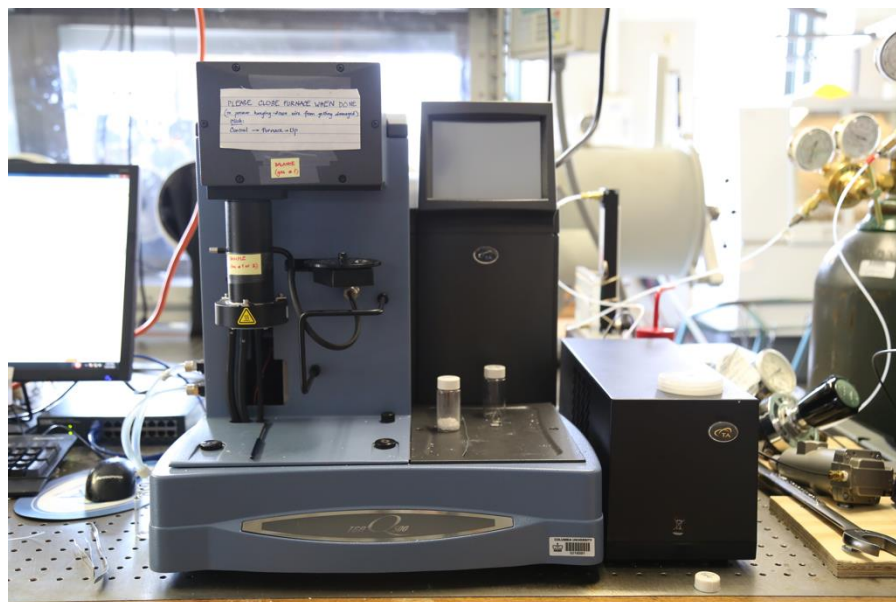




TA Instruments

Q500 Thermogravimetric Analyzer

Standard Operating Procedure



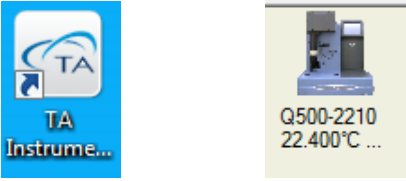
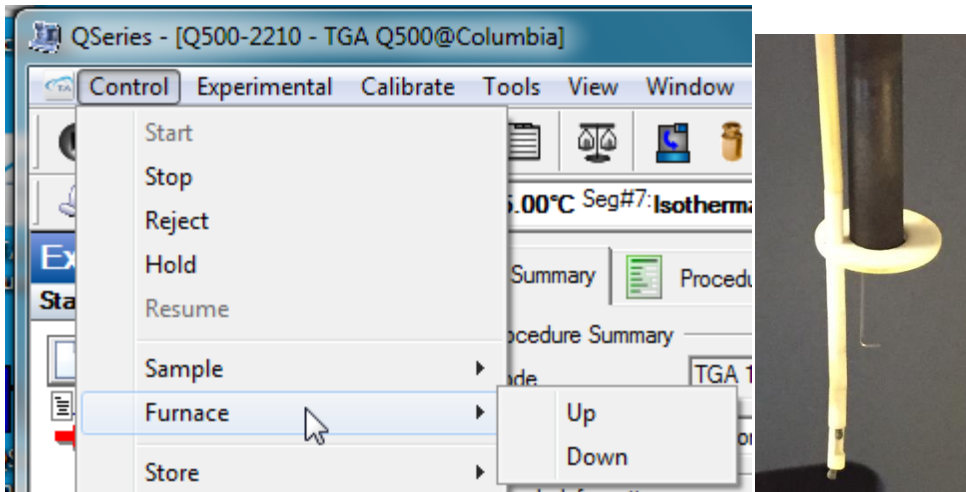
These instructions are intended for reference only, and will *not* replace the thorough training required for proper system operation. Contact staff with questions or to report a system problem.

SOP prepared by Thu Vi.


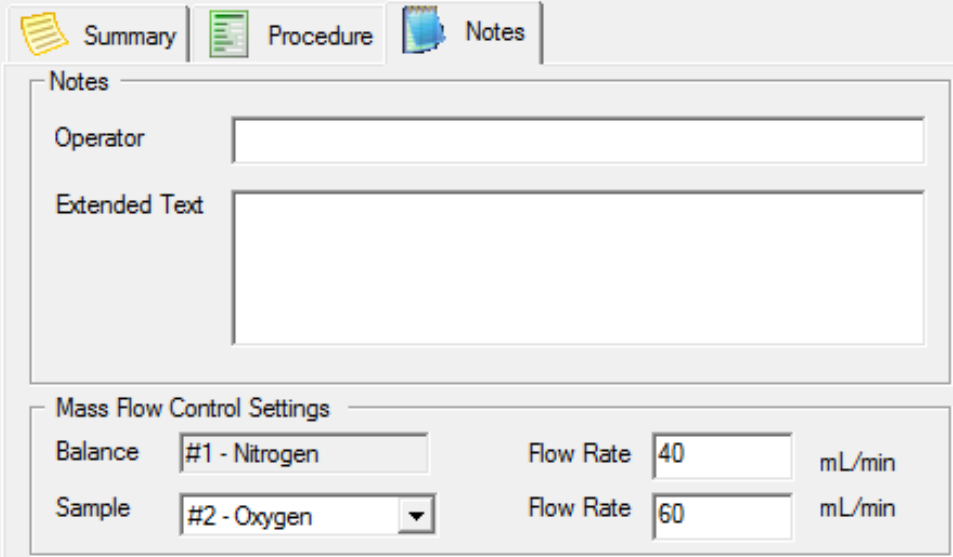
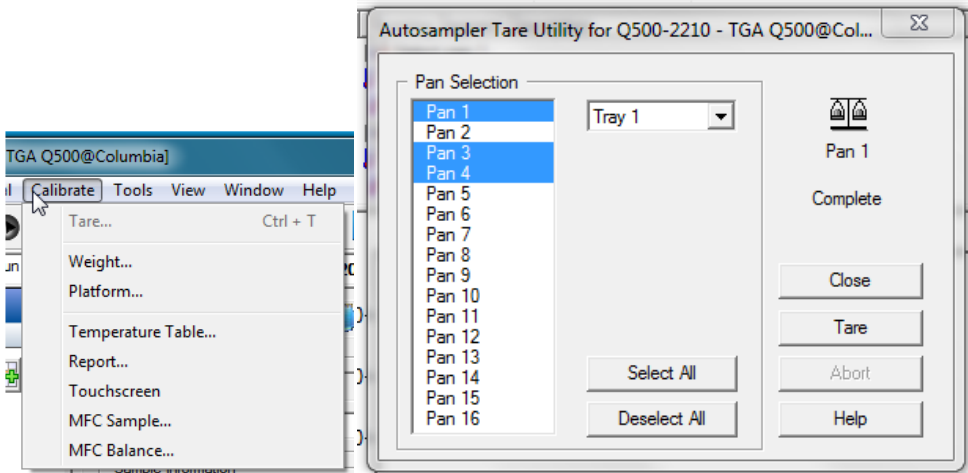


<p>1</p>	<p>Enable the instrument in Badger.</p>																								
<p>2</p>	<p>Fill out the logbook and complete all the information before starting experiment entries (SOP steps 5-8).</p>	<table border="1"> <thead> <tr> <th rowspan="2">Out</th> <th rowspan="2">Gases Used (N2, O2, CO2)</th> <th rowspan="2">Max. Temp. (Celcius)</th> <th colspan="2">Before Starting Experiment:</th> <th rowspan="2">Is sample pan in good shape?</th> <th rowspan="2">After E (Are gas turned, Are bo flows a)</th> </tr> <tr> <th>Balance</th> <th>Sample</th> </tr> </thead> <tbody> <tr> <td> </td> <td> </td> <td> </td> <td> </td> <td> </td> <td> </td> <td> </td> </tr> <tr> <td> </td> <td> </td> <td> </td> <td> </td> <td> </td> <td> </td> <td> </td> </tr> </tbody> </table>	Out	Gases Used (N2, O2, CO2)	Max. Temp. (Celcius)	Before Starting Experiment:		Is sample pan in good shape?	After E (Are gas turned, Are bo flows a)	Balance	Sample														
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<p>3</p>	<p>Ensure gas cylinders are closed.</p>																								
<p>4</p>	<p>Check that the sample pan is not bent or damaged. If there is a problem, contact the staff/superuser.</p>																								

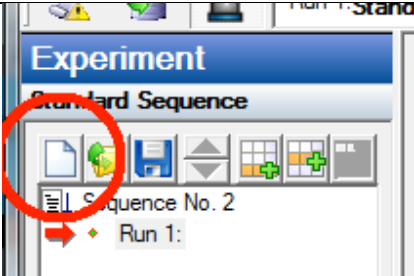
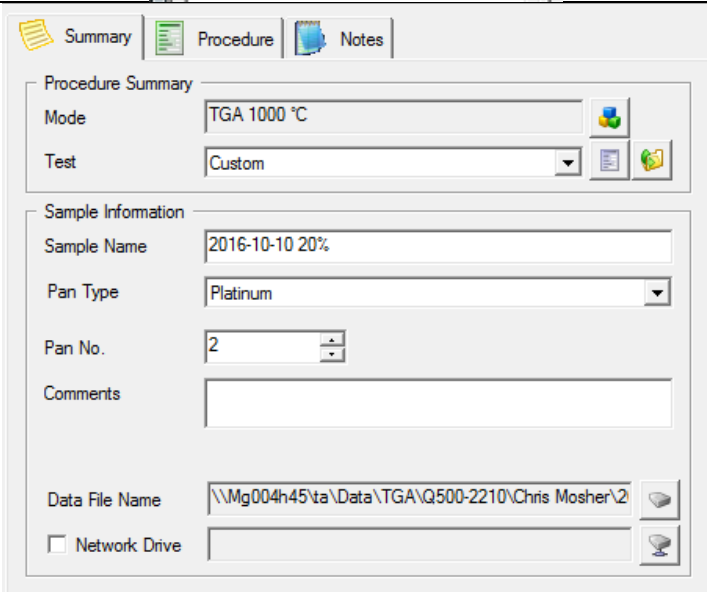
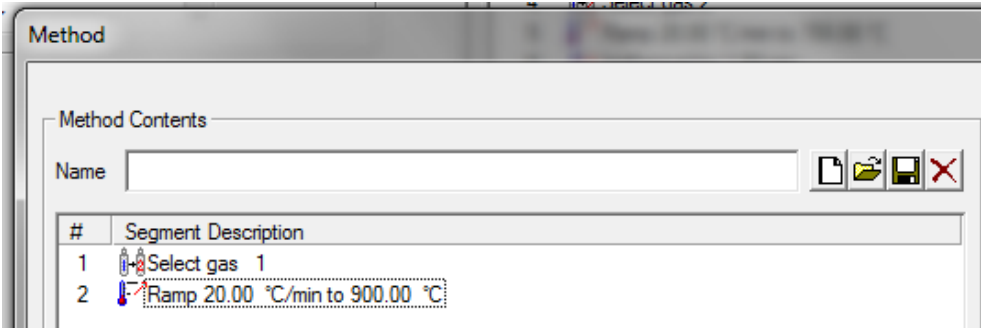


<p>5</p> <p>Open TA Instrument Explorer. Click on TGA logo. TA Software should open.</p>																																			
<p>6</p> <p>Sample Purge Flow and Balance Purge Flow should read 0 mL/min. You may have to dismiss some errors about the flow rate.</p>	<table border="1"> <thead> <tr> <th>Signal</th> <th>Value</th> </tr> </thead> <tbody> <tr><td>Method Time</td><td>0.00 min</td></tr> <tr><td>Segment Time</td><td>0.00 min</td></tr> <tr><td>Remaining Run Time</td><td>0 min</td></tr> <tr><td>Temperature</td><td>22.24 °C</td></tr> <tr><td>Weight</td><td>-44.2649 mg</td></tr> <tr><td>Weight percent.</td><td>100.00 %</td></tr> <tr><td>Balance Purge Flow</td><td>0.07 mL/min</td></tr> <tr><td>Sample Purge Flow</td><td>0.00 mL/min</td></tr> <tr><td>Set Point Temp</td><td>0.00 °C</td></tr> <tr><td>Heater Power</td><td>0.00 W</td></tr> <tr><td>Modulation Period</td><td>0.0 s</td></tr> <tr><td>Modulation Amplitude</td><td>0.00 °C</td></tr> <tr><td>Temp Amp (+/-)</td><td>0.00 °C</td></tr> <tr><td>Modulated Weight Amplitude</td><td>0.0000 mg</td></tr> <tr><td>Avg Derivative Weight</td><td>0.0000 mg/min</td></tr> <tr><td>Deriv Weight Amplitude</td><td>0.0000 mg/min</td></tr> </tbody> </table>	Signal	Value	Method Time	0.00 min	Segment Time	0.00 min	Remaining Run Time	0 min	Temperature	22.24 °C	Weight	-44.2649 mg	Weight percent.	100.00 %	Balance Purge Flow	0.07 mL/min	Sample Purge Flow	0.00 mL/min	Set Point Temp	0.00 °C	Heater Power	0.00 W	Modulation Period	0.0 s	Modulation Amplitude	0.00 °C	Temp Amp (+/-)	0.00 °C	Modulated Weight Amplitude	0.0000 mg	Avg Derivative Weight	0.0000 mg/min	Deriv Weight Amplitude	0.0000 mg/min
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<p>7</p> <p>- On top left corner, click Control -> Furnace -> Down. Furnace should come down. Check if the hanging down wire and the thermocouple are straight. Stop if the thermocouple or hanging down wire are not straight. Contact the staff/superuser right away.</p>																																			



<p>8</p>	<p>Once all the conditions in the Before Measurement Requirement are met, open gas cylinders: N₂, O₂ or CO₂) and place a clean, empty sample pan on the carousel.</p>	
<p>9</p>	<p>In Notes tab, set the Sample gas. (Balance gas is always nitrogen.) The flow rates should be 40 and 60 mL/min.</p>	
<p>10</p>	<p>Click Calibrate -> Tare. Choose the correct sample pan (you can tare several at once). Wait for the taring process to finish.</p> <p>If the instrument fails to pick up a pan, it will try a second time.</p> <p>Wait for the second attempt to complete</p>	



	<p>before you make any adjustment of the pan.</p>	
<p>11</p>	<p>Click on New Sequence. Say "No" to "Do you want to save the last sequence?"</p>	
<p>12</p>	<p>Under Summary tab: - Chose "Custom" for Method. - Enter Sample Name and Pan Number used. - Choose where to save the sample file.</p>	
<p>13</p>	<p>Under Procedure tab, click on Editor to set up Procedure. - Gas selection must be the first step. (Gas 1 is nitrogen; gas 2 is oxygen or any other alternative.) - Add segments by double-clicking in the right panel and adjusting parameters as needed.</p>	

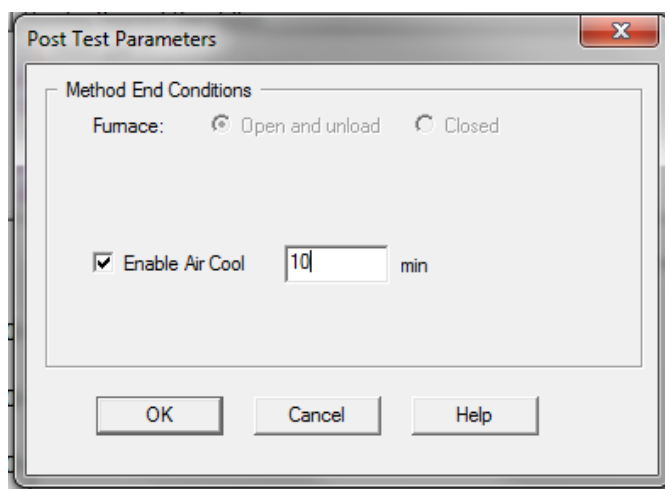


- Click Save logo to save procedure.
Consult the superuser if you are unsure of your experiment setup. Maximum temperature is 1100°C but please try not to go over 1000°C if possible. Also keep the total run time below 3 hours if running at high temperature (900 to 1000 °C).

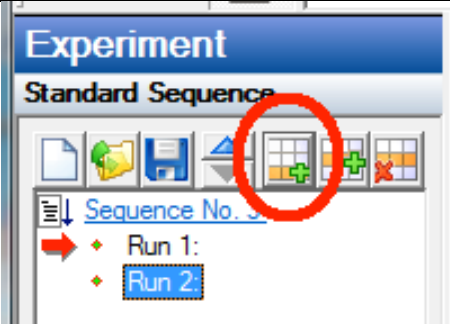

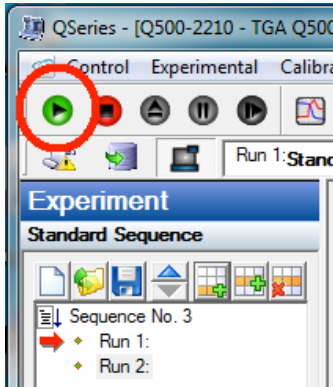
14

Still in Procedure tab, click on Post Test. This will set up an air flow to cool down the furnace after the run. This is important when performing multiple runs.

To help estimate the correct cooldown time: 10 min is sufficient to cool from 900 °C to 50 °C. Adjust accordingly if you are cooling down




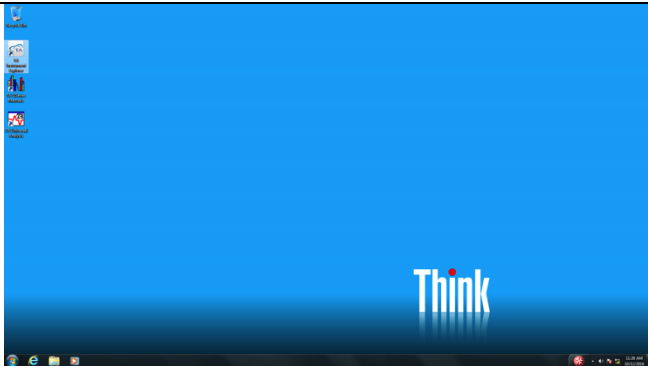



	<p>from a higher or lower temperature.</p>	
<p>15</p>	<p>To set up multiple runs, click Add Run and repeat steps 12-14 for each run.</p>	
<p>16</p>	<p>Ensure that the tare procedure is finished, remove the pan(s) from the carousel and load sample onto pan(s). Do not load the pans while they are on the carousel. Use 5-15 mg samples. Load powdered samples in the fume hood. Clean up after yourself!</p>	
<p>17</p>	<p>After the run is set and the sample is loaded, click Start Run. Watch to make sure sample pan is loaded properly and the running man logo appears next to the run.</p>	



<p>18</p>	<p>Once the run is finished, the sample might be unloaded automatically. If not, click Control -> Sample -> Unload.</p>																																			
<p>19</p>	<p>Turn off gas tank used. WAIT UNTIL GAS FLOWS IN THE PROGRAM REACH 0 mL/min. You can analyze your data while you wait.</p>	<table border="1"> <thead> <tr> <th>Signal</th> <th>Value</th> </tr> </thead> <tbody> <tr> <td>Method Time</td> <td>0.00 min</td> </tr> <tr> <td>Segment Time</td> <td>0.00 min</td> </tr> <tr> <td>Remaining Run Time</td> <td>0 min</td> </tr> <tr> <td>Temperature</td> <td>22.34 °C</td> </tr> <tr> <td>Weight</td> <td>-44.2649 mg</td> </tr> <tr> <td>Weight percent.</td> <td>0.00 %</td> </tr> <tr> <td>Balance Purge Flow</td> <td>0.09 mL/min</td> </tr> <tr> <td>Sample Purge Flow</td> <td>0.00 mL/min</td> </tr> <tr> <td>Set Point Temp</td> <td>0.00 °C</td> </tr> <tr> <td>Heater Power</td> <td>0.00 W</td> </tr> <tr> <td>Modulation Period</td> <td>0.0 s</td> </tr> <tr> <td>Modulation Amplitude</td> <td>0.00 °C</td> </tr> <tr> <td>Temp Amp (+/-)</td> <td>0.00 °C</td> </tr> <tr> <td>Modulated Weight Amplitude</td> <td>0.0000 mg</td> </tr> <tr> <td>Avg Derivative Weight</td> <td>0.0000 mg/min</td> </tr> <tr> <td>Deriv Weight Amplitude</td> <td>0.0000 mg/min</td> </tr> </tbody> </table>	Signal	Value	Method Time	0.00 min	Segment Time	0.00 min	Remaining Run Time	0 min	Temperature	22.34 °C	Weight	-44.2649 mg	Weight percent.	0.00 %	Balance Purge Flow	0.09 mL/min	Sample Purge Flow	0.00 mL/min	Set Point Temp	0.00 °C	Heater Power	0.00 W	Modulation Period	0.0 s	Modulation Amplitude	0.00 °C	Temp Amp (+/-)	0.00 °C	Modulated Weight Amplitude	0.0000 mg	Avg Derivative Weight	0.0000 mg/min	Deriv Weight Amplitude	0.0000 mg/min
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<p>20</p>	<p>Make sure all sample pans are removed from furnace and carousel. Click Control -> Furnace -> Up to close furnace.</p>																																			



<p>21</p>	<p>To analyze data, open TA Universal Analysis software on Desktop. See the brief Data Processing SOP for further details.</p>															
<p>26</p>	<p>Ensure gas flows are at 0 mL/min. Close TA Instrument Explorer and TA Universal Analysis Software.</p>															
<p>27</p>	<p>Fill out log book for the After Experiment requirements.</p>	<table border="1"> <thead> <tr> <th colspan="4">After Experiment:</th> <th rowspan="2">Comment</th> </tr> <tr> <th>an</th> <th>Are gas tanks turned off? (Are both gas flows at 0?)</th> <th>Is sample taken out?</th> <th>Is furnace closed?</th> </tr> </thead> <tbody> <tr> <td> </td> <td> </td> <td> </td> <td> </td> <td> </td> </tr> </tbody> </table>	After Experiment:				Comment	an	Are gas tanks turned off? (Are both gas flows at 0?)	Is sample taken out?	Is furnace closed?					
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<p>28</p>	<p>Log off from personal account on TGA Computer.</p>															
<p>29</p>	<p>Disable the instrument in Badger.</p>	