UHV System
Standard Operating Procedure

Lab: Davenport 270
Department: Chemical and Biomolecular Engineering
PI/Manager of Space: Prof. David W. Flaherty
Written by: Abinaya Sampath

Section 1: Overview

Type of SOP: ☒Process ☒Hazardous Material ☐Hazardous Class of Materials ☒Equipment

Synopsis:

The SOP will help future group members to understand the basic UHV system operation along with its full capabilities. There are a number of important equipment attached on to the UHV system, such as LEED, AUGER, Sputter Gun, Manifold system, Metal Evaporator, and TPD system etc. For more details on specific equipment look on the Lab Wiki>UHV related documents.

Section 2: Risk Assessment Summary (Hazards and control measures)

Information obtained from performing a risk assessment should be entered into this section.

Materials:

<table>
<thead>
<tr>
<th>Material (name, CAS #, other ID)</th>
<th>Hazards</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon monoxide</td>
<td>Toxic, flammable, asphyxiant</td>
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<tr>
<td>Liquid nitrogen</td>
<td>Cryogenic, can cause fire hazard, asphyxiant</td>
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<tr>
<td>H₂</td>
<td>Flammable, asphyxiant</td>
</tr>
<tr>
<td>O₂</td>
<td>Flammable, cause burns and frostbite, strong oxidizer</td>
</tr>
<tr>
<td>Ar</td>
<td>Gas under pressure, asphyxiant</td>
</tr>
</tbody>
</table>

Relevant References for Material Hazards:

- Carbon monoxide
- Liquid nitrogen
- H₂
  [https://www.airgas.com/msds/001026.pdf](https://www.airgas.com/msds/001026.pdf)
- O₂
  [https://www.airgas.com/msds/001043.pdf](https://www.airgas.com/msds/001043.pdf)
- Ar gas
Equipment Hazards:

- Power supply DLM 8-75, Auger electronics, sputter gun electronics, LEED electronics, mass spectrometer electronics, thermal evaporator electronics pose potential electrical shock hazard.
- Variacs and heating tape—Ignition source, hot surfaces, electric shock
- Diffusion pumps and turbo pumps – Hot surfaces, electric shock
- Liquid nitrogen probe, dewer – cold surfaces, explodable
- Gas lines – High pressure

Hazardous Conditions:

- CO is toxic. The gas lines are connected to high vacuum lines with control valves. These gas lines are not to be removed without proper precautions.
- H₂ line connected to UHV should be disconnected only after vacuuming the gas line
- If any of the electronics are wet due to water spillage will cause electrical shorts, which could shock individuals without. Make sure no electrical cables are lying on floor

Technique Hazards:

- Filling probe with liquid nitrogen requires steady hands and all of the liquid nitrogen handling technique, cryogenic gloves, and safety goggles are a must
- Cleaning of high vacuum chamber requires lifting of heavy objects which are to be taken take of

Personal Protective Equipment

- Safety goggles, cryogenic gloves if needed for handling liquid nitrogen
- All chemicals to be placed in the manifold are to be handled in fume hood with gloves and safety goggles
- Gloves and lab coats should be used when handling the chambers of UHV when cleaning to avoid the non-hazardous pump oil stains

Engineering Controls

- Gas lines are to vacuumed into UHV before disconnection as most of them are hazardous
- All the liquid dosers in the manifold should be tight enough to not to be exposed to atmosphere.
- Carbon monoxide and Hydrogen should be stored in gas cabinet with ability to purge gases prior to removing gas regulator.
- Variacs and furnace should be plugged into outlets with a ground fault circuit interrupter (GFCI).

Section 3: Procedures

- UHV system operation: Prior to system turn on, a check list should be run to ensure all CF and VCR connections are fully tight. The UHV chambers are fully sealed when all the following are either closed/tight
- Gate valve is closed
- The liquid nitrogen probe is tightened to the chamber
- The turbo pumps are in working condition
- All the UHV equipment (QMS, LEED, Thermal evaporators, QCM, Sputter gun, AES) are attached to the UHV chamber and sealed
- The ion gauge is connected and working
- The leak valve attached to the UHV chamber is closed
- The turbo pumps are connected to the roughing pump line
- All the transparent windows are attached and sealed

**UHV Startup**

- Once the UHV is completely sealed from atmospheric conditions, the roughing pump in pump house can be turned, within 8-10 minutes the pressure in the roughing pump line monitored by the thermal conductivity gauge should reach 5E-2 torr
- At this point you may turn on both turbo pumps simultaneously and wait for ~ 20 minutes, (Both controllers are on the blue electronic rack). By this time pressure in UHV should be around 6E-7torr
- Turn on the ion gauge to check the pressure reading
- If there’s leaks the pressure would be higher than 6E-7 after 20 minutes

**UHV Shutoff**

- Prior to vacuum shutoff, all other electronics must be fully shut off (LEED, AES, Ion gun, QMS, Evaporators)
- The circuitry of flag, gate and shutter are shut off from power
- The power supply for heater if shut off
- The liquid nitrogen dewers are to be emptied
- Make sure the three leaks valves (one with manifold, one in the phosphine line and one to the rear of chamber 4) are completely closed
- Switch off the channel for ion gauge of chamber 1 and 2 and unplug the ion gauge for chamber 1 and 2
- Unplug both the diffusion pumps and approximately after an hour when the body of the pumps are cold enough, by pass the cooling water for the pumps and cut off the water to pumps
- Switch off the manometer attached to the manifold and close the vacuum outlet of manifold
- Close the line to roughing pump from chamber 1 and 2 and after about 10 minutes switch off the roughing pump of chamber 1 and 2
- Now, switch off the ion gauge of chamber 4 and shut off the both the turbo pumps
- The turbo pumps will take ~ 1.5 hours to slow down and once they do, close the roughing pump line
- When the Pfeiffer pump reads 200 HZ and below, you can turn off the roughing pump for chamber 3 and 4
- Make sure to monitor for a while till all the pumps calm down and there is no abnormal noise from the system

Note: the Varian V-301 does not display the blade speed, it displays only power and current
- If opening the chamber to atmosphere, it is advisable to wait for at least half an hour before opening the windows
**UHV system bakeout**: This step should ONLY be done with Abinaya’s full supervision. It should be noted that all power being used for bakeout should not be shared with any other individuals in the lab. Total of seven variacs are used to power up three bakeout lamps and five heating tapes.

- The bake out procedure is carried out when the vacuum has been initiated (after making sure there are no leaks in the chamber) in UHV for a while now (at least a day) and we want the pressure to get to as low as possible
- Bake-out is essentially heating the outer walls of the chamber and the parts in by conduction with heating tapes and also by radiation using heating lamps inside the chamber
- The UHV system is baked to get the contaminants adsorbed onto the surfaces of the UHV part of system to minimize the degassing of them and slower pump down of chambers of UHV
- Before one starts the bake-out procedure, we should ideally monitor the contaminants in UHV and see that water is one of the main contaminant (18 amu QMS signal)
- All the equipment inside the vacuum chamber (QMS, QCM, LEED, AES, sputter gun, metal evaporators are switched off before the procedure begins
- Make sure the variacs connected to the power outlets. Also connect all the lamps and tapes to the variacs. It is better to have the variacs plugged into the outlets which share the circuit with Hongbo’s system for safe operation of variacs at their maximum capacity
- Cover the chamber windows and the walls with aluminium foil to retain heat better
- To start the bake out, increase the power output of variacs by 10 % every 10 minutes and not suddenly
- There are two variacs controlling three lamps and four variacs controlling five heating tapes
- Monitor the temperature at different locations on UHV and the crystal to increase the power of heating and achieve desired temperature
- Typically, the length of bake out depends on the base pressure of UHV before bake-out.
- Once the required bake-out is carried out, gradually decrease the power output of variacs to terminate the procedure
- The UHV will take 4-5 hours to cool down completely and only after it cools down will the base pressure be known

**Placing chemical in the liquid doser and testing its purity:**

- Remove the liquid doser from the manifold after closing the valve joining the doser and manifold. Clean the necessary liquid doser of the manifold with acetone thoroughly
- The sample holder in manifold is sprayed with acetone to make sure no old chemical is stuck in there. Ensure to have a Kim wipe when doing it so that acetone is not accidentally sprayed on the chamber walls. It should be left for at least an hour to dry
- In the fume hood place it in a stand. Clean a new copper gasket which is used to tighten it to the manifold. Every time the tube is inserted to the manifold, we use new gaskets. We use a soft gasket for a hard metal. In this case, we use copper with steel. The copper is cleaned with acetone too
- Before attaching the tube to the manifold, we have to clean the chamber 1 from the previous reactants. In this case, we use CO. Removal of CO is very important as it has high vapor pressure. Make sure the dosage valves are closed. Open and close the leak valve (check) and open the vacuum valve minimally swiftly and close it (we can observe the white vapors in the chamber – If we do not do it in small increments, the CO may escape to the UHV areas). Make sure the pressure of UHV is stabilized. Close all the valves. Once the pressure in the gauge comes down and the UHV pressure is stabilized we are ready
- Attach the tube to the manifold using nuts and bolts. Make sure that the liquid dose valve is closed
- Next step would be the removal of the contaminants from the doser and the solved contaminants. Get an ice bath ready to remove the dissolved contaminants. Immerse the doser in the ice bath
- Open and close the liquid dosage valve with the vacuum and the leak valve closed
- Open and close the vacuum valve. Make sure the UHV pressure is stabilized. Dose the liquid again by opening and closing the dosage valve. Repeat it a couple of times. Open the leak valve and check the pressure using the gauge. If the pressure equals the vapor pressure of the liquid, we have a purer gas
- Once we are done, vacuum it slowly by opening the vacuum valve
- With the introduction of the desired chemical in the manifold, we are to test the purity of it before we begin the runs
- Turn on mass spectrometry boxes, bottom first, and quadrupole control second
- Open the liquid doser valve completely and close it with the vacuum valve closed. Open the leak valve for till we achieve desired vapor pressure (desirably not more than 1 torr as we could increase the UHV pressure greatly otherwise and not less than 0.8 torr as we will not see contaminants easily) we start scanning for the contaminants
- We perform the mass spectroscopy after we open each of the valve. We do not let the gas to the sample by opening the flag. We perform a continuous scan (For all the compounds possible between 2 and 90 of molar masses). We do not heat the sample in this case. It is just a scan for the compounds present
- Open the ‘Merlin’ software and go to tune menu and change multiplier voltage from 850 to 1150 V. There we would find four kind of graphs in the home page. Find the one to the bottom left. Use the ‘Profile View’. Scan for 1 – 100 (changes depending on the fragmentation of the chemical used. Check NIST) of molar masses. Click ‘Averaging’ ->’Continuous’ (There would be the number of scans to be performed entered, typically 20). In the ctrl option below, enter ‘prof_to_list’. The file is saved as a text file. Export data from that and process it in Origin. Stop ‘Averaging’ by clicking the option ‘None’
- Open the gate valve. Change the ‘Averaging’ to ‘continuous’. Change it to ‘None’ again.
- Open the ‘shutter’ valve. Change the averaging to ‘continuous’. Once the 20 cycles of averaging is done, we change the averaging to ‘None’. Close the valves
- Now plot the QMS intensities of shutter corrected for contribution from gate valve open as a function of m/z+
- You should see that the relative intensities should match the fragmentation provided by NIST

*Temperature Programmed Reaction (TPR)*:
- Open labview program ‘2D film fabricator 6.0 Pd(111)’. Make sure the minimum crystal temperature is 77 K by adjusting the CJC (80 – 150 mV) or above. The output high voltage should be 2 V. Clean the crystal by first heating it to 900 K from the current temperature at 5 K/s. Once the crystal is being heated, change the output high voltage to 2.5 V. Hold the crystal at 900 K for ~ 10 minutes in the presence of oxygen. Cut off the oxygen supply once the crystal cools at the end of the procedure
- Once the base pressure goes below 9 E-10 torr, turn on the mass spectrometer by turning on mass spectrometry boxes, bottom first, and quadrupole control second and then the Merlin software.
- Close and open the labview program ‘2D film fabricator 6.0 Pd(111)’
- With the vacuum valve and leak valve in manifold closed, open the liquid doser valve as desired and open the leak to achieve the desired beam pressure as monitored by ion gauge
- Make sure the minimum crystal temperature is 77 K by adjusting the CJC (80 – 150 mV) or above. The output high voltage should be 2 V. Clean the crystal by first heating it to 1000 K from the current temperature at 5 K/s. Once the crystal is being heated, change the output high voltage to 2.5 V. Hold
the crystal at 1000 K for ~ 50 seconds. Meanwhile adjust the multiplier voltage to QMS to 1150 V in the Merlin interface and also enter the fragments to scan for in profile mode in Merlin interface.

- Once the crystal cools down from 1000 K, add desired quantity of liquid nitrogen to the probe depending on the temperature of the single crystal before annealing. For temperatures > 280 K add 1.5 tanks, 150 – 280 K add 1 tank, < 150 K add 0.5 tanks of liquid nitrogen. After anneal, make sure the temperature is down to a temperature less than or equal to 77 K.

Note: The crystal should cool down to <77 K in less than 6 minutes from the time liquid nitrogen is added

- Open the labview program ‘Andy’s TPD 21.0’. Also switch on the dash board with the controls for gate, shutter and flag
- Name the file as ‘dose_MM_DD_number of trial according to lab notebook’ for ease of reference
- Adjust the value of CJC in the labview interface to get the temperature to be in between 77 – 78 K (0.8 – 0.15 V)
- Enter the temperature ramp conditions and the dosing conditions accordingly. Make sure the output voltage is 2 V initially. After heating the crystal to the desired dosing temperature change the output voltage maximum to 2.5 V, open the gate valve, wait a few seconds for the pressure to stabilize, and turn on shutter, and wait another few seconds for the pressure to stabilize and open flag for the stipulated time of exposure and then close it
- Turn off shutter after a few seconds, close the gate valve, and open the flag again
- Copy the results found in Excel file into Origin file

Reactive Molecular Beam Scattering (RMBS):

- Open labview program ‘2D film fabricator 6.0 Pd(111)’. Make sure the minimum crystal temperature is 77 K by adjusting the CJC (80 – 150 mV) or above. The output high voltage should be 2 V. Clean the crystal by first heating it to 900 K from the current temperature at 5 K/s. Once the crystal is being heated, change the output high voltage to 2.5 V. Hold the crystal at 900 K for ~ 10 minutes in the presence of oxygen. Cut off the oxygen supply once the crystal cools at the end of the procedure
- Once the base pressure goes below 9 E-10 torr, turn on the mass spectrometer by turning on mass spectrometry boxes, bottom first, and quadrupole control second and then the Merlin software.
- Close and open the labview program ‘2D film fabricator 6.0 Pd(111)’
- With the vacuum valve and leak valve in manifold closed, open the liquid doser valve as desired and open the leak to achieve the desired beam pressure as monitored by ion gauge

Note: For RMBS experiments, the beam pressure should be maintained constant as we are trying to monitor the temperature dependence of the reaction

- Make sure the minimum crystal temperature is 77 K by adjusting the CJC (80 – 150 mV) or above. The output high voltage should be 2 V. Clean the crystal by first heating it to 1000 K from the current temperature at 5 K/s. Once the crystal is being heated, change the output high voltage to 2.5 V. Hold the crystal at 1000 K for ~ 50 seconds. Meanwhile adjust the multiplier voltage to QMS to 1150 V in the Merlin interface and also enter the fragments to scan for in profile mode in Merlin interface.
- Once the crystal cools down from 1000 K, add desired quantity of liquid nitrogen to the probe depending on the temperature of the single crystal before annealing. For temperatures > 280 K add 1.5 tanks, 150 – 280 K add 1 tank, < 150 K add 0.5 tanks of liquid nitrogen. After anneal, make sure the temperature is down to a temperature less than or equal to 77 K.

Note: The crystal should cool down to <77 K in less than 6 minutes from the time liquid nitrogen is added

- Open the labview program ‘Andy’s TPD 21.0’. Also switch on the dash board with the controls for gate, shutter and flag
- Name the file as ‘dose_MM_DD_number of trial according to lab notebook’ for ease of reference
Adjust the value of CJC in the labview interface to get the temperature to be in between 77 – 78 K (0.8 – 0.15 V)
- Enter the dosing temperature conditions accordingly. Make sure the output voltage is 1 V or less initially. Immediately after the start of heating, change the output voltage maximum to 2 V
- Once the desired temperature of crystal is reached, open the gate valve - wait for 10 seconds for the pressure to stabilize, and turn on shutter - wait another 10 seconds for the pressure to stabilize and open flag for the stipulated time of exposure and then close it
- Turn off shutter after 10 seconds, close the gate valve 10 seconds later
- Copy the results found in Excel file into Origin file

Using the thermal evaporator:
- Move the crystal to the evaporator position by taking care of the Y and Z position and do not hit the chamber walls with the crystal or the QCM
- Make sure the cooling water lines are on for the evaporator
- Turn on the thermal evaporator (one with the C-Type thermocouple as we are heating to a higher temperature ~1100 C)
- Make sure the shutter for the evaporator is closed
- Open the Ez-zone configurator and connect it to the evaporator using USB cable (COM-13)
- Program the evaporator using the configurator to heat the metal sample to required temperature (never use the temperature ramp of more than 25 C/min) and make sure you let the evaporator stabilize at intermittent temperatures
- Once the desired temperature is reached, open the shutter and expose the single crystal to the evaporated beam and align the crystal based on whether you can see the melted metal for desired temperature

Sputtering of the single crystal:
- Get picoammeter from the electric shop in Noyes lab from John
- Move the crystal to the sputter position by taking care of the Y and Z position and do not hit the chamber walls with the crystal
- Take out the copper leads for heating and connect the microammeter to one of the ends with alligator clips and read the emitted current. (As soon as the microammeter is switched on it goes to maximum current and it drops after a while)
- Connect the Argon gas cylinder to the chamber via the leak valve and maintain a constant pressure of Ar (from 7 – 8 E-08 torr when the base pressure is ~ 1 E-09 torr)
- Turn on the ion gun control and the beam voltage. Increase the beam voltage to 3 keV
- Turn on the emission very slowly to 5 A (approximately 1 turn)
- The switch the Raster from off to external. As soon as this is done we should see the current in microammeter to go up to typical value of 1.5 – 2.5 microamps
- Using the labview program ‘2D film fabricator 6.0 Pd(111)’ to heat the crystal to required temperature when sputtering the single crystal

LEED imaging of single crystal:
- Move the single crystal to the LEED position by beginning with changing y coordinate to 15 and z coordinate to 20 mm
- Rotate the crystal with the help of rotating platform to θ~271°
- Change the y coordinate to 24 and the z to 26 mm
- Switch on the power for LEED control
- Change the filament current to ~1 mA (~3 turns) gradually
- Increase the beam voltage to 40 eV and the screen voltage to 4 keV
- Ground the crystal with an alligator clip attached to one of the terminal of LEED instrumentation on UHV by clipping it onto the chamber
- You must be able to see LEED pattern

Note: Cover yourself with a black opaque fabric by going as close as possible to the window opposite to LEED to get a high resolution image
- To shut off LEED, unground the crystal and turn the filament current control completely counter clockwise
- Decrease the beam voltage to get to the minimum value possible
- Reduce the screen voltage to 0
- Switch off the power supply to the LEED

For Equipment: All the equipment has been described in Wiki which includes low energy electron diffraction, Auger electron spectroscopy, sputter gun, quadrupole mass spectrometer

Section 4: Waste Disposal/Cleanup

Liquid waste from the pump should be disposed of in the Organic Waste container. Be sure to include all of the components on the side of the waste container. Gloves should be worn when handling the liquid and should be kept in a fume whenever it is in an open container

Section 5: Emergency Response

For any emergency and requires UHV to be shut off, please look at the sheet attached to side of the blue electronic rack. Also, call Abinaya at 217-419-9224 to ensure no harm comes to any of the UHV equipment

Section 6: Additional Information for Troubleshooting

Advice:

1. Ion gauge should NEVER be turn on when pressure is above 1E-3 torr, this will burn out the filament
2. Diffusion pump must have cooling water on prior to being switched on (Double check the cooling water always)
3. Quadrupole mass spectrometer should never be turned on when oxygen pressure more than E-9 Torr is being dosed in the background of UHV
4. There are over 50 wires running in every direction near the UHV chamber, please be careful never to disconnect or damage any wire.
5. Thermocouple on near the probe is very fragile, special care MUST be taken to avoid bumping into it
6. Turbo pumps and diffusion pumps should NEVER be switched on before the roughing pumps are switched on
7. Acetone should NEVER come in direct contact with the walls of chambers or the rubber gaskets or windows. It can dissolve the sealant used on the chamber walls, windows or gaskets
8. The dashboard for gate and shutter should be turned off when chamber 1 and 2 are not under vacuum and chamber 4 is under vacuum
9. The liquid dosers are very fragile. Make sure you handle them with steady hands
10. Do not open the gas lines connected to manifold with the vacuum valve or leak valve open
11. When there is a high vapor pressure fluid in the manifold, open the vacuum valve slowly to pump out that fluid.
12. Make sure the manifold is empty as much as possible and not with any fluid dosed unnecessarily to avoid clogging of manifold.
13. Please check the vapor pressure of the fluid to be more than 0.8 torr to be used in UHV.
14. Take proper precautions when using corrosive fluids as they might damage UHV lines.
15. Make sure to get only vacuum compatible components for UHV.
16. Make sure you do not touch any switch on the heating supply DLM 8-75.
17. If the temperature reading is off and is not stable after the addition of liquid nitrogen to the probe, stop heating and to remove the top part of the probe gently to make sure the thermocouple wires are intact and put the cap back. This mostly solves the problem.
18. Spray sealant inside the probe if the base pressure is going up with liquid nitrogen.
19. Make sure the cooling water is on for all the lines to the diffusion pumps and the thermal evaporators all the time.
20. Make sure the QMS files (tune, configurator for UIUC and the modified ones by you) from the Merlin folder are backed up and the software installation folder is also backed up.
21. Make sure all the Labview Vis are backed up too with the changes made (the QMS related Labview files work with only Labview 2013 version and with Merlin 4.0 version). So, back up the download files of Labview 2013 which was bought from webstore by Abinaya.
22. Follow the instructions in the Merlin>manuals to connect the Merlin software to the QMS hardware and use only Ethernet port to establish a connection.
23. Very important point to note is that Labview 2013 and Merlin 4.0 work only with Windows 7. Hence do not upgrade the version of Windows without the purchase of new version of Merlin (which will cost more than $1500).
24. The chances of roughing pump failures are large when starting them up:
   a. If the pump does not work, and there is a humming noise, the motor is damaged.
   b. If the belt part is not vibrating, the belt is damaged and needs to be fixed.
   c. If the pressure is not low enough, check oil and after which you might have to talk to Webb Kyle who is generally available up to 9 PM in Noyes Lab.

Checklist to use on UHV system on a regular basis:

- [ ] UHV pressure to be $-2 \times 10^{-10}$ torr
- [ ] The vacuum line connecting turbo pumps to roughing pump should have a pressure of $-3 \times 10^{-2}$ torr
- [x] The vacuum line connecting diffusion pumps to roughing pump should be under the pressure of $-2.2 \times 10^{-2}$ torr
- [ ] The roughing pumps have sufficient oil and there is no smoke or weird noise coming out of them.
- [ ] All cooling water lines are maintained at constant flow rate of 20 GPH prior to metal deposition and diffusion pump operation.
- [ ] The temperature of small turbo pump <40 deg C and the cooling fan is switched on.
- [ ] The pressure in chamber 1 and 2 should be E-09 – E-07 torr.
- [ ] The ion gauge for beam pressure reads 0 torr.
- [ ] Shutter, gate and flag are connected.
- [ ] Thermocouple for single crystal reads room temperature.
- [ ] Ensure Pd crystal is aligned and hit any component (Coordinates Y≤21 mm, Z≤ 26 mm, X≤ 25.75 mm)
- [ ] The pressure on oxygen line is not more than 200 psi.
☐ Oxygen line is connected to the leak valve near sputter gun
☐ The vacuum lines are open to roughing pumps
☐ The liquid doser in manifold is not broken and the fluid inside looks normal

References:
Refer to Wiki > UHV related documents for any doubts on specifications of any equipment or procedures like cleaning of chamber 1 and 2 or the switching procedure of single crystal
Other manuals are with Abinaya
Training Documentation
Signing this document means that you have read and understand all aspects of this Standard Operating Procedure. The supervisor is the person that acknowledges you took the training and understand the procedure. They can be a lab manager or researcher assigned by the PI to oversee this particular SOP.

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<th>Name (Printed)</th>
<th>Name (Signed)</th>
<th>Supervisor</th>
<th>Date</th>
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