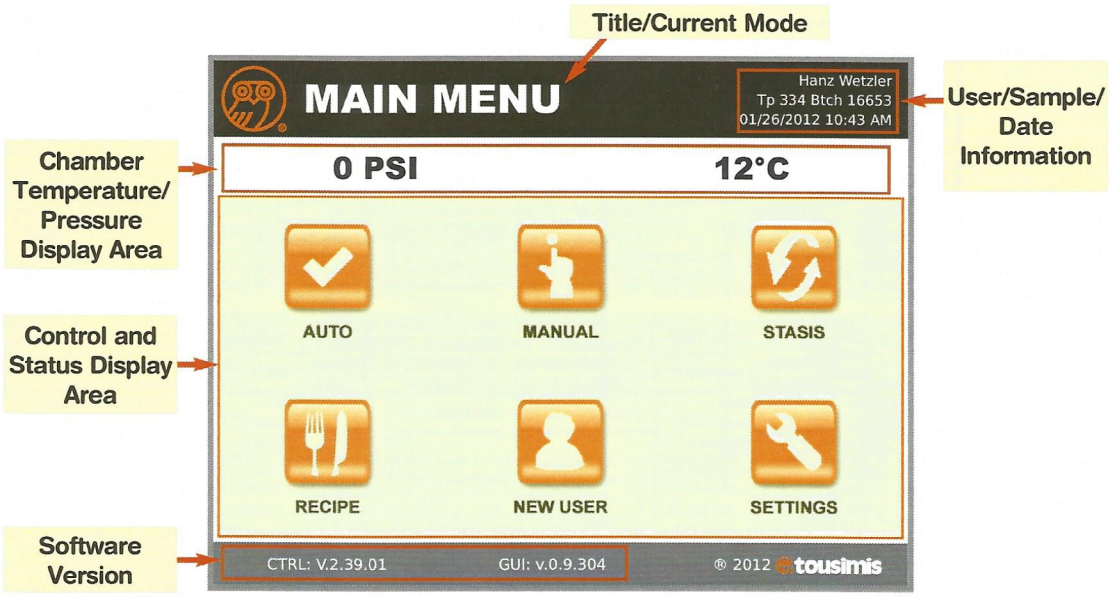
CHAPTER 4

tousimis® 931 Operation

4



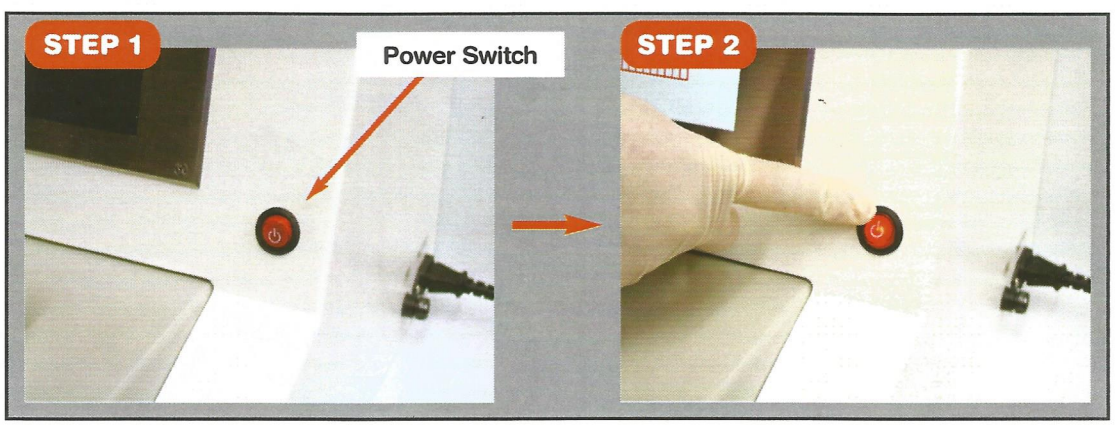
tousimis® 931 Touch Screen Display Overview

4.1 Start Up

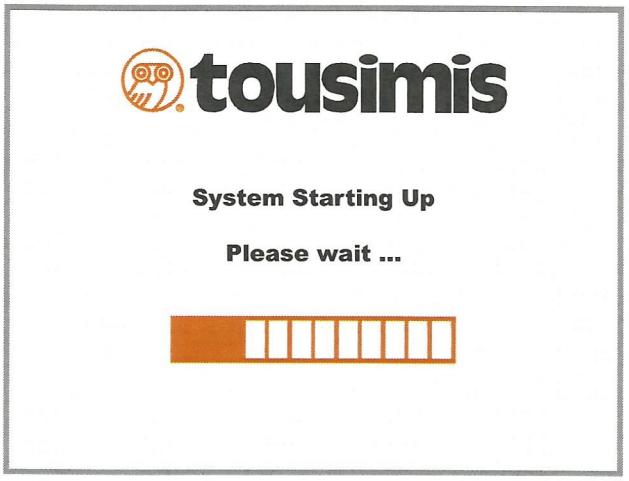
1. All 5 metering valves (COOL, SLOW FILL, FILL, BLEED, PURGE/VENT) have been factory preset (See Chapter 5 Illustration for Metering Valves).
There is no need to adjust these Metering Valves.
Open the main LCO_2 Tank valve. The Syphon (Dip) Tube LCO_2 Tank should have between 25 lbs - 50 lbs (12.5 kg -25 kg) net weight of LCO_2 in order to operate the 931.
(Subtract the tare weight of the tank from the tank weight to get the net weight of LCO_2).

2. Turn power switch ON.

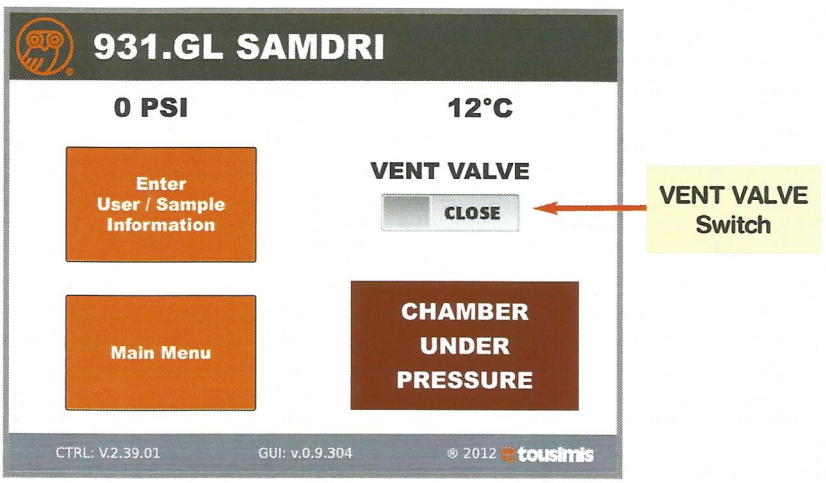
NOTE:
Pressure measurement is not indicative of the amount of LCO_2 remaining in tank. The same "PSI" reading may be noted even after the liquid CO_2 drops below the level of the Syphon (Dip) Tube.
Weight is the best way to properly indicate remaining LCO_2.



3. The 931 will start up with the following screen display.



4. The 931 is ready to operate when the following screen is displayed.



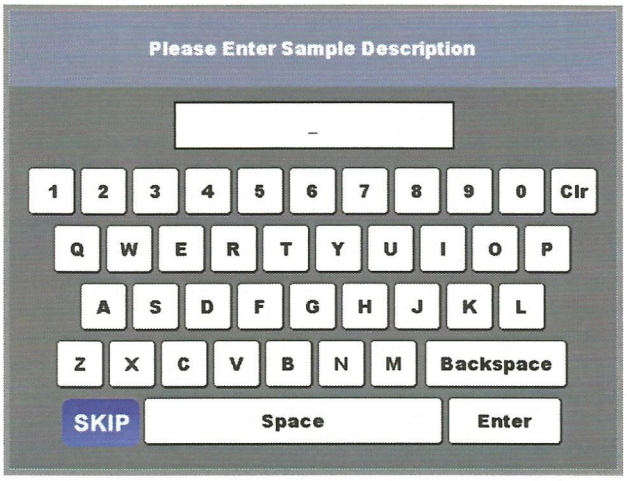
Press "Enter User/Sample Information" to enter User/Sample Information.
You may also press "Main Menu" to start operation without entering User & Sample Information (See #5 & #6).

If PROCESS CHAMBER is under pressure (above 50 PSI), the 931 will display "CHAMBER UNDER PRESSURE."
Press VENT VALVE Switch to open VENT VALVE in order to start a new process run.

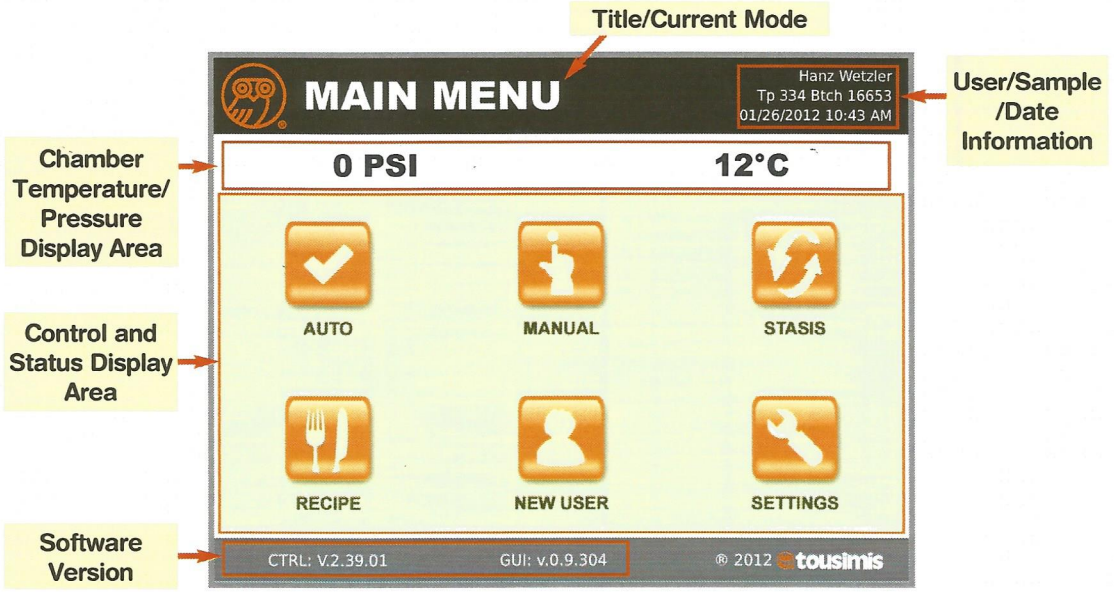
If the previous process run was interrupted by any unexpected reason such as a power outage during the process run, DO NOT OPEN VENT VALVE. You may resume the rest of process run steps using "Manual Mode" from Main Menu screen (#7).

5. Enter User Name or ID (maximum 18 spaces long) and press Enter.

6. From the following screen, Enter Sample ID or Description (maximum 18 spaces long) and press Enter.

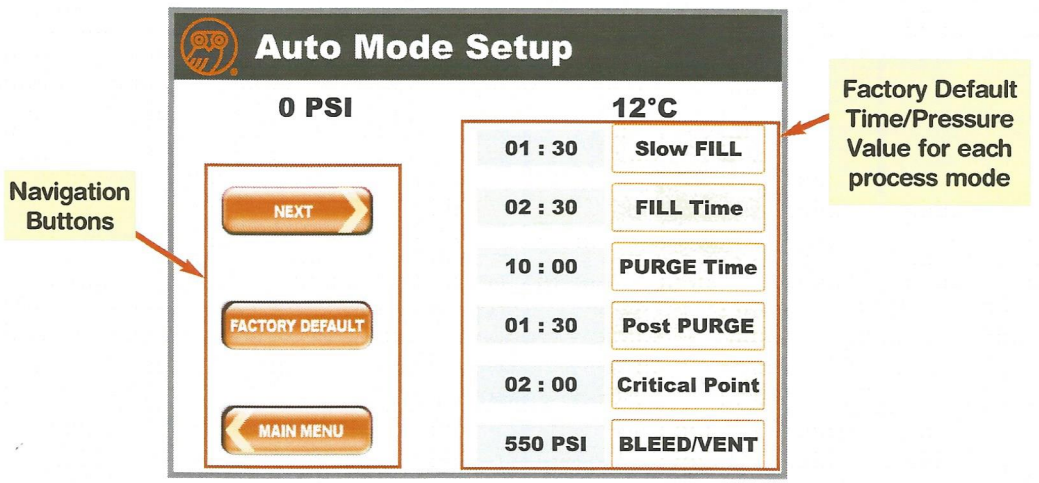


7. Main Menu will then be displayed. Press any button to initiate desired mode.



4.2 Auto Mode

1. Factory Default Time and Pressure Values may be used or updated from the Auto Mode Setup Screen.



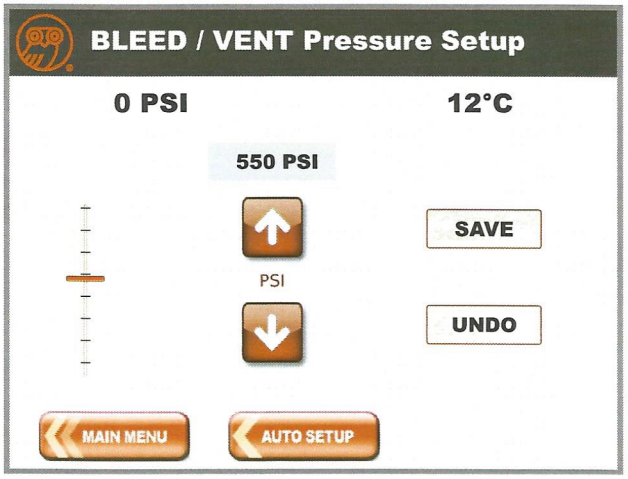
Press NEXT to advance screen or to modify preset values press appropriate buttons.
For example, to modify "Slow FILL" time, press Slow FILL button; to update BLEED/VENT Pressure, press BLEED/VENT button.

Press NEXT button to start "Auto Mode."

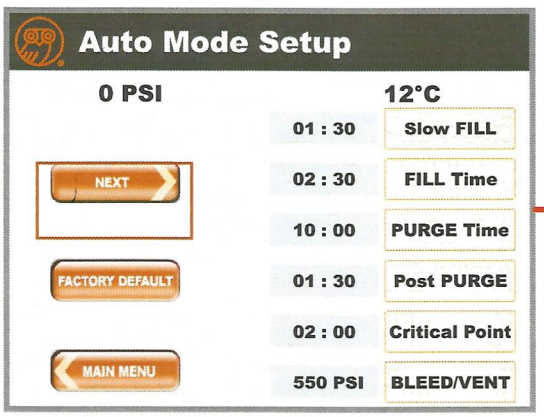
To restore tousimis® recommended preset values, press FACTORY DEFAULT button.
Exit the mode by pressing MAIN MENU button.

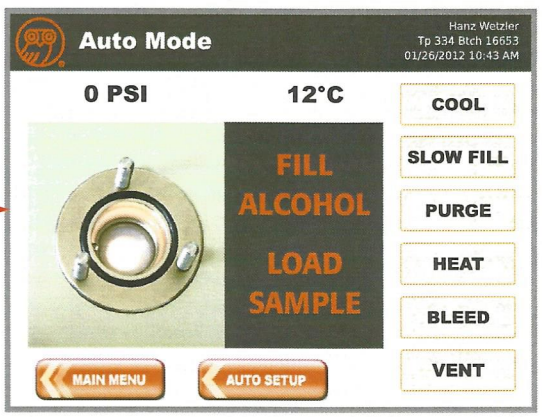
2. To update Time, use arrow buttons or slide bar to change value and press SAVE.
UNDO button restores tousimis® recommended default values for typical process run.
Press MAIN MENU or AUTO SETUP button to exit without saving.

3. To update BLEED/VENT Pressure, use arrow buttons or slide bar to change value and press SAVE. UNDO button restores tousimis® recommended value for typical process run.
Press MAIN MENU or AUTO SETUP button to exit without saving.



4. Press NEXT button from AUTO MODE SETUP page once Time/Pressure modifications are input to satisfaction.



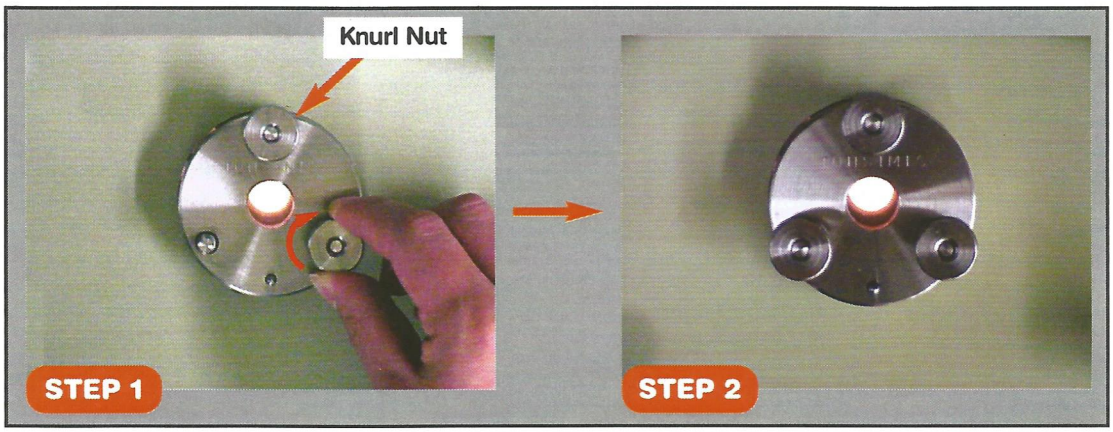


NOTE:
* Use Ultrapure Alcohols only!
(i.e. I.P.A., Methanol, Ethanol).

Use minimum 99.5%+ purity Alcohols for best results.

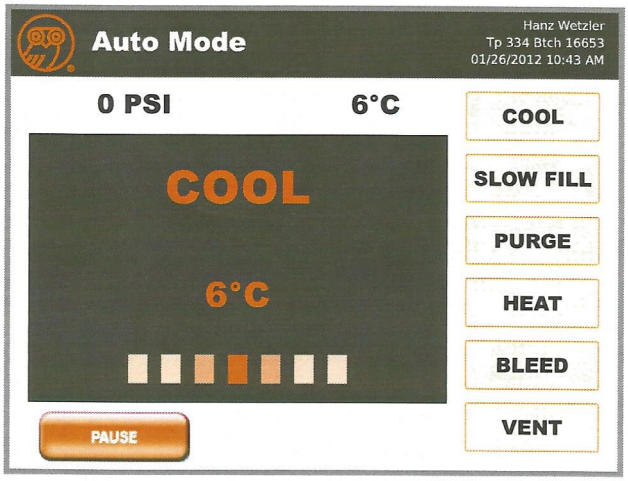
5. Pour sufficient alcohol into process chamber (Typically 10-15ml) to ensure complete sample coverage. Next, transfer the sample holder into the open process chamber.
Make the transfer quickly in order to avoid the samples' exposure to air and moisture.
The chamber O-Ring should lie completely in its groove. NEVER grease the O-Ring. Place the chamber lid down over the chamber using the 3 knurl nuts. Tighten each knurl nut hand tight with equal pressure. Once lid is secure press COOL button.

NOTE:
DO NOT introduce Acetone into the Process Chamber unless Acetone Kit (#8770-91) has been previously installed.

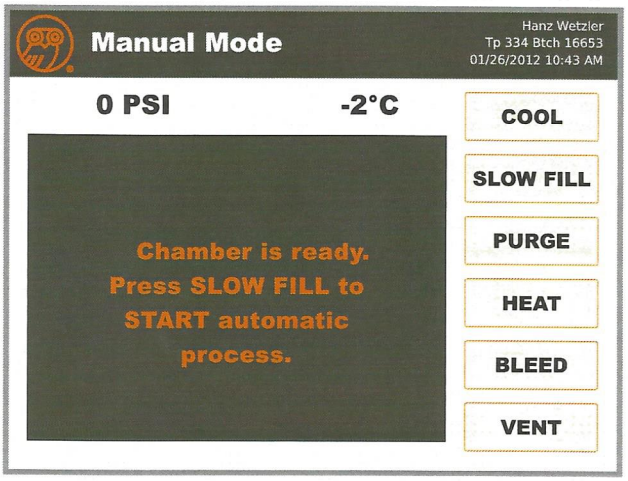


NOTE:
Never use channel locks or pliers for tightening the knurl nuts.
Always hand tighten!

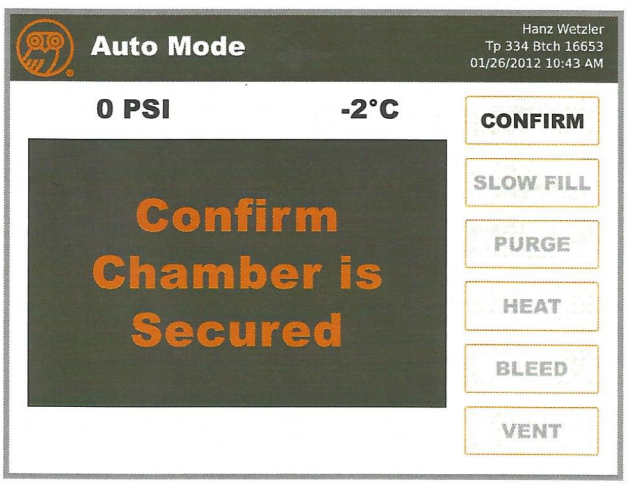
6. As the chamber temperature slowly begins to drop, you may hear the LCO_2 circulating through the unit. The 931 will continue cooling by itself until the chamber temperature reaches 0°C (±5°C).



7. At this cut off point, the cooling will automatically stop and display the following message:

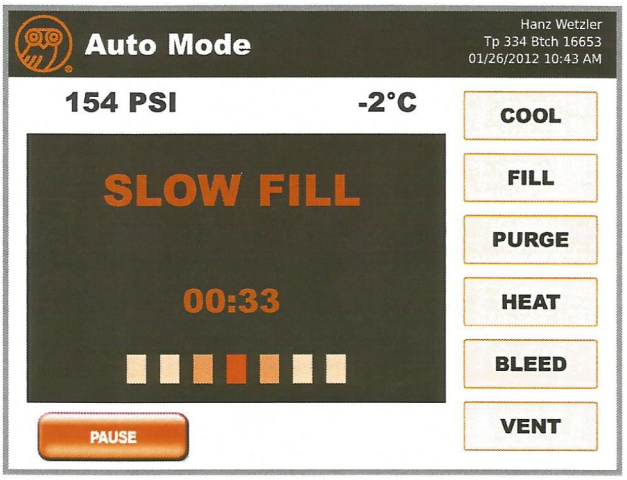


8. Press the SLOW FILL button to advance. The 931 requires confirmation of chamber security to further continue process. Double check chamber security and press CONFIRM.

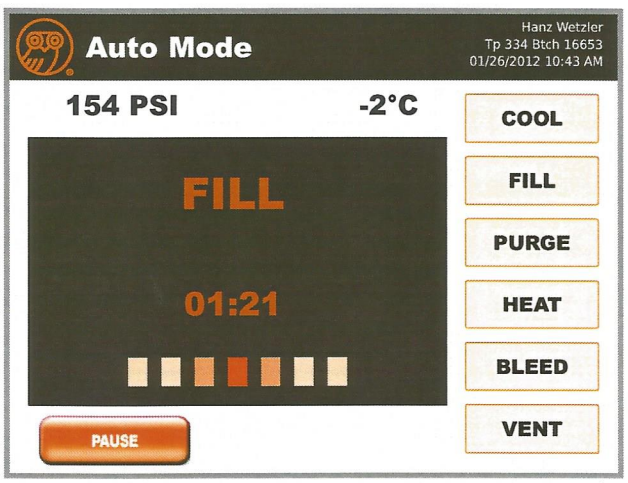


NOTE
PAUSE button will pause operation and give option to change preset value during process run. See Chapter 4.5 "Pause & Resume during Process Run."

9. The 931 will begin to fill the chamber with LCO_2. ***From this point forward, the 931 will automatically advance through all process modes sequentially until completion.*** During the SLOW FILL mode, the LCO_2 will enter and fill the Process Chamber slowly.

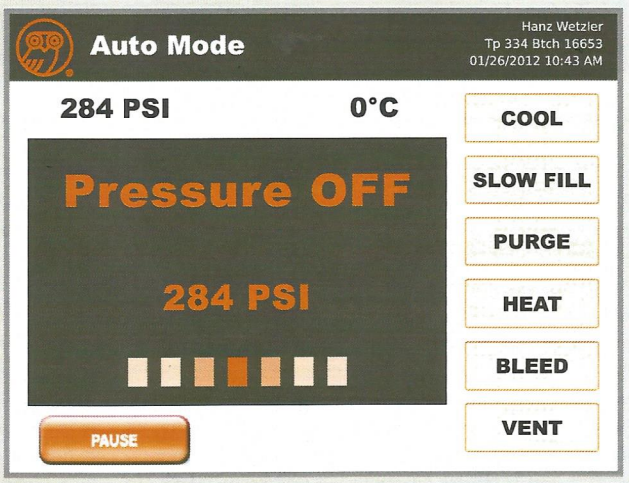


10. The 931 will advance to FILL Mode. The chamber temperature is automatically maintained below 10°C (±5°C). This is normal.



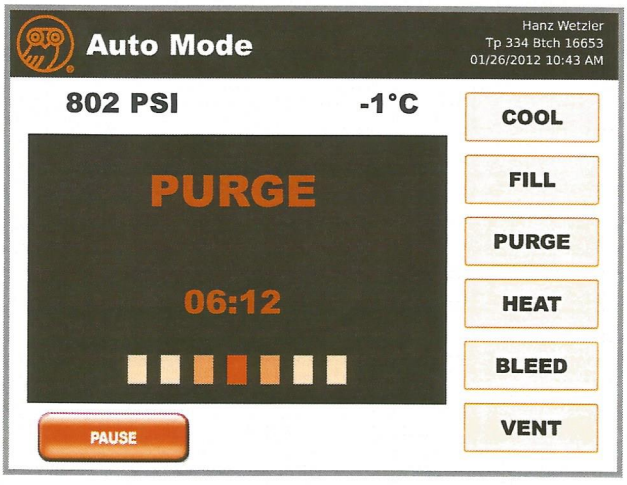
NOTE
Pressure OFF button may be used in case of poor chamber sealing. Chamber O-Ring is able to replaced without sample exposure to the air.

* Pressing the PRESSURE OFF button will slowly reduce chamber pressure while allowing alcohol to remain in the Process Chamber (if necessary).

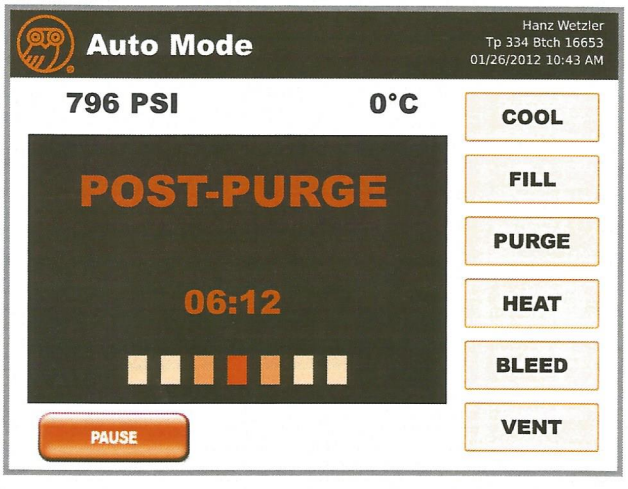


NOTE
The waste alcohol will exit the 931 Chamber via the PURGE/VENT Exhaust into the Carboy or Solvent Drain.

11. After the FILL mode expires, the 931 will automatically advance into the PURGE mode. The 931 will remain in the PURGE mode for the duration of process time.

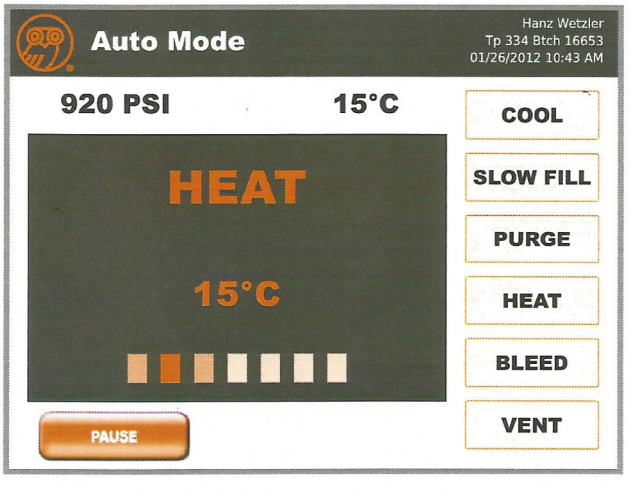


12. Upon Completion of the PURGE mode, the unit will automatically advance into a POST-PURGE FILL mode in which the chamber fills with LCO_2.



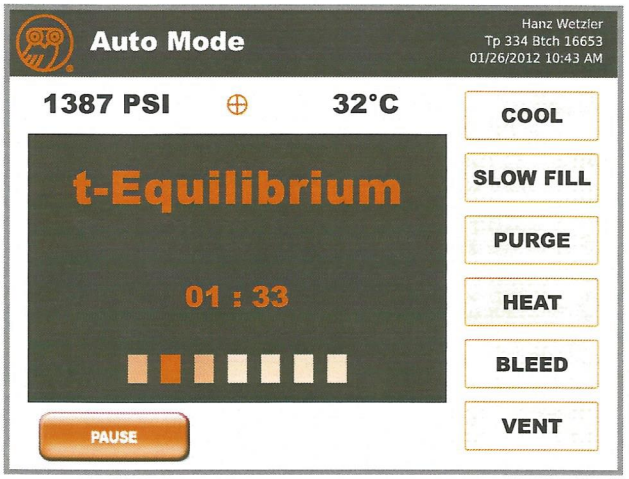
NOTE
Consult Check-Out Data Sheet in the Appendix of this 931 User Manual.

13. Upon completion of the POST-PURGE FILL mode, the HEAT mode will activate. The HEAT mode is when the samples are carried through the *"Critical Point."* Both the pressure and temperature will steadily increase.

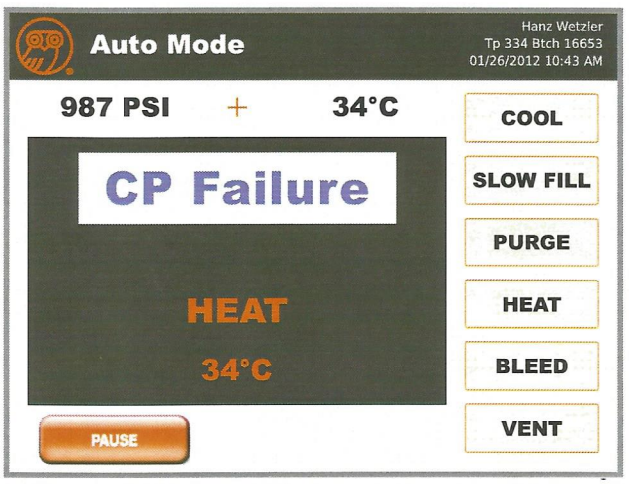


NOTE:
+ : CP Temperature
◯ : CP Pressure
⊕ : Critical Point

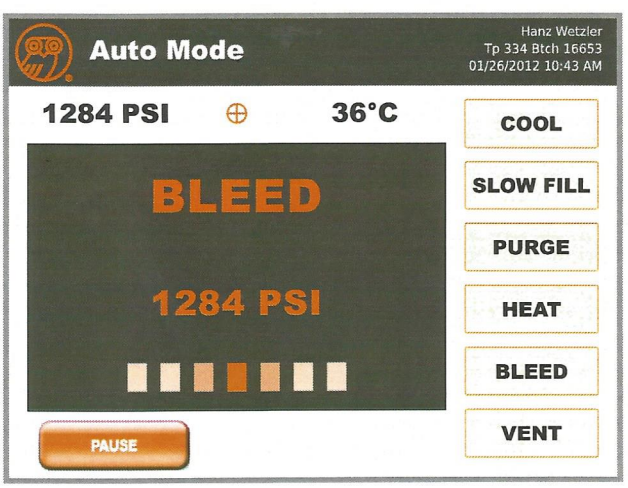
14. When the chamber pressure reaches and goes beyond 1072 PSI, it will stabilize in the range of 1350PSI (±5% @ 20°C). As the temperature reaches 31-35°C, the unit has achieved the "critical point" and this is where the 'tousimis equilibrium'* cycle starts. The + sign between the Pressure Temperature display indicates the Process Chamber has reached critical point temperature. The ◯ sign indicates the Process Chamber has reached critical point pressure.



15. If chamber pressure has not reached critical pressure prior to critical temperature "CP Failure" will be displayed. Refer "Critical Point Failure Correction (Chapter 4.7)" section.



16. At the end of the 'tousimis equilibrium' period, the 931 will automatically advance into the BLEED mode.

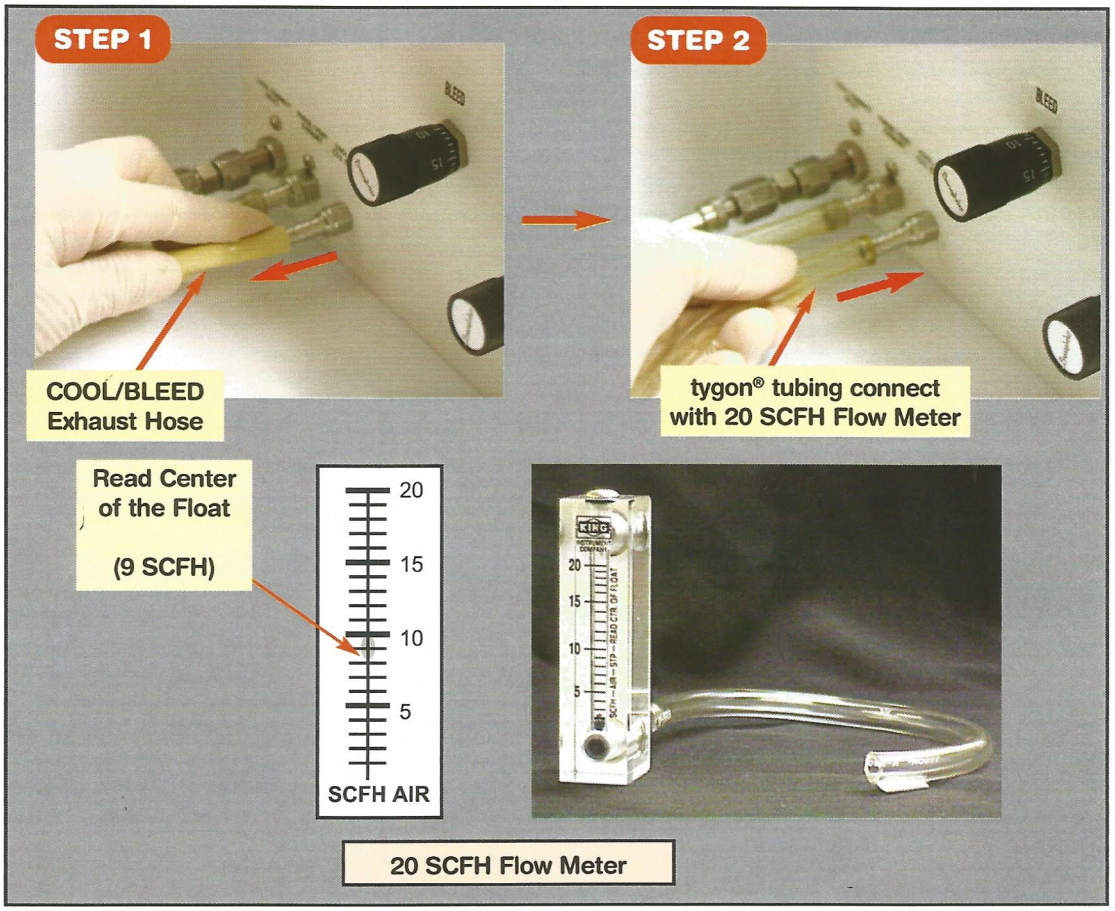


NOTE:
Step #17 is optional and NOT necessary with each process run.
This is more of a preventive maintenance detail.

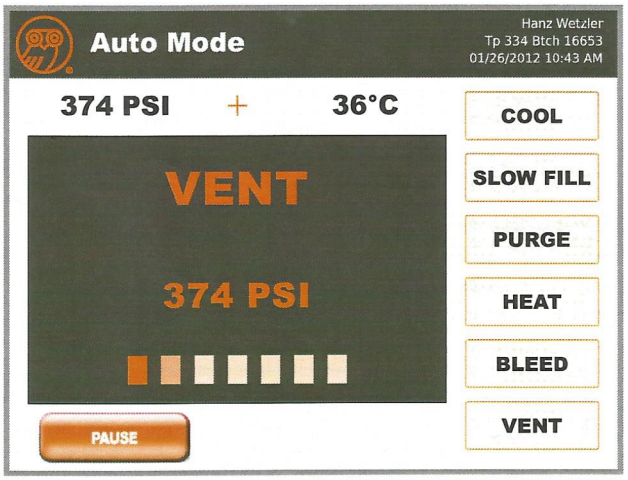
17. At this point, you can measure the BLEED rate (if desired) using the 20 SCFH Flow Meter supplied, by attaching the Flow Meter to the outlet of the COOL/BLEED EXHAUST. The BLEED rate has been factory preset to decompress the chamber at a rate of 100-150 PSI/min.

The flow rate should read 9-12 SCFH at the onset of the BLEED mode. This setting should yield an average decompression rate of approximately 100-150 PSI/min reduction in pressure. This pressure reduction flow rate is the desired decompression rate between 1300 PSI to 550 PSI.

Once the BLEED rate has been adjusted, remove the Flow Meter and reconnect COOL/BLEED Exhaust Hose.

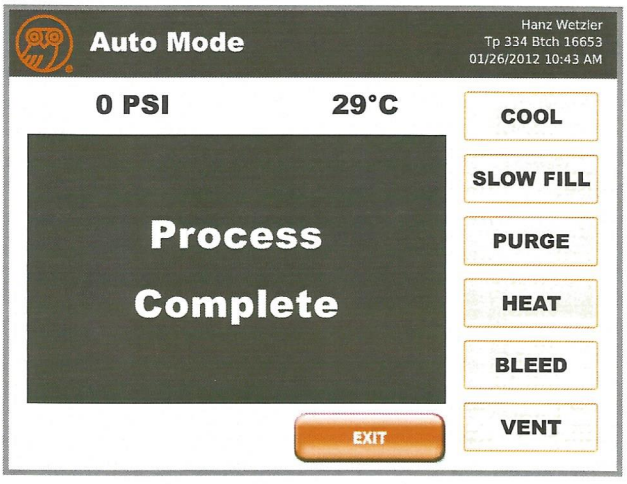


18. At the preset pressure (factory default: 550 PSI), the 931 will automatically advance from the BLEED mode into the VENT mode.



19. It is not necessary to re-adjust the PURGE-VENT metering valve flow rate as it is preset. The Process Chamber should then come to atmospheric pressure after approximately 3 minutes in this VENT mode.

20. When chamber pressure reaches less than 10 PSI, the 931 will display "Process Complete."



21. At this point, the chamber lid may be removed by alternatively and evenly loosening the Knurl Nuts (Never attempt to 'force' open).

22. The sample(s) can then be removed from the Process Chamber. Reseal the Process Chamber with the chamber lid to help keep it clean and moisture free.

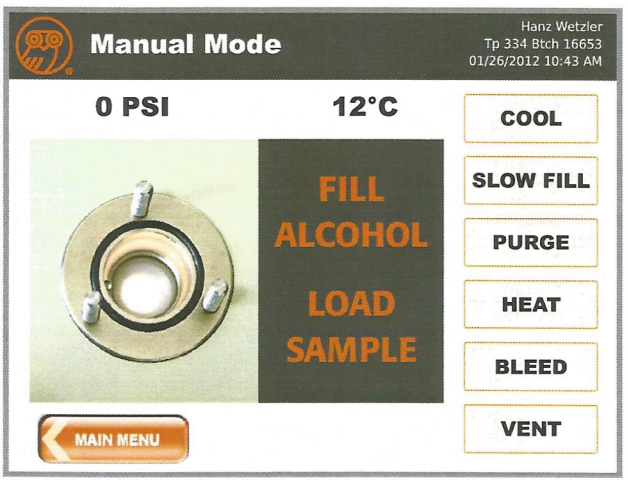
23. The next process run may be initiated provided there is sufficient LCO_2. Otherwise, the system power may be turned OFF.

4.3 Manual Mode

1. The "Manual Mode" enables the 931 to operate without a preset process run sequence. User may press buttons to advance each mode.

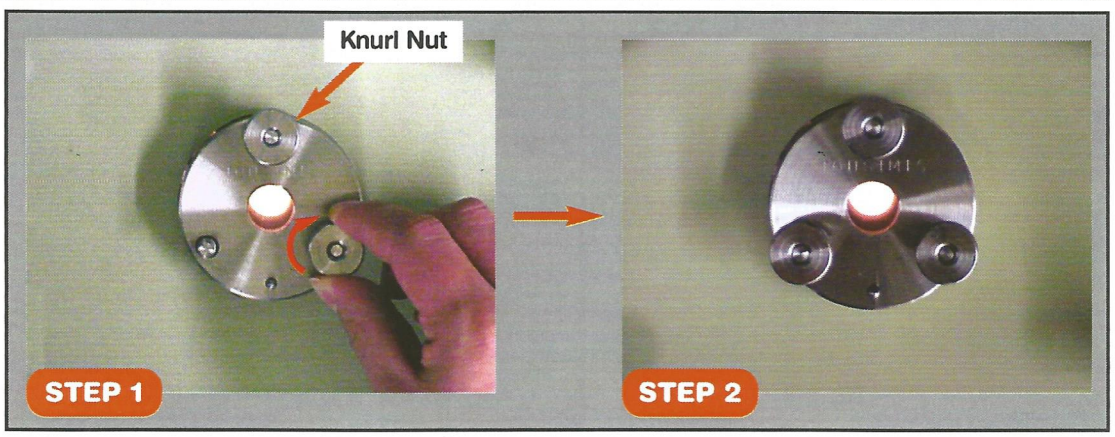
2. Prior to initiating the COOL mode, pour sufficient alcohol (Typically 10-15ml) into Process Chamber to cover your sample. Quickly transfer sample holder(s) into the Process Chamber.
The chamber O-Ring should lie completely in its groove. NEVER grease the O-Ring. Place the chamber lid down over the chamber using the 3 knurl nuts. Tighten each knurl nut hand tight with equal pressure. Once lid is secure press COOL button.

Actual Manual Mode Initial Screen Display

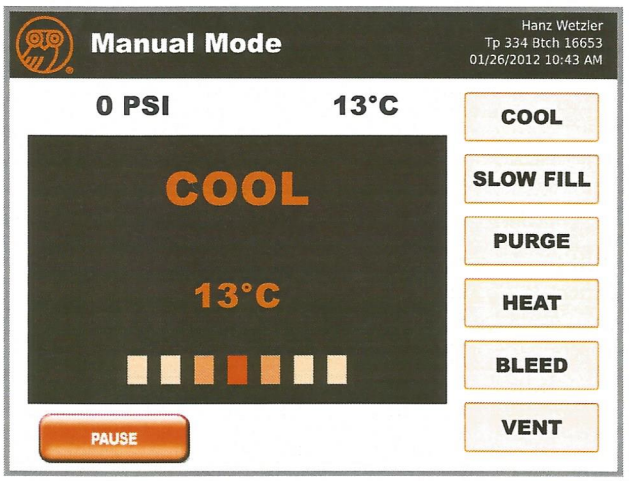


Secure Chamber

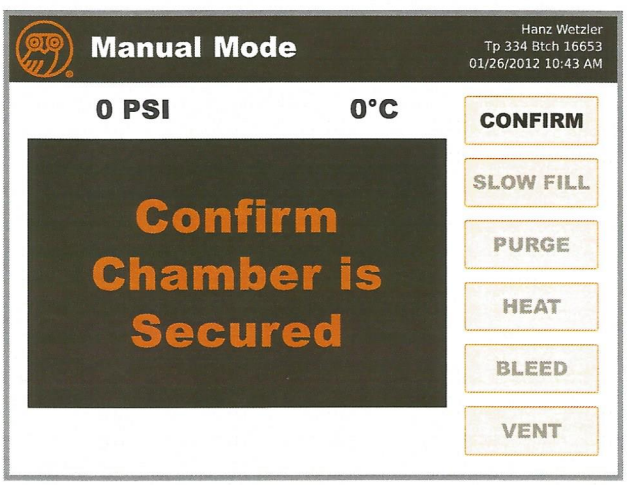
NOTE:
Never use channel locks or pliers for tightening the knurl nuts.
Always hand tighten!



3. The 931 will continue cooling and maintaining the chamber temperature near 0°C during manual COOL Mode until next mode has been selected by user.

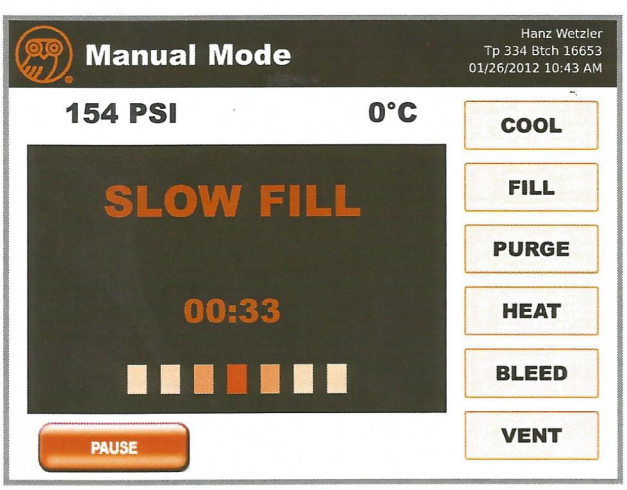


4. Press the SLOW FILL button to advance the process. The 931 requires Confirmation of "Chamber Security" to continue the process.
Double check "Chamber Security" and press CONFIRM.



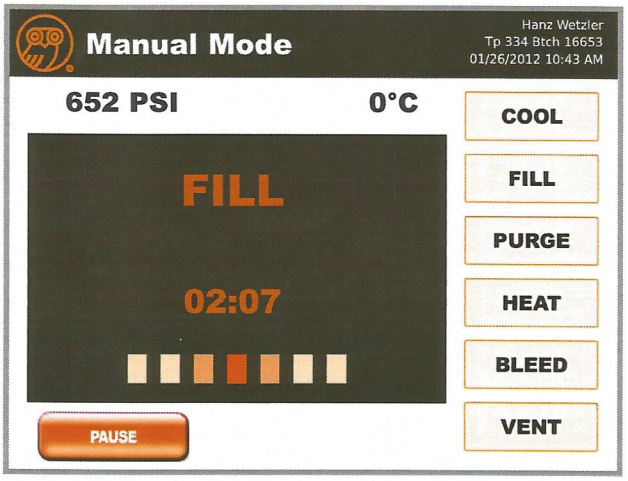
NOTE
Chamber Security is important to maintain chamber pressure. Chamber Lid must be properly sealed as per p.30 "Star Pattern" Chamber Closure Sequence.

5. The 931 will begin to fill the chamber with LCO_2. During the SLOW FILL Mode, the LCO_2 will enter and slowly fill the Process Chamber.



NOTE
One may observe the chamber through the Chamber Lid Window to make sure LCO_2 covers sample completely.

6. Press the FILL button to advance to FILL Mode.

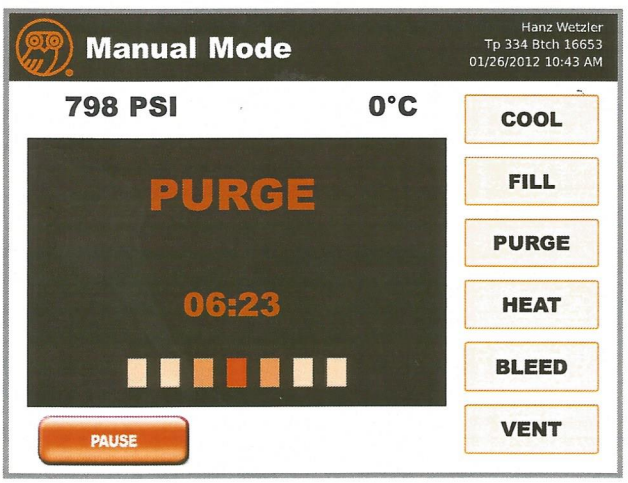


NOTE
Pressure OFF button may be used in case of poor chamber sealing. Chamber O-Ring is able to be replaced without sample exposure to the air.

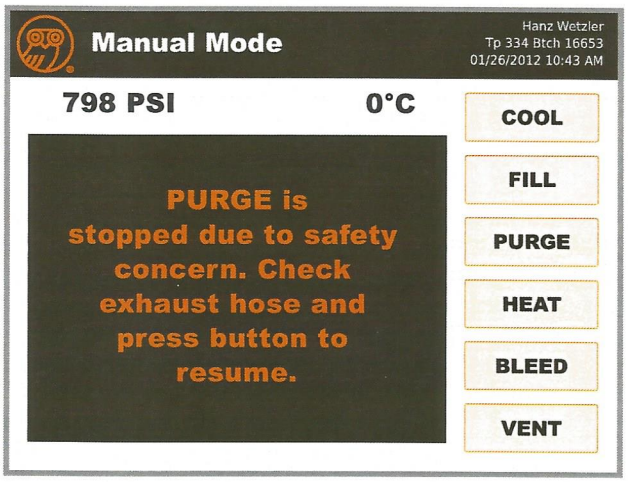
* Pressing the PRESSURE OFF button will slowly reduce chamber pressure, while allowing alcohol to remain in the Process Chamber (if necessary).

NOTE
PAUSE button will pause operation and give option to change preset value during process run, "Resume" operation or terminate the process run. See section 4.5 "Pause & Resume During Process Run."

7. Press PURGE button to advance into the PURGE Mode. During PURGE Mode the waste alcohol will exit the 931 Chamber into the Carboy or Solvent Drain.

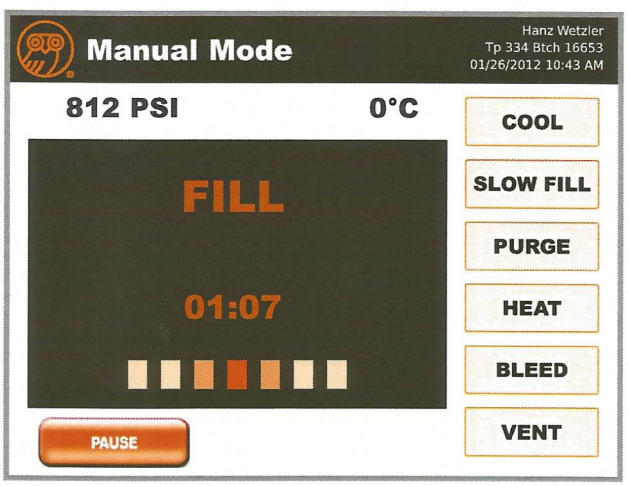


8. The 931 will shut down after 15 minutes of PURGE Mode. Checking exhaust tubing at this point is recommended. The 931 can resume any operational mode by pressing the appropriate button.

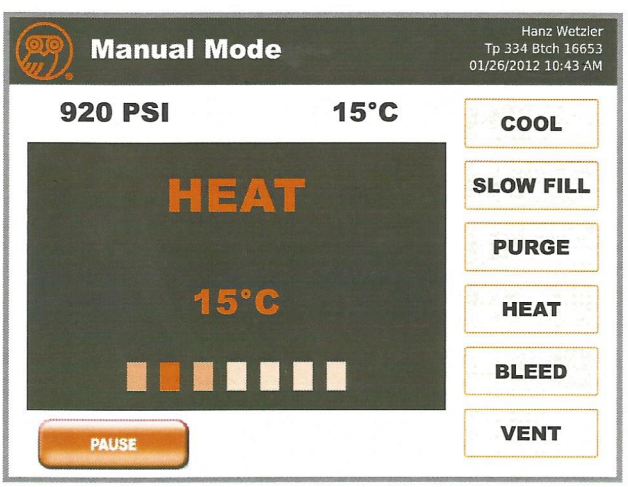


NOTE
A two minute POST-PURGE FILL time is recommended or until the chamber liquid level is full.

9. Upon completion of the PURGE Mode, you may press the FILL button to accomplish POST-PURGE FILL Mode, during which the chamber only fills with LCO_2.

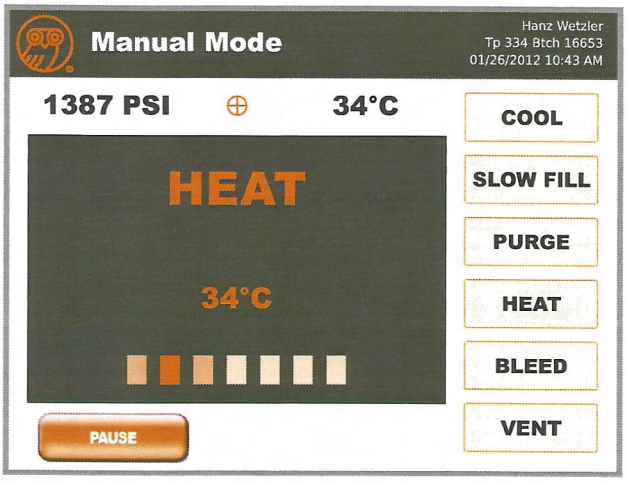


10. Press HEAT button to advance into the HEAT Mode. The HEAT Mode is the stage in which the samples are carried through the *"Critical Point."*
Both the pressure and temperature will steadily rise as the Process Chamber has active HEAT.



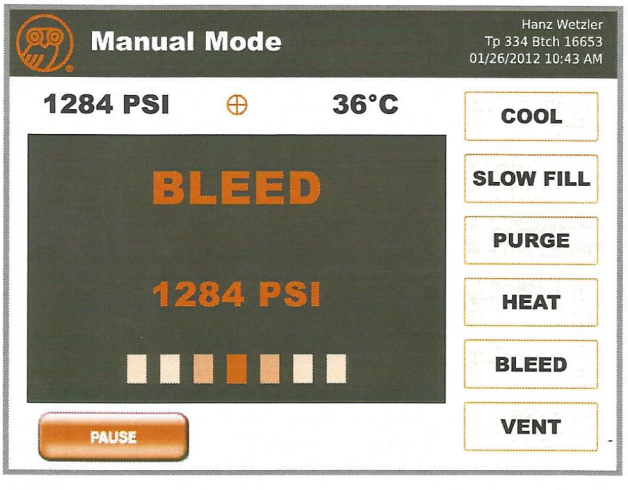
NOTE:
+ : CP Temperature
◯ : CP Pressure
⊕ : Critical Point

11. When the Process Chamber pressure reaches and goes beyond 1072 PSI, it will stabilize in the range of 1350 PSI (±5% @ 20°C). As the temperature achieves 31°C, the Process Chamber has achieved the "critical point". The + sign on the Pressure/Temperature display indicates the Process Chamber has reached the critical point temperature, the ◯ sign indicates the Process Chamber has reached the critical point pressure.

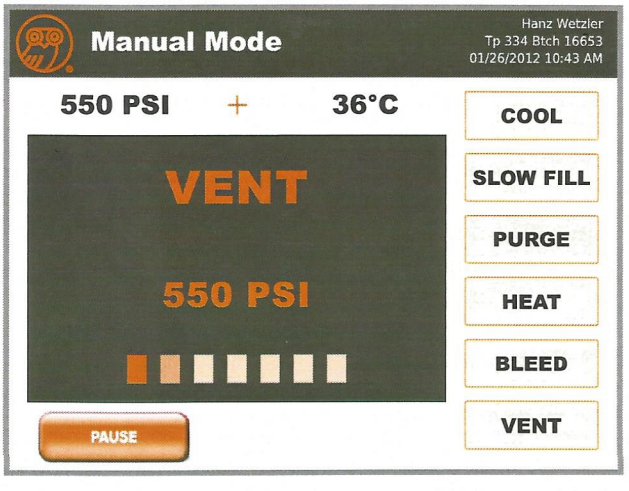


NOTE:
If needed, measure BLEED rate using "Auto Mode" BLEED Instructions (Chapter 4.2 Step #17).

12. It is recommended to wait for at least 2 minutes during the 'tousimis equilibrium' period prior to pressing BLEED button to advance into the BLEED mode.
It is recommended to maintain BLEED Mode down to 550 PSI.



13. Press VENT button to advance VENT Mode to complete a process run.



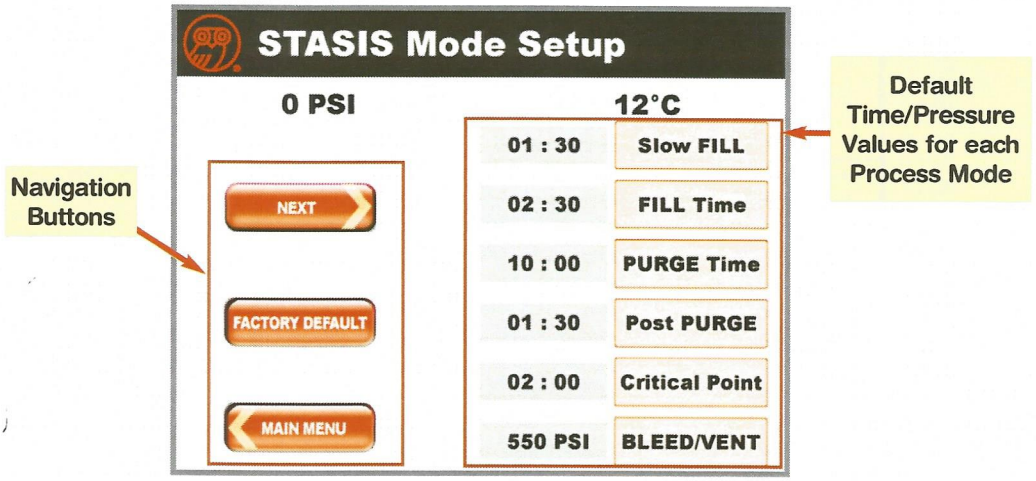
4.4 Stasis Mode
(See Chapter 4 prior to proceeding)

1. Stasis Mode processes samples using the identical Process Steps as the "Auto Mode" except for an additional "Stasis Step" (see Chapter 4 Stasis Diagram).

The Stasis Time and the number of Stasis Cycle need to be setup (see #3) to operate "Stasis Mode".

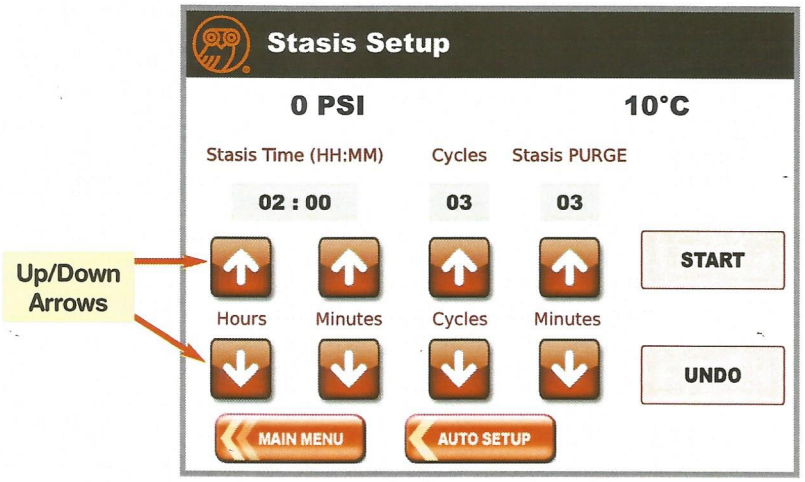
NOTE
With the "Stasis Mode", the user is able to program the times that the sample will sit in STASIS.
After the PURGE Mode the Post PURGE STASIS will repeat.

2. You may either use the Factory Default settings or Custom Setup the desired Time and Pressure Values for each Process Mode and press NEXT (See Chapter 4.2).



NOTE
Maximum Time for Slow FILL, FILL, PURGE and Post-PURGE Fill Mode is 15 minutes.
Maximum CP Time is 180 Minutes.

3. Using the Up/Down arrows, you may setup the desired "Stasis Times", "Stasis Cycles" and "Stasis PURGE Time". Press START to initiate the Stasis Mode.



Press RESET button to restore the factory default values.
Press either MAIN MENU or AUTO SETUP buttons to exit without saving values.

For the STASIS Process, Autosamdri®-931 will use two different PURGE Time.
Normal PURGE Time will be used for the initial PURGE Process before the 931 advances into Stasis Cycle.
Stasis PURGE Time will be used during Stasis Cycle.
Most Alcohol will be exit from process chamber during Normal PURGE Time.
The Stasis Purge Time recommended is to set it to less than 30% of normal PURGE Time to help reduce LCO_2 consumption.

Example: STASIS Process Settings

Slow FILL: 3 Min
FILL: 6 Min
Normal PURGE Time: 10 Min
Post-PURGE FILL: 4 Min
Critical Point: 10 Min

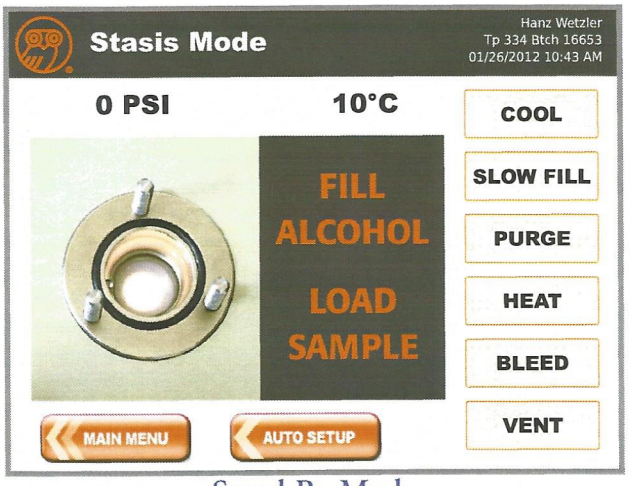
Stasis Time: 2 Hours
Cycle: 3 Times
Stasis PURGE Time: 5 Min

Process Sequence Example:

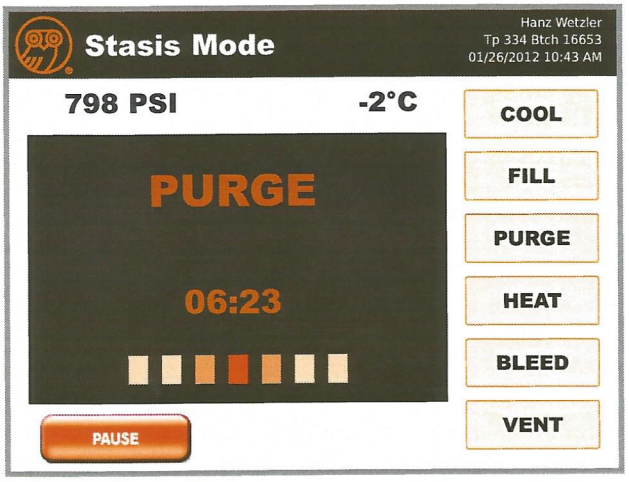
COOL → SLOW FILL (3 MINS) → FILL (6 MINS) → PURGE TIME (10 MINS)
→ POST-PURGE-FILL (4 MINS)
→ STASIS #1 (2 HOURS) → COOL → SLOW FILL (3 MINS)
→ STASIS PURGE TIME (3 MINS) → POST-PURGE FILL (4 MINS)
→ STASIS #2 (2 HOURS) → COOL → SLOW FILL (3 MINS)
→ STASIS PURGE TIME (3 MINS) → POST-PURGE FILL (4 MINS)
→ STASIS #3 (2 HOURS) → COOL → SLOW FILL (3 MINS)
→ STASIS PURGE TIME (3 MINS) → POST-PURGE FILL (4 MINS)
→ HEAT → CRITICAL POINT (tousimis®-EQUILIBRIUM 10 MINS)
→ BLEED → VENT

NOTE:
Total PURGE Time = 19 Min
10 Mins (Normal PURGE Time) + 3 x 3 Mins (Stasis PURGE Time) = 19 Mins

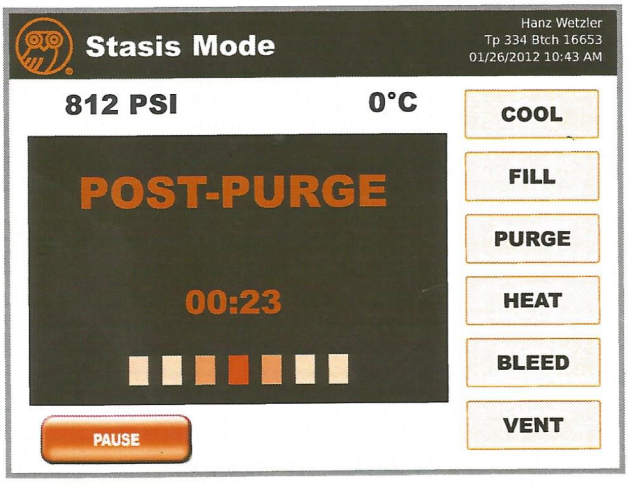
4. Once the START button is pressed, the 931 will operate as per Auto Mode from this point forward until POST-PURGE FILL Mode.



Stand By Mode



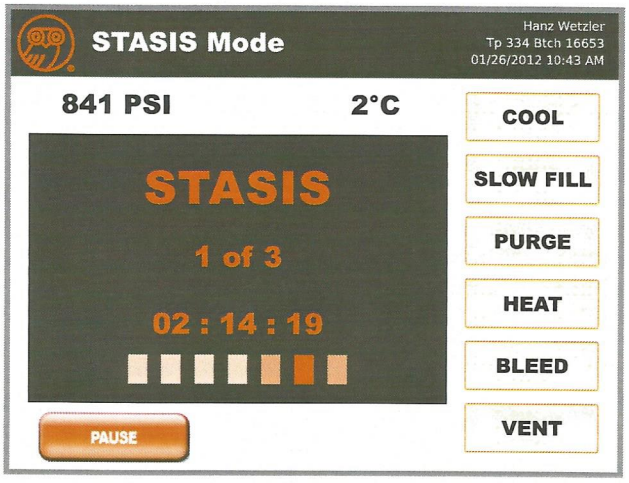
PURGE Mode



POST-PURGE FILL Mode

NOTE
All valves leading into and out of the Process Chamber are closed to maintain LCO_2 inside of chamber during "STASIS Mode".

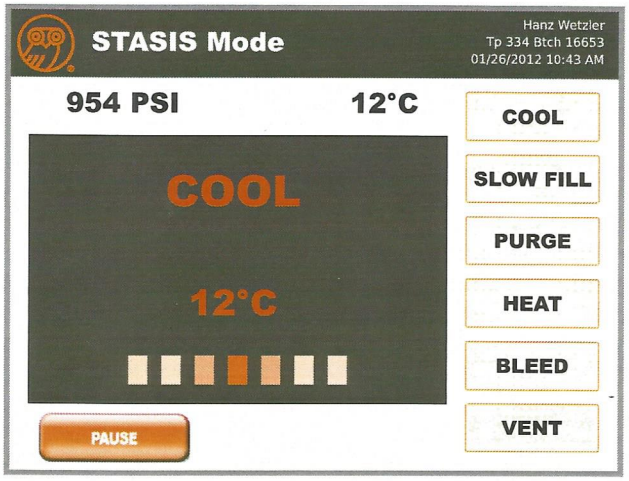
5. Upon completion of the POST-PURGE FILL Mode, the 931 will automatically advance into the first cycle of the STASIS Mode.



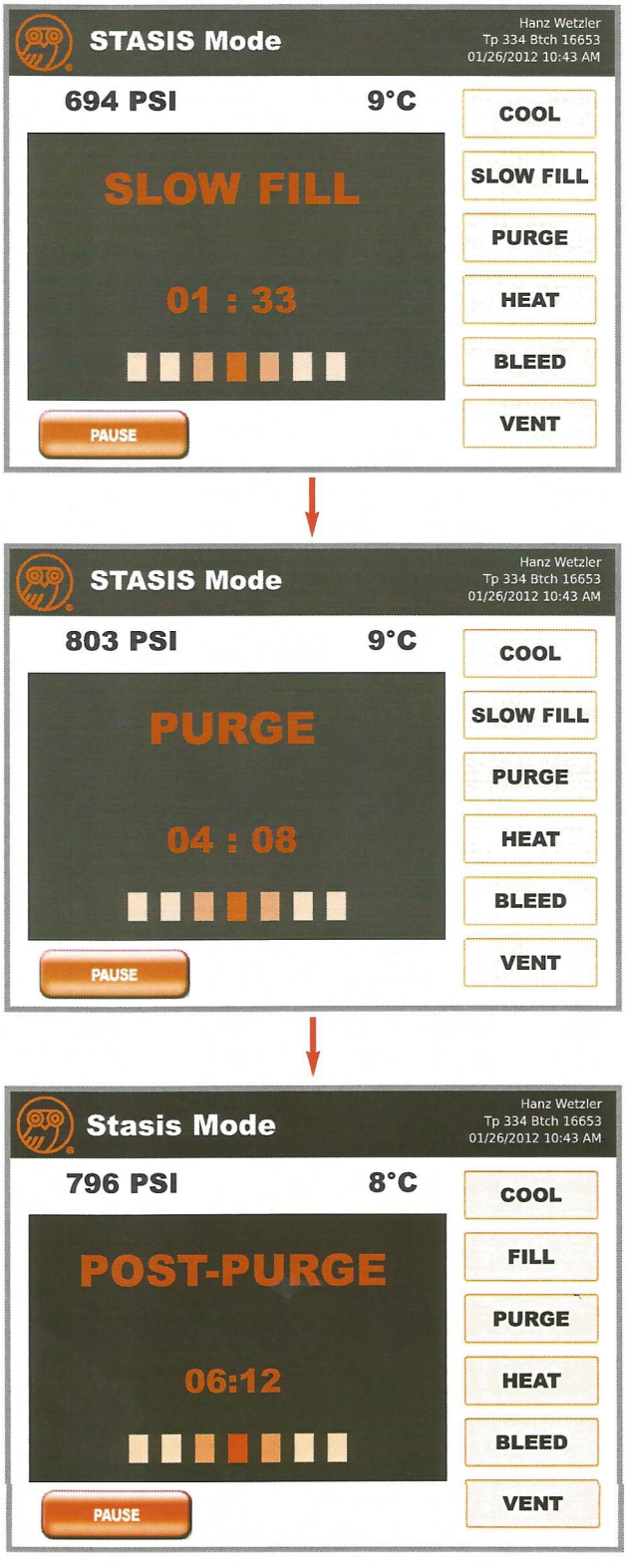
The sample will now stay submerged in LCO_2 allowing time for the intermediary fluid to dissipate from sample interior out into the surrounding LCO_2.

NOTE
It is normal to see the chamber temperature to gradually increase during Stasis Mode.

6. After the initial STASIS Mode is completed, the 931 will advance into COOL Mode and resume the process.



7. The 931 will then automatically advance into the SLOW FILL Mode and then into the Stasis-PURGE Mode, followed by POST-PURGE FILL Mode.

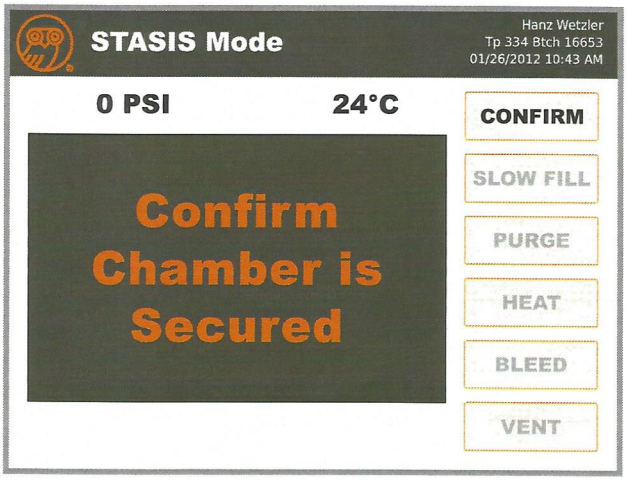


NOTE
After Post-PURGE FILL Mode, if any additional STASIS Cycles are programmed, the 931 will re-enter STASIS Mode.

8. Upon completion of the last preset STASIS Mode cycle, the 931 will then advance into the COOL Mode, SLOW FILL Mode, PURGE Mode and POST-PURGE FILL Modes sequentially just as if it were in an Auto Mode.

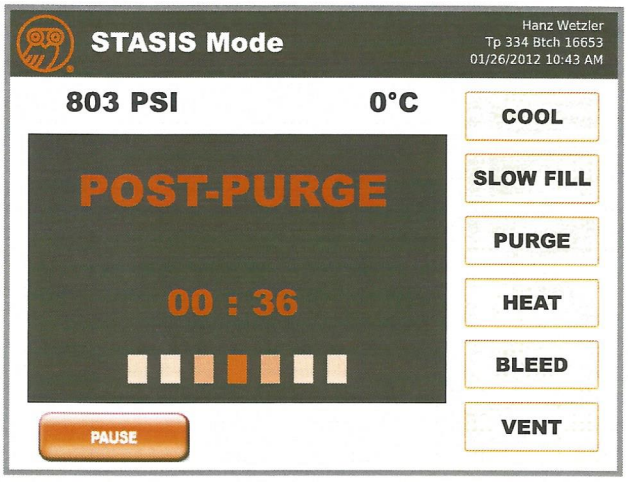
NOTE:
Chamber Lid may not be secured properly or damaged O-Ring could cause chamber pressure to drop during STASIS Mode.

9. During STASIS Mode, if chamber pressure drops to 0 PSI, the 931 will pause and display a Chamber Security Check Sign (Security Feature) stating "Confirm Chamber is Secured".

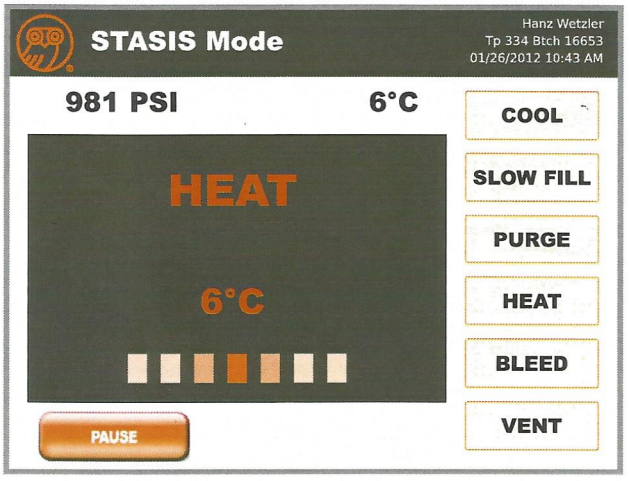


NOTE:
Please refer to "Auto Mode" (Chapter 4.2) for detailed instructions.

10. At this point until the end of the process run, the 931 will process each Mode Automatically.

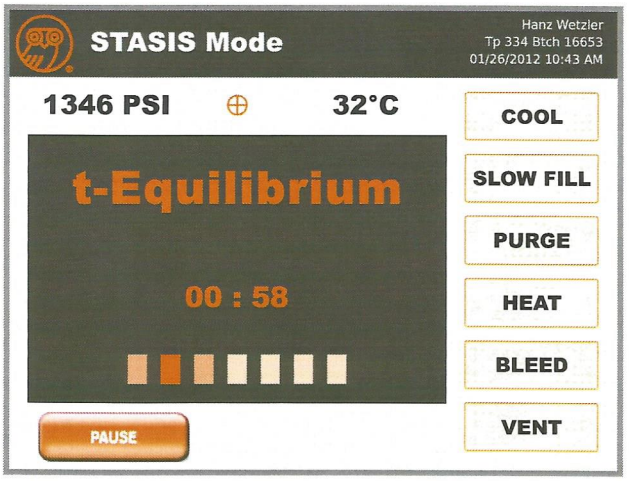


POST-PURGE Mode

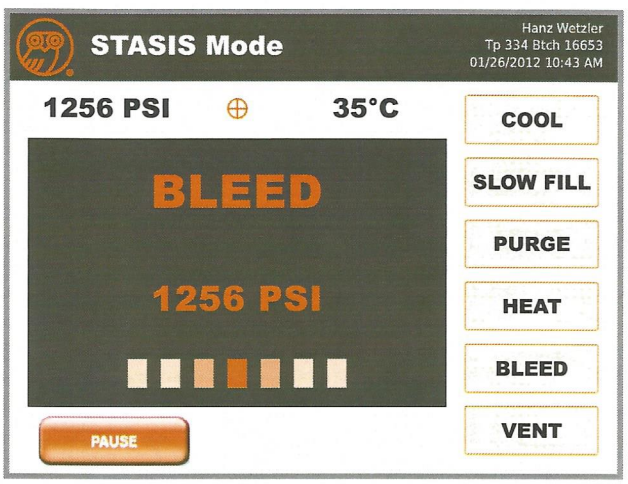


HEAT Mode

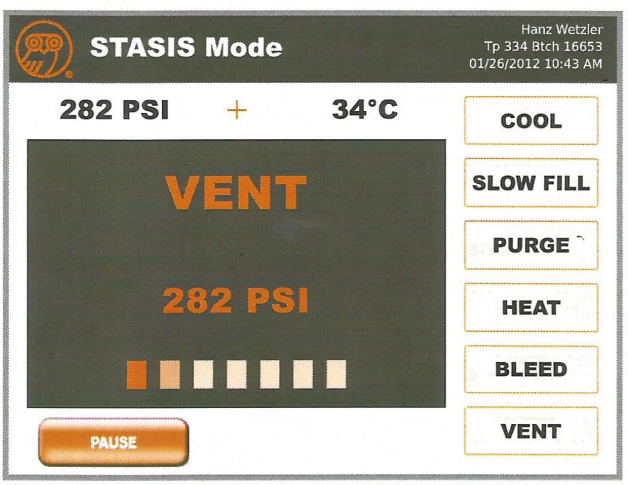
NOTE
tousimis-Equilibrium Time (CP Time) may meed to be extended as per Ternal Conductivity of the sample.



tousimis Equilibrium



BLEED Mode

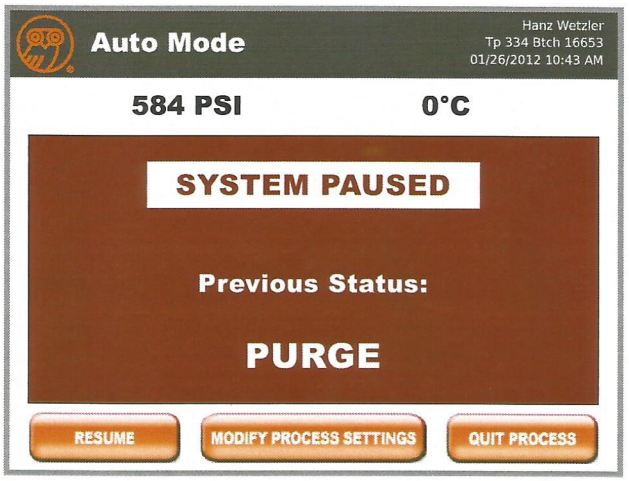


VENT Mode

4.5 Pause & Resume During Process Run

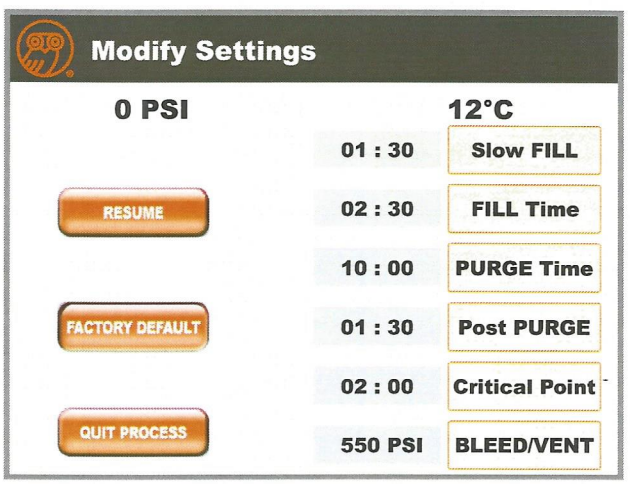
NOTE:
If LCO_2 Tank runs out during a process run, press PAUSE and replace the LCO_2 Tank.
Press RESUME to reactivate process run after the new LCO_2 Tank has been installed.

1. At any time during the Auto, Manual, or STASIS Modes the PAUSE button will allow the 931 to enter into the PAUSE Mode.



The 931 can then resume the process by pressing the RESUME button.

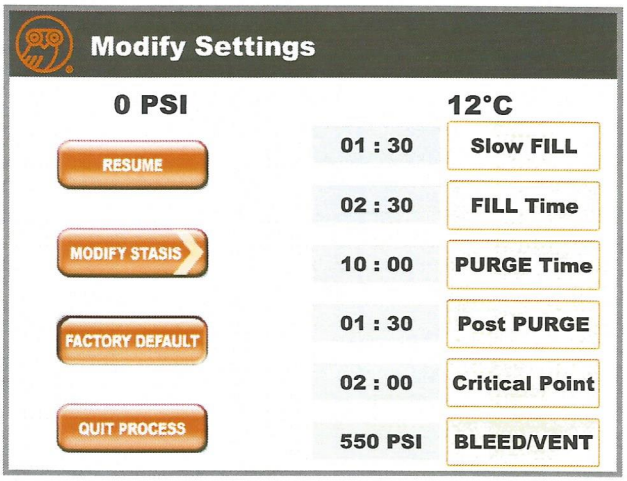
2. The MODIFY PROCESS SETTINGS button will redirect the display view to the "Modify Settings" page where the 931 Auto Mode Settings may be modified.



See instructions in "Auto Mode" (Chapter 4.2) to modify Auto Mode Settings and press RESUME to continue process run.

Pressing MODIFY PROCESS SETTINGS during "Manual Mode" will advance 931 from "Manual Mode" to "Auto Mode." The remaining process run steps will RESUME as Auto Mode.

3. During STASIS Mode, there will be an extra button for STASIS Setting Modification. See "STASIS Mode" (Chapter 4.4) instructions to modify settings.



4. Press QUIT PROCESS button on the Pause Screen and Modify Settings Screen will display a BLEED PORTS and VENT PORTS option to reduce chamber pressure in order to terminate the current process run.

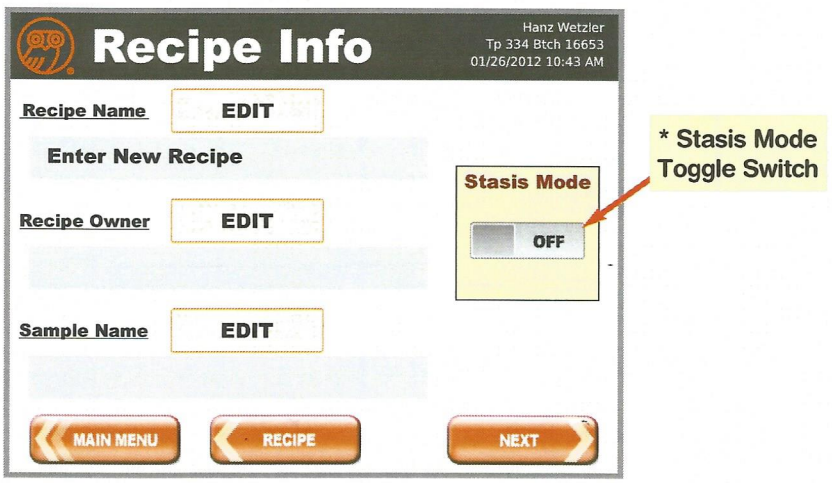
BLEED PORTS: This vents the chamber pressure back to 0 PSI while allowing intermediary fluid (Ultrapure Alcohol) to stay within the Process Chamber. Therefore, your sample will not be exposed to gas.

VENT PORTS: This vents the chamber pressure out and it is often set at a quicker flow. However, all intermediary fluid (Ultrapure Alcohol, if present) in the Process Chamber will be discharged through these ports.

4.6 Recipe Mode

1. The "Recipe Mode" allows the input and storage of up to 10 custom process run protocols. Press RECIPE button from "Main Menu" page to enter Recipe Mode. To create a new recipe, press ENTER NEW RECIPE button. To run or modify a previously saved recipe, press previously saved recipe name.

2. Press EDIT button next to either "Recipe Name", "Recipe Owner" or "Sample Name" to update the appropriate information for the Recipe.



* Press Stasis Mode Toggle Switch to set Stasis Mode ON/OFF for the recipe.

3. Type "Recipe Name" and press Enter to update information.

4. Press NEXT when the complete information has been entered.
Press MAIN MENU or RECIPE button to exit without saving.

5. If the Stasis Mode Switch is OFF:
You may modify the preset values. Pressing START will save recipe and start the "Auto Mode" immediately. SAVE RECIPE will Save the recipe only.

Press RECIPE and MAIN MENU to exit without saving.

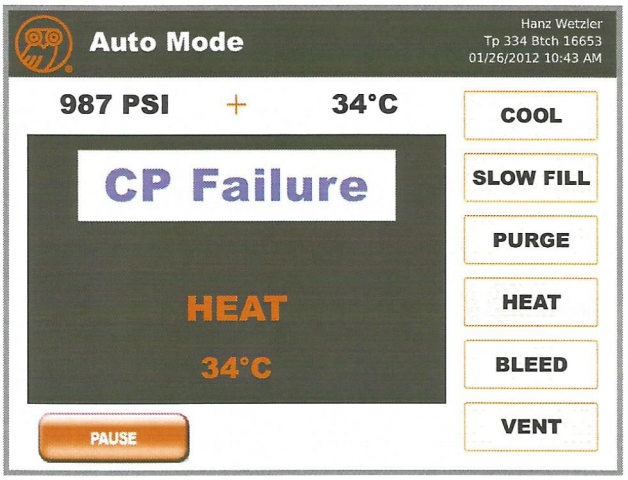
6. If the Stasis Mode Switch is ON:
You may modify the preset values and press NEXT. Press RECIPE and MAIN MENU to exit without saving.

7. Set "Stasis Time" and "Stasis Cycle" (See Chapter 4.4 "Stasis Mode") and press START to save recipe and start "Stasis Mode" immediately.
Press SAVE to save the recipe only.

Press GO BACK and RECIPE to exit without saving.

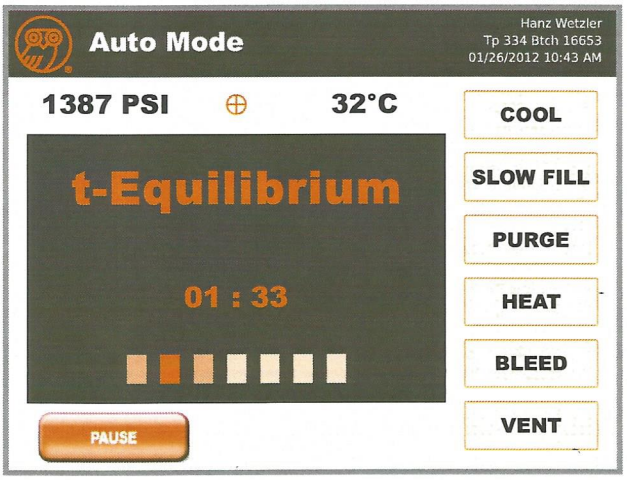
4.7 Critical Point Failure Correction

Symptom: The "CP Failure" sign will be displayed if the Temperature and/or Pressure in the Process Chamber has not reached the *"Critical Point"* in the proper sequence during HEAT Mode.



A successful "*Critical Point*" process (tousimis Equilibrium) is achieved when the internal micro processor receives the following two signals in this sequence:

1. Pressure Signal sent via High Pressure Sensor between 1150-1200 PSI.
2. Heat Signal sent sent via Heat Thermostat shutting OFF typically between 34-38°C



The + sign on the Pressure/Temperature display indicates that the Process Chamber has reached critical point temperature. An ○ sign indicates that the Process Chamber has reached critical point pressure.

NOTE:
+ : CP Temperature
○ : CP Pressure
⊕ : Critical Point

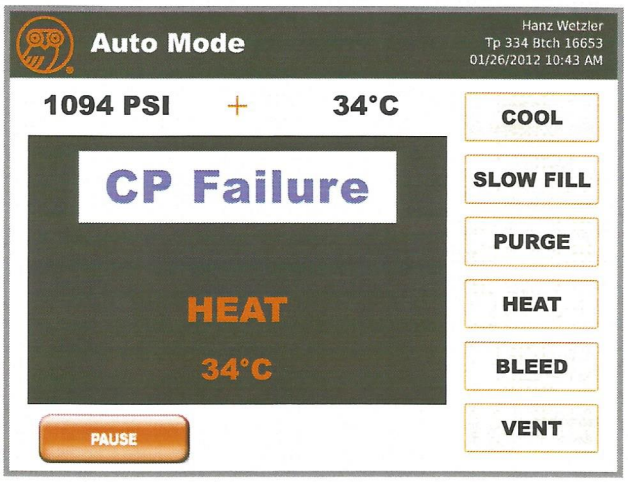
Critical Point Failure Correction Options:

Provided the micro processor receives the signals in the previously mentioned sequence, there will not be a 'Critical Point Failure.'

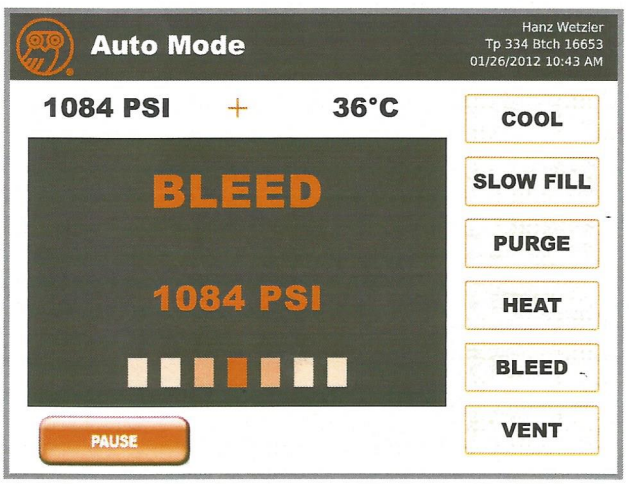
However, should the micro processor receive these signals in the reverse order - Heat Signal (1st) followed by Pressure Signal (2nd) - a failure will be indicated by 'CP Failure' flashing.

Solutions:

Scenario #1: Pressure is greater than 1072 PSI.



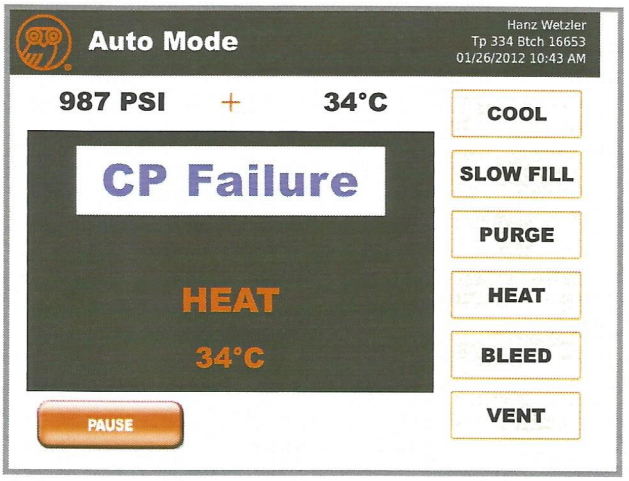
Press BLEED. 'CP Failure' display will be turned off and the 931 will default back to the normal Auto-Mode and resume in BLEED Mode moving on to completion as normal.



The LCO_2 tank should be replaced with a new LCO_2 tank prior to commencing the next process run.

Scenario #1 may be deployed as the theoretical physical completion of the critical point (1072 PSI + 31°C) for CO_2 has been attained even though the system's sensors did not detect it. The system's sensors are designed to over-ride both pressure and temperature minimums for the CO_2 critical point.

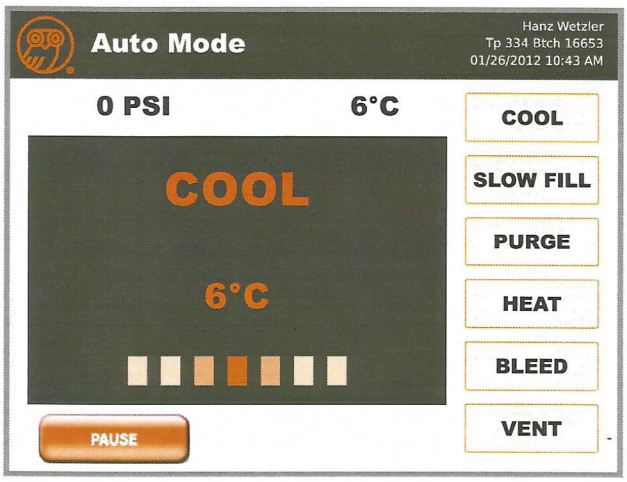
Scenario #2: Pressure is less than 1072 PSI.



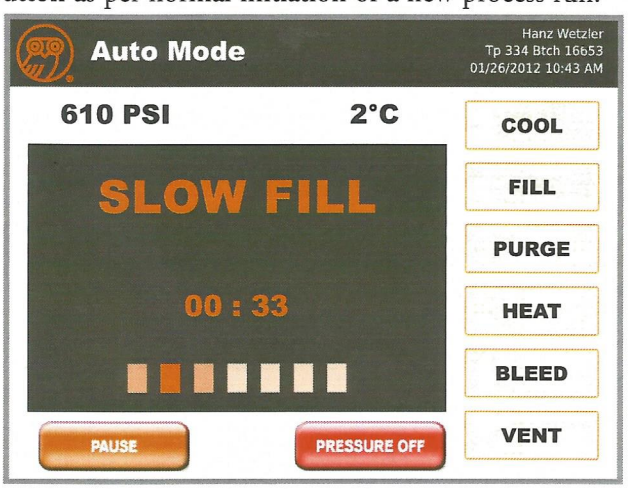
Replace LCO_2 tank with a new LCO_2 tank.

DO NOT OPEN CHAMBER AT THIS POINT.
KEEP SAMPLE PRODUCT WITHIN PROCESS CHAMBER.

Press COOL button to re-initiate the process.



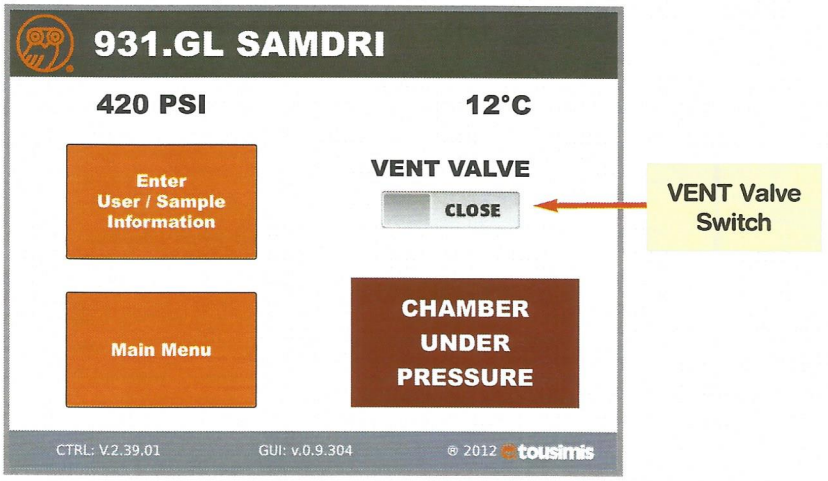
Press SLOW FILL button as per normal initiation of a new process run.



4.8 Power Failure Recovery

Symptom: Power outage during process run.

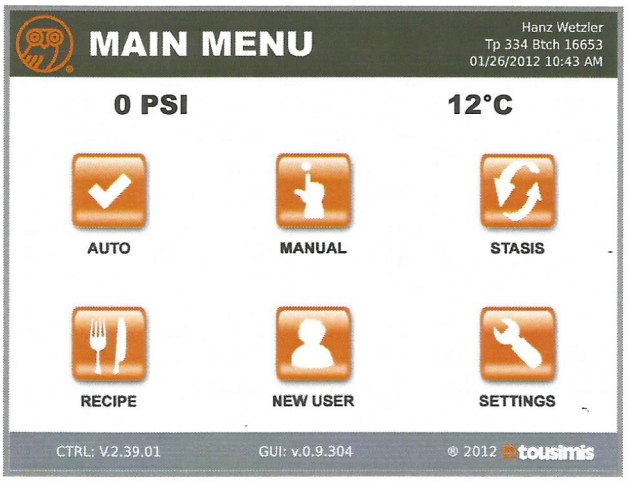
1. Power ON when the facility power is back ON.
Leave VENT Valve switch in CLOSE position.



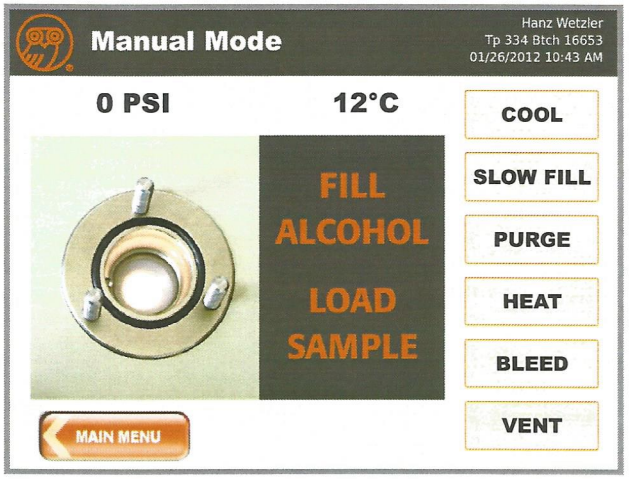
Press Enter User/Sample Information button to enter User/Sample Information.

Press Main Menu button to start operation without setup User/Sample Information.

2. Press MANUAL button to initiate "Manual Mode" process.

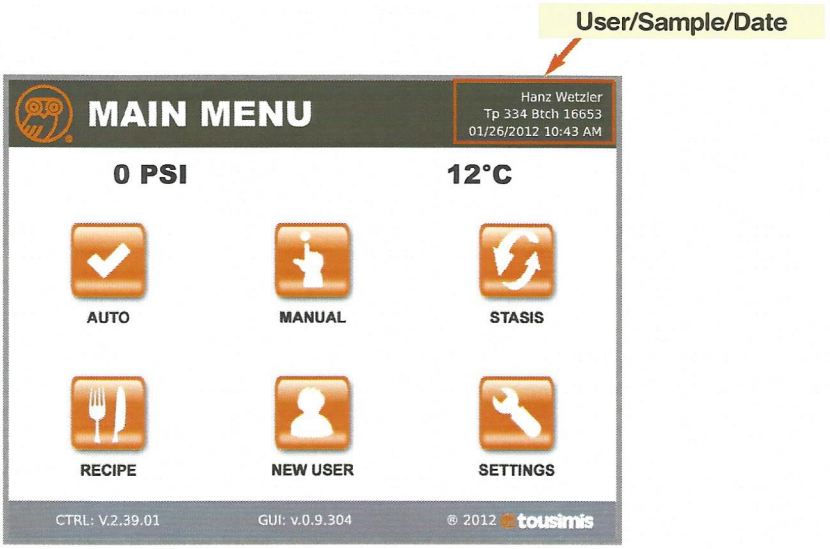


3. Resume process by pressing the button of the process mode (COOL, SLOW FILL, PURGE, HEAT, BLEED, VENT) at which the process had stopped during power outage.



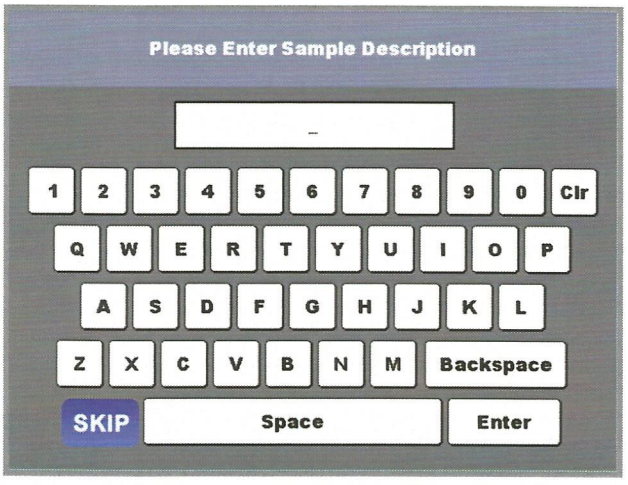
4.9 New User

1. To update User & Sample Information, press NEW USER button.



2. Enter User Name or ID (maximum 18 characters) and press Enter.

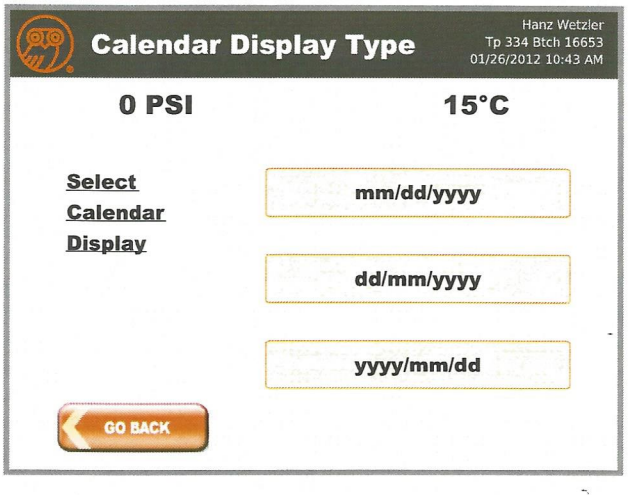
3. In the following screen, Enter Sample ID or Description (maximum 18 characters) and press Enter.



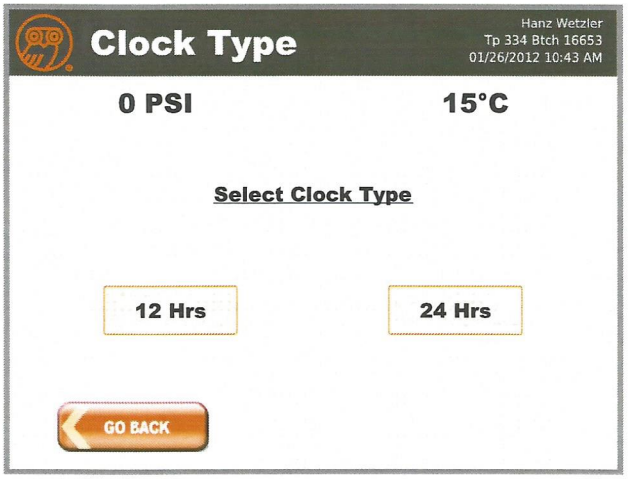
4.10 Settings (Date/Time)

1. To update Date/Time information, press DATE/TIME button.

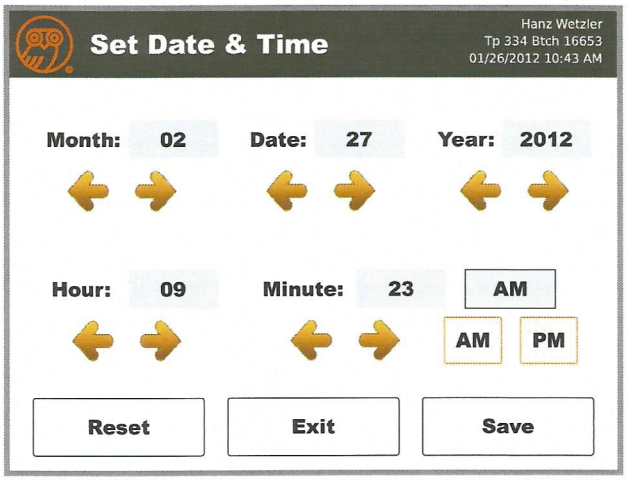
2. Select Calendar Display Type.



3. Select Clock Type.

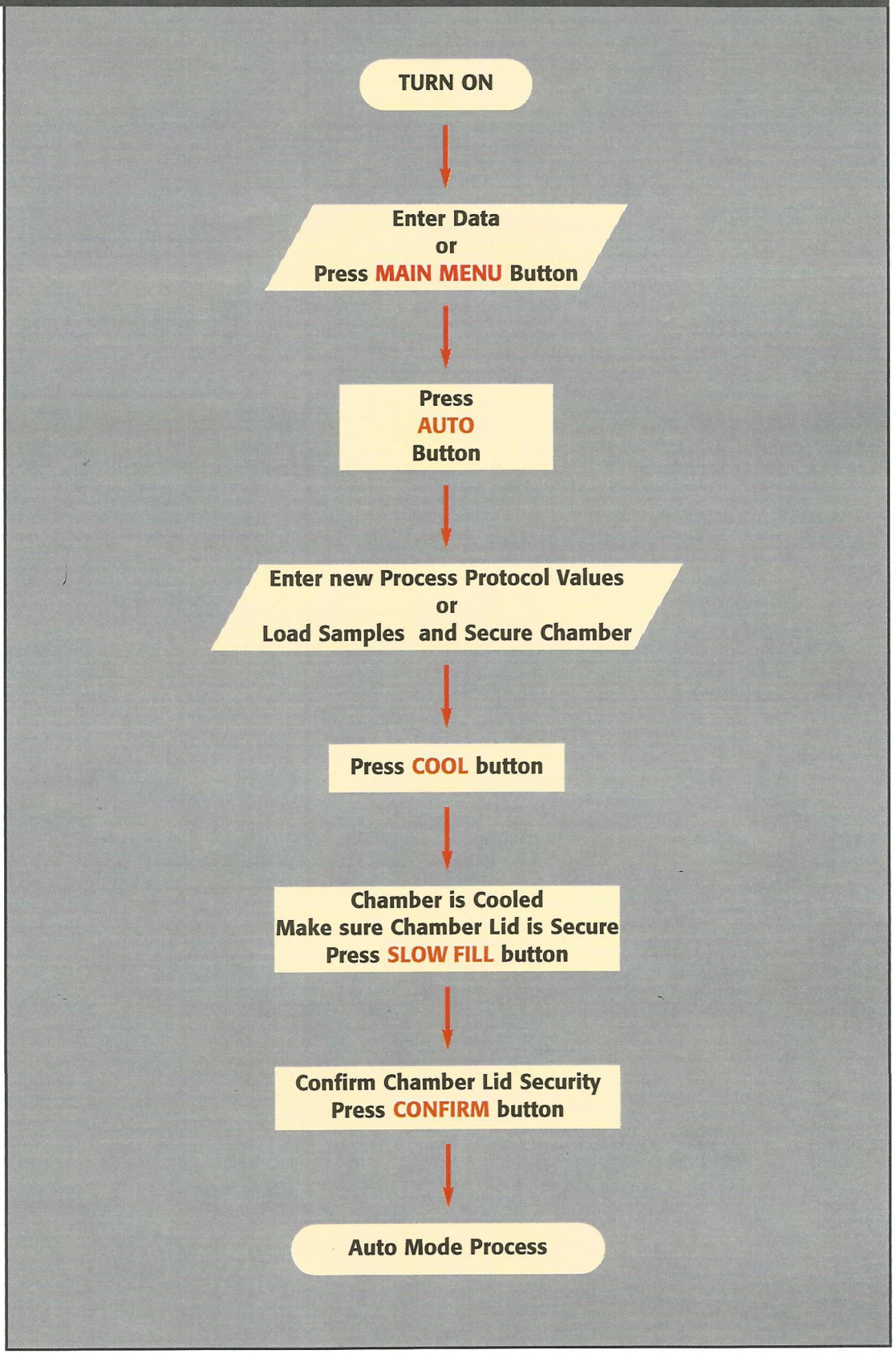


4. Using arrow buttons below each time variable, adjust time. Press Save to update time. Reset to reset the time. Press Exit to go back to "Settings" menu.

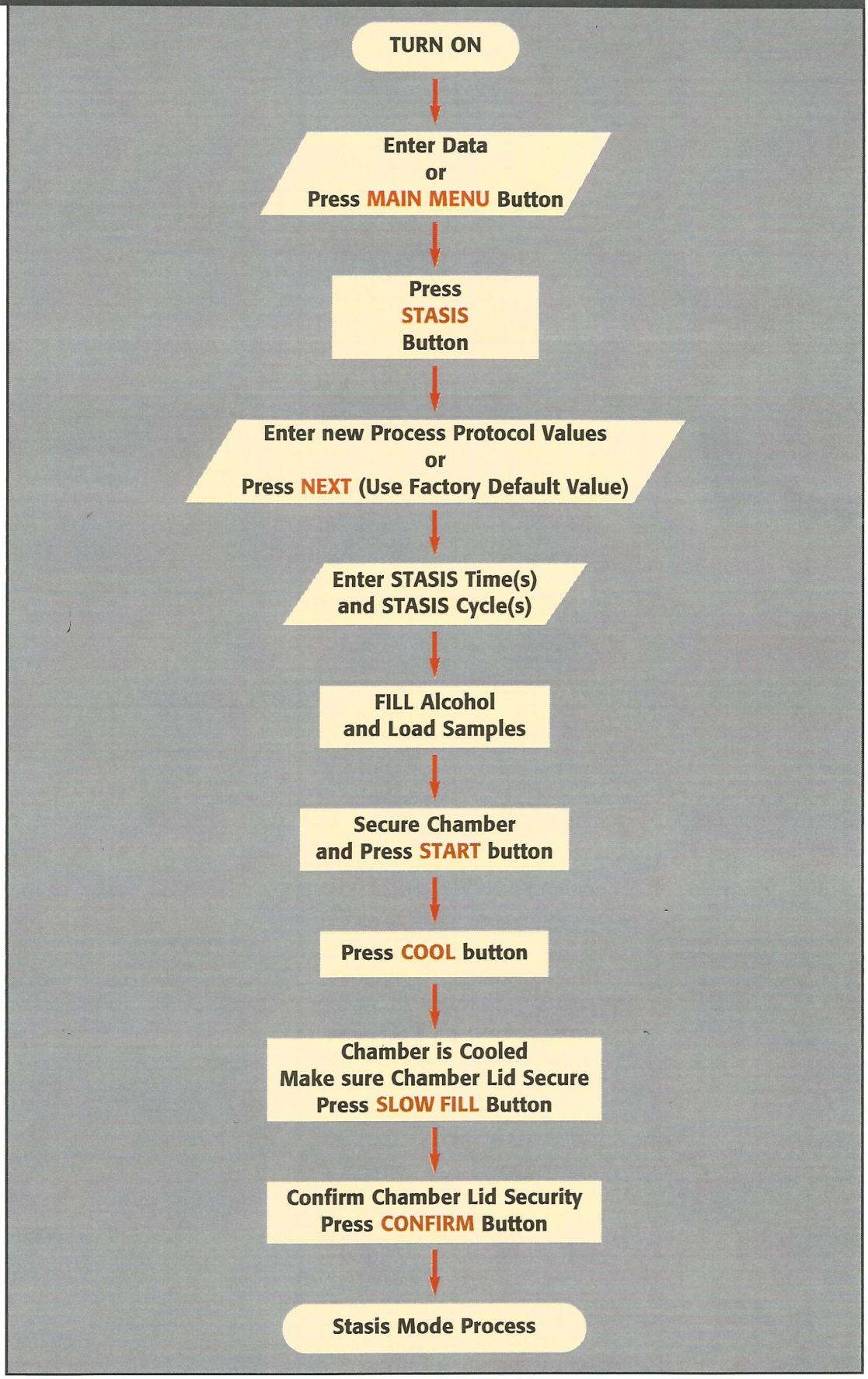


4.11 Flow Charts

Auto Mode



Stasis Mode



Recipe Mode