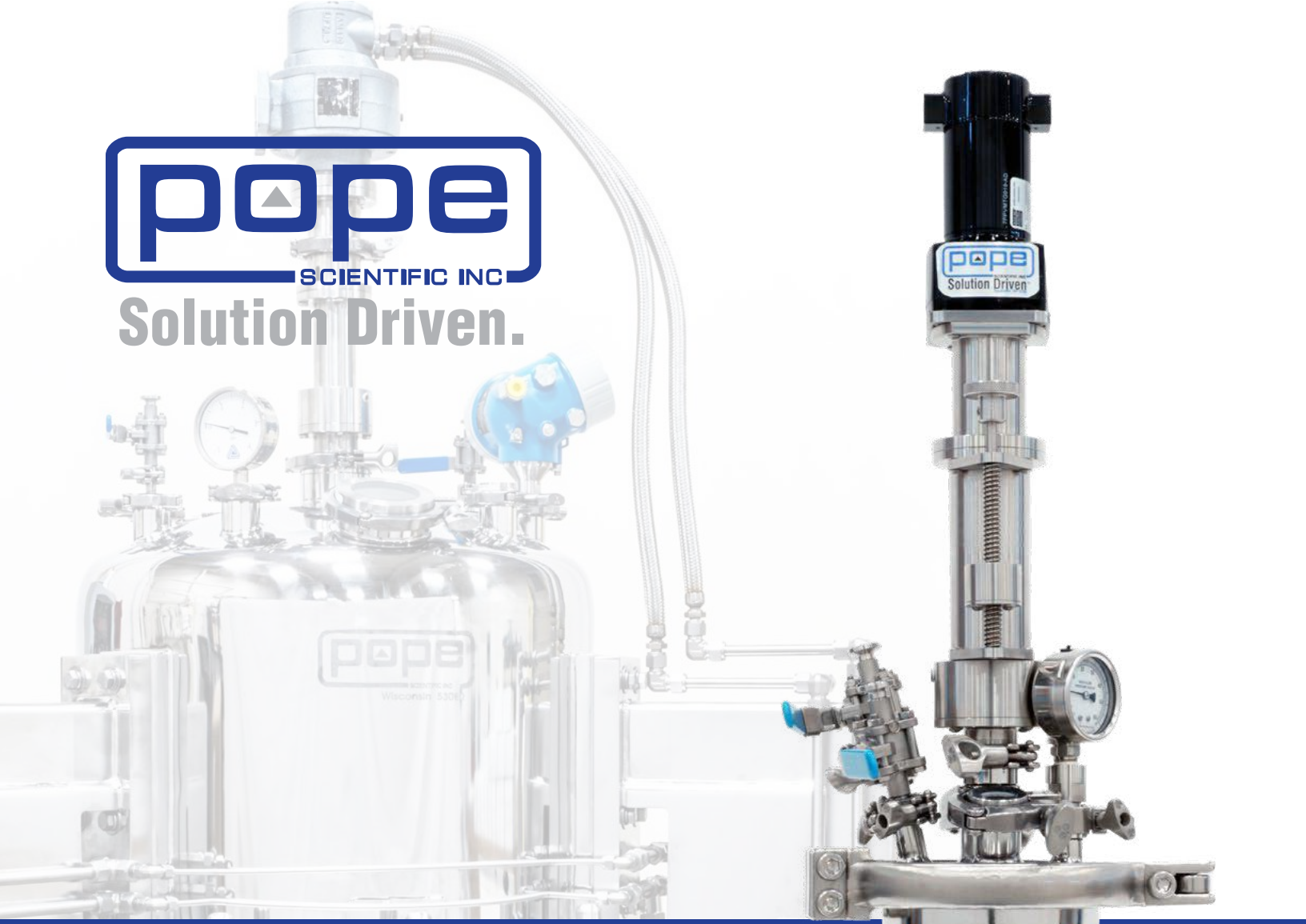
pope SCIENTIFIC INC

Solution Driven.

Nutsche Filter Dryers

Ideal For Processing Ultra-High Purity Substances



V1.0 - 2022

Nutsche Filter-Dryer Overview

A Nutsche Filter-Dryer is a specialized version of a pressure vessel, (normally stainless steel or other alloy), designed for separation of liquids and solids from a mixture of both, known as a slurry. Each Nutsche has a full diameter filter disc positioned at the vessel bottom to collect solid particles on top and allows liquid to pass through below. In most applications, the collected solids are the product, often being highly pure crystals from a reaction and precipitation operation, remaining suspended by agitation as a solvent slurry. Capabilities include solids filtering, washing, re-slurrying and drying in a single vessel, reducing process time and worker and environmental exposure. Pressure capability facilitates filtering rate and vacuum capability facilitates filter cake drying rate which can be additionally aided by heat and cake agitation. (Nutsches wth agitators, or Agitated Nutsche Filter Dryers, also known as ANFDs are common.)

Nutsche processing is a critical operation because it is often the final purification step in an overall manufacturing process, prior to packaging. Processed end-products include pharmaceuticals, bio-materials, electronics grade materials, fine specialty chemicals, and many others. Pope Nutsche filter-dryers are designed and built accordingly, with the highest quality materials, welding, components and surface finishes following ASME BPE guidelines, and are known, respected and utilized worldwide.

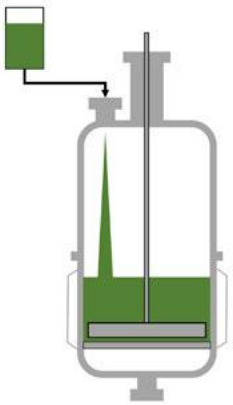
Did you know?

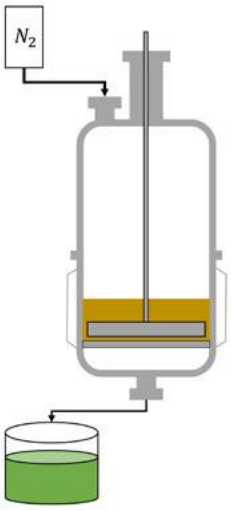
Nutsche is usually pronounced in English like “nooch”, (with oo being the ü sound), rhyming with “mooch”.
\ □nüch- \
variants: or nutsch
plural -es
Definition of nutsche filter:
a filter of a simple type adapted to batch operation
especially : such a filter operated by suction

NUTSCHE OPERATION PROCESS

A typical operating sequence involves transporting a slurry from a completed crystallization step into a Nutsche via either pumping, reactor pressurization, gravity, or simply pouring. (Figure 1.) All Nutsche ports are then closed except for a bottom drain which is connected to a liquid holding vessel, (Figure 2.) and a top port connected to a pressurized air or nitrogen source. The pressure, which is carefully controlled by a regulator acts as a driving force to push liquid past the solids. The solids begin to form a growing layer called a filter cake on top of the filter screen and then go through the filter plate, out the bottom drain and into the holding vessel. The pressure used is normally 1 - 4 bar, resulting in a faster liquid discharge rate than is possible via a vacuum alone, pulling from bottom (as is per-ormed with Buchner funnels, where the maximum force equates to 1 bar or less.) Eventually, all free liquid will be pushed out, resulting in a “wet” filter cake, i.e., solids saturated with solvent.

1. Filling





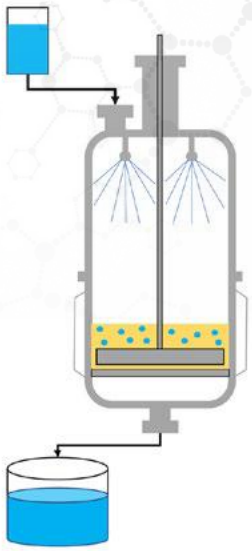
2 . Filtration

Nutsche Filter-Dryer Overview

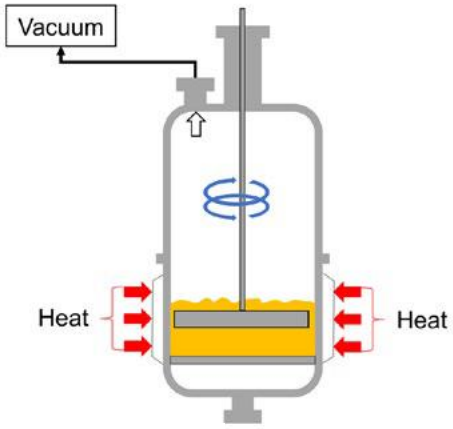
The next step is to wash the solids (Figure 3), which besides being wet, are contaminated with some amount of other chemical species from the preceding reaction. This is accomplished by pouring or spraying in fresh solvent from the top of the Nutsche to a level above the filter cake, mixing it into a slurry again, then once again, forcing the free filtrate out of the vessel. This can be repeated as necessary.

In most cases, the desired product is solids in dry form. This is accomplished within the same Nutsche vessel, without the need to move the wet solids to another processing device. Once again, all Nutsche ports are closed, except for a top port to which a vacuum line is connected. The vacuum condition will start to evaporate the saturating solvent from the wet cake. Usually, this is aided via introducing heat via a circulation heater connected to a liquid jacket outside of the vessel walls. (Supplementary heating means can include a controlled passage of heated air or nitrogen through the Nutsche.) Depending on the nature of the solids, lumps may form within the cake similar to dried mud which can significantly hinder the overall drying rate and duration. This may be addressed by use of an optional cake agitator, (Figure 4) a mixer of special design with a full diameter, angled impeller blade which can be raised and lowered while rotating, churning up and mixing the solids as they dry.

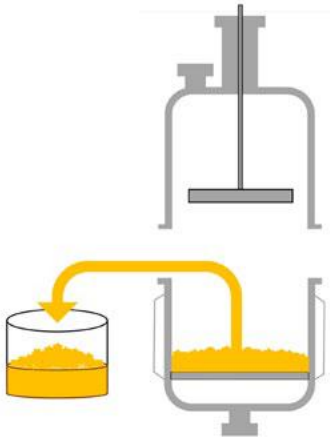
When the solids have reached an acceptable level of dryness, they may be "harvested", by removing them from the Nutsche to another container or directly into final packaging (Figure 5). There are options on how this may be accomplished, covered in the "Harvesting" section. After harvesting, any remaining slurry batch may be be added, otherwise, the Nutsche is cleaned, dried and readied for future processing.



3. Washing



4. Drying



5. Harvesting

Benchtop Nutsche Filter-Dryers

POPE SCIENTIFIC'S BENCHTOP NUTSCHE FILTER-DRYER IS AN ALL-IN-ONE SOLUTION

High purity chemicals, pharmaceutical intermediates, specialty solids and crystals can be: Filtered; Washed; Reslurried; Mixed; and Dried in this versatile jacketed, portable lab scale unit— providing chemists and researchers a powerful tool for testing, piloting and small production.

- The efficient operation reduces process time and minimizes worker and environment exposure.
- ASME Code Section VIII Division 1 And CE/PED Stamped For Group 1 Liquids. CRN Is Available Upon Request
- Solvent removal can be aided by pressurizing; drying can be enhanced by vacuum plus optional heating and solids agitation.
- It's the logical leap forward from laboratory Buchner funnels.
- Ideal for Pilot Work in Simulation of Larger Scale Nutsches
- Choice of Agitators—Conventional or Cake Churning
- Immediate Delivery—Various Sizes in Stock/Larger Pilot Production models also available.
- Rental units available.

CUSTOM BENCHTOP NUTSCHES

Also offered in sizes as small as 2" diameter, volumes as small as 0.2 liters. Extent of componentry depends on size and other considerations. Ideal for lab and small scale studies, you can also scale up your benchtop unit to pilot plant and industrial.

Pope Custom Benchtop Nutsches can be designed for various requirements, including such features as custom stands, funnel heads, instrumentation, heating/cooling circulators, alternate alloys and coatings, pumps, or other customization.

Specifying a Nutsche Filter-Dryer

A specialty at Pope is provision of Nutsche systems optimized for customers' requirements and preferences. This is best initiated utilizing the Pope Nutsche Application Questionnaire which goes through all of the points to be considered in the quotation process.

The first item to be addressed is the sizing of the Nutsche vessel. Considerations include the total volume of slurry to be fed to the Nutsche, the mass of solids within the slurry, particle size and behavior of the solids as they accumulate. Other considerations include preferences regarding equipment handling and support, type of harvesting, electrical hazard class, extent of optional extra functionality, instrumentation and control and mixer type. Interior surfaces are mechanically polished with welds flush and smooth then passivated and electro-polished, resulting in 25 Ra or better surface finish. If required, additional polishing is available down to 8 Ra and better. Exterior surface finish can also be specified.

A very important issue is the specification of materials of construction. 316L stainless steel for vessel body, filter and other components, and Viton for elastomers are the defaults. However, many customers may have applications there the processed materials are no compatible with those. Where highly corrosive materials are processed, higher alloys than 316L may be required such as Hastelloy C-22 or C-276 or others. Coatings may also be applied to inner surfaces such as PFA or ECTFE, covering 316L or even the higher alloys. Where solvents are aggressive, alternate elastomers may need to be considered, including Teflon encapsulated silicone or viton O-rings, and in extreme cases, Kalrez or similar O-rings are available.

Harvesting Designs

Harvesting type refers to the means by which the solids (normally dried) of the filter cake will be collected and transferred. Various options are covered below.

SINGLE BREAK DESIGN:

Here, the vessel has a bottom flange at which both the filter is held and the vessel is opened either by lowering the bottom portion or by raising the top portion. There is no upper or middle main flange. It is possible that some small amount of the filter cake can fall to the ground during opening, however, usually there is a top access port through which a tool can be inserted to scrape the solids away from the vessel wall to help prevent this. This is the simplest and least cost harvesting type.

TOP ACCESS DESIGN:

This is similar to the Single Break type but in addition, there is a full diameter top head or lid which can be removed for full access of the filter cake for sampling or harvesting, without the need to open the bottom filter flange. The top connection may be a sanitary flange type, (up to 12" max), or it can be a heavier bolting or C-clamp closing type. All standard Benchtop Nutsche Filter-Dryers are of this design.

Harvesting Designs

MID FLANGE DESIGN:

This design utilizes a flange break point within the vessel side wall, typically positioned someplace between a few inches above the filter flange to the midway point of the vessel wall. This allows for easier close proximity access to the filter cake. There may or may not be an upper top flange incorporated.

ROTATING STAND DESIGN:

The entire Nutsche is held in place by a portable rotating stand with locking stops at various degrees of rotation from vertical, such as 90° to 100°, sometimes to 180°. The typical arrangement utilizes a Top Access flange, with the entire top head and mixer first removed, then the Nutsche rotated to an angle appropriate for the customers' preferred position and the solids are scooped or dropped out into a receiving vessel. Movement can be manual (smaller size units only) or powered electrically or pneumatically.

REMOVABLE "BASKET" DESIGN:

The concept provides for a removable open top filter section nesting into the bottom head of a Mid Flange type Nutsche. Once the flange is opened and the bottom section lowered, the filter basket is removed, and the solids are removed from it separately in any desired location. This is a convenient feature.

SPECIAL ISOLATION DESIGN:

Pope can create custom isolation systems to suit individual customer's processing application needs. In the example below, the Nutsche is fitted with a clear disposable polypropylene shroud, which when the bottom vessel half is lowered, provides a clear, cylindrical, full diameter working zone with a means of handling and transferring material via gloves and sealable transfer tubes built into the sides of the shroud. This method allows for safe harvesting of the solids under fully contained conditions, with no exposure of the operator to hazardous materials nor contamination of those materials from the environment. The inside of the entire system can also be kept blanketed with an inert gas. This system is also CIP equipped and riboflavin tested for washing coverage, is Div. II electrically rated and all support and power lift surfaces are polished stainless steel.

Harvesting Designs

SIDE DISCHARGE DESIGN:

This method allows for harvesting the solids without opening any full diameter flanges to break apart the vessel. Instead, solids are moved out through a side port, and this can be done while keeping the material isolated from the environment. If required, material can also be kept blanketed with an inert gas while being transported from the Nutsche vessel into a closed receiving vessel. The design incorporates a piston contoured to the interior vessel wall, which at drying completion, is pulled back, allowing solids to be pushed out through a cylinder and down through a chute leading to a receiver. The solids are pushed out via rotation of a specially designed agitator blade. In some applications, additional batches of slurry may be processed subsequently, without having to open and clean out the Nutsche.

Means of Lifting

Nutsches and sections of Nutsches larger than the 6” Benchtop Nutsche series or 8” sizes can become quite heavy, making manual movements difficult and unsafe. Thus, where necessary, Pope will incorporate into the scope of delivery, a means of lifting and moving which can vary depending on the size, harvesting design and customer’s preferences.

SCISSORS TABLE LIFT

Rolls between vessel legs, lowers bottom section, moves it elsewhere for harvesting

ELECTRIC SCISSORS LIFT

Heavy duty, all stainless, precise vertical positioning, XP Div.II motor and controller

MANUAL FORK LIFT

Lowers bottom head for harvesting, top head or entire Nutsche & stand can also be lifted. (Stand is also rotating type)

PNEUMATIC LIFT

With swing-out of bottom head for harvesting

BUILT-IN MANUAL CRANK LIFT

Lowers bottom head with swing-out for harvesting

BUILT-IN PNEUMATIC LIFT

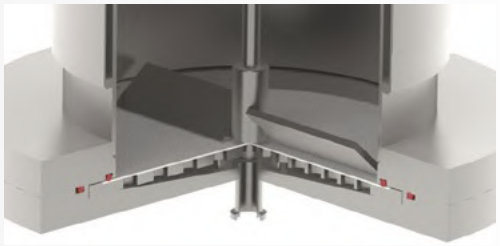
Lowers bottom section to ground, can be wheeled away for harvesting

Agitators and Mixers

To perform their role in filtering, Nutsche filters do not necessarily require any type of stirring device; some units are produced and used without them. With these Nutsches, slurry is fed into the vessel, which is then pressurized, forcing the liquid (filtrate) out through the bottom drain. See example to the right. This particular unit also has no jacket for heating – in this application, the client neither needs to mix nor dry their filter cake which is harvested wet.

There are other applications in which the customer requires that the slurry be mixed, sometimes vigorously. For this duty, Pope pressurizable mixers are utilized. One has an electric gear motor while the other an air motor, the latter sometimes chosen for explosion proof properties in rated electrical hazard (Div. I or II) classification areas. Explosion proof electric motors are also available, though for smaller scale Nutsches, the motors and gear reducers can wind up being out of scale in size and top heavy. Pope mixers are reconfigurable, allowing interchanging between motor types and speeds and various styles, sizes and numbers of impellers, etc. There are also related specialized types called homogenizers or disintegrators which operate at very high speeds, with rotors and stators. These are utilized for breaking larger particles into smaller ones of controlled size.

Many Nutsches utilize a Filter Cake Agitator with full diameter, angled blades and a mechanism for raising and lowering the blade height while rotating. These operate at slower RPM than the above described mixers. Their main function is to dig up and churn the wet filter cake during the heated vacuum drying cycle, breaking up lumps and evenly mixing the solids for efficient drying. After the churning, the blade is raised, the direction is reversed and lowered onto the top of the solids to smooth the cake. The churning process can be kept operating continuously until the end of the drying cycle, followed by harvesting. Most of these agitators have motorized rotation but special manual “pepper grinder” version are available. A special “S” design blade is incorporated in Nutsches with side discharge to push the solids out of the vessel. Typical Cake Agitators have a motorized rotation and manual up and down vertical movement. Vertical movement can also be motor or pneumatically driven as an option. Div. I or II electrical class versions are available.



Filter Types

To perform their role in filtering, Nutsche filters do not necessarily require any type of stirring device; some units are produced and used without them. With these Nutsches, slurry is fed into the vessel, which is then pressurized, forcing the liquid (filtrate) out through the bottom drain. Below is an example. This particular unit also has no jacket for heating – in this application, the client neither needs to mix nor dry their filter cake which is harvested wet.

PORE SIZES: There are different ways in which filters can be described and quantified regarding their filtering characteristics. The most commonly used is micron size, (sometimes designated as µ). 1 Micron = 1 thousandth of a Millimeter = 1 millionth of a Meter. Many particles in processing are somewhere between 1 and 100 µ, except for very large crystals or microscopic dusts, bacteria or viruses which can be down to 0.1 µ and smaller. For example, a user may need to filter particles with and average size of 40 µ. There is also the concept of particle size distribution to be considered; a slurry with a wide range could have, for example, anywhere from <1 to > 100 µ, a tight range might be 20 to 60 µ. The role of the filter is to capture as much of the particles in a slurry as possible and the µ value indicates that it will capture nearly all particles larger than that size. If a user choses a µ micron filter for almost all applications in the hope of capturing 100% of product, a 1 µ filter may do it, but being so fine, may slow the filtering rate to an extremely long time. Thus there is often a trade-off to examine where liquid will flow out at a reasonable, acceptable rate, while at the same time, result in an acceptably low loss quantity of total product (acceptable yield).

Pope filters are available from 1 to 100 µ and easily interchangeable. The micron size is typically selected to be a bit smaller than the nominal particle size. For example, if the average particles are 27 µ, in size and the distribution is not overly wide, a 10 µ filter may be a good choice to start with. If the filtering rate with this is too slow, the user subsequently might try a 20 µ filter instead, checking to see if the yield is acceptable. It is a good idea to purchase a few filter disk spares when a Nutsche is purchased, including alternate pore sizes, (unless the optimal filter choice has been previously tested and established).

STAINLESS STEEL SCREEN: Various filter styles and materials are utilized. Pope's default is a multilayer 316-SS screen, and these also must be used when an up/down cake agitator is incorporated in the Nutsche because non-metal types can be stretched out and torn by the agitator blade. These are typically formed with 5 layers of mesh of differing micron sizes sintered together via compression and high heat. The "control" layer is the second screen down from a protective top screen and is the one which has the critical micron pore size, (µ). The lower layers are for strength and support.

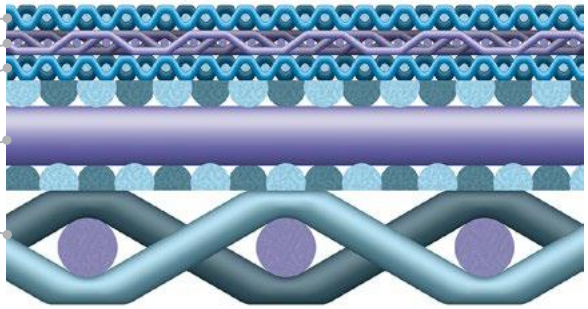


Image source: BOPP.com (BOPP - Sintered Wire Cloth)

Absolute filter fineness: 5 – 120 microns

Image source: BOPP.com

Filter Types

NON-METAL FILTER MEDIA: There are many materials from which filters are fabricated. Polypropylene is often utilized for its resistance to a wide range of chemicals. Woven cloths made of this and others such as Polyester, Rayon, Nylon, Fiberglass, etc. are made in large rolls from which required shapes are cut – in the case of Nutsche filters the shapes are simply circles of different diameters required for the different size Nutsches offered. Teflon and Teflon-family materials are also available for more extreme ranges of solvents though the physical characteristics are not as friendly as others to wettability and passage of liquids. The tightness of the weave and fiber diameters used determine the nominal pore sizes, though the precision of pore size cutoff is not as accurate as is possible with metal screens. However, the relatively low cost makes these suitable for single use. In addition to cloths, non-woven filter membrane rolls are made from these and other materials as well, including cellulosic "filter papers". These are much thinner than cloths and can be made with smaller submicron pore sizes such as 0.45, 0.2 or even down to 0.02 µ, and sometimes have very specific physical characteristics and applications.

POPE STOCKS: Polypropylene filter cloth screens in different pore sizes. These require a polypropylene coarse screen between the filter and stainless steel support plate for support because without it, the soft cloth would be pushed into the support plate holes. In addition, Pope can provide alternate materials such as those mentioned above. Customers are also free to cut circles of their own in-house materials to the proper diameter; these must also use the coarse screen for support. As previously mentioned, cloths and membrane filters are normally not compatible with up/down cake agitators due to possible stretching and tearing.

Pope's unique design allows for use of either non-metal or stainless steel filters. (shown)

Turnkey & Crystallizing Nutsche Reactor Systems

Pope Nutsche Filter-Dryers are known and used worldwide for final purification of pharmaceutical intermediates, high purity fine chemicals and other critical applications. Pope Reactors and Vessel Systems have been utilized for decades in the same industries. In many cases, Pope supplies reactors utilized for crystallization and a companion Nutsche for filtering, washing and drying the crystal slurries transferred from the reactor.

The advantages of pairing a reactor and Nutsche Filter-Dryer include:

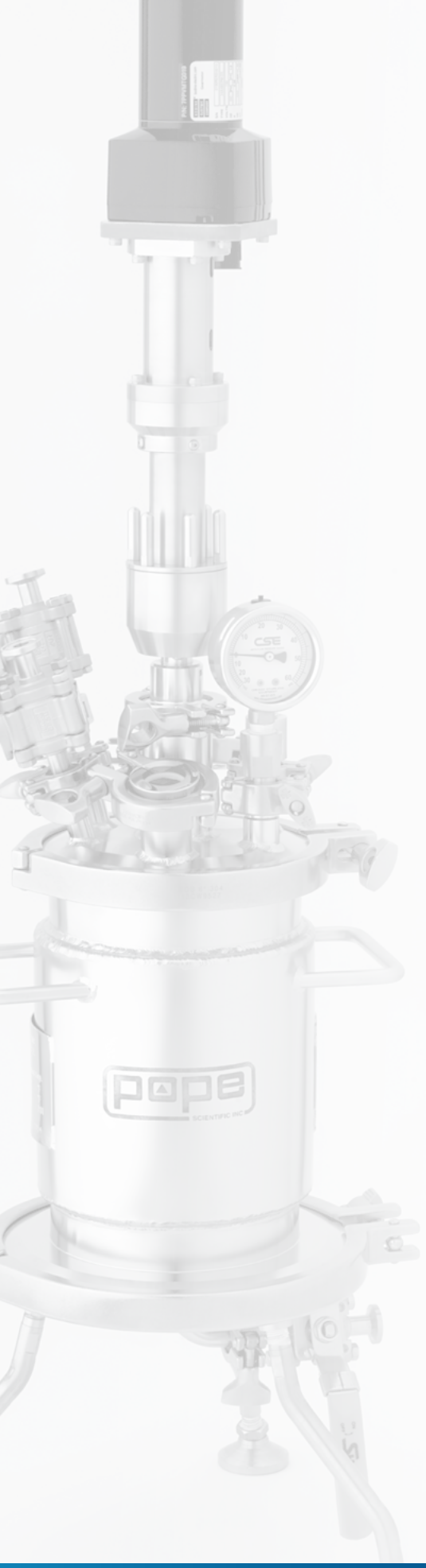
- Each vessel is optimally designed for performing these two different processes
- Saves time by having both processes running simultaneously
- Can be performed with no open handling of the raw material or slurry

Pope has developed a variety of turnkey systems including:

- 5L Reactor and 4L Nutsche (Benchtop scale)
- 30L Reactor and 25L Nutsche
- 60L Reactor and 50L Nutsche
- 120L Reactor and 100L Nutsche
- 200L Reactor and 170L Nutsche
- 400L Reactor and 350L Nutsche
- Custom sizes and designs available upon request

NUTSCHE CANNABIS APPLICATIONS:

Isolation is a critical operation, and often the final step in the entire manufacturing process. Pope Scientific's Nutsches are preferred-proven for CBD crystallizing isolation to 99.9%, with less than 0.3%, to undetectable THC levels.



pope SCIENTIFIC INC
Solution Driven.

LABTEX™
The UK's Laboratory Equipment Experts

POPE SCIENTIFIC, INC
351 N. DEKORA WOODS BLVD.
SAUKVILLE, WI 53080 USA

INFO@POPEINC.COM
+1-262-268-9300
WWW.POPEINC.COM

NEW MILLS, BROUGHAM ROAD,
MARSDEN, HUDDERSFIELD,
WEST YORKSHIRE, HD7 6AZ,
UNITED KINGDOM

INFO@LABTEX.CO.UK
01484 600 200
WWW.LABTEX.CO.UK

For over 50 years, Pope Scientific Inc. has been at the forefront of manufacturing chemical processing equipment, engineered systems and laboratory apparatus for science and industry. Internationally known, Pope provides specialized technical expertise in the design and manufacturing of chemical processing equipment.

SOLUTIONS FOR CHEMICAL PROCESSING EXCELLENCE

Lab, Pilot & Large Scale Production Capabilities

- Wiped-Film Molecular (Short Path) Stills and Evaporators
- Batch and Continuous Fractional Distillation Systems
- Hybrid Wiped-Film / Fractional Distillation Systems
- Pressure Vessels, Reactors, and Process Vessel Systems
- Nutsche Filter – Dryers
- Sanitary / Pressurizable Mixers
- Cannabis Specialties
- Toll (Contract) Distillation, Process Development and Testing Services

POPE – WHEN PROCESS EXPERTISE AND SUPPORT MATTER.