

## QOne 仪器匀场方法

### 匀场用到的标准样品: 0.1 mg/ml GdCl<sub>3</sub> in 1% H<sub>2</sub>0 in D<sub>2</sub>0 和 1% CHCl<sub>3</sub> in Acetone-d<sub>6</sub>

一. 测试 3D 匀场标样(0.1 mg/ml GdCl<sub>3</sub> in 1% H<sub>2</sub>0 in D<sub>2</sub>0)的氢谱

1. 将 3D 匀场标样(0.1 mg/ml GdCl<sub>3</sub> in 1% H<sub>2</sub>0 in D<sub>2</sub>0)放入磁体

在命令行输入 *aij(int)*进样(配备自动进样器)。在弹出的对话框中,Sample 栏输入样品信息,Solvent 栏选择样品溶剂 D2O。

Samp	le	
Sample		
Solvent	D2O	~
	OK	Cancel

#### 2. 新建实验

菜单栏点击 Acquire,在弹出的下拉菜单中点击 New Experiment,在右侧子菜单中点击 Create。

File	Acq	uire	Process	Analysis	V	iew	Op	tions	To	ols I	Help	8	
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此时软件界面弹出新建实验(New Experiment)对话框,填入或选择以下内容:

Dir: 数据存放目录,点击右侧 指定存储路径,例如 admin/Shim。

Exp Name:填入实验名称,例如1。

Params Type: 选择 template, 点击右侧, 在弹出的 Templates 对话框中选择 PROTON1。 Solvent: 点击右侧下拉箭头选择 D2O。

Title: 填入针对当前实验的说明信息。

点击 << Advanced ,	勾选 <b>☑</b> Get	Probe Parameters
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Experiment         New Experiment         Dir       admin/Shim         Dir       admin/Shim         Dir       admin/Shim         Exp Name       1         Params Type       template         PROTON1          Solvent       D20         D20       PROTON1         Solvent       D20         Solvent       D20         Copy Parameters       20 HI/C13 HSQC with Cho/An.         Solvent       D20         Solvent       D20 NOSVW With TPIm	
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	PROTON1.J
First Court	

点击 Finish 按钮。此时软件主界面显示新建的1工作区。



3. 调谐

选择刚刚新建的1工作区,在命令栏输入命令 stm,等待自动调谐结束。

4. 锁场

在命令栏输入命令 alock, 等待锁场结束。

5. 匀场

在命令栏输入命令 smartshim, 等待匀场结束。

6. 参数设置与采样

在命令栏输入命令 getprobe, 读取探头参数;输入命令 again, 自动调整接收机增益;输入命令 go, 开始采样。

7. 数据处理

采样完成后在命令栏依次输入命令 ft, aph, dc (必要时需进行手动相位调节)。

8. 查看水峰半高宽

在命令栏输入命令 res, 查看 50% 对应的值。



将该值与安装时工程师测的值进行比较,如果此次测试的值较大,则进行第二步。如果此次测试的值 与之前的相当,则进行第三步操作。

二. 3D 匀场

9.3D 匀场

菜单栏点击 Acquire,在弹出的下拉菜单中点击 Shim,在右侧子菜单中点击 3D Smart Shimming。软件 左侧将显示 3D 匀场界面,点击 Start,等待匀场过程结束。

					3D Smart Shir	mming 🛛 🖴 NMR Data	Navigator
File	Acq	uire Process Analysis	Q	nmr View Options Tools He		3D Smart	t Shim
Ē	U	Sample	>	↓ ∫ 八≝ ┯ ┉ ᠕ ┍	Nucleus	D	Phase Code 4x4
Þ	∎ ∤	New Experiment Tuning and Matching	>			Browse	Show Map
<b>झ</b> s	S.	Shim	>	Manual Shimming	Channels	Default	Edit
	-	Lock	>	Search Shimming	Before	off	
× 1	Ŷ	Spin		Smart Shimming	After	off	
	m	Acquisition	>	3D Smart Shimming	AutoLock		
	0	Automation				Start	Stop

10. 重新采集氢谱,查看水峰半高宽

在软件左侧 NMR Navigator 的 1 号实验节点点击鼠标右键,选择 New Experiment。在弹出的 New



Experiment 对话框只需填入 Exp Name 如 2。确认 Params Type 为 current node parameters。点击 Finish 按钮。 软件主界面显示 2 工作区。输入命令 *go*,开始采样。采样完成后在命令栏依次输入命令 *ft, aph, dc, res*。 查看水峰半高宽是否变小。

			🚺 New Expe	riment			×
			Experiment New Experin	nent			
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	/ •••		Exp Name	2			
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v 🖴 2.nmr [s1pu		New Experiment					
🖬 acqupara	6	Password					
procpara		Show Log		Fin	ish 🔄	Cano	el

11. 如果水峰半高宽变小,则重复 9-10,直至水峰半高宽与安装时工程师测的值相当。

12. 如果水峰半高宽不变或者变大,则在 3D 匀场界面选择 Make Map,点击 Start,开始匀场。再重复步骤 9-10 直至水峰半高宽降到合适范围。

	3D Smar	t Shim				
Nucleus	D	Phase Code 4x4	~			
Shim Map	default					
	Browse	Show Map				
Make Map	Show Workspace	e Setting				
Channels	Default	Edit				
Before	e off					
After	off					
Evaluate FIDArea						
AutoLock						
	Start	Stop				

三. 测试线形指标

13. 将线形标样(1% CHCl<sub>3</sub> in Acetone-d<sub>6</sub>)放入磁体

在命令行输入 aij(int)进样。在弹出的对话框中, Sample 栏输入样品信息, Solvent 栏选择 Acetone。

😏 Samp	le	×
Sample		
Solvent	Acetone	~
	ОК	Cancel

#### 14. 新建实验

在软件左侧 NMR Navigator 的 1 号实验节点点击鼠标右键,选择 New Experiment。在弹出的 New Experiment 对话框填入 Exp Name 如 3; Params Type 选择 template,选择 H1 Lineshape Non-Spinning;点击

<< Advanced, 勾选 Get Probe Parameters; 点击 Finish 按钮。软件主界面显示 3 工作区。



New Exper	riment			×
New Experim	nent			
Dir Exp Name	admin/Shim 3			
Params Type	template ~	H1 Lineshape Nor	n-Spinnii	ng
Title	d			*
Get Prob	e Parameters ameters ord			
		Finish	Cance	el

15. 调谐

选择刚刚新建的3工作区,在命令栏输入命令stm,等待自动调谐结束。

16. 锁场

在命令栏输入命令 alock, 等待锁场结束。

17. 匀场

菜单栏点击 Acquire,在弹出的下拉菜单中点击 Shim,在右侧子菜单中点击 Smart Shimming。



在软件左侧显示的匀场界面中, After 栏选择 After z1\_z2\_x1\_y1\_zx\_zy\_z1, Evaluate 栏选择 Evaluate FIDArea, 勾选 AutoLock, 点击 Start, 开始匀场。

	Smart Smin	
Solvent	Acetone	
Shim Map	default	
	Browse	Show Map
O Make Map	Show Workspace	Setting
Before	off	~
After	z1_z2_x1_y1_zx_zy_z1	~
Evaluate	FIDArea	~

18. 参数设置与采样

点击软件主界面进入3工作区,在命令栏输入命令getprobe,读取探头参数;输入命令go,开始采样。

19. 数据处理

在命令栏依次输入命令*ft, aph, dc*(必要时需进行手动相位调节)。

20. 在命令栏输入命令 res, 查看氯仿信号 50%, 0.55%, 0.11%对应的值。

Resolution		X				
<ul> <li>Spec line width: 50% = 0.47 0.55% = 4.6 0.11% = 8.0 DigitalRes = 0.015 Hz Peak: 8.04 ppm</li> </ul>						
	 		L.,			

- 21. 如果没达到指标可以重复匀场一次(步骤17)。
- 四. 保存场值
- 22. 保存场值

在命令栏输入命令 *ssh*,在弹出的 CheckPassword 中输入管理员密码,在 Select Save Path 中将两个选项都勾选。

🔄 Check Password 🛛 🗙	Select Save Path X
Password:	Please select shimvalue path you want to save! ✓ Save to default system path. ✓ Save to current solvent path.
OK Cancel	OK Cancel



# 自动化实验 Automation 中与匀场相关的设置

1. 用 admin 用户登录 Automation,点击自动化实验工具栏 Preference 选项,弹出 Preference 窗口。

Automation 
 Automati

2. 点击左侧 General Options,在右侧 Load Shim Value 选择 Always use shimvalue in system path。

Preference			×
<ul> <li>Automation Configuration General Options Auto sample changer External Setup Accounting</li> <li>User Settings User Manager User Preference</li> </ul>	General Options		
	Data Directory Automation data root directory: D:/Da	ta/1/Automation	
	Data format 1 root directory: D:/Data/1/Automation/Data1		
	Data format 2 root directory: D:/Data/1/Automation/Data2		
	Max Experiment Size Maximum number of displays for autor	natic experiments: 500	
	Experiment Directory Default Experiment Directory: Always t	ise default	~
	Subgroup Information: Show		
	Load Shim Value Load shimvalue before GradShim: Alwa	ys use shimvalue in system path	~
	Default Priority Priority Sample Handling: Wait until cu	irrent sample completed	~
	Resubmit the failed experiment Web Server		
	Web Server Login Address: 127.0.0.1		
	Web Server Port: 8081		
	Web Server Timeout(s): 1800		
	Default Time Settings		
	AutoSample Time(s): 62	Lock Time(s): 60	
	STM(H1) Time(s): 50	STM(X) Time(s): 55	
	Gmapshim Time(s): 120	Gshim Time(s): 70	
	Gain Time(s): 11	Search Shim Time(s): 200	
	Temperature Time(s) 300	Spin Time(s) 300	
			01
			UK

3. 点击左侧 User Preference,在右侧勾选 Use default shimming value before gradient shimming。注意每一个 用户都需要勾选。

<ul> <li>Automation Configuration General Options Auto sample changer External Setup Accounting User Settings User Manager User Preference</li> </ul>	User Preference		
	User Infomation Group	1	Change
	User: Default Experiment: Default Solvent:	admin	~
		PROTON16	~
		CDCI3	~
	Default Experiment I STM After a s	Procedure: solvent $\checkmark$ AutoLock After sample c $\checkmark$	
	Default Shim Proced	Interest of the second se	
	Use default shim Evaluate: LOCKLEVER	ming value before gradient shimming	
	Default Data Process Window Function Auto Phase Corre NUS reconstructi Algorithm of recons ST	s Procedure: n & Fourier Transform cetion Z Auto Baseline Correction on truction O Deep learning	
	Send Email		
	From Email Protoco	smtp.exmail.qq.com	~
	From Email Account From Email Passwor	d:	
	Target Email Accour	nt:	

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