

纯化学位移实验测定



1. 一维实验测定(以¹H为例)

做pureshift实验,先测普通氢谱,勾选force tune,得到氢谱。

▶ 脉冲选择: (delta5.3.1以上版本可直接调出)



→pureshift→pureshift_1d_psyche



➤ 实验参数设定: Acquisition

中心频率与谱宽根据普通氢谱信号的化学位移分布范围确定







Delta v5.3的界面

➤ 实验参数设定: Acquisition

x_offset 与spectrum_width根据普通氢谱信号的化学位移分布范围确定 x_acq_time: 20[ms], x_acq_time越小,灵敏度越好,分辨率越差

y_points: 32, y_points越大, 谱图分辨率越好, 但测样时间越长

Header Instrument	Acquisition Pulse Diagram 🏠 Favorites			
x_offset	4[ppm]			
x_sweep	8[ppm]			
x_acq_time	20[ms] 推荐使用默认值20ms			
j_calc	25[Hz]			
x_points	64			
x_resolution	49.97277[Hz]			
scans	4			
x_prescans	4			
mod_return	4			
y_points	32 推荐设为32			

▶ 实验参数设定: Pulse

band_width_psyche、chirp_pulse_psyche及chirp_atn_psyche



设定完成提交实验即可。去耦效果不好或灵敏度太低则优化以上三个参数

▶ 实验示例



^{*}指chirp_atn_psyche_calc+

Delta v6.0以上的界面

▶ 实验参数设定: Acquisition

x_offset 与spectrum_width根据普通氢谱信号的化学位移分布范围确定 x_acq_time: 20[ms], x_acq_time越小,灵敏度越好,分辨率越差 y_points: 32, y_points越大,谱图分辨率越好,但测样时间越长

Header Instrument Acquisition Pulse Diagram 🕎 Favorites				
x_offset	5[ppm]			
spectrum_width	20[ppm]			
x_acq_time	20[ms] 推荐使用默认值20ms			
j_calc	25.0[Hz]			
x_points	640			
x_resolution	49.97277[Hz]			
scans	4			
x_prescans	4			
mod_return	4			
y_points	32 推荐使用默认值32			

▶ 实验参数设定: Pulse

obs_psy1_m_fsweep、obs_psy1_m_pulse及obs_psy1_beta_deg



▶ 数据处理:

实验结束,打开数据如以下界面,直接点开数据书写板 🛓 即可查看。



▶ 数据处理:

调相位、标峰、积分等处理可直接在此界面进行。





如需更改傅里叶变换前的处理列表,请按以下步骤操作:





鼠标选中此窗口

在此界面可进行与普通一维谱图相同的处理



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示例样品: 0.5mM Sucrose的重水溶液 测试参数参考: x_acq_time: 20[ms]; y_points: 32; obs_psy1_m_fsweep: 4[kHz]; obs_psy1_m_pulse: 60[ms]; obs_psy1_beta_deg: 15[deg]。

2. 二维实验测定(此处介绍常用的TOCSY及HSQC实验)

▶ TOCSY实验脉冲选择:

pureshift_2d_f1_psyche_tocsy.jxp

pureshift_2d_f1_tocsy.jxp

Delta v5.3

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Delta v6.0

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➤ 实验参数设定: Acquisition(与普通TOCSY测试相同)

x_offset 、x_sweep、 x_acq_time根据一维氢谱设定

x_points: 1024; y_points: 256; 也可视谱图要求自行设定,其他使用默认值。

Header Instrument Acquisition Pulse Diagram ☆ Favorites				
x_domain	Proton			
x_offset	3.97619[ppm]			
x_sweep	4.08066[ppm]			
x_points	1024			
scans	4			
x_prescans	4			
mod_return	1			
y_points	256			
x_acq_time	0.62769[s]			
x_resolution	1.59314[Hz]			

▶ 实验参数设定: Pulse

设定mix_time, PSYCHE条件参数与一维pure shift ¹H谱中参数相同,其他参数默认

Header Instrument Acquisition Pulse Diagram 🏠 Favorites					
mix_time	80[ms]				
mix_time_loop	169				
total_mix_time	80.06844[ms]				
	PSYCHE condition —				
obs_psy_shape	SALTIRE_CHIRP				
obs_psy1_m_fsweep	4[kHz]				
obs_psy1_sweep_ppm	10[ppm]				
obs_psy1_m_pulse	60[ms]				
obs_psy1_beta_deg	15[deg]				

设定完成提交实验即可。

TOCSY实验之前应做好一维氢谱的pure shift实验, 完成参数优化,二维实验不再优化参数。 ▶ 数据处理:

实验结束,打开数据如以下界面,①点击 🔃,回到数据处理界面; 鼠标选择Y轴处理列表,②添加Covariance,③修改为X。



> 数据处理及实验结果:

再点开2D浏览器 🛅 即可查看处理。



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2. 二维实验测定(此处介绍常用的TOCSY及HSQC实验)

▶ HSQC实验脉冲选择:

pureshift_2dacq_edit_cpmg_hsqc_pn.jxp

pureshift_2dacq_cpmg_hsqc.jxp

Delta v5.3

Delta v6.0

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	Experiment File Categories: 2d, hsqc, pure_shift, liquids File Filter: ^.*\.(jxp ex2)\$				pureshift_2dacq_ghsqcad.jxp Experiment File Categories: 2d, hsqc, pureshift, liquids		

➤ 实验参数设定: Acquisition(与普通HSQC实验参数相同)

x_offset 、x_sweep、 x_acq_time、 x_points、y_points同上述设定相同。
y_offset 、y_sweep根据碳谱信号化学位移分布范围而定。

▶ 实验参数设定: Pulse

设定acq_loop: 32; 其他参数与普通HSQC参数相同,使用默认值即可。

Header Instrument Acquisition Pulse Diagram 🕎 Favorites				
Acquire parameter				
acq_loop	32			
pureshift_x_points	5120			
pureshift_x_resolution	0.78082[Hz]			

设定完成提交实验即可。

▶ 数据处理:实验结束,打开数据如以下界面 delta V5.3:



> 数据处理:实验结束,打开数据如以下界面

delta V6.0:



▶ 数据处理:





▶ 实验结果:



(a) Sucrose的HSQC谱图; (b) Sucrose的F1维pure shift的HSQC谱图

其他二维实验设定与上述实验类似,如遇问题请联系工程师。