrum of **34**, in CDCl_3 ; **b** DEPT135 spectrum; **c** selective INAPT spectrum, irradiation at the signal at $\delta = 13.79$. The exact values of the ^{13}C chemical shifts are: $\delta = 191.9, 160.9, 159.7, 148.4, 146.8, 144.6, 130.5, 128.1, 127.4, 123.5, 117.7, 115.9, 114.9, 114.1, 110.1, 109.4, 108.2, 77.8, 56.0,$ and 28.4 .

Fig. 4.25.3. Expanded section of $^1\text{H},^1\text{H}$ -COSY

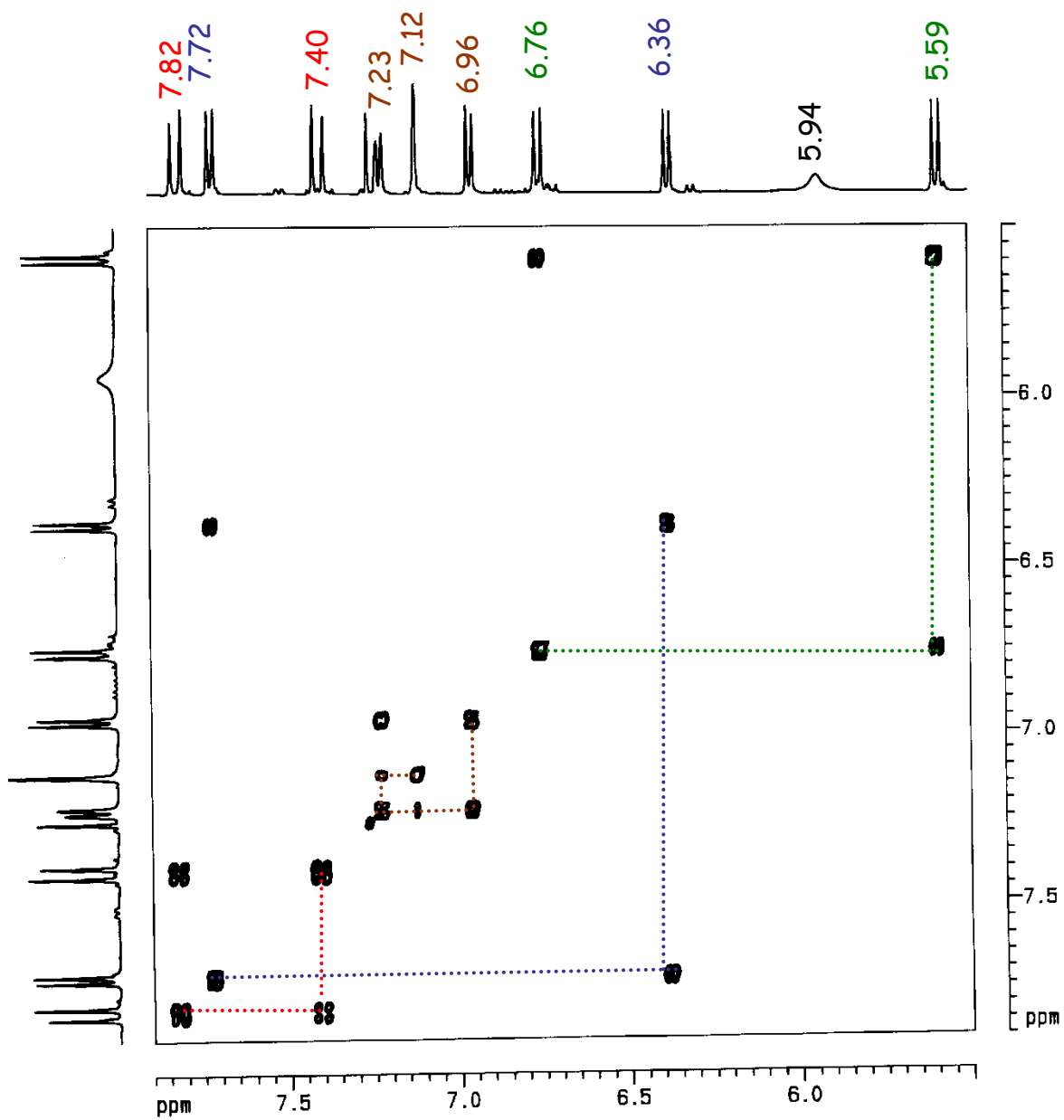


Fig.4.25.4. HMQC

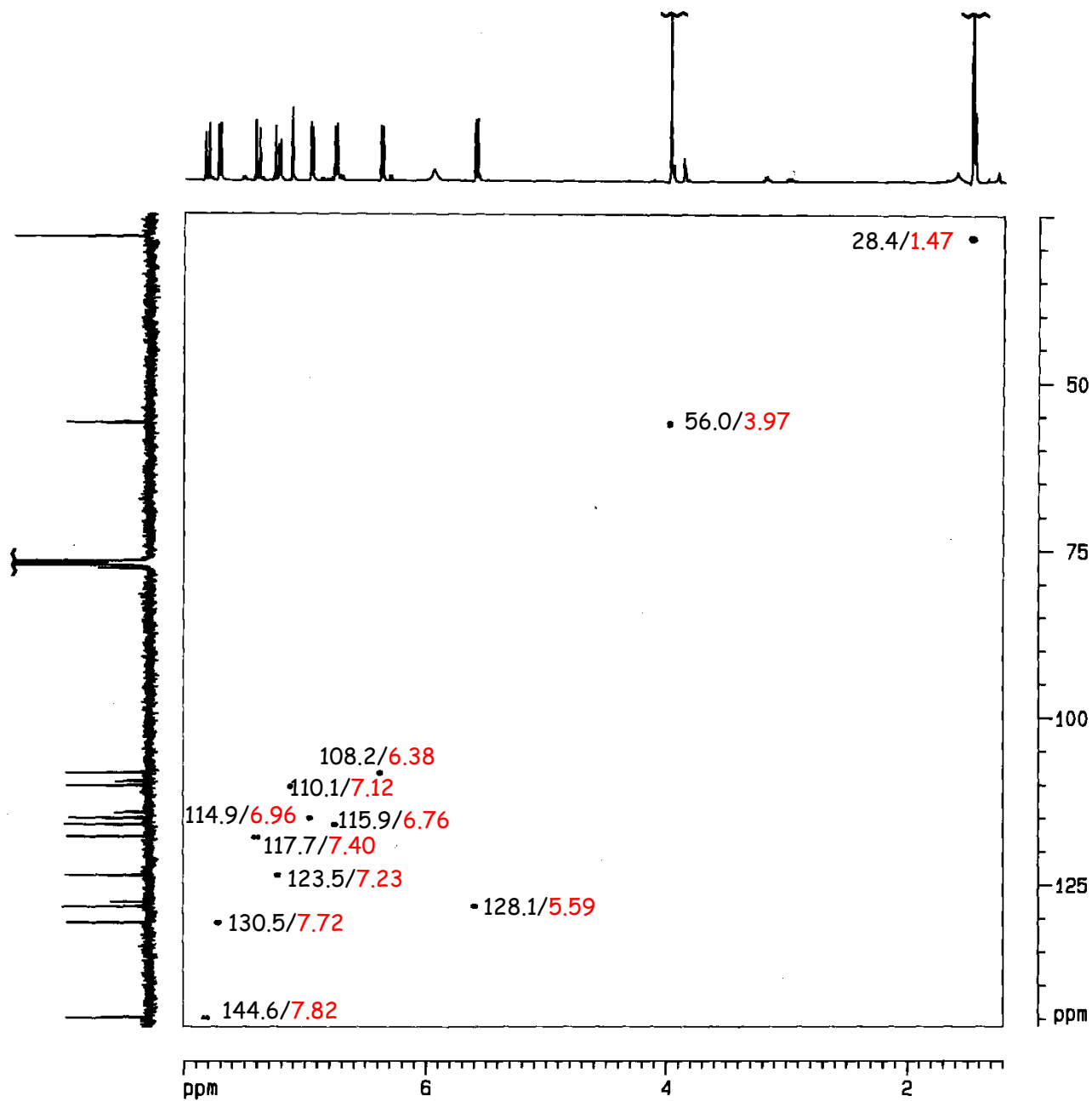


Fig. 4.25.5 HMBC

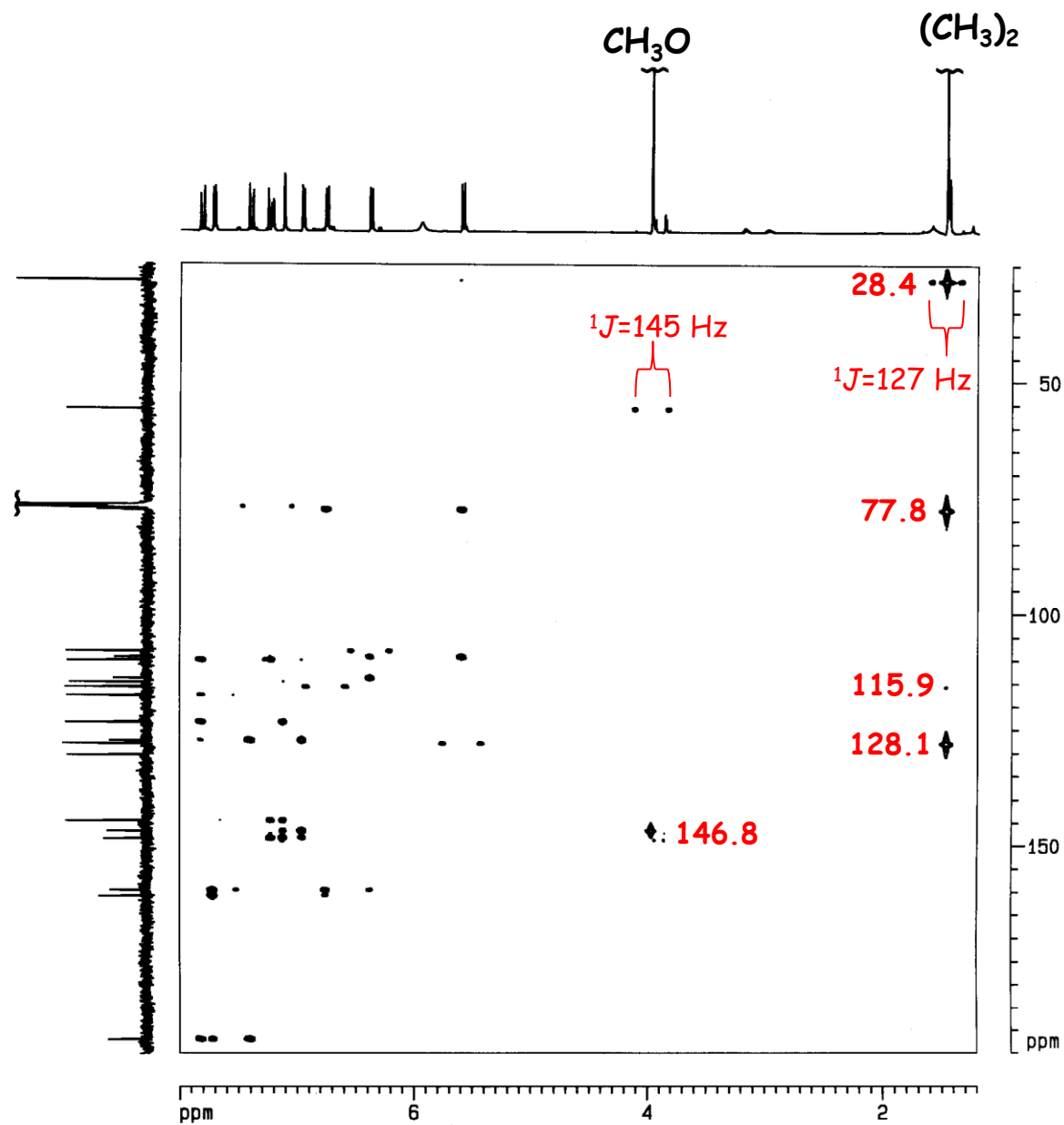


Fig. 4.25.6 HMBC széthúzás

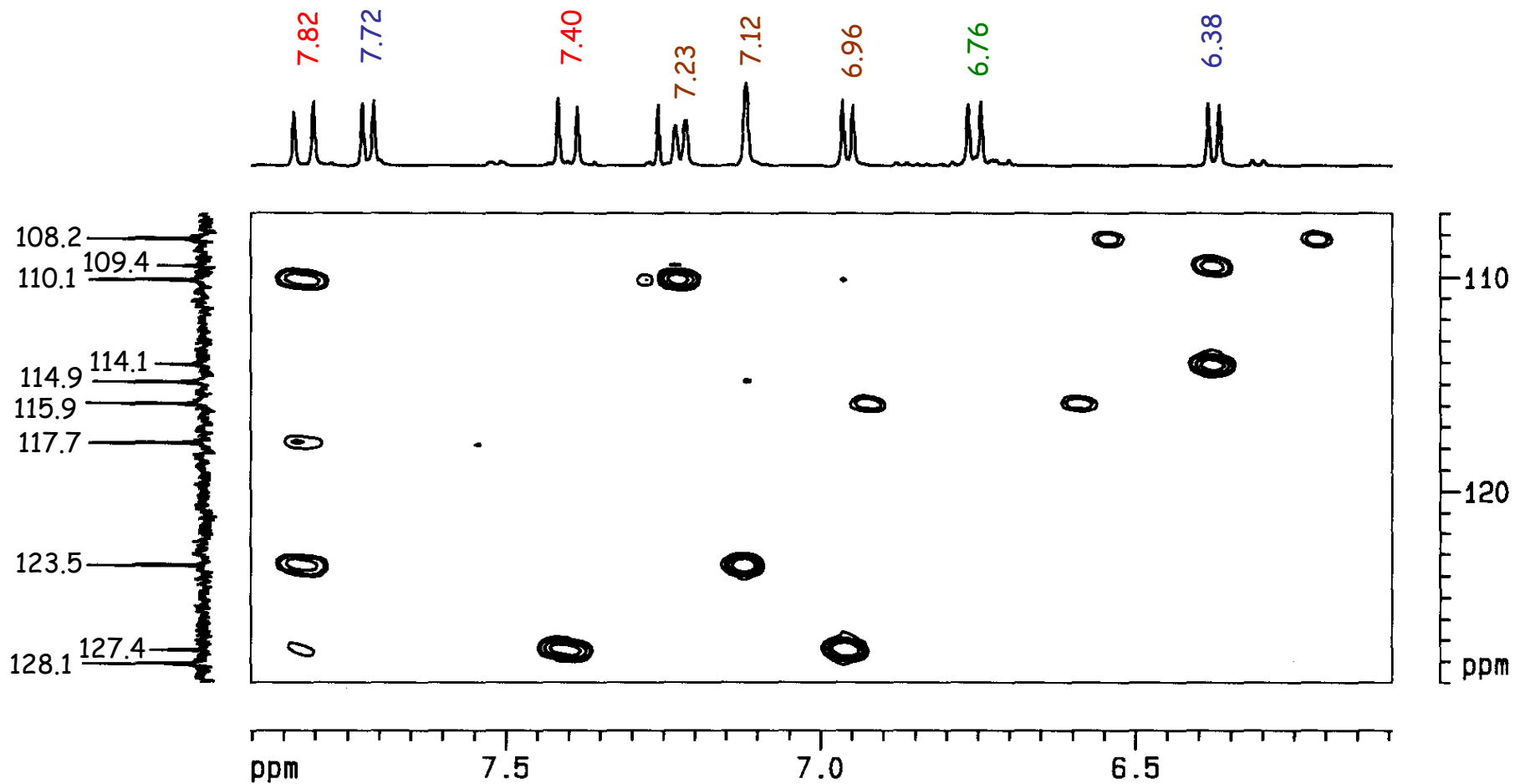
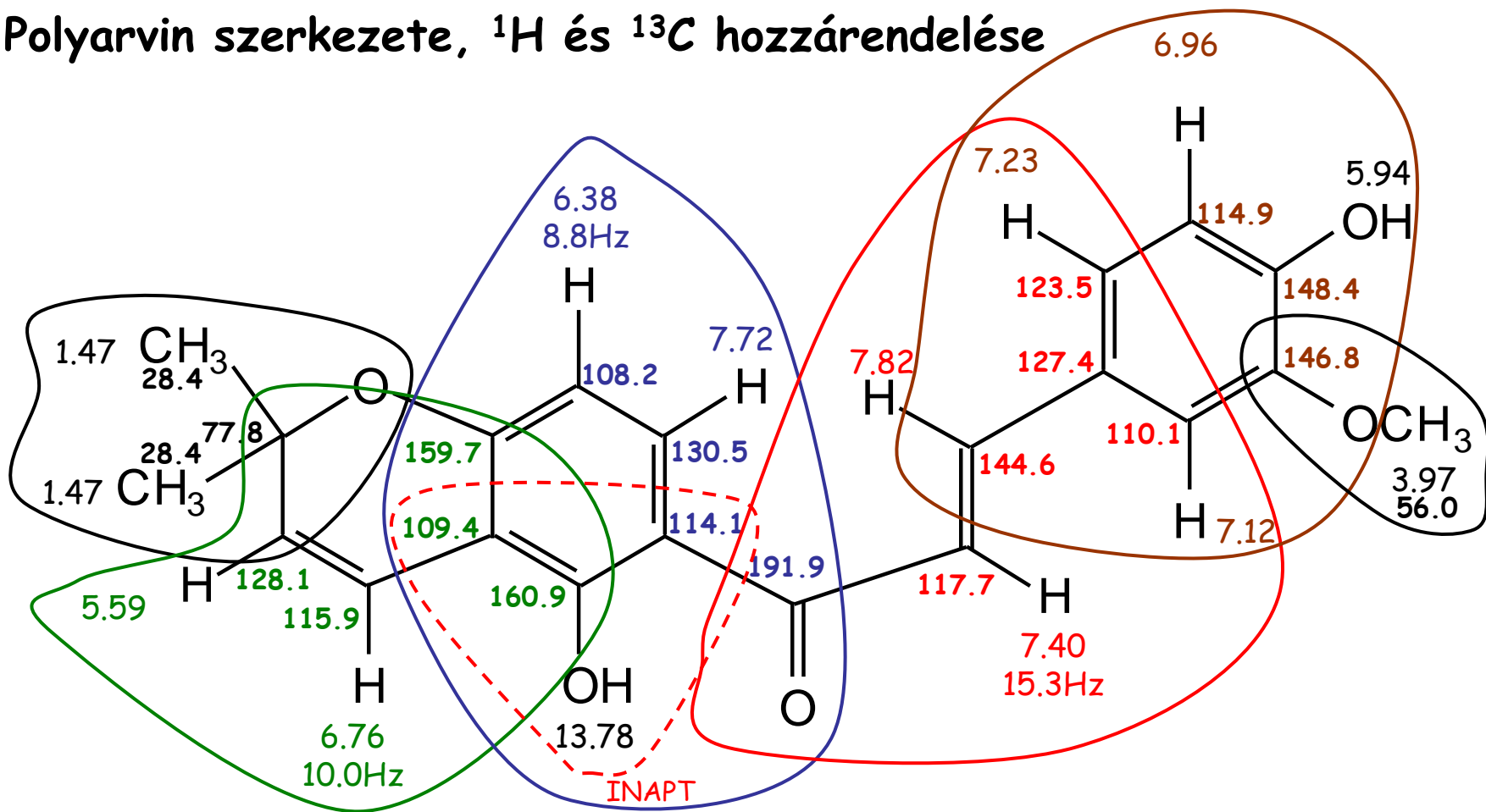


Table 6.25.1. Listing of ^1H correlations in the HMQC and HMBC spectra of **34**. $\text{C}_{21}\text{H}_{20}\text{O}_5$ DBE=12

^1H signals at $\delta =$	^{13}C signals at $\delta =$ (HMQC)	Long-range correlations at $\delta =$ (HMBC)
1.47		28.4
3.97		
5.59		
6.38		
6.76		
6.96		
7.12		
7.23		
7.40		
7.72		
7.82		
13.78	1D INAPT	109.4; 114.1; 160.9

Polyarvin szerkezete, ^1H és ^{13}C hozzárendelése



M. S. Rao, P. S. Rao, G. Tóth*, B. Balázs, H. Duddeck:
 Isolation of Polyarvin, a Chalcone from *Polygala arvensis*.
 Nat. Prod. Letters 12, 277-280 (1998)