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Technical Guidance on Use of the Linseis Vertical Dilatometer L75

Gretchen K. Toney
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April 2022

OAK RIDGE National Laboratory

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Radioisotope Science and Technology Division

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OAK RIDGE NATIONAL LABORATORY
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TABLE OF CONTENTS

FIGURES

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ABSTRACT

Linseis vertical Dilatometer L75 thermal expansion equipment is a complete system which can be used for measuring the change of the absolute length of a sample, determination of the thermal expansion coefficient (CTE), shrinkage, and sintering. The ^{238}Pu Supply Program is interested in re-establishing the capability to determine CTE and measuring the change of the absolute length of various ^{237}Np Al/cermet samples during heating. The change in length of the measured sample is recorded using a highly sensitive Linear Variable Differential Transformer (LVDT). The dilatometer is user-friendly and simple to operate which will allow technicians to be trained easily on its use. This equipment was designed to be used specifically for ceramics. It has a highly vacuum tight push rod dilatometer, a high-resolution inductive transducer using innovative and comprehensive temperature controls which provide excellent performance as a measurement system yielding high accuracy, reproducibility, and long-term stability.

1. VERTICAL DILATOMETER L75 INSTRUMENT INSTALLATION

1.1 L75 INSTRUMENT INSTALLATION

1.1.1 Equipment Set-up

It is important to identify a good working space for the L75 dilatometer. The area it is placed in should be stable and the area should minimize mechanical vibrations, including vibrations from its own operation. Other information pertaining to the equipment set-up should be taken into consideration as followed:

- The dilatometer should be placed in a location with access to the following: power, cooling water, and gas outlets. Access to these features allow full use of the dilatometer. Ideally the dilatometer should be located in an area with a fairly constant temperature that doesn't suffer from drastic temperature fluctuations while the instrument is running.

- The dilatometer requires a USB cable connection to the computer where the dilatometer data is collected. It is best to have the computer/monitor on a separate worktable.

- The water chiller and power supply should be placed out of the way of the dilatometer such as under the table or workspace.

- If the dilatometer needs to be transported place a vacuum clamp over the flanges at the separation point between the flanges and the furnace lift point.

- The vacuum clamp must be removed prior to operation, or the furnace element will be damaged.

- There is also foam installed in the measuring system for transportation purposes which must be removed prior to operation.

- Connection of the cooling water system uses two hoses which are color coded red and blue and need to be connected properly to ensure the water flow loop between the chiller and dilatometer.

- Water hose set up should follow the diagram in Figure 1 below. Instrument water flow “out” connects to water chiller flow “in” and water chiller flow “out” connect to instrument water flow “in”.

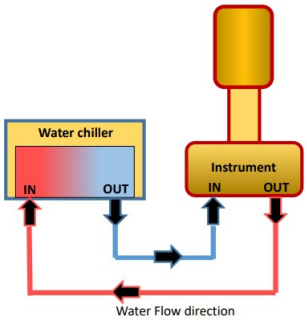


Figure 1. Water chiller hook-up.

1.1.2 Measuring System Installation

The measuring system is made up of the measuring system tube, the pushrod, S-type thermocouple, fish hose, and a reference sample. To install the measuring system the following steps should be completed:

1. Raise and move the furnace out of the way.

2. Ensure the vacuum clamp has been removed, this is a very important step to ensure the furnace element does not get damaged.

3. The thermocouple needs to be secured low on the measuring system tube out of the furnace hot zone.
   - Lay the S-type thermocouple on the tube first then attach it in place being careful not to bend and break the thermocouple.

4. The thermocouple’s tip should be positioned on the right-hand side of the sample window of the measuring system tube.

5. Look inside the top of the measuring system flange to identify the center hole (which is where the measuring system tube passes through), and the right-hand side opening is where the thermocouple wires need to pass through.

6. Insert the ball end of the pushrod into the top of the measuring system and open the clamp screw to allow the ball to slip in the clamp.

7. The clamp screw should be tightened to hold it in position but not to secure it.

8. Check that the pushrod can be moved left to right but does not flop around on its own.

9. Connect the thermocouple wires to the terminal connector.

10. Check to see the polarity of the thermocouple wires is correct by adding heat to the tip of the thermocouple.
    - If the temperature increases, then the polarity is correct.
    - If the temperature decreases, then the wires need to be switched on the terminal block.

11. Gently center the push rod and tighten the screw to make sure the pushrod is only touching the ball connection.
    - Check the placement by displacing the pushrod by a few millimeters and watch the pushrod to make sure it bounces.

12. Center the furnace back over the measuring system and begin to lower the furnace while visually inspecting the alignment to ensure the measuring system does not touch the furnace flange when lowered.

2. VERTICAL DILATOMETER L75 SAMPLE REQUIREMENTS

2.1 SAMPLE PREPARATION

2.1.1 Sample Dimension Limits

The L75 dilatometer detects the change in length of a sample that is exposed to a controlled temperature by the furnace. The samples need to have flat parallel ends for proper measurement. The samples must not exceed the 7 mm inner diameter of the sample holder. The sample length should not exceed a maximum length of 50 mm.

3. EXPANSION ARTIFACT REMOVAL IN VERTICAL DILATOMETER L75

3.1 CORRECTION FILE

3.1.1 Purpose of the Correction File

In dilatometry the expansion artifact of the sample holder needs to be removed to have only the actual expansion of the sample. The L75 Linseis dilatometer is set up to place the sample between the pushrod and the tube which are both parts of the measuring system. The pushrod and the tube exit the furnace and pass through the chiller collar and enter the thermal mass stabilizer which means the pushrod and tube exhibit a temperature gradient between the furnace and the thermal mass stabilizer. The thermal mass stabilizer maintains a temperature of 35ºC to ensure room temperature fluctuations do not affect the measuring system. The displacement of the pushrod end is measured inside the thermal mass stabilizer at the LVDT.

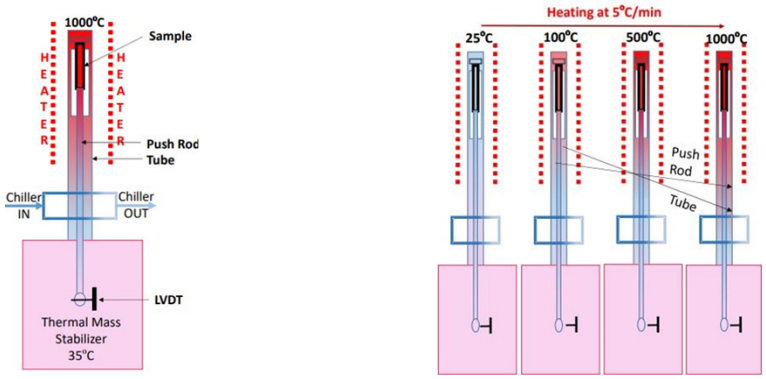


Figure 2. Sample set-up and thermal gradient of pushrod and tube as temperature increases.

As the system increases the temperature during a run, it is important to note the thermal gradient of both the pushrod and tube varies over the length of each piece. At any position down the length of the pushrod and the measuring tube the temperature gradient may not be the same and it is strongly dependent on the heating rate of the furnace. The pushrod displacement that is measured at the LVDT represents the expansion of the tube which is made up of the sample and the pushrod. Therefore, the sample displacement measurement is only accurate if the contributions made by the pushrod and tube are identified as a function of time and temperature.

Using a quartz reference material provides a way to identify the quartz pushrod and tube expansion contributions in the LVDT measurement. When the reference and the measuring system are the same material (quartz) the LVDT measurement should be zero, but this is not the case due to the unequal rating of the components. A correction run of the quartz reference material creates the set of correction factors as a function of time and temperature to identify the real sample expansion.

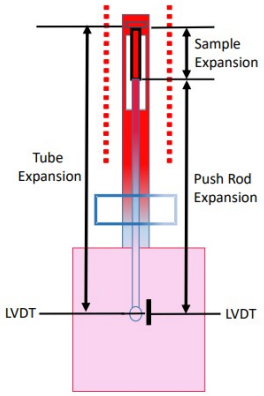


Figure 3. Thermal expansion of sample, pushrod, and tube.

4. RUNNING THE VERTICAL DILATOMETER L75

4.1 DATA COLLECTION AND EVALUATION

4.1.1 Quartz Correction Run

Linseis software is a comprehensive user-friendly suite of tools which supports the measurement functions through a program called Dilatometer and the analysis functions through a program called Evaluation. The reference material should be the same size as the sample + or – 5 mm in length. The heating and cooling profile that is created for the sample run must also be the same for the correction run.

1. Open the dilatometer program to create a correction file for a quartz sample.

Figure 4. Measurement icon.

2. Select OK when the Linseis TA Software Acquisition window appears. Username is Admin and no password is necessary.

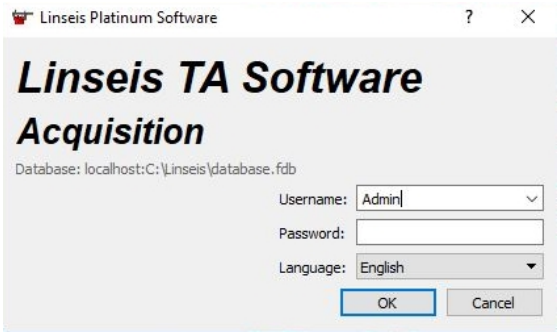


Figure 5. Acquisition window.

3. Check the top left corner of the screen displays L75 PT 1000 and the word Demo in brackets is not present.

Figure 6. Demo not present vs demo present.

- If the word Demo appears in the top left corner, then double check the chiller and dilatometer to make sure they both are turned on and connected to the computer via USB connection.

- If the USB connection between the equipment and the computer is correct and there are still issues, then check if another Dilatometer Program window is open and running. This Dilatometer program window will need to be closed before moving forward with the data collection set up.

- Generally, if there is an issue with the USB connection the following window will appear:

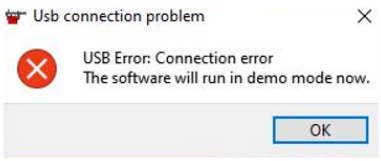


Figure 7. USB error window.

- After clicking OK the Demo mode warning window will appear.

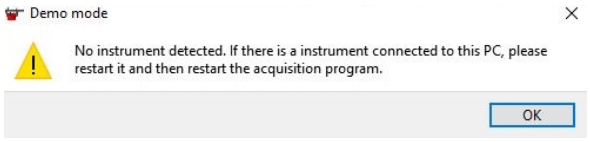


Figure 8. Warning in demo mode.

4. The program should open to the setup page of the general settings tab.

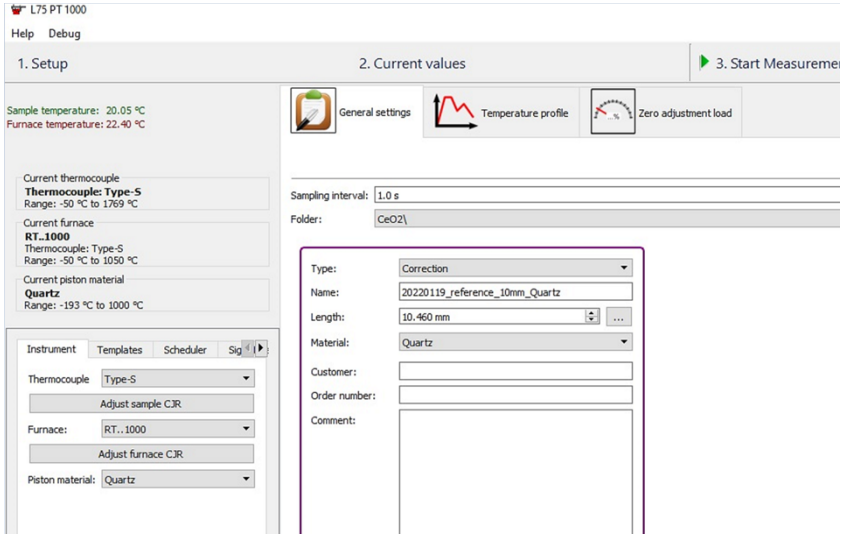


Figure 9. General settings tab in set-up.

5. On the general settings tab, select the data folder to store the sample information in from either the drop-down arrow or the … as seen below in Figure 10:

Figure 10. Data folder.

6. If you need to create a new folder select the … (Figure 10), once the select folder window appears, right click to create a new folder, enter the folder name, and click ok. The folder should be in the list. Select the folder and click ok.

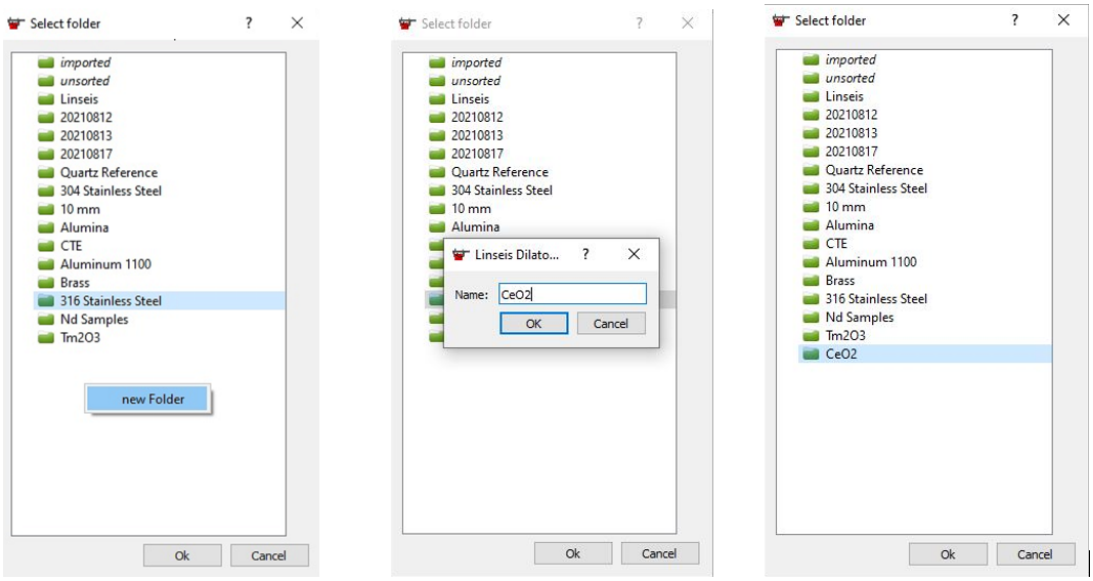


Figure 11. Create and name folder.

7. In the general settings tab of the setup page make sure to enter the type, name, length, and select the material from the drop-down box.

Select correction for the type from drop-down box.
Enter the name.
Enter the length in mm.
Select quartz as the material

- The length should be an average of 3 measured points on the sample.
- The length of the reference material should be +/- 5 mm of the sample material.

Figure 12. Correction curve settings information.

8. Select the temperature profile tab in the setup page.

Figure 13. Temperature profile.

- Use the same temperature profile for the correction run and the sample run. Select the same rate, temperature, and dwell time for both the heating and cooling segments.

- Line 1 in the example above is the heating segment and line 2 is the cooling segment with a room temperature dwell time of 200 minutes.

- The room temperature dwell time is needed to allow the instrument to cool to room temperature.

- Ensure enough time to allow the sample to cool to room temperature particularly if samples are running through the scheduler.

- At low temperatures the furnace will cool slower than the programmed rate.
- If the room temperature dwell is not added, then no data will be collected the whole time the furnace is cooling.
- Profiles can be manipulated by using the buttons on the right side: add segment, insert segment, and delete segment.

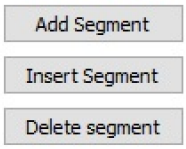


Figure 14. Temperature segment buttons.

9. Once the temperature profile has been created select save on the lower right side. Enter the name and any description for the newly made profile and click save.

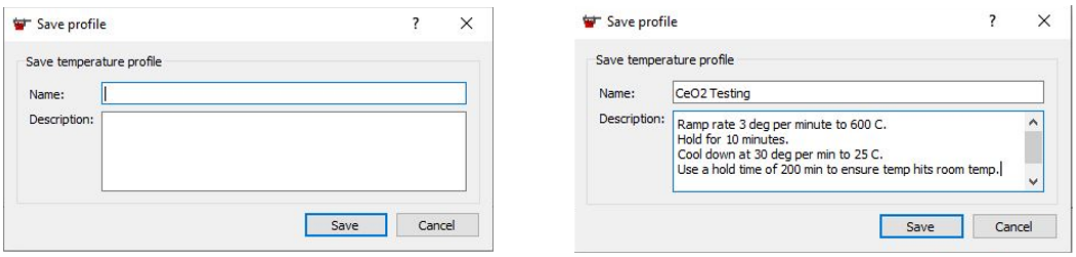


Figure 15. Save profile add name and description.

10. To select a previously used temperature profile, click on load and then when the profile administration window appears select the desired profile and click ok.

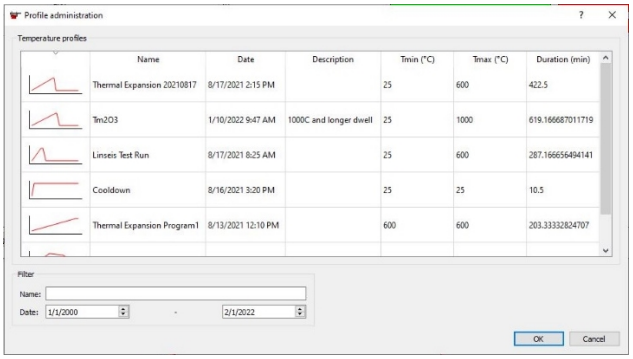


Figure 16. Saved temperature profiles.

11. Select the scheduler tab in the left side of the screen.

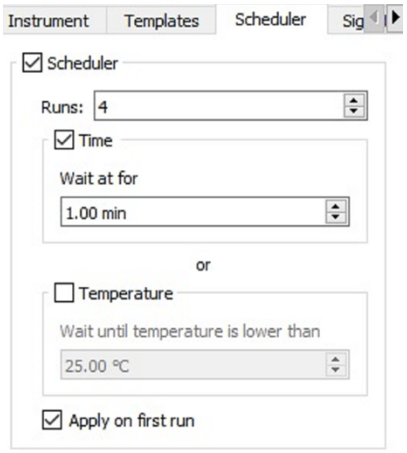


Figure 17. Scheduler.

- Use the scheduler to set up 4 repeat runs.
- Four runs are suggested to ensure a good run-to-run repeatability.
- The data will be saved in separate data files for each repeat run.
- Select either time or temperature, but not both.
- Time or temperature is used as the start trigger for the next scheduled run.
- If the dwell was programmed in the cooling segment, then select the time option and set it to 1.00 min.
- The temperature option should only be used if room temperature does not vary throughout the day.
  - If a low room temperature is entered, then the furnace may never get to the triggered temperature to run the next sample.
  - If a high room temperature is entered, then the sample may never reach the room temperature expansion value before the next run starts.

12. Select the zero adjustment load tab in the setup page, see Figure 18, the zero adjustment load tab is in the upper right corner of the pop-up window.

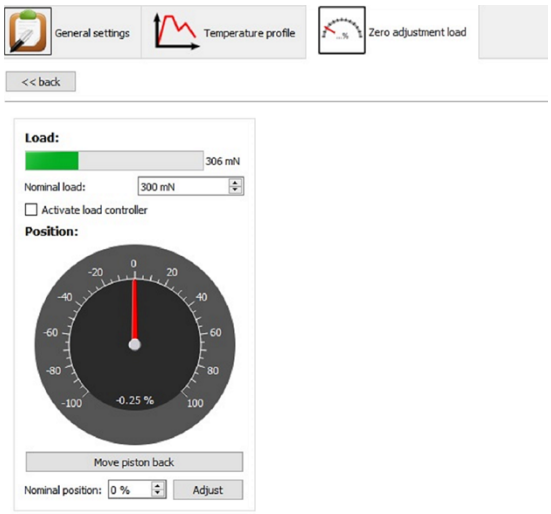


Figure 18. Zero adjustment load tab.

13. Raise the furnace on the dilatometer's touch screen page 3, image of the touch screen is in Figure 19. Press the up arrow on the right above the "Furnace Lift". Lower the push rod using the same screen. Press the down arrow on the left below the "Push Rod DIL1".

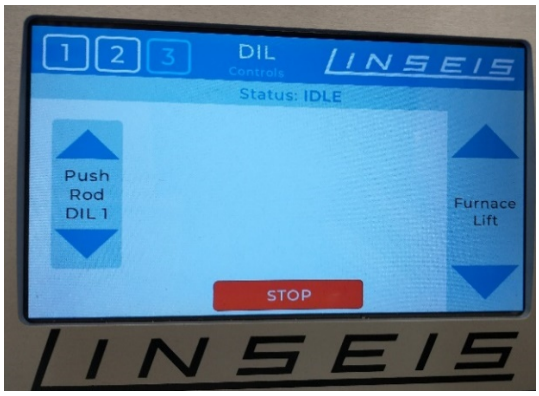


Figure 19. Dilatometers touch screen page 3.

14. Displace the push rod manually to see if the zero adjustment load's red dial moves properly, before loading the sample, to confirm the computer is communicating with the LVDT.

15. Before loading and unloading a sample ensure the activate load controller box is not selected, see Figure 20.

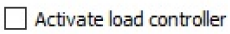


Figure 20. Load controller.

16. Load a quartz sample onto the push rod, see Figure 21.

Figure 21. Sample.

17. Verify the nominal load entered is 300 mN on the zero-adjustment load tab, see Figure 22.

Figure 22. Nominal load.

18. Raise the push rod on the dilatometer's touch screen until the position is reading -10% to -15%. A typical pushrod read out is shown in Figure 23.

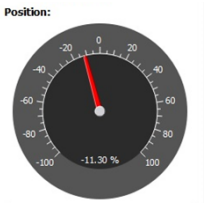


Figure 23. Pushrod at -11.30 %.

19. Click the adjust button to zero out the LVDT. The needle will adjust to the new % position (close to 0%). A typical zeroed pushrod read out is shown in Figure 24.

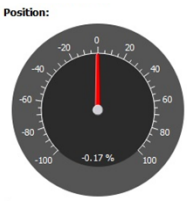


Figure 24. Pushrod adjusted.

20. Select the start measurement green side arrow to start the run and collect the data. See Figure 25 for how this should look.

Figure 25. Start dilatometer.

- Verify the scheduler has started, by looking under the setup tab and finding the word scheduler highlighted in a yellow box, see Figure 26 for how this should look.

Figure 26. Started scheduler.

- Take note of the room temperature for the sample and furnace (Figure 26).

- Verify the measurement has started, the word scheduler will be replaced by the word measurement highlighted in a red box, see Figure 27.

Figure 27. Started measurement.

- The sample temperature and the furnace temperature should be increasing (Figure 27).

21. Select the current values tab to view the measurement in progress. Figure 28 is an example of the measurement progress.

    - The whole temperature profile and the corrected profiles may be viewed by selecting the boxes in the upper right corner. The temperature, temperature profile, and length are color coded.

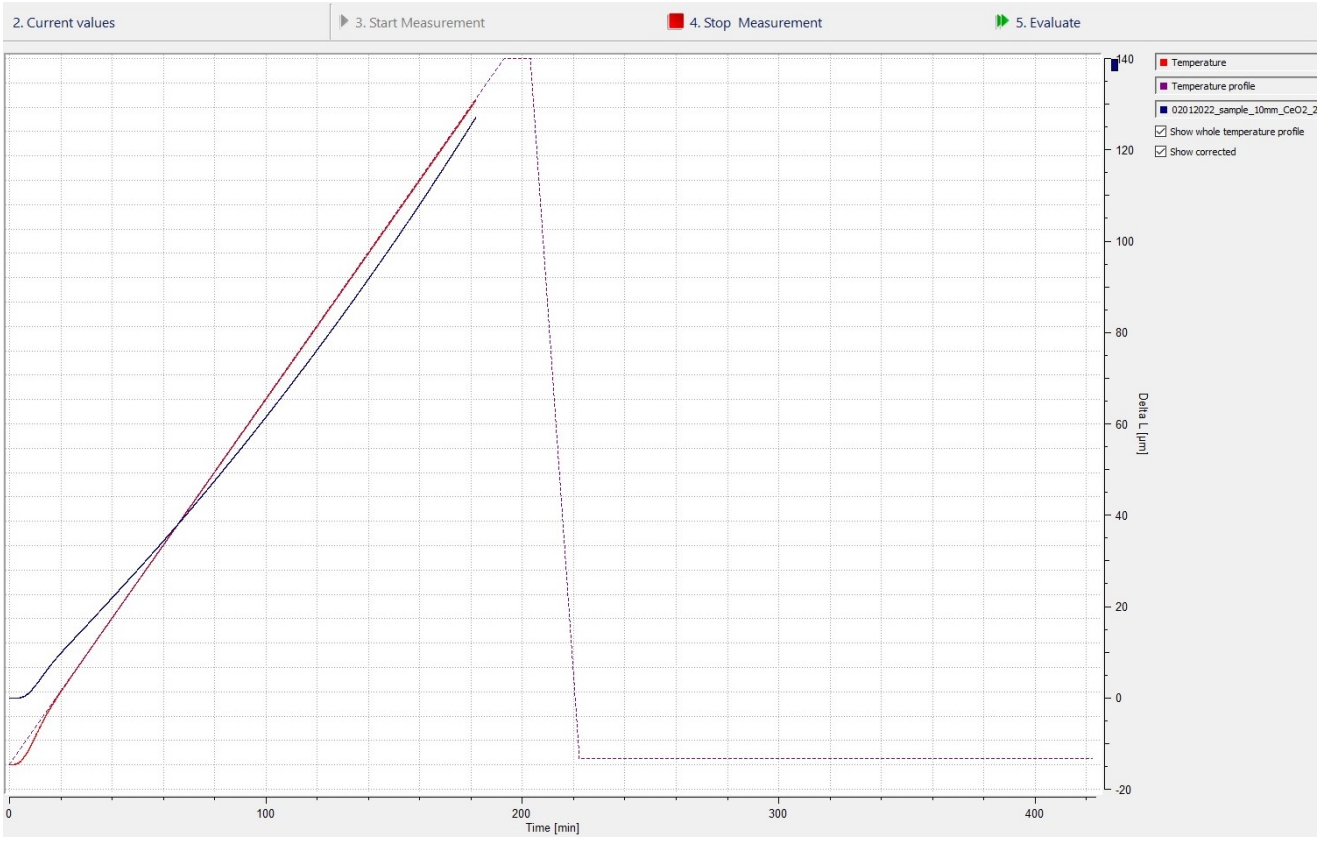


Figure 28. Current values page.

- Remember the expansion measured at the LVDT is the net sum of the reference sample, the push rod, and the tube expansion. The expansion vs time/temperature profile may appear odd with several up and down inflections.

4.1.2 Create a Mean Correction File using Quartz Reference

1. Open the evaluation program (see Figure 29) to analyze the correction curves for the quartz sample.

Figure 29. Evaluation icon.

2. On the tool bar select the icon  to load a curve.

3. Select the curves that were run on the dilatometer under the quartz reference folder, use the Ctrl key to select multiple curves, and click OK. See Figure 30 for an example of multiple selections.

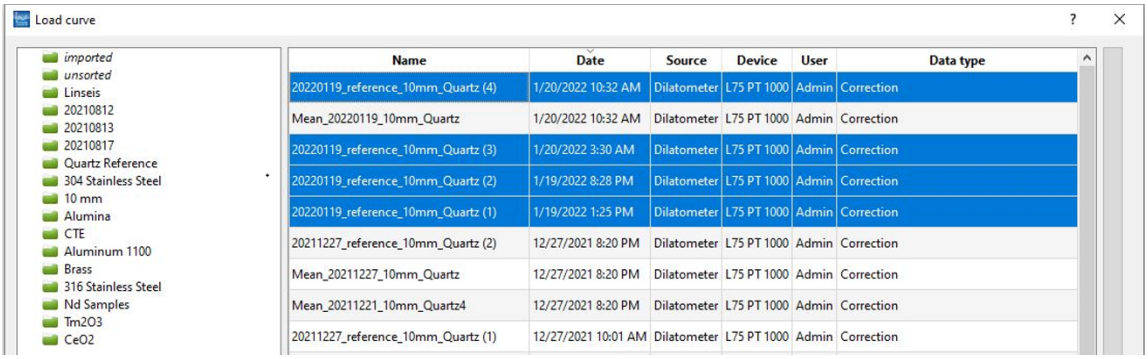


Figure 30. Loading reference curves.

- The channel selection window will appear, then select your x (Time) and y axis (Delta L) for each loaded curve. Click OK and the curves will be populated in the main screen.

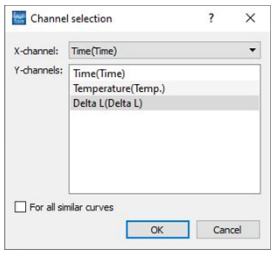


Figure 31. Select X and Y axis parameters.

4. Compare the data of the quartz correction runs to look for repeatability. See Figure 32.

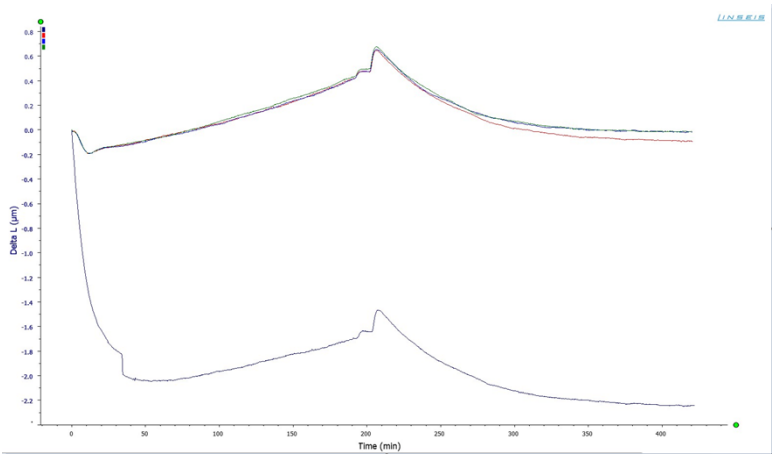


Figure 32. Quartz correction curve data

- Eliminate any curves which are outliers and are not to be included in the mean, notice the difference between Figure 32 and Figure 33. Figure 33 shows the deleted outlier curve. Delete the curves by first highlighting them in the elements window under the project name then right click and select delete.

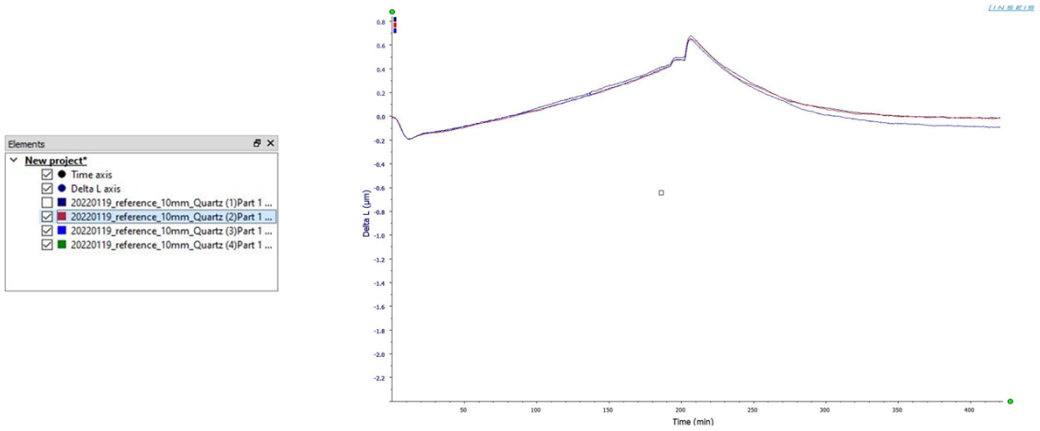


Figure 33. Curves listed in elements and deleted outlier.

5. Select a curve and separate the heating and cooling segments using the icon in the tool bar.
   - As needed adjust the visible axis to enlarge the graph with the icon in the tool bar.
   - Delete the cooling segment or deselect the cooling segment in the project to only show the heating segments for each curve, see Figure 34.

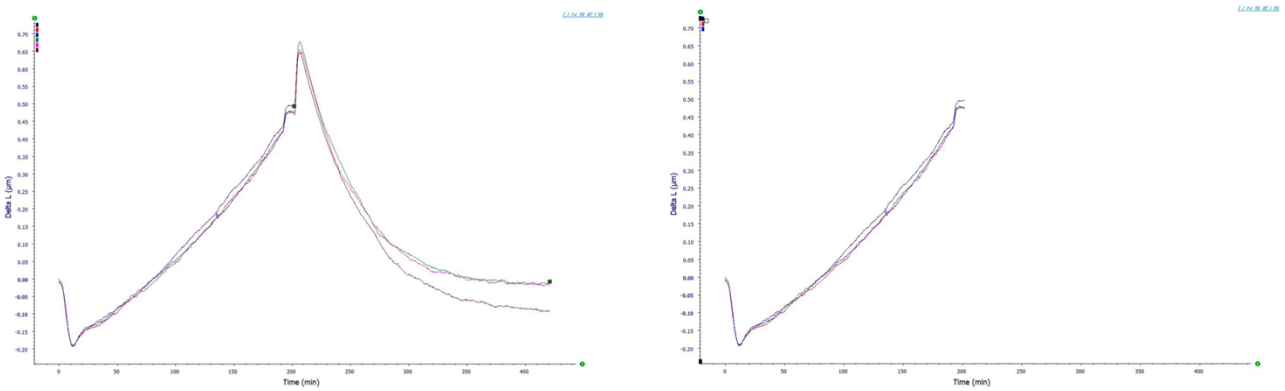


Figure 34. Curves separated into heating/cooling then cooling segments deleted.

6. Select all the correction curves and get the mean curve (see Figure 35) for those highlighted by using the icon  in the tool bar which evaluates the mean curve of a couple of curves.
   - The mean curve is now populated in a new color on the graph, and it has been automatically added to the project list under the elements window.

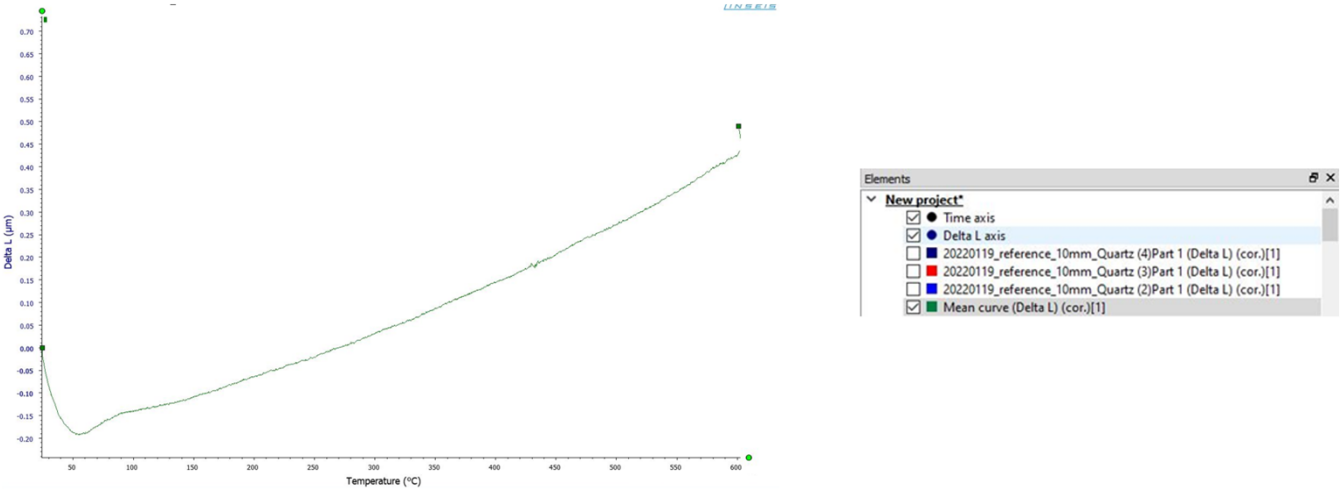


Figure 35. Mean curve.

7. Save the mean curve by highlighting it and right click to save as a measurement curve, see Figure 36.

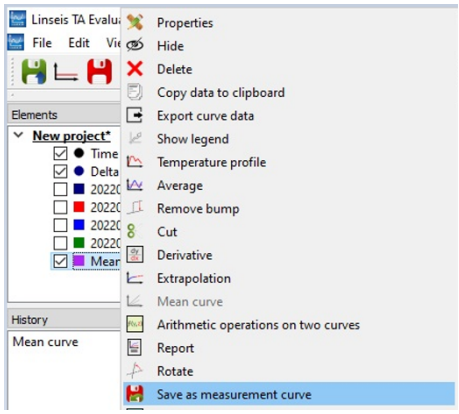


Figure 36. Save the mean curve.

- Enter a name for the curve and click OK, see Figure 37 left.
- Select the type of curve (in this case it is a correction), see Figure 37 right.
- Verify the curve has been saved by clicking ok on the pop-up window stating the curve was saved.

Figure 37. Name mean curve and select correction curve.

8. Save the project used to create the mean curve, by selecting the project under the elements window and clicking on save project, see Figure 38.

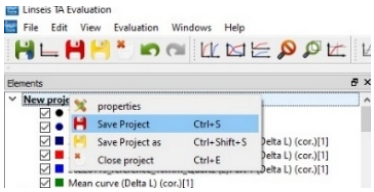


Figure 38. Save project.

- Select the folder to be used to save the project, next type the name of the project, and click OK, see Figure 39.

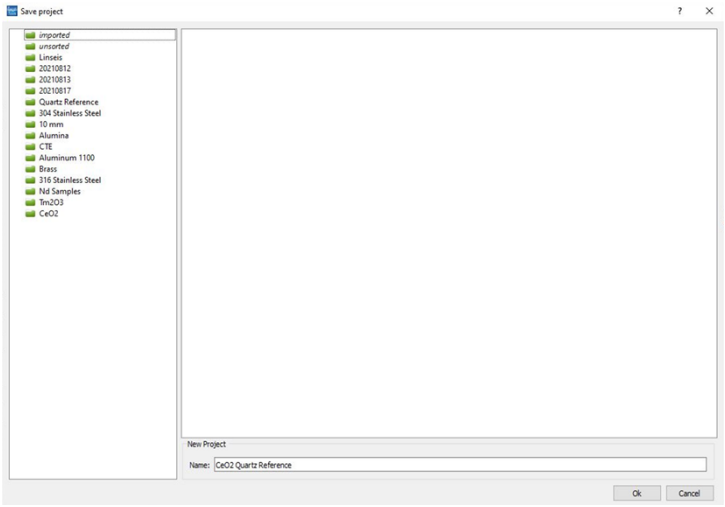


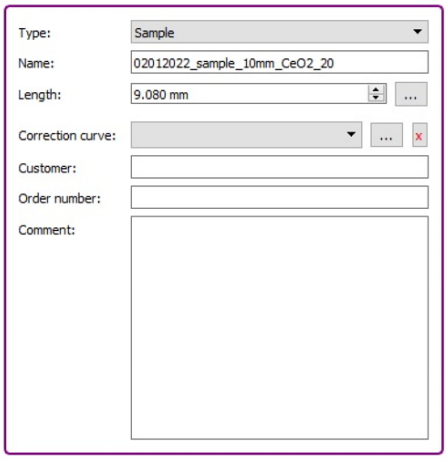
Figure 39. Select folder and name project.

4.1.3 Setting up a Sample Run

1. Open the dilatometer program to set-up the sample runs.

2. On the general settings tab, select the data folder to store the sample information in from either the drop-down arrow or the … Create a new folder as needed (refer to figures 9, 10, and 11).

3. In the general settings tab of the setup page make sure to enter the type, name, length, and correction curve (the correction curve may be left blank at this time).



Select sample for the type from the drop-down box.
Enter the name
Enter the length in mm
Correction curve doesn't need to be selected at this time.

- The length should be an average of 3 measured points on the sample.
- The length of the reference material should be +/- 5 mm of the sample material.

Figure 40. Sample settings information.

Select the temperature profile tab in the setup page (refer to figure 13).

- Use the same temperature profile for the sample run as the correction run. Select the same heating and cooling segments with the same rate, temperature, and dwell times.

- To select a previously used temperature profile, click on load and when the profile administration window appears select the desired profile and click OK (refer to figure 16).

4. Select the scheduler tab (refer to figure 17).
   - Use the scheduler to set up 4 repeat runs.
   - Four runs will ensure a good run-to-run repeatability.
   - The data will be saved in separate data files for each repeat run.
   - Select either time or temperature, but not both.
   - The time or temperature is used as the start trigger for the next scheduled run.
   - If the dwell was programmed in the cooling segment, then select the time option and set it to 1.00 min.
   - The temperature option should only be used if room temperature does not vary throughout the day.
     - If a low room temperature is entered, then the furnace may never get to the triggered temperature to run the next sample.
     - If a high room temperature is entered, then the sample may never reach the room temperature expansion value before the next run starts.
5. Select the zero adjustment load tab in the setup page (refer to figure 18).
6. Raise the furnace on the dilatometer's touch screen page 3. Press the up arrow above the "furnace lift". Lower the push rod using the same screen. Press the down arrow below the "push rod DIL1" (refer to figure 19).
7. Displace the push rod manually to see if the zero adjustment load's red dial moves properly, before loading the sample, to confirm the computer is communicating with the LVDT.
8. Before loading and unloading a sample make sure the activate load controller box is not selected (refer to figure 20).
9. Load a sample onto the push rod as done with the quartz reference sample (refer to figure 21).
10. Verify the nominal load is 300 mN on the zero-adjustment load tab (refer to figure 22).
11. Raise the push rod on the dilatometer's touch screen until the position is reading -10 to -15% (refer to figure 23).
12. Click the adjust button to zero out the LVDT. The needle will adjust to the new % position close to 0% (refer to figure 24).

13. Select the start measurement green side arrow to start the run and collect the data (refer to figure 25).
    - Verify the scheduler has started, by looking under the setup tab and finding the word scheduler highlighted in a yellow box (refer to figure 26).
    - Take note of the room temperature for the sample and furnace.
    - Verify the measurement has started, the word scheduler will be replaced by measurement highlighted in a red box (refer to figure 27).
    - The sample temperature and the furnace temperature should be increasing.
14. Select the current values page to view the measurement in progress.
    - The whole temperature profile and the corrected profiles may be viewed by selecting the boxes in the upper right corner. The temperature, temperature profile, and length are color coded (refer to figure 28).

4.1.4 Correct Sample Data Using Mean Correction File

1. Open the evaluation program to analyze the sample curves with the mean quartz correction data.
2. On the tool bar select the icon to load a curve.
3. Open the folder where the sample curves were saved and select the sample curves to be evaluated, use the Ctrl key to select multiple curves, and click OK.

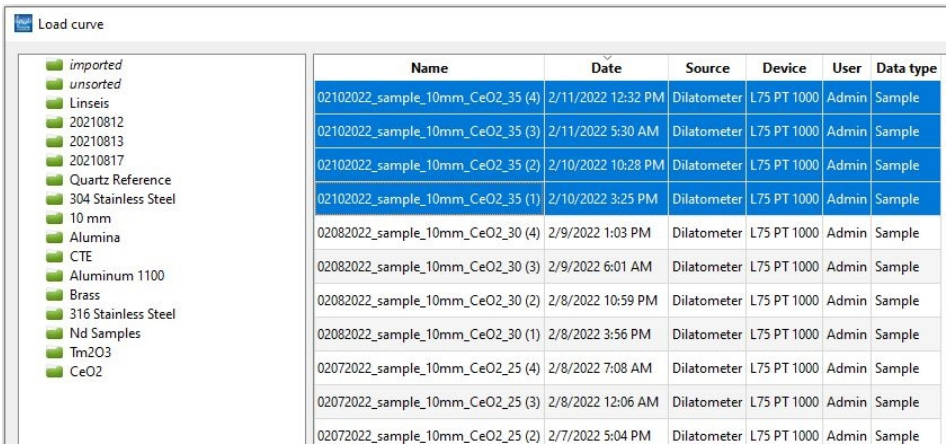


Figure 41. Loading sample curves.

- The channel selection window will appear, then select the x (Time) and y (Delta L) axis for each loaded curve. Click OK and the curves will be populated in the main screen (refer to figure 31).

- The dilatometer correction window will appear, verify the piston correction and subtraction of a correction curve is selected.

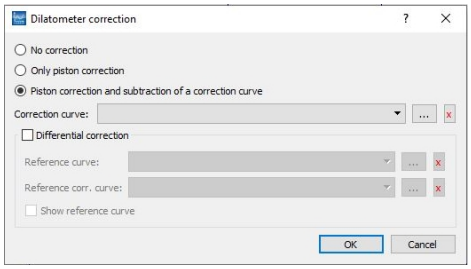


Figure 42. Loading correction curve.

4. To load the mean correction curve, select the … in Figure 42.
   - Open the folder where the mean correction curve was saved, select the file, and click OK.

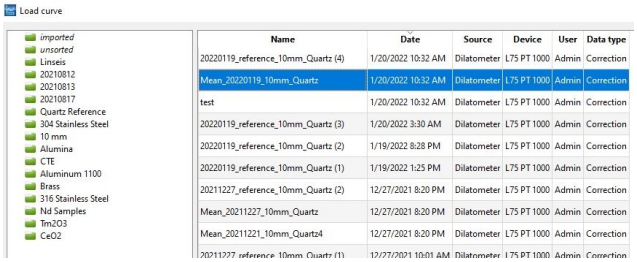


Figure 43. Select correction curve.

- The mean correction curve is populated in the correction curve drop-down box in figure 42.
- Click OK and repeat this process for all the sample curves selected.
- A pop-up window indicating a different start temperature will appear. Click yes to continue.

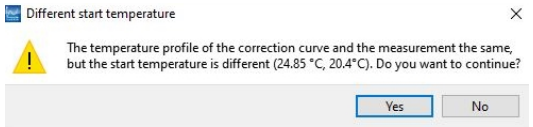


Figure 44. Start temperature.

5. Compare the data of the sample runs to look for repeatability.

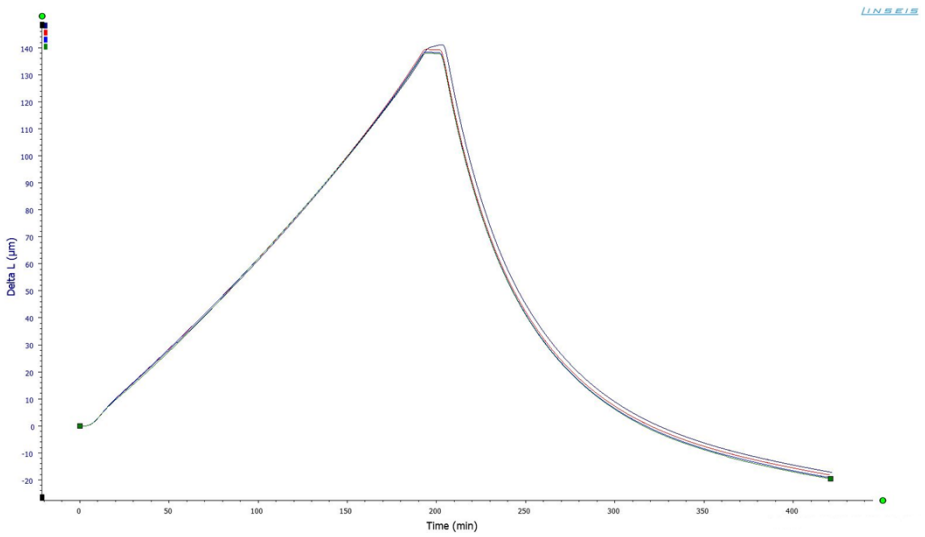


Figure 45. Sample curves.

- Eliminate any curves which are outliers and are not to be included in the data. Delete the curves by first highlighting them in the elements window under the project name, then right click and select delete.

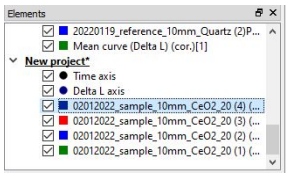


Figure 46. Delete sample outlier.

6. Select a curve and separate the heating and cooling segments using the icon in the tool bar.
    - Delete the cooling segment or deselect the cooling segments in the project to only show the heating segments for each curve.

    - As needed adjust the visible axis with the icon in the tool bar.

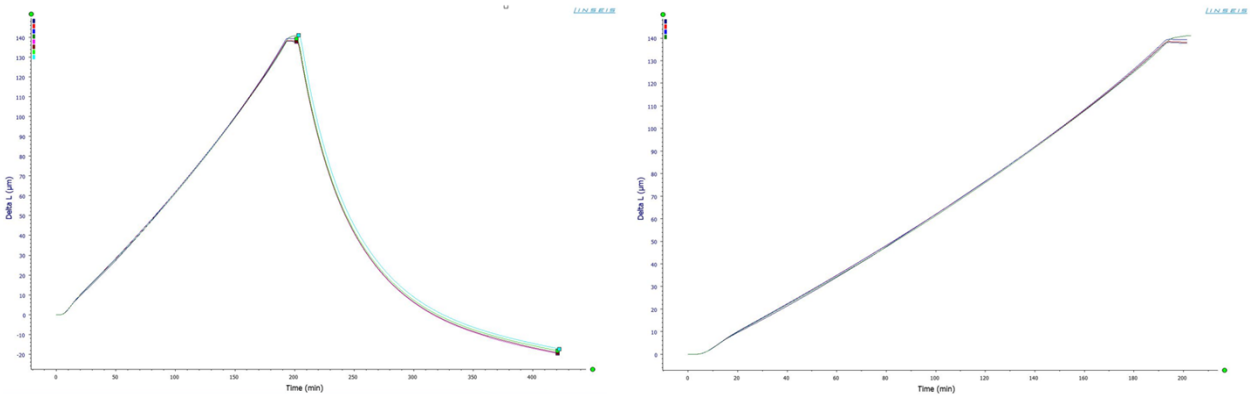


Figure 47. Split segment into heating/cooling and remove cooling segment.

7. Change the x axis as needed from time to temperature by right clicking on temperature in the x axis and select time from the pop-up window.

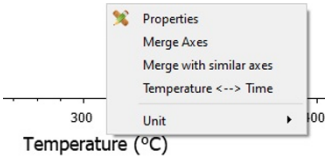


Figure 48. Change X-axis units.

8. Select a sample curve and click on the CTE icon  , which is no longer greyed out.

9. A pop-up window will appear asking for a reference temperature to be used. Enter the desired reference temperature (for example 30.0000ºC) and click Ok.

Figure 49. Reference temperature changed to 30.0000 ºC.

10. Repeat steps 8 and 9 as needed for each sample curve.

4.1.5 Export Data

Data can be exported as an image of the Linseis graph or as a table for use in excel.

1. To export data as an image, select the curves to be exported from the project in the elements window.

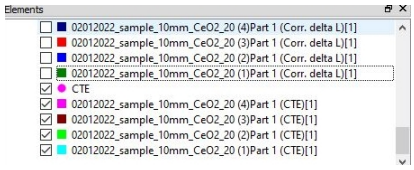


Figure 50. Check information to export.

2. Select the diagram tab and then click create diagram button.

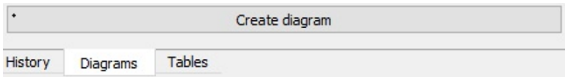


Figure 51. Create diagram.

3. Enter the name of the diagram in the pop-up window that appears.

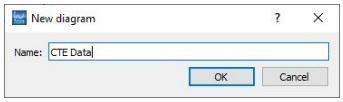


Figure 52. Name diagram.

4. After entering a name and clicking OK the diagram will populate under the diagrams window.

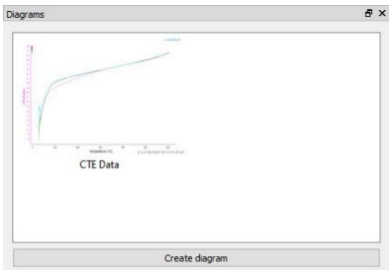


Figure 53. Created diagram.

5. To export the diagram, right click the diagram and select export.
    - Save the diagram in a folder of your choice and use the image type of your choice (for example JPEG image *.jpg).

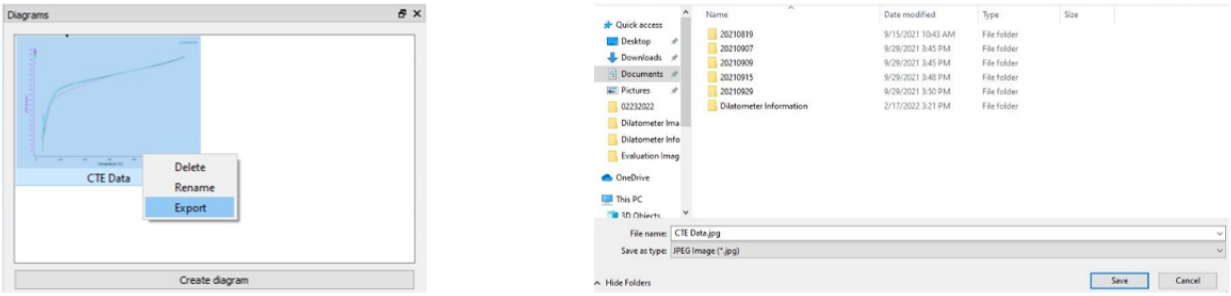


Figure 54. Exporting a diagram.

6. To export data as a table, select the curve to be exported from the elements window (refer to figure 50).

7. Select the table tab and click on the create table button.

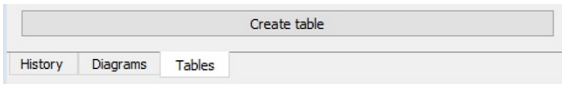


Figure 55. Create table.

8. Verify a table creator window appeared.

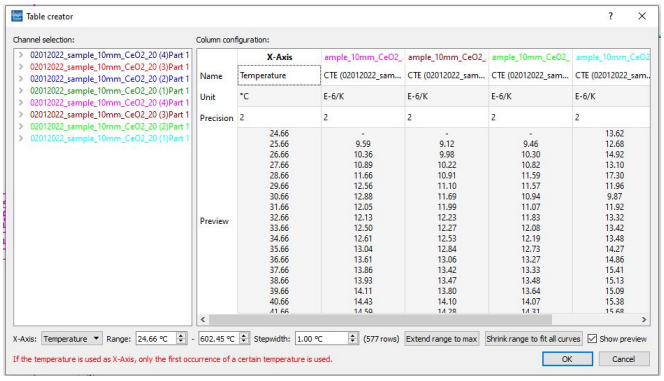


Figure 56. Table creator window.

9. Select the desired units (time or temperature) for the X axis.
10. Verify the range and step width for the x axis are set to the desired quantity.
    - If necessary, change the range or step width as needed.

11. Select Ok and a pop-up window will appear to name the table.

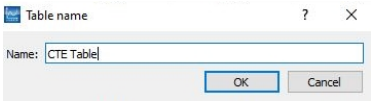


Figure 57. Name table.

12. After entering the name and clicking OK, verify the table populated under the tables window.

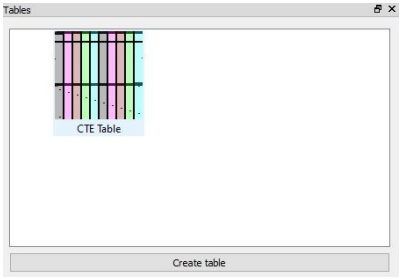


Figure 58. Created table.

13. To export the table, right click the table and select export.
    - Save the table in a folder of your choice and save as an MS Excel File (.xls).

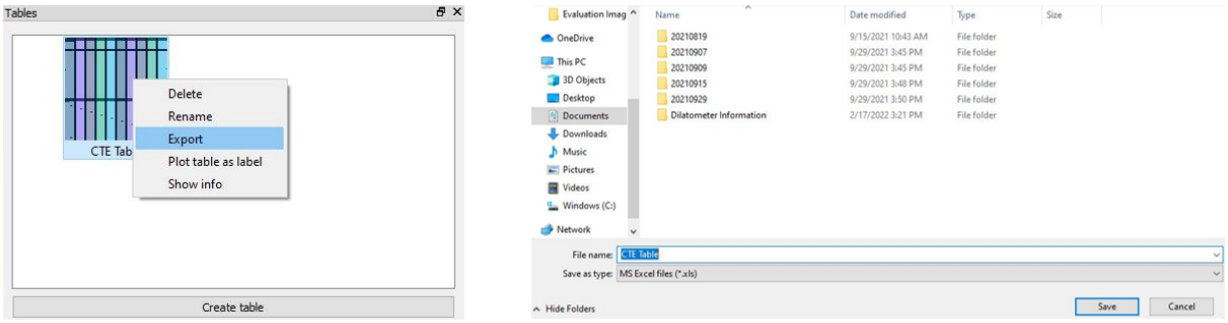


Figure 59. Exporting a table.