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Detox Demonstration Plan

The purpose of this plan is to verify that the process removes the PCBs from mineral oil via dichlorination with sodium metal in compliance with the current EPA permit.

The system will be operated as outlined in the attached Operators Guide.

The process operates using batches of oil. Only oil with 3,000 PPM or less PCBs is processed in this system. Oils with greater PCB concentrations are sent off-site for disposal in a TSCA permitted hazardous waste incinerator.

Each batch of oil is 1,000 gallons. For the demonstration, three (3) batches of oil will be processed. The target concentration of oils will be 2,000-3,000 PPM PCBs, near the maximum levels processed.

Each batch will be tested for PCB content prior to the start of processing using method ASTM Method D-4059 to verify the PCB levels.

For the demonstration, after the batch has been processed and determined to be <2 PPM PCB (ASTM Method D-4059) additional samples will be collected and sent to an outside laboratory to be tested for PCB (SW-846 Method EPA 8082) and total chlorine (SW-846 Method 9075).

The expected total time to complete the planned demonstration is approximately 3-5 days as the processing time for the highest level of PCBs can be significantly longer than the lower concentration batches.

For the demonstration, all processed batches of oil will be held in a common storage tank until results from the outside laboratory verify that the PCBs are <2 PPM.

Operator's Guide

Removing PCB from Transformer Oils with the NaX Process



Processing SOP Introduction

This document is the standard operating procedure for the NaX Process HMI (human-machine interface) software. Because there are inherent risks to operating machinery and handling chemicals, potential process operators should read and understand this procedure before attempting to treat PCB contaminated oil with the NaX Process.

Process Safety

It is important the Operator begins each day with a safety inspection. This means thoroughly inspecting all parts of the process and associated machinery to be sure they are in good working condition.

Before operating process equipment, the operator should:

- Inspect all safety guards, including tank and pipe insulation, mixer, and pump shaft guards. If any guards are missing or broken, replace or repair immediately.
- Check all the pipelines and sight glass tubes to make sure they are securely fastened, operating properly and in good condition. Immediately replace or repair any defective connections.
- Keep machine parts clean and free of accumulation oil or oil absorbent material, dirt or debris.
- Make sure all electrical cabinets are securely closed to prevent electrical shock hazards.

Never operate any equipment that is not in safe working condition.

Don't make a practice of leaning on or resting against equipment. You could be burned. Process tanks and associated piping, valves, and other devices can reach temperatures of 250 F.

Watch for possible causes of accidents and always stay alert. Process safety is a s full time job!

See the company's SDS for additional concerns and specific safety practices should follow.

HMI Conditional Elements

The process control software (also called the Human Machine Interface or HMI) is designed to inform the operator of processing steps, equipment function as well as status of the various processing tanks and equipment. These indicators display flow rate, temperature, pressure, on/off status, tank volumes and heater operation.

Equipment icons (pumps, valves, heaters, pressure switches) indicate the ON condition when they are colored green. These same process icons indicate the OFF condition when they are colored red. All [On] and [Start] buttons indicate ON when a green dot appears on the button. All [Off] or [Stop] buttons indicate OFF when a red dot appears on the button. Unless otherwise indicated, close buttons remain inactive (i.e. gray) until the operator clicks the [Stop] button or the processing set point value is reached. It may be necessary to click on the Stop button to cause the Close button to activate. This is required to prevent again until the processing steps are reset by clicking the [Reset Process Steps] button located at the bottom of each Processing Menu.

Tank volumes are displayed in gallons (or tenths of gallons) along with temperature and pressure where applicable. These values are typically accurate within 2-3% of scale but should always be confirmed with another measurement. In case of volume, the operator has access to totalizing flow

meters for PCB oil and water. Many tanks are equipped with sight-gauges. These clear tubes show the volume within a particular tank. Temperatures can periodically be confirmed by observing thermometers located on heated tanks and cooling lines. By periodically checking HMI display values against these measuring devices, the operator can detect any malfunction sensors or devices that require recalibration before such devices can cause a problem.

HMI Process Step Buttons

Processing Menus are located across the top of the HMI screen. These menus contain menu buttons that may open a processing pop-up or an additional processing sub-menu. A pop-up can only be opened once at a time. Buttons are white when available and gray when they are locked out. A red dot on the menu button indicates the step has been completed. When all the steps on a menu have been accomplished, all the menu button dots will be green. These can be reset by clicking that menu's [Reset Steps] button.

There are four Processing Menus on the HMI screen for the dehalogenation of PCBs in transformer oils. The menus are segregated by the area they correspond to (Reactor 1, Reactor 2, and Quench Tank) as follows:

Sodium Processing Menu

Manual Steps

Some steps in the Sodium Dispersion Formulation process are performed manually. These steps involve initial emptying of the Sodium Mix Tank, loading the tank with sodium bricks, transferring dispersion to the Sodium Storage Tank, and finally rinsing the Mix Tank once the sodium dispersion has been transferred to the Sodium Storage Tank.

It is best to start making dispersion as early in the day as possible and let the mixer run as long as possible. Longer mix times produce the smallest sodium dispersion particles. Smaller particles produce the reagent and the most effective PCB destruction. Typically, the Sodium Mix Tank will contain 20 gallons of very dry oil from the Blue Tank prior to making dispersion. This oil is left in the tank when not in use to prevent sodium residue from reacting with any air that may get into the tank.

The Sodium Mix Tank is designed to hold 200 pounds of sodium and 55 gallons of oil. The total volume of this mixture is about 100 gallons. The sodium mix tank should already contain approximately 20 gallons of very dry oil. As the sodium bricks are loaded in the tank they submerge in this oil. The oil prevents the bricks from reacting in the event air enters the tank prior to processing. It is important that the bricks be placed as low in the tank as possible so that the minimum amount of oil (20 -25 gallons) is needed to cover the bricks. During processing, additional oil is added to the tank to prevent overheating. Once a total of 55 gallons of oil has been added to the tank – no matter how coarse the dispersion may be – processing cannot continue due to the risk of overheating. This is why it is so important to place the bricks at the bottom of the tank when loading.

Bricks and oil can be added the day before melting the sodium. This will allow the tank to be preheated overnight to 150 F prior to melting. Pre-heating can cut 4 hours off the melting process. CAUTION: Do not run mixer when pre-heating. Do not add additional oil if you do not plan to complete making dispersion that day or the tank may overheat. Prior to adding sodium brick addition to the Mix Tank, the operators must wear the proper protective equipment (PPE). The PPE requirements for this task are specific to the operations performed by the operators as follows:

Tank Loader – Chemical resistant rubber gloves (24 inches in length minimum), nitrile gloves, and clear safety glasses.

Assistant – Nitrile gloves. A bucket of mineral oil (<300 ppm water), and a diaper absorbent material to assist cleaning the Sodium Mix Tank and or the Tank Loader's gloves.



Procedure

- Operator Drain any condensation from the nitrogen gas line filter/dryer prior to making sodium dispersion in the Sodium Mix Tank.
- Operator There should already be 20 gallons of very dry oil in the sodium mix tank. If the Mix Tank is empty, add 20 gallons of very dry oil at this time.
- Loader Open the manual vent valve (if closed). Turn on Hi-Flow Nitrogen Purge valve.
- Loader Remove lock/bolt securing the Fill Hatch and carefully open the Fill Hatch. LOADER MUST HAVE GOOGLES AND A FACE SHIELD IN PLACE FOR THIS OPERATION!
- Loader Add 200 lbs. of sodium to the Mix Tank.

IMPORTANT: Stack the bricks as neatly and as low within the tank as possible so that no additional oil is needed to cover the bricks.

Assistant – Wipe off any sodium residue on loader's sleeve(s) and hatch seals. As he places the bricks into the tank with a sponge that has been dampened (not dripping!) with very dry mineral oil, then close and seal the hatch. This procedure must be continued throughout the tank loading process.

• Loader – Latch the Fill Hatch and secure it with a bolt or padlock.

HMI Steps

The following steps for Sodium Dispersion Formulation are controlled by the Operator using the HMI processing menus and screens. These steps involve adding mixed oil to the Mix Tank, pre-heating the Tank, adding dispersing agents to the Mix Tank, and melting the sodium to form dispersion.

Before implementing the following software procedures, make sure the appropriate chemical tank manual effluent valves and manual nitrogen supply valves are open. Also, make sure the nitrogen supply is sufficient to provide tank blanketing and to power pumps during these steps.

Adding Mix Oil – Procedure

- Make sure Nitrogen valve supplying support tanks and pumps are open. Open manual valve at the bottom of the Mix Oil Tank. Make sure Fill and Vent Valves on Sodium Mix Tank are open.
- Click on the [Add Mix Oil] button on the Sodium Processing Menu.
 - The Add Mix Oil to Na Mix Tank pop-up window appears.
 - Type in volume to fill or accept default (20 gallons). Keep in mind that you want to use the minimum amount of oil necessary to just cover the bricks.
 - Click the [Start] button.
 - Once the default is reached, the filling will be stopped automatically by the PLC program. Or, the operator can click the [Stop] button to halt the addition if less-than the accepted default amount is to be used because the bricks are well stacked in the bottom of the tank.
 - Click on the [Close] button to close the Add Mix Oil pop-up window.

Tank Pre-Heating – Procedure

- Click on the [Pre-Heat Na Mix Tank] button on the Sodium Processing Menu.
 - The Pre-Heat Na Mix Tank pop-up window appears.
 - Type in the pre-heat temperature set-point, typically 150 F.
 - Click the [Start] button and leave the window open while Pre-Heat is in progress (even if the Tank is to Pre-Heat overnight) so that the heater controls can be quickly accessed if necessary.
 - Click on the [Close] button to close the Add Mix Oil pop-up window.

Adding Mix Oil – to complete transfer of oil to Sodium Mix Tank

- Make sure Nitrogen valve supplying support tanks and pumps is open. Open manual valve at the bottom the Mix Oil Tank. Make sure the Fill and Vent Valves on Sodium Mix Tank are open.
- Click on the [Add Mix Oil] button on the Sodium Processing Menu.
 - The Add Mix Oil to Na Mix Tank pop-up window appears.
 - Type in volume required (2 gallons). On to two gallons of Mix Oil should be sufficient to clean-out the transfer line and make sure all additives are in the Sodium Mix Tank.
 - Click the [Start] button.
 - Once the default is reached, filling will be stopped automatically by the PLC program. Or the operator can click the [Stop] button to halt the addition if less-than the accepted defaults amount is to be used because the bricks are well stacked in the bottom of the tank.
 - Click on the [Close] button to close the Add Mix Oil pop-up window.

Melting Sodium

- Click on the Sodium Mix Tank Mixer icon atop the tank graphic on the HMI screen to turn ON the Mix Tank Mixer. (Red indicated the mixer is off. Green indicated the mixer is on.)
- Click on the [Melt Sodium] button on the Sodium Processing Menu.
 - The Melt Sodium on the Mix Tank pop-up window appears.
 - $_{\odot}$ Type in the temperature set-point. Initially the set Set-Point will be 175 F.
 - Click on the [Shell] button to display the temperature of the Mix Tank Shell.
 - As core temperature reaches 175 F, increase the shell set point by 10 F. As the next Set-Point is reached, continue increasing the Shell Set-Point by 5-10 F increments until the Core reaches 200 F.
 - IMPORTANT! The HMI software uses the displayed temperature to control the Shell Heater. Display ONLY the Shell temperature until the Core reaches 200 F to prevent overheating of the Mix Tank Shell.
 - Once the core reaches 210 F, TURN HEATER OFF. Residue shell heat and the exothermic reaction between the sodium and dispersion additives will complete the melt and warm the tank to at least 215-220 F. Watch temperature closely from this point on as it can increase quickly once the sodium has melted. IMPORTANT! Keep Jacket temp <250 F during heat-up and core temp <225 F at all times.

Tank Cooling

Once melt temperature (208-210 F) has been exceeded and the core and jacket reach equilibrium temperature, the exothermic reaction between molten sodium and the oil will continue to add heat to Mix Tank, **even with the heater turned off.** Temp must be maintained below 225 F, and preferably below 222 F. Adding 0.5 - 1.0 gallons of the MixOil to the Mix Tank when the core temperature reaches about 222 F will be sufficient to maintain the temperature at an optimal level. Be careful not to add so much cooling oil that the temperature drops below 215 F as this may cause the molten sodium to solidify in the tank.

The bottom section of the pop-up window provides cooling addition controls to regulate Tank temperature and prevent overheating. The following instructions pertain to preventing overheating of the tank and the controls are located the lower portion of the Melt Na in Mix Tank pop-up.

- Type in the Gallon to Add or accept the default (2 gallons).
- Click the [Start Add] button to transfer the preset Gallons to Add to the Sodium Mix Tank. Adding 0.5 – 1.0 gallons of Mix Oil can cool the tank between 1 and 3 F.
- The Gals Added counter tracks the gallons of Mix Oil transferred during the current addition. It automatically resets when each new transfer is started.
- Clicking the [Manual Stop] button can be used to stop the oil addition at any time during the addition.
- The Total Gals Added counter keeps track of all Mix Oil additions since the current sodium formulation batch was begun. A batch of sodium dispersion will require 55 gallons of oil during this step. The Gals remaining counter shows the Operator how many gallons are yet to be added of the 55 gallons required.

Monitoring Dispersion Particle Size

Before the operator can determine sodium particle size, they should become acquainted with the microscope used to measure particle size. The following diagram shows the basic components of the compound light microscope used to determine particle size.



Fig: Parts of Compound microscope

The microscope used for sodium particle size determination is equipped with 5X, 10X, 40X objectives. The 40X objective should be used for final particle size determination. The coarse adjustment knob should be used to bring the image nearly into focus. The fine adjustment knob is then used to make the image viewed in the ocular clear enough to accurately determine particle shape and size. The ocular has also been equipped with the measurement scale. See the following figure for details on how to use the scale for particle measurements. The diaphragm is outfitted with a light polarizing filter. This filter can aid in removing visual clutter from the sample. Such clutter is usually gas bubbles created when the sodium particles react with air and have become trapped between the slide and cover slide.

- Each hour for the first two hours of mixing, draw a sample of sodium dispersion from the Mix Tank using the Ram Seal sample valve. Thereafter, sampling every 30 minutes is suggested.
 - Allow enough dispersion to flow through the sample port to assure a fresh sample.
 - Catch a drop of sodium dispersion on a glass microscope slide.
 - Add a drop of clean mineral oil on top of the sodium dispersion drop to aid in viewing.
 - Place a cover clip slide over the fluids on the microscope slide.
 - Clip the prepared slide onto the microscope stage and observe the sodium particles.
 - If the average particle size is greater than 10um, continue mixing.
 - The ideal average particle size is <3 um.



Transfer Dispersion to Sodium Storage Tank

- Drain any condensation from the nitrogen gas line filter/dryer prior to transferring contents of the Sodium Mix Tank to the Sodium Storage Tank.
- Add 110 gallons of very dry, very good quality non-PCB oil to the Sodium Storage Tank.
- Click on the Sodium Mix Tank Mixer icon atop the tank graphic on the HMI screen to turn OFF the Mix Tank Mixer.
- Transfer the contents of the Sodium Mix Tank to the Sodium Storage Tank as follows.
 - Latch the Fill Hatch and secure it with a bolt or padlock.
 - Close the Mix Tank Vent, Transfer and Low Flow Nitrogen manual valves.
 - Open the High Flow Nitrogen manual valve.
 - When the pressure gauge shows 5 psig, open the the Transfer Valve to allow the Mix Tank contents to flow to the Storage Tank.
 - When the transfer is complete the pressure gauge will read nearly zero. Open the vent manual valve and close the High Flow Nitrogen manual valve.
- Add 55 gallons of oil to the Sodium Mix Tank according to the Adding Mix Oil procedure listed previously. NOTE: The Mix Oil Addition screen only allows additions up to 30 gallons at a time. Therefore, the Operator will have to add 30 gallons followed by an additional 25 gallons to make up the needed 55 gallons for this step.
- Click on the Sodium Mix Tank Mixer icon atop the tank graphic on the HMI screen to turn ON the Mix Tank Mixer. Allow the tank to mix for 15 minutes to rinse any sodium residue from the tank and mixer.
- Transfer the contents of the Sodium Mix Tank to the Sodium Storage Tank using the procedure indicated in the transfer step above.
- Click the mixer icon atop the Sodium Storage Tank on the HMI screen to turn ON the Sodium Storage Tank mixer.
 - Allow no more than 2 minutes of mixing to blend the concentrated sodium dispersion with the dilution and clean out oils.
 - Hot dispersion is susceptible to shear shock so do not over mix. Shear shock occurs when soft or molten sodium micro particles collide with similar particles creating larger, less reactive particles. In extreme cases the larger particles can reach several inches in diameter or larger.

- $_{\odot}$ The Sodium Storage Tank now contains 12.85% sodium dispersion ready for use in the NaX PCB dehalogenation process.
- Add 20 gallons of Mix Oil to the Sodium Mix Tank according to the Adding Mix Oil procedure listed previously.
- Purge the Sodium Mix Tank with nitrogen for at least 15 seconds to make positive the headspace is inert.
- Close manual valve at bottom of Mix Oil Tank. Close nitrogen valve supplying support tanks and pumps.

Refilling Sodium Storage Tank

NOTE: Before proceeding, make sure the Blue Oil Tank has sufficient volume of good quality dry (<300 ppm water), non-PCB oil to accomplish the refilling of Sodium Storage Tank, then open manual valves.

Procedure – for refilling the Sodium Storage Tank from the Blue Oil Tank

- Open the manual valves on the inlet side of the transfer pump beside the Blue Oil Tank.
- Open the manual valve atop the Sodium Storage Tank on the refill line.
- Click on the [Refill Na Pro Tanks] button on the Sodium Processing Menu.
 - The Refill Na Pros Tanks pop-up window appears.
 - Click on the [Add Oil to NaSto Tank] button.
 - Then Add Oil to NaSto Tank window appears.
 - Type in volume to fill or accept default (110 gallons).
 - Click the [Start] button.
 - Once the default is reached, filling will be stopped automatically by the PLC program.
 Or, the operator can click the [Stop] button to halt the addition if less than the accepted default amount is to be used because the bricks are well stacked in the bottom of the tank.
 - Click on the [Close] button to close the Add Oil to NaSto Tank pop-up window.





Drawing 14.1: Manual Valves for Refilling NaSto Tank

Reactor Processing Menus

NOTE: The procedures that follow, although they pertain to Reactor 1 processing, can be applied to processing Reactor 2 since the menus and equipment for both tanks are identical.

Fill Reactor 1

Procedure

- Record the total value on Tank 705 before proceeding.
- Open the manual valve on the Vacuum Dehydrator Unit to achieve the desired flow (75 gpm).
- Click the [Fill Reactor 1] button on the Reactor 1 processing menu.
 - The fill reactor pop-up window appears.
 - Click the [Start] button.
 - On the Vacuum Dehydrator, turn the pump to the ON position to begin filling Reactor 1.
 - Once you have filled Reactor 1 with your intended set amount, turn the pump OFF on the Vacuum Dehydrator.
 - Change the [Gals Added] to the number of gallons taken from 705's previous measurement to the existing measurement.
 - Click on the [Close] button to close the Fill Reactor pop-up window.
- Confirm volume transferred from 705 was the intended amount using the plastic sight glass.
- Close the manual valve on the Vacuum Dehydration Unit.

Reactor 1 In-Tank Heater

Each Reactor has an internal heater that is used to speed Reactor Pre-Heating and maintain Reactor temperature during NaX process. Reactors must contain >750 gallons minimum and an oxygen level of <3% to energize heaters.

Procedure

- Turn on Reactor 1 Mixer by clicking on the tank mixer icon atop the tank graphic on the HMI screen.
- Turn on Reactor 1 In-Tank Heater by clicking on the In-Tank Heater icon on the tank graphic.
 - \circ The In-Tank Heater pop-up window appears.
 - Type in set point temperature or accept default (220 F)
 - Click [Heater ON] button to engage the In-Tank Heater.
 - Click the [Close] button and the Tank Heater pop-up windown.
- Clicking on the In-Tank Heater again causes the pop-up window to appear so that the operator can observe the heater set-point, change the set-point, or to turn the In-Tank Heater OFF. If the operator elects to change the set-point, the heater must be stopped and re-started for the change to take effect.

Pre-Heat Reactor 1

- Make sure heaters are off by clicking on the [Heaters Off] button at the bottom of the screen and selecting [Stop].
- Click the [Pre-Heat Reactor 1] button on the Reactor 1 Processing Menu.
 - The Pre-Heat pop-up menu appears.
 - Type in set point temperature or accept default (220 F).
 - Click the [Start] button to engage the Circulation Heater (and pump).
 - When Reactor 1 is up to temperature (i.e. temperature set point is reached) the system automatically turns off the Circulation Heater.
 - Circulation Pump continues for two minutes after the heater is turned off.
 - At the end of the two minutes, pump automatically turns off and circulation valves close.
- Close the Preheat Reactor 1 pop-up window by clicking on the [Close] button.

Add Sodium Dispersion to Reactor 1

Do not add sodium until the preheat step is complete and oil is no longer circulating through the circulation heater. The In-Tank Heater can remain on throughout the sodium addition and subsequent dehalogenation processing to maintain the temperature of the oil within the Reactor.

- Open the manual valve at the bottom of the Sodium Storage Tank and the manual valve supplying nitrogen to the Sodium Storage Tank.
- Click on [Add Na to Reactor 1] button on the Reactor 1 Processing Menu.
 - The Add Na pop-up window appears.
 - Type in volume to fill or accept default (20 gallons).
 - Click on the [Start] button.
 - The Sodium Storage Tank vent valve is automatically closed. The Sodium Storage Tank pressurizes with nitrogen gas. When the pressure in the tank reaches 5 psig, the Sodium Storage Tank effluent valve automatically opens, the sodium valve atop Reactor 1 automatically opens, and sodium dispersion begins to flow to Reactor 1.
 - Once the volume to fill Set-Point is reached, [Start] button turns white and the [Stop] button turns red to indicate filling has been stopped automatically by the PLC program. The operator can also click [Stop] at any time to halt the addition of sodium to the Reactor.

- Close the Add Na pop-up window by clicking on the [Close] button when this operation is completed.
- Close the manual valave at the bottom of the Sodium Storage Tank and the manual valve supplying nitrogen to the Sodium Storage Tank if tou don't intend to treat more oil today.

Cleanout Sodium Dispersion Line with Dry Non-PCB Oil

Every three weeks, the Detox Operation's Manager will be held responsible for ensuring preventative maintenance is done by cleaning out the sodium dispersion line from the Sodium Storage Tank to the Reactor(s).

Procedure

- Add 60 gallons of dry (<300 ppm), non-PCB oil to the mix tank.
- Turn the mixer on.
- Turn the in tank heater to the Mix Tank.
 - Allow the temperature to rise to 140 degrees Fahrenheit and then turn off the heater. Not to exceed pass 170 degrees Fahrenheit.
- Allow the tank to mix for an additional 15-30 minutes to free up any potential residues along the walls.
- Turn the mixer off and begin to transfer to the Sodium Storage Tank.
 - NOTE: You have created a less potent batch of sodium dispersion so when adding sodium to the reactor(s), it will take more gallons than normal.

Testing to Confirm PCBs in Reactor 1 have been Destroyed

Procedure

- Mix Reactor 1 for at least 45 minutes after the sodium dispersion has been added.
- Sample and test Reactor 1 contents by using the GC. Once the PCB concentration is less than 2.0 ppm, the contents of Reactor 1 is ready for transfer to the Quench Tank.

Transfer Reactor 1 Contents to Quench Tank

The In-Tank Heater must be allowed time to cool to prevent damage to the heating elements. Then the oil can be transferred to the Quench Tank for subsequent processing.

- Turn OFF In-Tank Heater.
- Allow tank to continue mixing for at least 5 minutes after the In-Tank Heater is turned OFF to make sure heater has cooled sufficiently before transferring contents to Quench Tank.
- Turn off the mixer in Reactor 1.
- Allow the Reactor contents several minutes to settle prior to transfer to Quench Tank. This settling reduces the chances of sodium residue remaining in the tank once it has been emptied.
- Click on the [Transfer Reactor 1 to Quench Tank] button on the Reactor 1 Processing Menu.
 - The Transfer Reactor to Quench Tank pop-up window appears.
 - Click on the Gallons Added] button.

• Click on the [Start] button.

NOTE: As the Reactor empties, the control software automatically introduces a 5second blast of inert gas to keep the tank pressure above 2 inches of

water column. These short blasts typically occur every 10-15 seconds during emptying. Automatic transferring continues until 20 seconds after the flowmeter between the Reactors and Quench Tank no longer senses flow. The operator should confirm the tank is empty by observing that it has been more than 30 seconds since the last inert gas blast and or the pressure gauge on the effluent pipeline reads zero while the pump is on. The operator can then click the [Stop] button to halt the transfer from the Reactor.

• When the transfer is complete, close the Transfer Reactor to Quench Tank pop-up window by clicking the [Close] button.

Add 100 Gallons of PCB Oil or Refill Reactor

If the Reactor is not to be used immediately after it has been emptied, it must be partially refilled to cover with oil any sodium residue that may remain at the bottom of tank. Covering this residue prevents air from reacting with any sodium residue. Sodium generates hydrogen gas as it reacts with air. This gas can be explosive! DO NOT LEAVE A REACTOR COMPLETELY EMPTY WHEN NOT IN USE!

Procedure

- Record the total value on Tank 705 before proceeding.
 - Open the manual valve on the Vacuum Dehydrator Unit to achieve the desired flow (75 gpm).
 - Click the [Fill Reactor 1] button on the Reactor 1 processing menu.
 - The fill reactor pop-up window appears.
 - Click the [Start] button.
 - On the Vacuum Dehydrator, turn the pump to the ON position to begin filling Reactor 1.
 - Once you have filled Reactor 1 with your intended set amount, turn the pump OFF on the Vacuum Dehydrator.
 - Change the [Gals Added] to the number of gallons taken from 705's previous measurement to the existing measurement.
 - Click on the [Close] button to close the Fill Reactor pop-up window.
- Confirm volume transferred from 705 was the intended amount using the plastic sight glass.
- Close the manual valve on the Vacuum Dehydration Unit.

Sparge Contents of Quench Tank with Inert Gas

The sodium residue in the oil is converted to inert salts by sparging with inert gas. Sparging also reduces the amount of hydrogen has generated during the subsequent water addition step. Make sure the inert gas flow rate is at least 4 cfm throughout purging to get the most efficient transfer gas into the oil.

- Turn on the Quench Tank Mixer by clicking on the tank mixer icon atop the tank graphic on the HMI screen.
- Click on [Neutralize Treated Oil] button on the QT Processing Menu.
- Click on the [Sparge Quench Tank] button on the Neutralize Oil Menu.
 - The sparge pop-up window appears.

- Type in the desired sparge time or accept the default (60 minutes).
- Click on the [Start] button. The sparge timer counts down from the set point sparge time until it reaches zero. At zero, the sparge valve closes, but the oil continues mixing in the Quench Tank.
- Test the contents of the Quench Tank to determine if sufficient inert gas has sparged into the oil. PROCEDURE – Draw a 100 ml sample from the Quench Tank and add 2 ml of water. Mix thoroughly. If the oil reactes over 150 ml, continue sparging for an additional hour until the oil does not react over 150 ml.

Add Water to Quench Tank

Water is added to the oil to wash out the salts generated in the dehalogenation and sparging processes. This water also pulls a great deal of the color out of the oil, leaving clear, bright yello oil. Water addition generates hydrogen gas. The operator should ADD WATER SLOWLY at first. Start with 0.5 gallon additions every 5-15 minutes until at least 3-5 gallosn of water have been added. Larger additions of water may follow once the residual sodium has been reacted. If the temperature increases, the water is reacting with the sodium. If the temperature decreases, the water is cooling the oil.

Procedure

- Turn on the Qunehc Tank Mixer by clicking on the tank mixer icon atop the tanke graphic on the HMI screen.
- Record the Totalizer Value on the Water Meter before procedding.
- Open the manual water valve to about 1/4 to 1/2 of full open position.
- Click on the [Add Water to Quench Tank] button.
 - The Add Water pop-up window appears.
 - Type in the volume of water to add or accept default (0.5 gallons).
 - Click on [Start] button.
 - When the set point is reached, the water addition automatically stops.
 - Subsequent additions of water can be made after allowing sufficient mix time.
 - Typically, 25% water is added to the total gallons of sodium used in the Reactor processing stage. 20 gallons of sodium would be 5 gallons of water. For ultimate safety, the operator will do a minimum of 6 gallons of water.
 - Click the [Close] button to close the Add Water pop-up window when sufficient water has been added.
- Record the total value on the water meter. Compute actual gallons of water added by subtracting the initial value from the final value.
- Allow the tank to mix for 15-30 minutes after the last water addition in order to be sure all sodium has reacted.

Transfer Contents of Quench Tank to Clean Oil Storage Tanks

The Quench Tank may contain residual water from prior batch processing. THIS WET OIL MUST BE REMOVED FROM THE TANK PRIOR TO ADDING HOT, SODIUM ENRICHED OIL FROM THE REACTORS. Otherwise, the moisture will immediately react with the sodium and hot oil to produce hydrogen gas and steam and over pressurization of the Quench Tank could occur. Operator MUST transfer any wet oil in the Quench Tank to Clean Oil Storage prior to introduction of hot oil from Reactors.

Procedure

The Quench Tank oxygen concentration must be less than or equal to 3% before a Reactor can be transferred into the Quench Tank's headspace with inert gas using short (15 second) blasts every 5 minutes until the oxygen concentration is within safe limit.

- Click on the [Transfer to Storage] button. A caution window pops up to remind the operator to check the Manual Transfer valve located at the Quench Tank effluent pump. Close the caution window when the valve status has been confirmed.
- Select the destination Clean Oil Storage Tank by clicking on the desired [Transfer Quench Tank To...] button on the Post Treatment menu screen.
 - The Transfer to... pop-up window opens.
 - Verify the volume in the Quench Tank is properly posted on the Transfer pop-up window. If the value is incorrect, it can be changed on the Process Hardware Status screen.
 - The transfer function is equipped with an Auto Stop Override. To engage this feature, click the [Override] button on the Transfer QT to... window. A caution statement will pop up so the Operator can confirm his intention to override the automatic stop.





- Click the [Start] button on the Transfer pop-up window to begin emptying the Quench Tank. Initially, the Quench Tank's inert gas purge will be turned on to build a slight positive pressure in the Quench Tank. After an 8 second delay, if the pressure on the Tank is positive and oxygen concentration is less than or equal to 3%, the effluent valve will open and the transfer pump will turn on.
- Emptying continues until the counter reaches zero or the operator interrupts the process by clicking on the [Stop] button on the Transfer pop-up window. Emptying will also automatically stop if oxygen concentration exceeds 3% or the inert gas purge cannot maintain positive pressure on the Quench Tank. Note: The gallons transferred value displayed on the window shows the volume of oil removed from the Quench Tank. The operator can confirm that the transfer is complete when the gallons transferred displayed value stops changing and when the effluent pump flow rate is zero.
- Close the Transfer pop-up window by clicking on the [Close] button.
- Close the Transfer Quench Tank to... pop-up Menu by clicking on the Menu's [Close] button.

Add Clean Oil to Quench Tank

Once emptied, if the Quench Tank is not to be used again immediately, transfer 300 gallons of dry non-PCB oil into the Quench Tank after emptying. This practice can prevent any un-reacted sodium residue at the bottom of the Quench Tank from being exposed to air, which could create hydrogen gas.

- Mark the sight tube on the non-PCB oil tank at initial volume and target volume (4" represents approximately 200 gallons using a 700's non-PCB oil tank sight tube.)
- Open correct manual valves to route oil from the non-PCB oil tank to the transfer pump.
- Set transfer pump station in Auto mode. Auto/Manual mode selector switch is located on the lower right side of the pump station control panel.
- Click on [Add Oil to Quench Tank] button.
 - The Add Oil pop-up window appears.
 - Type in volume to add or accept default (300 gallons).
 - Click on the [Start] button.
 - Once the default is reached, the [Start] button turns white and the [Stop] button turns red to indicate filling has been stopped.
 - Click on [Stop] button to stop adding oil before the volume to add is reached.
 - Click the [Close] button to make the Add Oil pop-up disappear.
- Confirm oil level in non-PCB oil tank is at or near target volume indicator on sight tube.
- Close manual valves routing oil from non-PCB oil tank to transfer pump.
- Set transfer pump station in Manual mode.
- Each morning, transfer 5-10 gallons from QT to 600 and 601 to flush any wet oil and salt from the prior day's processing. This will prevent blockages due to salt build up.

Tank Dimensions

Reactors R1 and R2



Height - 6' Diameter - 6' Cone Depth - 1' (130 gallons ea) Gallons per Inch - 17.4 gp1 Working Volume - 1100 gallons Notes - RTD at 300 gal Sample Port at 400 gal Heater RTD at 600 gal New 9-08, 3/8" steel



Height - 9'

Diameter - 8'

Cone Depth – 1'-4" 320 gallons in cone

Gallons per Inch - 31.5 gpi (cylinder section)

Working Volume – 2000 gallons (4000 gallons max.)

Notes - Steel, New 7-01

PCB Oil Storage - 300 through 304



Height - 12'

Diameter - 12'

Gallons per Inch - 71 gpi

Working Volume – 8,000 gallons (10,165 gallons max.)

Notes - Steel, New 8-01



Gallons per Inch - 48 gpi

Working Volume – 4800 gallons (6000 gallons max.)

Notes - Steel, New 8-01

Clean Oil Storage - 700 through 704



Height - 15'

Diameter - 10'

Cone Depth – 4' ~ 450 gallons in cone

Gallons per Inch – 48 gpi

Working Volume – 4800 gallons (6000 gallons max.)

Notes - Steel, New 8-01

Mix Oil Tank	Height - 5'
	Diameter - 3' (260 gal)
	Cone Depth - 0'-6" (16 gal)
	Gallons per Inch - 4.33 gpi
	Working Volume – 230 gal
	Notes - Steel, New 9-08
Sodium Storage Tank	/ Height - 5'
	Diameter - 3'-6" (350 gal)
	Cone Depth – 0'-7" (25 gal)
	Gallons per Inch - 5.8 gpi
	J Working Volume – 320 gal
	Notes - Steel, New 8-01
Sodium Mix Tank	Height - 3'
	Diameter - 2'-6" (110 gal)
	Cone Depth – 0'-5"
	Gallons per Inch - 3 gpi
	Working Volume – 110 gallons
	Notes – Keep fill-hatch closed and secured with a padlock or bolt when not in use. Steel, New 8-01
"Blue" Oil Tank	Height - 6'-6"'
	Diameter - 4'-6' (820 gal)
	Cone Depth – 0'-6" (75 gal)
	Gallons per Inch - 10.5 gpl
	Working Volume – 775 gal (970 gal total)
	Notes - Steel



<u>Safety</u>

Disclaimer: The following information is intended to alert the operator to the importance of workplace safety. This information is not intended to equip the operator to workplace safety. The Occupational Safety and Health Act of 1970 requires employers to provide workplaces free from serious recognized hazards and to comply with occupational safety and health standards. Consult your company's EHS Manager immediately to acquire the necessary training and equipment to safeguard your health and safety.

Chemical Safety

Chemical use has the potential to harm the health and safety of people in the workplace. The term use encompasses the production, handling, storage, transport, and disposal of substances in the workplace. A chemical can be in any form (solid, liquid, or gas). Chemical by-products or mixtures may also be regarded as hazardous if they contain ingredients that are hazardous and are in concentrations above defined cut-off levels. If chemicals are not properly managed, the effects on health and associated costs can be significant.

The health risk in handling any particular hazardous substance will vary with each substance and the way in which the substance is used. Factors such as route of exposure, type of disease, or injury, the relative toxicity and concentration of the hazardous substance is used. Factors such as route of exposure, type of disease or injury, the relative toxicity and concentration of the hazardous substance are required to be considered in the assessment. Chemical container labels and Safety Data Sheets contain information on chemicals. The SDS for all chemicals may be found in the Detox laboratory. For immediate concerns, please feel free to contact the EHS Manager. The SDS for a substance describes its identity, relevant health hazard information, precautions for use safe handling information, and first aid measures.

The operator should read, understand, and follow the guidelines listed within the SDS for each chemical used in the lab or the NaX Process before attempting to use or handle the chemical or its container. The operator should pay particular attention to the SDS sections pertaining to PPE. Do not handle any chemical until you understand the following:

- What PPE is necessary;
- How to properly put on, take off, adjust and wear the PPE;
- The limitations of the PPE; and,
- Proper care, maintenance, useful life and disposal of PPE.

Electrical Safety

Working with electricity can be dangerous. Licensed electricians and plant maintenance professionals work with electricity directly, including working on overhead lines, cable harnesses, and circuit assemblies. Others, such as laboratory technicians and process operators, work with electricity indirectly and may also be exposed to electrical hazards.

Many workers are unaware of the potential electrical hazards present in their work environment, which makes them more vulnerable to the danger of electrocution. The following references aid in recognizing hazards associated with electrical work.

Curnt passing through your body can cause electrical shock, resulting in 3 types of potential injuries:

- 1. Burns (arcs burn with heat and radiation);
- 2. Physical injuries (broken bones, falls, and muscle damage). For example, at 10 mA, the muscles clamp on to whatever the person is holding. Larger current can cause severe burns, blindness, and other injuries.
- 3. Nervous system effects (stop breathing at 30 to 75 mA alternating current 60Hz, fibrillation at 75 to 100 mA at 60Hz). Fibrillation = heart is "twitching" and there is no blood flow to the body.

NOTE: The heart can be damaged because it is in the path of the most common routes electricity will take through the body:

- o Hand to hand
- o Hand to foot

WARNING! If you are not specifically trained to perform electrical installations, troubleshooting, or maintenance, do not attempt these activities. Instead, contact your supervisor, your company's electrical maintenance, or a licensed electrician.

If you are trained to perform electrical installations, troubleshooting and maintenance, be sure to observe all warnings and caution posted on the various electrical devices. Also, be sure to read, understand and follow all applicable technical specifications, manuals and safety guides associated with the equipment prior to conducting these activities.

Troubleshooting

In the majority of cases, and equipment malfunction can be traced to a disturbance in the control logic or feedback circuit. The effects of these disturbances can easily be cleared by resetting the PLC. The sequence for resetting the PLC must be performed as follows: (1) turn off power to PLC-02 cabinet using the key switch on the side this cabinet; (2) turn off power to the Sodium Room PLC using the key-switch on the side of this cabinet; after waiting about 10 seconds, (3) turn on power to the Sodium Room PLC using the key switch on the side on the side of the cabinet; then, (4) turn on power to PLC-02 cabinet using the key switch on the side of the cabinet. Within a few moments, the

computer display window should report correct values for temperatures, volumes, and pressures. Confirm the PLC is fully functioning by clicking on the tank purge buttons. The operator should be able to hear gas entering the tank being purged if the PLC is back online.

If the previous procedure does not return function to all process equipment, the operator should check to see that all the necessary breakers disconnect(s) are turned on. Confirm gas lines have pressure and manual gas valves are open. It may become necessary to restart the process control computer. Exit out of the control software and restart the computer. When the computer restarts, reload the process control software.

If these measures fail to resolve the malfunction, additional troubleshooting may be required.

Before the operator begins to troubleshoot any piece of equipment, they must be familiar with TDS's safety rules and procedures for working on electrical equipment. These rules and procedures govern the methods that can be used to troubleshoot electrical equipment (including lockout/tagout procedures, testing procedures, etc.) and must be followed while troubleshooting.

The operator SHOULD NOT open PLC, starter or heater controller cabinets, valve actuators, process sensors, or any other device or electrical junction box when the items contained within them or connecting wire are energized.

Next, the operator needs to gather information regarding the equipment and the problem. It is also important to understand how the equipment is designed to operate. It is much easier to analyze faulty operation when you know how it should operate. Make notes such as briefly describing the event, the time of the event, processing step(s) being performed at the time of the event, and equipment or reading affected. The operator should also note any steps taken to correct the condition such as manually adjusting regulators or flow meters, closing, or opening manual valves, resetting the PLCs, etc.

Troubleshooting – Supplemental Info for TDS

The basic steps involved in trouble shooting consist of the following:

- 1. Observe
- 2. Define the problem area
- 3. Identify possible causes
- 4. Determine the most probable cause
- 5. Test and repair
- 6. Follow up
- 1. OBSERVE

Most faults provide obvious clues as to their cause. Through careful observation and a little bit of reasoning, most faults can be identified as to the actual component with very little testing. When observing malfunctioning equipment, look for visual signs of mechanical damage such as indications of impact, chafed wires, loose components or parts lying in the bottom of the cabinet. Look for signs of overheating, especially on wiring, relay colls, and printed circuit boards.

Don't forget to use your other senses when inspecting equipment. The smell of burnt insulation is something you won't miss. Listening to the sound of the equipment operating may give you a clue to where the problem is located. Checking the temperature of components can also help find problems but be careful while doing this, some components may be alive or hot enough to burn you.

Pay particular attention to areas that were identified either by past history or by the person that reported the problem. A note of caution here: Do not let this information mislead you. Past problems are just that – past problems, they are not necessarily the problem you are looking for now. Also, do not take reported problems as fact; always check for yourself if possible. The person reporting the problem may not have described it properly or may have already made his own incorrect assumptions.

When faced with equipment which is not functioning properly you should:

- Be sure you understand how the equipment is designed to operate. It makes it much easier to
 analyze faulty operation when you know how it should operate. Equipment installation and
 operation manuals are located in a notebook in the Process Operator's Office;
- Note the condition of the equipment as found. You should look at the state of the relays (energized or not), which lamps are lit, which auxiliary equipment is energized or running etc. This is the best time to give the equipment a thorough inspection (using all your senses). Look for signs of mechanical damage, overheating, unusual sounds, smells etc.;
- Test the operation of the equipment including all of its features. Make note of any feature that
 is not operating properly. Make sure you observe these operations very carefully. This can
 give you a lot of valuable information regarding all parts of the equipment.

2. DEFINE THE PROBLEM AREA

It is at this stage that you apply logic and reasoning to your observations to determine the problem area of the malfunctioning equipment. Often times when equipment malfunctions, certain parts of the equipment will work properly while others not. The key is to use your observations (from step 1) to rule out parts of the equipment or circuitry that are operating properly and not contributing to the cause of the malfunction. You should continue to do this until you are left with only the part(s) that if faulty, could cause the symptoms that the equipment is experiencing.

To help you define the problem area you should have a sketch or diagram of the processing area in addition to your noted observations.

Starting with the whole process diagram as the problem area, take each noted observation and ask yourself, "what does this tell me about the process operation?" If an observation indicates that a section of the process appears to be operating properly, you can then eliminate it from the problem area. As you eliminate each part of the circuit from the problem area, make sure to identify them on your diagram. This will help you keep track of all your information.



Fig AIII-4: Areas of NaX Process where fault/failure is occurring.

 IDENTIFY POSSIBLE CAUSES Once the problem area(s) have been defined, it is necessary to identify all the possible causes of the malfunction. This typically involves every component in the problem area(s).

> It is necessary to list (actually write down) every fault which could cause the problem no matter how remote the possibility of it occurring. Use your initial observations to help you do this. During the next step you will



Fig AIII-5: PLC-02 Devices can be responsible for faults and failures.

eliminate those which are not likely to happen.

4. DETERMINE MOST PROBABLE CAUSE

Once the list of possible causes has been made, it is then necessary to prioritize each item as to the probability of it being the cause of the malfunction. The following are some rules of thumb when prioritizing possible causes.

Although it could be possible for two components to fail at the same time, it is not very likely. Start by looking for one faulty component as the culprit. The following list shows the order in which you should check components based on the probability of them being defective:

- First look for components which trip, burn-out or have a tendency to wear . out, i.e. mechanical switches, fuses or circuit breakers, relay contacts, or light bulbs. (Remember, in the case of fuses and circuit breakers, they "pop" for a reason. You should find out why before replacing/resetting them.)
- The next most likely causes of failure are coils, motors, transformers and . other devices with windings. These usually generate heat and, with time, can malfunction.
- Connections should be your third choice, especially screw type or bolted type. Over time these can loosen and cause a high resistance. In some cases this resistance will cause overheating and eventually will burn open. Connections on equipment that is subject to vibration are especially prone to coming loose.
- Electrical noise or interference can cause problems with analog signals from telemetry. Make sure all signal wires are shielded and the shield tap is grounded. Also, improper grounding and ground loops can cause problems. Neutral to Ground voltage potential should be less than 5 VAC. Higher values can indicate poor or absent grounding. USB to serial connections require isolation to prevent ground loops.
- Finally, you should look for is defective wiring, especially on a new piece of equipment.. Pay particular attention to areas where the wire insulation could be damaged causing short circuits. Wire size is another important area to consider when tracking down electrical faults. Undersized wiring can cause thermal overloads on motor starters to trop.

5. TEST AND REPAIR

Testing electrical equipment can be hazardous. The electrical energy contained in many circuits can be enough to injure or kill. Testing and repairs MUST ONLY BE PERFORMED BY TRAINED PERSONNEL. If you have not been trained in testing, repairing or maintaining electrical equipment, DO NOT ATTEMPT THESE ACTIVITIES. When trained personnel do perform these activities, make sure to follow all your company's safety precautions, rules and procedures while troubleshooting.

Once you have determined the most probable cause, you must either prove it to be the problem or rule it out. This can sometimes be done by careful inspection however, in many cases the fault will be such that you cannot identify the problem component by observation and analysis alone. In these circumstances, test instruments can be used to help narrow the problem area and identify the problem component.

The multimeter is a general testing device which can be used on most electrical equipment and is the troubleshooting tool of choice at this point. A typical multimeter (see example to right) can measure AC and DC Voltages, Resistance, and Current. A very important rule when taking meter readings is to

Fig AIII-6: Thermal Overload



Fig AllI-7: Motor Starter



Fig AIII-8: Multi-Meter

predict what the meter will read BEFORE taking the reading. Select the proper meter scale – generally 10X the expected reading. If the reading is anything other than your predicted value, you know that this part of the circuit is being affected by the fault.

Use a "divide and eliminate" testing approach to eliminate parts from the problem area. The results of each test provide information to help you reduce the size of the problem area until the defective component is identified.

Once you have determined the cause of the fault you can proceed to replace the defective component. BE SURE THE POWER FEEDING THE COMPONENT AND ALL OF ITS CONTROLS IS LOCKED OUT AND YOU FOLLOW ALL SAFETY PROCEDURES BEFORE DISCONNECTING THE COMPONENT OR ANY WIRES.

After replacing the component, you must test operate all features of the component to be sure you have replaced the proper component and that there are no other faults in the process or circuit.

6. FOLLOW UP

Although this is not an official step of the troubleshooting process it nevertheless should be done once the equipment has been repaired and put back in service. The Operator and Trinity Technician should try to determine the reason for the malfunction:

- Did the component fail due to age?
- Did the environment the equipment operates in cause excessive corrosion?
- Are there wear points that caused the wiring to short out?
- Did it fall due to improper use?
- Is there a design flaw that causes the same component to fail repeatedly?

Addressing these questions can prevent or minimize further failures.