NMR對樣品製備要求

1. 溶劑應大於0.5 mL，若溶劑量少，掃描速率會變慢，得到峰形不佳
2. 樣本不應有固體微粒或沉澱，應先過濾去除
3. 樣本濃度要求：H譜 10 mg/0.5 mL；C譜 >10 /0.5 mL且濃度越大越好
4. 樣本中不應含任何磁性物質
5. 樣本管應平直，粗細均勻，不能有任何金屬存在，亦不能用金屬捆綁固定；儘量不要貼標籤紙，以免影響樣本管在磁圈內旋轉；樣本管必須使用專屬的管帽，並蓋緊以防泄漏或揮發，可使用封口膜再密封
6. 重覆利用的樣本管清洗後可使用氮氣吹乾，烘乾時溫度不超過100℃
7. 實驗室室溫過高時，可能會影響磁場穩定性，建議暫停實驗
8. (1) D-solvent 是用來鎖定磁場，FT-NMR 一定得使用D-溶劑

(2) D-solvent是用來去除背景。

1. 一般測1H 譜時，溶劑不能用含1H成分之溶劑(需 <1 %)。

否則1H 譜將僅只能見到又大又寬之溶劑峰，這時就需要做溶劑抑制實驗(solvent suppression)

樣品可用溶劑表

|  |  |
| --- | --- |
| Acetic | Acetic Acid-d4 |
| Acetone | Acetone-d6 |
| C6D6 | Bezene-d6 |
| CD2Cl2 | Dichlormethane-d2 |
| CD3CN | Acetonitrile-d3 |
| CD3CN\_SPE | LC-SPE solvent (Acetonitrile) |
| CD3OD\_SPE | LC-SPE solvent (Methanol-d4) |
| CDCl3 | Chloroform-d |
| CH3CN+D2O | HPLC solvent (Acetonitrile/D2O) |
| CH3OH+D2O | HPLC solvent (Methanol/D2O) |
| D2O | Deuterium oxide |
| D2O\_salt | Deuterium oxide with salt |
| DMF | N,N-dimethylformamide-d7 |
| DMSO | Dimethylsulfoxide-d6 |
| Dioxane | Dioxane-d8 |
| Juice | Fruit Juice |
| MeOD | Methanol-d4 |
| None | No solvent |
| Plasma | Blood plasma |
| Pyr | Pyridine-d6 |
| TFE | Trifluroethanol-d3 |
| THF | Tetrahydrofuran-d8 |
| T\_H2O+D2O+Me4NCL (CD3)4NCl in 90%H2O and 10%D2O | for NMR thermometer |
| T\_H2O+D2O+NaAc sodium acetate in 90%H2O and 10%D2O | for NMR thermometer |
| T\_H2O+D2O+Pivalate pivalate –d9 in 90%H2O and 10%D2O | for NMR thermometer |
| T\_MeOD | Metanol-d4 for NMR thermometer |
| Tol | Toluene-d8 |
| Urine | Urine |
| oC6D4Cl2 | *ortho-*dichlorobenzene-d4 |
| pC6D4Br2 | *para-*dibromobenzene-d4 |

NMR手動調諧流程

指令碼

打樣：

ej 升起樣品

ij 放入樣品

lock 鎖場，選擇溶劑

edlock 加入新溶劑在列表中

new建譜，更改參數，可調ns (number of scan)

getprosol 使用新設置的參數

rga增益

topshim 勻場

atma 調諧

halt 樣本掃描中途急停

zg 採樣

增加掃描次數(例)：ns 1000;go 額外再加1000次掃描次數

 ns 500;zg 重打譜，掃描次數500

lock off 解場：每手動打一個譜前先lock 鎖場

 打完譜要結束便lock off解場

看數據：

 1D：etp; apk; abs

 2D：xfb; abs2d

ATMA/ATMM ( 中文版)

|  |
| --- |
| ATM 的使用及注意事項ATMA = Automatic Tuning Matching Automatically (全自動)ATMM = Automatic Tuning Matching Manually(以指令代替手動)目的：調諧探頭之最佳操作頻率 方法：利用atma或 atmm 指令完成 配合：先 rpar;再 atma(rpar是讀入實驗參數; atma是探頭調諧)正確之使用後果：探頭核種因atma變換後與par(參數巨集)一致，異核實驗才有訊號不正確使用後果：探頭核種頻率可能與par不一致，異核實驗沒有訊號。另探頭及射頻放大 (transmitter)器將因能量不當反射而耗損結論：BBFO探頭作異核實驗時，請記得ATMA以保護儀器ATMA 注意事項：1. 選擇含有要調整之Nucleus 之一維之parameter set

Read a Par set for Nucleus to be tuned (rpar)2.  ATMA 是比較安全的，  惟有時執行過快，無法及時見到結果時，  可以ATMM 檢視之或微調之3.  只作一維1H譜時，可不用調ATM (除非特稀 diluted samples)4.  因為是寬頻，異核之間的切換得靠ATMA 來完成5.  作一維13C譜時，不確定BB 頻率是否在13C時，   需要調整ATM，可以tr data 後，    efp 檢查是否確實為13C 圖譜For 13C-nucleus detection, ATMA must be done and data should be transferred using tr and efp during the beginning of acquisition to check if the solvent peak come out or not.(except for D2O)6.  作二維實驗時，  需將所有相關核種(包括1H) ATMA 做好，  再測90度pulse width. ATMM 注意事項：1.   ATMM 需要一邊觀察，一邊調整，若任意調整將嚴重損害探頭。  若需送修，儀器就需停機，因探頭貴重只買一只2.   ATMM 以(手按)指令(代替手轉動調桿)去啟動馬達來調整 bbfo探頭之tuning 及 matching，執行不當時是會損害探頭，請務必小心再小心。ATMM is function through clicking shift > or < in tune table.(Try either direction)3.   不論ATMA或 ATMM，若 wobble curve 不出現，可先停止。   Exit Topspin 再重新來過。4.   調整ATMM時，壓完位移鍵，wobble curve有移動時可繼續壓下一次位移，   若wobble curve沒有移動，   請等2~3 秒再按下一次位移，   切不可連續不停的按。Wait for the reaction of wobble curve after clicking the shift key in ATM table. (It is suggested to wait 2~3sec for each clicking of the shift key)5.   tuning 控制橫向，matching 控制縱向，最佳位置在X與Y交點6. 若樣品從有機溶劑換成水溶液時，   請使用ATMM，因ATMA較易超出範圍。何時作ATMA ?( When)a) doing X-Nucleusb) measuring 90 pulse width of 1Hc) doing 2D experimentd) changing X Nucleus from one another (e.g. from 13C to 31P) 何時作ATMM ? (When)a) for checking or observing onlyb) for precise tuning and matching (need only with 2D expts) Procedure of ATMA or ATMM1) rpar bbfo\*\_\_\_\_(Parameter Set) , [e.g.  rpar  bbfo\_13c (tune for 13C and 1H)]2) getprosol3) atma4) atmm  (for checking only)5) stop  (select the exit (x) mark) getprosol此指令將儀器矯正後之相關各樣脈衝能量 (power level and pulse width)從prosol表中代入ParSet 參數巨集中 SoftwareNucleus frequency can be checked by command sfO1 (console) Pulse Program can be checked by command pulprog 提醒:請勿隨意使用Bruker Default大寫之PAR。如否，後果自負(需要自己檢查)。有特別實驗需要者，請先尋求協助討論參數之設定。 |

1H experiment of BBFO Probe

**1.  在BSMS panel按lift,**

**(Press Lift on BSMS panel with DPX 400 in Room A525 or find icon bsmsdisp with AVIII NMR in room B662)**

**2. 將樣品管擦拭乾淨，套入轉子，量好深度並於空氣浮力中置入磁體之探頭中(深度=2.0cm )**

**Wipe the NMR tube with tissue, insert to the spinner, adjust the depth with gauge and place it on the cushion of air at top of magnet.**

**3.  再按一次lift, 解除浮力**

**Click again the lift to release the air pressure for the sample to drop down to the probe gently.**

**4.  鎖場lock (選擇solvent)**

**Use field to find out the Deuterated**

**signal of solvent , or type in lock (solvent name)**

**5. Shimming(5F DPX) 或 topshim(6F AVIII)**

**Use manual shimming z, z2, for DPX and topshim for AVIII 400NMR**

**6.  shim x, y (with nonspinning  mode)**

**7.  edc ( Note: expno為置放實驗fid處, procno為置放數據處理區）**

**expno in edc means place to keep fid. For each new experiment changes a new expno.**

**procno means different place for storing different way of processing data with the same expno(raw data).**

**8.  rpar bbfo\_1h (read in the parameter set of expt, eg: acqu,proc,plot, title etc)**

**9.  getprosol (get the correct pulse width with the current probehed)**

**10.**  **atma (automatic tuning and matching )**

**11.**  **rga  (receiver gain auto-adjust)**

**12.**  **ns =1 (for shimming checking)**

**13.**  **zg;ft;pk**

**14.**   **ns = 16**

**15.**   **zg**

13C Experiment of BBFO Probe

1. **Take a normal 1H to check the condition of shimming.**
2. (**先測 1H, 將shimming 勻好)**
3. **edc ( Note: expno為置放實驗fid處,**
4. **procno為置放數據處理區）**
5. **(expno : set before acquisition, procno : set after acqusition.)**
6. **rpar  bbfo\_13c  (read in the parameter set of expt, eg: acqu,proc,plot,title etc)**
7. **getprosol (get the correct pulse width with the current probehed)**
8. **atma  (automatic tunning and matching )**
9. **rga  (receiver gain auto adjust)**
10. **(For DPX-400MHz skip the rga step, set rg =16k )**
11. **ns = 1k or else**
12. **td0= 9 ~10  (for overnight)**
13. [ td0=n (n>1), td0 is used to store and loop for n times of NS]
14. **zg**

**ps1. Data should be check if solvent peak comes out or not**

**after ns=1 (except D2O solvent),use tr before efp.**

**ps2. Use halt to terminate the expt, use tr before efp**

**to monitor the experiment.**