

## 9 Two-Dimensional NMR Spectroscopy

Two-dimensional  $\pi$ -resolved spectrum

جابجایی‌های شیمیایی در یک محور فرکانس، و ثابت‌های جفت شدن در محور دیگر  
فرکانس رزم می‌شوند

Two-dimensional (shift)-correlated NMR spectrum

هر دو محور جابجایی‌های شیمیایی را نشان می‌دهند

روشهای دو بعدی بر اساس جفت شدنهای بین دو قطبهای هسته ای هسته  
(جفت شدنهای اسکالر، تأثیرات متقابل، و قطب مانده از طریق نفاذ)

1971 J. Jeener امکان انجام 2D NMR را مطرح کرد  
با تلاشهای R.R. Ernst و R. Freeman و همکاری آن  
این روش کاربردی شد

preparation, Evolution and Mixing, Data Acquisition

n آزمایش با تداوم  $t_1$  (با شروع از  $t_1=0$ ) انجام می‌دهد  
n  $t_2$  interferogram (FID) حاصل می‌شود  
دو سیگنال فریبست به  $t_1$  و  $t_2$  طیف دو بعدی را پدید می‌آورد

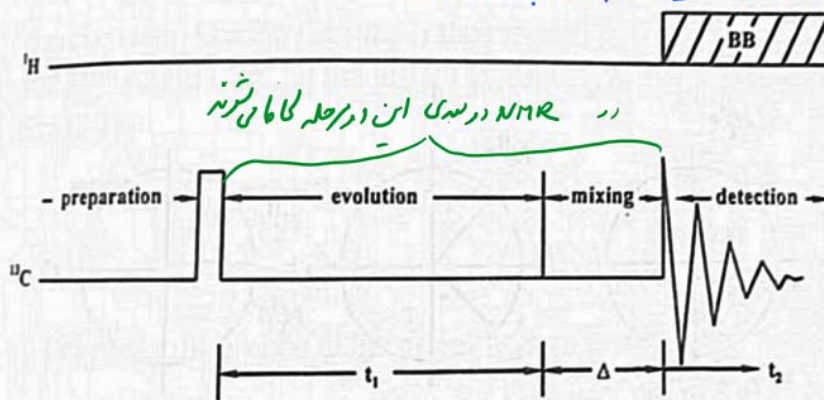
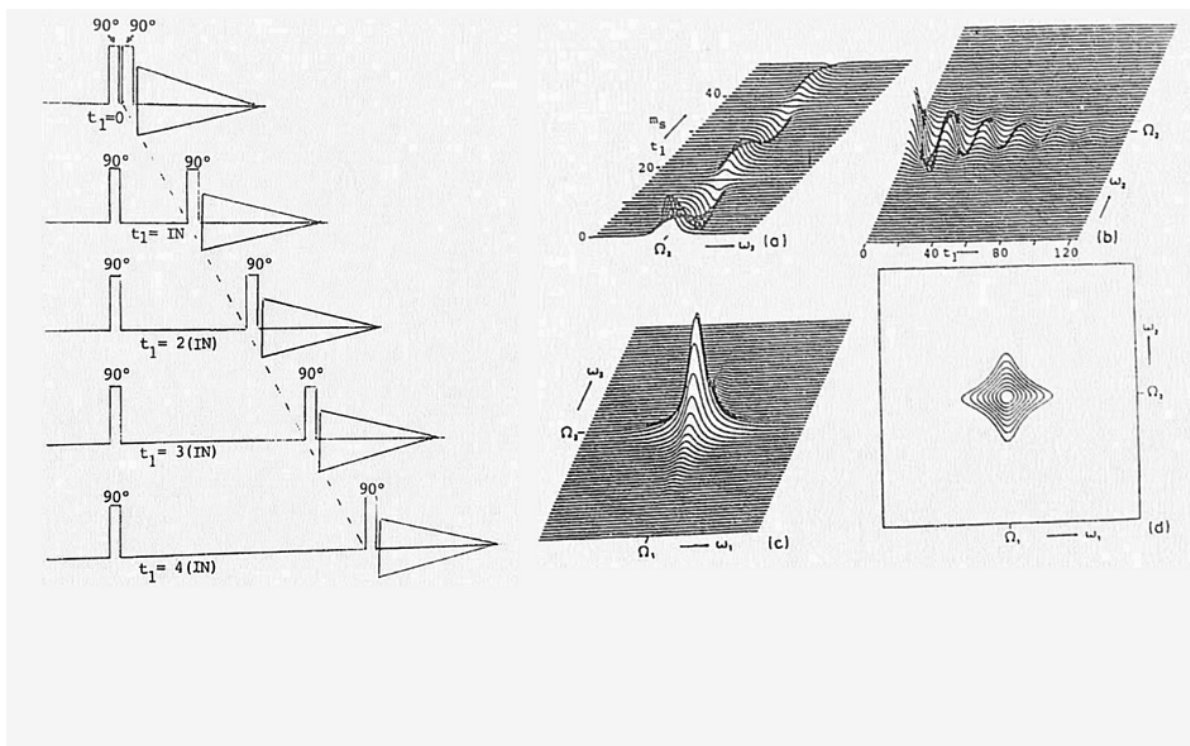
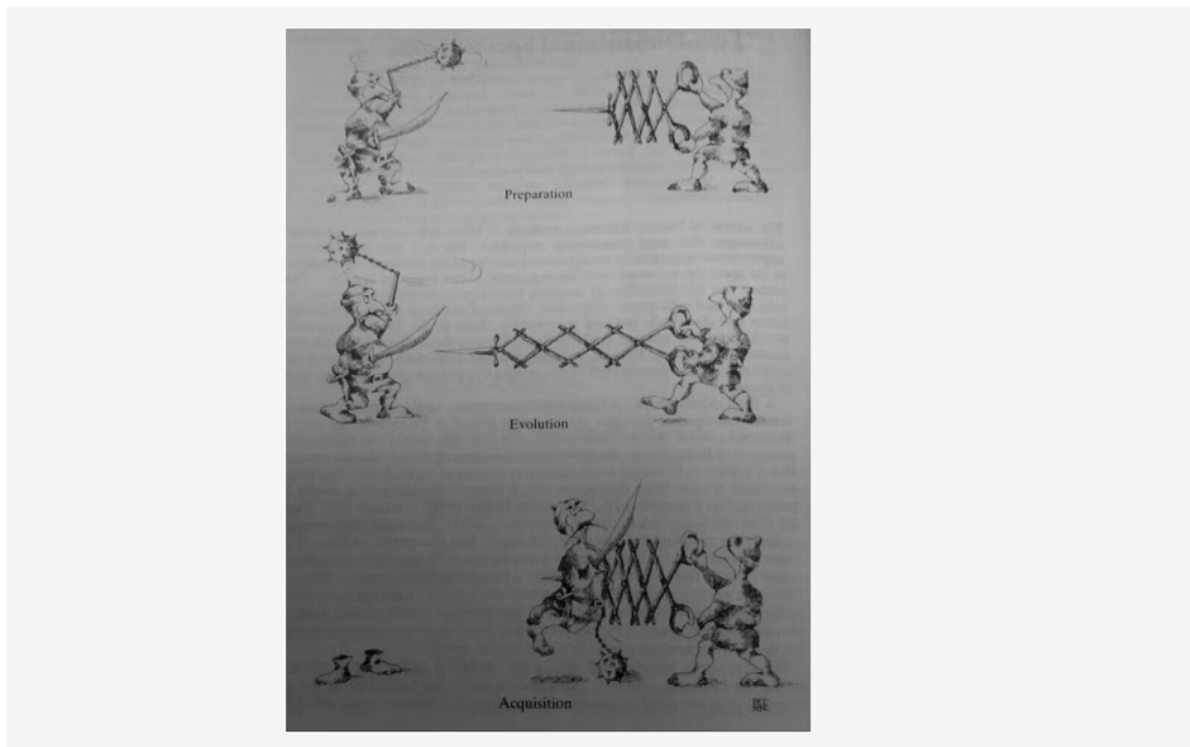
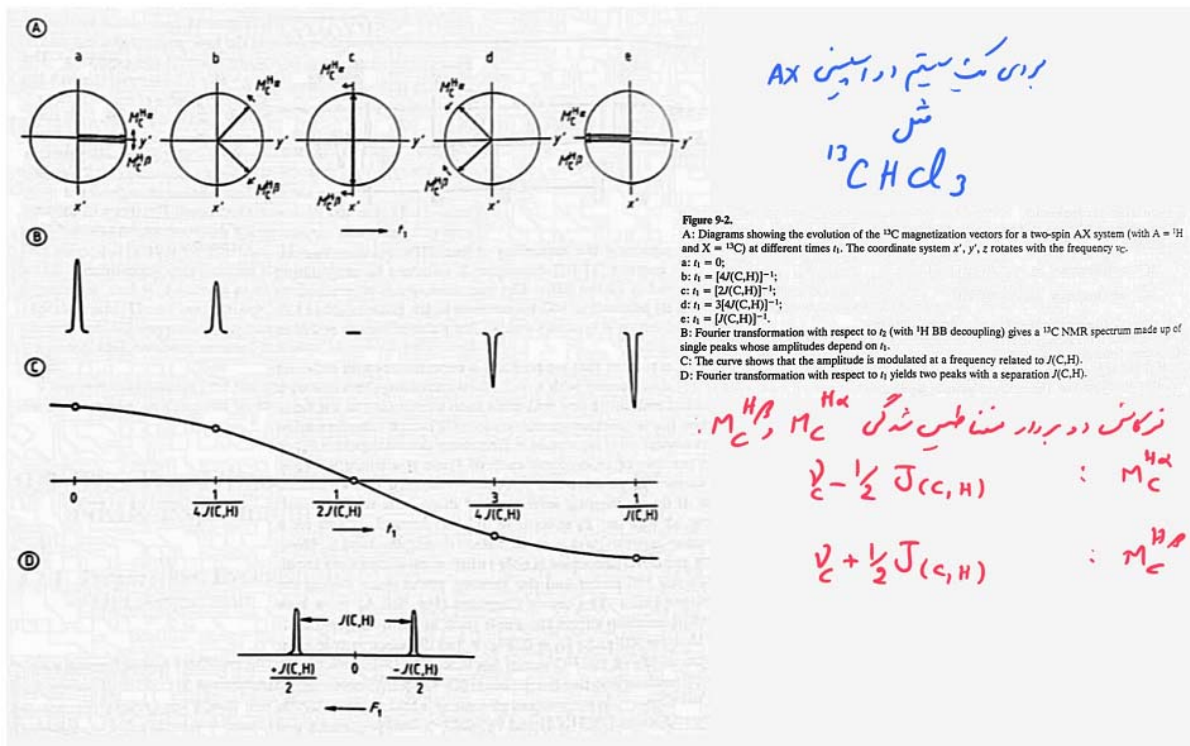
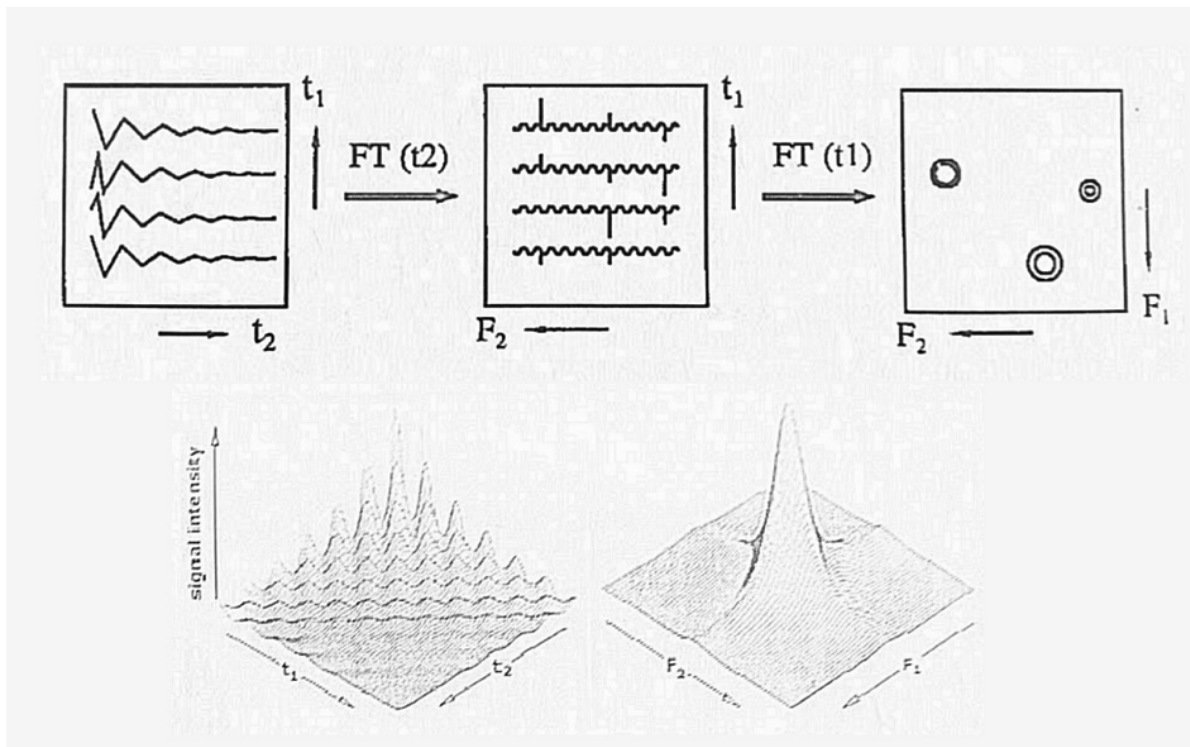


Figure 9-1. The basic principle of a two-dimensional NMR experiment. The variable is the interval  $t_1$ , which can be of the order of milliseconds to seconds. During the period  $t_1$  the system "evolves".  $\Delta$  is the mixing time, a period during which the spin states are allowed to "mix", and is kept constant throughout the experiment. The choice of a value for  $\Delta$  depends on the nature of the experiment.





ژدایای حرکتی از دو بردار نشان‌دهنده در من زمان  $t_1$  :

$$\varphi_{\alpha} = 2\pi \left( \nu_c - \frac{1}{2} J_{(c,H)} \right) t_1$$

$$\varphi_{\beta} = 2\pi \left( \nu_c + \frac{1}{2} J_{(c,H)} \right) t_1$$

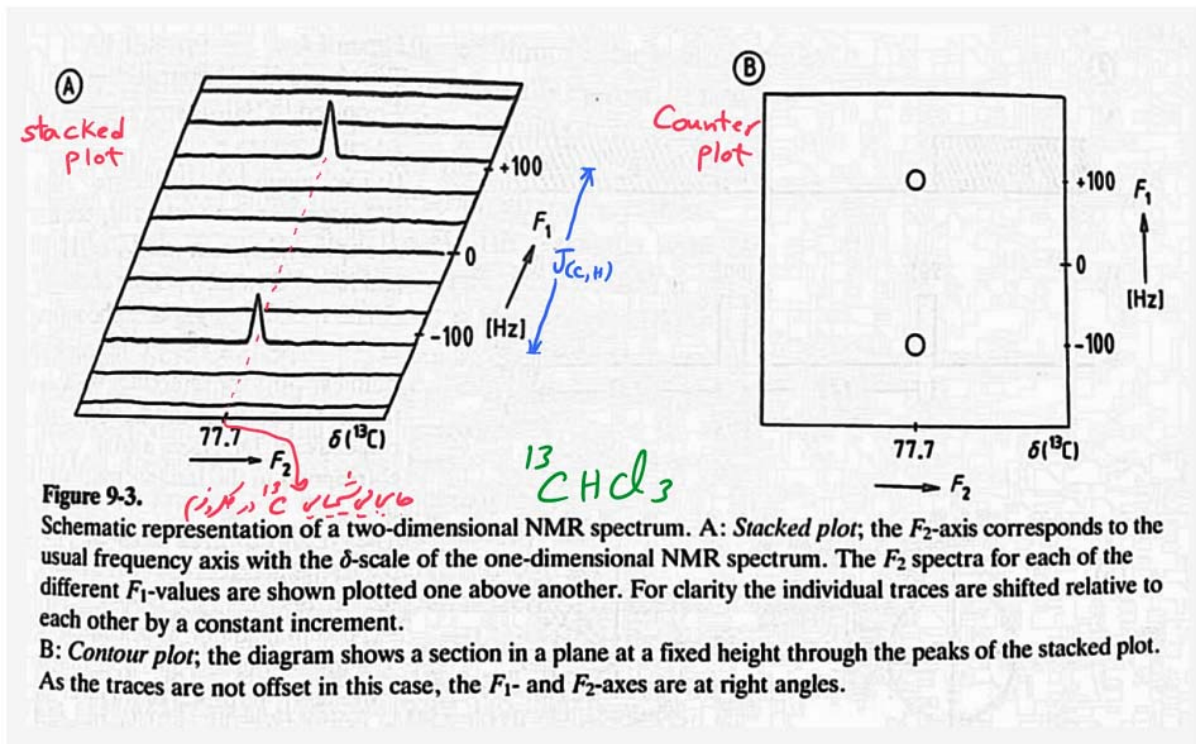
اختلاف فاز ( $\theta$ ) بین دو بردار برابر است با :

$$\theta = \varphi_{\beta} - \varphi_{\alpha} = 2\pi J_{(c,H)} t_1$$

$$t_1 = \frac{1}{4J_{(c,H)}} \quad \theta = 90^\circ$$

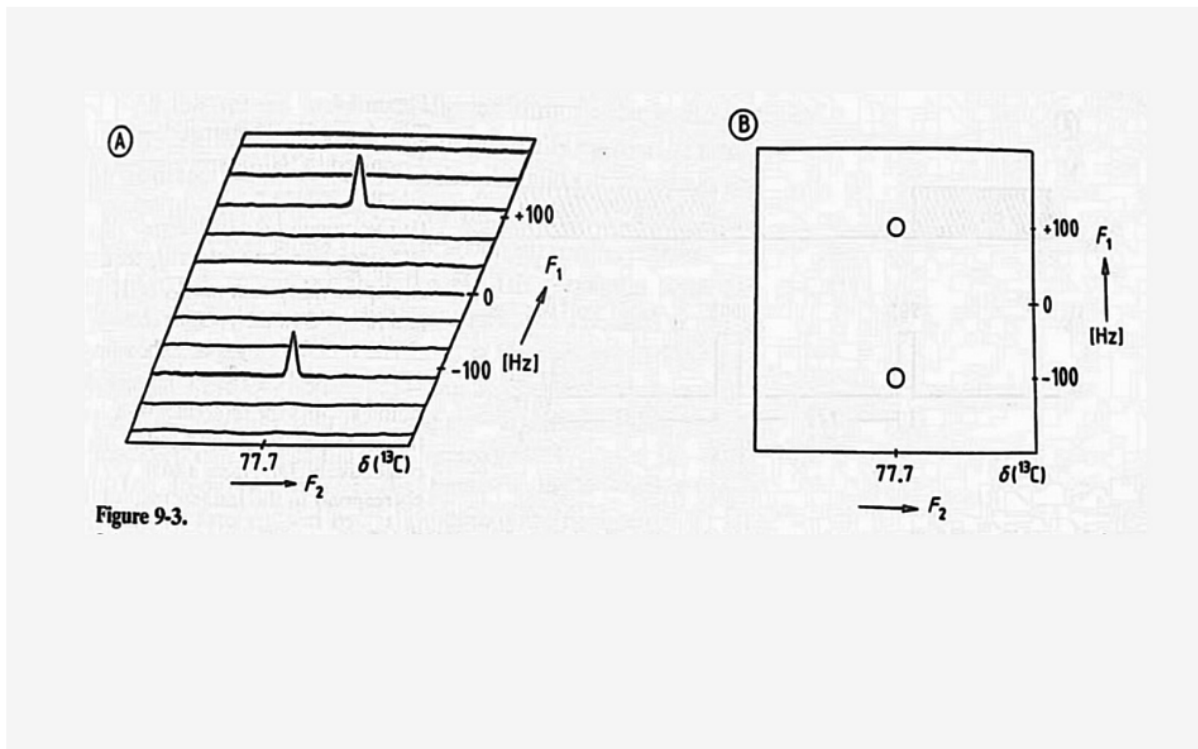
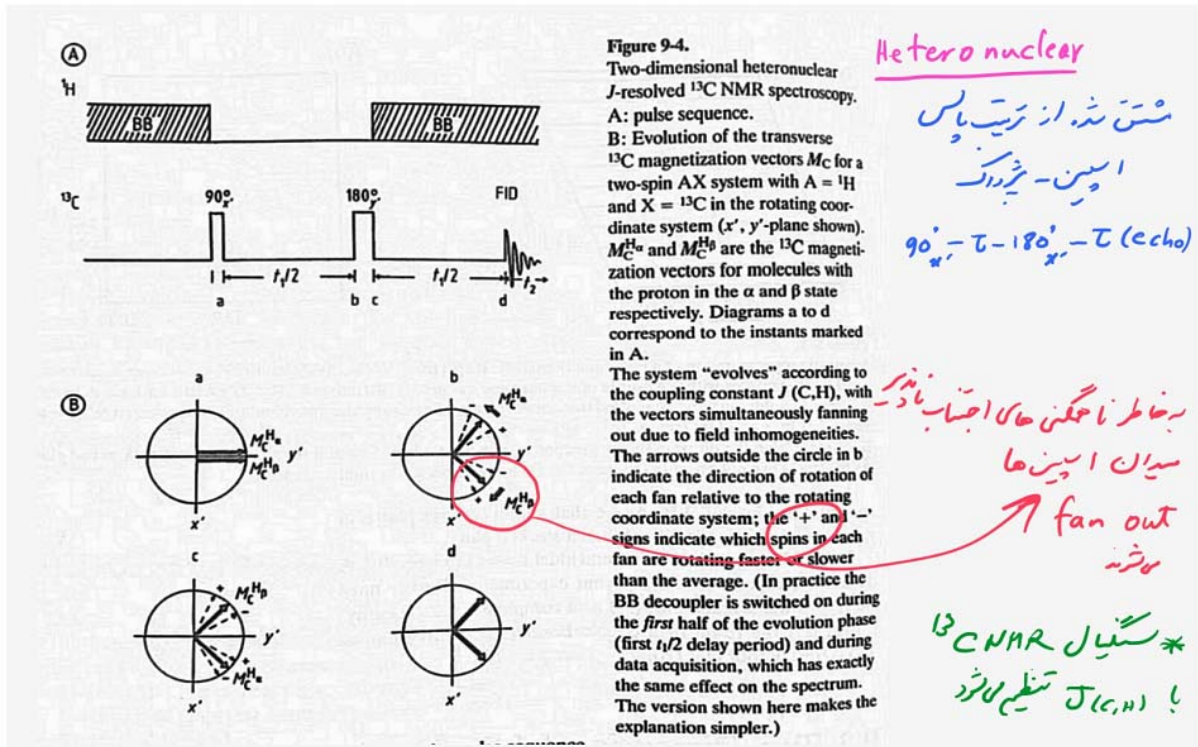
$$t_1 = \frac{1}{2J_{(c,H)}} \quad \theta = 180^\circ$$

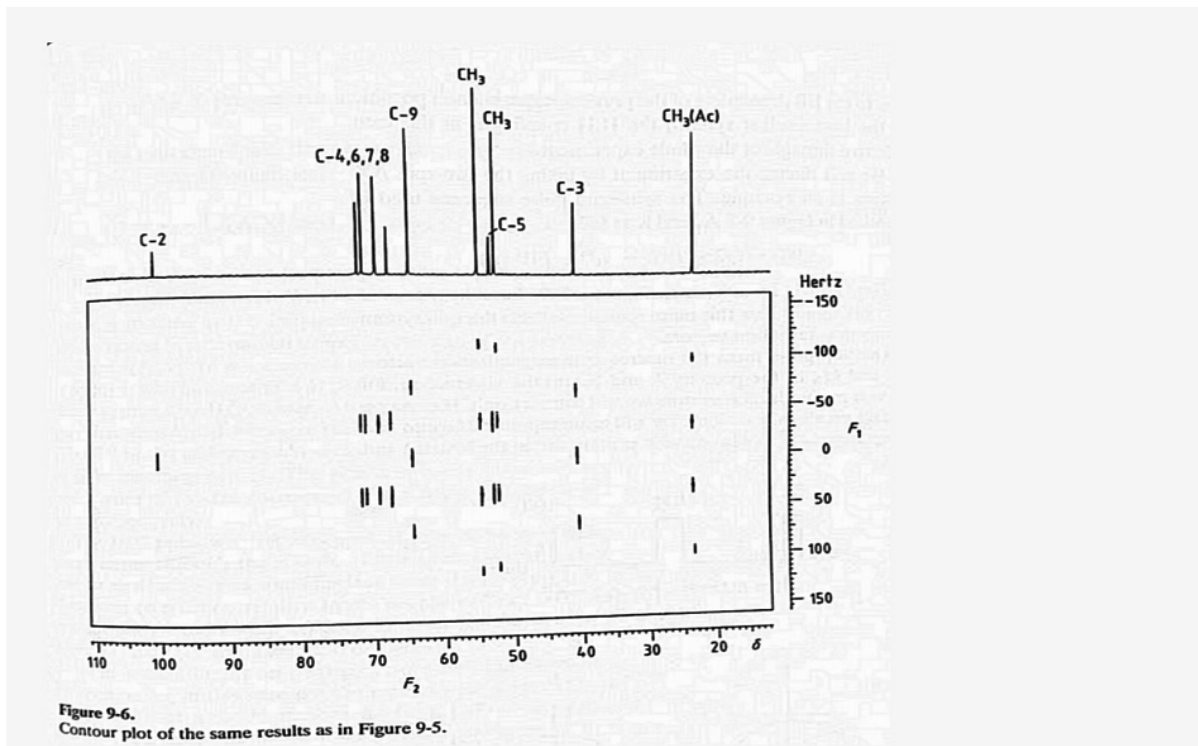
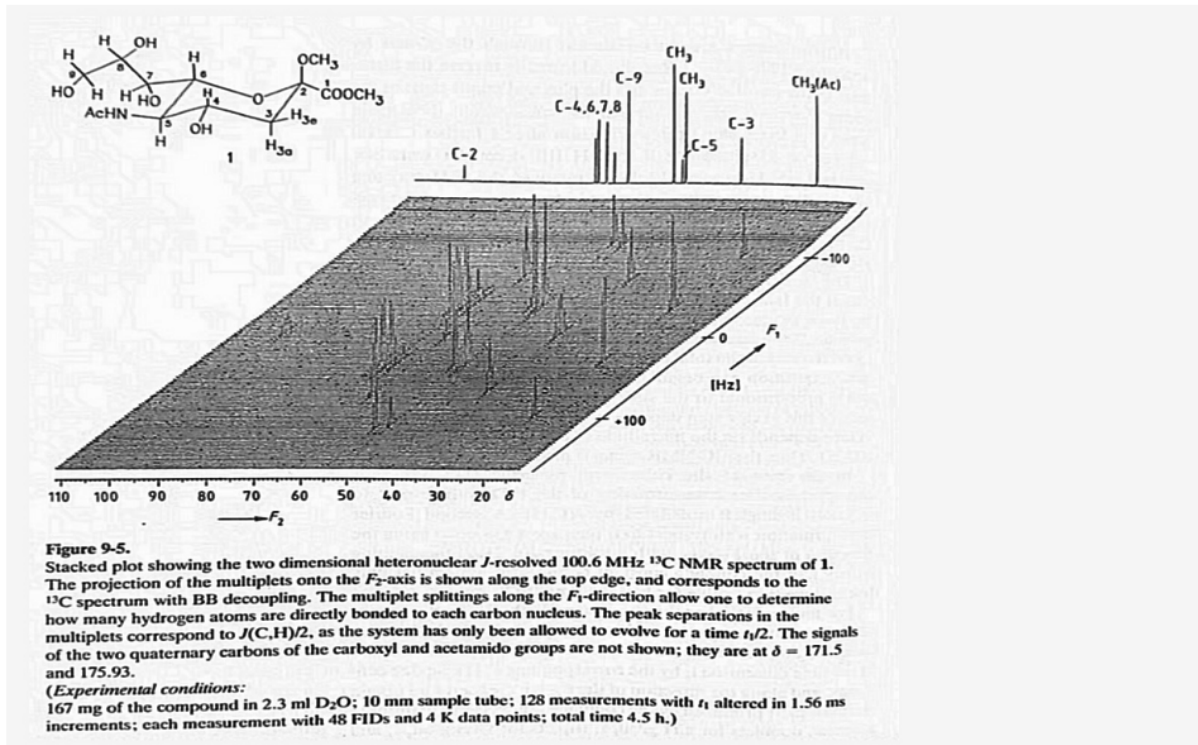
$$S(t_1, t_2) \xrightarrow{FT} S(t_1, F_2) \xrightarrow{FT} S(F_1, F_2)$$



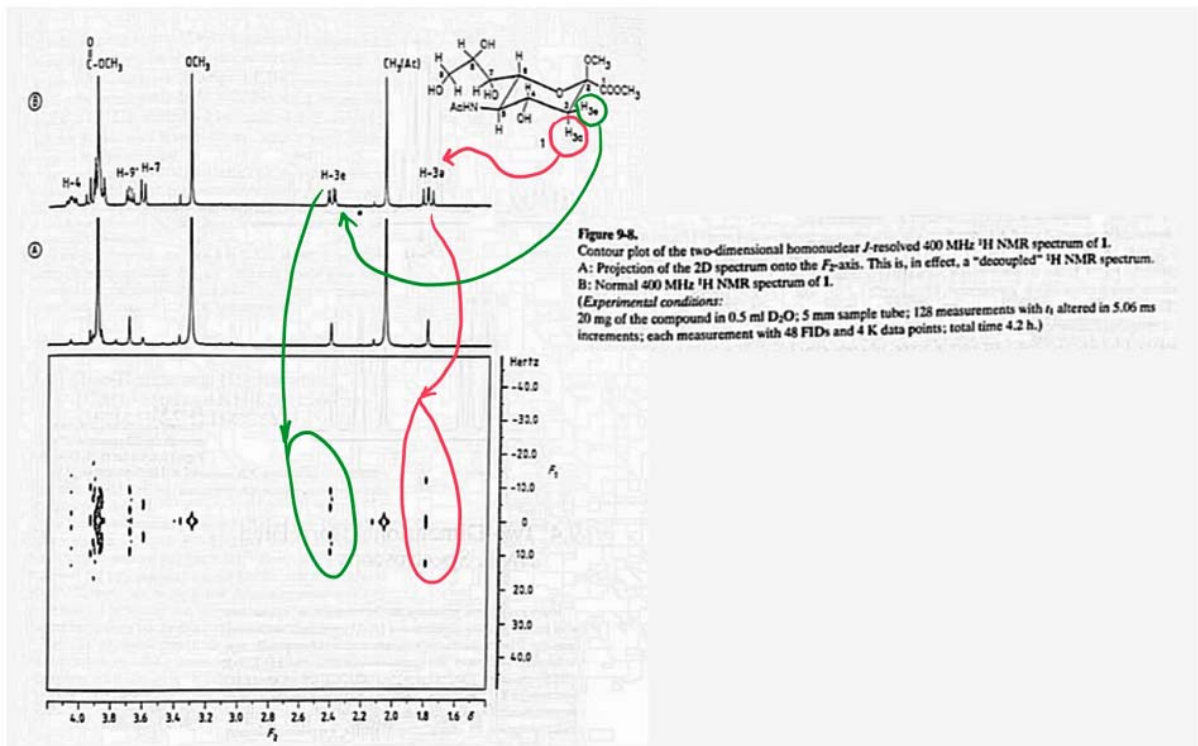
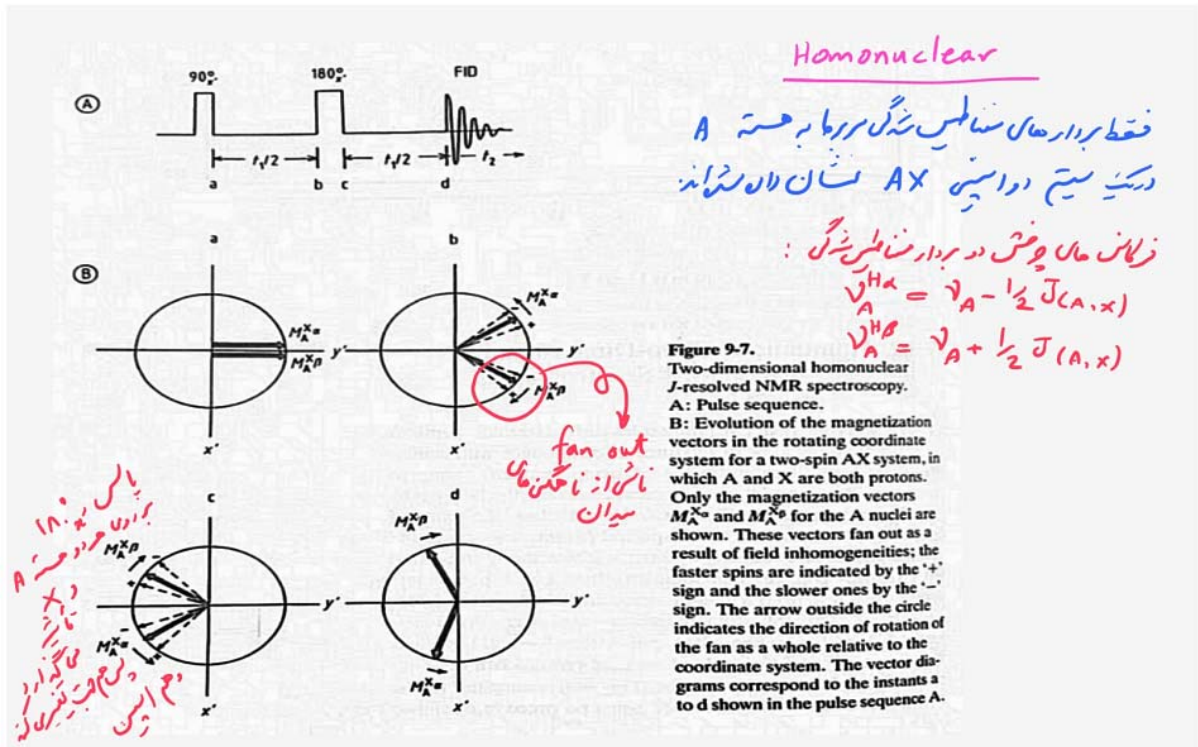
## Two Dimensional j-Resolved NMR Spectroscopy

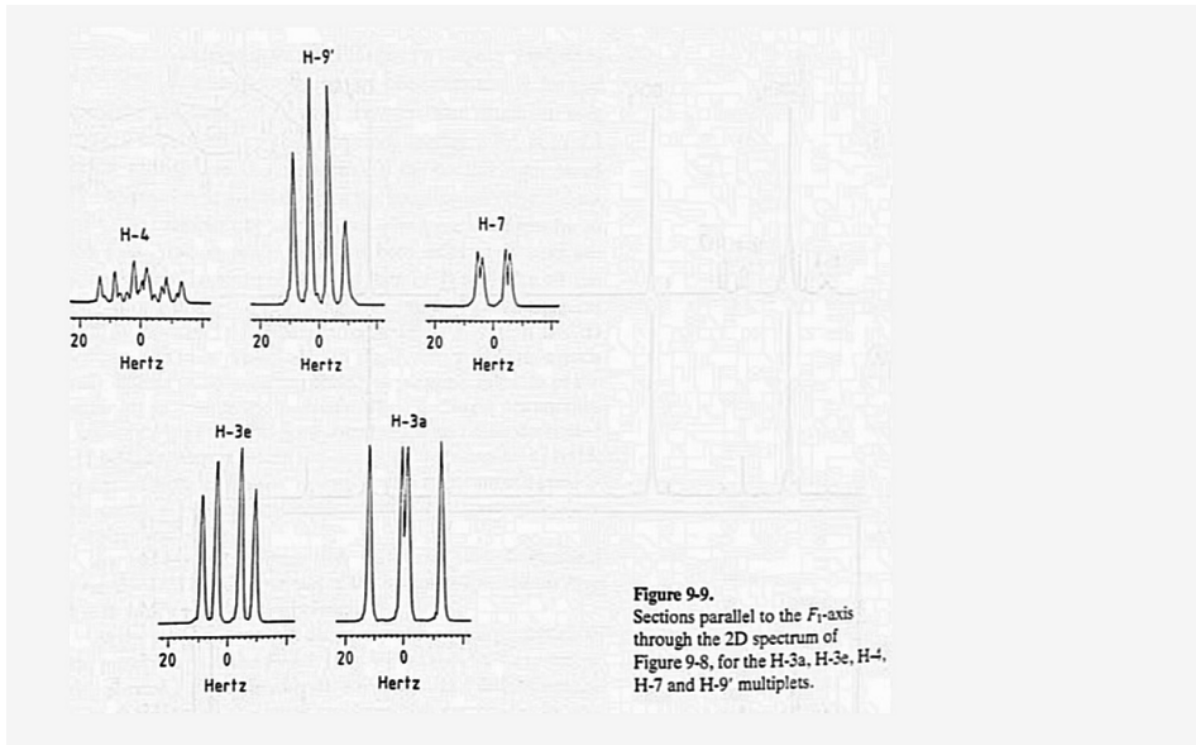
- Heteronuclear
- Homonuclear











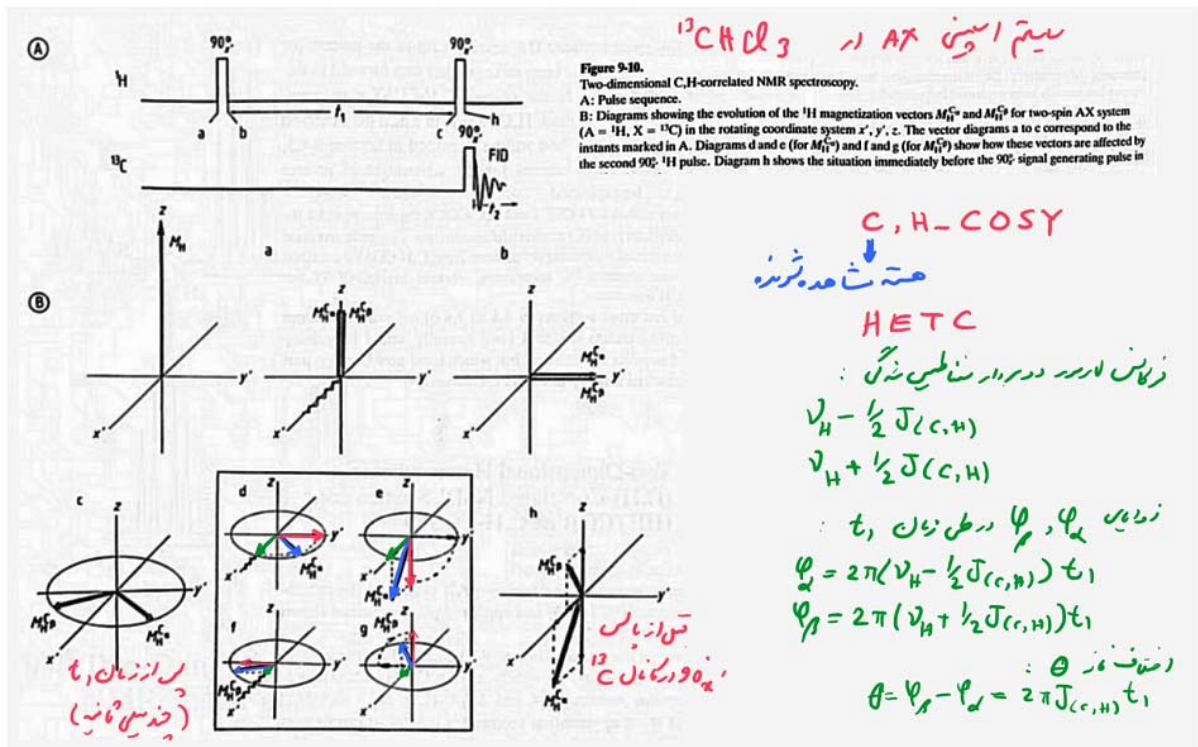
## Two- Dimensional Correlated NMR Spectroscopy

HETeronuclear CORrelation

HETCOR

CORrelated Spectroscopy

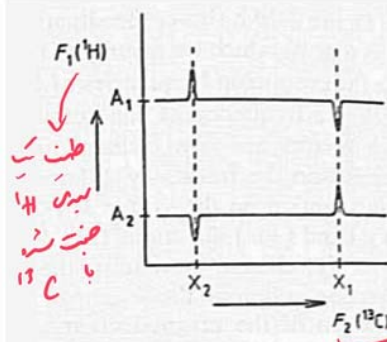
C, H-COSY



← سیگنال  $^{13}\text{C}$  NMR به عنوان تابعی از  $t_1$  برسد فرکانس حال لایزر پدرونها تنظیم شده است.

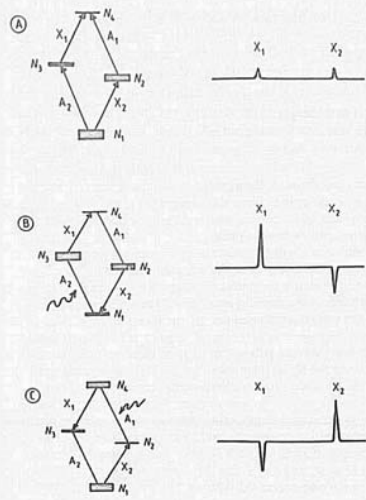
← این سرعت را نیز توان بطور عموداری به روشی تشریح نمود

← آنالیز ریاضی نشان میدهد که بردار حال نشاء طیس شدن  $M_H^{Ca}$  و  $M_H^{Cp}$  و هم چنین  $M_C^{Hx}$  و  $M_C^{Hp}$  که از نظر سطح انرژی به هم مرتبط هستند، همواره به میزان یکسان و با علامت متضادی تغییر میکنند

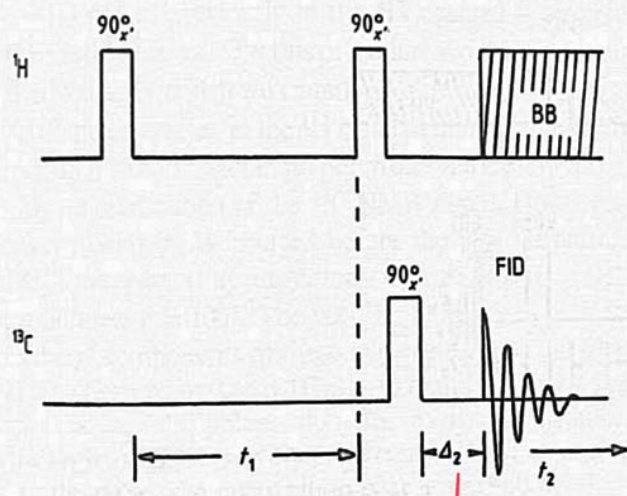


**Figure 9-11.** Schematic two-dimensional C,H-correlated NMR spectrum of a two-spin AX system (for pulse sequence see Fig. 9-10). The two signals along the  $F_2$ -direction correspond to the one-dimensional  $^{13}\text{C}$  NMR spectrum without decoupling, except that the signals have opposite signs. Along the  $F_1$ -direction is seen the doublet of the  $^1\text{H}$  NMR spectrum with the C,H coupling (the  $^{13}\text{C}$  satellites, also with opposite signal amplitudes).

خط 13  
خط 1H  
جفت نشد با 1H  
جفت نشد با 13C



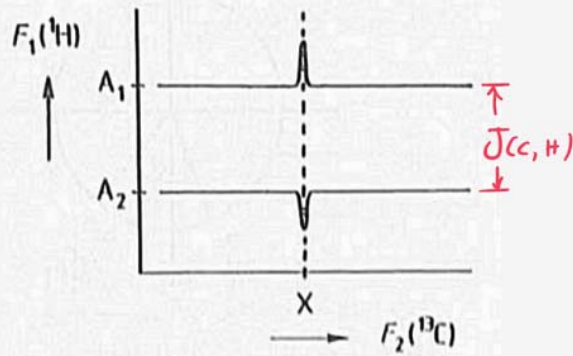
**Figure 8-14.** Energy level scheme for a two-spin AX system such as  $^1\text{H}$ - $^{13}\text{C}$  ( $A = ^1\text{H}$ ,  $X = ^{13}\text{C}$ ) showing schematically the resulting  $X$  ( $^{13}\text{C}$ ) spectra. A: Equilibrium state. B: A selective  $180^\circ$  pulse exciting only the  $A_2$  transition inverts the populations of levels 1 and 2, as indicated by the thicknesses of the slabs. The  $X_1$  transition then gives an enhanced absorption signal, while the  $X_2$  transition gives an enhanced emission signal. C: A selective  $180^\circ$  pulse exciting the  $A_1$  transition inverts the populations of levels 2 and 4. In the  $^{13}\text{C}$  NMR spectrum we again obtain two enhanced signals, an absorption signal for the  $X_2$  transition and an emission signal for the  $X_1$  transition. (The argument can also be worked through as a numerical simulation by inserting the values  $N_1 = 4$ ,  $N_2 = 5$ ,  $X_1 = 2$  and  $X_2 = 1$  for the equilibrium populations. Using these numbers leads directly to the enhancement factors of +2 and -2 for the two-spin  $^1\text{H}$ ,  $^{13}\text{C}$  system.)



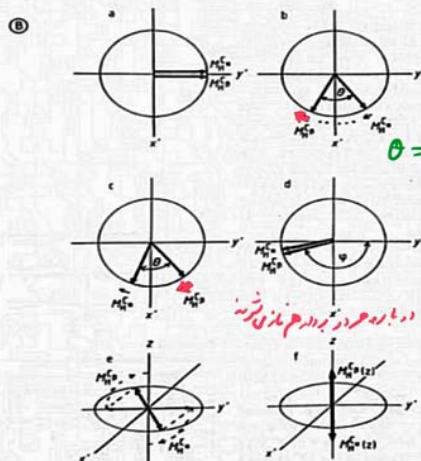
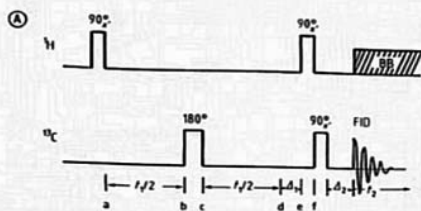
**Figure 9-12.** Extended pulse sequence for simplifying the two-dimensional C,H-correlated NMR spectrum of Figure 9-11. After the  $90^\circ_x$  pulse in the  $^{13}\text{C}$  channel there is a delay  $\Delta_2 = [2J(\text{C,H})]^{-1}$  before the start of data acquisition and simultaneous  $^1\text{H}$  BB decoupling.

اگر این تاخورد را نگذارند شد و ما تاخورد بعد از دایس بودیم  
BB decoupling اعمال شود سنگینا حذف خواهد شد.

جای سنگین به دو سنگین  
گواهی می آید



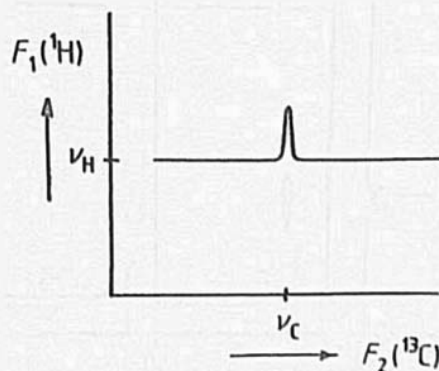
**Figure 9-13.** Schematic two-dimensional C,H-correlated NMR spectrum of a two-spin AX system (pulse sequence as in Fig. 9-12). The 2D spectrum is reduced to two signals with opposite signs; their separation along the  $F_1$  frequency axis is equal to  $J(C,H)$ .



$\theta = \pi J(C,H) t_1$  ;  $t_{1/2}$

**Figure 9-14.** A: Pulse sequence for a two-dimensional C,H-correlated NMR experiment which reduces the 2D spectrum of a two-spin AX system to only one peak. B: The vector diagrams a to f show the positions of the  $^1H$  magnetization vectors  $M_H^+$  and  $M_H^{+*}$  or their z-components ( $I$ ) at the instants indicated in A; in diagrams a to d only the  $x', y'$ -plane is shown.

← اولین تبدیل فریب نسبت به  $F_2$  سیگنال در  $\nu_C$  دهد  
 ← اگر کیت سری طیف با  $\Delta_1 = \Delta_2 = \frac{1}{2J_{(C,H)}}$  در تراز  $\nu_C$  تک سیگنال  
 شد که سیگنال برسد زمانس  $\nu_H$  تنظیم شده است  
 ← تبدیل فریب دوم نسبت به  $F_1$  کیت طیف در سببی  $(F_1, F_2)$  را می دهد که  
 تنها حاصل کیت سیگنال با مختصات  $(\nu_H, \nu_C)$  است.



**Figure 9-15.** Schematic two-dimensional C,H-correlated NMR spectrum of a two-spin AX system (pulse sequence as in Fig. 9-14). The 2D spectrum which is obtained consists of only one signal with the coordinates  $(\nu_H, \nu_C)$ .

شدت سیگنال برابر  
 $\Delta_1 = \frac{1}{2J_{(C,H)}}$   
 حد اکثر است  
 در برسد انتقال سینا طیف شکل  
 از  $^1H$  به  $^{13}C$   
 کیت هرگز

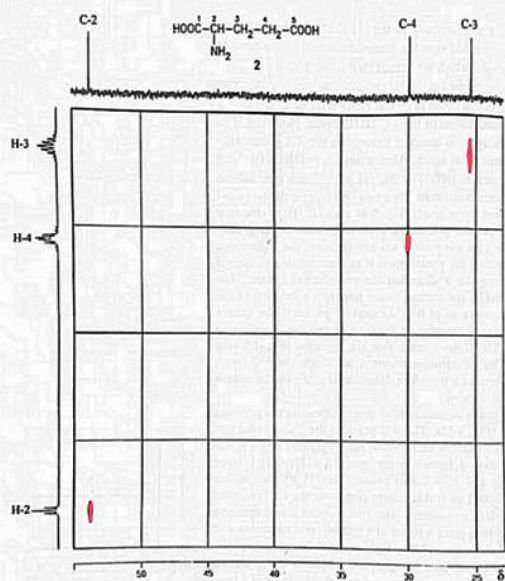
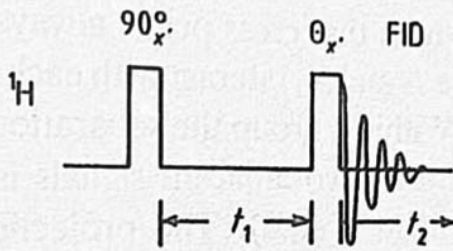


Figure 9-16. Two-dimensional C,H-correlated spectrum of glutamic acid (2) in D<sub>2</sub>O. The one-dimensional 500 MHz <sup>1</sup>H NMR spectrum is shown at the left-hand edge and the 125 MHz <sup>13</sup>C NMR spectrum at the top edge. (Experimental conditions: approx. 20 mg in 0.5 ml D<sub>2</sub>O; 5 mm sample tube; 128 measurements with *t<sub>1</sub>* altered in 312 μs increments; each measurement with 16 FIDs and 1 K data points; total time approx. 1 h.)

• در هسته کربن نیز جابجایی آروماتیک  
 یک ماه Correlation می دهه  
 بنابراین در این ظاهر شده اند

• مشاهده 13C در بلور مستقیم به پردازش  
 متصل شده و می توانند

## Two Dimensional Homonuclear (H, H)-Correlated NMR Spectroscopy H, H- COSY Long-Range COSY



**Figure 9-17.** Pulse sequence for the two-dimensional homonuclear H,H-correlated NMR experiment COSY. The variable is  $t_1$ . The pulse angle  $\theta$  is usually  $90^\circ$  or  $45^\circ$ , or occasionally  $60^\circ$ .

چار برداشت طیف شدگی  
 $M_A^{X_A}, M_A^{X_B}, M_X^{A_A}, M_X^{A_B}$

فرکانس رزونانس:

$$\nu_A \pm J(A,X)/2$$

$$\nu_X \pm J(A,X)/2$$

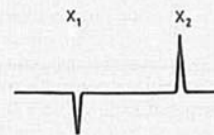
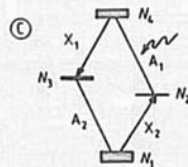
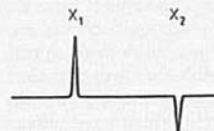
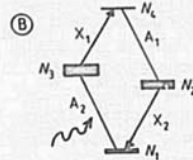
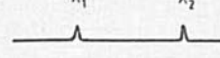
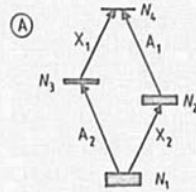
در ضمن زمان  $t_1$ ، چار برداشت طیف شدگی به خاطر اختلاف فرکانس fan out می‌شوند

پس می‌توانیم درم، می‌تواند حال کنی هر یک از برداشت‌ها  
 شدگی را به سمت  $+Z$  و  $-Z$  می‌خواند  
 راست لقطه به چپ می‌خواند

میشه حال کنی برداشت‌ها طیف شدگی  
 چرخش خود را در ضمن کنی، که اداره  
 می‌کنه. کیت FID می‌خواند  
 از تبدیل فریب است به  $t_2$   
 طیف از اینج

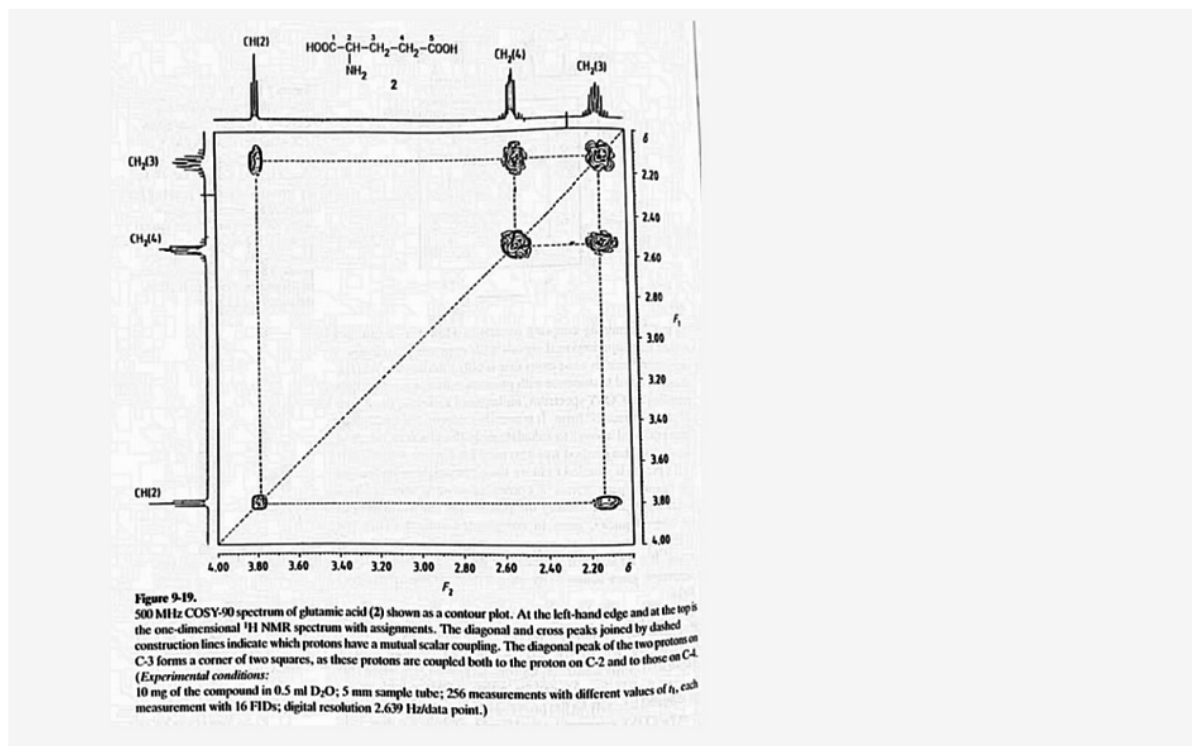
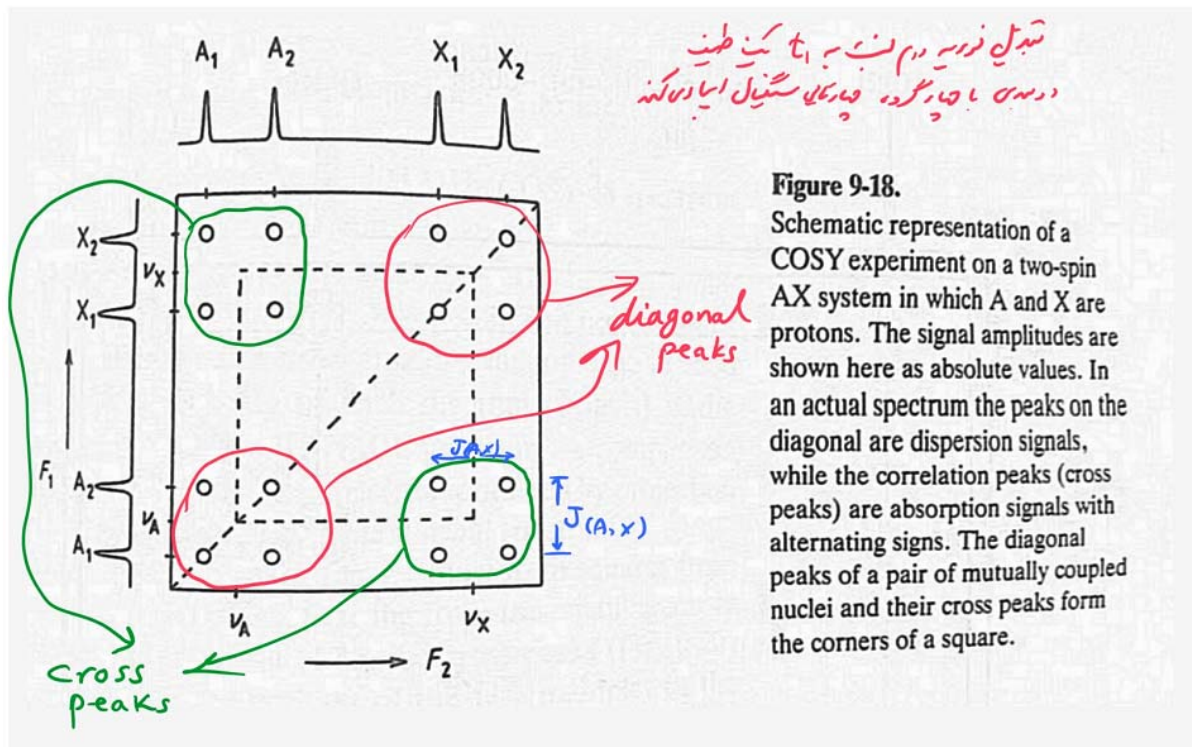
four-line AX-type  
 با فرکانس زیر ایجاد می‌کنه

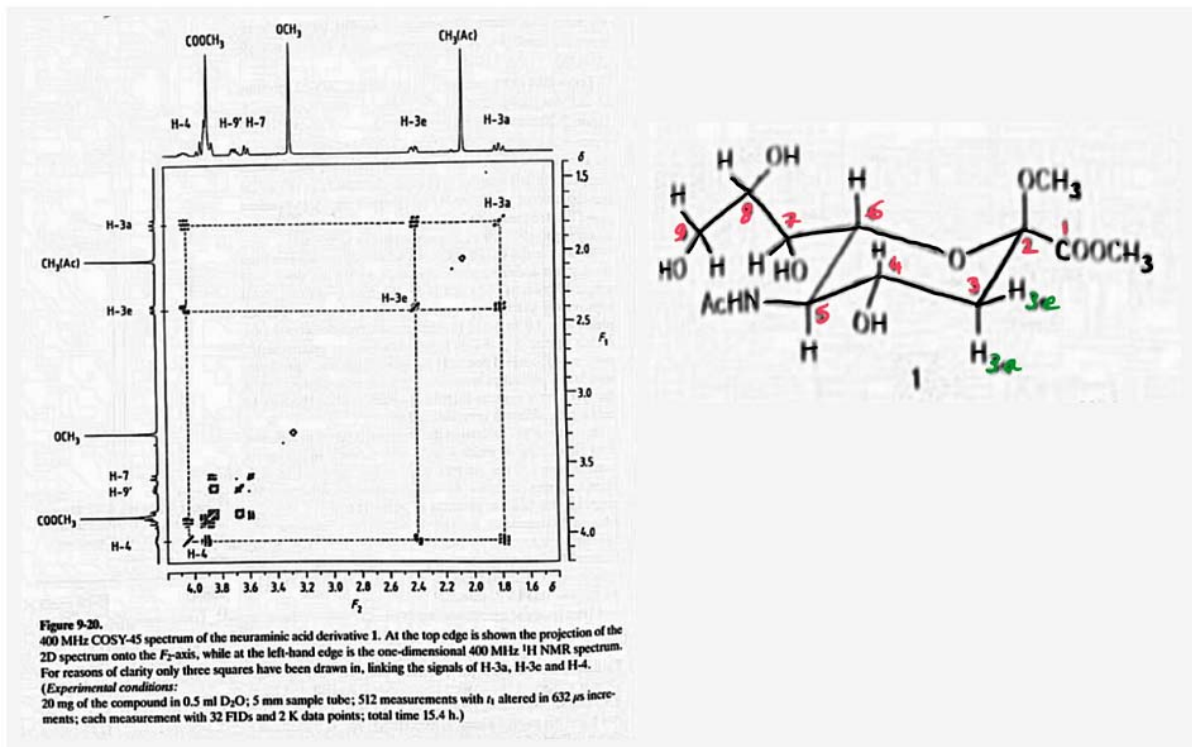
$$\begin{aligned} \nu_A + \frac{1}{2} J(A,X) & (A_1) \\ \nu_A - \frac{1}{2} J(A,X) & (A_2) \\ \nu_X + \frac{1}{2} J(A,X) & (X_1) \\ \nu_X - \frac{1}{2} J(A,X) & (X_2) \end{aligned}$$



**Figure 8-14.** Energy scheme for a two-spin AX system such as  $^{13}\text{CHCl}_3$  ( $A = ^1\text{H}$ ,  $X = ^{13}\text{C}$ ) showing schematically the resulting X ( $^{13}\text{C}$ ) spectra. A: Equilibrium state. B: A selective  $180^\circ$  pulse exciting only the  $A_2$  transition inverts the populations of levels 1 and 3, as indicated by the thicknesses of the slabs. The  $X_1$  transition then gives an enhanced absorption signal, while the  $X_2$  transition gives an enhanced emission signal. C: A selective  $180^\circ$  pulse exciting the  $A_1$  transition inverts the populations of levels 2 and 4. In the  $^{13}\text{C}$  NMR spectrum we again obtain two enhanced signals, an absorption signal for the  $X_2$  transition and an emission signal for the  $X_1$  transition. (The argument can also be worked through as a numerical simulation by inserting the values  $N_1 = 6$ ,  $N_2 = 5$ ,  $N_3 = 2$  and  $N_4 = 1$  for the equilibrium populations. Using these numbers leads directly to the enhancement factors of +5 and -1 for the two-spin  $^1\text{H}$ ,  $^{13}\text{C}$  system.)







**Table 9-1.**  
 Summary of the procedure followed in analyzing the two-dimensional H,H-correlated NMR spectrum of **1**.

starting point	newly assigned
H-3a/H-3e	H-4
	H-5
	H-6*
H-7	H-8*
	H-9*
	H-9'

\*The experiment gives only approximate positions for these signals.

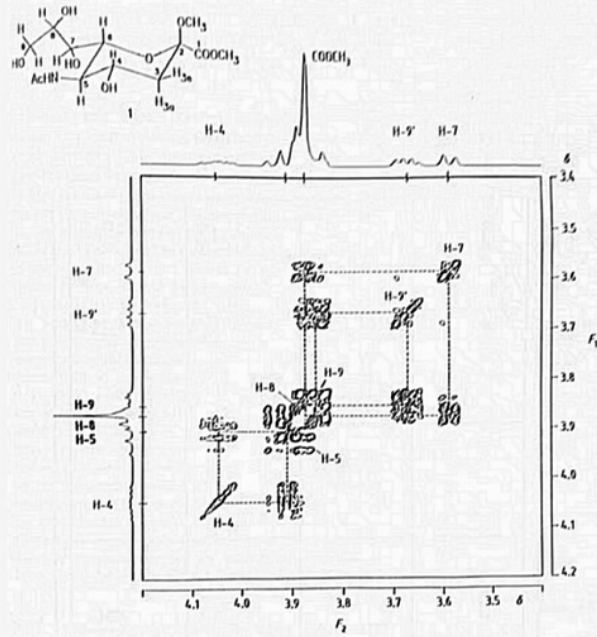


Figure 9-21. Expanded plot of a portion of Figure 9-20. Starting from the signals of H-4, H-7 and H-9', which are already assigned with certainty as shown in the top spectrum, the cross peaks lead us to the chemical shifts to H-5, H-8

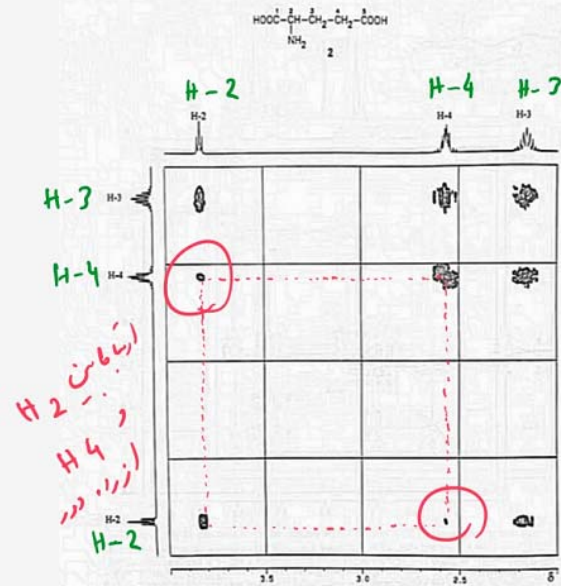


Figure 9-22. 500 MHz long-range H,H-COSY spectrum of glutamic acid (2). The one-dimensional  $^1\text{H}$  spectrum is shown at the top and on the left. Compared with the COSY-90 spectrum (Fig. 9-19) two new signals at approximately (2.6,3.8) and (3.8,2.6) have now appeared. These provide evidence of a correlation between the protons on C-2 and C-4; in the one-dimensional 500 MHz spectrum it is not possible to detect a coupling between these protons. (Experimental conditions: approx. 20 mg in 0.5 ml  $\text{D}_2\text{O}$ ; 5 mm sample tube; 128 measurements with  $t_1$  altered in 600  $\mu\text{s}$  increments; each measurement with 8 FIDs and 1 K data points; total time approx. 40 min.)

Long-range  
H,H-COSY  
Spectrum

## Reverse Two-Dimensional Heteronuclear (H,C)-Correlated NMR Spectroscopy

### HSQC; HMQC

- در شرایطی که در آنجا هسته غیر حاکم باشد، روشی وقت‌گیری هسته (زمان طیف‌گیری طولانی است)
- روش طیف‌سنجی HETCOR، اطلاعات مفیدی ملاحظه (دارای عیب بالا است)
- مانند روش reverse INEPT در آنجا نیز برای رفع عیب بالا هسته حاکم تر باشد خواهد بود
- در نتیجه به مقدار کمتر زمان و زمان کوتاه‌تر طیف‌گیری نیاز خواهد بود

### HSQC

#### Heteronuclear single Quantum Coherence

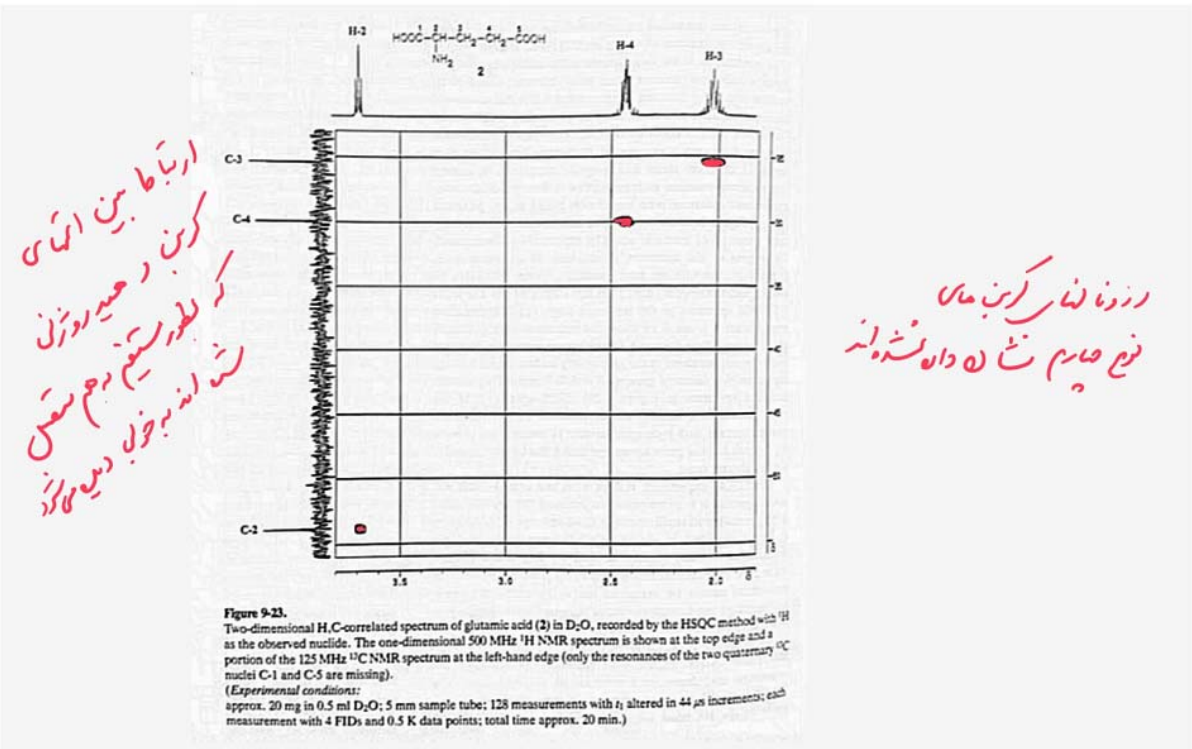
<sup>1</sup>H:

$90^\circ_x - \tau - 180^\circ_x - \tau - 90^\circ_y - t_1/2 - 180^\circ_y - t_1/2 - 90^\circ_x - \tau - 180^\circ_x - \tau - \text{FID}$   
( $t_2$ )

<sup>13</sup>C:

$\tau - 180^\circ_x - \tau - 90^\circ_x - t_1 - 90^\circ_x - \tau - 180^\circ_x - \tau - \text{BB}$

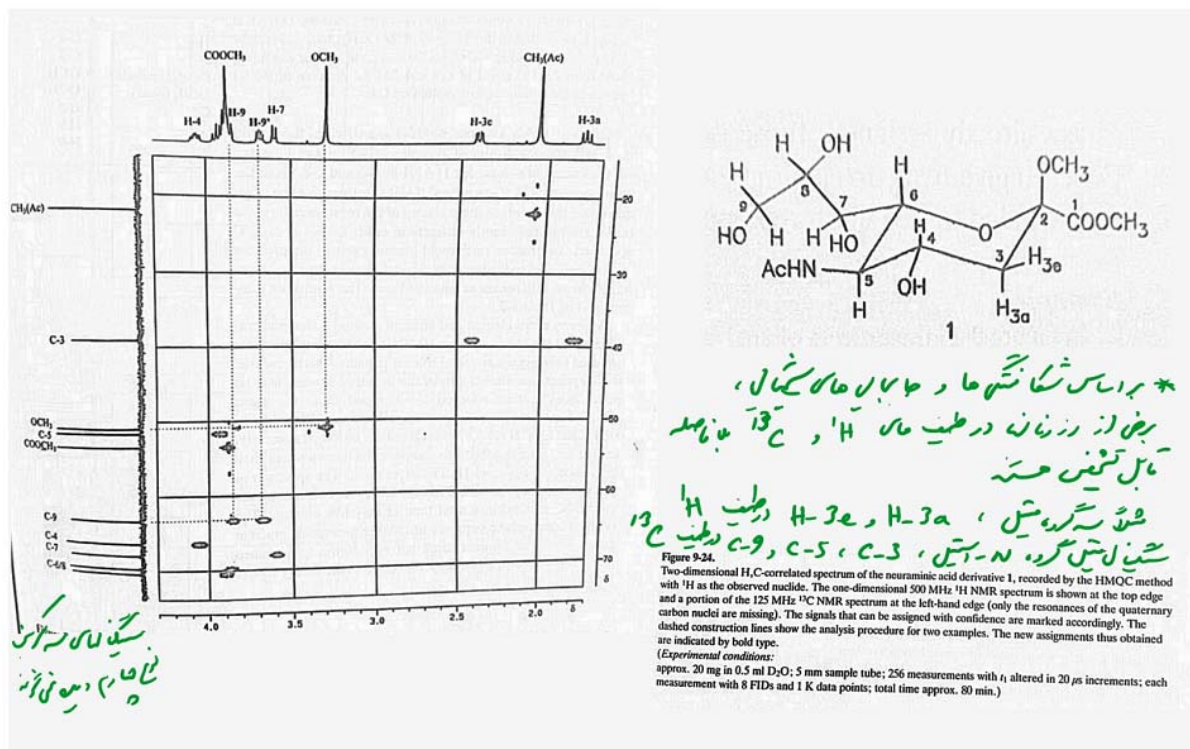
- در مرحله اول انتقال قطبیت  $^1\text{H}$  سربر پایه منطقی شدگ  $M_H$   
 از طریق ترتیب پاس INEPT مدل که تقویت شدن منطقی شدگ  $M_C$  را در اول دارد  
 - در مرحله دوم بردارهای  $M_C$  اجازه ترکه یافتن در زمان  $t_1$  را میبایند  
 - در مرحله نهای قطبیت حاصل برای  $^{13}\text{C}$  از طریق ترتیب پاس reverse INEPT  
 دوبار به برودرکنا منتقل میگردد  
 - در خانه رزونا  $^1\text{H}$  ثبت میگردد



آزمون HSQC دارای یک عیب است و آن این است که به پالهای  $90^\circ$  و  $180^\circ$  دقیق نیاز دارد و به خط‌های کوچک در تنظیم این متادریسار حس است.

این مشکل با یک‌بارگویی روش  
 HMQC (Heteronuclear multiple Quantum Coherence)  
 تا حد زیادی رفع شده است.

در هر دو روش ذکر شده سگنال‌های پروتون‌ها مضعف شد به اتم‌های  $^{12}\text{C}$  از طریق چرخه‌های ناز مناسب حذف می‌شوند



**Table 9-2.**

Summary of the procedure followed in analyzing the two-dimensional (H,C)-correlated NMR spectrum of 1.

starting point	newly assigned
H-4	C-4
H-7	C-7
$\text{OCH}_3$ (ketoside)	$\text{OCH}_3$
$\text{OCH}_3$ (ester)	$\text{OCH}_3$
C-5	H-5
C-9	H-9
C-9	H-9'

# The Gradient-Selected (gs-)HMBC Experiment

(gradient selected)

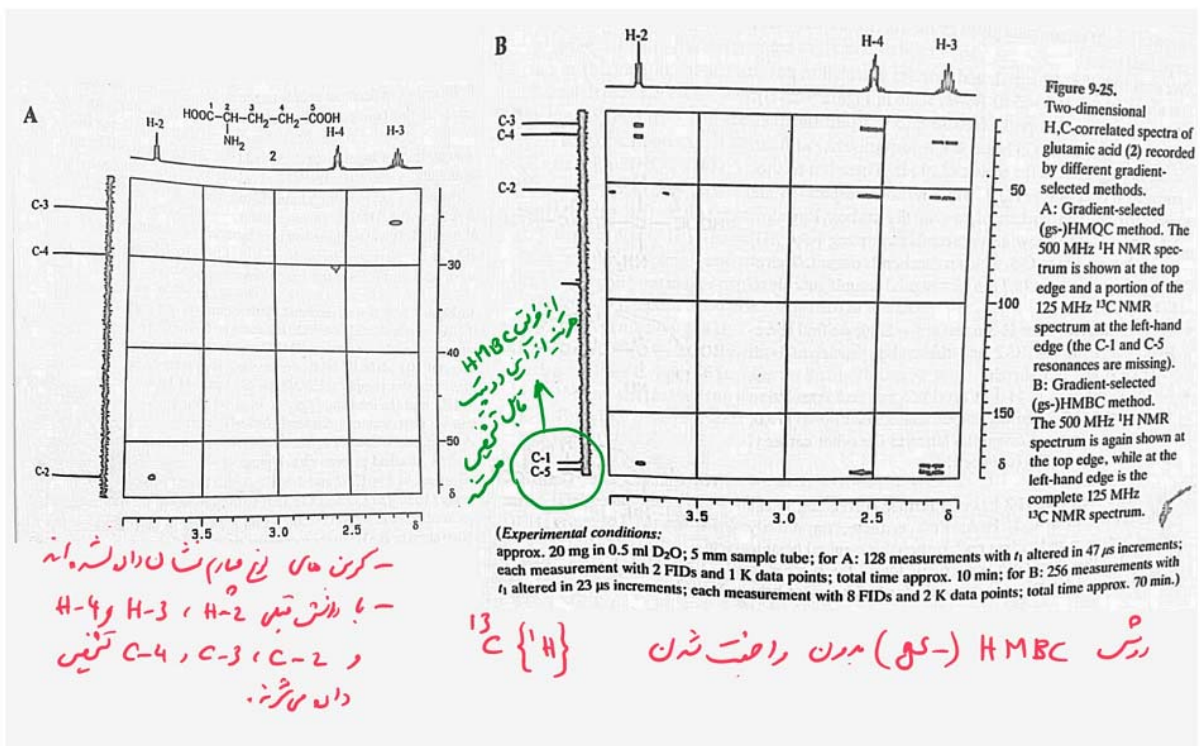
در دورش گزیده شد پس یک ها ارتباط بین پروتونها و کربن ها رخ می دهد و این امر به نفع است.

بنابراین دورش قبل از آن تغییر جهت شد تا بزرگتر  $J(C, H)$  ظاهر شود.

پس کمتر به تغییر سگنالها کربن رخ می دهد.

دورش HMBC ارتباط بین هسته های  $^1H$  و  $^{13}C$  که از طریق دریا می بیند با هم جهت داشتن دستاورد  $J$  کوچک تر دارند نیز می باشد.

Homonuclear Multiple Bond Correlation





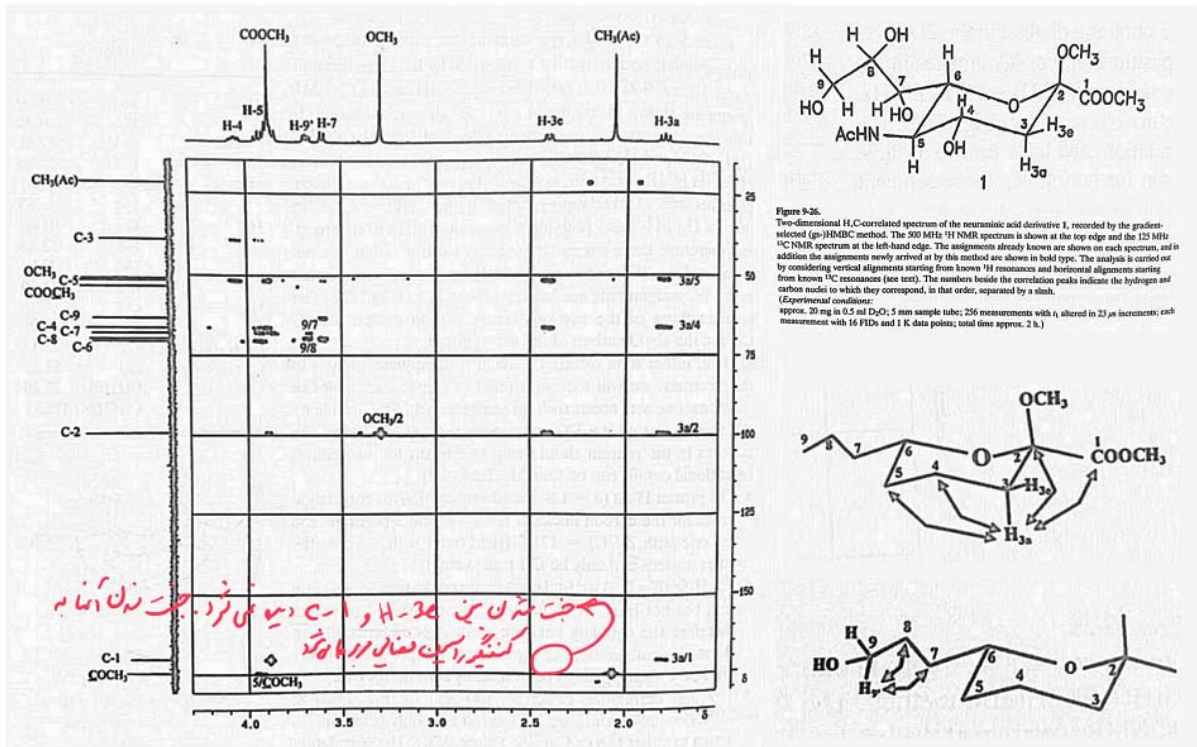
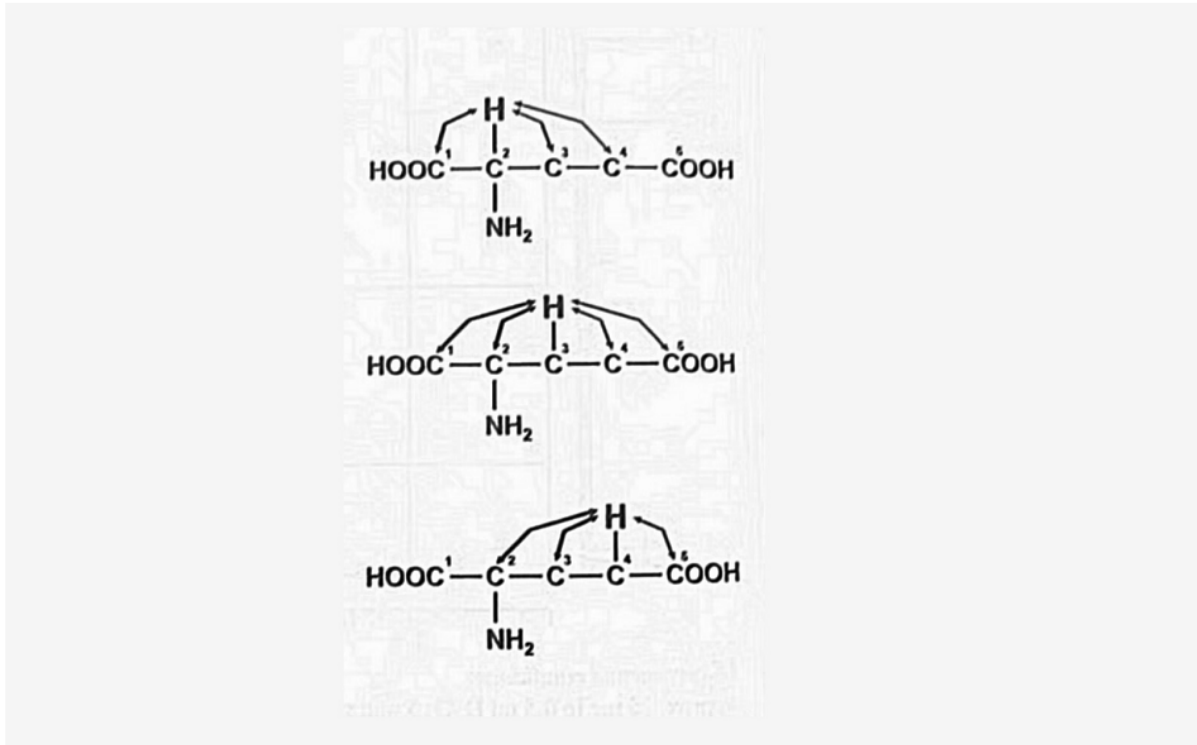
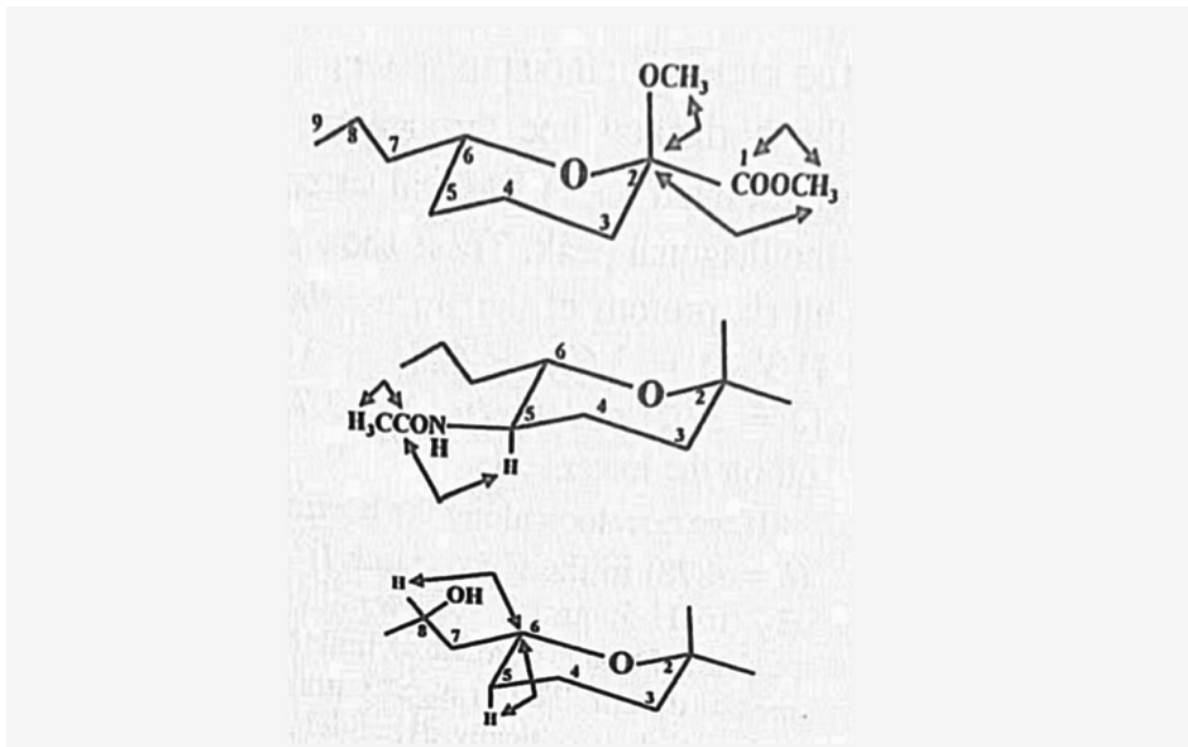


Figure 9-26. Two-dimensional  $^1\text{H}$ - $^{13}\text{C}$  correlated spectrum of the neuraminic acid derivative 1, recorded by the gradient-selected (gs)-HMBIC method. The 500 MHz  $^1\text{H}$  NMR spectrum is shown at the top edge and the 125 MHz  $^{13}\text{C}$  NMR spectrum at the left-hand edge. The assignments already known are shown on each spectrum, and in addition the assignments newly arrived at by this method are shown in bold type. The analysis is carried out by considering vertical alignments starting from known  $^1\text{H}$  resonances and horizontal alignments starting from known  $^{13}\text{C}$  resonances (see text). The numbers beside the correlation peaks indicate the hydrogen and carbon nuclei to which they correspond, in that order, separated by a slash.

(Experimental conditions: approx. 20 mg in 0.5 ml  $\text{D}_2\text{O}$ ; 5 mm sample tube; 256 measurements with  $t_1$  altered in 23  $\mu\text{s}$  increments; each measurement with 16 FIDs and 1 K data points; total time approx. 2 h.)



**Table 9-3.**  
Summary of the procedure followed  
in analyzing the two-dimensional  
(gs-)HMBC spectrum of 1.

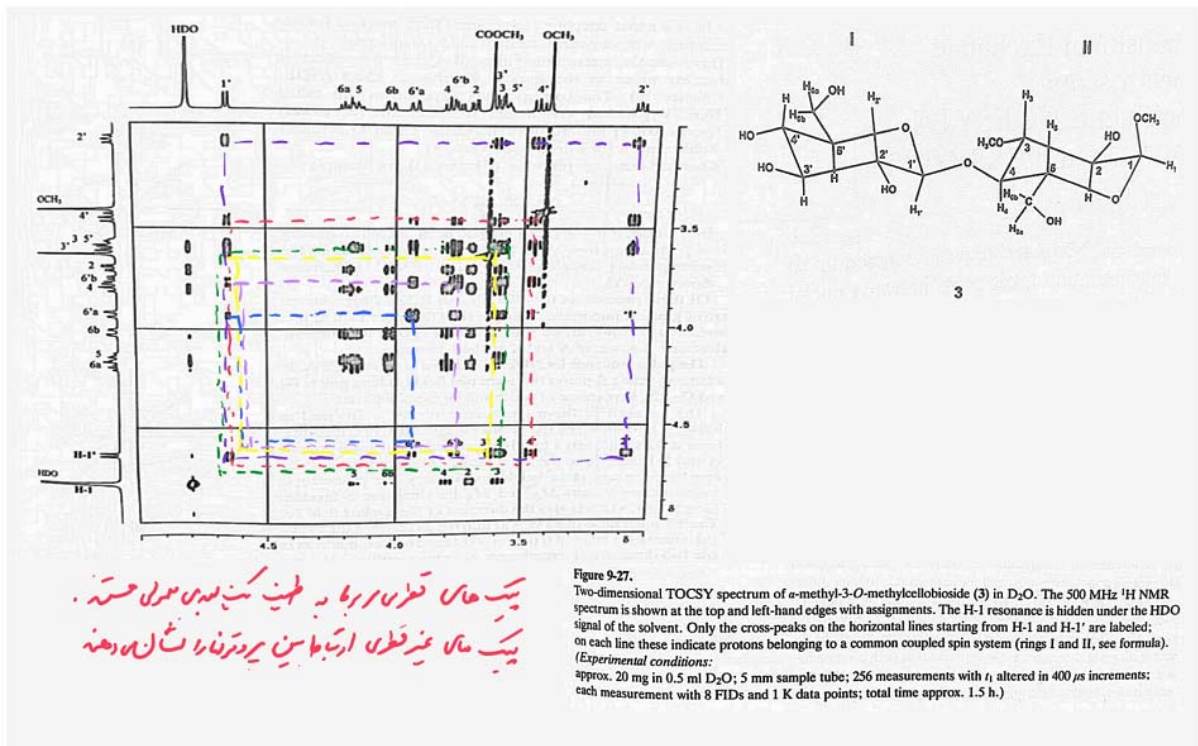
starting point	$^{13}\text{C}$ NMR signals assigned	$\delta(^{13}\text{C})$ [ppm]
H-3a	C-1	171.50
	C-2	100.32
	C-4	67.51
	C-5	52.83
H-4	C-3	40.31
	C-5	52.83
H-5	C-6	71.67
H-7	C-5	52.83
	C-8	70.98
	C-9	64.50
H-9'	C-7	69.18
	C-8	70.98
	C-8	70.98
OCH <sub>3</sub> (ketoside)	C-2	52.12
OCH <sub>3</sub> (ester)	C-1	54.65
CH <sub>3</sub> (Ac)	CH <sub>3</sub> (d)	23.20
	C=O (ac)	175.93

## The TOCSY Experiment

$90^\circ - t_1 - \text{Spin-lock} - \text{FID}$

n اندازه گیری با افزایش تداوم  $t_1$  انجام می شود

\* با حرکت اشغال ردیف‌ها می‌توان در مکتول، امکان تشخیص ایزومرهای  
تاسی بر دترهای که در یک سیستم اسپین با آن جفت می‌شوند فراهم می‌گردد



انتقال تأثیرات متقابل در قطب - در قطب از طریق نفا

NOESY

Nuclear Overhauser Enhancement (or Effect)  
Spectroscopy

ROESY

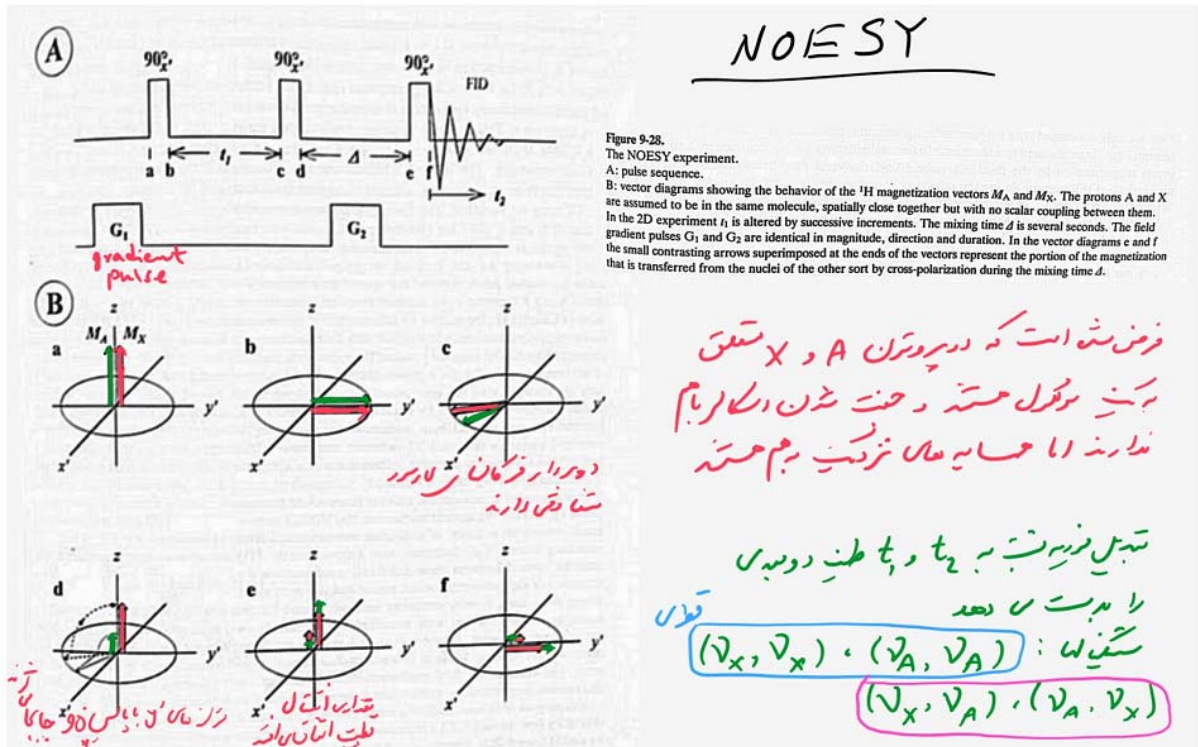
Rotating frame Overhauser Enhancement  
(or Effect) Spectroscopy

EXSY

EXchange Spectroscopy



اگرچه A پایداری شود، انتقال قطب از A به X  
بنا به سرعت k انجام خواهد شد.



## The Two-Dimensional INADEQUATE Experiment

**I**ncredible  
**N**atural  
**A**bundant  
**D**ouble  
**Q**uantum  
**T**ransfer  
**E**xperiment

$$1D: 90^\circ_x - \tau - 180^\circ_y - \tau - 90^\circ_x - \Delta - 90^\circ_\phi - FID(t_2)$$

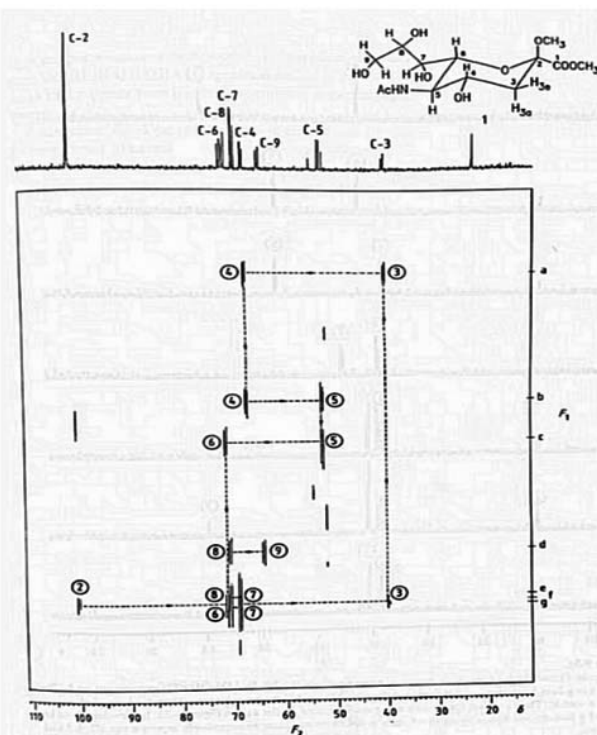
$$2D: 90^\circ_x - \tau - 180^\circ_y - \tau - 90^\circ_x - t_1 - 90^\circ_\phi - FID(t_2)$$

establish double quantum coherence

converted into single quantum transitions

double quantum coherence evolves

برای هر جفت هسته  $^{13}C$  که بطور مستقیم  
بدعم مستقیم شده اند در دامنه حاصل می شود  
A و X



- بیشتر از طریق  $^{13}C$  در این نشان داده شده است  
- سیگنال مربوط به کربن نوع جدید C-2 در آنجا  
قابل تشخیص است  
- با کمک این سیگنال و با توجه به جهت شدن سیستم  
C-2 با C-3 میزان سیگنال C-3 را تشخیص داد  
در همین ترتیب سیگنالها که هسته ها  $^{13}C$  دیگر تشخیص  
داده شده اند  
- جهت تشخیص سیگنالها، ساختار اسکلت کربن مولکول  
بسیار مهم بود (که اصل آنها کربن به هم متصلند)

Figure 9-29. A portion ( $\delta = 10$  to  $110$ ) of the two-dimensional  $100.6$  MHz  $^{13}C$  INADEQUATE spectrum, with  $^1H$  BB decoupling, of the neuraminic acid derivative 1. The projection of the 2D spectrum onto the  $F_2$ -axis is shown at the top. The horizontal construction lines link the doublets of coupled pairs of  $^{13}C$  nuclei. The identification numbers alongside the signals correspond to the numbering of the carbon atoms in 1. Starting from C-2 and following the horizontal and vertical construction lines in the direction of the arrows leads to unambiguous assignments of all the  $^{13}C$  resonances.

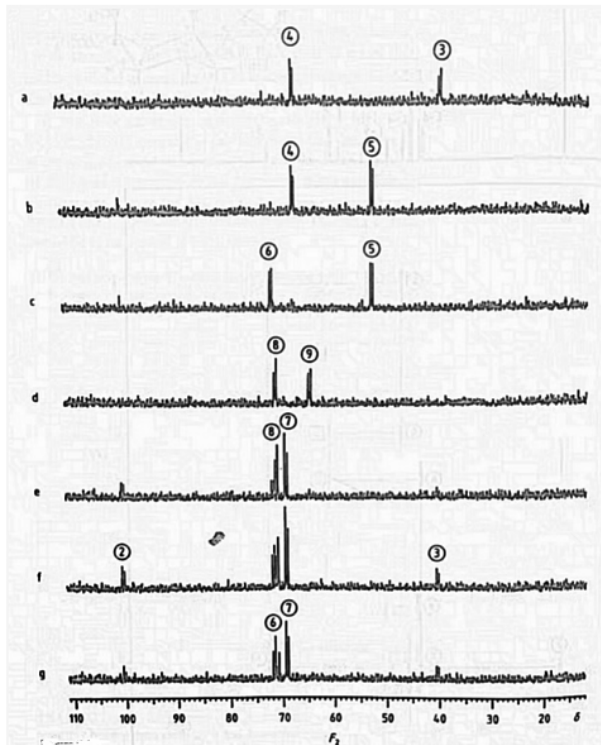


Figure 9-30.

Individual  $F_2$ -spectra for a series of different  $F_1$ -values from the 2D-INADEQUATE experiment on 1. The traces a to g have been chosen so that in each case an AB- or AX-type satellite spectrum for a coupled pair of  $^{13}\text{C}$  nuclei is visible. The  $F_1$ -values for spectra a to g are indicated on the axis in Figure 9-29. It is evident from traces e, f and g that the resolution along the  $F_1$ -axis is not quite adequate to completely separate the 2/3, 6/7 and 7/8 pairs.

(Experimental conditions:

167 mg of the compound in 2.3 ml  $\text{D}_2\text{O}$ ; 10 mm sample tube; 128 measurements with  $t_1$  altered in 50  $\mu\text{s}$  increments; each measurement with 576 FIDs; total time 66 h.)

## 9.6 Summary of Chapters 8 and 9

Table 9-4 summarizes the types of information that can be obtained using the various techniques described in Chapters 8 and 9. This makes it easier to compare the different methods.

Table 9-4.

Types of information obtained from different multiple pulse experiments.

Experiment	Nuclides observed	Types of information and applications
<b>One-dimensional (1D) techniques</b>		
<i>J</i> -modulated spin-echo (attached proton test, APT)	$^{13}\text{C}$	CH and $\text{CH}_3$ carbon nuclei give positive signals, quaternary and $\text{CH}_2$ carbon nuclei give negative signals (an aid to assignment).
INEPT	$^1\text{H}/^{13}\text{C}$	The INEPT pulse sequence is used as a component of many two- and multidimensional experiments (example: HSQC).
DEPT	$^{13}\text{C}$	Tells how many hydrogen atoms are directly bonded to a carbon nucleus: CH, $\text{CH}_2$ , $\text{CH}_3$ . Disadvantage: no signals from quaternary carbon nuclei.
Selective TOCSY	$^1\text{H}$	Allows one to identify all the protons belonging to a common coupled spin system.
1D-INADEQUATE	$^{13}\text{C}$	C,C coupling constants.

**Two-dimensional (2D) techniques**

Heteronuclear <i>J</i> -resolved <sup>13</sup> C NMR spectroscopy	<sup>13</sup> C	C,H coupling constants, number of directly bonded hydrogen atoms (as in DEPT).
Homonuclear <i>J</i> -resolved <sup>1</sup> H NMR spectroscopy	<sup>1</sup> H	Determining $\delta$ -values in complicated spectra, identifying the peaks of a multiplet.
H,H-COSY	<sup>1</sup> H	Assigning signals in complicated spectra.
Long-range COSY	<sup>1</sup> H	Assigning signals of protons separated by four or more bonds where the couplings are small.
H,C-COSY	<sup>1</sup> H/ <sup>13</sup> C	Assigning signals in the <sup>1</sup> H and <sup>13</sup> C spectra, starting from known signals.
Heteronuclear multiple bond correlations HMBC	<sup>1</sup> H/ <sup>13</sup> C	Assigning <sup>1</sup> H and <sup>13</sup> C signals on the basis of <sup>2</sup> <i>J</i> (C,H)- and <sup>2+n</sup> <i>J</i> (C,H)-values.
2D-TOCSY	<sup>1</sup> H	Allows one to identify all the protons belonging to a common coupled spin system.
NOESY, ROESY	<sup>1</sup> H	Gives evidence for spatial proximity of nuclei.
EXSY	<sup>1</sup> H	Qualitative evidence of exchange processes.
2D-INADEQUATE	<sup>13</sup> C	Assigning signals by detecting couplings between adjacent <sup>13</sup> C nuclei.