

JEOL – ECS 400 – Delayed Data Acquisition

1. Insert the sample, select the solvent, auto-lock and gradient-shim as described in 1D NMR Measurement Set-up and Protocol.

User Shims

button and then on **III**. This will open

In the Sample window, click on the Save Shim dialog box:

💋 Save Shim File		<u> </u>	
Path: C: • C:			
Format: JEOL : Delta	♣ Filter: ▼		
Directory	Filename	Version	
- Favorites -	eclipsel.ttu.edu.im_cache .scc.im_cache 040312EB 092212postpoweroutage 092212postpoweroutageA 2692_start a_temp dwp031312 dwp041415 dwp041415A		
Name: delayed_acq			
Ok Info	Delete Refresh	Cancel	

In this box, click on the button in order to save shims to your home directory, enter *Name* (in this example *delayed_acq*), and click on **OK**.

- 3. As described in *1D NMR Measurement Set-up and Protocol*, create the experiment you need to run and adjust parameters like number of scans, relaxation delay...etc.
- 4. In the **Experiment** window / **Header** tab, click on the **button**. The following box opens:

Ø.		x
round_points sawtooth scaling settle_vt slot sn_border sn_noise_start sn_noise_stop sn_signal_start sn_signal_start sn_signal_stop sn_skip start_time tsc_er_tool unload_slot		
Value: 15-FEB-2017 23:30:00		
Add	Done	

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Scroll down the list to **start_time**, enter the time that you want your experiment to start (in 24-hour format – in this example I requested February 15th, 11:30 pm). Click on **Add**.

[If you need to run several experiments on the same sample, enter the same **start_time** for all of them. The **start_time** is just the earliest time the experiment can start.]

REMEMBER TO RESERVE SPECTROMETER TIME IN THE FACES SYSTEM!

Scroll down to **slot**, enter the number for your sample location (e.g. 5), click on **Add**, and then on **Done**. After this, **Experiment/Header** should look like that:

修 Experiment Tool: si	ngle_pulse_dec.ex2		
File Tools View Options			
😸 😹 📰 💓 📰 🕼 Add 😰 Submit			
Get Acq. View: XYZABCDE			
Header Instrument Acquisition Pulse			
sn_ratio			
auto_filter	Ø		
auto_gain	0		
filter_limit	8		
force_tune	0		
save_aborted	Ø		
start_time	15-FEB-2017 23:30:00		
slot			
scc Total Collection Time: 00:50:57			

5. Switch to the **Experiment/Instrument** tab and select **File->Get Shims** menu item. This will display **Open Shim File** dialog box:

侈 Open Shim File				
Path: C: + C:\Users\delta/Documents/delta/instrument				
Format:	🕴 Filter: 🚹 🔍			
Directory	Filename Version			
- Favorites -	012313 04172014old			
 scc.backup scc.backup_01-09-2013 scc.backup_10-3-2012	102914 11132012 11132012A 11142012start 12072012 CHrysch accy dwmaw			
Info: Unknown				
Ok Info	Delete Refresh Cancel			

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Find in your home directory the shim file you saved in step 2 (e.g. *delayed_acq*). Select it and click on **OK**.

6. Click on the button. Find and select lock_state, click Add. Find and select autoshim_mode, click Add and Done. In the Experiment/Instrument tab, set lock_state to AUTOLOCK and autoshim_mode to Z1Z2. Your Experiment/Instrument tab should now look more or less like that:

September 2 Experiment Tool: single_pulse_dec.ex2			
File Tools View Options			
	E Submit		
Get Acq. View:	Get Acq. View: X Y Z A B C D E		
Header Instrument Acquisition Pulse			
shim_yz	<mark></mark> -151[Hz]		
shim_yz2	94.8[Hz]		
shim_yz3	4.4[Hz]		
shim_y2	42[Hz]		
shim_y2z	√7[Hz]		
shim_y3			
solvent	CHLOROFORM-D		
	D2O DMF-D7		
	DMSO-D6		
recvr_gain	50		
lock_state	AUTOLOCK		
autoshim_mode	[Z1 Z2 +		
scc Total Collection Time: 00:50:57			

7. Click on the **Submit** button. The experiment is added to the queue which is confirmed in the Spectrometer Control window:



🔗 Spectrometer Control		
Tools Config Queue Machine Options		
Info Connect Monitor Unlink Free		
Connect : scc		
Queue State : OWNED	Selected Job : UNKNOWN	
00_774 # delta 000:50 S#409421 single pulse 15-FEB-2017 :	32 5 decoupled gated NO 23:33:18	
Prio Slot Job Submit Time +6		
Sample Expmnt Auto Sawth View Copy		

Note that the time displayed for this job is slightly different from your setting. This results from lack of time synchronization between the workstation and the spectrometer console which usually causes no problems.

You can now submit other experiments. They will be added to the queue but will all display the same start time. Of course, they are going to be executed consecutively, according to the submission order.

8. If needed, the experiment can be removed from the queue by selecting corresponding job and



- 9. You can now reload standard sample, disconnect from the console, exit Delta and logoff from the Windows account.
- 10. After the experiment(s) is done, log back in, open Delta, and reconnect to the console. **Upload Manager** window opens automatically:



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Click on the green square under **Select** in order to select it and then click on the **Upload** button. Your data will be uploaded to the workstation and the spectrum will open in data processor. Remember to remove your sample from the probe or (if someone already loaded another sample) from the sample changer.