Temperature Controlled Stages

Standard Operating Procedure for the Dimension series non-magnetic heater/cooler & magnetic heater only stages

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Introduction to the Two Stages

The particular experiment being performed will determine which stage should be used. The non-magnetic heater/cooler stage contains a Peltier-based heater/cooler element with a maximum accessible temperature range of -35°C to +100°C (although extremely small thermal masses and a constant flow of dry nitrogen purge gas to prevent condensation are necessary to reach the lower temperature limit). For temperatures >100°C, the magnetic heater only stage must be used. It is capable of achieving setpoint temperatures ranging from ambient (~22°C) to +250°C. Both stages contain an embedded K-type thermocouple to measure the temperature of the stage and communicate with the controller. There are pros and cons to each stage. Things to consider when deciding which stage to use include: whether magnetism of the stage is a potential problem, if the accessible temperature range is adequate, and sample size and thermal mass. Although the non-magnetic heater/cooler stage can handle the same size pucks as the magnetic heater stage, it requires more thermal compound (discussed further in the SOP). Also, the non-magnetic heater/cooler stage is in reality designed for a smaller samples compared to the magnetic heater stage. The optimum sample size for the heater/cooler stage is 6 mm in diameter, for which there are no pucks commercially available. In contrast, the magnetic heater stage can accommodate samples up to 12 mm in diameter. The link to the Bruker User Guide is as follows:

https://wpwww-prod.s3.us-west-2.amazonaws.com/uploads/sites/551/2019/07/SUPPORT-NOTE-DIMENSION-HEATER-COOLER-E-013-441-000.pdf

Two additional materials may be needed to ensure a good thermal connection between the stage and the sample: 1) thermally conductive, electrically insulating pads (Bruker Part # 499-000-261), 2) Wakefield Engineering Thermal Compound (Wakefield Engineering Part # 120-2). The thermally conductive pads are only for use on the non-magnetic heater/cooler stage; do not use them with the magnetic heater stage.

General Procedure (Both Stages)

The generalized procedure below is for either stage. When special steps are needed for a particular stage, they will be clearly indicated.

1) Find the desired 5-pronged stage in the blue storage bin kept in the bottom of the rolling cabinet located next to the Dimension Icon AFM. The different stages are shown in Figure 1 below.

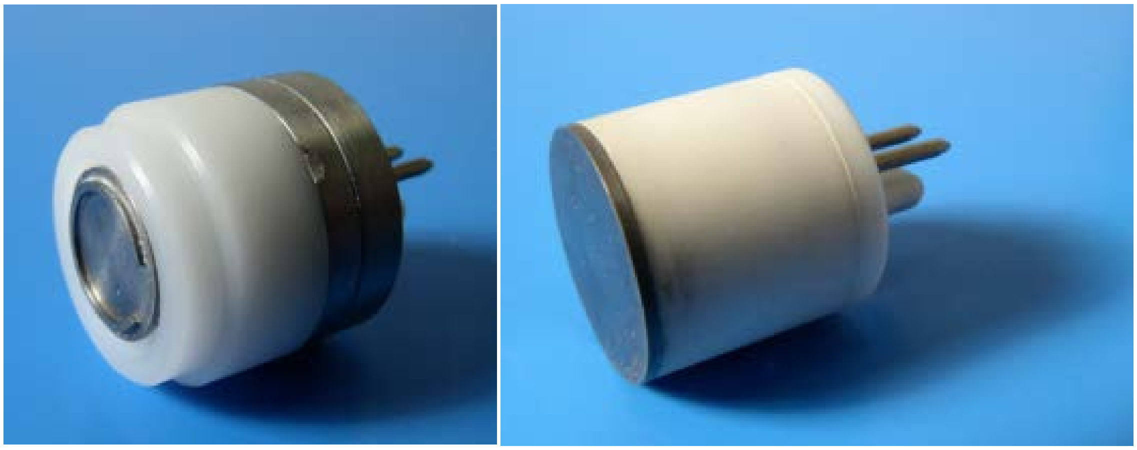

Figure 1. Left: The non-magnetic heater/cooler stage (-35°C to +100°C). Right: The magnetic heater stage (ambient to +250°C).

NOTE: *The non-magnetic heater/cooler stage requires placement of a thermally conductive, electrically insulating pad placed at its base (around the prongs) as shown in* Figure 2.

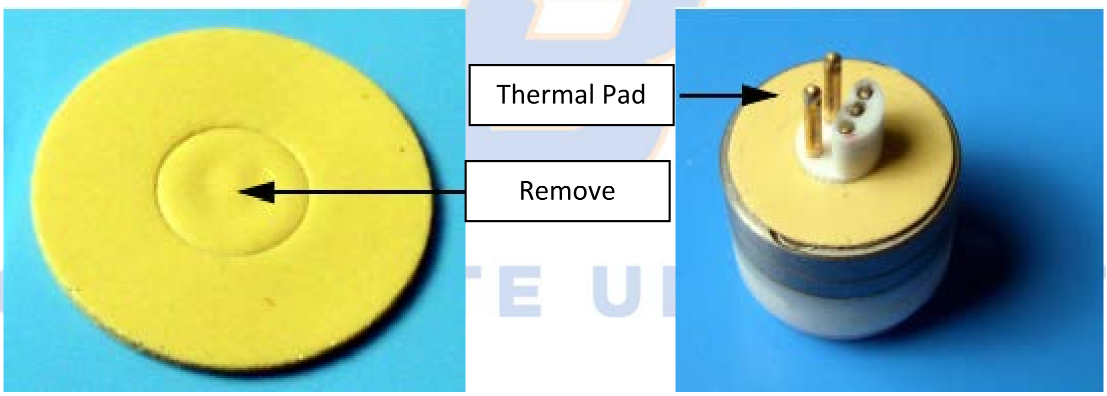

Figure 2. A new, unused thermally conductive, electrically insulating pad (left), and one attached to the base of the non-magnetic heater/cooler stage (right).

2) Carefully plug the desired stage into the temperature controlled stage base that is attached to the back left of the AFM stage as shown below in Figure 3.

   WARNING: **Be sure not to bend or otherwise damage the leads on the bottom of the element! Also note that the tungsten cap atop the non-magnetic heater/cooler stage is easily dislodged from the underlying leads, thereby destroying the heater/cooler element.**

Figure 3. The magnetic base and surrounding gas purge ring for the temperature controlled stages without (left) and with (right) the non-magnetic heater/cooler stage installed.

3) Turn on the AFM.

   WARNING: Do not power cycle the computer with the AFM on; this could cause severe damage to the piezo(s).

   NOTE: *Allow the laser to warm up for approximately 30 minutes prior to imaging if exceptionally precise measurements are required for your experiment.*

4) Login on the Excel sign in sheet located at the upper right of the AFM computer desktop.
5) Turn on the TAC (Thermal Applications Controller) by flipping the toggle switch on the back left of the controller (Figure 4). This controller is used to control both stages.

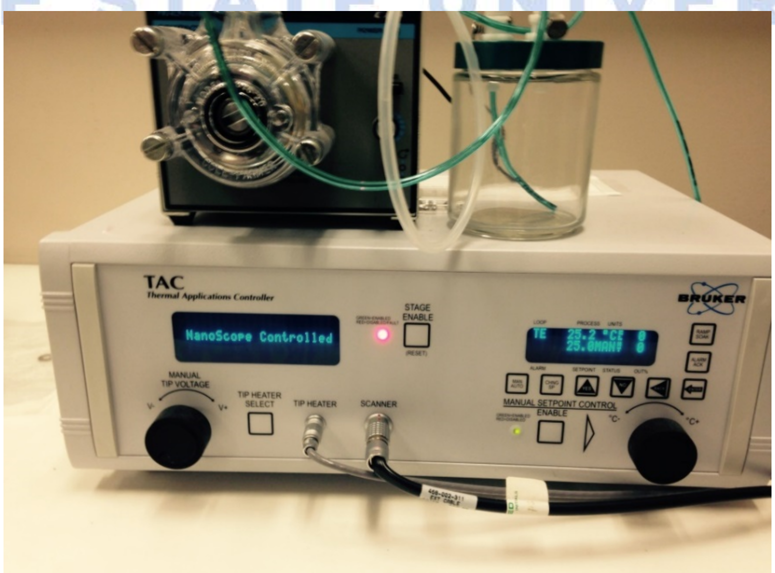

Figure 4. Thermal Applications Controller (TAC) with peristaltic pump and fluid reservoir (used when cooling the sample below ambient temperature) located on top.

6) Open the AFM control software by double clicking the NanoScope icon.
    a. Ensure that the Dimension Icon head is both physically attached and displayed in the software (Figure 5). If not, use the *Change Scanner* option in the software to switch from the FastScan to the Icon head.
    b. Under *Change Microscope Setup*, select the *Dimension Icon H/C* from the *Microscope Select* menu. This establishes communication between the TAC and AFM (Figure 5).
    c. Load the desired experiment (e.g., MFM) under *Select Experiment*.

NOTE: *Ensure that **both** the AFM and TAC are turned on prior to starting NanoScope and selecting the Dimension Icon H/C option. Not doing so will cause a software error due to the inability of the AFM and TAC to communicate.*

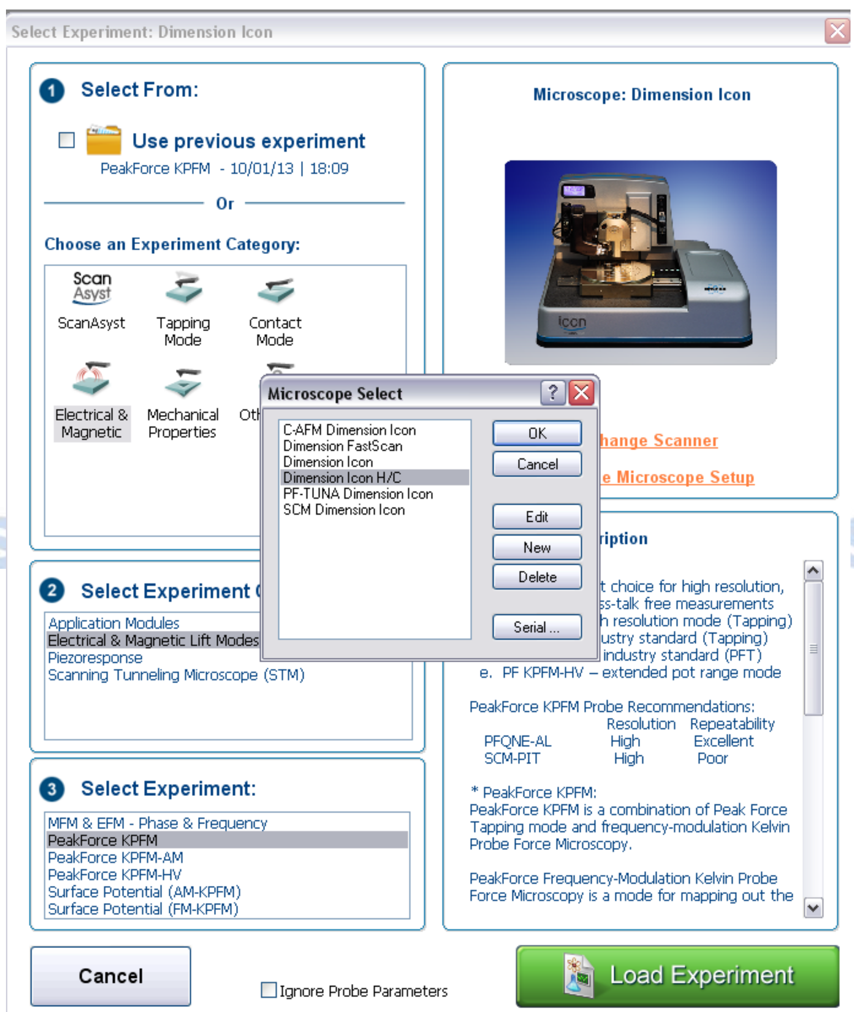

Figure 5. The *Select Experiment* interface with the *Microscope Select* window opened. The *Dimension Icon H/C* profile must be selected prior to running a temperature controlled experiment.

7) Prepare the sample to be used for the temperature controlled experiment.

NOTE: *Preparation of the sample will vary based upon the type of sample being used (e.g., metal, ceramic, polymer, etc.). If you are unsure, you should meet with an SSL employee prior to starting your experiment to discuss how the sample should be mounted. A generalized preparation technique is outlined below for reference.*

a. Obtain a sterilized cotton swab from the plastic storage cabinet located on the desk next to the optical microscope used for AFM probe mounting.
b. Use the swab to apply a thin coating of Wakefield Engineering Thermal Compound (located in the blue bin) until the entire top surface of the stage (i.e., tungsten cap for the non-magnetic heater/cooler and the metal puck for the magnetic heater) is uniformly covered. This will ensure there is good thermal conductivity between the temperature control element and the sample.

NOTE: *Even with good thermal conductivity established, the precise temperature at the surface of the sample for a given nominal (i.e., setpoint) temperature will vary based upon a variety of factors, including the thickness, mass, and thermal conductivity coefficient of the sample, whether or not a sample holder (e.g., 6, 12, or 15 mm stainless steel puck) is placed between the element and the sample, etc.* Table 1 *is the actual sample temperature for a variety of nominal setpoint temperatures as measured empirically using a thermocouple and a NiMnGa sample for the non-magnetic heater/cooler stage.* Table 2 *is the actual sample temperature for a variety of nominal setpoint temperatures as measured empirically using a thermocouple and a sample puck for the magnetic heater stage.*

Table 1. Non-Magnetic Heater/Cooler Stage Nominal versus Actual Temperature.

Nominal Setpoint (°C)	Actual Temperature(°C)[1]	Δ (°C)
25	23.5	-1.5
35	30.9	-4.1
45	38.2	-6.8
55	45.5	-9.5
65	53.9	-11.1
75	61.5	-13.5
85	69.1	-15.9
95	76.5	-18.5
100	81.2	-18.8

[1] Note that the exact value will vary based upon sample composition/properties (e.g., thermal conductivity), size (thermal mass), and mounting. Values quoted are for NiMnGa as measured by Chad Watson and Courtney Hollar.

Table 2. Magnetic Heater Stage Nominal versus Actual Temperature.

Nominal Setpoint (°C)	Actual Temperature(°C)[2]	Δ (°C)
25	22.8	-2.2
35	31.8	-3.3
45	41.6	-3.4
55	51.1	-4.0
65	60.7	-4.3
75	69.7	-5.3
85	78.8	-6.2
95	87.9	-7.1
105	96.9	-8.1
115	106.3	-8.8
125	115.4	-9.7
135	125.2	-9.8
145	134.6	-10.4
155	144.2	-10.9
165	153.9	-11.2
175	162.2	-12.8

c. Turn on the purge gas flow to prevent condensation (cooling) and/or oxidation (heating) as well as assist in maintaining thermal equilibrium and environmental control.

   i. Turn on the UHP (ultrahigh purity) nitrogen tank located against the back wall of the lab to allow gas flow from the tank.

   ii. Open up the line and regulate the flow of purge gas by turning the knob on the flow meter counter-clockwise (Figure 6). Allow the small bead to rise approximately to the line marked 10 mL/min (for heating). This will be sufficient to generate laminar flow without affecting the temperature or creating turbulence. An ~10x higher flow rate is necessary to prevent condensation on the probe when cooling the sample.

[2] Note that the exact value will vary based upon sample composition/properties (e.g., thermal conductivity), size (thermal mass), and mounting. Values quoted are for a 12 mm sample puck as measured by Lance Patten.

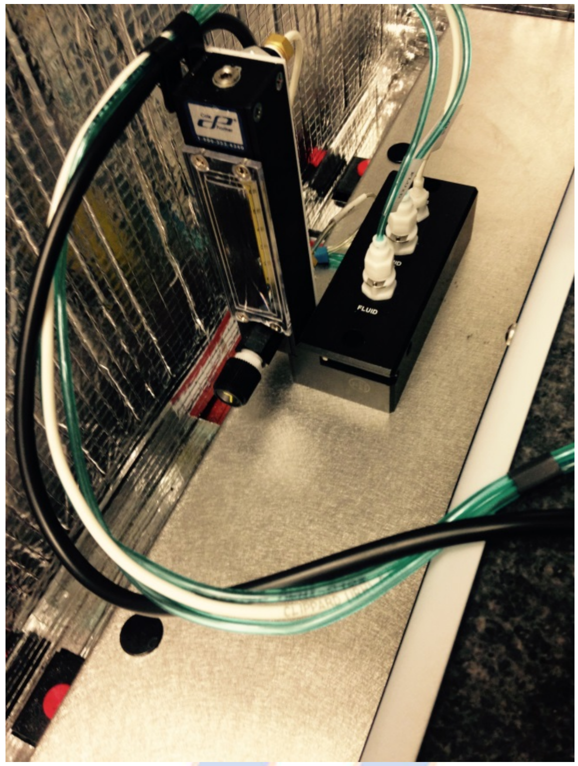

Figure 6. The purge gas/Peltier cooling fluid manifold showing the flow meter, adjustment valve, and associated tubing for the dry nitrogen purge gas.

8) Load a probe into the special ceramic probe holder (also located in the blue bin). The ceramic probe holder is seen at left in Figure 7 below.
   a. The ceramic probe holder uses a special stand, shown at right in Figure 7. Place the ceramic probe holder on the correct probe loading station. Pressing the ceramic probe holder down on the correct probe loading station will cause the pin to release the spring clip on the holder. Slide the probe under the clip to the normal probe location, just off the back of the groove. Release the pressure on the ceramic probe holder to allow the spring clip to hold the probe in place.
   b. When attaching the ceramic probe holder to the Dimension Icon head, also attach a silicone rubber gasket in order to seal the chamber for a controlled atmosphere.
   c. This special ceramic probe holder has several important features needed for both stages.
      i. It enables heating of the probe tip so that the tip temperature more closely matches that of the sample.
      ii. It thermally insulates the Icon head's piezoelectric scanner tube from the heated tip and sample, as the piezoelectric response (and hence calibration) of the scanner is temperature dependent.
      iii. The silicone rubber gasket seals around the gas ring, helping to maximize thermal stability, minimize turbulence, and create a controlled (dry) environment for imaging.

NOTE: *Do NOT use the normal Icon probe holder! The heater/cooler stage will not function properly without this special ceramic heater/cooler probe holder.*

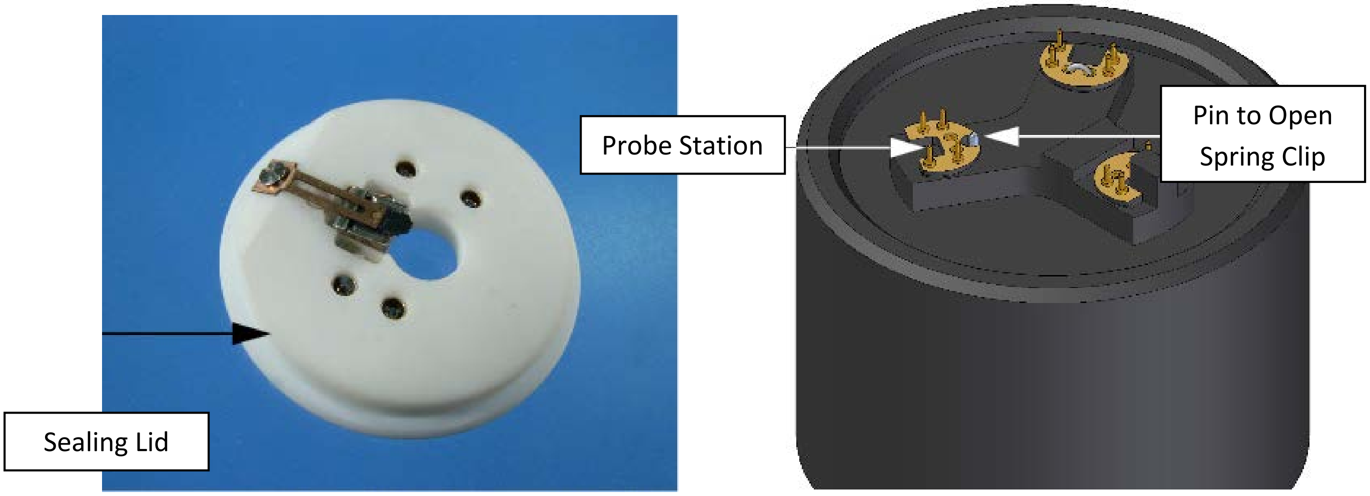

Figure 7. The ceramic probe holder (left) and the ceramic probe holder stand (right).

9) Set up the NanoScope software in accordance with the appropriate experimental SOP.
10) Prior to engaging, ensure the temperature control parameters are set properly (Figure 8).
    a. Set *Temp Stage Setpoint* to the desired value for imaging. The *Stage Temperature* parameter will update in real time to show the actual temperature of the unit (***not*** that of the sample).
    b. Due to the difference in mass between the special ceramic probe holder and the normal Icon probe holder, change the X and Y IGains from 12 to 8. These can be accessed via the *Calibrate|Scanner* menu, which will open up the *OptoXY Closed Loop Calibration* window. *Remember to change these back to the default value of 12 when you are done with your experiment!*

WARNING: For both stages, if the desired temperature is far from room temperature (~25°C), gradually change the temperature in small increments (≤10°C per step). This will protect the sample and stage from rapid temperature changes, which can lead to cracking due to thermal expansion or contraction. For example, if the desired imaging temperature of the element is 100°C (corresponding to ~81.2°C at the sample surface according to Table 1 for the non-magnetic heater/cooler stage), then initially increase the *Temp Stage Setpoint* to 35°C. Monitor the *Stage Temperature* parameter as it gradually approaches 35°C. Hold for ~5 minutes, then continue this procedure of increasing the setpoint in 10°C increments until the final desired setpoint of 100°C is obtained. Gradually cool the stages in the same manner, but in reverse.

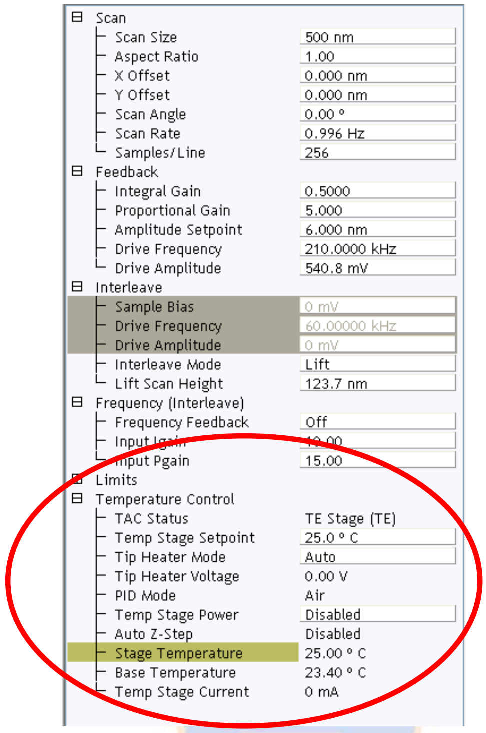

Figure 8. Temperature Control parameters (circled). Note that the ceramic probe holder has a built in heater for the probe tip. This special holder serves to insulate the thermally sensitive piezoelectric scanner tube from the heated probe and sample.

11) Engage the surface and proceed as normal.

WARNING: It is not advisable to change the temperature of the sample while imaging. Instead, withdraw, change the temperature setpoint by a small amount, and wait 3-5 minutes for the temperature to equilibrate before re-engaging and imaging.

12) After the experiment has completed, withdraw the probe and start equipment take down.
13) Turn off nitrogen flow, if used.
14) Change the X and Y IGains back to 12 from 8. This is accessed via the *Calibrate|Scanner* menu, which will open up the *OptoXY Closed Loop Calibration* window.
15) Click on *Experiment* in upper left, scroll down to *Select Experiment* and click.
    a. Go to *Change Microscope Setup* and select the *Dimension Icon* from the *Microscope Select* menu. This removes communication between the TAC and AFM (Figure 5).
    b. Load the desired experiment if needed (e.g., MFM) under *Select Experiment* or close the NanoScope Software
16) *After* the NanoScope software has been closed, turn off TAC controller.
17) Remove the ceramic probe holder from the Dimension Icon head. Remove the probe from the ceramic probe holder using the probe station on the probe holder stand. (Remember to press the ceramic probe holder down to cause the pin to release the spring clip on the holder.) Return the holder and silicone gasket back to the blue bin.

18) Remove the sample and clean the stage that was used. Wet a KimWipe and/or q-tip with a minimum amount of mold organic solvent (e.g., an alcohol such as methanol or ethanol, *not* acetone) and gently clean the stage. Return the stage back to its container in the blue bin.

WARNING: Be careful cleaning the heater stage. Do not use acetone! This can ruin the stage by damaging the heating elements or destroying the adhesives used to cement the stage together.

19) Ensure all other components have been returned to the original locations if experimentation has been completed.