Contents

Basic Protocol for Using Gas Bench II

Okay, so you are now ready to run your first batch of samples in the Gas Bench. Here are the instructions to walk you through the steps.

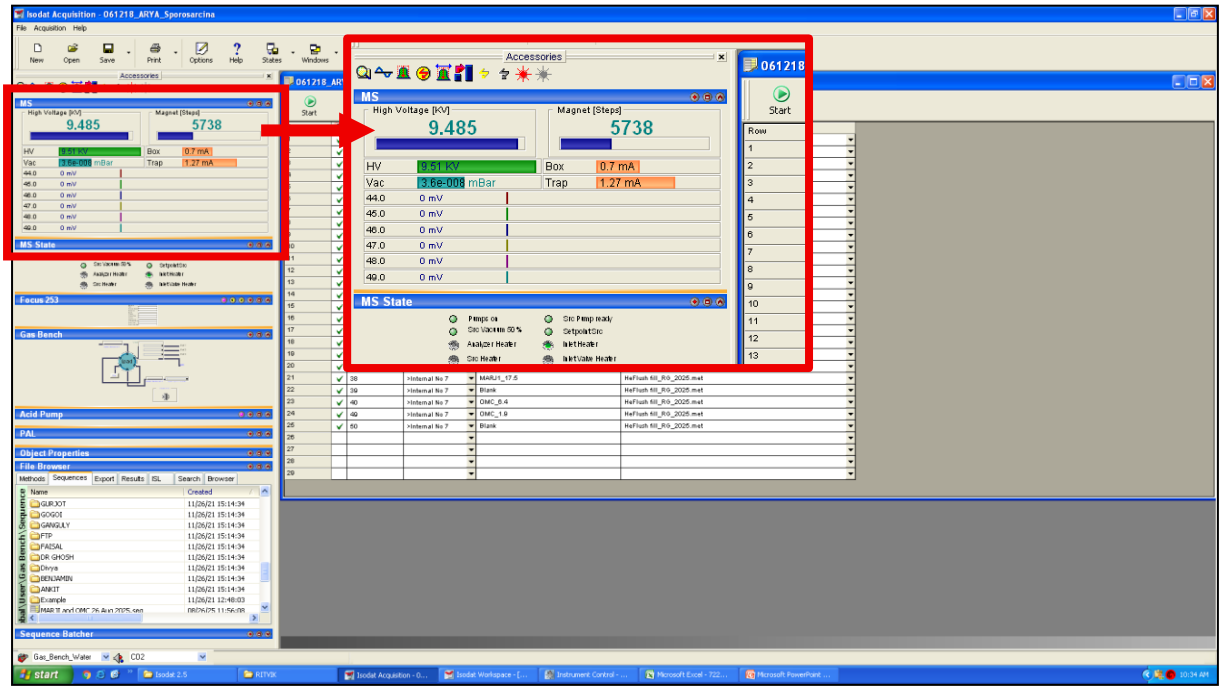
1 Consumables required

You will need:

1. Your carbonate (or water) sample or standard
2. Exetainer vials with (undamaged) caps and septa
3. Orthophosphoric acid (H_3PO_4, for carbonate analyses)
4. Syringes or small glass crucibles (‘floating boats’)
5. Isodat Acquisition and Isodat Workspace software modules

2 Initial checks

1. First things first: take a quick look at the top left corner of the left sidebar in Isodat Acquisition.

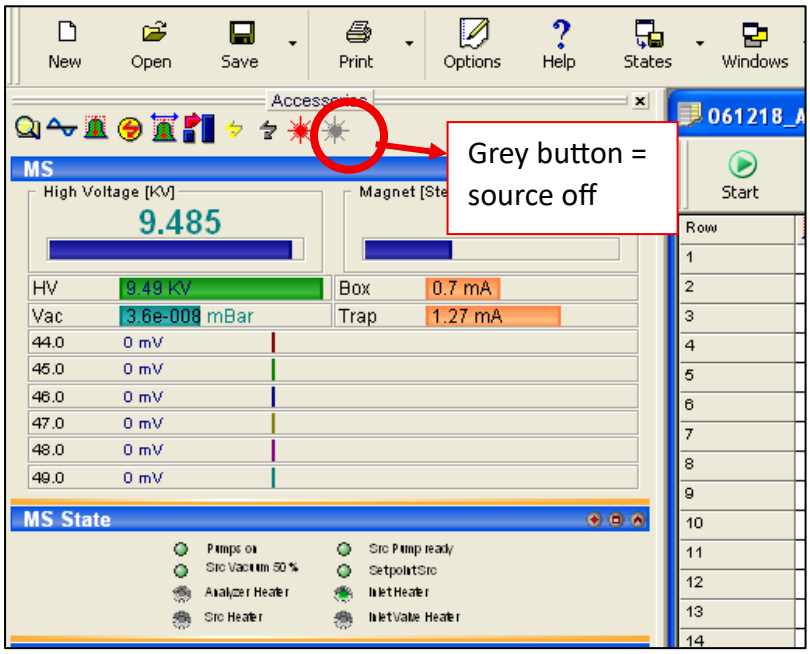


2. If the voltage (milli volts) values for 44, 45, 46 etc. are 0 mV, then it likely means that the needle valve *or* the ion source *or both* are ‘off’. In that case the vacuum value will be of the order of 10^{-8} mbar.
3. If they are even a little more than zero, then it means that both the needle valve and the

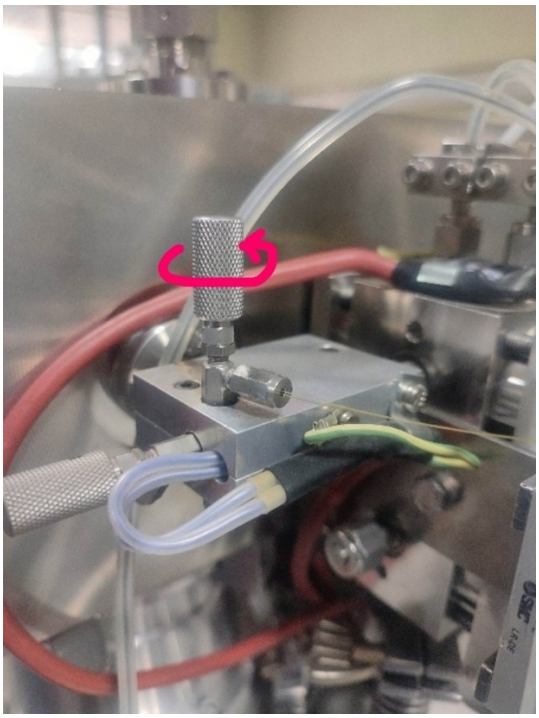
ion source are ‘on’.

If the voltage is >0 mV, then go straight to step 7 (page 4). If the voltage is 0, then proceed from step 4.

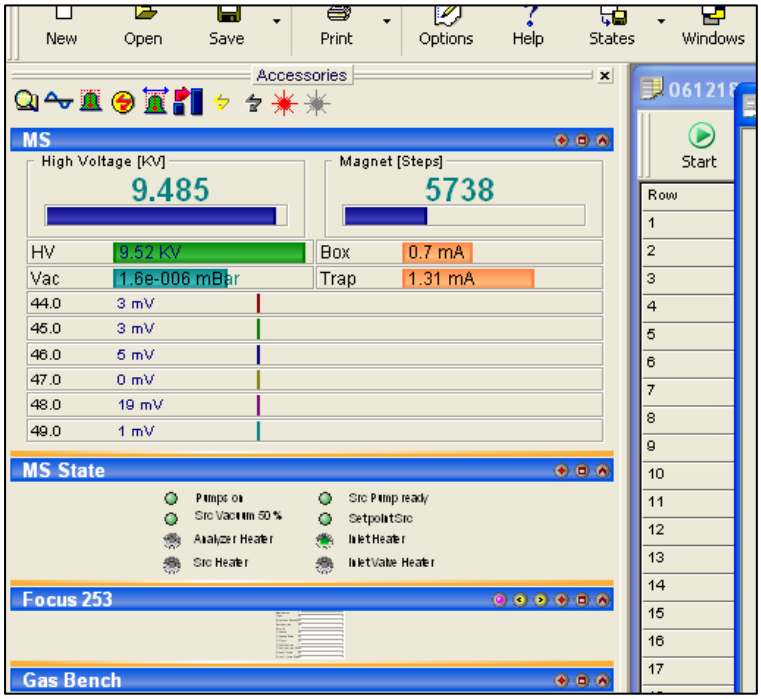
4. If the voltage = 0 mV and the vacuum is of the order of 10^{-8} mbar, it is difficult to know whether it’s due to (a) source being ‘off’ or (b) needle valve being closed or (c) both. That is why, in the event of voltage = 0 mV and vacuum = ~10^{-8} mbar at the start, switch the ion source ‘**off**’.



5. Then, walk over to the needle valve and turn it anti-clockwise (arrow shown in image).



6. At this point, you should see the voltage *increase* and the vacuum *decrease* from 10^{-8} to 10^{-6} mbar[1].

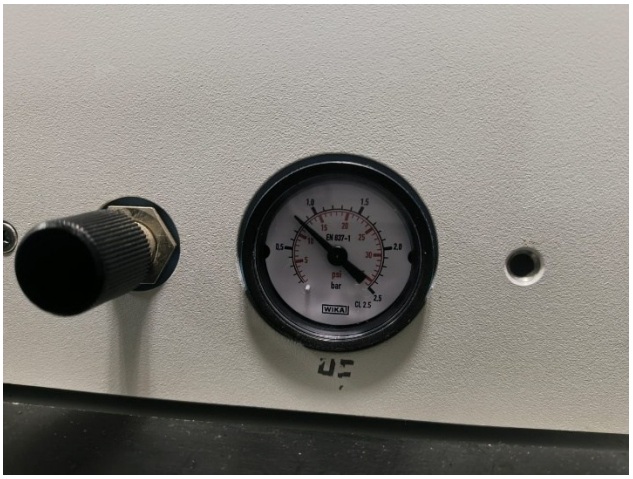


7. Once this is done, glance at the pressure gauges mounted on the wall. They are feeding CO_2 to Ref 3 in the Gas Bench[2] and Helium as a carrier gas[3] to Gas Bench.

8. Next, glance over to the helium gauge in the Gas Bench itself. Ensure that this is roughly

[1] Note that a change from 10^{-8} to 10^{-6} mbar is an *increase* in pressure and a *decrease* in vacuum.
[2] CO_2 has been connected to Ref 3 port at the time of writing this document (Mon 15 Sep 2025). Ref 1 and Ref 2 are empty – not connected to any gas.
[3] As on Mon 15 Sep 2025, helium output from the wall mounted pressure gauges is only serving carrier gas. Helium for flushing is being served by a separate helium cylinder altogether.

0.2-0.3 bar. This is reflecting helium values *as a carrier gas* (not helium values as a flushing gas – those are shown *only* in the cylinder supplying helium for flushing, nowhere else) so this value is important for analyses (not for flushing).

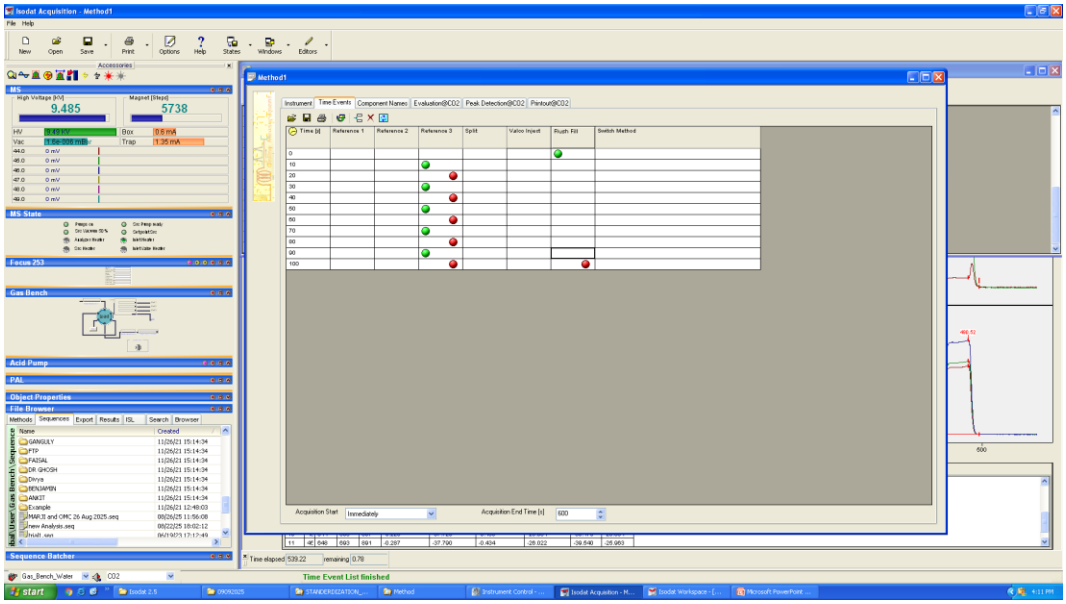


9. Now, take a look at the pressure gauge in the helium cylinder for flushing. This is kept *inside* the room, right besides the Gas Bench. Pressure reading on this cylinder is expected to be zero at the start of the day, as the cylinder is routinely switched off each day following the completion of the daily flushing process. If off (as it should be), switch the cylinder on and bring the pressure to 4 kg/cm^2 (≅ 4 bar).

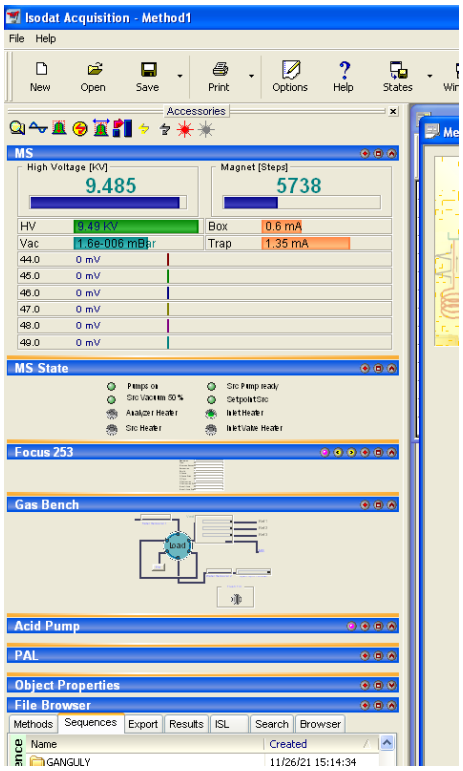
3 Creating a new ‘Method’

3.1 For Flushing

1. Go to the bottom left corner of Isodat Acquisition and select a ‘Gas Bench’ configuration from the dropdown menu if that is not already selected. Typically we have used ‘Gas Bench + Water’ even when analysing carbonates[4].

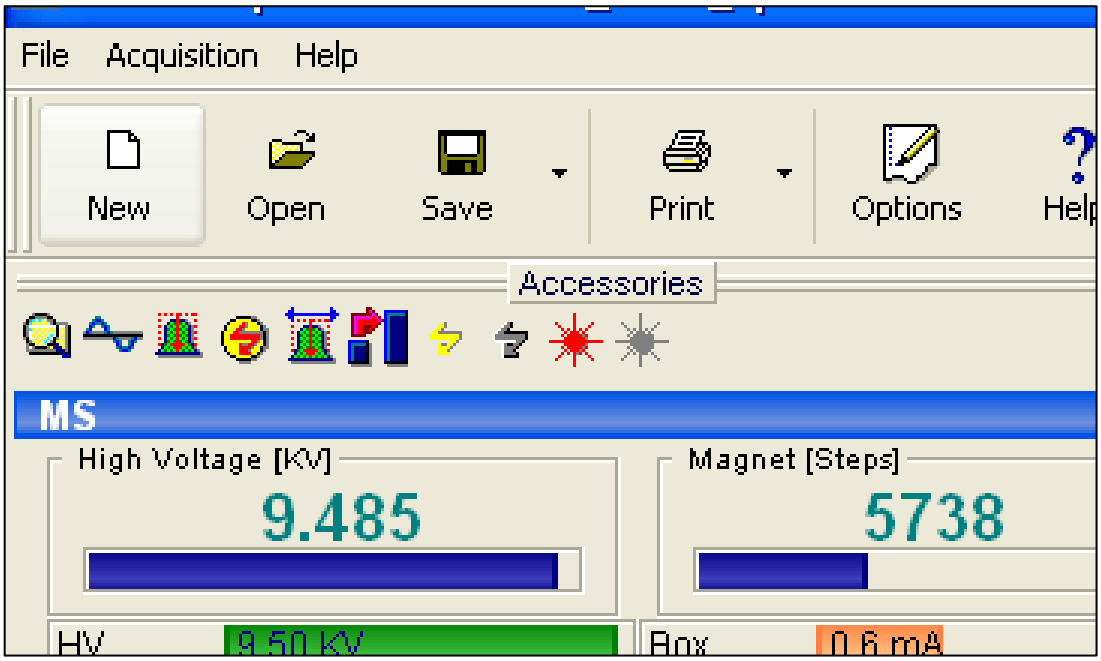


2. Once you see the Gas Bench configuration, your left sidebar should look something like this:

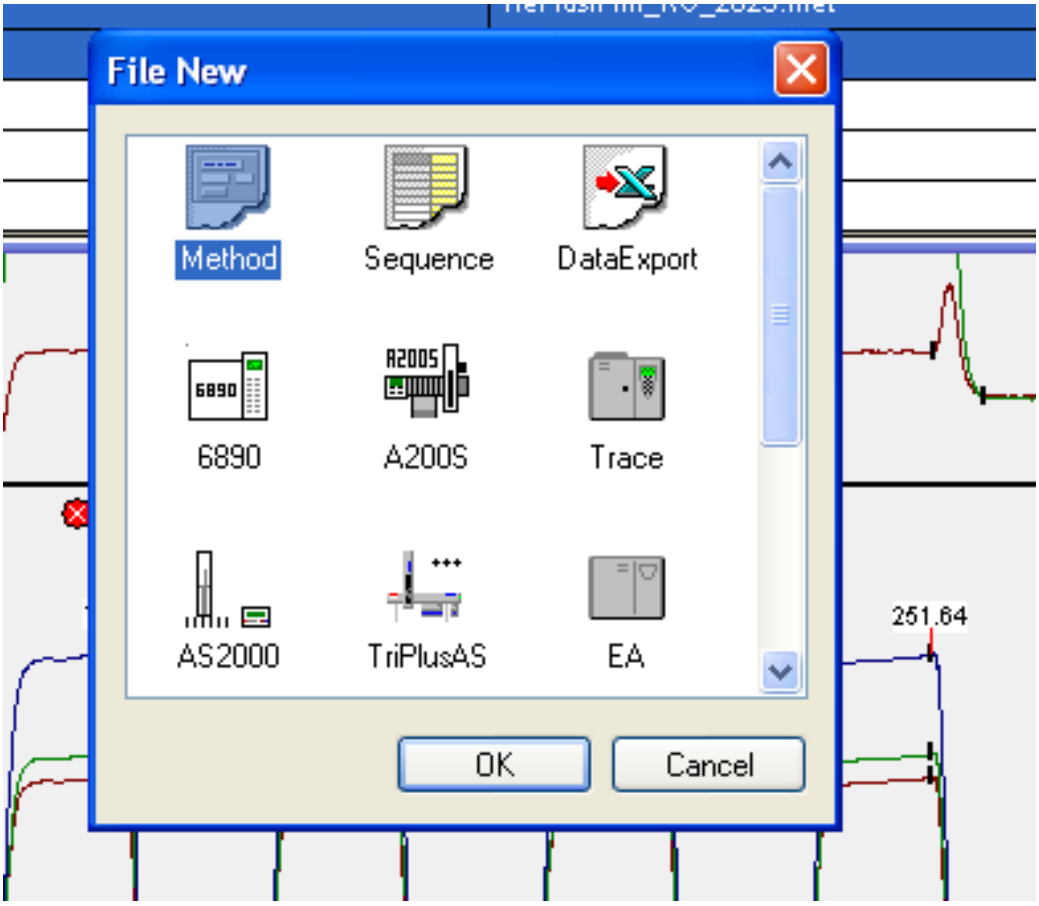


[4] At the time of writing this document, I do not know why that is the case. I have personally never tried ‘Gas Bench + Carbonate’ or ‘Gas Bench,’ and am not sure what would change if I were to do that.

3. To create a new method, in the top header menu/toolbar, click ‘New’.

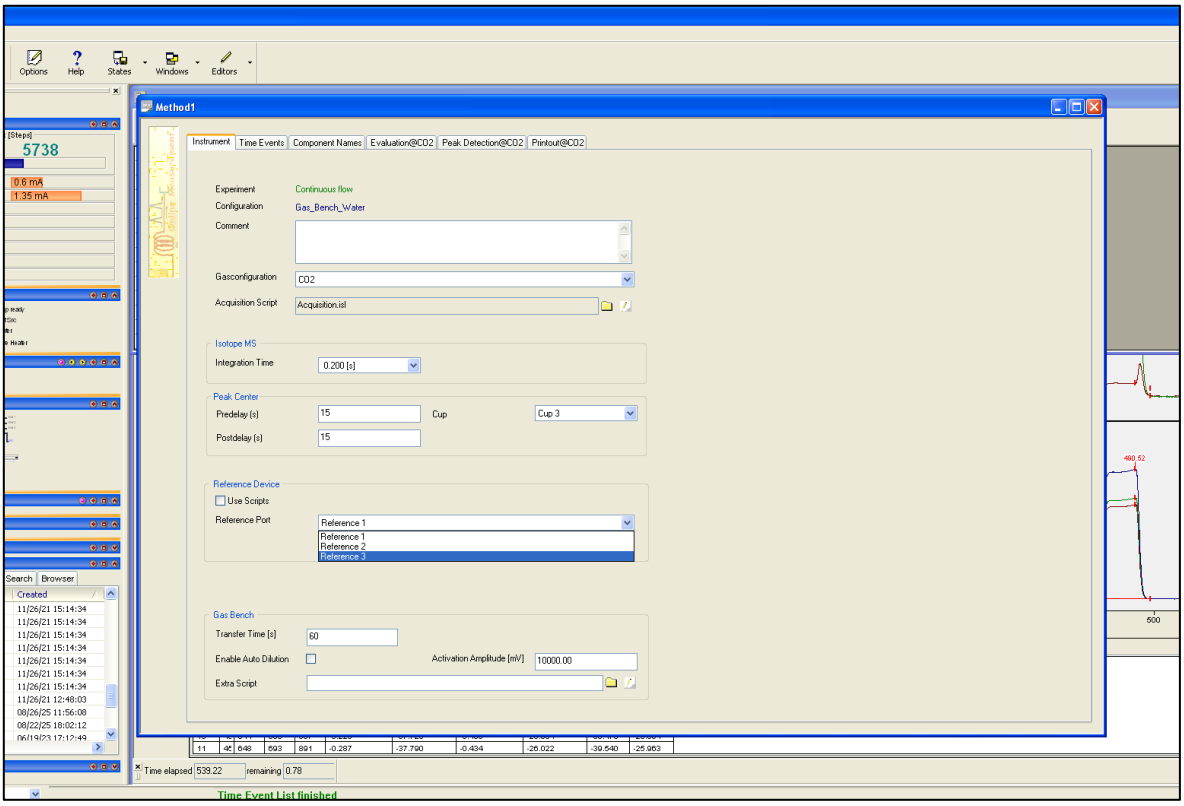


4. You will see a new dialogue box like this. Click on ‘Method.’ Then click ‘OK’.

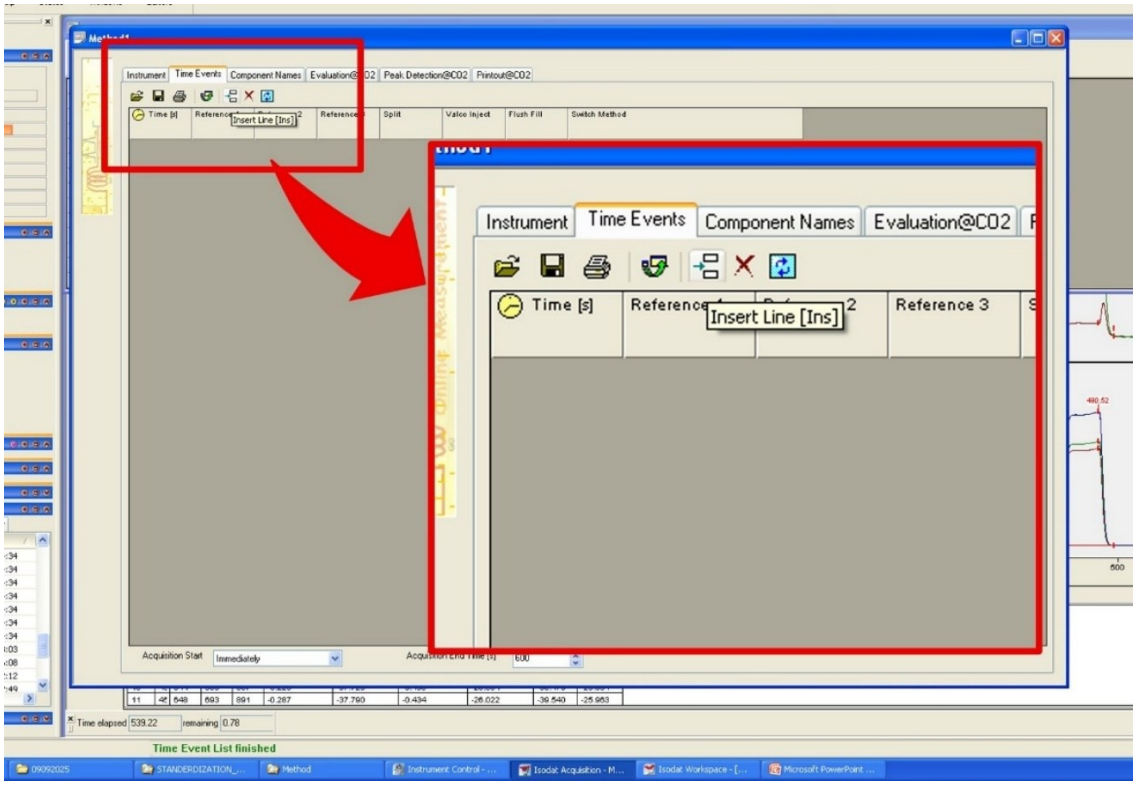


5. This will open a new ‘Method’ dialogue box, titled ‘Method 1’ by default. You will see six tabs: ‘Instrument,’ ‘Time Events,’ ‘Component Names,’ ‘Evaluation @ CO_2,’ ‘Peak Detection @ CO_2,’ ‘Printout @ CO_2’.

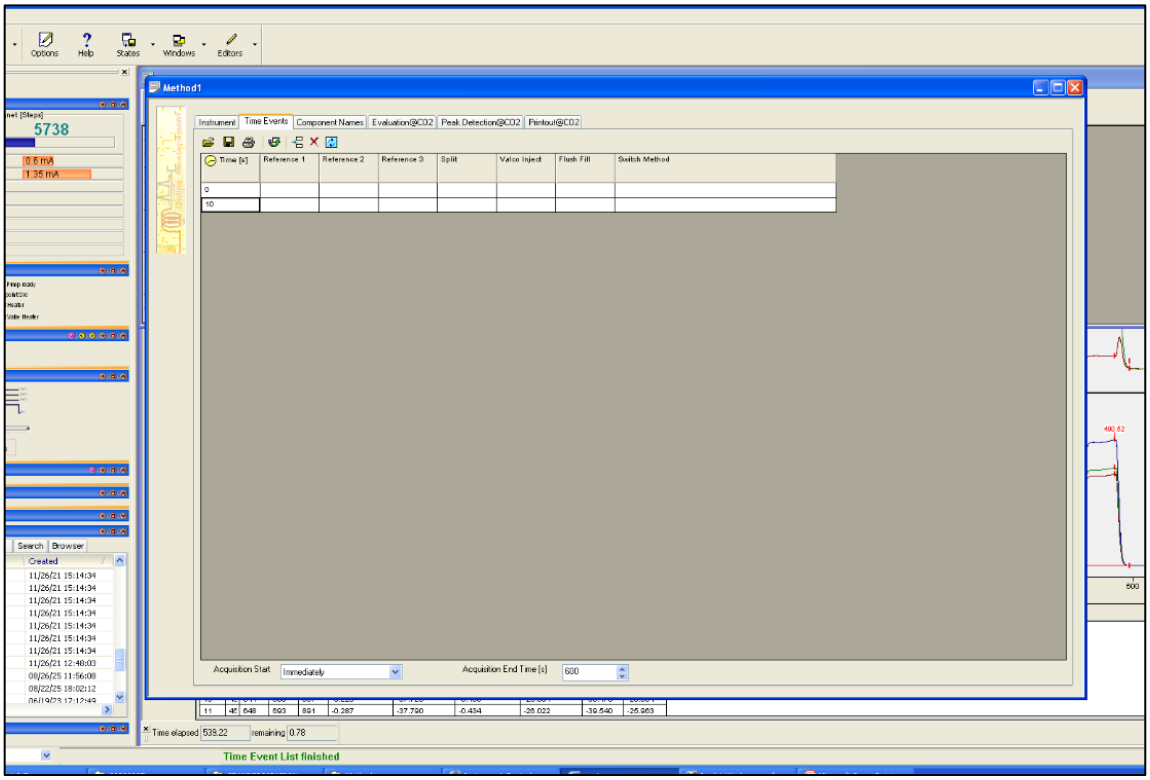
In the ‘Instrument Tab,’ select ‘Reference 3’ in the Reference Device dropdown list. This is because CO_2 is feeding Ref 3.



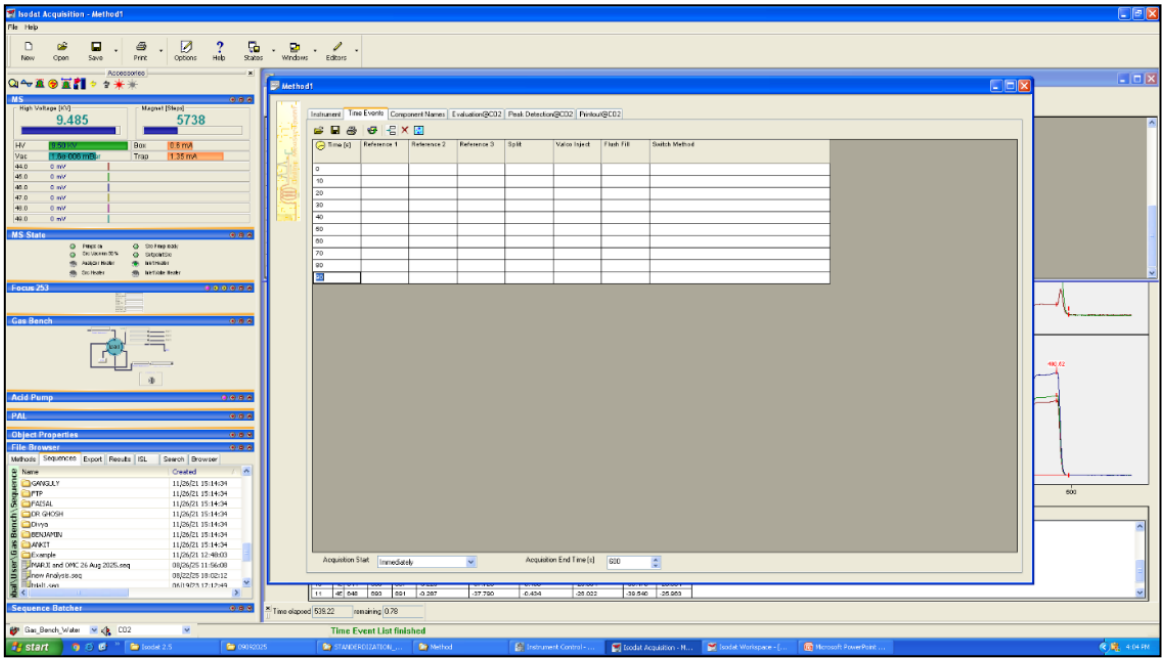
6. Then, go to the ‘Time Events’ tab. The table will initially be empty. Click on the ‘Insert Line’ button or the ‘Insert’ key on the keyboard.



7. Insert **<u>two</u>** lines. The ‘Time (s)’ value in the first row should be 0. In the second row, you can write any number. In this example, I have taken ’10 (seconds)’.



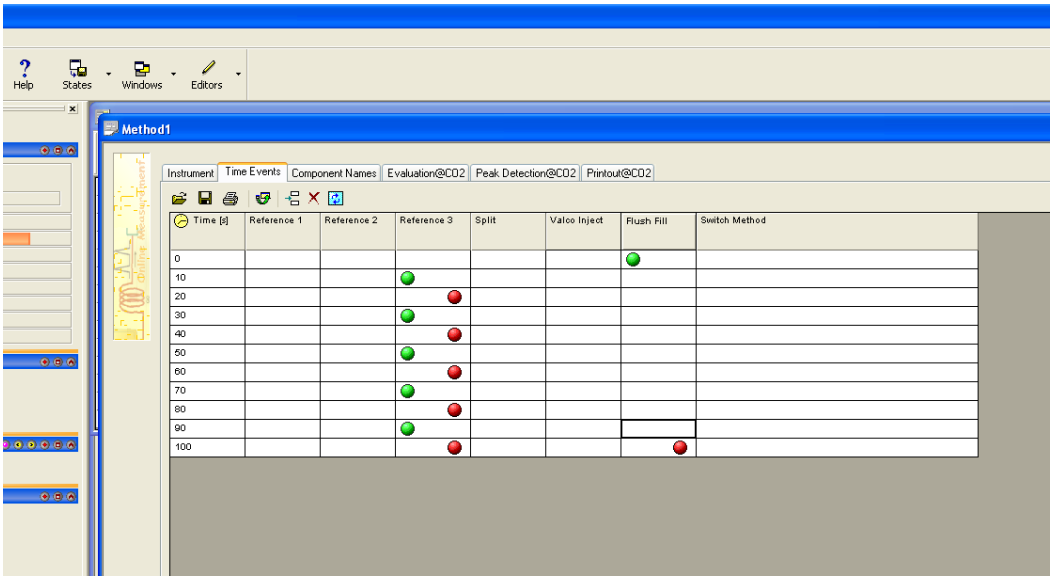
8. Then, keep pressing the ‘Insert Line’ button or the ‘Insert’ key on the keyboard. It will keep adding rws that are automatically populated with time-stamps equivalent to the difference in the first two rows (here, 0-10-20-30… 90). You can keep adding more rows.



9. Now we need to tell the software programme when to switch on/off the reference and when to switch the flushing on/off.

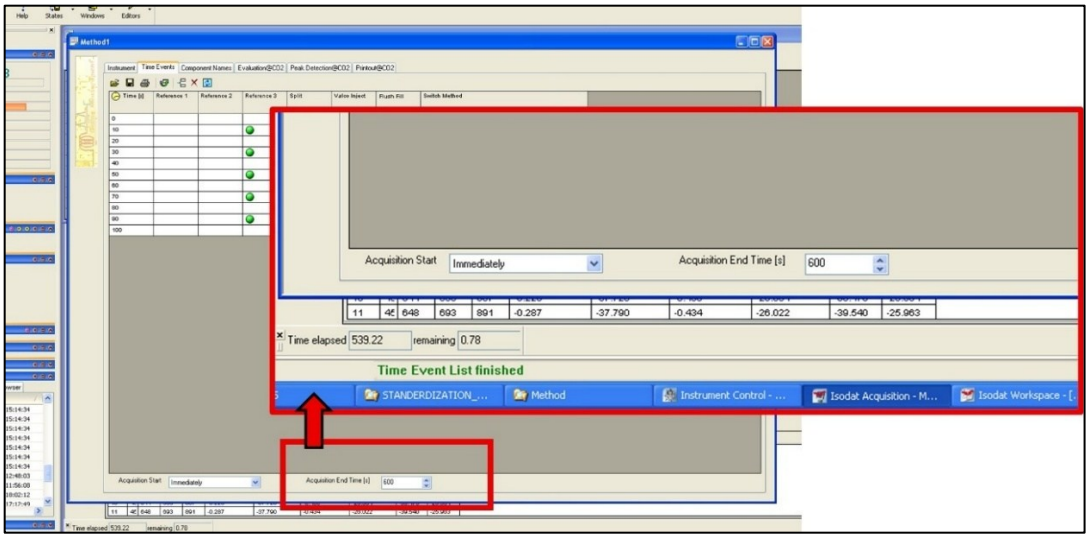
In order to do this, click on a cell in the Reference 3 column at a time stamp at which you want to switch it on. Clicking on it once will turn it green (‘on’) and clicking it twice will turn it red (‘off’). You can also alternate between green and red by clicking the space bar on the keyboard.

Now, do the same for ‘Flush fill,’ since this is a method for flushing. In the end, you will have a table that looks something like this. *Please note that this is just an example for demonstration.*

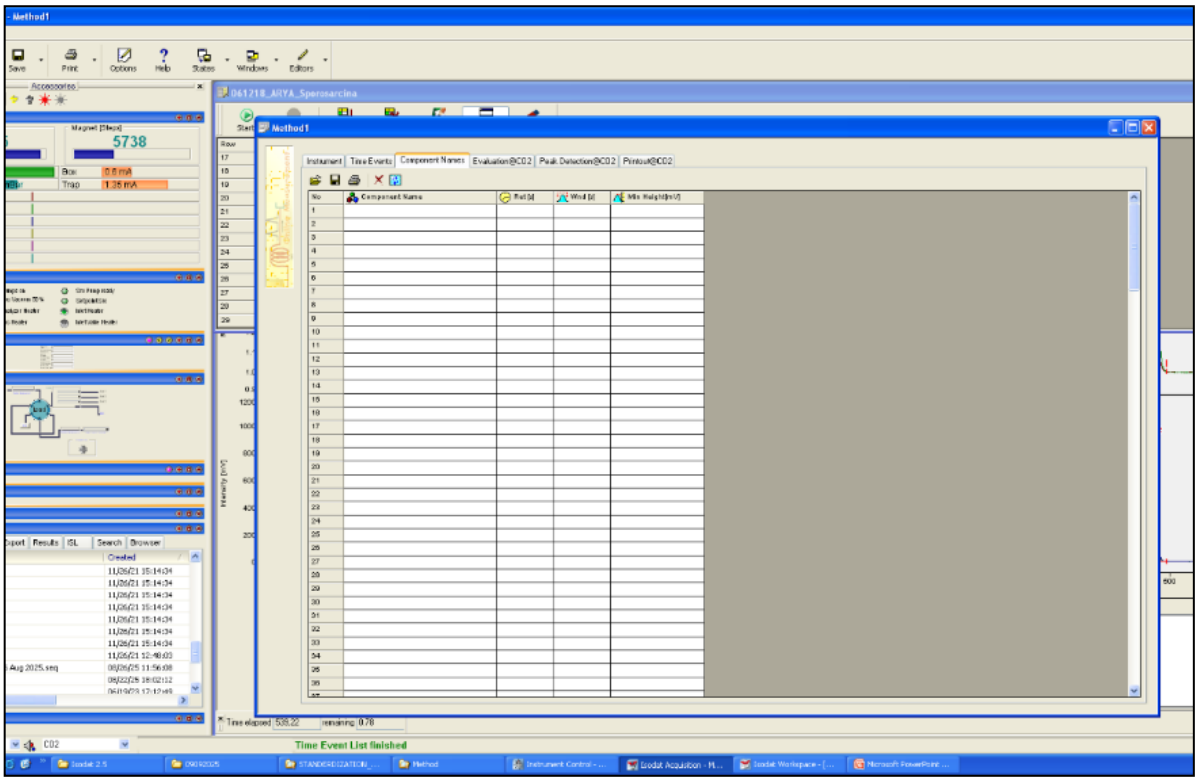


10. Do note that the Reference 3 gas undergoing ‘on or off’ is independent of the helium flushing. Even if the Reference gas was off throughout, flushing would still take place. We use the time for helium flushing to carry out what is commonly known as ‘standard on-off’ in order to find out whether the Gas Bench and Mass Spectrometer are working fine.

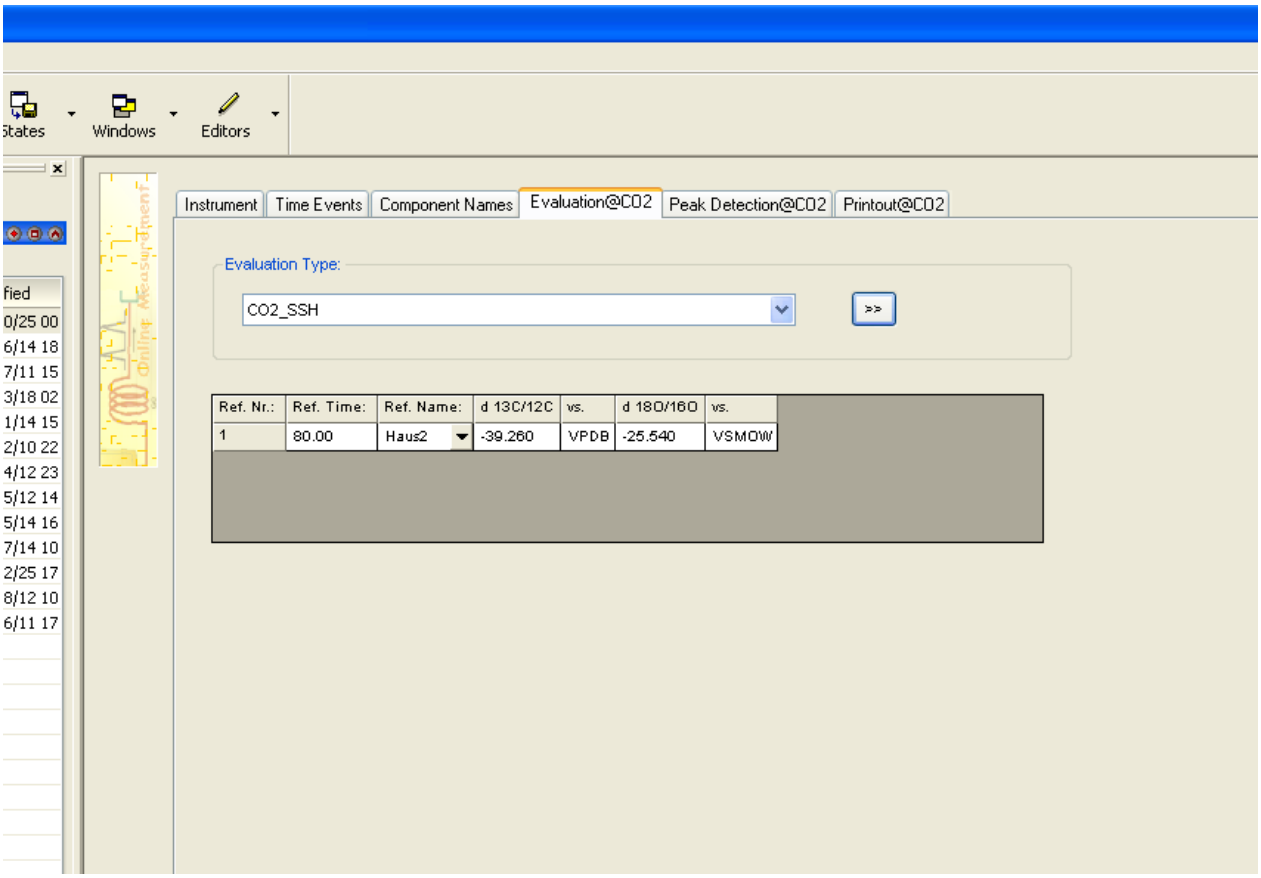
11. Also that the *time stamp in the last row is not the time at which it is going to stop flushing for that particular sample*. The time at which it stops the flushing operation for that particular sample is at the bottom of the ‘Time Events’ tab in the Methods dialogue box. You will see that in this example, the ‘End Time is 600s.



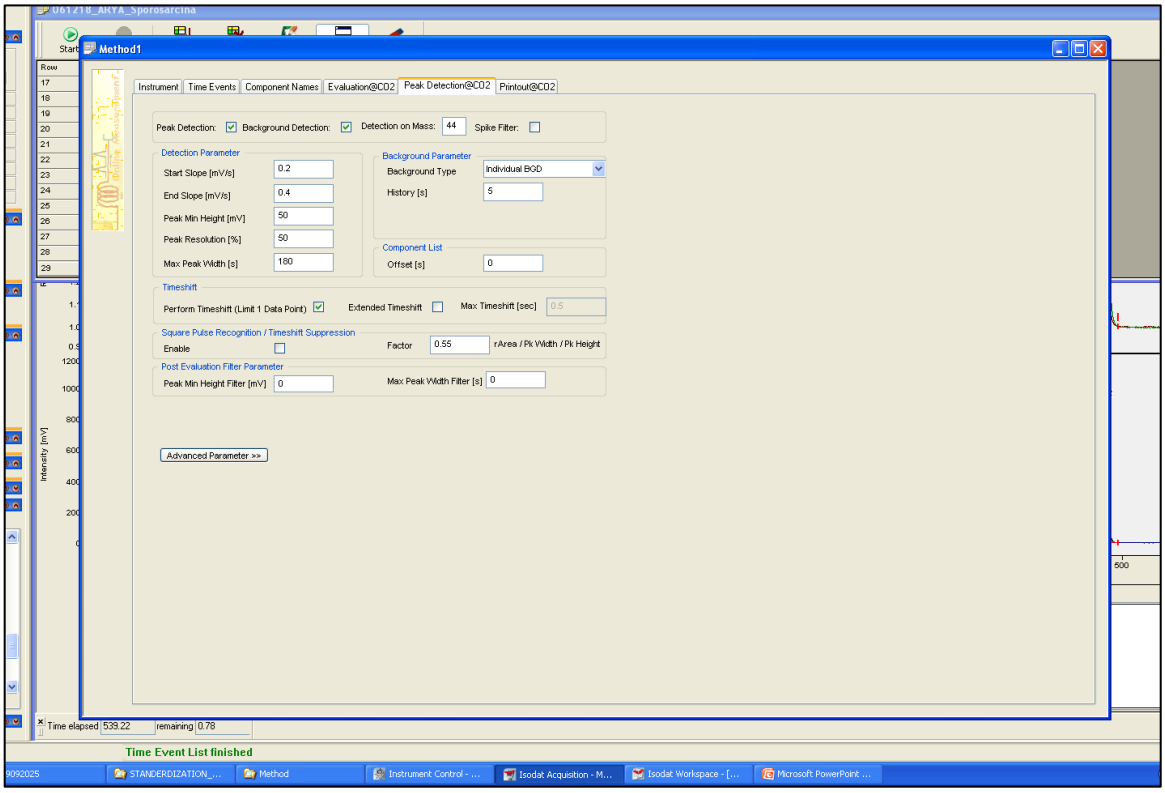
12. Now, head over to ‘Component Names.’ This is empty, and just leave it so.



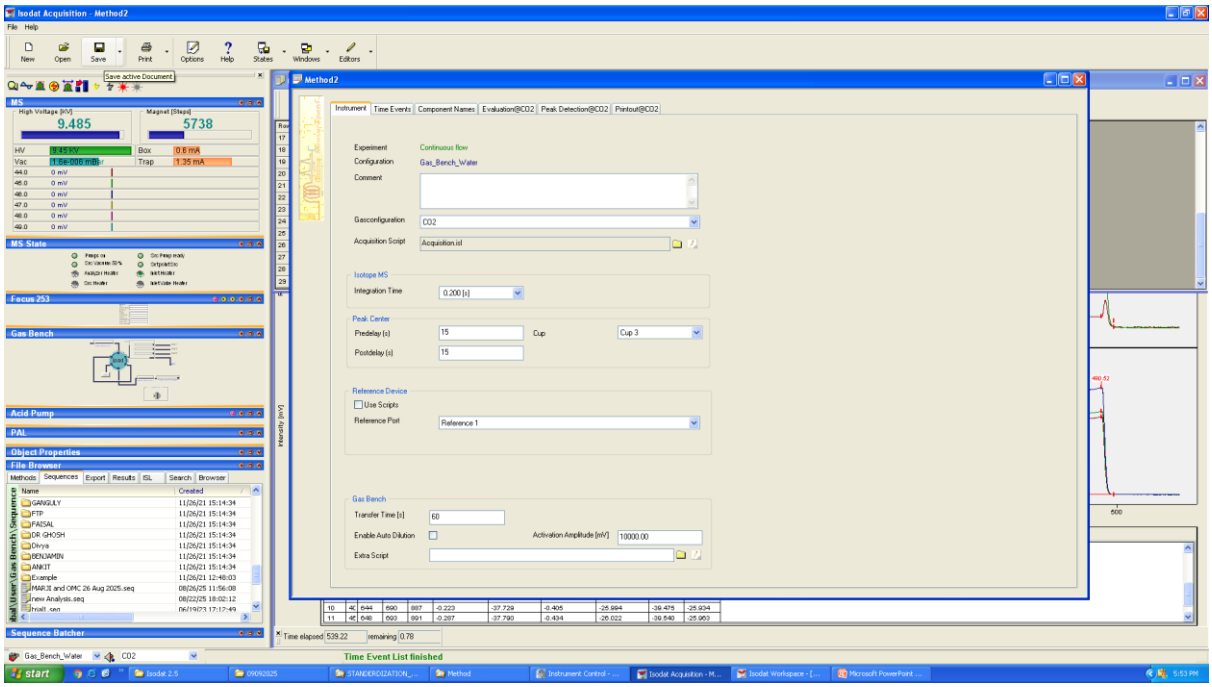
13. Go to ‘Evaluation @ CO_2’ tab. You can select anything in the ‘Ref Name’ dropdown. We have hitherto used ‘Haus 2’.



14. Go to ‘Peak Detection @ CO_2’. The values here are automatically populated. Leave them as such.



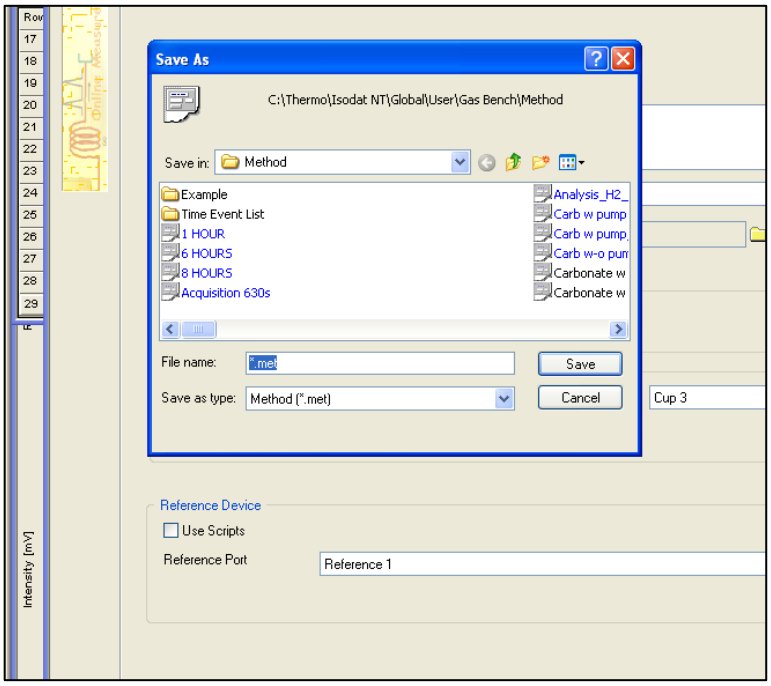
15. Once everything is done, now it’s time to save the ‘Method’ as a ‘.met’ file. For that, click on the ‘Save As’ button in the main header menu[5].



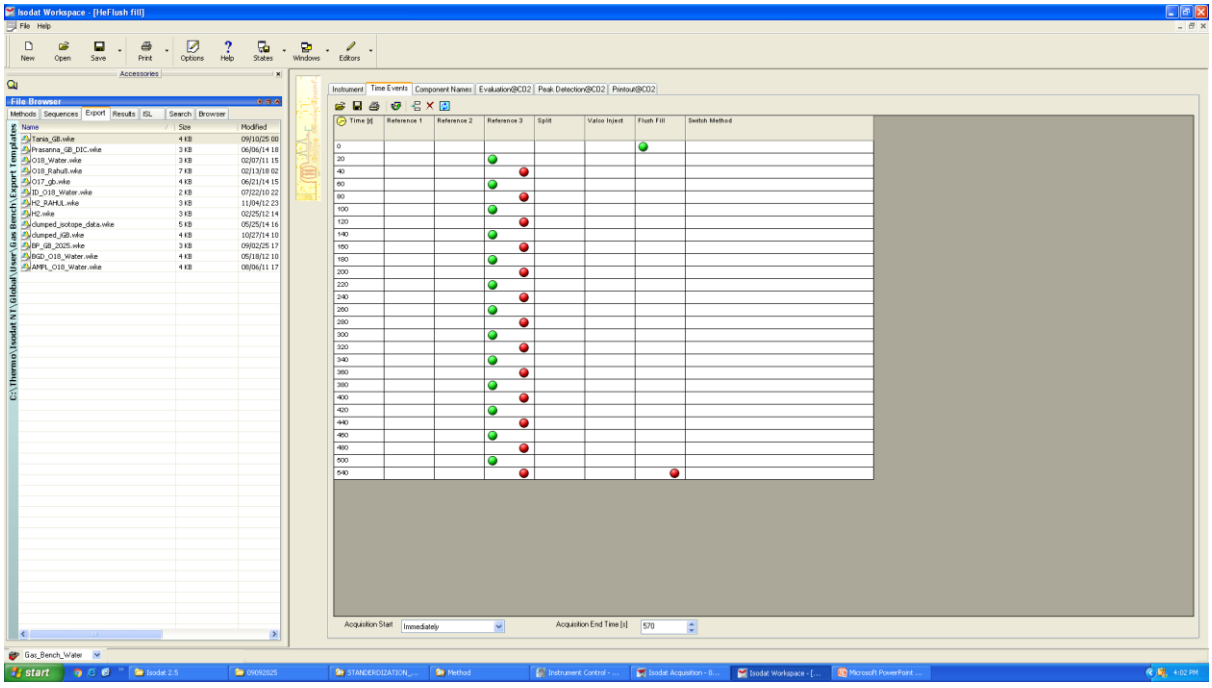
16. This will open a dialogue box. You can save your new flushing method by any name. In

[5] Do *not* select the save button in the ‘Time Events’ tab within the Method dialogue box. It will just save it as a time series, and not as a ‘Method.’

our Win XP PC, all '.met' files are saved in **`C:\Thermo\Isodat NT\Global\User\Gas Bench\Method`** by default It is advised that you save your new method here. (Tip: The 'File Name' has **`*.met`** by default. You may remove this as the file inherently carries the file extension while being saved.)



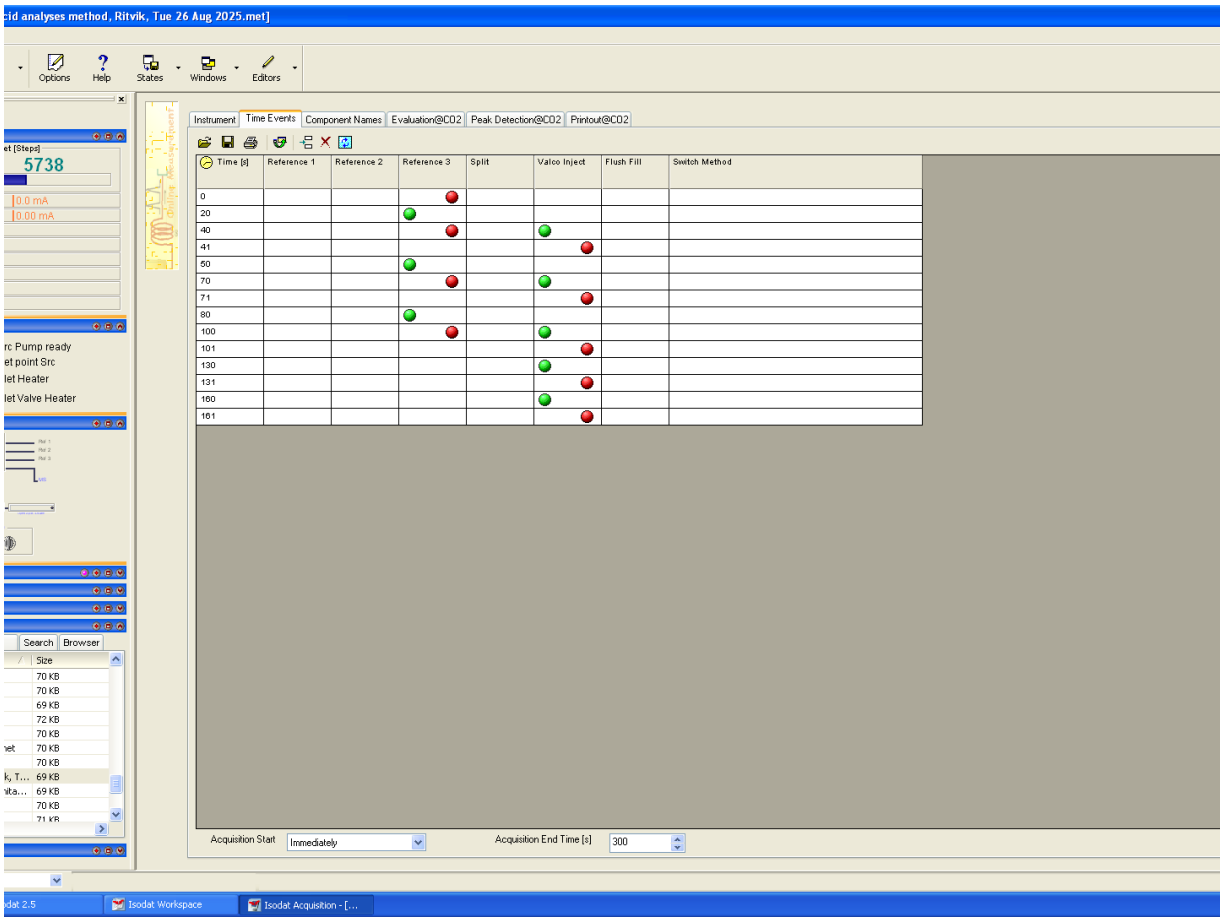
17. For the time being, however, a flushing method has already been created in the same folder by the name **`HeFlush Fill.met`** and the same ought to be used for your carbonate flushing. The 'Time Events' in this **`.met`** file looks like this.



3.2 For Analyses

1. The steps to create a Method for ‘Analyses’ is the same as that of ‘Flushing’. The only difference is the ‘Time Events’ tab. Here, instead of switching the ‘Flush Fill’ on and off, we shall switch the ‘Valco Inject’ option on and off. Everything else is the same.

2. At the time of writing this document (Mon 15 Sep 2025), **`C:\Thermo\Isodat NT\Global\User\Gas Bench\Method`** contains a file just for that: ‘Carbonate w acid analyses method, Ritvik, Tue 26 Aug 2025.met’. Its time event list looks like this:



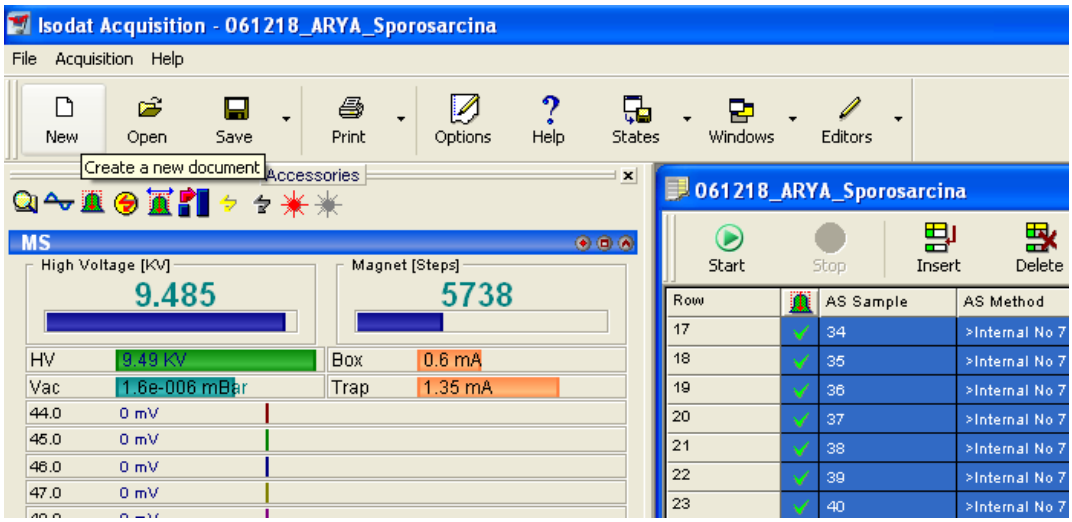
4 Creating a new ‘Sequence’

It’s easy to get confused between ‘Method’ and ‘Sequence’. A ‘Method’ in this software programme is just the series of steps that the machine carries out on each sample. A ‘sequence’, on the other hand, specifies which ‘Method’ to apply in a list – and the same ‘Method’ will be carried out for each entry in the sequence list.

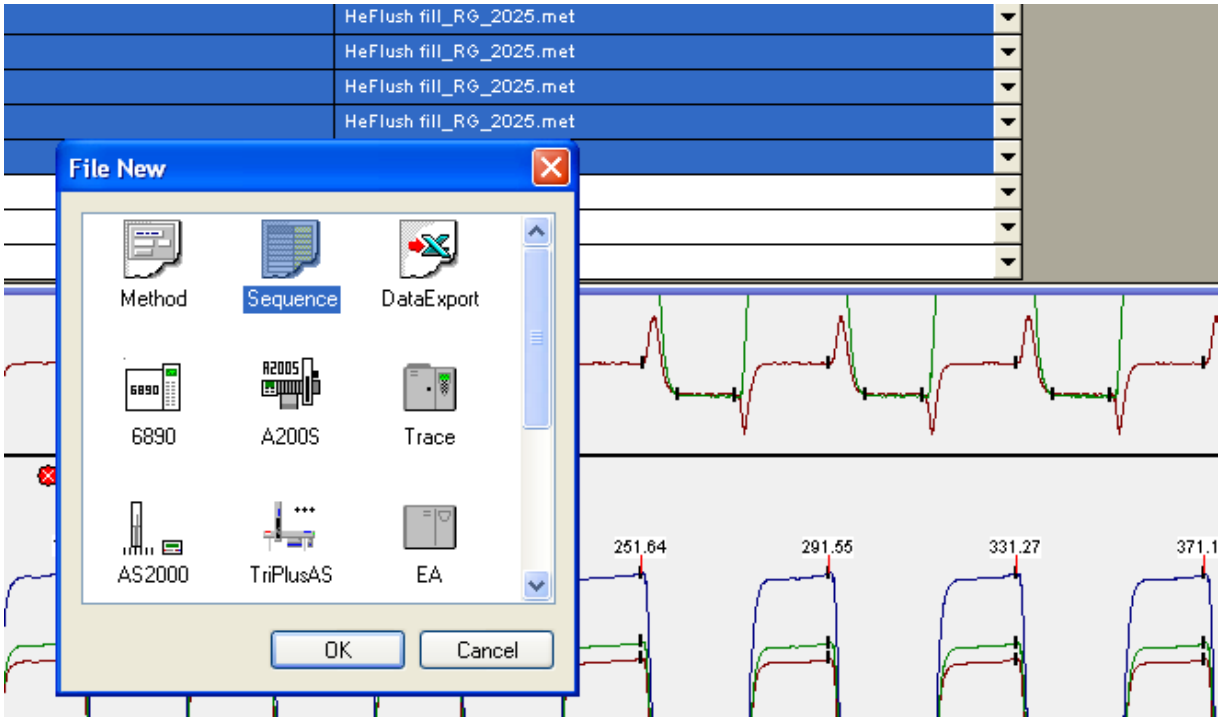
Here are the steps for the same.

4.1 For Flushing

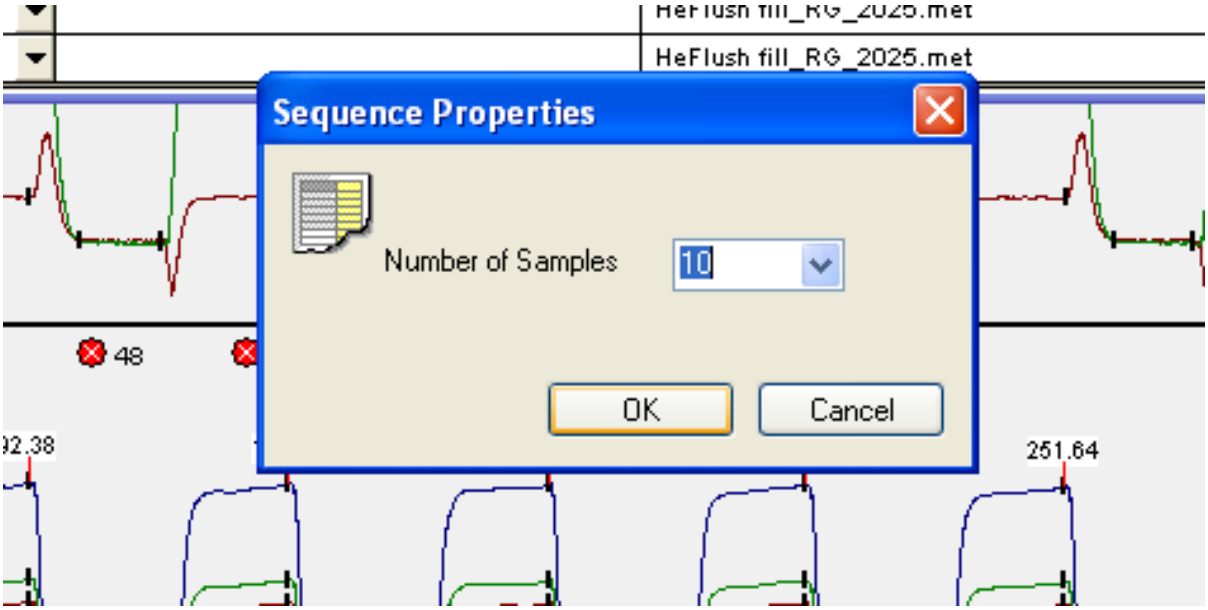
1. Having created Methods, we again start at the main header menu of Isodat Acquisition. Click ‘New’.



2. This time, click ‘Sequence’.

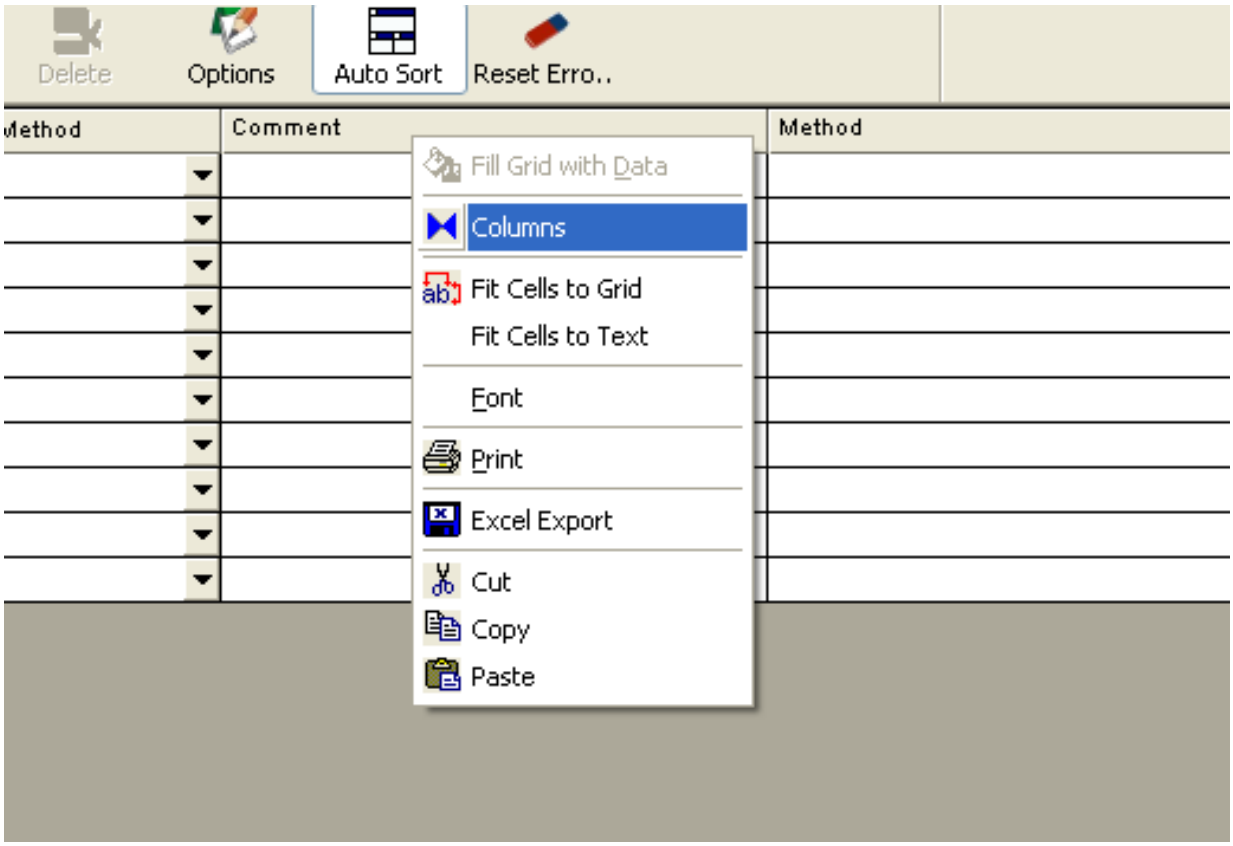


3. By default, the 'Sequence Properties' dialogue box will take ten rows. That's nothing to worry about, for you can add/delete as many rows as you wish.

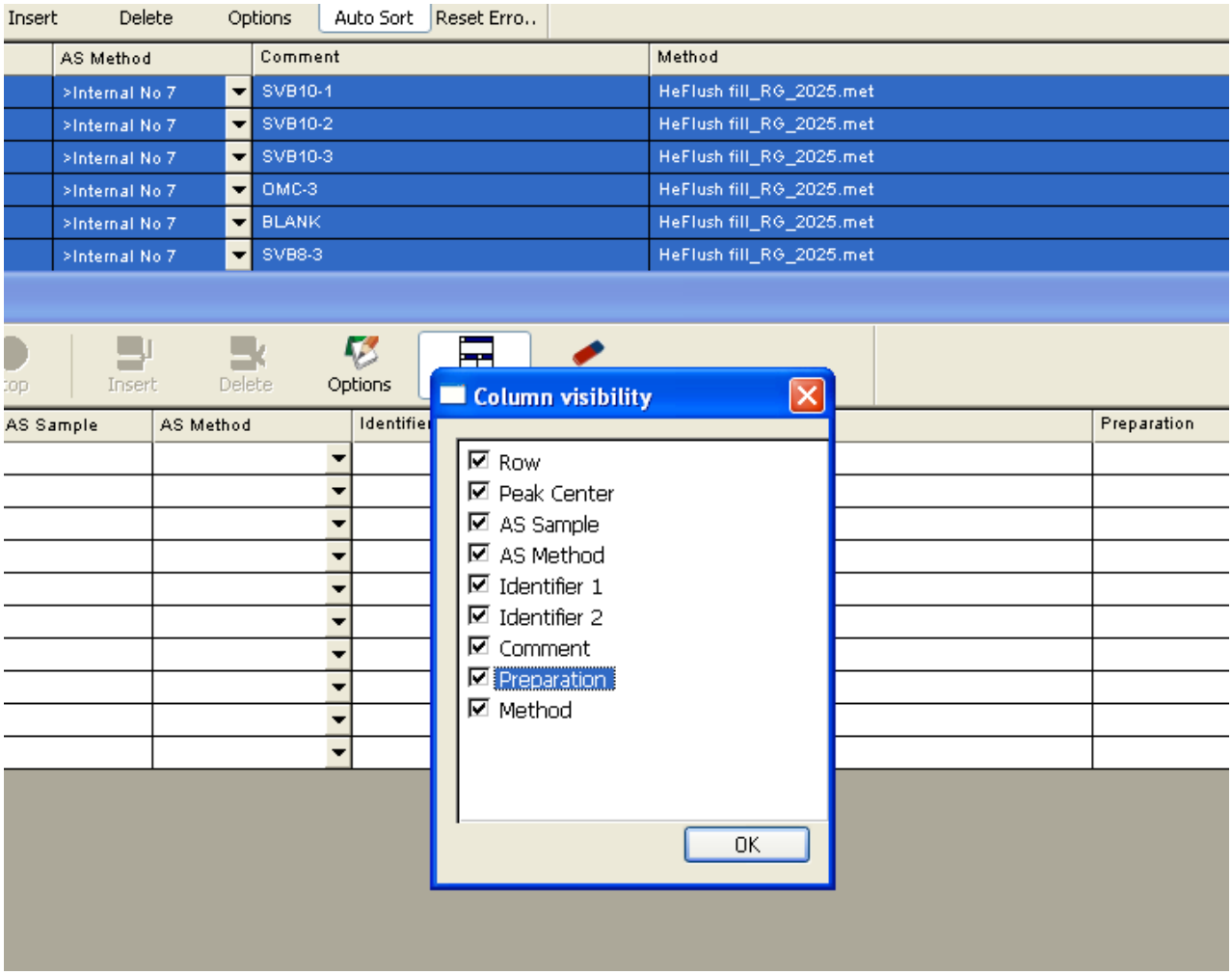


4. The new Sequence dialogue box is titled 'Sequence 1' by default. You will see the following columns: 'Peak Centring,' 'AS Sample,' 'AS Method,' 'Comment,' and 'Method.'

5. While making Sequences, it is a good idea to try to have as much information about the sample as possible. This is super helpful in retrieving information later on, in case any mistakes occur. Therefore, right click on any column header, and select 'Columns.'



6. Then, I tend to select pretty much all the options available to me.



7. Now, to fill out the columns: The essential columns, without which the Sequence simply won't run, are: 'AS[6] Sample,' 'AS Method' and 'Method.' It is optional, but advisable to fill out other columns , such as Identifier 1/2 etc. for your own records.

8. The 'AS Sample' column corresponds to the slots in the autosampler tray. In other words, the 'AS Sample' number directly dictates which needle goes where during a particular Sequence. Therefore, I will digress a bit to discuss needle positioning in the autosampler tray (Figure 1, P2-15, Finnigan Gas Bench II Operating Manual).

   In order to have the correct numbers in the 'AS Sample' column, we need to know the needle configuration in the autosampler arm. In our lab, the analyses needle is on the left and the flushing needle is on the right. Do note that this could be the other way around in a Gas Bench elsewhere.

   It is important to note that the autosampler itself does not know which needle has been placed on which side. Therefore, whichever slot we specify in 'AS Sample' (let's say, '9') it will by default lower the needle on the left (in our case, the analyses needle) in that slot.

[6] 'AS' here stands for 'Autosampler.'

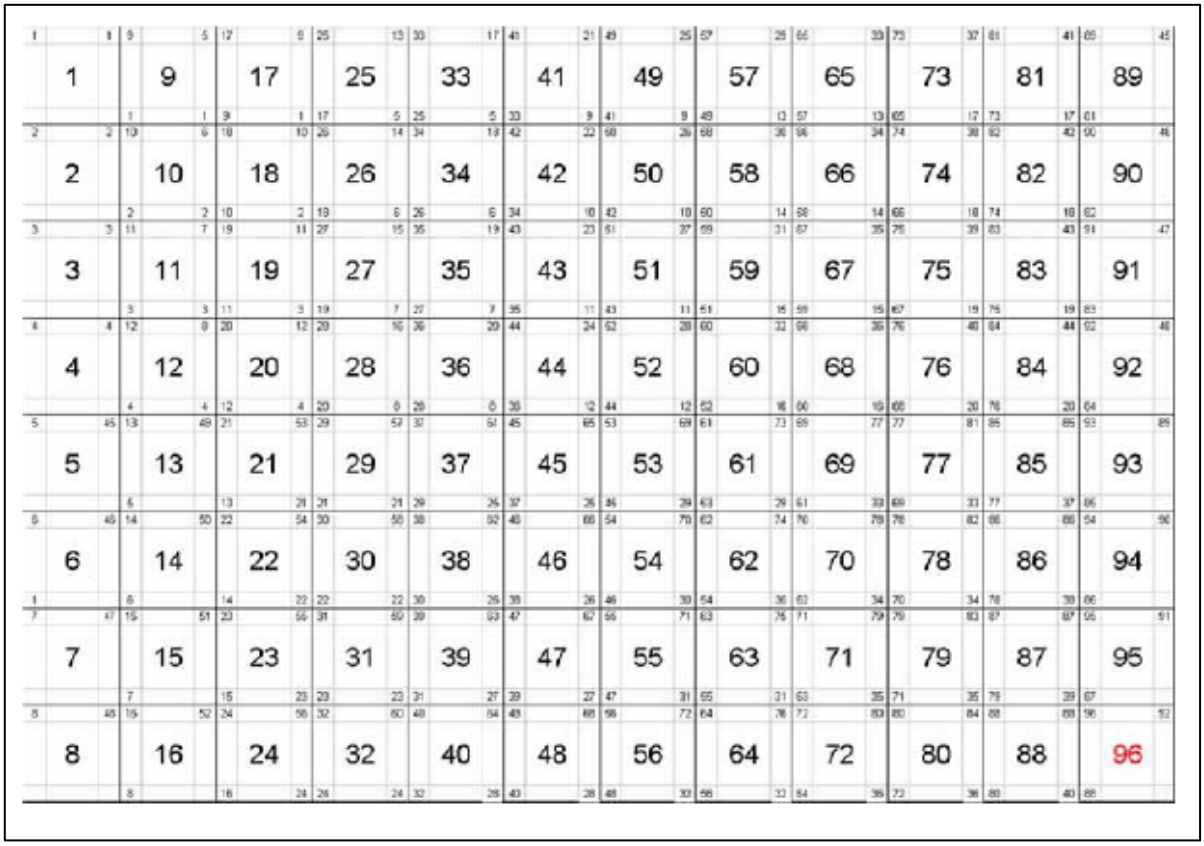


Figure 1 Sample numbering in AS tray

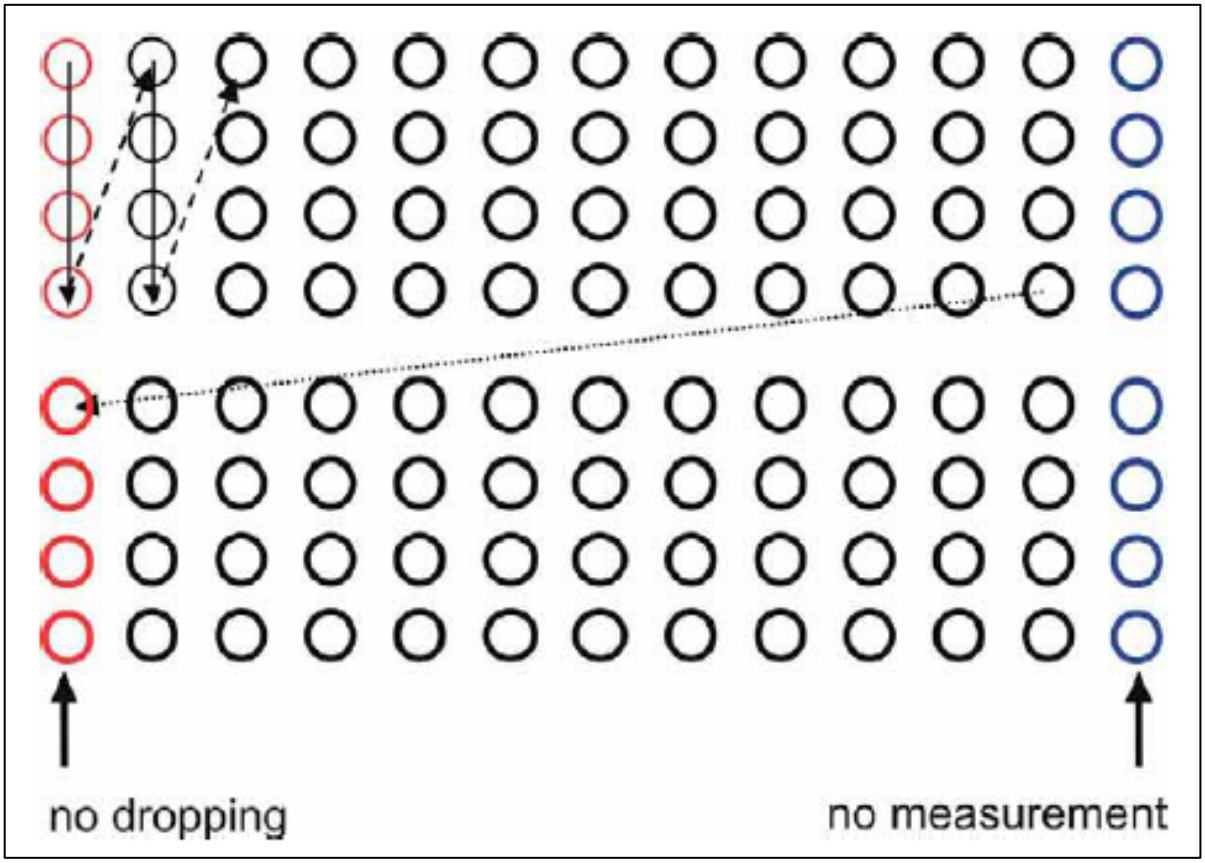
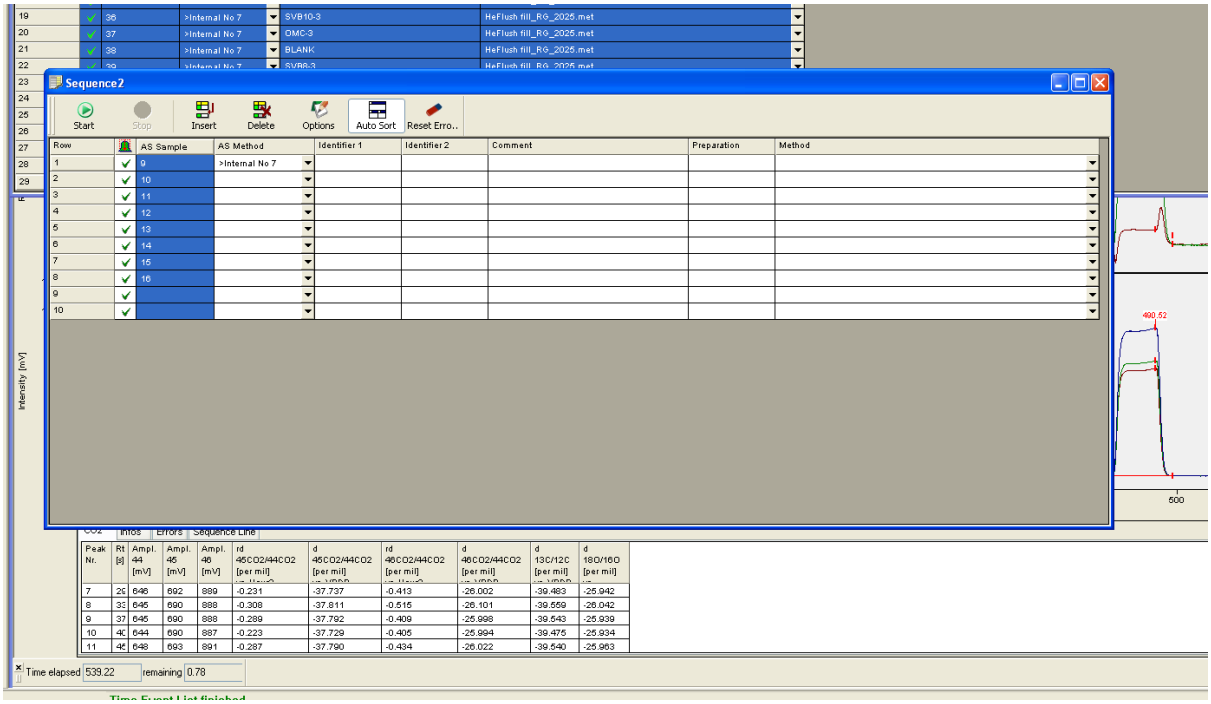


Figure 2 Sampling protocol in AS tray

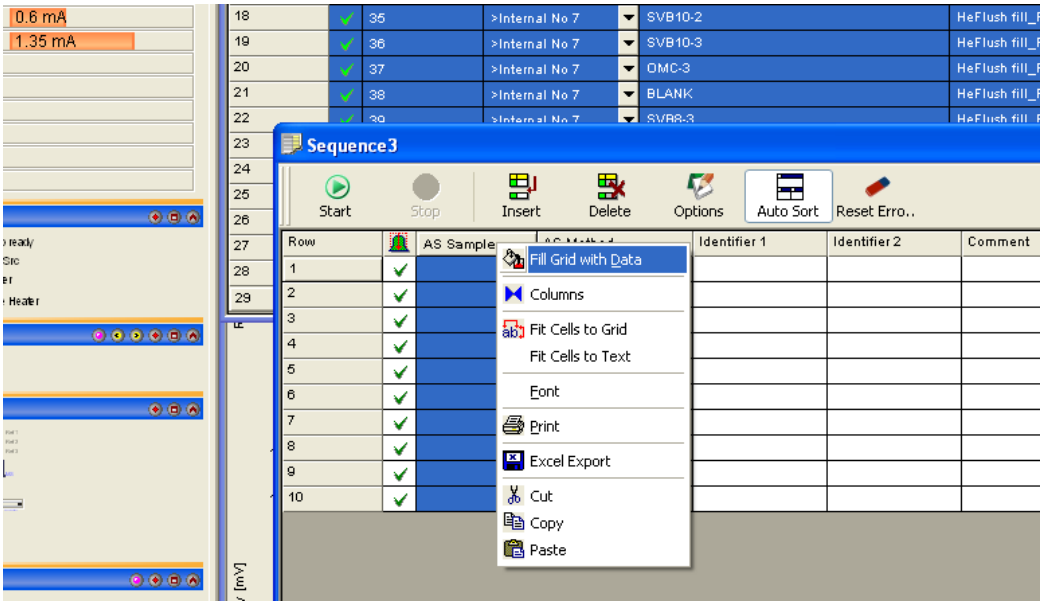
Since the tray has only 8 slots in each column, a slot immediately to the right of any slot has a position of +8. So, a slot immediately to the right of #9 will be #17. If we would like the flush needle (i.e. the one on the right) to be lowered, into slot #9, the 'AS Sample' value should be '#1'.

This is the reason that odd numbered columns are typically left completely blank, so that either needle can have space to lower and not be damaged. This is advised in the official Gas Bench manual as well (Figure 2, P2-7, Finnigan Gas Bench II Operating Manual).

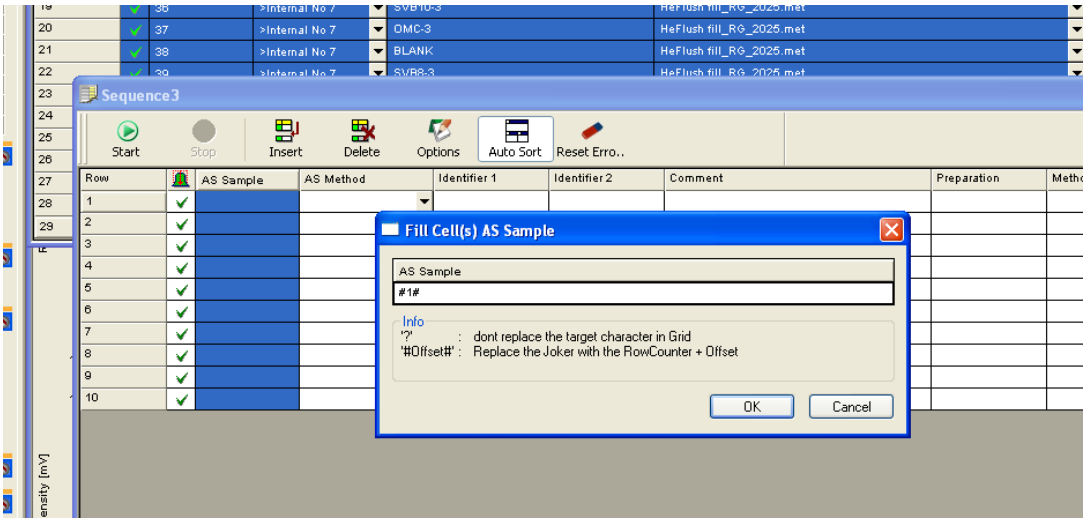
9. Therefore, we fill rows in 'AS Sample' column from 9 through 16. Either we can do it manually…



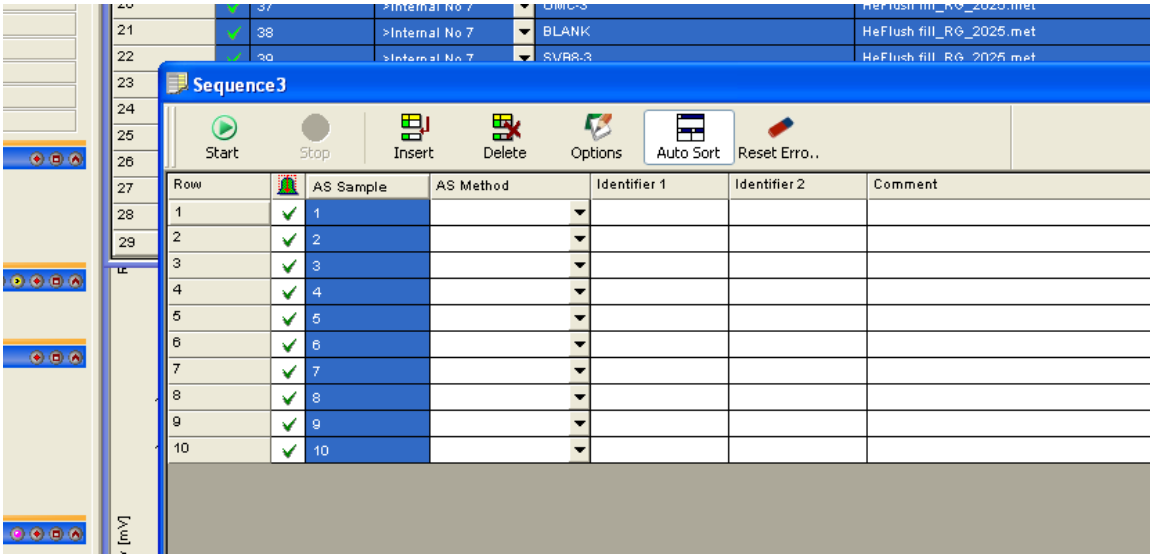
10. …or a convenient way to do it is to click on the header of any column, right click and select 'Fill Grid with Data.'



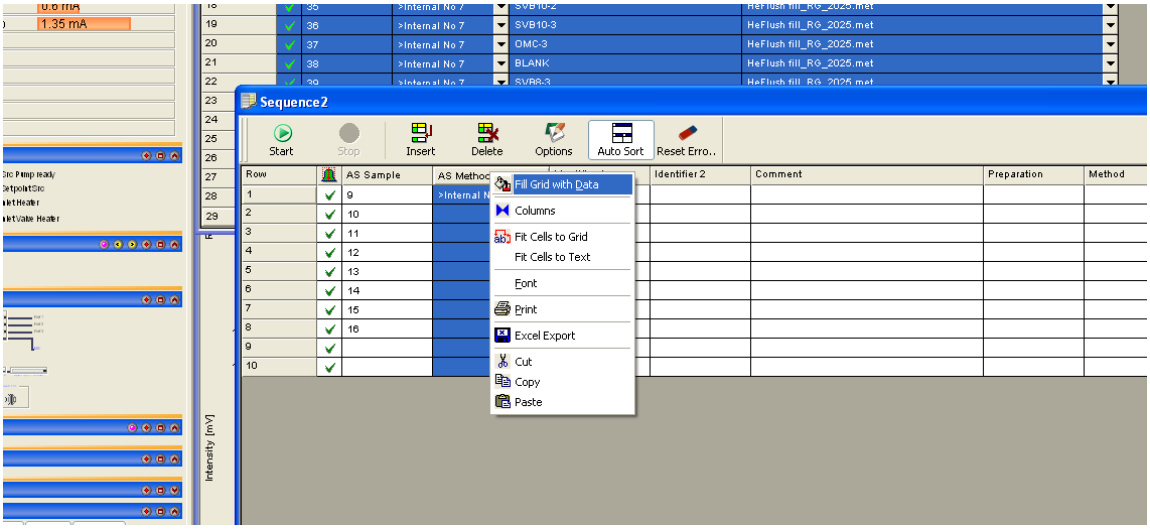
11. Write #1# (you can write any number instead of 1 in the software).



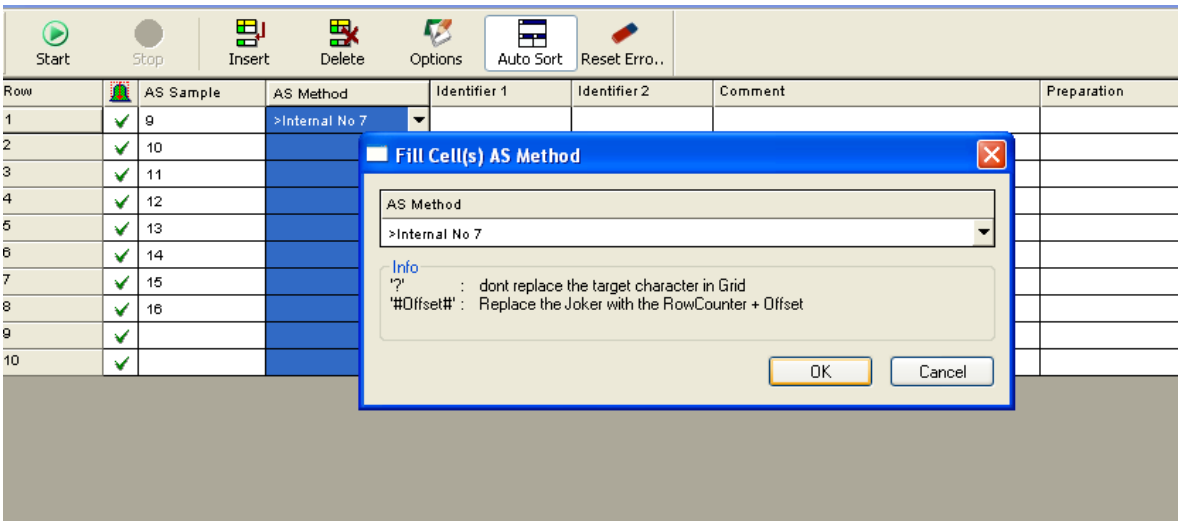
12. It automatically fills up all the subsequent rows with consecutive numbers (here, 1 to 10).



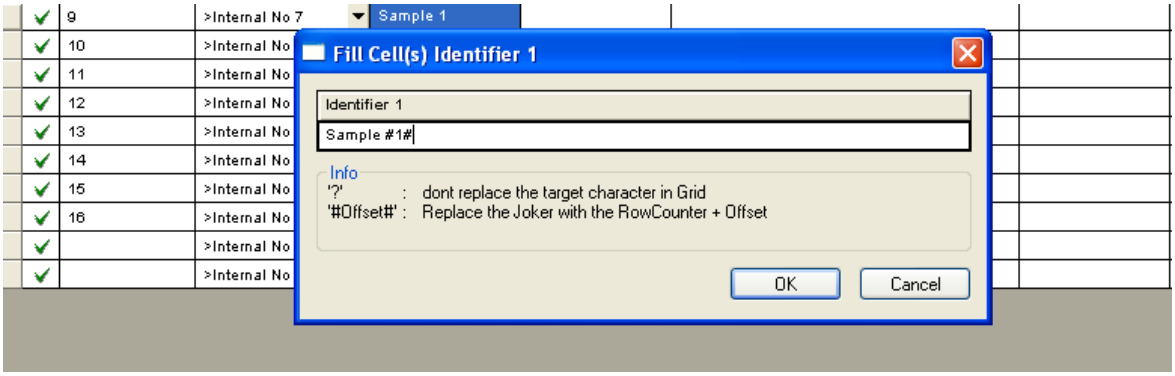
13. The next column is 'AS Method'. Again, click on the header of the row, select 'Fill Grid with Data.'



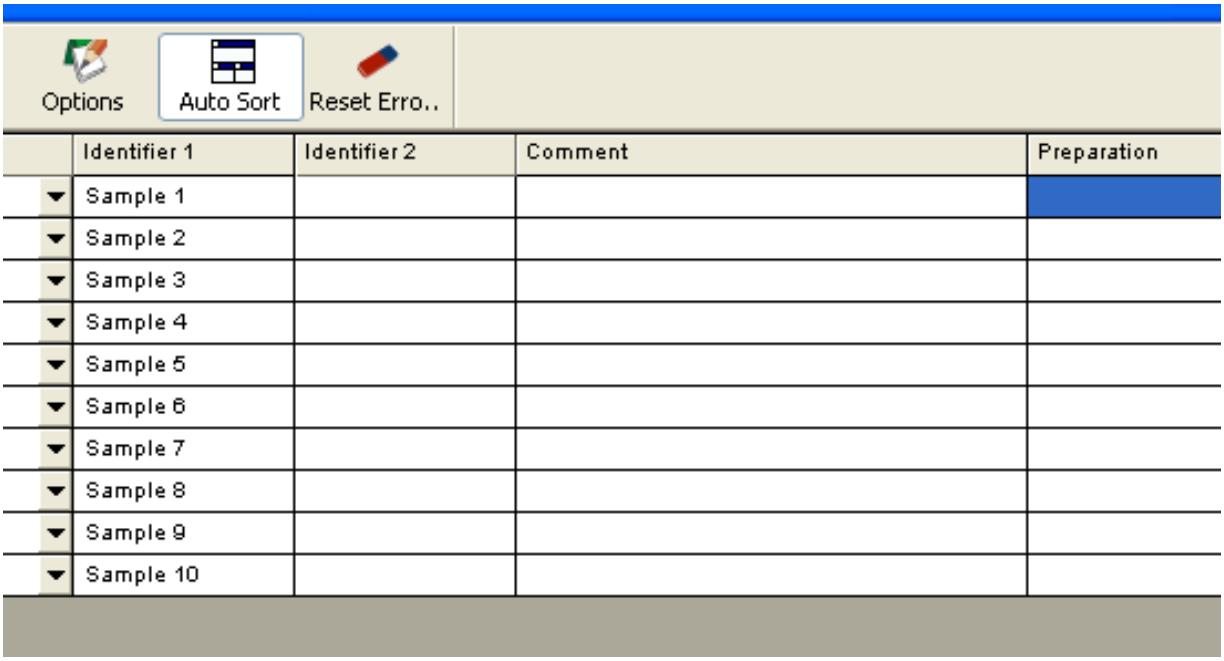
14. Then, select '**Internal No. 7**' from the dropdown menu. Remember that for flushing, 'Internal Method' is always No. 7.



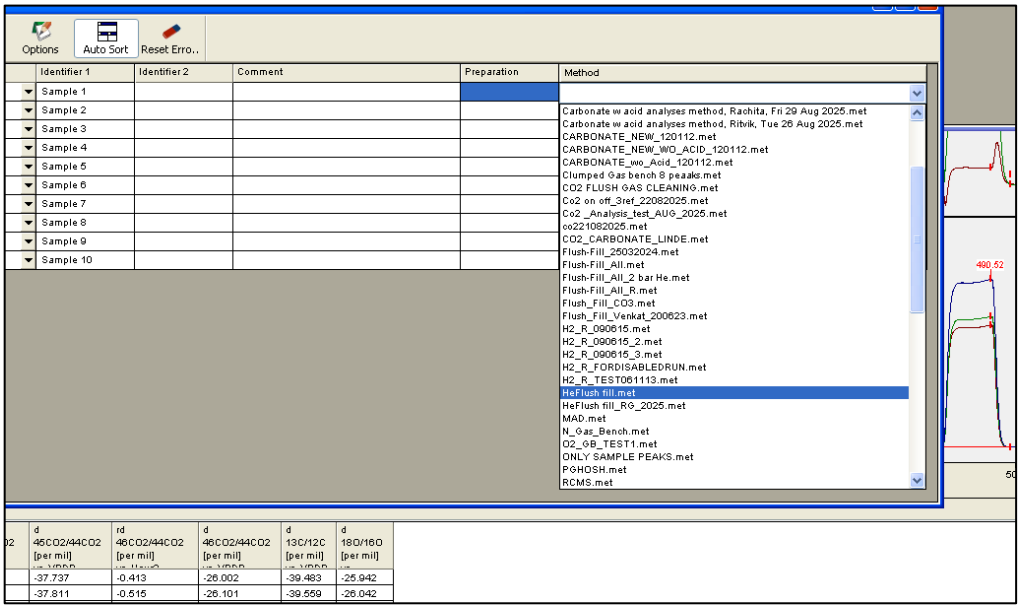
15. Next, we fill up the 'Sample Identifier' column. Just like the previous steps, we right click the header of the column, select 'Fill Grid with Data' and fill up the details. Here, just for demonstration, I am writing 'Sample #1#'.



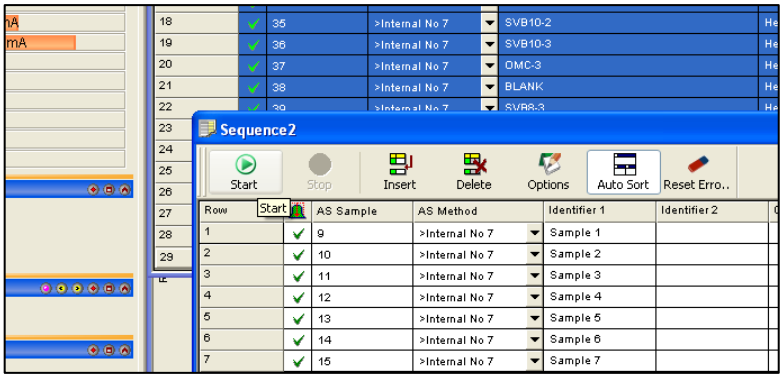
16. This fills up the cells from 'Sample 1' to 'Sample 10'.



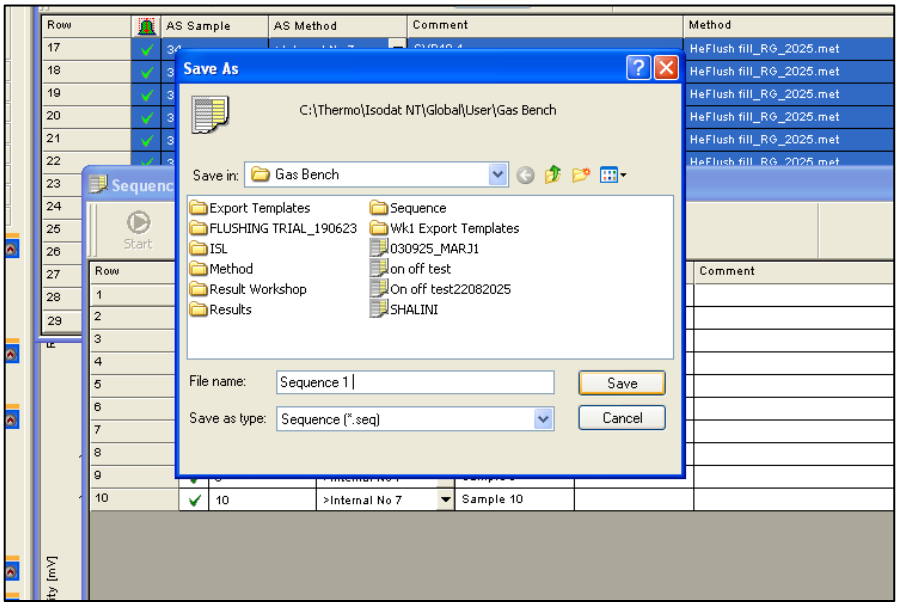
17. For Method, select ‘HeFlushFill.met’. You may select some other flush fill method as well, as per your preference. If you are first time user, stick to a pre-made method i.e. ‘HeFlushFill.met’.



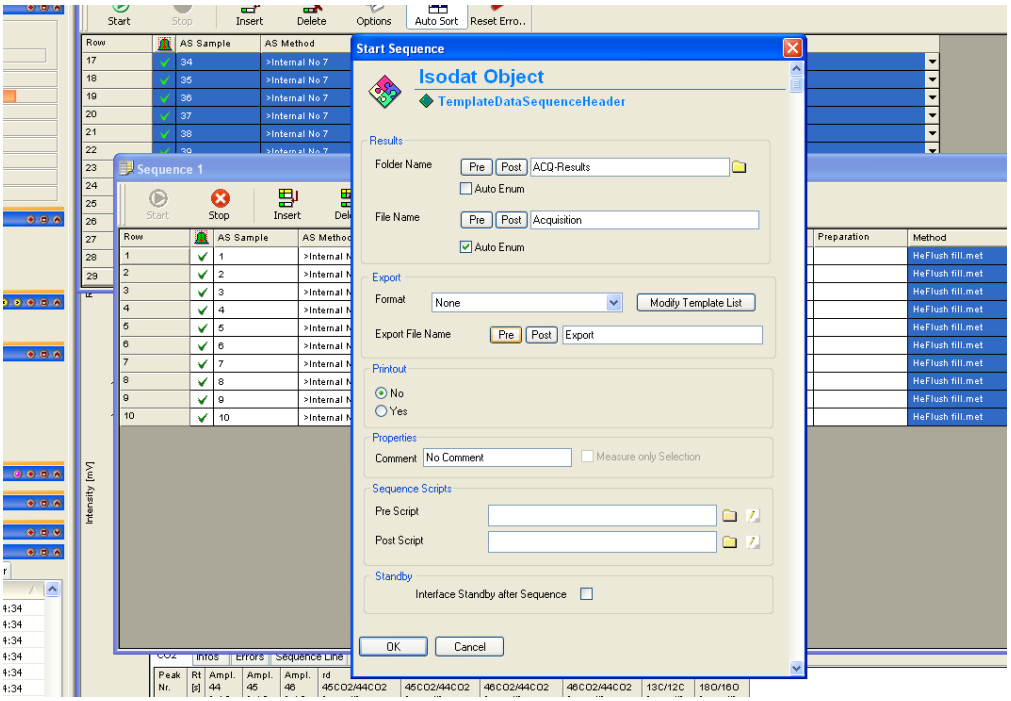
18. Then, click on the ‘Start’ button at the top of the dialogue box.



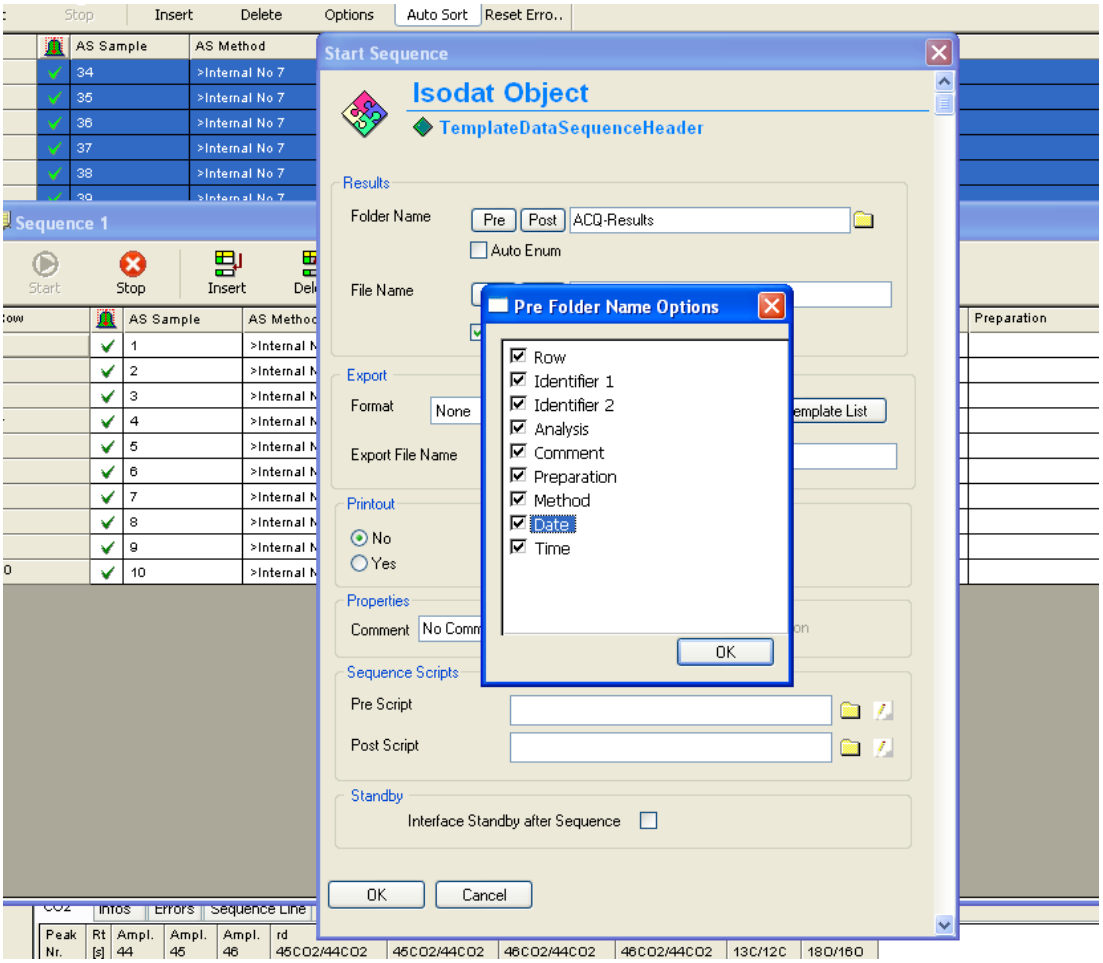
19. This will first prompt you to save the Sequence as a .seq file.



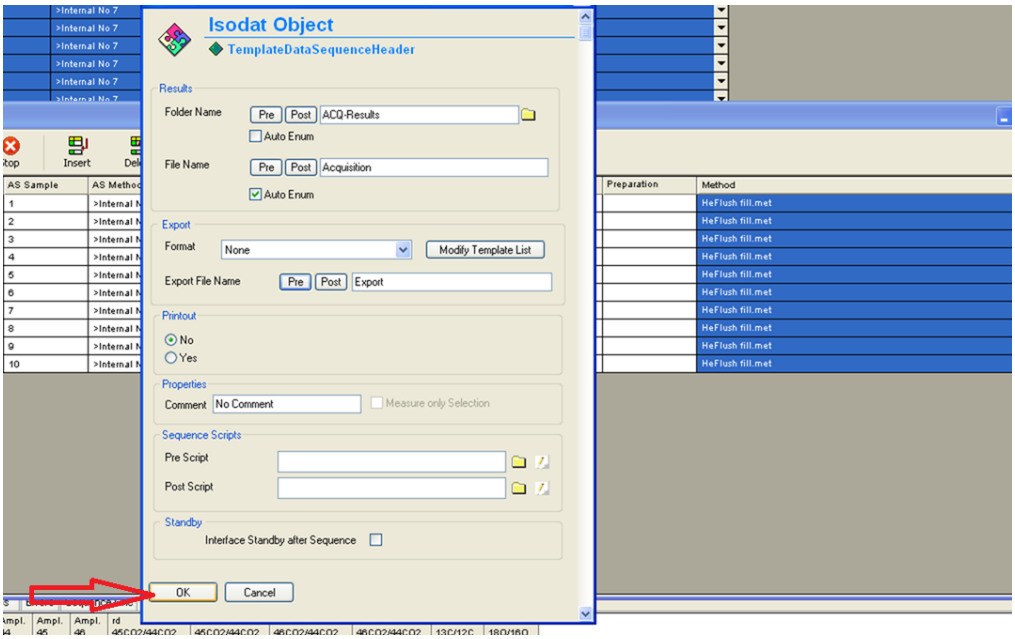
20. In the ‘Start Sequence’ dialogue box, it is advisable to select ‘Pre’ or ‘Post’ in the ‘Export File Name’ list…



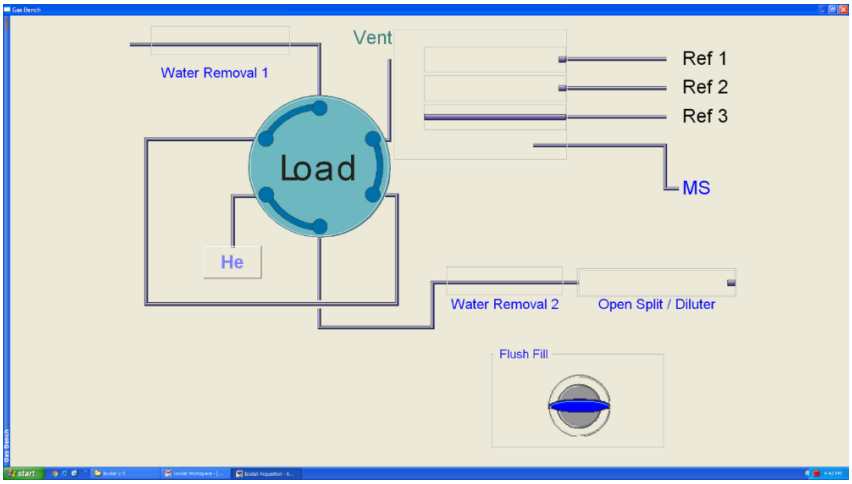
21. …and check all the options. This ensures that your results file (.dxf) carries all the details you have put in the table in the file name.



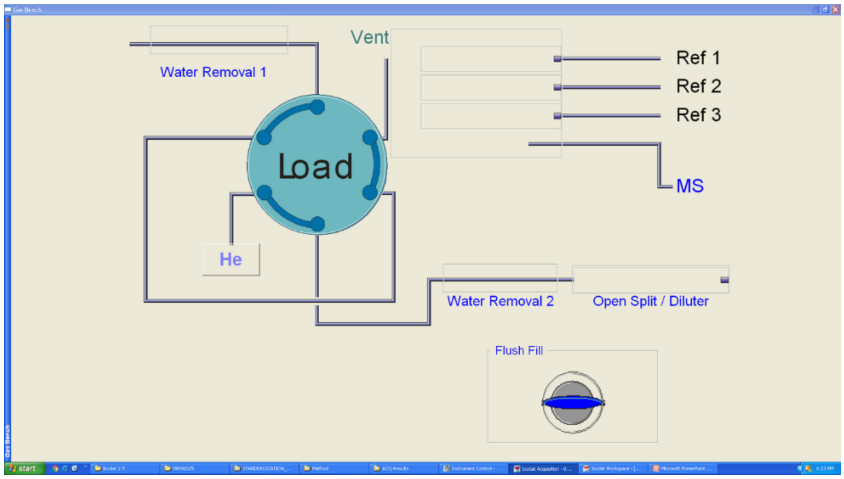
22. Then, click ‘Okay’ and your sequence will start.



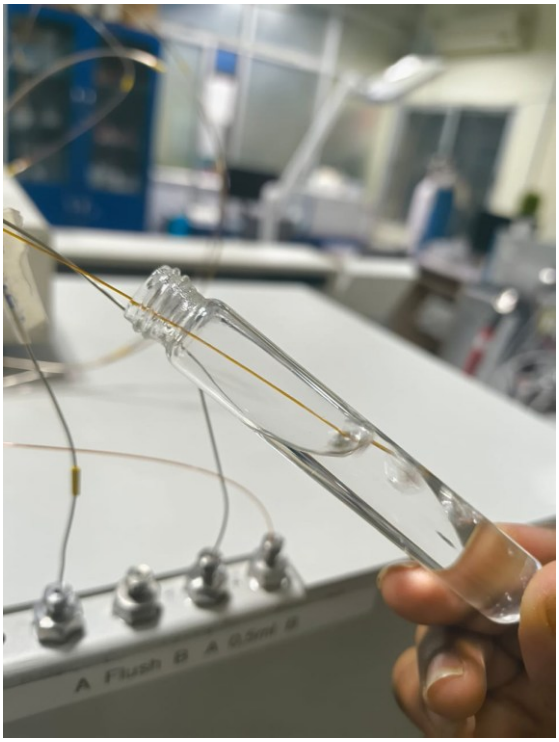
23. You will observe that at the time-stamps you had selected for ‘Reference on,’ the reference (here, 3) split will turn on.



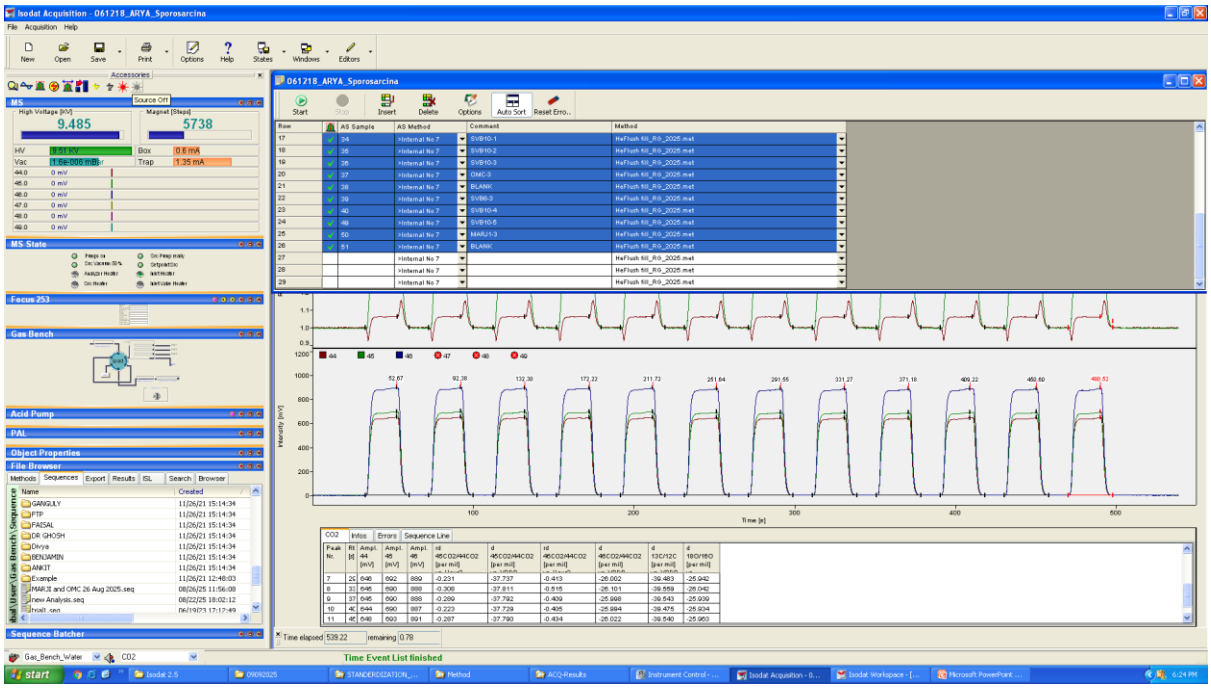
24. At other times, the reference split will remain off. Flush fill remains on throughout the sequence.



25. While flushing is going on *in the software*, it is a good idea to check whether it is *actually* going on in the exetainer vials. An easy way to check it is through the backflow. Simply take a container filled with ethanol or isopropyl alcohol and immerse the 0.32 mm silica capillary from the flushing needle into it. If flushing is actually taking place, you will observe heavy bubbling.



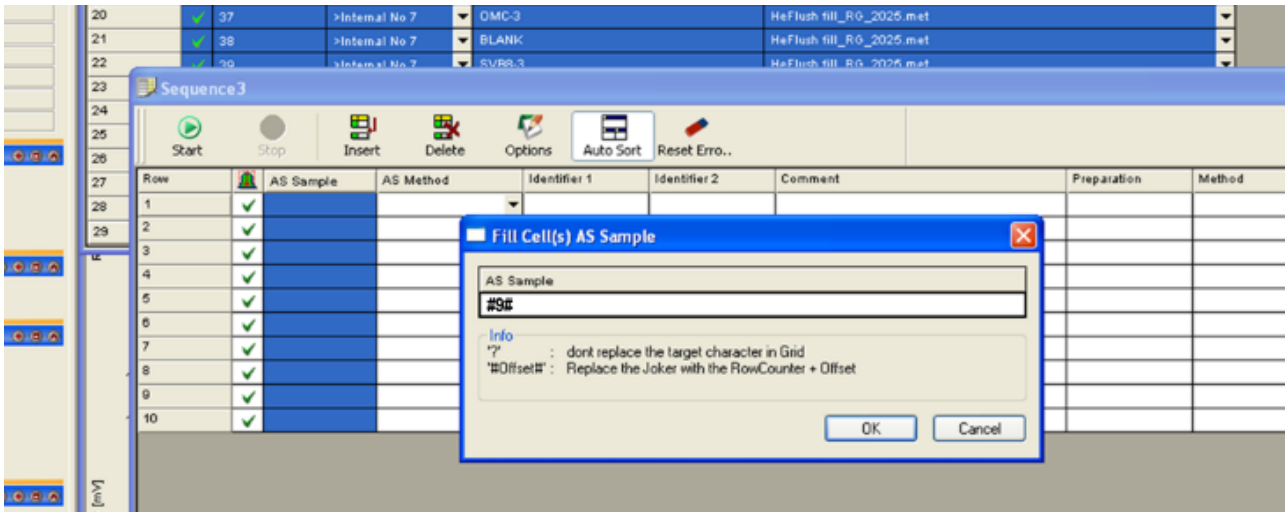
26. In the end, your *flushing* sequence (.dxb) file should look something like this.



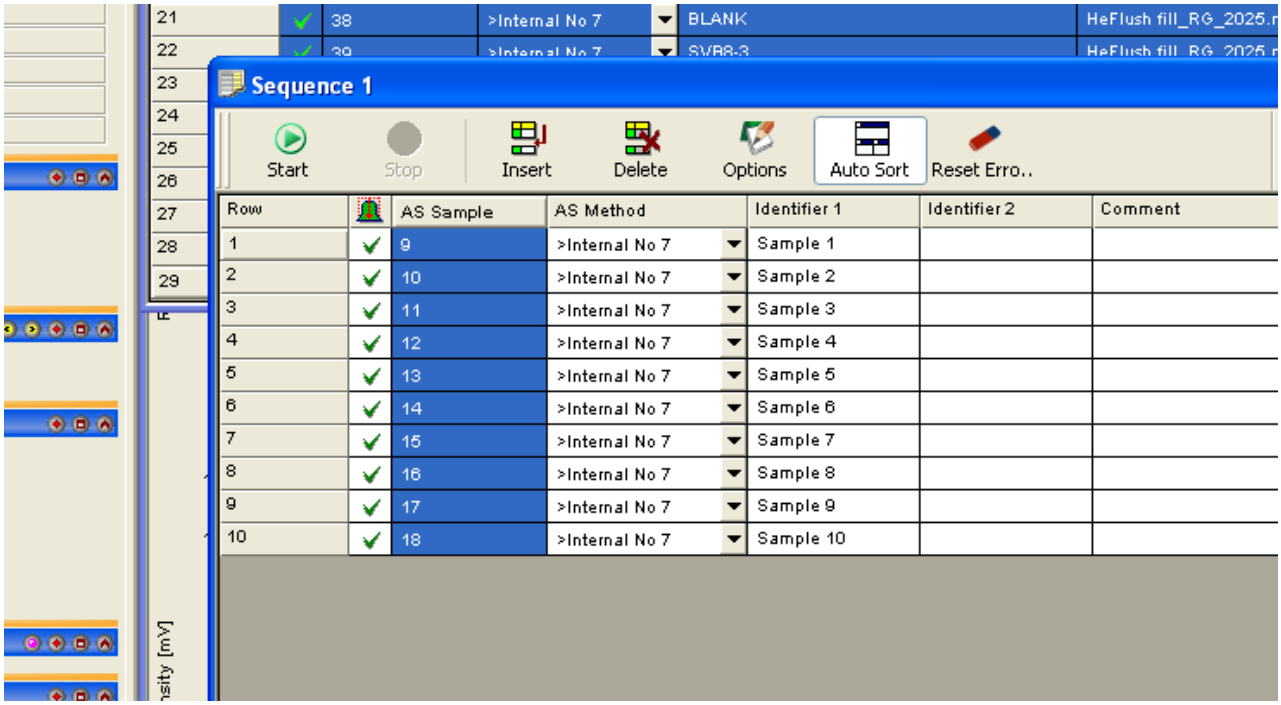
4.2 For Analyses

1. To create a sequence for analyses, follow the same steps as given from page 15. But, there are a few changes.

2. One, in the 'AS Sample' column, the column values should reflect the position for the left needle (remember: the autosampler does not know which needle is on the left – and *we* have kept the analyses needle on the left hand side). So, if you have kept exetainer vials from 9 to 16 (let's say), right click on the header of the 'AS Sample' column and write '#9#' as shown in the screenshot below.

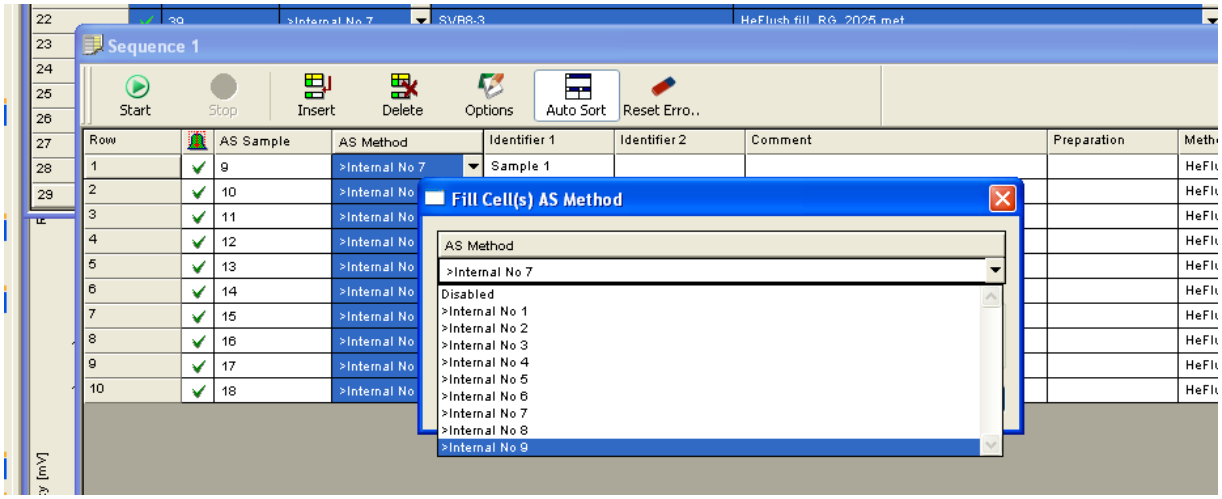


3. This will fill the column with values from 9 to 16[7].

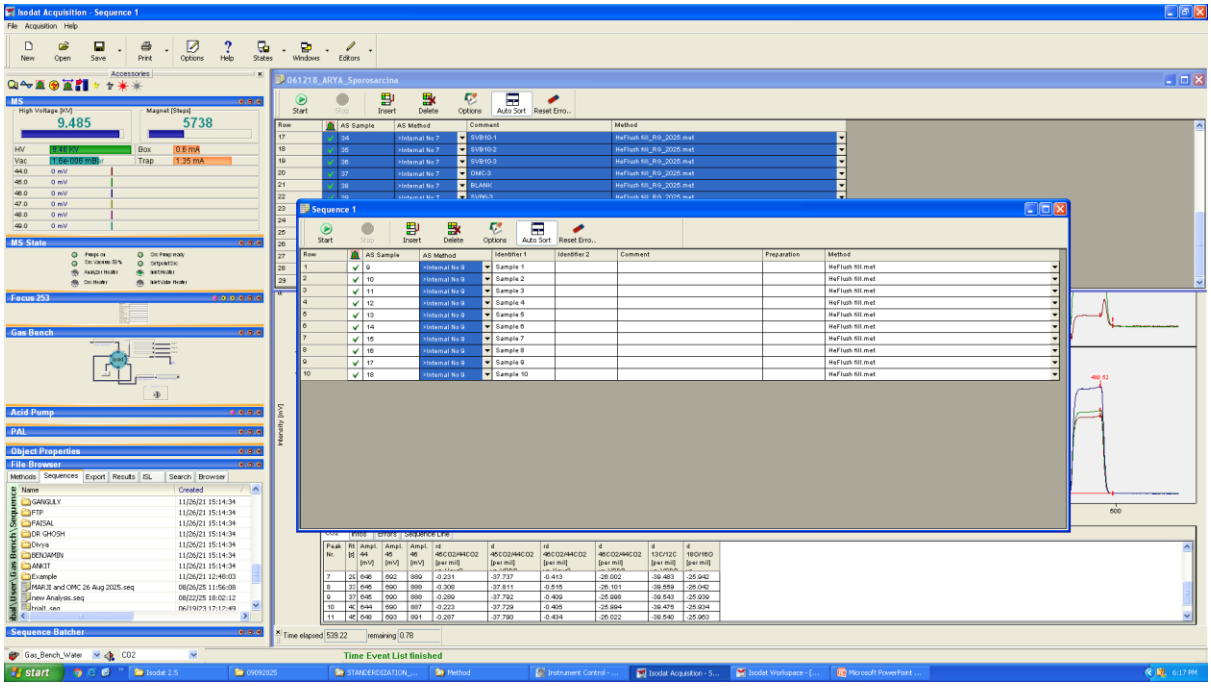


[7] Alternately, you may also keep the values same and change the position of the exetainers themselves in the autosampler tray in a way that they correspond to the needle you want to work with. But, this is not advisable for a variety of reasons.

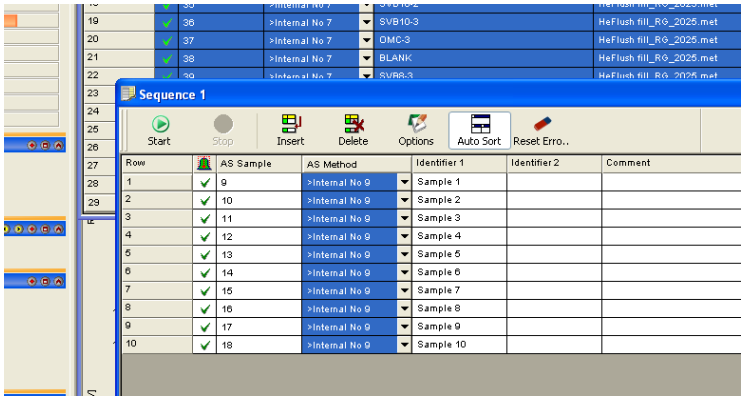
4. Then, you need to change ‘Internal No. 7’ to ‘Internal No. 9’ (Remember: Internal No. 7 is for flushing and Internal No. 9 is for analyses). We adopt the same approach as before; and right click the column header to select ‘Internal No. 9’.



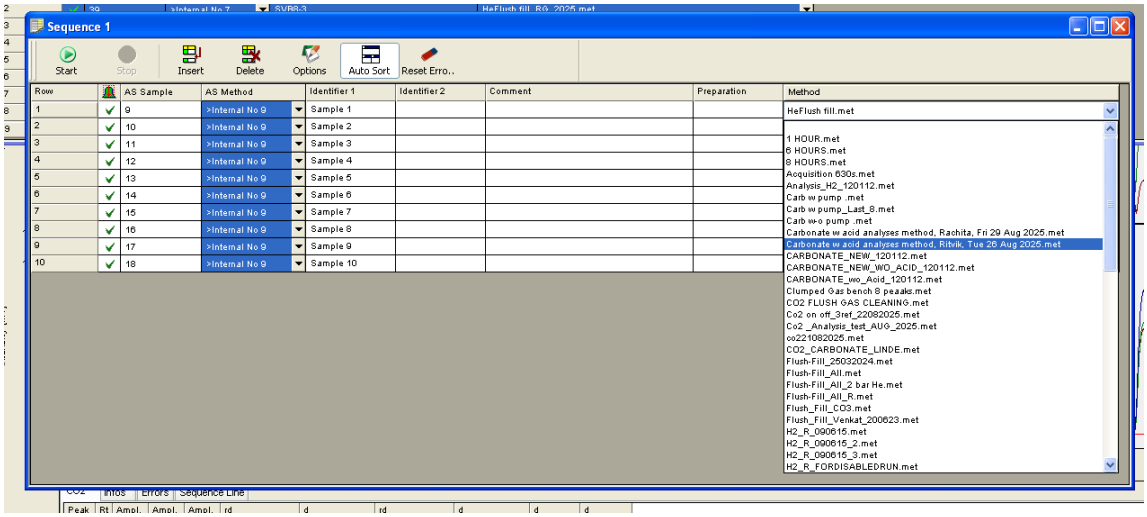
5. Now the entire list is filled with ‘Identifier No. 9’.



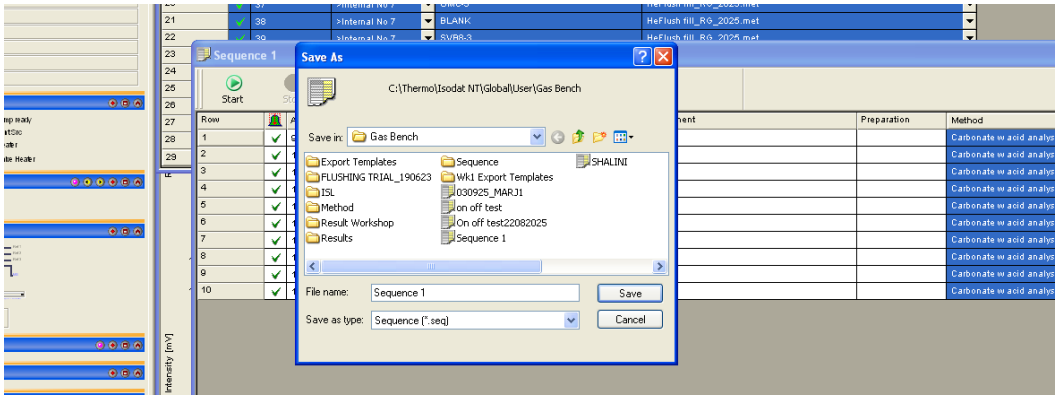
6. We will keep the Sample Identifiers same as before, of course.



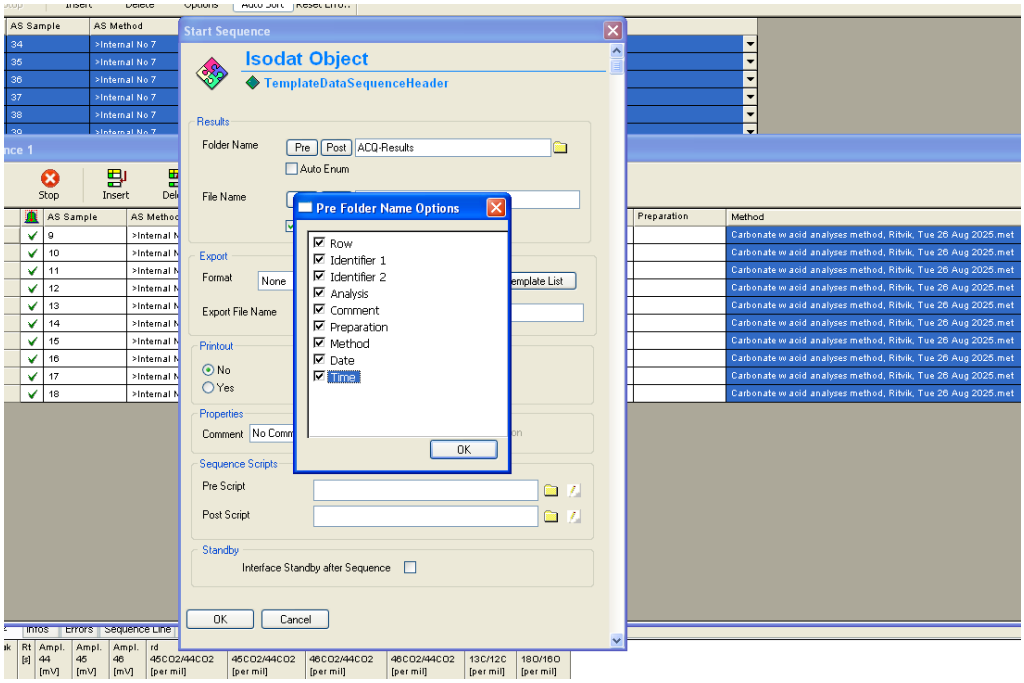
7. Then, we change the ‘Method’. I am using ‘Carbonate w acid analyses method, Ritvik, Tue 26 Aug 2025.met’. You may choose another method as per your preference, sample quantity etc.



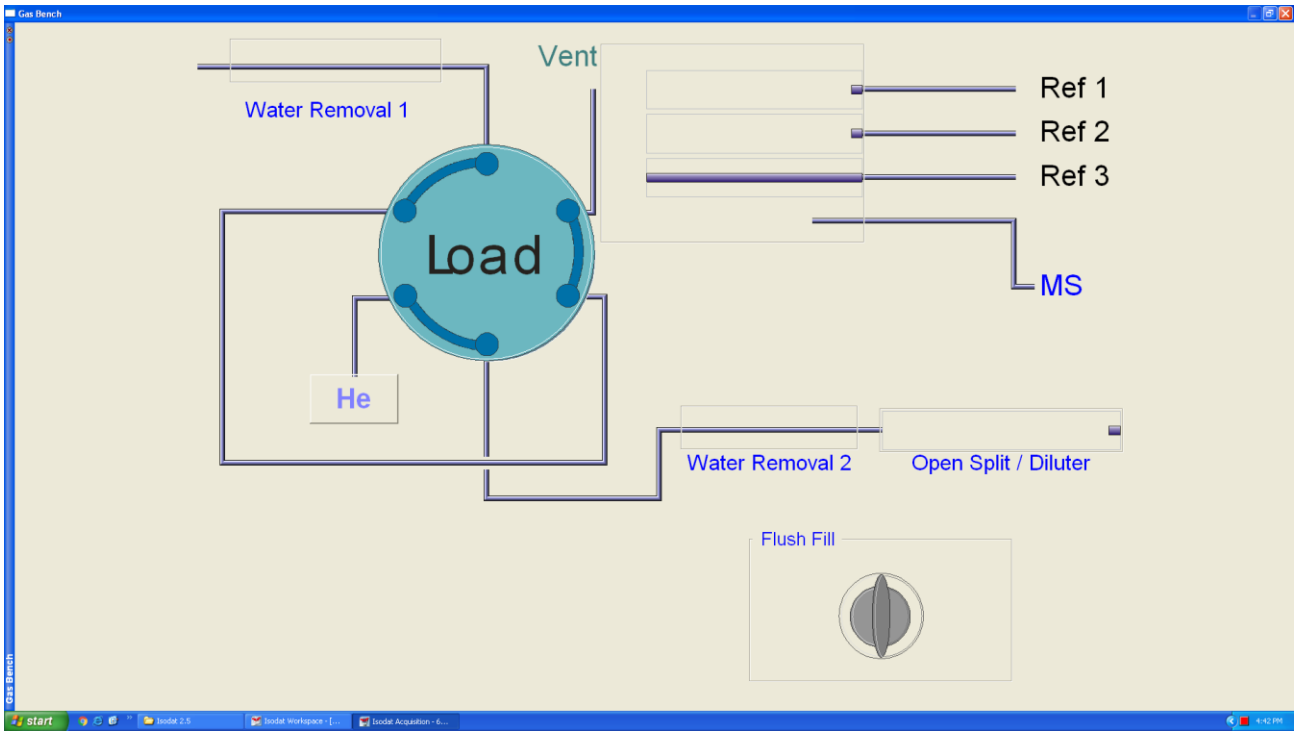
8. Again, as before, save the file…



9. …and make any changes to the file name at this step.

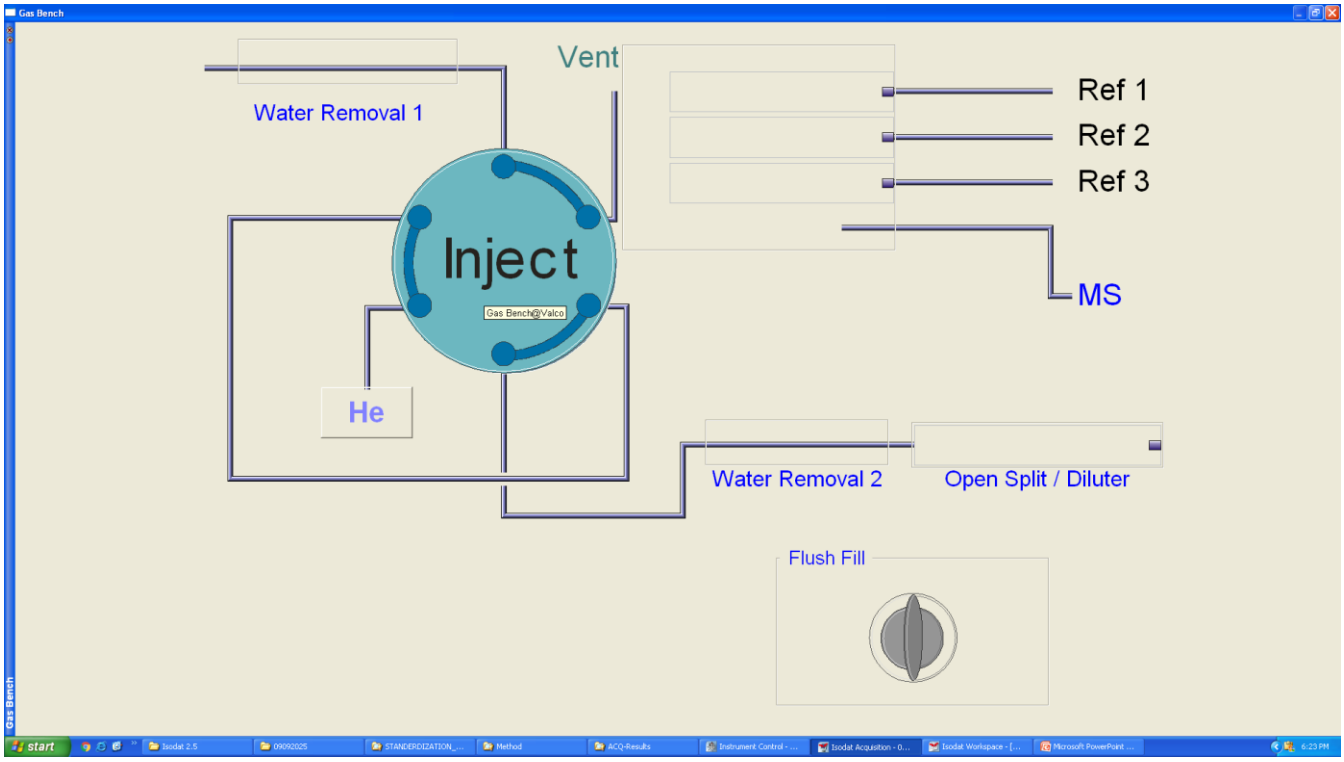


10. You will observe that at the time stamps you specified in ‘Method’ for Reference 3, the Ref 3 open split will turn on.

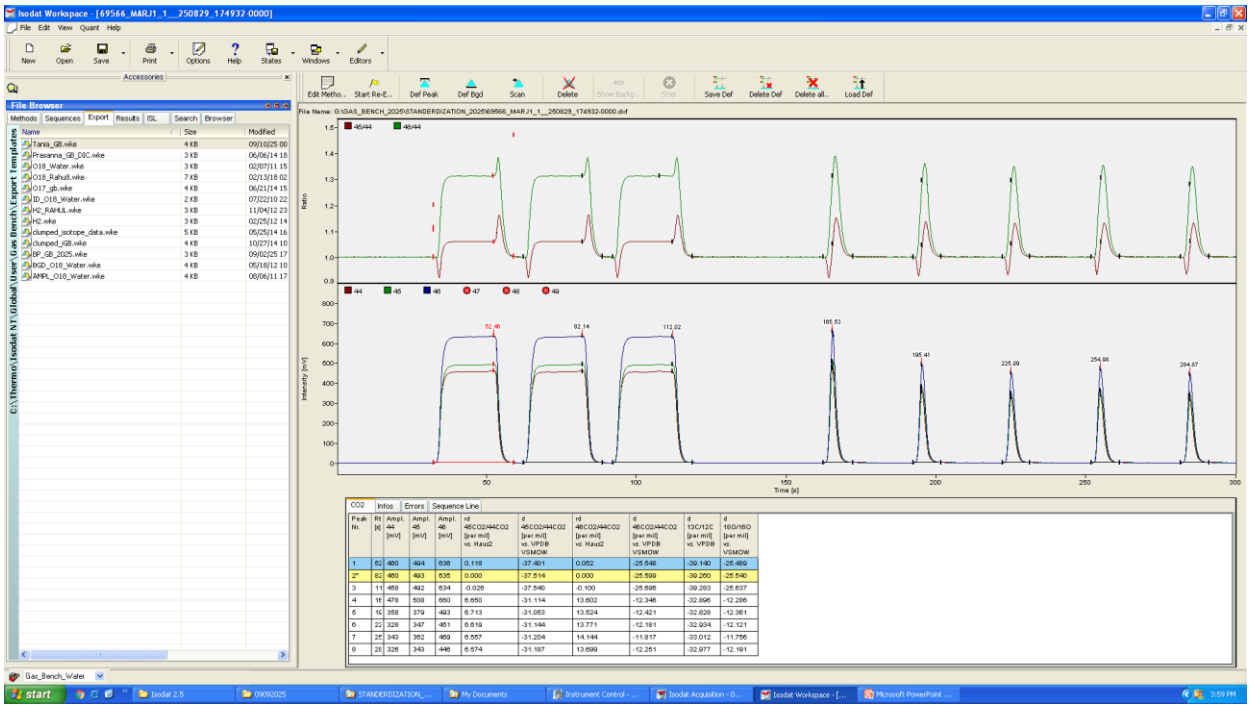


11. At other times, it will remain off.

12. You will also observe that the Valco 8 port valve symbol on the software stays in ‘Load’ mode constantly, and changes to ‘Inject’ only at the time stamp you specified. This is when the sample finally gets transferred to GC/Water Removal 2 etc. Note the connections of capillaries during ‘Load’ versus ‘Inject’



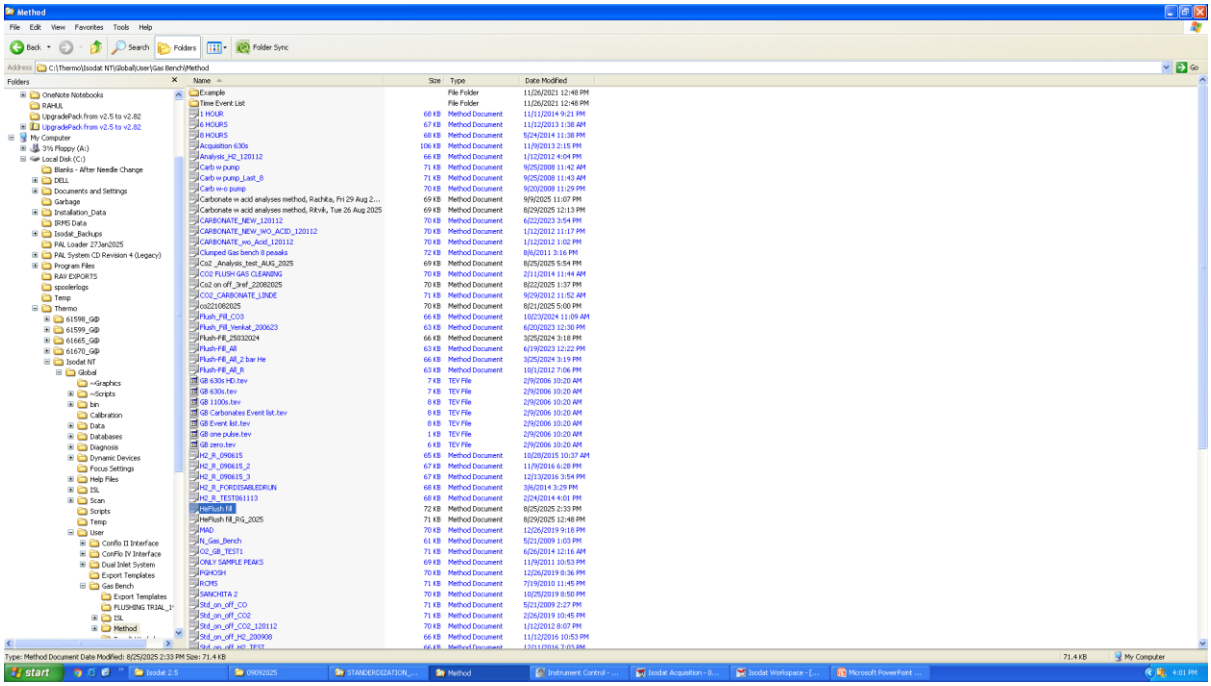
13. Once your analyses is done, your results should look something like this:



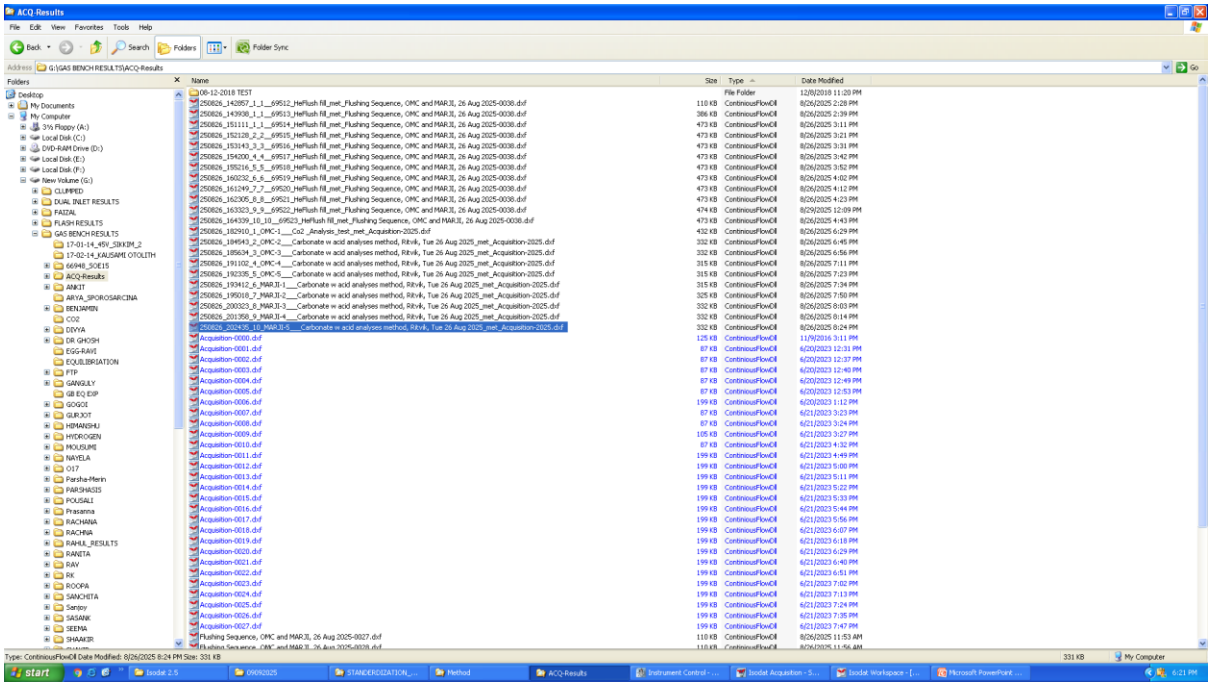
5 Viewing already created ‘Methods’ and ‘Sequences’

Viewing a ‘Method’ or ‘Sequence’ created long ago is easy as long as you remember the file path (which is why it is important to note it down when you are saving/creating a file). It is also advisable to take a copy of your file on your personal USB drive from time to time.

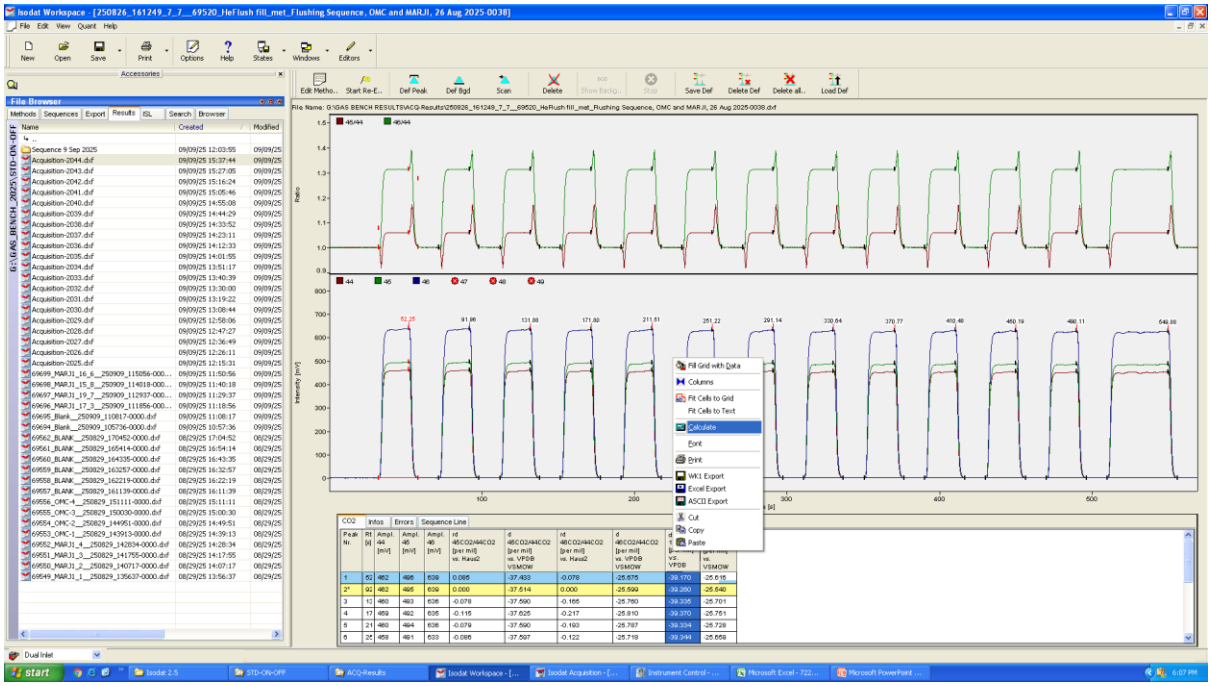
Simply go to Isodat Workspace > Click Open > Navigate to the file path and open the file of your choice. For e.g., for Methods, I navigate to this folder and open it in Isodat Workspace (I can do the same for a .seq file as well).



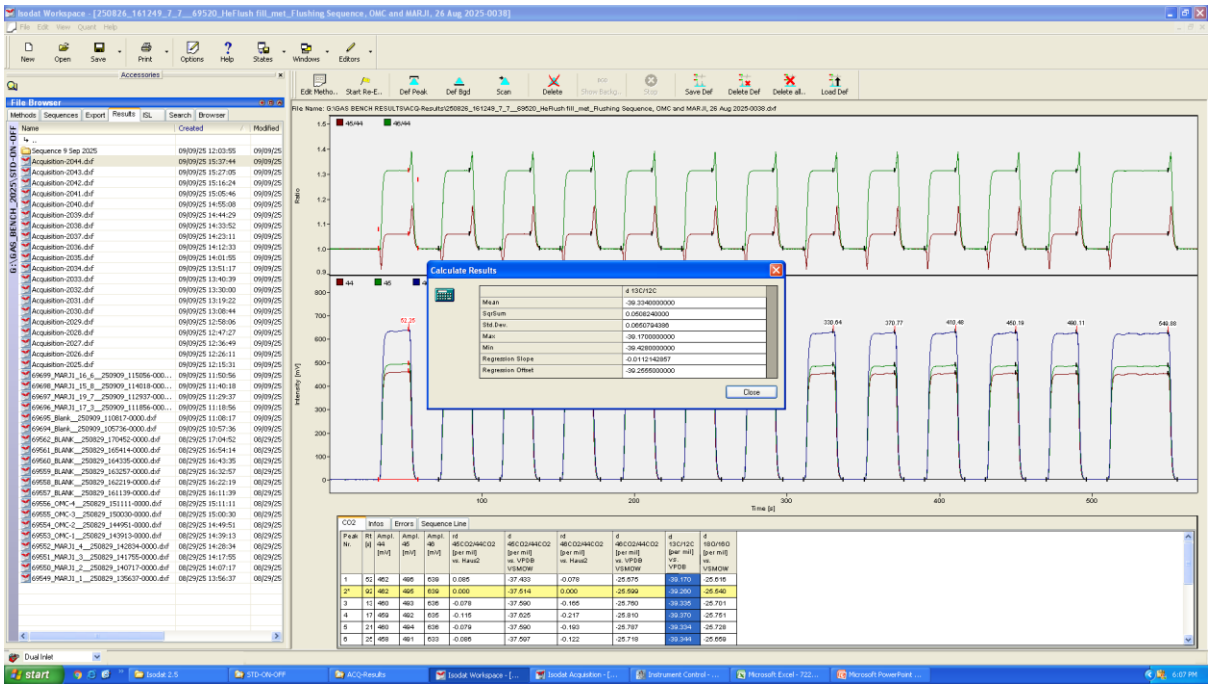
For viewing a Conflo results (.dxf) file, I navigate to a folder where I have saved it and view it in Isodat Workspace.



To see the standard deviation of a particular ‘Result’, right click on the $\delta^{13}C$ or $\delta^{18}O$ column and click ‘Calculate’



This will show you a standard deviation and other statistical parameters.



6 Frequently Asked Questions (FAQs) and Troubleshooting

When the instrument is in standby mode (i.e. no analyses or flushing is taking place), is it okay to switch off CO_2 completely?

Yes

When the instrument is in standby mode (i.e. no analyses or flushing is taking place), is it okay to switch off flushing helium completely?

Yes, in fact we switch on the flushing helium cylinder immediately before we start flushing; and switch it off immediately after flushing has terminated.

When the instrument is in standby mode (i.e. no analyses or flushing is taking place), is it okay to switch off carrier helium completely?

No, the pressure gauge for carrier helium in the Gas Bench should be at 0.3-0.4 bar even when there is no analyses running on the system.

The analyses I was running has suddenly stopped midway. What should I do?

Click the 'X' icon that says 'Stop'. Abort the operation immediately and proceed one row at a time.

In case the autosampler needle/arm is stuck or shows an error on the autosampler handheld display terminal, under no circumstances should you plug out the LAN cable that feeds the display terminal and plug it back in. Doing so may result in the display going completely defunct altogether. In case autosampler needs to be rebooted, remove the syringe cartridge from the arm and keep it separately. Then, switch off the power supply for the autosampler (not the Gas Bench!!), wait for 1-2 minutes and switch it on back. Reinsert syringe cartridge.