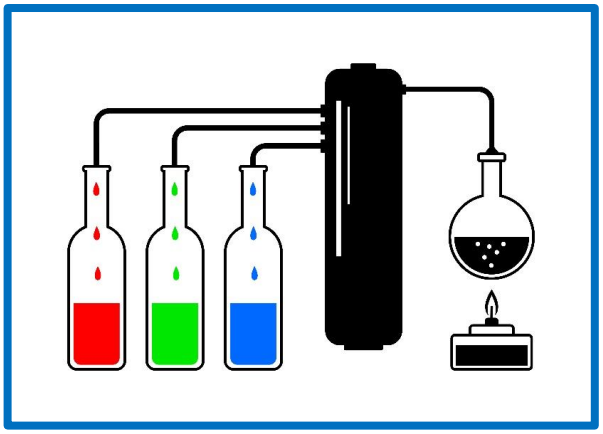
BENEFITS OF VACUUM DISTILLATION IN BOTANICAL PROCESSING

Abstract: One of the *primary purposes of using vacuum in distillations is to lower the temperature required to achieve a boiling point*. The target product in a distillation could either be the remaining product, the distilled product, or a purified product. The vacuum associated with a distillation depends on the product to be distilled.



Volatile substances are likely to undergo vacuum distillations above 1 Torr.

Composite distillations typically require precise pressure control in the region above 1 Torr.

This paper focuses on the benefits of vacuum distillation, how rotary evaporation differs, and details vacuum instrumentation with proven results in botanical processing.

Introduction:

What is distillation? It is the process of driving gas or vapor from liquids or solids by heating and condensing to liquid products. It is used especially for purification, fractionation, or the formation of new substances.

Methods for vacuum distillations are taught at the college level*, and there are a variety of apparatuses developed for distillations. These include Schlenk lines, fractional distillation, packed distillation, Perkin triangle distillation and rotary evaporation to name a few.

A common practice for volatile substances is fractional distillation. Volatility refers to the tendency of a substance to pass from the liquid phase into the gas phase. In this process, a vapor from a heated liquid mixture is condensed as it rises through a vertical column.

This column includes a series of theoretical plates which help distill the substance over and over as it passes up the condensing column. The most volatile fraction of the mixture emerges from the top of the column, while less volatile fractions

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are withdrawn at lower points. In petroleum refining, this method is very efficient for removing naphtha, kerosene, and gas oils from crude oil. †

In addition, fractional distillation is a method used when processing botanicals like essential oils, cannabinoids, CBD and hemp. The botanical oil is evaporated in the fractionating column and its vapors are made to condense at different temperatures (cold traps may be used in this process).

Rectification is the process of increasing purity: If the distillate obtained during distillation is processed again, purity increases. The concentration of volatile components in the distillate decreases on each pass. THC and CBD fractions can be isolated. Repetition of this process enables highly pure concentrates, devoid of residual solvents, and other contaminants.

Difference between Rotary evaporation and Short Path Distillation: Rotary evaporator is ideal for removing solvents. Solutions in solvents can be processed through a rotary evaporator and evaporate the solvent. If you want to purify a solvent or other chemical which is liquid at normal conditions, you use distillation. Short Path Distillation (SPD) or its cousin Wiped Film Distillation provides a cannabinoid potency of up to 99%.
In rotary evaporation, you do not care what happens with the vapors of the solvent, but distillation setup is specifically built for collecting vapors, and even separating vapors of different compounds.

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.

Common Applications for Rotary Evaporators Are: medical or pharmaceutical, chemical engineering, biological and botanical products processing, and for the purpose of scientific research and development. Evaporation, crystallization, drying, separation and solvent recovery process are necessary for any of the industries that handle reaction solvents.

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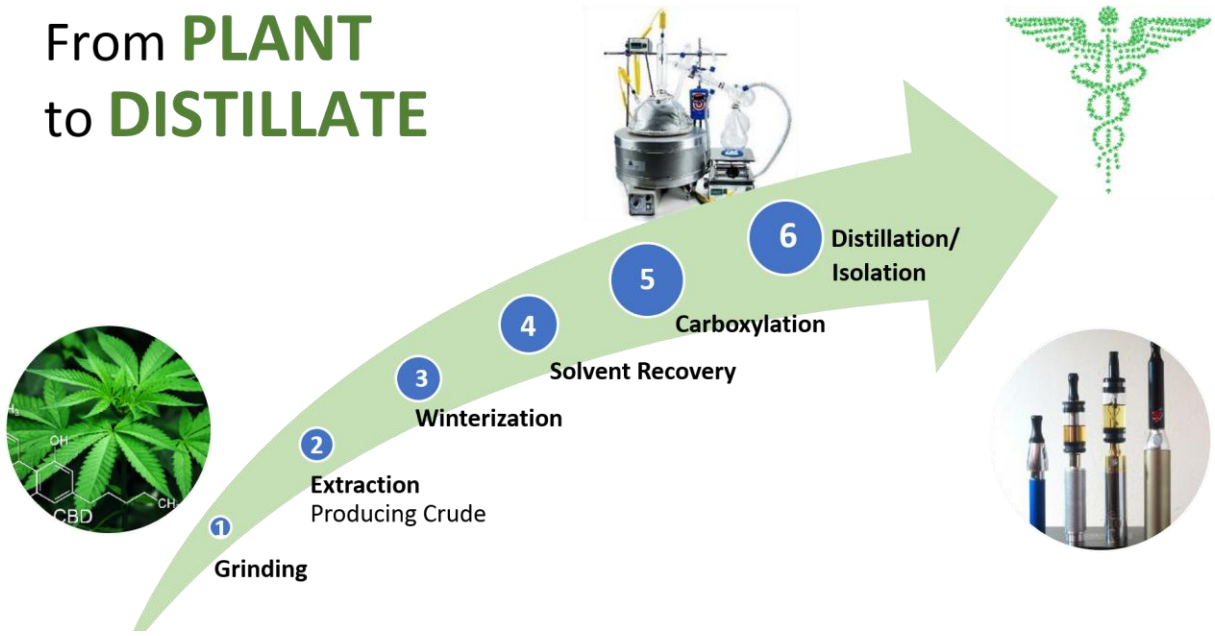
Vacuum distillations in high pressure/low vacuum and medium vacuum regions:

Vacuum distillation is a method of *distillation* whereby the pressure above the liquid mixture to be distilled is reduced to less than its *vapor pressure* (usually less than *atmospheric pressure*) causing evaporation of the most *volatile* liquid(s) (those with the lowest *boiling points*). The atmospheric pressure in the distillation tank is reduced making it possible to boil the liquid at a lower temperature.

Compounds with a boiling point of greater than 150°C will likely need vacuum distillation.

Note: pressure at atmosphere is 760 Torr, however atmospheric pressure could be different depending on your location since it drops as altitude increases. Atmospheric pressure is the force exerted on a surface by the air above it as gravity pulls it to Earth.

Distillation relies on the basic principle that boiling occurs when vapor pressure exceeds ambient pressure for a liquid. The pressure above the liquid being distilled is reduced via vacuum to below its vapor pressure, causing it to boil and rise through the column. This may or may not require heating.



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Three benefits of Vacuum Distillation:

1. Faster processing time
2. Effective distillation while keeping the process under the maximum temperature of the distillation unit's heater
3. Effective processing of higher boiling point solvents without igniting them or causing thermal breakdown (maintaining the integrity of the distillate)

Vacuum distillation is sometimes called *low temperature distillation*.
The target product in a distillation could either be the remaining product, the distilled product or a purified product.

The vacuum pressure associated with a distillation depends on the product to be distilled. For example, volatile substances like those used in oil refineries are likely to undergo vacuum distillations at above 1 Torr, perhaps 20-50 mmHg.

Note: 1 millitorr = 1 micron = .001 Torr

Organic distillations may happen at 10 microns, 100 microns or 400 microns, where the rule of thumb is that the more volatile the solvent, the higher the residual pressure. For example, the residual pressure in a Schlenk line distilling an aodic organic solvent might be about 10 microns. Ideal gauges for monitoring these distillations that occur at less than 1 Torr, are the DigiVac Bullseye Precision Gauge® with Bluetooth and for process control StrataVac Touch or StrataVac vacuum system monitoring and control.

* http://www.chem.umass.edu/~samal/269/distill.pdf † http://science.howstuffworks.com/environmental/energy/oil-refining4.htm

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Understanding the product design:

DigiVac gauges that have applications in the medium vacuum range operate on the principle of indirect pressure measurement where it senses the cooling of the surrounding gases. The less gas (the lower the pressure) the hotter the thermocouple gets and vice versa; this temperature is correlated to a pressure for air, nitrogen, and other similar gases.

High Pressure/Low Vacuum (Above 1 Torr to ATM)-Volatile Organic Compounds found in oil refineries

- Vacuum Regulator | Model 450
- VPC | Vacuum Pressure Controller
- StrataVac with 775i sensor
- Bullseye Precision Gauge Piezo

Medium Vacuum (Below 1 Torr and above .5 milliTorr)-organic compounds like solvents and aodic molecules utilized in the life sciences industry for biomedical or biological research, and botanical processing

- Bullseye Precision Gauge®
- StrataVac with Thermocouple
- Model 200
- Model 215V
- Model 801W

Product performance:

Vacuum analysis you can trust: DigiVac gauges are manufactured with thermocouple passive sensor technology which has proven reliability in field operations. Every DigiVac instrument is manufactured in the USA.

Tight controls: manufacturing process control driven by quality reviews and improvements.

Repeatability: The process of calibrating everything under real vacuum against a NIST standard insures repeatability and superior instrument accuracy.

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DigiVac
Scientific Measurement & Control

Over 30 years of Innovative Scientific Measurement and Control Designs

Product Information:

Vacuum Measurement and Monitoring: The **Bullseye Precision Gauge with Bluetooth** has patented vacuum graphing and has become the standard for measuring SPD processing. Combine with the **Vacuum Gauge App** to enable remote monitoring. Set low and high alarms plus email data, and graphs for process improvement.

Vapor Pressure Controller | 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.

StrataVac Touch is a premium vacuum controller with sweep gas capabilities. Comes programmed to deploy Sweep Gas control which increases flow (liters/per hour) at a lower temperature. Easily see a snapshot of up to 3 sensors and up to 2 valves (any configuration of valves and sensors up to 5) working on your distillation system with easy touchscreen functionality. Take your vacuum control and SPD or Wiped Film system monitoring and control to the next level with the StrataVac Touch.

StrataVac is a vacuum monitor and controller that is often installed in distillation systems including SPD and Wiped Film. The StrataVac can show multiple vacuum levels and deliver precise vacuum control. Simultaneously shows condenser, rough pump and diffusion pump vacuum pressures, which is critical for understanding and improving your process. This StrataVac model can be configured to drive up to three sensors and 2 valves. We offer bundles that can deliver either **Throttle Control** or **Bleed Vacuum Control**.

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