

Created on 07/05/2012 by Kevin A. Pollack

Updated 4/13/2015 by Lauren A. Link

Updated 2/13/2017 by Peter Wang

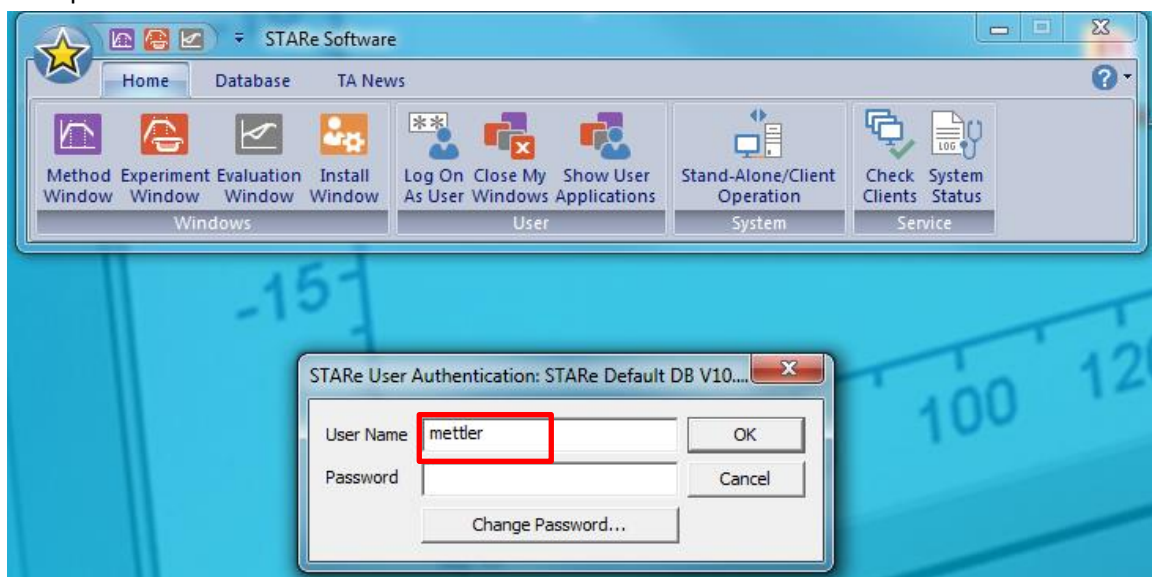
*The following is the Standard Operating Procedure (SOP) for the Mettler Toledo TGA/DSC 1. All Mettler Toledo instruments are operated using the STARe software v15.00a.*

## **I. General Use**

1. Double click **STARe Software**

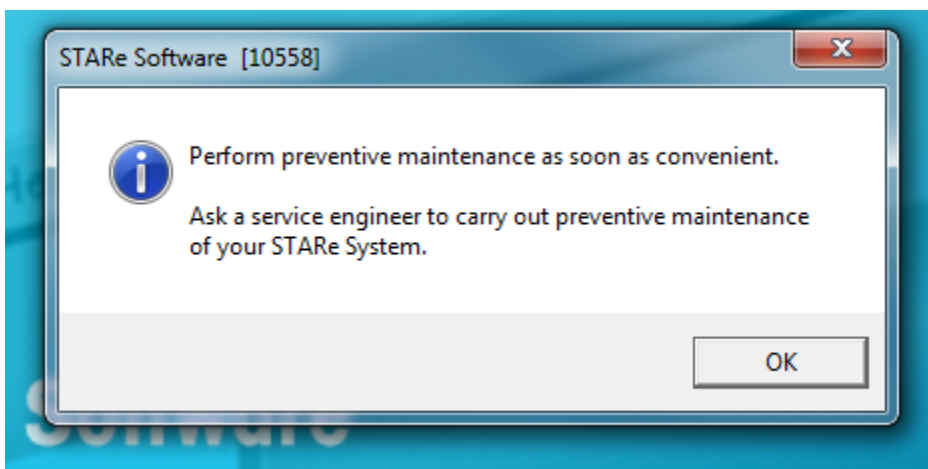


2. A prompt will appear asking for the User Name and Password. The User Name is **mettler**; there is no password. Hit **OK**.

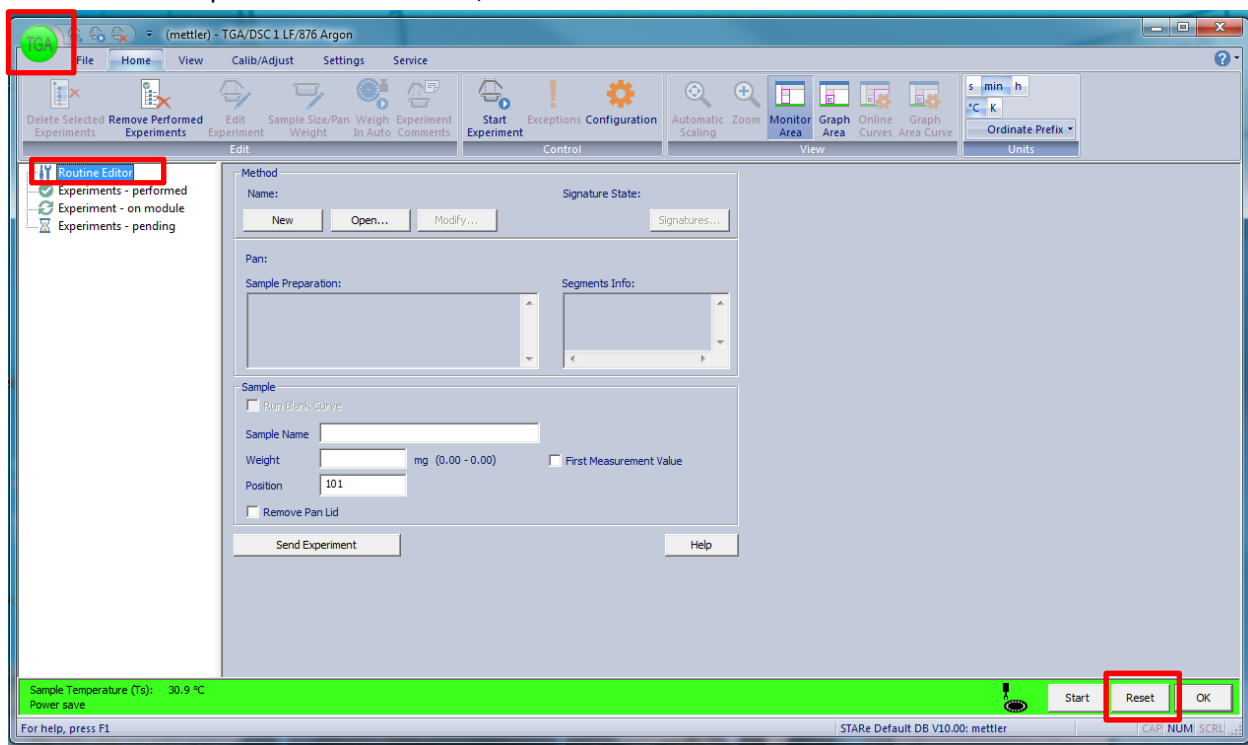


3. Sometimes there will be a window stating "Perform preventive maintenance as soon as convenient", you can just click **OK**. However, if you see any other warning message, please

contact the TGA team.



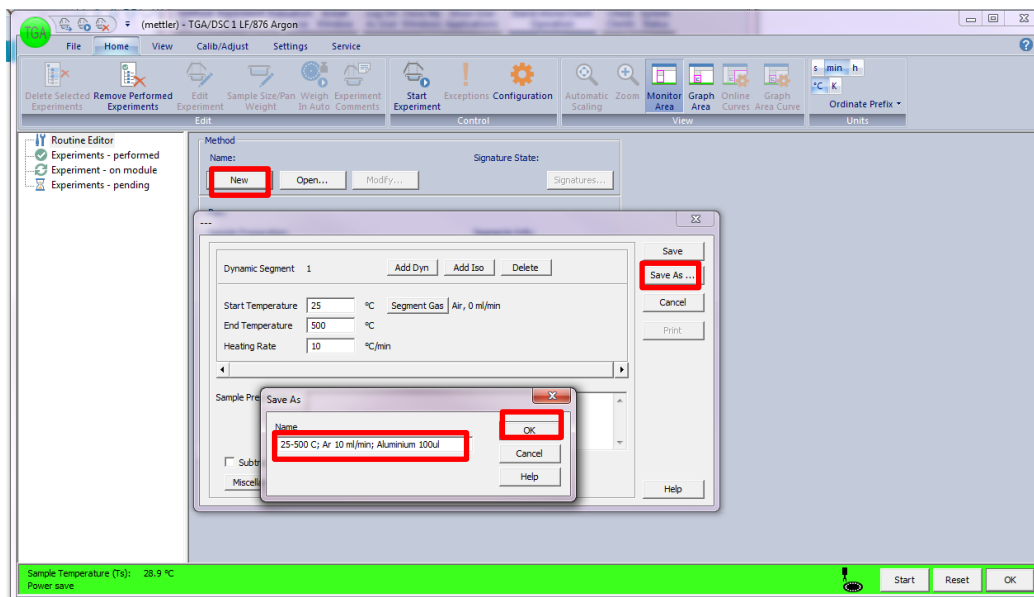
4. Open the window controlling the TGA (Green ball at the upper left corner states TGA)
5. On the left hand portion of the window, select **Routine Editor**.



6. Hit **Reset** in the lower right hand corner of the window.
7. If a method needs to be created, select **New** and create a program for the TGA (for specifics on creating a new Method, please refer to the person/s in charge of the instrument). **Save the method, and use "25-500C 10C/min; Ar, 10ml/min; Aluminum 100ul" as an example.** If a method is already created, select it using the **Open** tab.
  - The most common method for the TGA is 25-500 °C at a rate of 10 °C/min entitled 25-500C 10C/min; Ar 10 ml/min; Aluminum 100ul.
  - When creating a new method/using an old method, make certain that the pan being used matches the pan as outlined in the method. The most commonly used pans are the

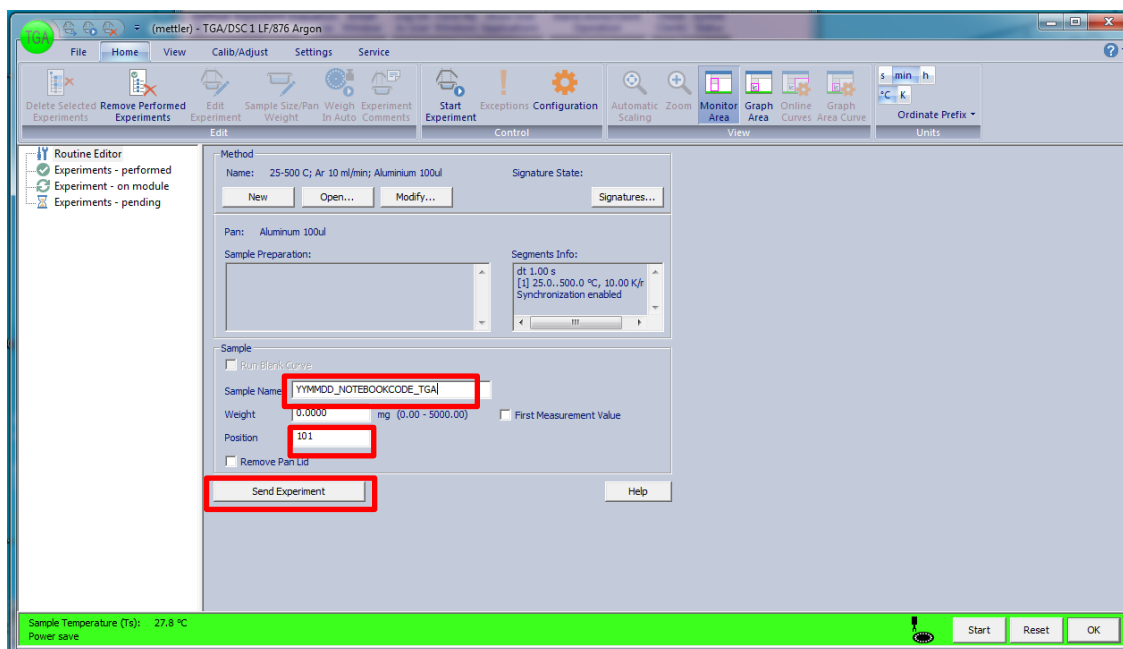
70µl Alumina pan and the 100µl Aluminum pan. The alumina pans are reusable and should be cleaned after use; the aluminum pans can be disposed.

- The maximum temperature for the alumina pans is 2000 °C; the maximum for the aluminum pans are 640 °C. **DO NOT TAKE THE PANS ABOVE 500 °C UNLESS APPROVED BY DR. WOOLEY!!!**

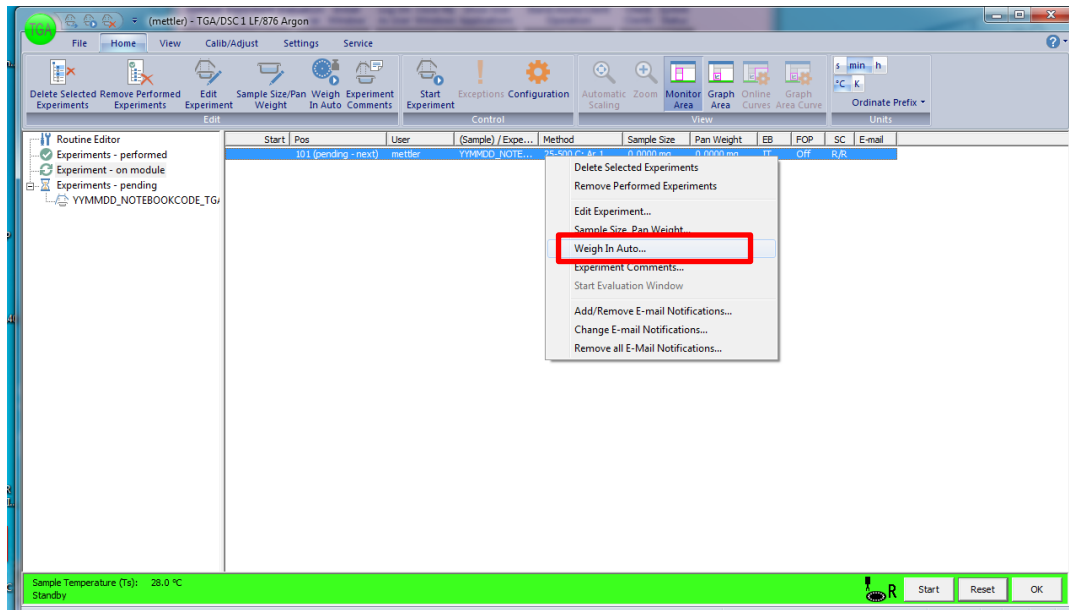


- Once a method is selected, place a sample name into the field. Sample name should be placed as follows: **YYMMDD\_NOTEBOOK CODE\_TGA**

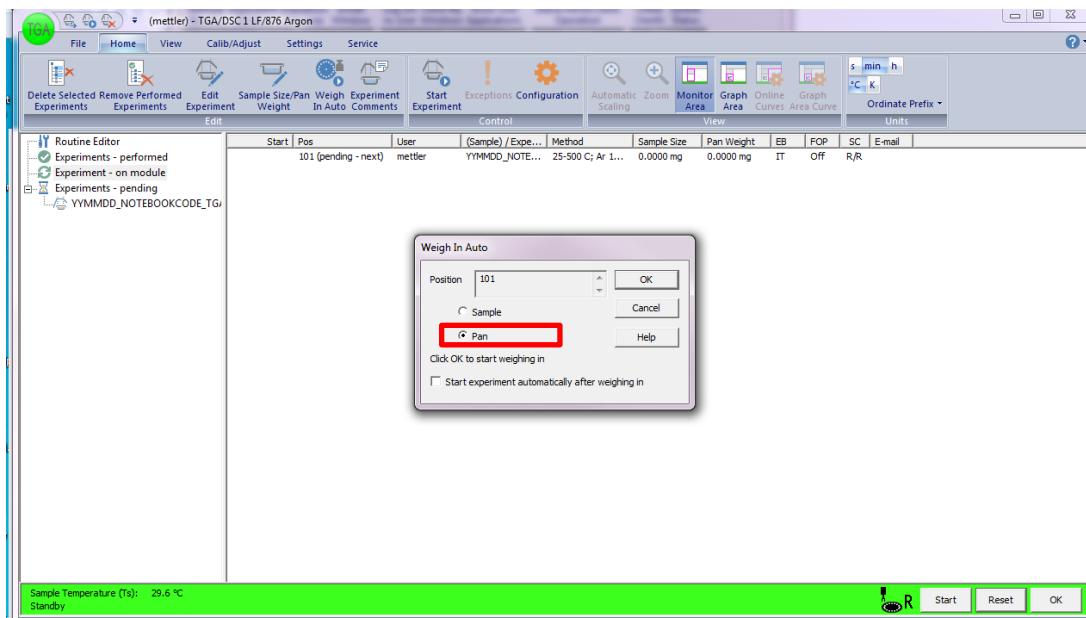
The weight can be left blank. The position should match the position on the carousel where you place the pan. Click **Send Experiment**.



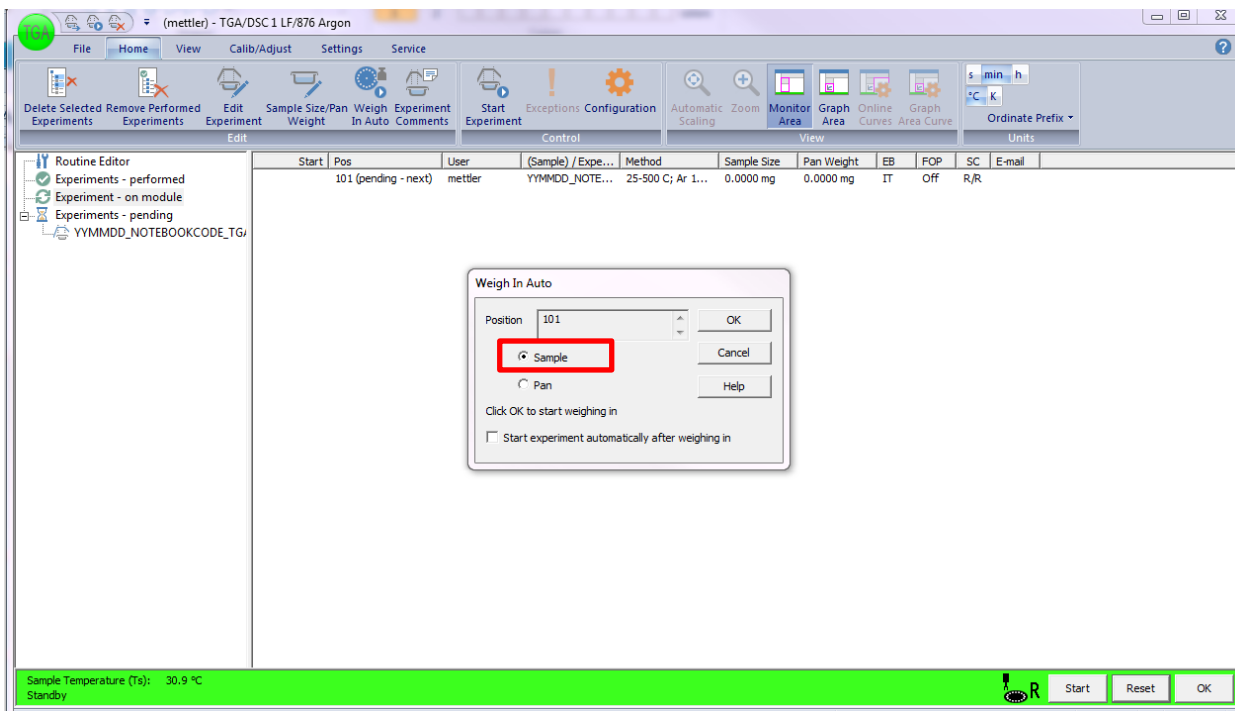
- For position 1 on the carousel, the position inputted is **101**, for position 2, **102**, position 10, **110**, etc.
- If more experiments need to be added to the queue, follow steps 6 & 7 (step 6 only needs to be followed if running a different routine).
  - Open up the **argon** tank. Click on **Experiment – pending** on the left tab of the window. Highlight all experiments and right click and select **Weigh In Auto...**



In the window, select **Pan** and hit **OK**. This will weigh out the pans for all experiments. Each pan takes between 3-5 minutes to weigh.



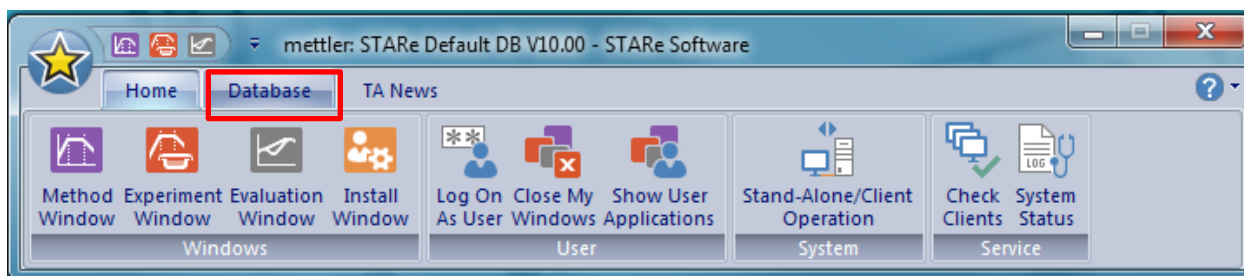
10. When the pans are weighed, add your samples to the pans. Right click on the experiments, select **Weigh In Auto...** then select **Sample** and hit **OK**.



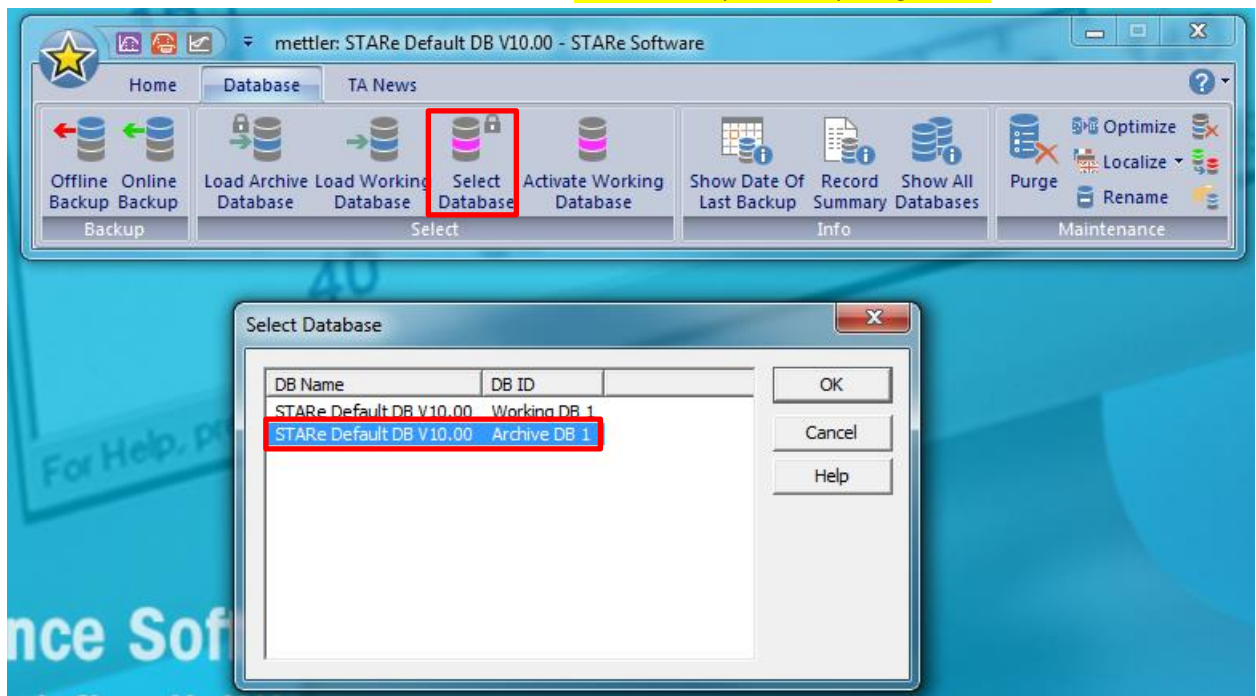
11. When all samples are weighed, select **Start** at the bottom right hand of the window. This will open the experiment on the module and will begin the experiment. Samples will run sequentially until all samples are finished.
- If the run needs to be stopped for any reason, press the **Reset** button in the bottom right of the window.
12. When the sample is done running, follow the procedures for cleaning the alumina crucibles below. If an aluminum pan was used, the crucible can be disposed.

#### Retrieving Old Data before 02/09/2017

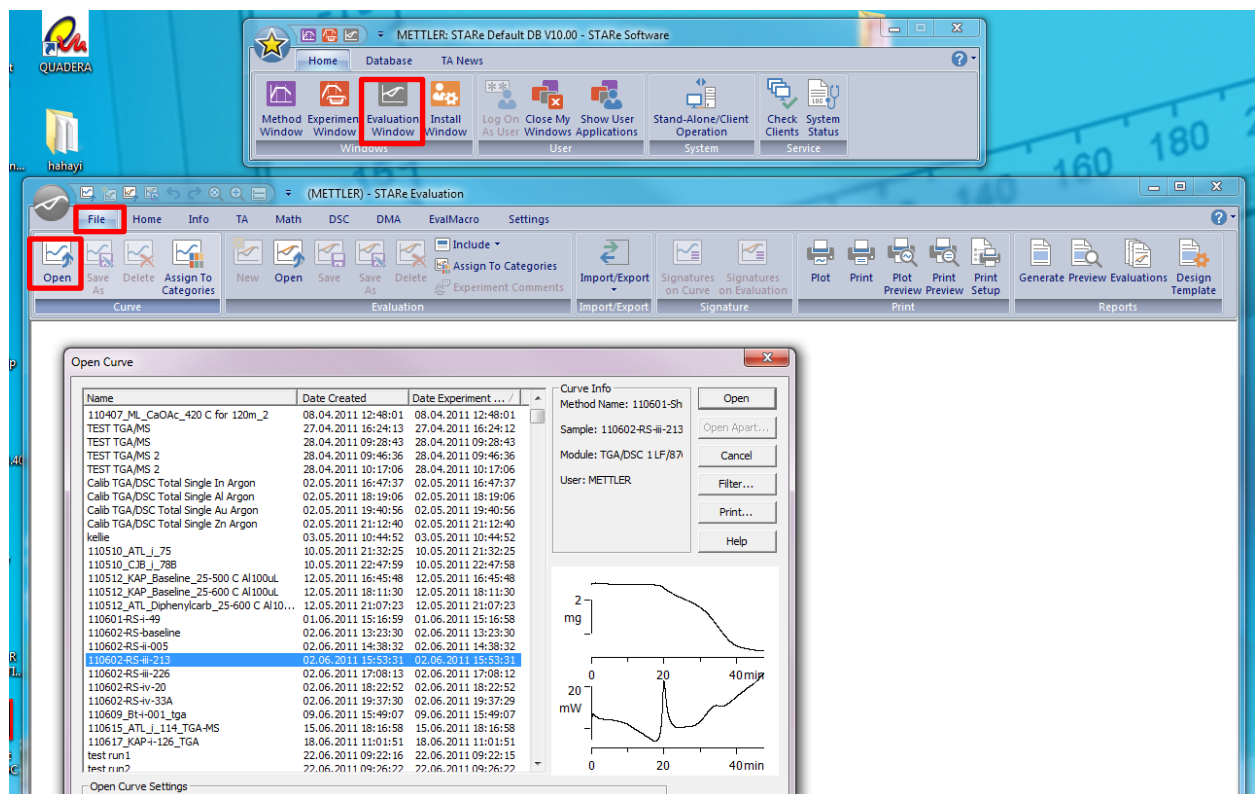
1. Click **Database** at the top part of the software



2. Click **Select Database** and chose **Archive DB1**. **Do not manipulate anything else!!!**

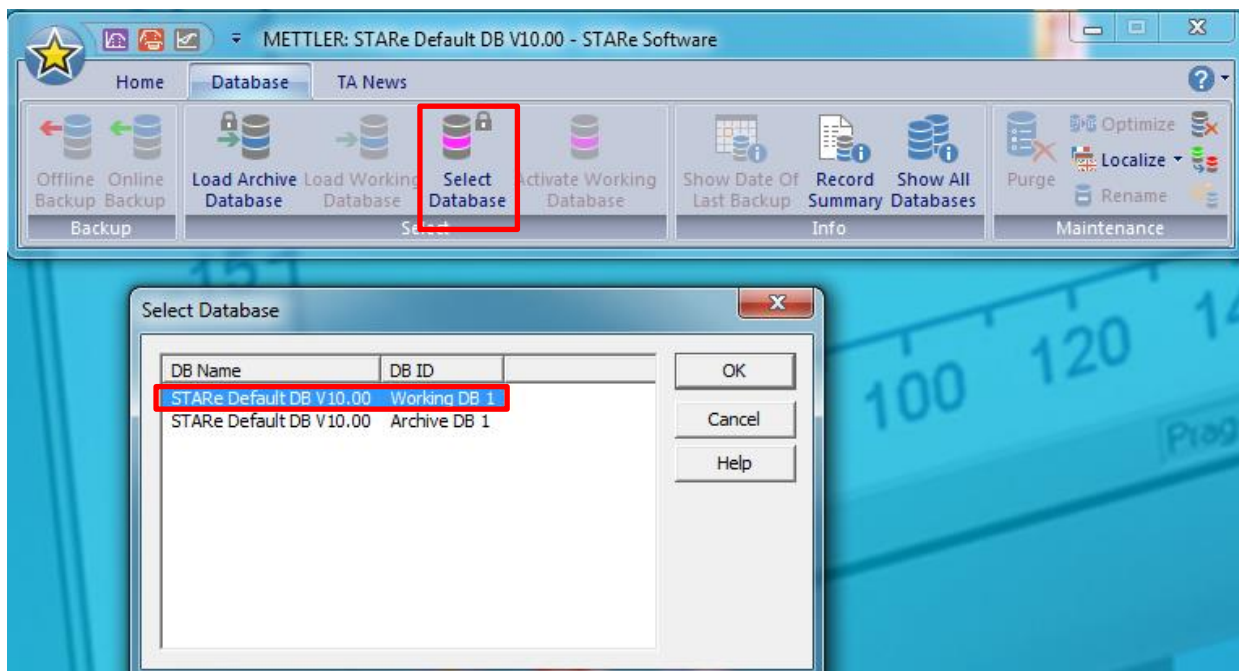


3. Now you can go back to the **Home** tab and click **Evaluation Window**. Click **File-Open Curve** and select your curve.





4. Following the same procedure as step 1-2 and switch the data base back to the original Working DB1.



### Alumina Crucible Clean Up

General Note: The white alumina crucibles are not disposable and should be reused. Please clean these after use.

1. After completion of the TGA program, remove the alumina crucible and place it in a solution of 1 M hydrochloric acid solution. Sonicate the solution to remove as much charred residue as in the crucible. Remove the crucible and rinse with water.
2. The alumina crucible should be placed back in the carousel of the TGA/DSC1. In the STARe software, open up the **Routine Editor**. Open the method entitled *BURN IN*. Use the name "BURN IN" as the sample name and click **Send experiment**. Repeat this step for as many crucibles that need to be cleaned. Hit the **Start** button in the lower right hand corner to begin the method.
3. Upon completion, examine the crucible to determine if it is clean. If the crucible is clean, place it back in the box with the alumina crucibles. If it is not cleaned, sonicate with 1 M HCl solution again. If this does not clean the crucible, organic solvents and running the BURN IN method again can be used to finish the cleaning process.