LABCONCO

Sample Preparation

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LABCONCO®

Lyophilization, evaporation or concentration

Which is best for my samples?

Lyophilization, vacuum evaporation and nitrogen blow-down evaporation are all methods to reduce sample volume; however, each method involves a different process and affects samples differently. It is important to identify which evaporation method is most appropriate for your sample in order to identify the right specific equipment to use.

Lyophilization, evaporation and concentration cause liquid or solid molecules to undergo phase changes, resulting in aqueous or solvent molecules leaving the sample as a vapor. The main force that drives the phase change is heat energy. The best evaporation method for a sample is mainly determined by how much heat the sample can withstand.

Forces that Catalyze Evaporation

If a sample is not affected by heat, simply place the sample on a hot plate and let it boil until the sample volume is reduced to the desired level. Unfortunately, most samples are affected by heat, with biological samples being the most sensitive. In heat sensitive samples, the amount of heat driving the phase change must be limited or the sample will be damaged. When heat is limited, other forces must be used to drive molecules through the phase change instead.

Concentrator

Evaporator

Lyophilizer

Applying a vacuum atmosphere is a common technique used to force molecules through the phase change during evaporation or lyophilization. Boiling points of liquids are greatly reduced under vacuum, so phase changes occur at lower temperatures and less heat input is required. As a result, **vacuum concentration** or **lyophilization** are the most optimal methods for biological or heat sensitive samples.

Another technique that expedites the phase change of solvents is introducing a nitrogen stream at the surface of samples. The nitrogen stream disrupts the balance between the liquid and vapor phase and encourages the molecules to move into the vaporous phase. As a general rule, nitrogen blowdown evaporation requires a higher heat input than vacuum evaporation. Because of this, **nitrogen blowdown evaporation** is commonly used for volatile solvents or samples that are not damaged at high temperatures.

Lyophilization (Also Known as Freeze Drying)

Lyophilizers use deep vacuum (< .200 mbar) and heat to remove moisture from a sample. The drying process, or phase change, in lyophilization is unique and is called sublimation. In sublimation, molecules go directly from the solid phase (ice) to a gaseous phase (vapor) without passing through the liquid phase. Lyophilization requires a frozen sample. If the sample's freezing point is suppressed by the presence of solvents, the solvents should be removed with vacuum concentration prior to lyophilization so that the sample can then be frozen solid.

By circumventing the liquid phase in sublimation, the biological viability of many samples is preserved. This makes lyophilization a unique type of evaporation as it also preserves biological samples.

Vacuum Concentration/Evaporation

In vacuum concentration/evaporation, the sample is dried by converting liquid to vapor. Vacuum concentrators and evaporators take the liquid sample to 99% dryness in a relatively short amount of time and can accommodate most samples regardless of their freezing or boiling point.

Vacuum concentrators and **evaporators** are commonly used in sample preparation, and use centrifugal force, heat and vacuum to remove moisture from a sample. What makes concentrators different from evaporators is how the centrifugal force is generated and how the heat is supplied to the sample.

In a vacuum concentrator, samples are held in a rotor that spins at 1700 RPM creating a centrifugal force. The centrifugal force prevents liquids from **bumping** (splashing, misting or otherwise breaking the sample's unified surface tension) out of the tube when vacuum is applied. Heat is applied indirectly through the walls of the vacuum chamber.

Vortex evaporators
for multiple, small to large sized liquid samples

Concentrators
evaporate many, small to medium sized samples

Lyophilizers or freeze dryers
evaporate any size or number of samples under deep vacuums that must be below 0.400 mBar

A **vortex evaporator** uses centrifugal force, heat and vacuum to remove moisture from a sample. Unlike the spinning motion of a vacuum concentrator, the vortex evaporator creates a vortex motion within the sample container so that centrifugal force holds the sample into its container while vacuum is applied to the liquid sample. Without an opposing force to hold the sample into its container, it would **bump**, or splatter out of its container. **Vortex motion**, washes the analyte (analyzed material) down the sidewalls of the glassware, increasing the recovery of the analytes being tested. In vortex evaporation, heat is applied directly into a sample through a block that holds the sample.

Nitrogen Blowdown Evaporation

Nitrogen blowdown evaporators use heat and a nitrogen stream to evaporate moisture from a sample. Evaporation rates can be increased if a vortex motion is created to increase the surface area of the sample. **Dry block heaters** are commonly used in these evaporators and offer many advantages over water baths.

	Lyophilizers	Concentrators	Evaporators
Sample Volume Range	Micro liters to 10L	Micro liters to 25 ml	4.5 - 450 ml
Sample Tube Size	Micro plates Microcentrifuge tubes Test tubes Flasks Serum bottles Bulk	Micro plates Microcentrifuge tubes Test tubes	Test tubes 170 ml and 600 ml tubes
Processing Time Range	12 hours to 1 week	25 minutes to 6 hours	25 minutes to 6 hours
Components Required	Freeze dryer Drying chamber Vacuum pump	Concentrator Rotor Vacuum pump	Evaporator Block Vacuum pump for system N_2 regulator for N_2 system
Accessories	Glassware Adapters	Cold trap CentriZap Glassware Secondary traps Additional rotors	Glassware Secondary traps Racks Additional blocks
Pump Type Required	Rotary vane Diaphragm	Rotary vane Diaphragm Hybrid	Diaphragm
Applications	Aqueous Protein peptide Vegetation Environmental HPLC Pharmaceutical	Peptides DNA Protein Oligonucleotides Pharmaceutical	Environmental EPA Toxicology Biological MIcrobiology Food chemistry

Introduction to freeze drying

Freeze drying, also known as lyophilization, is a process used for sample preparation. With its unique preservation properties, freeze drying has a large variety of applications with an ever-growing list of new uses each year.

What is freeze drying?

Freeze drying is a process in which a completely frozen sample is placed under a vacuum in order to remove water or other solvents from the sample, allowing the ice to change directly from a solid to a vapor without passing through a liquid phase. This process, called sublimation, along with the minimal heat input that is required, is ideal because of the long-term preservation properties it provides to the integrity of the sample's biological and chemical structure. Lyophilization can be achieved in various volumes, from small at-home freeze dryers all the way up to large, production-scale equipment.

Equipment required for freeze drying

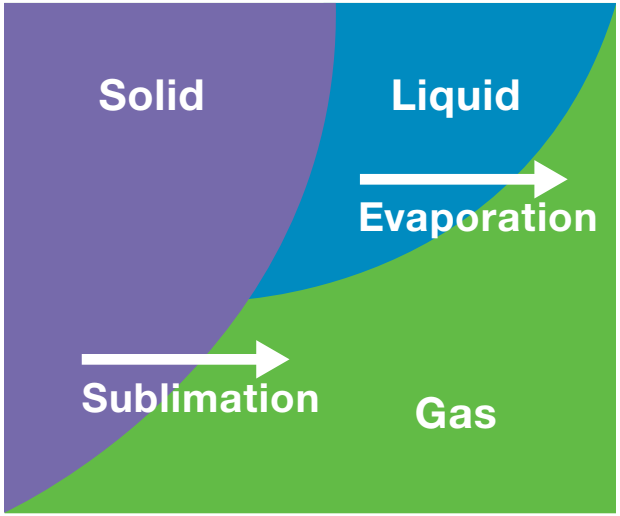
To successfully freeze dry a sample, your equipment will need to meet certain requirements. First, the collector coil of your freeze dryer will need to be 15-20 degrees colder than the freezing point of your sample in order to trap the water vapor that is being released. You will also need a vacuum pump that can reach a minimum of .002 mBar. This deep vacuum establishes negative pressure, forcing the free-flowing water molecules that have left the sample via sublimation to evacuate the environment around the sample and travel to the collector coil. You will also need to have a drying accessory, such as: a manifold, chamber or tray dryer, and glassware or trays to contain your sample. Although not required, an end-point detection system makes the (sometimes days-long) freeze drying method easier to manage by keeping you informed of when your sample has finished drying.

Required Equipment

- **Freeze dryer**
  a vacuum tight chamber with collector coil
- **Vacuum pump**
  that can reach an ultimate vacuum of .002 mBar or lower
- **Vacuum-tight sample holders**
  flasks or vials with adapters, or chamber with trays

Three stages of lyophilization:

- Pre-freezing
- Primary drying
- Secondary drying



A typical phase diagram

Pre-freezing

The pre-freezing stage is the most important stage of the freeze drying process. In this stage, sample material will need to be cooled to at least the temperature of the melting point for that sample. This ensures the sample will be completely frozen and can then undergo sublimation. If the sample isn't frozen solid, evaporation will occur and the sample will not achieve the same preservation properties that occur with sublimation.

The rate at which your sample freezes will affect the size of the ice crystals that form. If not done properly, it can impact the speed of reconstitution, length of the freeze drying process and integrity and stability of your sample.

Larger ice crystals facilitate faster and more efficient lyophilization because water molecules are able to move more freely out of the sample during sublimation. For samples like food or tissues, large crystals can break the cell walls and damage your sample. In these situations, it is best for freezing to be done quickly through flash freezing, creating smaller ice crystals.

Primary Drying

Primary drying begins when you start your freeze dryer and vacuum pump. With the low pressure environment, evaporative cooling of the sample begins, allowing for energy in the form of heat to speed the freeze drying process. At the end of primary drying, roughly 93% of the water in the sample is sublimated out. This stage can take several days, depending on the sample type and heat input. For laboratories that are using their freeze drying equipment for sample preparation and resuspension, primary drying is where the run would end. For long term preservation of the sample, the run would continue on to secondary drying.

Secondary Drying

In the secondary drying phase, water molecules that are bound to the sample are released. Additional heat is added in this stage to drive off excess moisture, leaving behind a moisture content of about 2%. Secondary drying is typically used in samples that are being prepared for long term preservation and storage.

Why freeze dry?

While freeze drying can accomplish many things, its primary goal is to protect the biological and chemical structure of a sample through the process of sublimation. Once complete, the sample can be used for testing, long term preservation, or in the case of food, eaten. In the laboratory, the applications of freeze drying are limitless.

How to determine end point during lyophilization

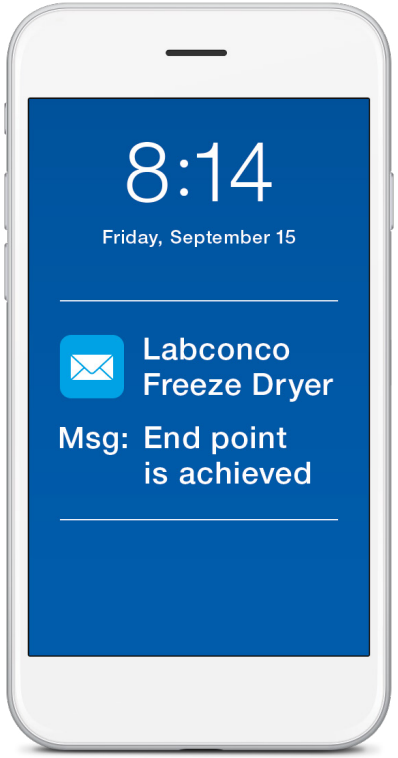
Freeze drying, or **lyophilizing**, is an ideal sample prep method for heat-sensitive samples, but it can be difficult to determine when the freeze drying process is complete. This "**end point**" can be most challenging to determine when freeze drying samples in flasks. The current method requires visual inspection to determine if a sample is fully lyophilized, offering no guarantee of completion.

If a sample has not reached end point and is removed from the lyophilizer prematurely, the ice will melt and rehydrate the sample that has been dried. For many applications, this would require the entire sample to be resuspended and the process to begin all over. Because of the need to start over and the inability to determine when freeze drying is complete, many freeze dry runs are extended beyond their end point, wasting processing time.

However, there is a solution using vacuum sensors to detect the end-point condition for optimum sample processing efficiency.

Video: Programming your freeze dryer with Lyo-Works OS

Labconco's End-Zone™ **End Point Detection System** takes the guesswork out of determining when end point is reached during flask freeze drying. How does it work? Utilizing two vacuum sensors, it detects the end point by comparing the vacuum level of a sample flask to the vacuum level of an empty freeze dryer valve which acts as a reference point. Because the presence of vapor molecules within the flask raises the vacuum level, the vacuum level inside of a flask that is undergoing lyophilization will be higher than the system's vacuum level. Once lyophilization has completed, vapor molecules are not present to elevate the vacuum level within the flask, thus the vacuum level in the flask becomes equal to the system vacuum level.



The **End-Zone Starter Kit** requires a freeze dryer with the **Lyo-Works**™ Operating System. The Lyo-Works OS will display an alert when end point has been reached. If the freeze dryer is connected to a network, it can send an email to notify the user that freeze drying is complete.

If you're frustrated trying to determine your samples' end points, contact Labconco for help to find a solution that works for you.

Evaporation solutions for all types of labs

Your samples are valuable, just like the equipment you use. This is why Labconco RapidVap Dry Evaporators were designed to protect both. The chamber and sample block are PTFE-coated while all mechanical components are isolated from chemical fumes. This resilience to strong chemicals, along with repeatable methods and programmability, gives you the confidence you need to run your samples unattended without issue. With four models of RapidVap Dry Evaporators to choose from, the evaporation process can be optimized to meet the needs of any application.

Efficiency

RapidVap Dry Evaporators are made to quickly reduce multiple samples to complete dryness or to an end point volume using direct heat, a vortex motion and either a vacuum or nitrogen stream. The vortex motion used by the RapidVap Vacuum, N_2, and N_2/48 Evaporators produces remarkable evaporation rates by dramatically increasing the surface area of your samples, saving you precious time. The constant washing of solvent down the sidewalls of the glassware also increases the recovery rate of the analytes being tested.

Vertex

Vacuum

N_2

N_2/48

With the RapidVap **Vertex**, evaporation occurs using a combination of **nitrogen blowdown** and **heat**. Nitrogen blowdown reduces the partial pressure directly over the liquid, using a slight vortex motion within the sample to speed evaporation, creating a gentler way to evaporate. The dry block supplying heat in the Vertex is angled to increase the surface area of your samples for faster evaporation.

All RapidVap Dry Evaporators feature a **dry block heater** that supplies a controlled amount of heat to the samples. Unlike water baths, the dry block heater also eliminates the potential for condensation accumulating on the lid and causing cross contamination. Additionally, the block heaters require no additional maintenance.

Versatility

Sample processing and methods can be fluid as your research changes over the years. That is why the RapidVap is designed to accommodate your needs with interchangeable sample blocks.

- RapidVap Vertex for sample volumes up to 60 ml
- RapidVap Vacuum, N_2 and N_2/48 Systems for volumes up to 450 ml

End Point Detection

For certain types of research, it is important the samples not evaporate to complete dryness. The RapidVap Vacuum, N_2 and N_2/48 **Evaporators** offer unique ways to ensure your samples are protected, allowing you to work on other things instead of watching your samples dry.

The **Cool-Zone**™ insulates samples in a glassware stem for end point, dramatically slowing down the evaporation process for the last few milliliters of sample. Glassware with stems are offered in a variety of end-point volumes.

As a backup to the Cool-Zone, the RapidVap monitors the system temperature in the block and in the heater. During operation, evaporative cooling of the solvent creates a differential between the block and heater temperatures. Once evaporation is nearly complete, the two temperatures equalize indicating end point is near. The alarm sounds and the Preheat/End Alarm indicator light flashes.

The operator can always set the end point time and when set time has expired an audible alarm sounds and the RapidVap automatically turns off all functions.

Vertex

- For sample volumes up to 60 ml

Vacuum

- For sample volumes up to 450 ml

N_2

- For sample volumes up to 450 ml

N_2/48

- For sample volumes up to 450 ml

Environmental Protection

As we continue to find new ways to help protect our environment, recovering solvents during evaporation has become increasingly important. The RapidVap Vacuum Evaporator can be used with a cold trap, in addition to the RapidVap Trapping Valve, to collect large volumes of volatile solvents. This lengthens the vapor dwell time within the cold trap, dramatically increasing trapping efficiency.

RapidVap Dry Evaporators are an efficient, versatile solution to evaporation for a broad range of sample preparation applications.

How to select the right vacuum pump

4 main types of lab pumps:

- **Rotary Vane**
- **Diaphragm**
- **Combination**
- **Scroll**

There are many vacuum pump options available, making it difficult to know how to select the best pump for your application. Vacuum pumps are available with varying displacement capacities and ultimate vacuum levels.

There are four main types commonly used in laboratories for freeze drying (lyophilization), evaporation and concentration.

Rotary vane pump

Traditional **Rotary Vane** (RV) vacuum pumps are often used because they can have a lower up-front cost, are smaller than other pumps, and can be used for multiple applications. RV pumps use oil to ensure a tight seal, lubricate the working parts and remove heat to cool the rotors.

If solvents are not trapped or recovered before they enter the RV vacuum pump, they can condense in the pump oil and damage the inside of the pump. It's necessary when using an RV pump to collect the evaporated vapors upstream of the pump. Condensers or Cold Traps are commonly used to collect these vapors and protect the pump.

Another downside of the RV pump is that the oil must be changed. This is a costly, and potentially messy, process. It is recommended that the oil in these pumps be checked regularly and changed approximately every 3,000 hours of use.

The life of the vacuum pump depends on the maintenance of the oil. Even if you use a system to automate oil changing, time is still lost while the pump is out of use for maintenance.

Rotary Vane vacuum pumps reach deep ultimate vacuum levels and have high displacement capacity. This makes them a good choice for freeze drying applications. RV pumps work especially well for aqueous samples and solvents with high boiling points that have vapors that can be easily trapped before they reach the pump.

Diaphragm pump

Diaphragm vacuum pumps are dry (oil-free) pumps that operate using a pulsing motion to open and close valves to move air. This design eliminates the need for oil. The valves are often made of polytetrafluoroethylene, also known as **PTFE**, making the pump resistant to corrosives and less susceptible to damage from vapors.

While they can have a higher up-front cost, they do not use any oil so operation and maintenance costs are significantly lower than pumps that require oil. Diaphragm pumps can handle highly viscous liquids and be used with a wide range of samples.

Ultimate vacuum levels of these pumps are not extremely deep and displacement capacities are much lower than other types of vacuum pumps. Therefore, compatible applications are limited to those requiring higher ultimate vacuum levels.

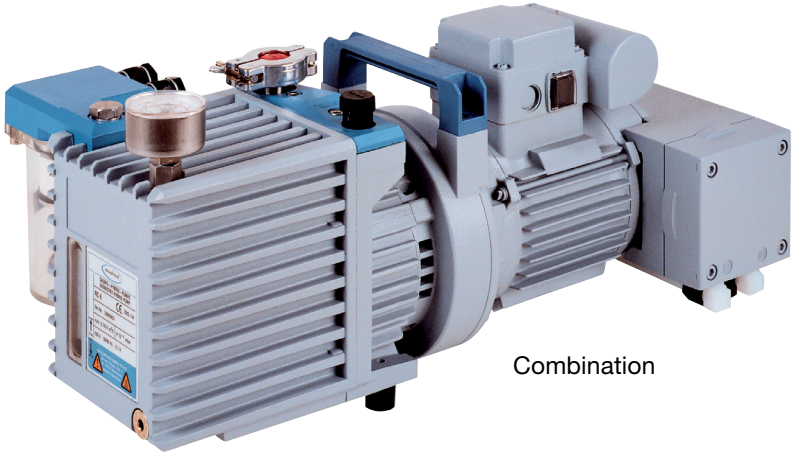
Diaphragm pumps are one of the most chemical- and corrosion-resistant types of pumps. Therefore, almost any type of sample, even those containing a combination of solvents and acids, can be used with these pumps, making them a good choice for both evaporation and concentration.

A diaphragm pump cannot be used for freeze drying since there isn't a deep enough depth of vacuum.

Did you know?

Diaphragm pumps are one of the most chemical- and corrosion-resistant types of pumps

Diaphragm



Combination

Combination/hybrid pump

Combination vacuum pumps, also known as Hybrid vacuum pumps, have both a Rotary Vane and Diaphragm pump together in the same vacuum pump. In a combination pump, the diaphragm pump keeps the oil of the RV pump under negative pressure to reduce or eliminate vapors going through it and condensing in the oil.

This design keeps the oil cleaner and allows for less frequent changing – oil lasts up to 10 times longer between changes compared to RV pumps. So while there may be a higher up-front cost than an RV pump, the operation costs are lowered as less replacement oil is needed and less time is lost to maintenance.

The ultimate vacuum level and displacement capacity with combination pumps is similar to those of RV pumps. Because the diaphragm pump is incorporated into the design, these pumps are better at handling acids and solvents than RV pumps.

Combination pumps are recommended for freeze drying corrosive or volatile samples as they can be used with acidic samples and those containing harsh chemicals such as TFA, acetonitrile, HBe and Nitric acid.

Scroll

Scroll pump (dry vacuum)

Scroll vacuum pumps are dry pumps (oil-free) that use two spiral scrolls to compress air and vapors and move them toward the exhaust. While they can have higher up-front costs, the lifetime operation costs are much lower because they don't require oil and very little maintenance is needed.

It is recommended that the scrolls be changed **every 40,000 hours** of use. By eliminating the oil, the hydrocarbon-free design means these pumps are environmentally friendly as well. Scroll pumps handle water vapor better than most types of pumps and make significantly less noise during operation.

Because the scrolls are made of metal, even on chemical- or corrosion-resistant models, only samples with **acids below 20%** are recommended. Compared to diaphragm pumps, scroll pumps can reach

Scroll pumps are recommended for freeze drying as they can be used with aqueous and solvent samples, including acetonitrile. They may be used with concentration applications also, however the cost tradeoff may make another type of pump more attractive.

No matter what type of pump you're using, one of the most important things to remember about vacuum pumps is that whatever goes in the pump will come out! Even with chemical traps, you need to take other precautions when samples include potentially hazardous vapors. Venting into a fume hood is required when using hazardous samples.

Download the Vacuum Pump Selection Guide

The Vacuum Pump Selection Guide provides helpful information on choosing the right pump for your laboratory product. Several types of pumps and their uses are discussed.

4 questions to consider before buying a lyophilizer

Lyophilization, commonly referred to as freeze drying, is used to process samples for long term storage or as a sample prep step. Using the process of sublimation where a sample goes from a solid to a gas, moisture is removed from the sample using heat and a deep vacuum. This vapor is then collected on a cold coil in the form of ice. In order to choose the correct lyophilizer for your sample throughput, consider the following questions:

1. What type of samples are you processing?

Knowing what components make up your samples is important. In order to effectively trap the vapor coming off your samples, you need a temperature differential of 15-20° between the samples' freezing point and the collector coil of your freeze dryer. If the collector is not cold enough the vapor will bypass the coil and enter the vacuum pump, potentially causing damage. It is also important to consider whether any of your samples are corrosive to stainless steel. Even in small concentrations, acids or other harsh solvents can damage stainless steel over prolonged use. In these cases, PTFE coating of the collector and coils is recommended to prevent rust and other irreparable damage.

2. What is the total sample volume per run?

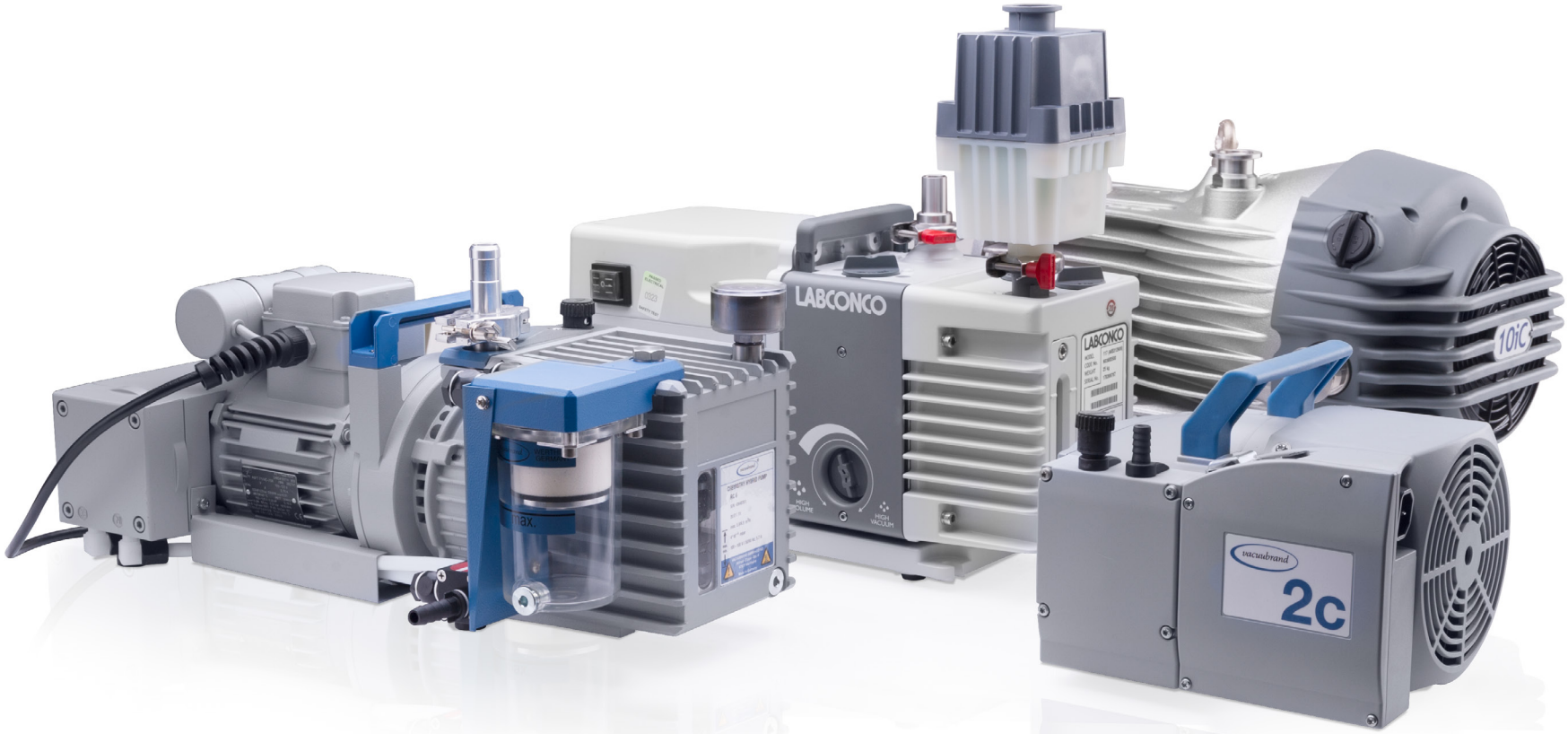
Generally, Labconco FreeZone Freeze Dryers handle a sample volume approximately half of the total ice holding capacity of the freeze dryer. For example, a 6L freeze dryer will be able to process about 3 liters of sample volume per run. If you were to put 6 liters of volume on the freeze dryer you would overload the system. This would result in poor vacuum, an increase in collector temperature and a low recovery of solvent on the coils. The samples would be unable to maintain their frozen state and the preservation properties of sublimation would be lost because the samples would begin to melt and evaporate.

3. How do you plan to freeze dry samples?

How you process your samples directly impacts the drying accessory needed for your freeze dryer. If you need to stopper samples under vacuum, a stoppering tray dryer or mini stoppering chamber is required. If you are freeze drying in test tubes or falcon tubes as part of a sample prep step, test tube holders are available to place in flasks. There are many accessories available to customize a freeze dryer to your specific needs.

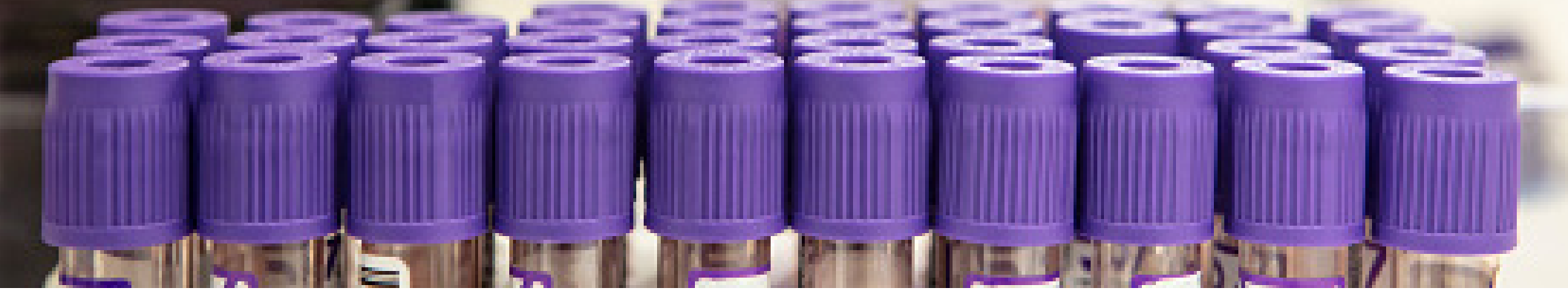
The format used to freeze dry samples can also impact the rate of lyophilization. Larger sample volumes freeze dried in flasks will take longer to complete than small vials or thin layers of bulk material. For flask freeze drying, the way you freeze the sample also makes a difference to the rate of sublimation. Samples that are slant or stub frozen will have slower sublimation rates compared to thinner, shell frozen samples. This is because freeze drying occurs from top to bottom in a sample, requiring moisture at the bottom of the flask to move through already dried material.

To ensure time is not lost when processing flask freeze dried samples, consider using the End-Zone End Point Detection System to take the guess work out of visual inspection to determine when samples are complete. End-Zone compares the vacuum level of an empty manifold valve to the vacuum level inside a sample flask to determine when the user specified level of dryness has been achieved, alerting the user when the run is complete.



4. What type of vacuum pump is required?

Freeze drying always requires the use of a vacuum pump. If you are working with aqueous samples, a rotary vane pump will work. However, if you are working with any acids or solvents, a hybrid or scroll pump is recommended. If you freeze dry solvents or acids with a rotary vane pump and any amount of vapor bypasses the collector, those solvents will wind up in the pump oil and damage the pump. With the use of any vacuum pump, make sure the pump meets the minimum specifications required for the operation of your lyophilizer.



4 factors to consider when selecting a concentrator

Labconco offers a wide variety of centrifugal concentrators to meet almost any need but determining which concentrator you need for your application doesn't have to be difficult. You just need to ask the right questions. The following information will help you select the concentrator that's right for your work.

1. What type of samples should I use?

Concentrators can be used for most sample types but are a great option for heat-sensitive samples. The most common way to evaporate something is by applying heat to the sample and boiling off the liquid. However, if you are working with a heat-sensitive application, such as biological samples, the heat that drives the evaporation process must be limited. With a vacuum concentrator, the vacuum atmosphere lowers the pressure level, allowing evaporation to occur at low temperatures. Heat is also applied from the chamber wall of the concentrator and does not come into direct contact with the sample. For samples that require additional protection from heat input, a chilled concentrator, like the Labconco Refrigerated CentriVap, is a great solution. The Labconco model allows you to set the chamber temperature down to -4° C.

Cold traps can be used to minimize impact on the atmosphere by trapping the solvents before they're exhausted out of the lab.

2. Can I use my concentrator as a centrifuge?

CentriVap Evaporators are not a centrifuges, so the quick answer is: no, you cannot. For a more detailed explanation see page 38 under, **Need to Know: CentriVap Centrifugal Concentrators**.

3. When do I need a cold trap?

Cold traps are needed to create a closed system. This means all the solvents that are being evaporated are collected in the cold trap. However, not all samples require a cold trap in order to evaporate. Cold traps are necessary if your lab does not have a fume hood to safely exhaust harmful evaporated solvents. They can also be used to minimize impact on the atmosphere by trapping the solvents before they are exhausted out of the lab.

However, in certain cases a cold trap is required. If you are using a rotary vane (oil) vacuum pump, a cold trap is necessary to protect the pump from liquids and vapors that could contaminate the oil and damage the pump. A cold trap is also required if you would like to lyophilize samples with your concentrator, along with a pump that can produce a deep vacuum.

If a cold trap is not being used when concentrating large volumes, a glass trap should be placed inline between the concentrator and vacuum pump. The trap will collect any liquids that are condensing in the vacuum tubing before they reach the vacuum pump, preventing pump damage.

Do all CentriVap Concentrators require a vacuum pump?

A vacuum pump is required to operate all centrifugal concentrators. However, certain Labconco models include a built-in chemical resistant diaphragm vacuum pump and sample rotor. These models include the CentriVap micro IR, DNA and the Complete. All others require the purchase of a diaphragm or rotary vane vacuum pump.

Labconco offers an extensive selection of centrifugal concentrators with models designed for a variety of applications. To ensure you get the best CentriVap for your needs, use the **Lab Evaporation Scout** on the Labconco website or contact one of our application specialists for additional support.

Freeze Dryers

Steps to Selection and Labconco Product Lines

1 Collector Temperature
(15 to 20 degrees colder than sample freezing point)

- -50° C — Aqueous Samples
- -84° C — Aqueous/Solvent Mixtures
- -105° C — Aqueous/Methanol Mixtures

2 Collector Size
(Total sample volume x2)

PTFE option for corrosive chemicals available on all models except Triad

- Aqueous Samples: 2.5 L Benchtop, 4.5 L Benchtop, 8 L Benchtop, 6 L Console, 12 L Console, 18 L Console
- Aqueous/Solvent Mixtures: 2.5 L Benchtop, 4.5 L Benchtop, 2.5 L Triad, 6 L Console, 12 L Console
- Aqueous/Methanol Mixtures: 4.5 L Benchtop

3 Accessories

- Tray Dryers: Vials, Trays
- Manifolds: Flasks, Manifolds, End-Zone
- Drying Chambers: Chambers, Add-ons

4 Vacuum Pump

- -50° C — Rotary Vane
- -84° C — Combination or Scroll
- -105° C — Combination or Scroll

Freeze Dryers

2.5 L, 4.5 L, 8 L

Benchtop

2.5 L

Triad Benchtop

6 L, 12 L, 18 L

Console and Console with Stoppering Tray Dryer

Accessories

Tray Dryers

Manifolds

Drying Chambers

Vacuum Pumps

Rotary Vane Pump

-50° C

Combination Pump

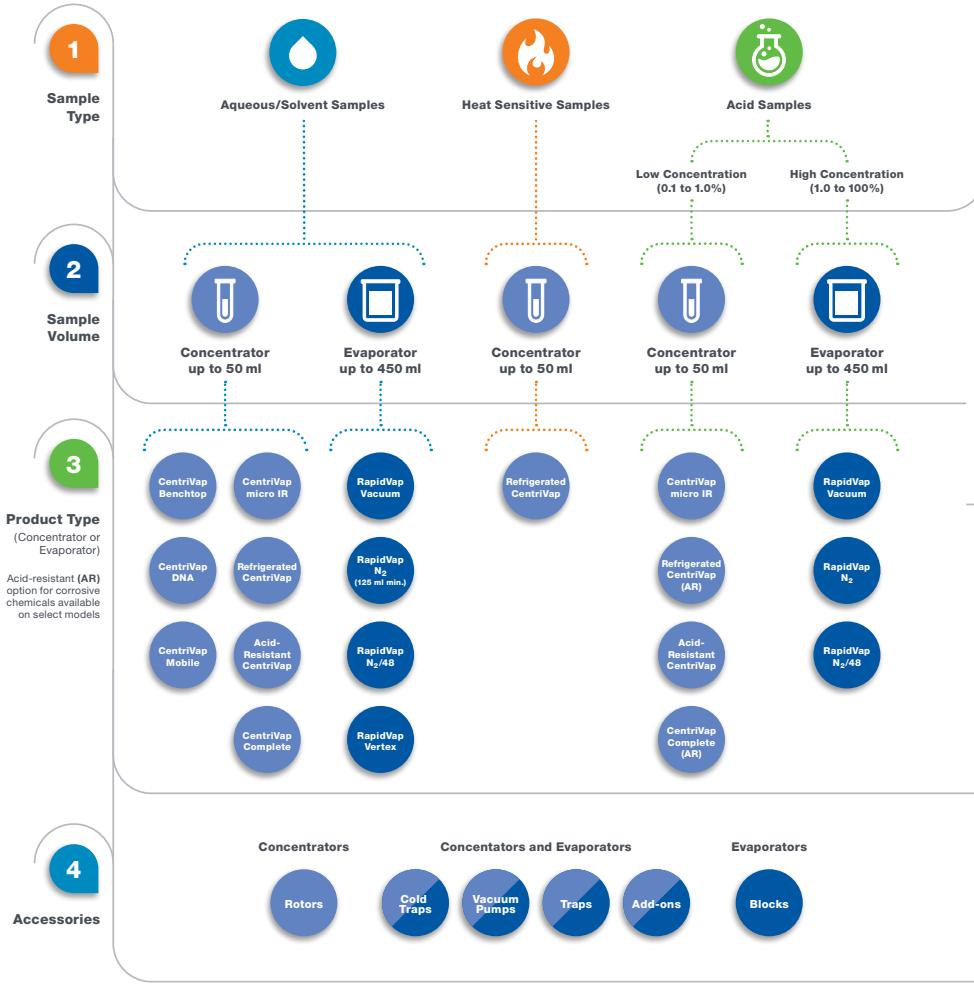
-84° C, -105° C

Scroll Pump

-84° C, -105° C

Concentrators and Evaporators

Steps to Selection and Labconco Product Lines



CentriVap Centrifugal Vacuum Concentrators


micro IR


Benchtop


DNA


Acid-Resistant


Refrigerated
AR


Complete
AR


Mobile

RapidVap Dry Evaporation Systems


Vertex


Vacuum


N_2
125 ml min.


N_2/48

Accessories


Rotors


Cold Traps


Vacuum Pumps


Traps


Add-ons


Blocks

Accessories that make freeze drying easier

What is the difference between the perfect freeze dryer for one 900 ml sample, and the perfect freeze dryer for nine hundred 1.0 ml samples?

The answer is in the drying accessories. Both freeze dryers in this example will accumulate the same amount of ice. The samples, however, are vastly different sizes. Therefore they require different accessories to accommodate them.

The base unit of a freeze dryer is clear: a vacuum chamber with a cold collector that reaches a certain temperature and holds a specific amount of ice. Drying accessories are added to the base. These accessories determine what types and sizes of samples can be lyophilized on the freeze dryer.

The correct drying accessory will ensure the most effective and efficient lyophilization of your sample.

Every freeze dryer has its own unique accessories as well as the standard **manifolds** and **drying chambers**. However, each manufacturer designs their freeze dryers to suit their own accessories so be sure to check compatibility before trying to mix and match brands.

Labconco offers more than 30 FreeZone® drying accessories that optimize the lyophilization process for a wide variety of sample types.

Accessory options from Labconco include the following, though common accessories like manifolds and chambers are available from many suppliers.

FreeZone Cart
Provides easy mobility of a complete freeze dry system

Ampule Pods
Allow up to 15 ampules to be lyophilized simultaneously on the same port

Manifolds
Ranging from four to 28 ports, for attaching flasks, Ampule Pods or a Mini Stoppering Chamber

Clear Chambers and Clear Chambers with Valves
Provide visibility of samples during lyophilization

Slant Freeze Flask Holder
Holds flasks at an angle during pre-freezing

Various Product Shelves
Hold a variety of sample sizes during pre-freezing and lyophilization

Test Tube Racks
Hold up to 104 tubes, and can accommodate microcentrifuge tubes

Amber Fast-Freeze Flasks
Protect light-sensitive samples

Microwell Plate Holder
For temperature stability on very small samples to help avoid sample melt back

Need to know: CentriVap Centrifugal Concentrators

Labconco offers a number of concentrators in our CentriVap® line but it can be difficult to determine the best model for each application. The following should provide you the basic information to help a customer select the CentriVap that's right for them.

How are CentriVap Concentrators different than centrifuges?

Both concentrators and centrifuges spin samples that are commonly placed in tubes or microplates. Centrifuges spin samples at variable rates creating G-forces that separate samples according to density. Concentrators spin samples at a fixed, low speed that creates just enough force to keep the liquid samples in tubes or microplates so a vacuum can be applied to liquids without losing any samples.

micro IR

Benchtop

DNA

What types of labs use CentriVaps?

CentriVap Concentrators are commonly used for biological samples. Any lab needing to evaporate biological samples could possibly use a CentriVap Concentrator. Biological samples are heat sensitive.

A vacuum atmosphere in the concentrator lowers the pressure level so that evaporation will occur at low temperatures and the samples will not be damaged from heat.

Does every CentriVap need a vacuum pump?

A vacuum pump is required to centrifugally concentrate. The CentriVap DNA, Complete and Micro IR models include a built-in chemical-resistant diaphragm vacuum pump and a rotor. All other CentriVaps require the purchase of a chemical-resistant diaphragm vacuum pump.

Acid-Resistant

Refrigerated

Complete

Mobile

Cold Traps are helpful when the lab does not have a fume hood to safely exhaust the evaporated solvents

When do you need a Cold Trap with a Concentrator?

Most samples do not require a Cold Trap in order to evaporate. Cold Traps are needed to create a closed system, meaning all of the solvents that are being evaporated are collected in the Cold Trap. This is helpful if the lab does not have a fume hood to safely exhaust the evaporated solvents. If the lab would like to lyophilize in addition to concentration then a cold trap is needed as well as a vacuum pump that can produce a sufficient, deep vacuum.

Is there an easy way to determine which CentriVap is best for my application?

To ensure your customer is getting the best CentriVap for their needs, just use the **Lab Equipment Selector** on the Labconco website or contact your Labconco Sales Representative. For more information on the CentriVap line or specific product questions, our Application Specialists are available at (800) 821-5525.

Trapping valve for solvent recovery

Many of you know the importance of having a solvent recovery option with a **vacuum evaporator**.

Evaporators with solvent recovery are often referred to as “closed systems,” meaning solvents are not escaping in the process. **Cold traps** are used to condense and collect solvents from evaporators.

When collecting evaporated solvents, it becomes harder to recover the solvent as the sample volume grows and the evaporation rate quickens. Because of these factors, solvent recovery with a vacuum evaporator has been difficult in the past.

It's important to use a trapping valve to recover solvents for many reasons:

1. Evaporating solvent is valuable and can be reused
2. Evaporating solvent is regulated, so only a limited amount can be dispersed
3. Collecting and properly disposing of the solvent protects the environment
4. Fume hood space is often limited and this allows users to bring their evaporator out of the hood
5. It's important to collect solvents before they reach a vacuum pump with oil

Labconco developed the RapidVap Trapping Valve to aid in solvent recovery using vacuum evaporation. The Trapping Valve increases the vapor dwell time within the Cold Trap and increases solvent recovery rates to >90%.

RapidVap Trapping Valve must be used with a Cold Trap and is compatible with RapidVap Vacuum Systems manufactured after September 2016. To learn more contact our Application Specialists

Top 5 lyophilization mistakes

It's been said that freeze drying is an art, not a science, but there are ways to help improve your artistic capabilities. Here are some of the top mistakes of the freeze dry process.

1. Not knowing your sample's melting point

Without knowing what temperature your sample melts at, you can't choose the correct lyophilizer for your needs, and your samples may melt during the process. A freeze dryer requires a **temperature differential** between the sample's **eutectic temperature** and the freeze dryer collector. The collector must be 20˚ colder than the eutectic temperature to allow for proper sublimation during lyophilization.

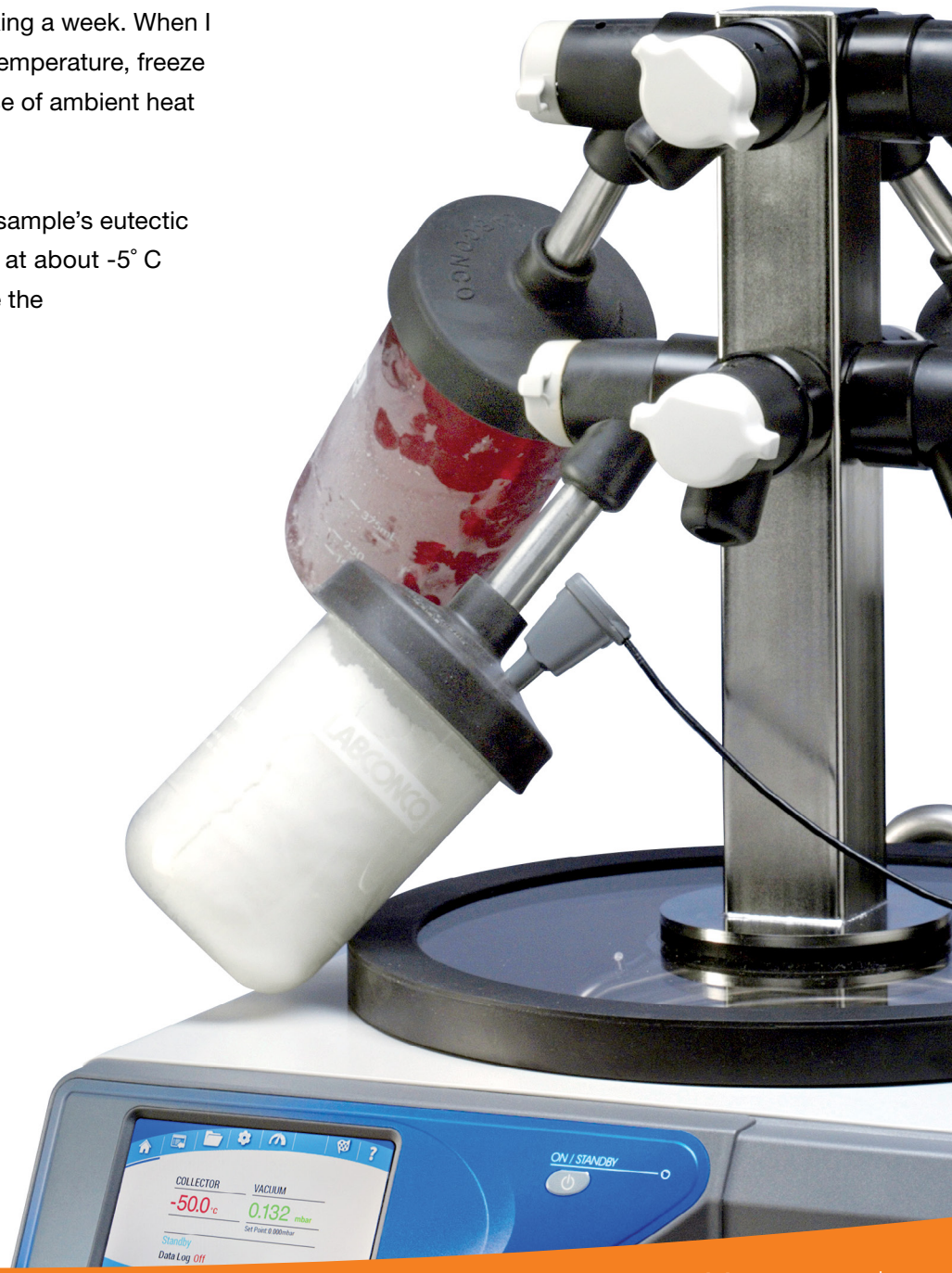
Example: Ethanol has a freezing point of -114˚ C. If used in a freeze dryer, the collector temperature would need to be -134˚ C. Unless you're using a liquid nitrogen freeze dryer, freeze drying a sample in pure ethanol would be impossible. In fact, just freezing pure ethanol is difficult. If you dilute ethanol with water, you can raise the sample's eutectic temperature to a point that it could be freeze dried using a -105˚ C freeze dryer.

2. Thinking colder is better when freeze drying on a shelf-type freeze dryer

I had a customer who thought the freeze dryer wasn't working because on the port-type freeze dryer the samples took 2 days to freeze dry, but in the tray dryer, they were taking a week. When I asked what the shelves were set at, I was told -40˚ C. Without an appropriate temperature, freeze drying is going to take a long time. The ports freeze dried more quickly because of ambient heat from the room.

During primary drying, you want to set the shelf temperature to just below the sample's eutectic temperature. With a water sample, this would mean the shelves would be set at about -5˚ C since water freezes at 0˚ C. Allow just enough heat to the shelves to encourage the moleculesof the sample to move, yet prevent melting.

DID YOU KNOW?
A freeze dryer requires a temperature differential between the sample's eutectic temperature and the freeze dryer collector.



3. Using the wrong equipment for your samples

Many freeze dryers are used in a group setting, so before purchasing one, make sure each person using it knows:

- Total sample volume per run
- What components make up the sample and the samples' eutectic temperature
- How to properly use the freeze dryer

If you need to process 10 liters of sample, a 2.5 liter freeze dryer is not the best option; an 18 liter would be recommended. Make sure you get the right **temperature collector** to prevent sample vapors from bypassing the collector and contaminating your vacuum pump oil.
If the unit is not used correctly, it could ruin everyone's samples. The **Laboratory Guide to Freeze Drying** can help.

4. Not maintaining the vacuum pump

Although it seems like a small piece of the freeze dry puzzle, the pump needs to be in optimal working order for freeze drying to work. There are a few things to remember about the vacuum pump. Running the pump with the gas ballast open 30 minutes before and after the freeze dry process will elongate the life of the pump. Opening the **gas ballast** purges contaminants out of the pump to prevent damage to internal components.

Check the pump oil often for discoloration and particles, and change the oil as needed. Doing regular oil changes keeps the pump pulling at optimum vacuum during the freeze dry process.

5. Having the wrong freeze drying accessories for your process

Do you need to stopper samples under vacuum? If so, a **stoppering chamber** is required. Are you freeze drying in flasks? Then a **drying chamber** with ports is needed. If you are doing solvents or acids, a **hybrid vacuum pump** is recommended.

By avoiding these mistakes, you can give your freeze dryer and pump a long life, and you'll have a masterpiece sample when your freeze drying is done.

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labconco.com/sampleprep