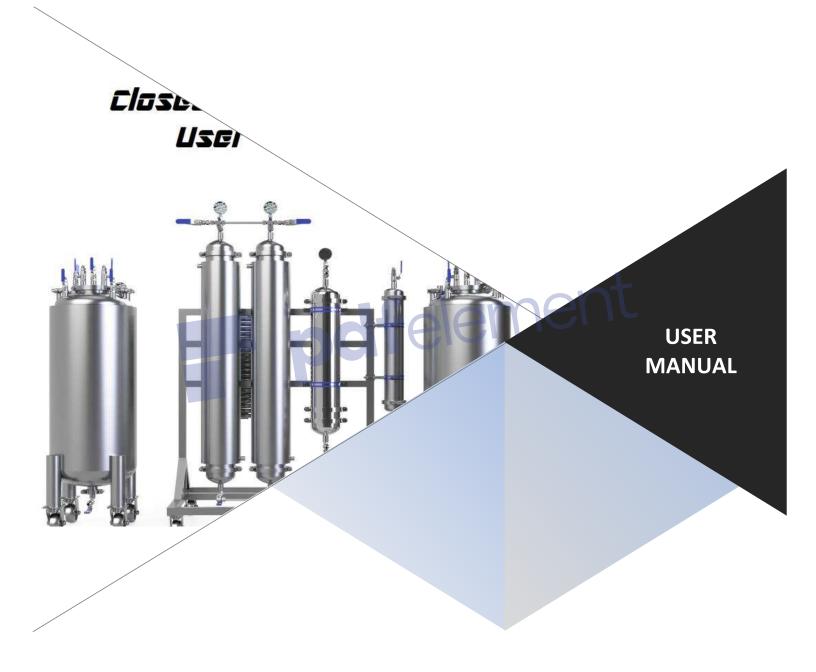


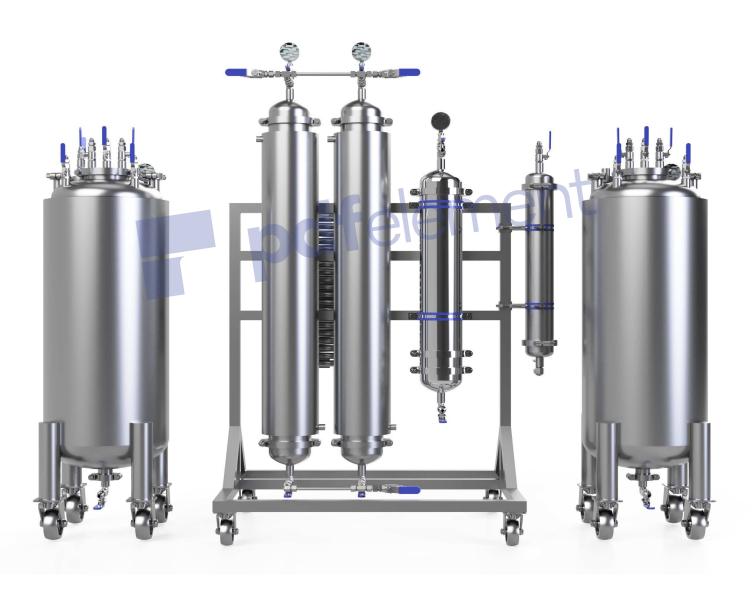
# STANDARD OPERATING PROCEDURE

# APPENDIX D





# 28 LB Closed Loop Extractor User Manual



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### Introduction to Closed Loop Extraction

Closed Loop Extraction also known as Hydrocarbon Extraction is performed by passing an alkane solvent over material to remove terpenes and other hydrocarbon compounds. The solvent is then distilled and recovered for later use, leaving the extracted compounds within the extractor base. Our Hydrocarbon Extraction systems are for use with butane (R-600) or iso-butane (R-600a) and a 70/30 butane propane mix only. Solvents will flow into the lowest pressure area of the system. Influencing thermals by chilling the solvent receiving vessel below the solvent's boiling point will allow the liquid solvent to condense in that vessel and reduce its pressure. This process in conjunction with our system allows the movement of solvent from one vessel to another without the loss or exposure of solvent.

### General Safety Information

If operated in accordance with this manual, common practices and safety procedures, this extraction system should provide a secure and dependable extraction process. Only operate this extraction system in extremely well ventilated areas. If this unit will be operated indoors, it must be operated in areas approved by your local Fire Marshal and be in accordance with local and state laws/ordinances. Always pressure Test your system before use to check for any possible leaks or breaches. Be sure to clean your gaskets only with compatible solvent, carefully inspect for wear or damage before each use.

Always wear static dissipative clothing discharging any potential static electricity before opening or interacting with the extractor.

This System may be used with Flammable Solvents. Use EXTREME CAUTION when operating this unit. Always operate in extremely well ventilated areas. Keep Away from any source of ignition.

ONLY USE THE RECCOMENDED WELCH CRV PRO 8 MODEL VACUUM PUMP OR OTHER <u>C1D1</u> RATED VACUUM PUMP.

# **Extraction Preparation**

The following are necessary supplies to operate the unit efficiently and safely.

- Various wrenches and sockets
- Refrigerant scale
- Nitrogen gas cylinder and regulator
- Combustible gas leak detector
- C1D1 booth / Explosion proof exhaust fan (recommended)
- Butane
- Dry ice
- Alcohol or Glycol (optional)
- Cleaning solvent (d-limonene recommended)



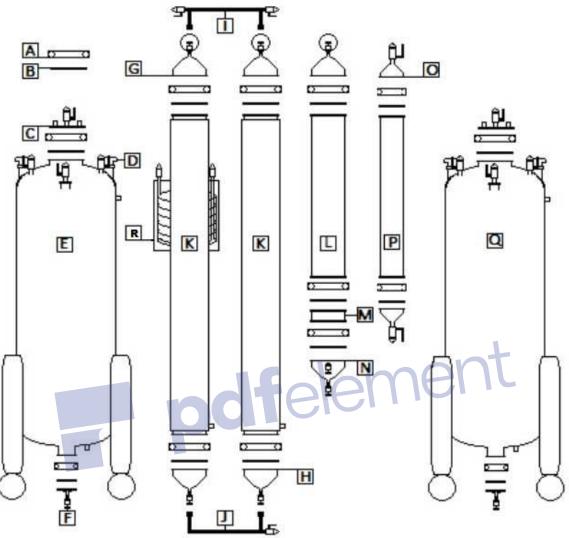
\*When assembling ensure clamps are seated and tightened properly (BUNA-N gaskets 20 inch pounds)

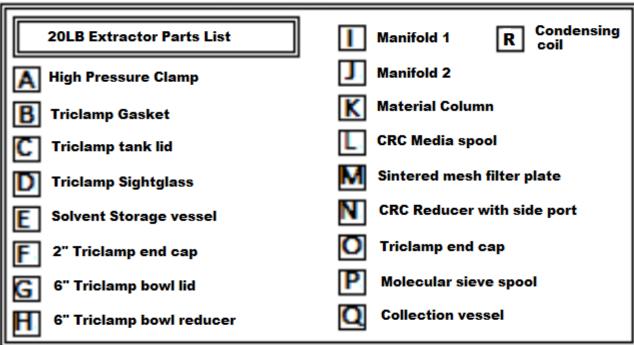
\*Be sure to tighten clamps evenly on each side. This unit must be pressurized and tested to 90 PSI prior to each use. Failure to do so may result in solvent leaks, exposing the solvent vapor to your working atmosphere.

-Please refer to the following diagram as a visual assembly guide.

#### Romovo Watermark No

### **ASSEMBLY GUIDE**





## **Pressure Testing Before Use**

Once the Unit is loaded with material and assembled, Pressure test the unit inspecting all gasket/clamp and hose connections. Pressurize the entire unit to 90 PSI with nitrogen gas or other safe means of pressure using one of the valves on the extractor lid. Allow the unit to rest under pressure for at least 15 minutes. Check your pressure gauge to ensure that no pressure is lost. If the unit does not show a change in pressure, Connect your vacuum pump and bring the unit to full vacuum.

\*When assembling ensure clamps are seated and tightened properly (BUNA-N gaskets 20 inch pounds)

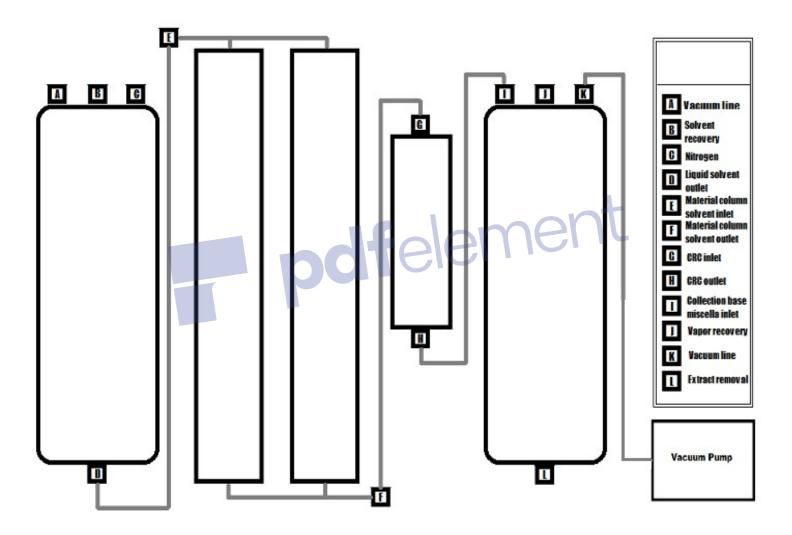
\*Be sure to tighten clamps evenly on each side. This unit must be pressurized and tested to 90 PSI prior to each use. Failure to do so may result in solvent leaks, exposing the solvent to your working atmosphere.





# **Valves and Components**

Valve and Hose Layout Please refer to the diagram below for valve and hose configuration.



### **Operation**

### **Influencing Thermals**

In order for solvent to flow throughout the system it must be influenced, you will need to make sure the supplying vessel is under higher pressure than the receiving vessel. One way of accomplishing this is to make sure the receiving vessel is colder than the supplying vessel. Solvent will always chase the cold temperatures and condense within the colder vessel. Temperature greatly affects pressure so maintaining proper temperatures is essential to the extraction process.

Nitrogen will be also be used to move solvent through the system by applying head pressure to the solvent tank.

For optimal results the solvent should be chilled before the extraction process. Note that the solvent should not be chilled below the boiling point of the chosen solvent, Removing pressure from the supply. Chilled solvent will assist in the dewaxing process, however this system uses a dewaxing chamber and will chill the solvent within the system to proper dewaxing temperature without prior solvent chilling. When distilling your solvent in the collection base it is important to note the boiling point of the most fragile compounds in your extracts. Recovery temperature should not surpass the boiling temperature of your lowest boiling compound.



It is important to have your tools and equipment readily available at all times (sockets, wrenches, scales, buckets). When assembling the unit we recommend that you apply all high pressure clamps in the same positions to ensure continuity if adjustments are needed during operation.

Always be sure to place the system on a level surface in a well ventilated area. Butane and other solvents are known to pool in cool stagnant areas so it is crucial to ensure all areas of your workspace have adequate air flow. If operating indoors your workspace must comply with the criteria for Class I, Division 1 work. Please consult your local fire marshal to ensure your workspace is in compliance with local laws and ordinances.

• Before the first run, it is important to clean all parts of the extractor. Oils, metal shavings and other contaminants may be present from manufacturing. Failure to properly clean the units parts can result in contaminated extracts.

### **Pre-run Testing**

Always test pressure test the extractor before every run. Load the material column with your choice of material, Assemble the rest of the extractor as pictured above. Be sure all filters and screens are in place and that filter plates have a paper filter.

For fresh frozen material, Subzero temperatures must be maintained during extraction so the any moisture solidifies and will not enter the collection chamber. This can be achieved by pre-chilling the solvent tank and by utilizing the dewaxing sleeve on the material column. (filling the sleeve with dry ice.)

The capacity of the material column can vary with the density of the packed material. It is recommended to firmly pack the material within the column to avoid solvent channeling ensuring all material has seen solvent. After the unit has been packed and prepared, attach nitrogen cylinder to manifold and perform pre-run pressure testing.

\*Every time you assemble the unit, it is crucial that the system is pressure tested to 90PSI with nitrogen gas. This ensures there are no leaks in the system and that all gasket, clamps and hoses are properly fastened and sealed. Allow the system to remain under pressure for at least 15 minutes to ensure there is no change in pressure before releasing the pressure and bringing the system to full vacuum.

• \*Always ensure that the nuts and bolts on all high pressure clamps are not worn or compromised by wear and tear. Failure to properly maintain clamps can result in unexpected clamp failure.

If no loss in pressure is observed you can begin to depressurize the system. Start by releasing the nitrogen pressure, then using your vacuum pump pull the system to full vacuum using multiple pressure gauges to ensure that full vacuum has been achieved. When finished the vacuum pump can be stored until its next use.

### **Introducing Solvent to the System**

Before you add solvent to the system it is vital the the solvent is pre weighed in your LP Tank to ensure the system is not overfilled. This also allows solvent to be weighed post run to ensure that all solvent has been recovered.

Solvent capacity of the 20LB Bi-Directional Extractor. Solvent capacity is directly related to the size of the collection base.

\*Solvent Capacity for this units Collection base = 105LBs

# **Beginning Solvent Flow**

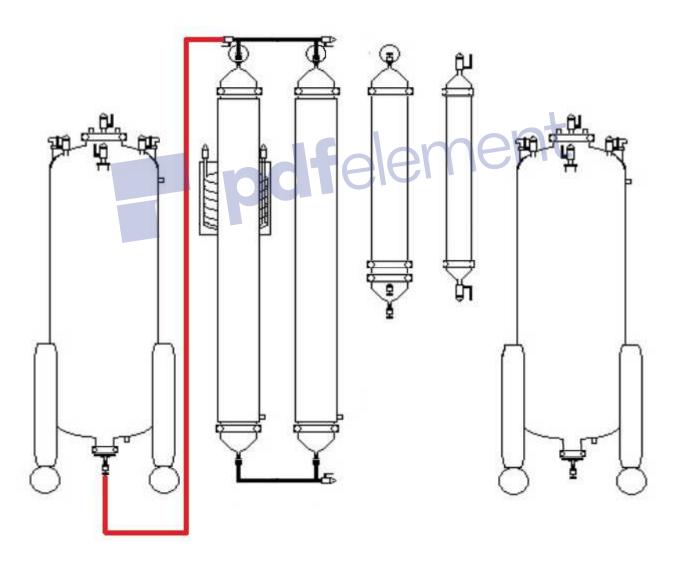
Connect the bottom port of the solvent tank to the material column manifold inlet on the top of the material columns using the SS line. When introducing solvent it is important to hook up to the bottom liquid port on your solvent tank. This port allows liquid solvent to be brought up rather than the gaseous contents. Nitrogen head pressure can be applied to help move the solvent through the system.

# **Top Fill Input**

Once connected, Open the valve on the bottom of the solvent tank and the valve on the material column manifold. Also ensure the valves on the material column lids are open.

# **Dewaxing**

Dewaxing is the process of coagulating fats and lipids within the material column to then be filtered out. Be sure to have dry ice in your material column sleeves, The extremely cold temperature will solidify fats and lipids. Stall the solvent within the material column for 30 minutes to 1 hour to allow for proper dewaxing. Keep the solvent tank chilled so that incoming solvent does not dissolve the solidifies fats.

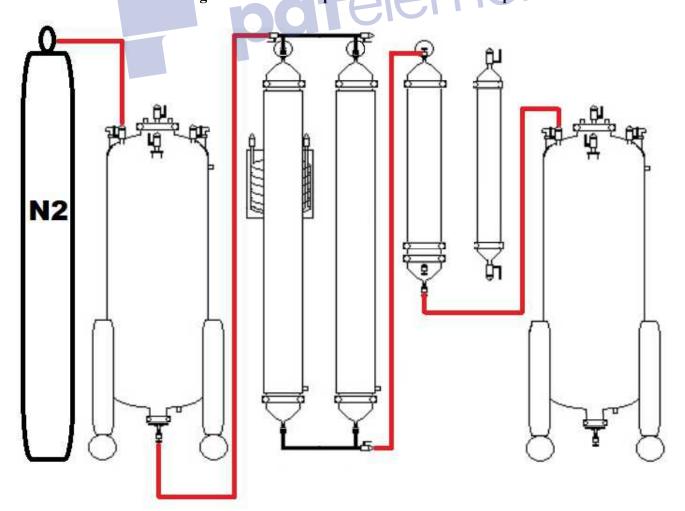


### Nitrogen Push through CRC

The Color remediation chamber is used to bleach or otherwise remediate the oil as is passes through this filtration system. The CRC uses a 1 micron filter plate. Due to the extremely small pore size of the filter, solvent flow rate is extremely restricted. In order to move solvent through the CRC adsorbent powders and 1 micron filter plate, Nitrogen vapor is used to create back pressure. The nitrogen is added to the head space of the solvent tank. Once empty of liquid solvent the vapor then creates head pressure and pushes liquid solvent through the material column and CRC chambers.

Turn your nitrogen regulator to 60-90 PSI and test your regulator before hooking to your system. Once you confirm your outgoing pressure add the nitrogen gas to the solvent tank. The nitrogen cylinder will be left open so that head pressure is maintained at the set pressure. Keep the nitrogen gas flowing until all of the liquid solvent has flowed into the collection base. Once the miscella has been transferred to the collection base the excess nitrogen pressure will need to be released. Keeping your solvent tank chilled allows the solvent to stay in a liquid state, This allows for easy removal of the nitrogen vapor without any volatile gases in the mix. Open a valve and use a hose to safely vent the nitrogen vapor. Pay attention to the state of the liquid solvent in the collection base. If the solvent is boiling then it is likely that you are releasing butane rather than nitrogen. In this case, Decrease the temperature of the collection chamber so that the butane condenses and then release the nitrogen.

\*throttling the inlet valve to the collection chamber allows users to slow the flow rate through the CRC in turn better utilizing the remediation potential of the adsorbent powders inside.



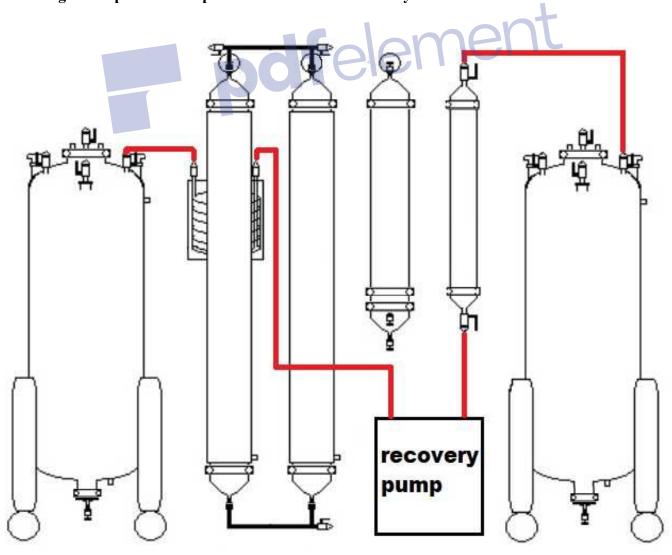
### **Recovery Process**

The recovery process involves the distillation of solvent in the collection base. Through distillation the impurities in the solvent will be left behind (in this case the Extract) while the gas moves to your LP solvent tank.

In order to efficiently recover solvent and preserve the full spectrum of the extract, A few simple rules must be followed.

First note the boiling point of your solvent, The LP tank must be chilled below this temperature to allow the solvent to condense. Keep the LP tank as cold as possible during the recovery process to allow the solvent to liquefy. The colder the receiving vessel is the faster the recovery process will be

Second, consider the compounds being extracted, It is important that the distillation temperature does not exceed the boiling point of your most fragile compound. In order to receive the full spectrum of your extract it is important that none of the extracted compounds are evaporated during the recovery process. Heat the collection vessel below the boiling point of your most fragile compound to evaporate the solvent for recovery.



### **Passive Recovery**

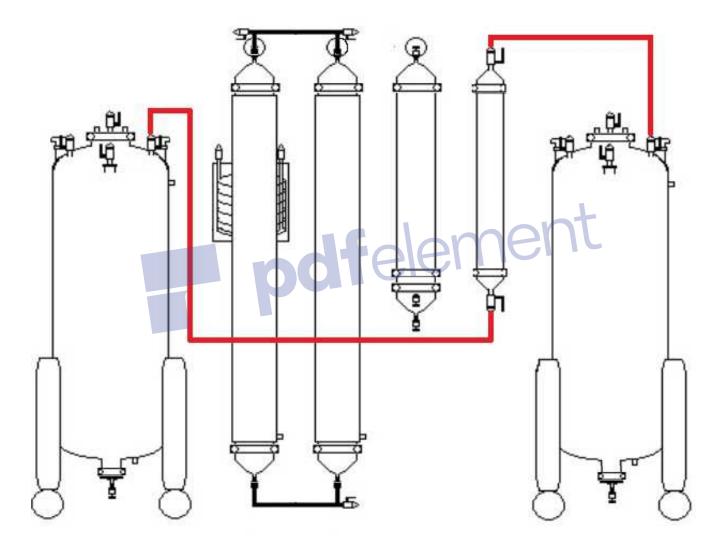
Passive recovery relies 100% on manipulating thermals to influence pressure within the collection base and LP tank. Since pressures are directly related to temperatures, we can raise and lower pressures with the use of heat and cold temperature baths. As temperatures rise, so do pressures.

As covered in the influencing thermals section, recovery is the distillation of the solvent that is used for extraction. You may refer to this section for recommended temperatures for recovery.

Before you begin recovery, please make sure all valves leading from the material column to the collection base are open so that solvent can be recovered from the entire system. Open the recovery valve leading from the collection base to the sieve. Turn on your circulation pump and heater to approximately 105F. The increase in temperature will cause the pressure within the extractor to rise. This changes the phase of the solvent. The solvent will then release any oils extracted and convert into its vapor form. As pressure rises the solvent vapor will transfer back into the LP tank. \* Turn the material column ball valves off if dry ice is being used around the material column. The cold temperature will attract butane during the recovery process. In this case the material column may hold onto some solvent through what is refereed to as vapor lock. The gas solvent inside the material columns will need to be recovered or released once the dry ice is removed and the column is back up to room temperature.

Turn on your recirculating chiller, set to the minimum temperature -30c or below. Once the temperature decreases, the pressure within the LP tank will decrease and create a vacuum effect. This will cause the solvent vapors to convert back into a liquid state. Maintaining a low temperature allows for faster and more efficient recovery.

# **Passive Recovery**



### **Efficient Recovery Methods**

In order to perform an efficient recovery process temperatures in the collection base and LP tank must be consistent. It is important that the majority of each vessel is mostly but not completely submerged in its warm/cold bath. Be sure to expose the bottom of the vessel to its warm/cold bath in order to maintain a consistent temperature without completely submerging the vessel.

To maintain warm temperatures the use of a submersion heater is recommended. Some submersible heaters can heat the water with a submersible heating coil while circulating the water throughout the bath, this allows for the most consistent temperatures while recovering. The collection bases temperature will cool due to the evaporation of solvent and the warm bath will quickly lose temperature without a heating element. It is important to maintain temperatures in order to perform and effective and efficient recovery.

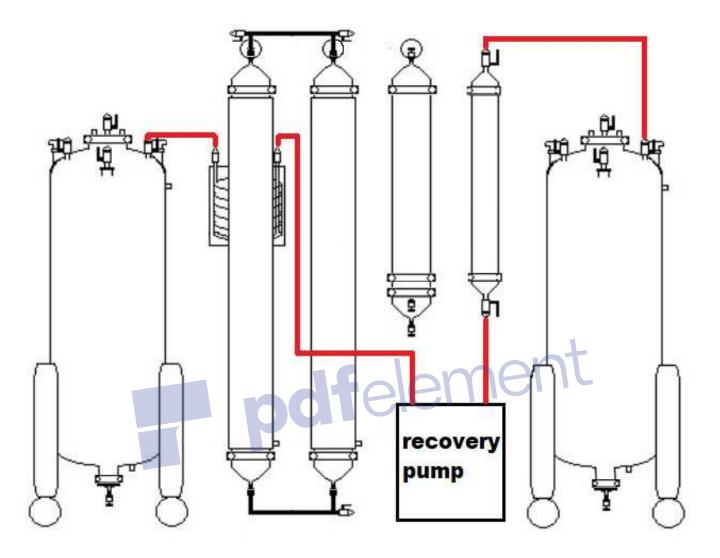
If not using a heating element the temperature of the bath must be closely monitored. To ensure that no compounds in the extract are evaporated it is NOT recommended to start with a higher temperature bath to compensate for temperature loss due to solvent evaporation within the collection base. Warm water will have to be replaced multiple times during the recovery process.

For cold baths, a dry ice/glycol slurry is optimal for maintaining subzero temperatures. This keeps the LP tank temperature lower then the solvents boiling point, ensuring no positive pressure will be present. When temperatures dip into the negative the LP tank pulls a vacuum on itself and will aid in the recovery process.

Note that incoming vapor from the heated collection base will bring warmer temperatures to the receiving LP tank, Thus you must also maintain cold temperatures as well as warm. Be sure the LP tank is submerged in the cold bath up to the valves. Replace dry ice as it evaporated during recovery.

Ice can be used instead of dry ice, However it will significantly increase the recovery time. Without reaching subzero temperatures the LP tank will not be able to pull a vacuum on itself. This causes a pressure increase in the LP tank, Slowing recovery.

# **Active Recovery**



Additional equipment consists of a molecular sieve (for removing moisture from solvent), recovery pump(to aid recovery process) and a cooling condensing coil (to condense solvent, aiding recovery). Further details on additional equipment can be found on the following page.

### Additional Equipment for Active Recovery

### Molecular sieve

The molecular sieve is used to remove moisture from the solvent as it is recovered. This component consists of a triclamp spool filled with desiccant beads that is hooked up in line with the recovery pump. Removing moisture prolongs the life of your pump and leaves you with a higher quality solvent to work with. It is important to check the moisture content of the beads and to regularly refresh the beads by drying them out. The moisture in the beads can be removed by baking them in the oven at 300F until the beads turn blue or to their original color.

### **Recovery pump**

Refrigerant recovery pumps are widely used for the extraction process and aids passive recovery by regulating pressures, speeding up the recovery process. Recovery pumps should be designed for use with flammable gas, be C1D1 rated, and be installed within the C1D1 area.

### **Cooling condensing coil**

The cooling condensing coil aids the recovery process by condensing vapors into liquid, reducing pressure to the LP tank. Cooling condensing coils are submerged in a cold bath to chill the solvent passing through the coil.

# **Active Recovery**

Once you are ready to begin the recovery process start by closing all valves except for the recovery valve and the valve on your LP tank. The collection base will need to be heated to start evaporation of solvent. Turn on your recovery pump to begin the recovery process. Keep in mind the same rules from the passive technique apply to active as well. IF the coil and LP tank temperatures are substantially lower than the boiling point of the solvent the LP tank will be able to pull a vacuum on itself, Aiding the recovery process. If these critical temperatures are not reached the recovery pump will be working against the pressures greatly increasing recovery time.

You can monitor the recovery process by using the sight glasses to check the level of solvent or by monitoring the weight of your LP tank, (Before beginning recovery tare the tank on your refrigerant scale and monitor the weight of the incoming solvent. The target temperature to preserve the full spectrum of your extracts is 105F. It is recommended to recover into negative pressures. This allows all of the solvent vapors to be recovered ensuring they are not exposed to your working atmosphere when opening the collection base.

\*\*\* Always perform regular tests and maintenance on your recovery pump. We recommend setting a schedule for routine cleaning and maintenance to ensure your pump functions properly.

### **Post Operation Procedure**

It is recommended to recover into negative pressures, ensuring that all solvent has been recovered. Failing to recover all of the solvent before opening the collection base will result in solvent exposure to your working atmosphere. When all of your solvent has been recovered you can begin to equalize the extractors pressure to atmospheric pressure. Depressurize the system by closing the valves on the LP and opening the recovery valve allowing the vacuum within the extractor to pull in air from your working atmosphere. Note that if your extractor has positive pressure solvent will be exposed to your working atmosphere. Completely dry your collection base before disassembling.

\*For rack mounted systems like the 20LB, 1-2 PSI of liquid solvent should be left in the collection base to ensure the oil is at the proper viscosity for removal.

### **Removing Extracts**

Always wear static dissipative clothing discharging any potential static electricity before opening or interacting with the extractor.

After recovering to 1-2 PSI dispense the extracts by slowly opening the ball valve on the bottom of the extractors collection base and dispense in an approved container for post processing. This process WILL expose you directly to solvent vapors. Use extreme caution and only perform extractions in a Class 1 Division 1 laboratory.

### **Maintenance**

It is important to break down the unit and clean the parts and gaskets individually. Be sure to use a compatible solvent to clean gaskets. Some solvents may degrade the gaskets. Isopropyl alcohol can be used but be sure to wipe gaskets dry to avoid them breaking down. Failure to properly clean the unit before or after each use can result in contaminated extracts.