Solvent Recovery Practices and Tips to Avoid Rotary Evaporation Mistakes

Solvent recovery is the process of extracting target products from waste or by-product solvents generated during the manufacturing process. These chemicals can then be repurposed, reducing the need to produce or purchase new solvents, while eliminating a large amount of waste and removing potentially hazardous substances from the primary processed material.

Solvent recovery can be done by several methods including fractionation, azeotropic distillation, and rotary evaporation.

The cannabis market uses three main extraction techniques: CO2, liquefied hydrocarbons, and ethanol extraction. In all processes, the plant material is subjected to a solvent to remove active compounds from the plant matter and filtered to yield a solution of the solvent with plant extracts.

All extraction methodologies yield an oil once the solvent has been removed. This oil contains plant lipids, possibly chlorophyll, waxes, fats, terpenes, and other cannabinoids.

The resulting ethanol solution is concentrated via vacuum distillation. The solvent-free, purified extract can be dried on trays in a vacuum oven to yield a yellow to amber extract.

Solvents Overview

The term 'solvent' refers to several chemical substances which are used to dissolve or dilute other substances or materials. They are usually organic liquids.

Solvents usually have a low boiling point and evaporate easily or can be removed by distillation, thereby leaving the dissolved substance behind. Solvents should therefore not react chemically with the dissolved compounds—they must be inert.

Solvent recovery methods are used to separate and recover solvent from other liquids. Removal efficiency can be very high using this process and can be used for solvent mixtures as well as single solvents.

Solvent recovery is best suited for applications dealing with expensive or easily recoverable compounds. Recovery is economical if the recovered compounds can be reused in a process.

Common Examples of Solvents in Industrial Manufacturing and Processing

- Aliphatic hydrocarbons | pentane, hexane, and heptane
- Alicyclic hydrocarbons | cyclohexane and methylcyclohexane
- Aromatic hydrocarbons | benzene, toluene, xylene, ethylbenzene and styrene
- Halogenated hydrocarbons | methylene chloride, chloroform, trichlorethylene, carbon tetrachloride, dichloroethane, freons and trichloroethane
- Alcohols | methanol, ethanol, isopropanol and butanol
- Glycols | ethylene glycol and diethylene glycol
- Ethers | methoxy ethanol and butoxy ethanol
- Esters | methyl acetate, ethyl acetate, isopropyl acetate and methyl methacrylate
- Ketones | acetone, butanone, hexanone and cyclohexanone

Currently, the most popular solvents for cannabis extractions are butane, propane, and ethanol. These hydrocarbon solvents are chosen over other solvents because they are economically superior for producing high-quality extracts at an accelerated rate, safely.

The most sustainable and economical solution for organic solvent recovery is distillation. The raw material is subjected to vacuum to reduce its boiling temperature and the heat required in the distillation equipment. Depending on the level of contaminants in the initial raw material, the temperature and vacuum pressure can be adjusted to efficiently achieve recovery of the solvent.

What is Solvent Recovery?

Solvent Recovery is a form of distillation and based on the principle that the vapor of a boiling mixture will be richer in low boiling point components such as acetones, ketones, aliphatic hydrocarbons and alcohols.

Chemical solvents, especially in the commercial cannabis industry, are expensive. The high cost of solvents makes solvent recovery an ideal way to save money. Not only is solvent a major processing expense, but resources must also be devoted to proper handling, storage, and disposal of any waste if the solvent is not used efficiently. Solvents such as hexane, ethanol, and isopropyl alcohol that are used in cannabis extraction processes can be recovered for recycling and later use.

To extract specific compounds from cannabis biomass, a solvent is used to help separate and isolate the desired substance. Once the oil has been isolated from plant materials into a crude form, the solvent must be removed to further refine and purify the oil. The solvent must be cleaned and collected to allow for reuse.

After use, the solvents are recovered and separated through distillation. Special distillery tanks are used to heat and separate the solvents when they reach boiling and condense into vapor. The vapor is collected and funneled to a condenser. Upon cooling, the solvents again turn into a pure liquid which can be reused.

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CO2 Extraction

The distillate can be isolated easily by reducing the pressure which evaporates the CO2, leaving a cannabis extract with no solvent. Tweaking the temperature and pressure affords CO2 systems the ability to yield extracts with a complete terpene profile.

Sophisticated extraction apparatus can also incorporate fractionation, which enables process tuning to isolate desired components. Refrigerated chillers that are integrated into these systems facilitate recycling of the CO2 by condensing the gas back to a liquid state.

Liquefied Hydrocarbons

Systems pressurize butane, propane, or other low molecular weight hydrocarbons to a liquid state. The liquid hydrocarbon passes through a bed of plant material and filter, yielding an extract solution of hydrocarbon and plant extract. Like the CO2 method, a reduction in pressure evaporates the hydrocarbon liquid, yielding a solvent-free plant extract.

This method requires great attention to safety due to the flammability of the hydrocarbon used. Maintaining the pressurized hydrocarbon in the liquid state requires low temperatures. Butane has a low boiling point and is the most common hydrocarbon solvent currently used in extraction. It is favored for its non-polarity, which allows the extractor to capture the desired cannabinoids and terpenes without co-extracting undesirables including chlorophyll and plant metabolites.

Ethanol Extraction

Systems use food grade or USP grade ethanol as a solvent to extract plant material. Ethanol is the second most important solvent (after water) in consumer products. This method varies from vessels to reactors, filter reactors, spinning vessels to barrels.

Ethanol is a polarized molecule. A polar solvent will readily mix with water and dissolve water soluble molecules. Chlorophyll is one of those compounds which will easily co-extract when using ethanol as a solvent. When a concentrate retains chlorophyll, it introduces a dark coloration and an undesirable bitter, grassy flavor. Activated carbon filters may be used to eliminate impurities.

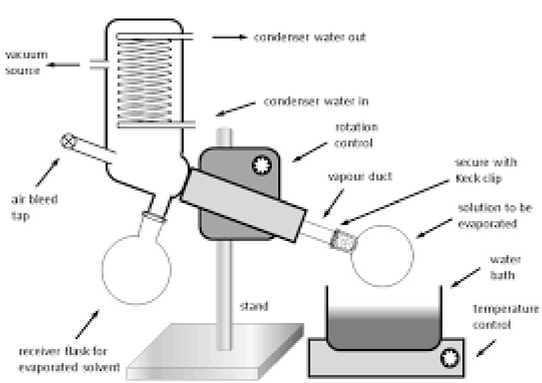
Ethanol extraction can be carried out by several methods including cold, warm, or room-temperature processing. Some ethanol extraction processes involve the use of a rotary evaporator setup to remove the ethanol from the extract solution.

Ethanol extraction is popular for several reasons:

- Can extract a full spectrum of compounds from cannabis plants
- The process can be adjusted to prevent certain compounds from solubilizing
- Generally, equipment costs for ethanol extraction are lower compared to other methods
- Ethanol is the least toxic of the alcohols
- Ethanol can be recovered and recycled
- The resulting ethanol solution is concentrated via vacuum distillation (falling film, reactor, or rotary evaporator)
- The solvent-free, purified extract can be dried on trays in a vacuum oven to yield a yellow to amber extract.

Rotary Evaporation

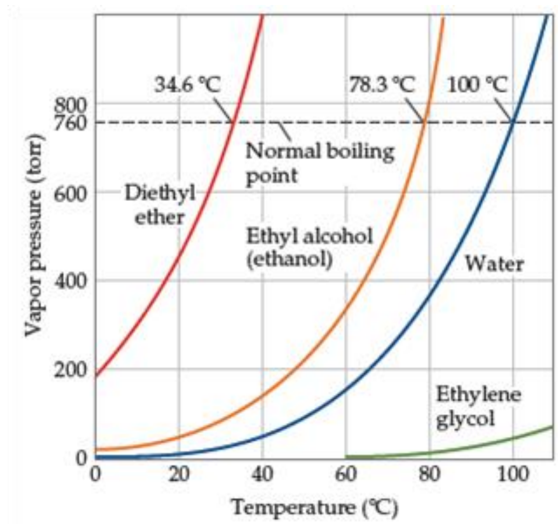
The rotary evaporator has been a relied upon scientific tool for solvent removal for many decades. They are found in almost every organic laboratory since they perform solvent recovery quickly. The rotary evaporator enables the removal of solvent in a controlled manner under vacuum. Sizes range from bench top (to 5 L flasks), pilot scale (20 L) on up to 50 to 100 L. The pressure is reduced in the rotary evaporator by combination of a vacuum pump and a vacuum controller to lower the boiling point of the solvent to be removed, specifically ethanol in the case of cannabis extract processing.



Typically, the distilling flask is filled to 50% volume. The water bath is heated to 30-40°C. The condenser temperature, controlled by a recirculating chiller, is set to -10°C to 0°C. Once the water bath and condenser have reached the set points, the distillation flask is rotated from 150-200 rpm.

This creates a thin film on the upper surface of the round glass flask, which increases the solution surface area and enhances the solvent evaporation rate. Applying an appropriate vacuum to the system lowers the boiling point.

In the lab, the house vacuum line or vacuum pump is used as source for the vacuum (40-50 torr). The application of vacuum means that the boiling points of the solvents are going to be significantly lower than at ambient pressure.



Here is a safety educational resource for proper rotary evaporator operation and includes an mbar boiling point table for your reference.

The 20/40/60 rule offers a general guideline for rotovap operating conditions. Under this rule, the bath should be set to around 20 degrees higher than your desired vapor temperature.

In turn, the condenser temperature should be set at around 20 degrees lower than the desired vapor temperature.

From a vacuum control perspective to keep your system tightly controlled within this range, we recommend piezoresistive vacuum technology. DigiVac uses Piezo sensors that are isolated and hermetically sealed stainless steel in our VPC, Concerto, and Bullseye Piezo products.

Examples of solvents used in the lab setting and their boiling points at 40 Torr*

Solvent	Boiling point (760 Torr/ATM)	Boiling Point (40 Torr)
acetonitrile	81.8 °C	7.7 °C
diethyl ether	34.6 °C	-27.7°C
ethanol	78.4 °C	19 °C
ethyl acetate	77.1 °C	9.1 °C
hexane	68.7 °C	-2.3 °C
heptane	98.4 °C	22.3 °C
methanol	64.7 °C	5.0 °C
water	100 °C	34.0 °C

Common Applications for Rotary Evaporators are:

- Medical or pharmaceutical

- Scientific Research and Development
- Chemical engineering and Laboratory
- Biological and botanical products processing
- Evaporation, crystallization, drying, separation and solvent recovery processes are necessary for any of the industries that handle reaction solvents

Before we get into some product recommendations, let us clear up some misconceptions in the market regarding vacuum.

1. I need as deep a vacuum as possible. False!

- Too deep a vacuum can result in the solvent having a boiling point that is too low
- You could end up with uncontrollable boiling and possibly bumping or foaming
- The product will escape the evaporation flask resulting in system contamination, possible product loses and having to restart your run…. stay tuned for a way that you can quickly and easily stop bumping in your system with Concerto.

2. I need to run my pump at full throttle. False!

- If the vacuum pressure is too high, the solvent will not have time to condense into the flask
- Which can lead to your vacuum pump being flooded with solvent and possibly damaging the pump, the use of a vacuum controller/regulator will help solve this
- This leads to poor solvent recovery since you cannot recover solvent from inside a pump
- Finally, running a vacuum pump above its base pressure for extended periods of time is not good for its longevity.

It is a good thing to make sure you have a precise vacuum controller that can help you avoid these problems. A precision vacuum controller can help you optimize your process as well as preserve the life of your vacuum pump by providing some isolation between your system and your vacuum pump as well as keeping it running happily nearer to its base pressure.

VACUUM CONTROLLER OPTIONS

DigiVac offers the Vapor Pressure Controller | VPC for Rotary Evaporation applications. The VPC provides innovative yet simple vacuum control for

rotary evaporator distillation and isolation of plant oils.
It delivers precise control of target vapor pressure and is designed to tolerate harsh chemicals.

DigiVac has [the SNAP Controller | Simple, Nimble, Automatic, Process](#) controls the vacuum chamber or equalizes the system, automatically venting or isolating the system with the touch of a button. It treats material in the chamber more gently by implementing the controlled ramp rate, ideal for freeze drying, vacuum drying or lyophilization applications.
The single chamber controller can set up to 8 recipes based on time and pressure setpoints.

CONCERTO Multi-Chamber Controller By Across International and DigiVac

Vacuum & Pressure Controller that can precisely control Multiple Rotary Evaporators at once (Up to 4) with only one vacuum pump.

- Simplify your lab & control more with Less
- Maximize throughput with a 17.5 mm vacuum path to each hose barb
- **Control Range:** 2 Torr to 760 Torr

- **Unique Vacuum Control:** proprietary dual valve designed by DigiVac offers proportional throttle and proportional bleed control (vent to ATM).
- Each of the four channels have a dedicated valve
  - The integrated bleed design allows you to quickly vent to atmosphere to stop bumping during processing
- **Recipes:** Program ramps and holds vacuum at different a duration and vacuum level
- Numerical readings right on the LCD screen and one touch start, stop and release buttons

Silver Innovation Award Winner for Instrumentation at PittCon 2020!

The rotary evaporator controller is particularly useful when you are looking to maintain pressure at a certain level as needed for botanical processing and rotary evaporation. Mounting is bench top.

See overview video here

See how to select recipes here

See how to customize recipes here

See how to easily create set-points here

Bullseye Piezo Rough Vacuum Gauge with Isolated 775i Sensor | UL, CSA, CE Certified

The Bullseye Precision Gauge Piezo-775i provides highly accurate and reliable vacuum readouts in the rough vacuum range 1 to 775 Torr and 11 additional measurement units.

- Visual Graphing: Patented digital graphing capabilities (Pump-down graph, Autoscaling line graph, and bar graph)
- Isolated Sensors: For use in dirty environments or where corrosive gases are in use
- Certifications: CE, CSA, UL, and RoHS
- Measures 1 – 775 Torr (+/- 2 Torr) without a capacitance manometer
- And it is available in a Bluetooth version as well to allow for remote monitoring from your phone or tablet.

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Contact Us by phone or email and one of our engineers can work with you to customize the best set-up for your application.

About DigiVac

We have been designing and manufacturing scientific measurement and control instruments since 1983. Proud to be Made in America.

Our Vision: To pioneer measurement and control innovations that simplifies science for the benefit of humanity.

Our Mission: Engineer, manufacture and deliver reliable measurement and control solutions for the research, laboratory and processing communities.

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