Missouri University of Science and Technology
Chemistry Department

TA Instruments Q50
Thermo-Gravimetric Analyzer (TGA)
Operation Guidelines

Developed By: J. Counsil	Release Date: Sep 2010
Revised By: W. D. Satterfield	Revision Date: May 2018

General:

The Q50 TGA, manufactured by TA Instruments measures the change in mass of a sample with respect to temperature. It utilizes a sensitive micro-balance and precision temperature control.

First, as with any of the shared departmental instrumentation, sign the logbook. Please write legibly and include your full name and the full name of your advisor so we may contact you if necessary.

Research users are required to purchase their own sample pan. Check with your advisor, your group may already have one. If you need to purchase one, contact either Dr. Leigh or Mr. Satterfield for a link to vendors/sources.

Load and unload the sample pan using the instrument software controls only. Do not manually load the pan onto the hang-down wire. Never touch the hang-down wire or thermocouple, as they can be easily damaged. In the event of a stuck pan or fallen hang-down wire, contact department technical support.

Inspect the inner wall of the furnace jacket. If significant residue is observed, the jacket should be cleaned before use. Report any problems (low gas cylinders, instrument errors, dropped pans, hang-down wire, dirty jacket, etc.) immediately to either Mr. Satterfield or Dr. Leigh. Make a note in the logbook with a brief description of the problem.

Start-Up:

Open N_2 cylinder and other gasses if required.

Instrument Power ON (switch is located on the back of the unit)

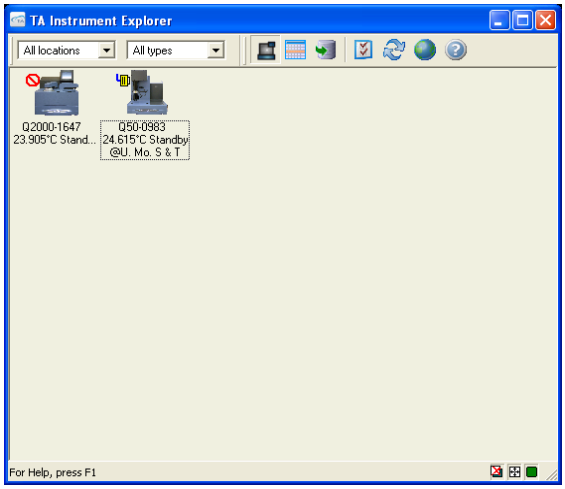
The Q50 will take approximately 2 minutes to boot up, run its self diagnostics and initialize. When the furnace has opened and lowered completely, the instrument is ready for use.

Start-Up *(cont'd)*:

Open TA Instrument Explorer. Double click on the Q50 TGA icon.

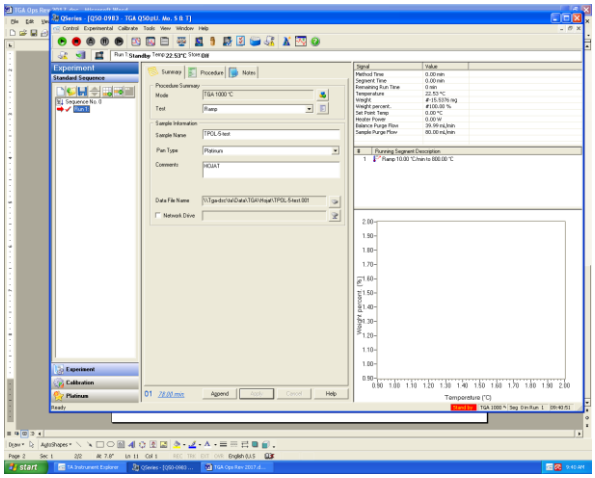
Sometimes the icon images of the instruments do not show in the TA Instrument Explorer window. The text will be present, and the "hotspot" will still be active. This is a known issue with the software. It does not affect the operation of the instrument.

The TA Advantage software supports both the TGA and DSC. Both instruments may be run simultaneously.



The program loads the last user's method when it opens. If you have a saved method, open/load it and double check that all the parameters are correct. Otherwise, continue to Build Method which will describe the steps necessary to create a new experiment.

The image below shows a typical start screen, with the previous user's method loaded.



A few things to note from the previous screen-shot:

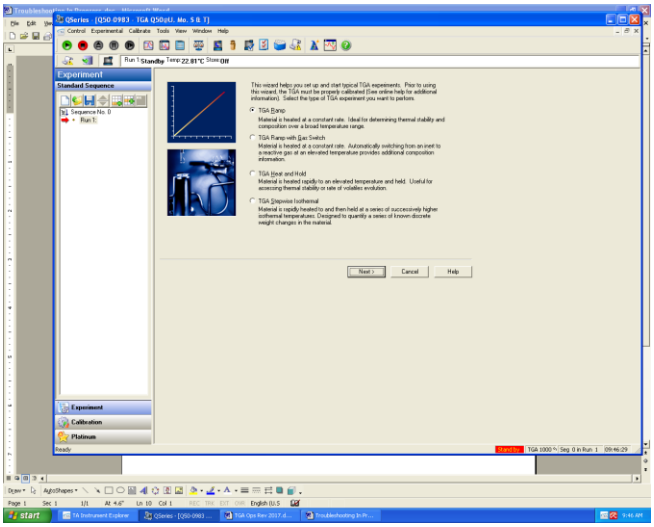
The image shows that the Q50 is in Standby mode in the status bar.

The Experiment/Standard Sequence pane on the left shows that Run 1 has been completed by the red checkmark. To start another run using the same operating parameters, simply double-click the checkmark to reset.

Remember to change the username, filename and folder, as appropriate, but do not change the entire file/data path.

Build Method:

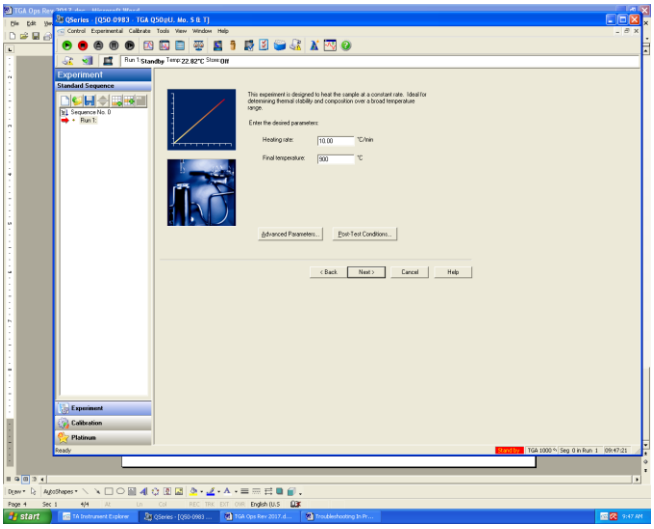
The easiest way to build a basic TGA method is to use the Experimental Wizard. The Wizard, which can be started by either clicking on the conical hat icon or via the Experimental menu, will ask a few questions and guide the user through the set-up of a basic TGA experiment.



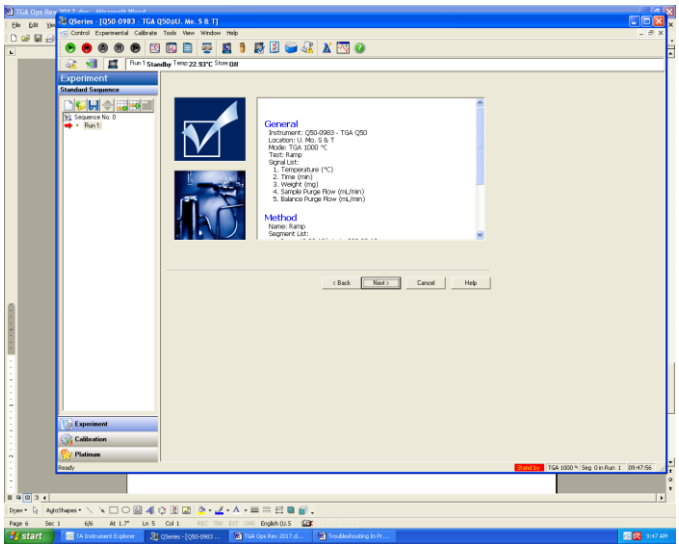
The most commonly used options are TGA Ramp and TGA Ramp w/ Gas Switch. The headings are fairly self-explanatory. Customizing the method will be described later in this guide.

Click **Next** to continue.

This window is where you can set the heating rate and final temperature. Do not make any changes to either the Advanced Parameters or the Post-Test Conditions.

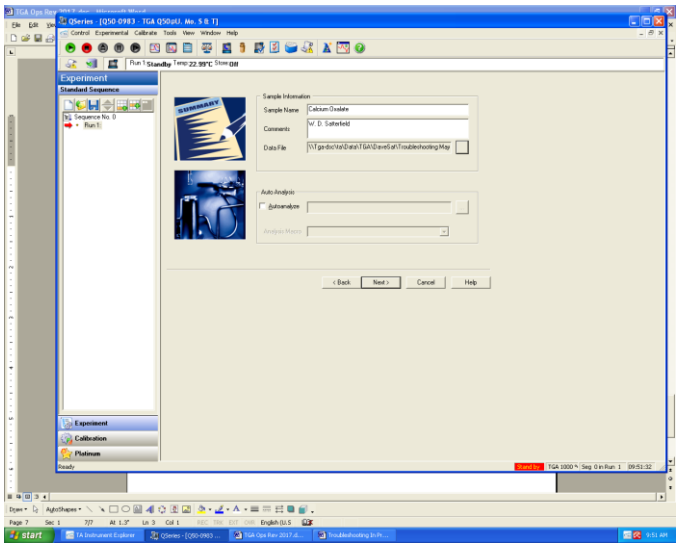


The next screen is simply a summary of the parameters and settings so far:



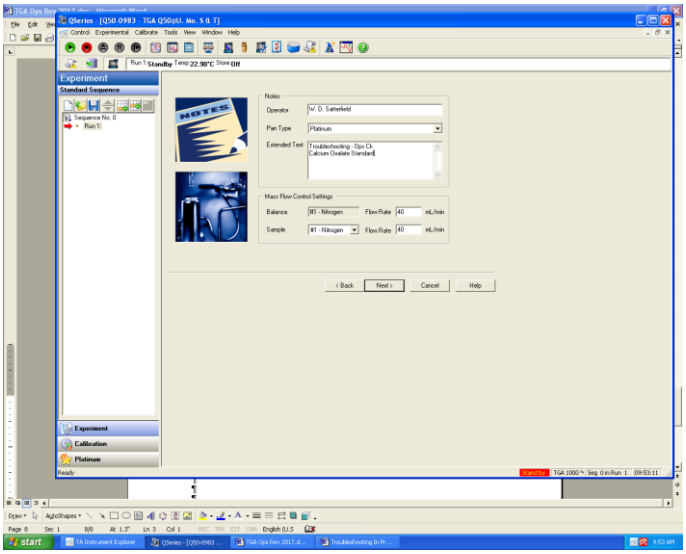
Click Next to continue.

This screen allows you to enter some basic sample information and name your data file.



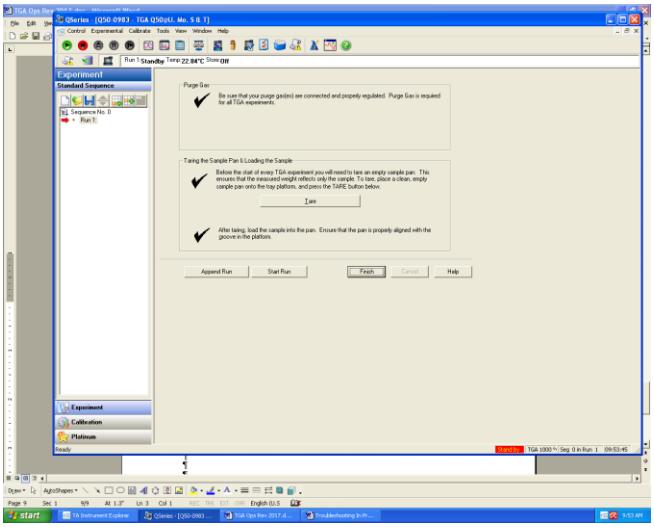
Click Next to continue.

Here you can enter your name and an explanatory note if desired. You can also select the sample purge gas and set its flow rate. The balance section of the Q50 is always purged with nitrogen at a flow rate of 40 mL/min. The furnace has several options for its purge.



Gas 1 is always nitrogen. Gas 2 is fed through a valved manifold by which you can choose between air, pure oxygen, or an alternative inert gas. You can also vary the furnace purge rate as necessary to clear the furnace chamber of evolved gases and/or combustion products.

This is the final screen of the Experiment Set-Up Wizard. It directs you to tare the pan, which defines zero mass for the instrument, and then to load your sample into the pan.



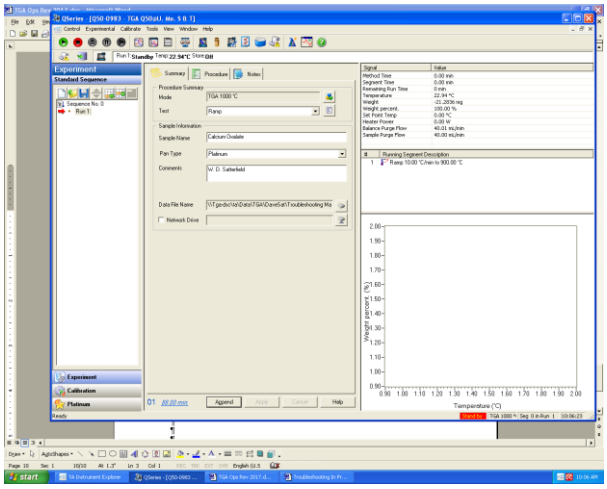
Never load your sample into the pan while it is hanging or while it is on the loading platform. Remove the pan, using brass tweezers, and set it on a clean, flat, stable surface to load sample.

How much sample is needed? The exact amount (mass) of the sample is not important to the TGA measurement, it will be defined to be 100%. However, in order to get a good result, the sample should undergo several 10s of µg of mass loss over the duration of the run. Typically a few mg is sufficient. Remember, the total capacity of the internal microbalance is only 1 g, which includes the pan and any secondary container you may use.

Click Finish to continue, which will close the Wizard and show the main TGA control window. From there you can double check that you have set up your experiment correctly or edit/modify your method as necessary.

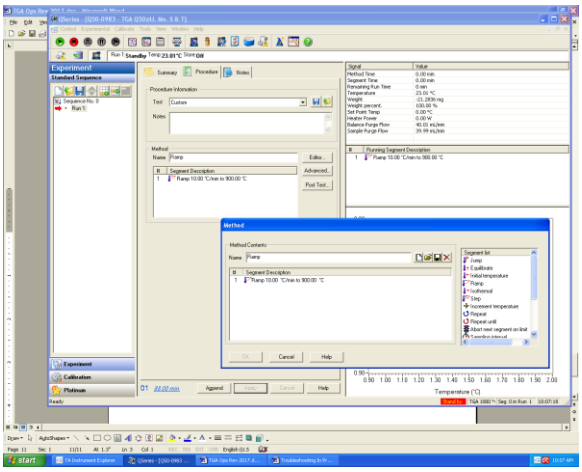
If you are 100%, absolutely, unquestionably certain that all the instrument and method parameters have been set up properly, you could start the run from the final Wizard screen. It is not recommended, however.

After setup via the Wizard is completed, the main instrument control window is shown. You can see that it is the same start screen as when the software was opened, except of course, it now shows your method instead of the previous one.



Here you have the opportunity to check the method information entered. If you are simply running a basic temperature ramp, load your sample by the control menu: ***Control → Sample → Load*** then ***Control → Furnace → Up.***

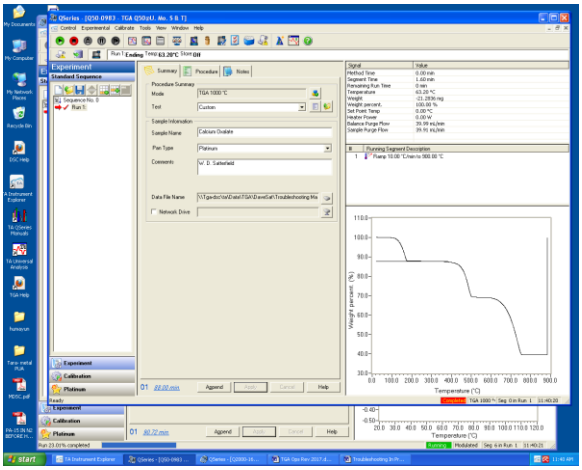
On the other hand, if you wish to customize your method – holding isothermal, changing heating rates or gases in mid-run, etc. – then next to the Test box, click on the arrow to open the drop down menu and change it from Ramp to Custom. Then click on the Procedure tab and you will see an Editor button in the Method box. Clicking on the Editor button will open another dialog box from which you can add additional steps and set their parameters.



Simply drag and drop the additional commands into the Segment Description window and change the values as required. To remove a step, highlight it and press Delete on the keyboard.

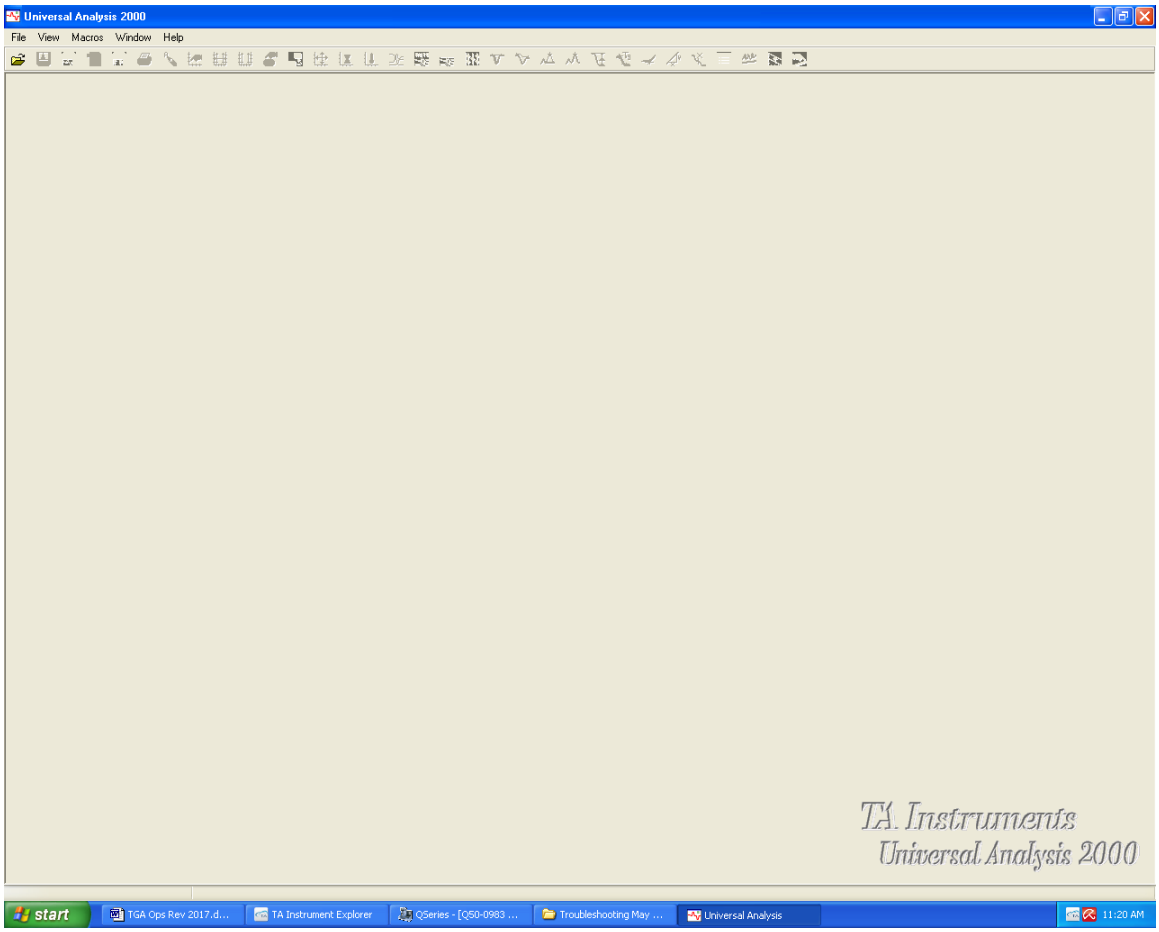
Once the sample is loaded and the furnace has closed completely, start the run by either clicking on the green ▶ button or from the menu bar ***Control → Start***

Observe the main window as the run begins:



In the lower left corner of the middle pane, it shows the approximate time that it will take to complete the run. Be back at the instrument when the run finishes.

If you wish to terminate the run early, do not use the red ■ stop button. Instead position the cursor in the Running Segment pane, right click and select “GoTo Next Segment” as required to step through the remaining segments until you reach the end. This will ensure that the instrument does not stop in an indeterminate state and that the Post-Test conditions execute properly.


Universal Analysis 2000
File View Macros Window Help
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QSeries - [Q50-0983 ...
Troubleshooting May ...
Universal Analysis
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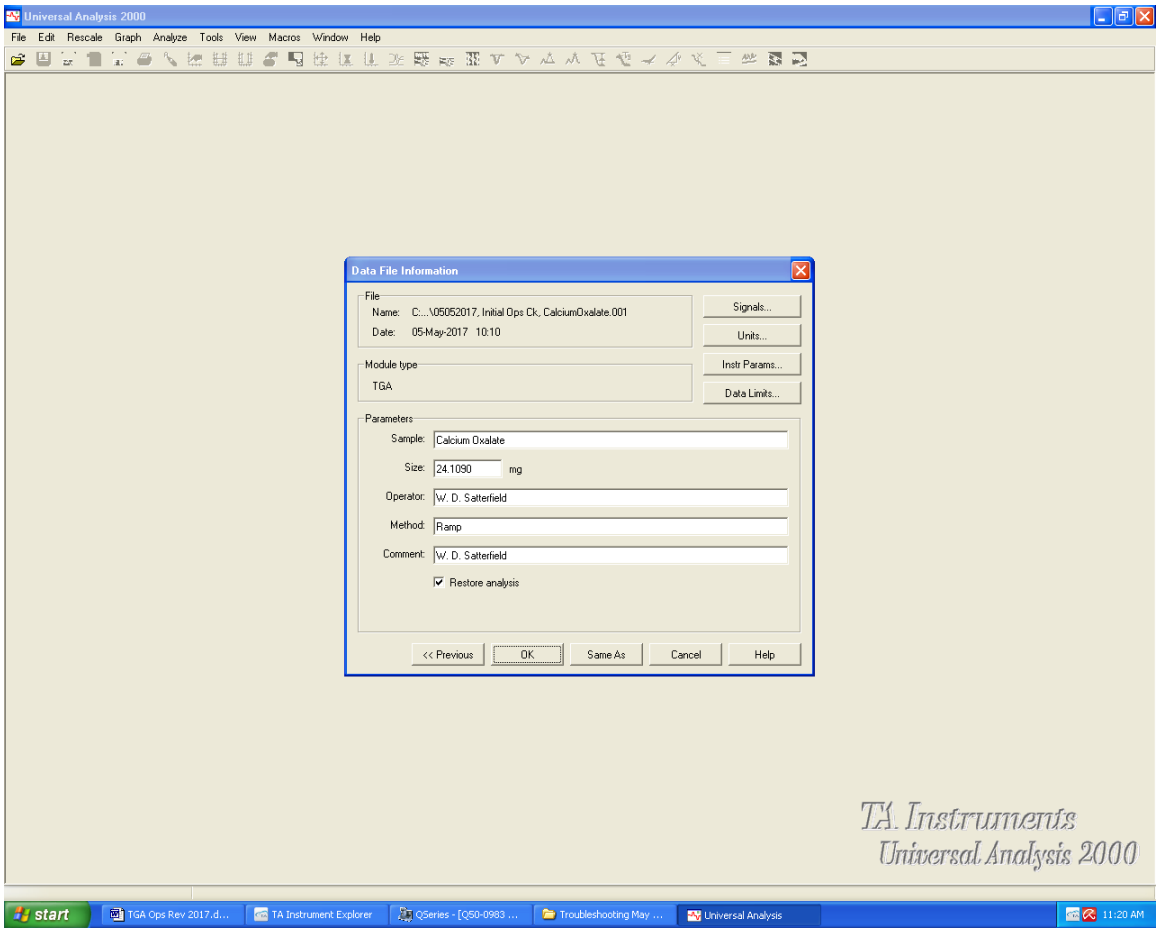
Universal Analysis 2000
File Edit Rescale Graph Analyze Tools View Macros Window Help
Data File Information
File
Name: C:...\05052017, Initial Ops Ck, CalciumOxalate.001
Date: 05-May-2017 10:10
Signals...
Units...
Instr Params...
Data Limits...
Module type
TGA
Parameters
Sample: Calcium Oxalate
Size: 24.1090 mg
Operator: W. D. Satterfield
Method: Ramp
Comment: W. D. Satterfield
Restore analysis
<< Previous
OK
Same As
Cancel
Help
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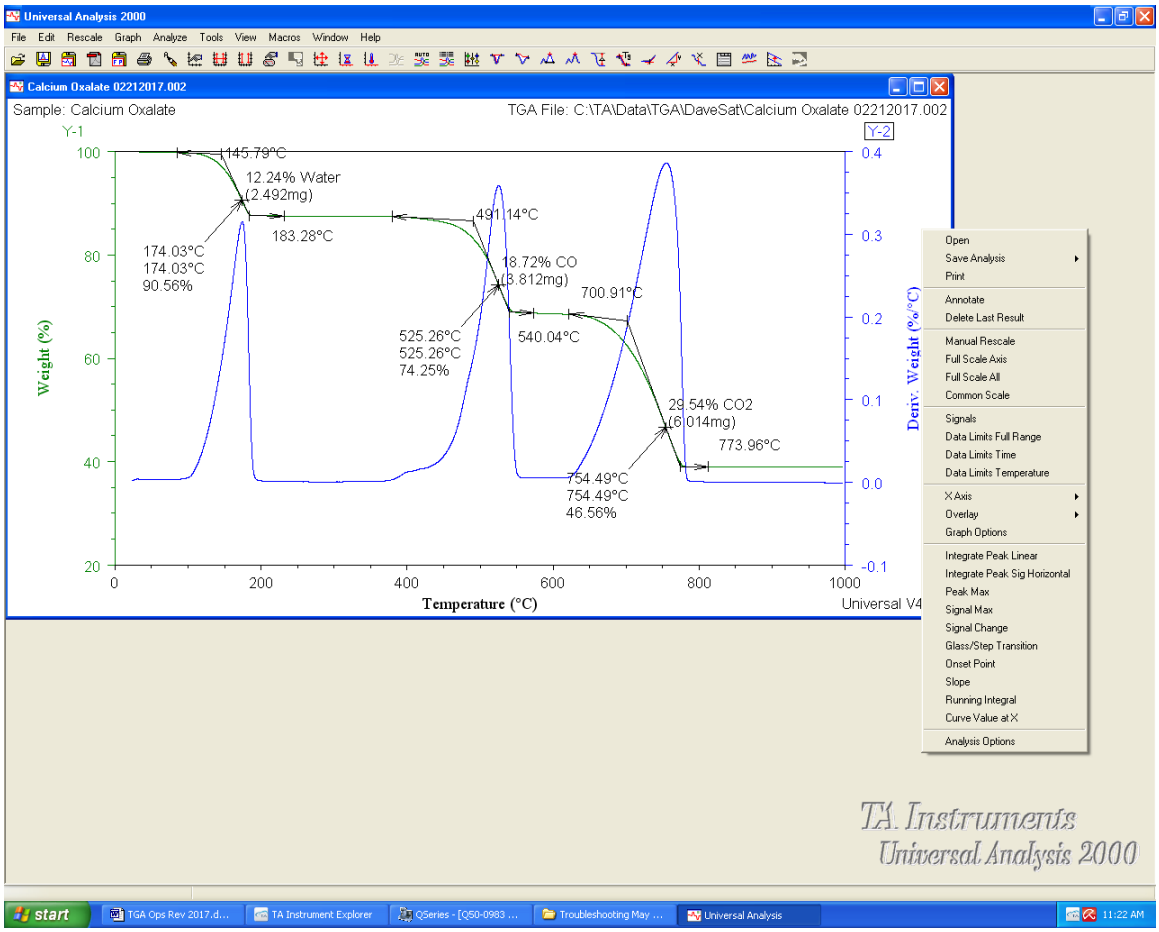
Universal Analysis 2000
File Edit Rescale Graph Analyze Tools View Macros Window Help
Calcium Oxalate 02212017.002
Sample: Calcium Oxalate
TGA File: C:\TA\Data\TGA\DaveSat\Calcium Oxalate 02212017.002
Y-1
Y-2
145.79°C
12.24% Water
(2.492mg)
183.28°C
491.14°C
18.72% CO
(3.812mg)
700.91°C
174.03°C
174.03°C
90.56%
525.26°C
525.26°C
74.25%
540.04°C
29.54% CO2
(6.014mg)
773.96°C
754.49°C
754.49°C
46.56%
Weight (%)
Deriv. Weight (%/°C)
Temperature (°C)
Universal V4
Open
Save Analysis
Print
Annotate
Delete Last Result
Manual Rescale
Full Scale Axis
Full Scale All
Common Scale
Signals
Data Limits Full Range
Data Limits Time
Data Limits Temperature
X Axis
Overlay
Graph Options
Integrate Peak Linear
Integrate Peak Sig Horizontal
Peak Max
Signal Max
Signal Change
Glass/Step Transition
Onset Point
Slope
Running Integral
Curve Value at X
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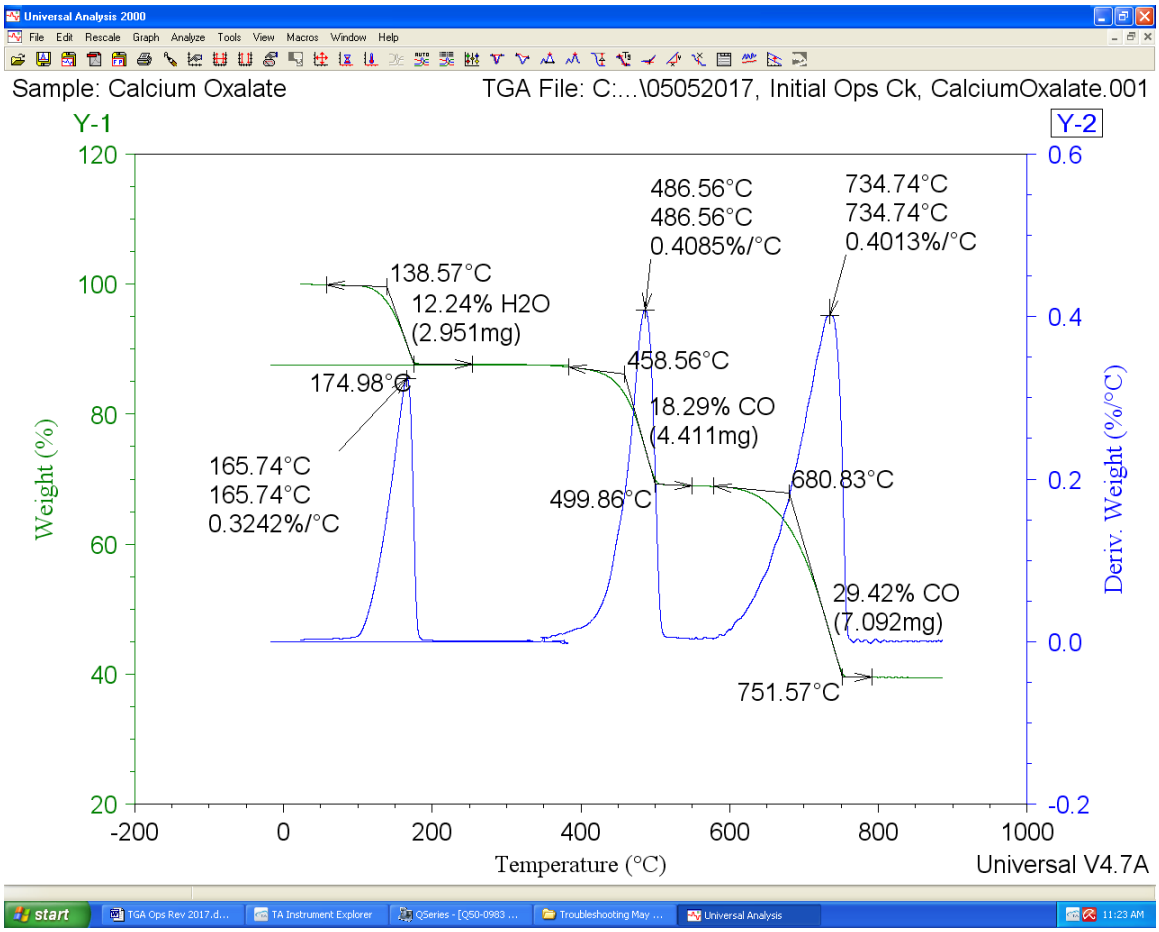
Universal Analysis 2000
File Edit Rescale Graph Analyze Tools View Macros Window Help
Sample: Calcium Oxalate
TGA File: C:...\05052017, Initial Ops Ck, CalciumOxalate.001
Y-1
Y-2
120
100
80
60
40
20
0.6
0.4
0.2
0.0
-0.2
Weight (%)
Deriv. Weight (%/°C)
138.57°C
12.24% H2O
(2.951mg)
174.98°C
165.74°C
165.74°C
0.3242%/°C
486.56°C
486.56°C
0.4085%/°C
458.56°C
18.29% CO
(4.411mg)
499.86°C
680.83°C
734.74°C
734.74°C
0.4013%/°C
29.42% CO
(7.092mg)
751.57°C
-200
0
200
400
600
800
1000
Temperature (°C)
Universal V4.7A
start
TGA Ops Rev 2017.d...
TA Instrument Explorer
QSeries - [Q50-0983 ...
Troubleshooting May ...
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