



# Technical Considerations in Selecting Vacuum Equipment for Chemical Processes

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# Introduction

Many processes involving chemicals or chemical reactions are best performed under vacuum. These processes are widely diverse. The incorrect selection and use of vacuum system components in processes involving chemical exposure can lead to expensive, time consuming, and even dangerous equipment failure. This guide is designed to help identify and understand some of the important considerations when designing a vacuum system for chemical processes.

After studying this guide, please contact us if you are still unsure or unclear about equipment selection. Please be ready with your process requirements and the identities of any significant corrosive, toxic, or otherwise difficult chemicals you will use. The more information you provide, the easier it is for our engineers and chemists to assist you in choosing the best equipment solution for your process.

#### Before starting system design, you should know:

- **1. The level of vacuum needed:** rough, medium, high, or ultra-high.
- 2. The type of process used: chemical, wet, dirty, or a combination.

A **chemical process** involves or produces corrosive, organic, toxic, or flammable vapors.

A **wet process** involves or produces liquid(s) or condensable vapors.

A **dirty process** involves or produces dust or chemicals that are gel, slime, grease, or similar (in the normal temperature range of the process).

A project could include any combination of chemical, wet, and/or dirty processes. The process attributes determine the types of pumps, gauges, seals, filters, and precautions that are needed.

# Levels of Vacuum

# Vacuum Levels

Vacuum Level	Pressure (mbar)	Pressure (Torr)	%of Atmosphere
Rough	10 <sup>3</sup> to 1	760 to 0.75	100% to 0.1%
Medium	1 to 10 <sup>-3</sup>	7.5 to 7.5x10⁴	0.1% to 0.0001%
High	10 <sup>-3</sup> to 10 <sup>-7</sup>	7.5x10 <sup>-4</sup> to 7.5x10 <sup>-8</sup>	0.00001% to 0.0000001%
Ultra-High	<10 <sup>-7</sup>	<7.5x10 <sup>-8</sup>	<0.000001%

**Table 1**: Vacuum Pressure ranges at the four commonly usedlevels of vacuum.

The vacuum levels described in **Table 1**, above. are often used colloquially in vacuum terminology. The exact pressure cut offs for each range are formally defined in ISO 3529-1:2019, but some organizations follow different standards.<sup>1</sup> For pressures near the transition pressures listed above, it may be best to cite both ranges to avoid confusion. For example, "rough-to-medium vacuum" is a more universally appropriate description for a pressure of 1.2 mbar.

#### **Rough Vacuum:**

#### Atmospheric Pressure to 1 mbar (0.75 Torr)

Rough vacuum is any pressure less than ambient atmospheric pressure, but greater than 0.1% of atmosphere. Processes performed in the rough vacuum range are the most common. Rough vacuum is used in any situation where air needs to be removed, but 0.1% gas remaining is acceptable.

Rough vacuum is used in packaging to extend the shelf life of food and chemical products, in polymers to remove air bubbles from resin, during manufacturing to prevent corrosion and oxidation, in altitude simulation to mimic high atmosphere conditions (up to approximately 190,000 ft), in insulation to prevent



thermal transfer, in natural product isolation as part of the distillation process, and in many chemical and analytical processes where the presence of oxygen or nitrogen is only acceptable in very small quantities.

There are many types of vacuum pumps that can create rough vacuum. These pumps fall into two main categories: **dry pumps** and **wet pumps**.

## **Dry Pumps**

Dry pumps do not use oil or water in the vacuum generation process and therefore do not introduce vapor contaminants into the vacuum system. They have solid seals (usually rubber or Teflon<sup>®</sup>) and very few contacting surfaces. Dry pumps are typically more expensive than wet pumps for the same pumping speed. The most common dry roughing pumps are scroll, diaphragm, and dry roots lobe pumps.

#### Wet Pumps

Wet pumps use oil or water in the vacuum generation process. The use of oil or water improves the vacuum seal at rough vacuum levels and lubricates the pump which increases pump longevity. However, the liquid used in wet pumps can backstream into the vacuum chamber causing contamination of anything inside, and pump oil must be periodically replaced or cleaned. Wet pumps are typically less expensive than dry pumps for the same pumping speed. The most common roughing wet pumps are rotary vane, lubricated roots lobe, and rotary piston.

## Gauges

There are many types of gauges that can be used to accurately measure rough vacuum. The most common types are piezo, capacitance manometer, Bourdon, convection-Pirani, and thermocouple.



**Figure 1:** A sample variety of rough-to-medium vacuum gauges. From left to right: MKS 626D capacitance manometer (**P103249**), Swagelok<sup>®</sup> PGI-63C-PC0-LA02 Bourdon (**P104860**), IVP convection-enhanced Pirani (**P1010258**), Teledyne-Hastings DV-4D thermocouple (**P102048**), Agilent Varian PCG-750 Pirani + capacitance manometer combination gauge (**P107188**).

# **Medium Vacuum:**

#### 1 mbar to 0.001 mbar (0.75 to 7.5 × 10<sup>-4</sup> Torr)

Medium vacuum extends from below rough vacuum to 0.001 mbar ( $7.5 \times 10^{-4}$  Torr), or one millionth of an atmosphere. Medium vacuum is used in chemical processes where 0.0001% residual gas pressure is acceptable. This includes processes such as coatings, vacuum welding, and very high altitude simulation (approximately 190,000 ft to 478,000 ft).

Most types of roughing vacuum pumps can also achieve medium vacuum. These include scroll, rotary vane, roots lobe, and rotary piston pumps. The same is true of gauges (except Bourdon gauges). Many gauge types are accurate only within a limited pressure range, so multiple gauges of the same or different types may be necessary to accurately measure the full range of process pressures.



**Figure 2**: A sample variety of roughing vacuum pumps for rough and medium vacuum use available on idealvac.com. From left to right; top: KNF Neuberger N938.50 diaphragm pump (**P1012409**), Edwards nXR60i dry roots pump (**P1010962**), Edwards nXDS20i dry scroll pump (**P105132**); middle: Welch 1400 rotary vane pump (**P103008**), Alcatel Adixen 2015I rotary vane pump (**P103925**), Agilent Varian DS402 rotary vane pump (**P102296**); bottom: Leybold WSU-251 roots blower pump (**P105158**), Edwards E2M175 rotary vane pump (**P101931**).



# **High Vacuum:**

#### .001 mbar to 10<sup>-7</sup> mbar (7.5 × 10<sup>-4</sup> to 7.5 × 10<sup>-8</sup> Torr)

High vacuum extends from immediately below the medium vacuum range to  $10^{-7}$  mbar (7.5 ×  $10^{-8}$  Torr), or one ten billionth of an atmosphere. High vacuum is often used in chemical processes where zero contamination is acceptable, such as in semiconductor manufacturing, space simulation, and other high purity processes.

High vacuum requires the use of at least two vacuum pumps, a roughing pump to initially remove most of the gas from the chamber and a second high vacuum pump that begins operating once medium vacuum is achieved. During high vacuum operation the roughing pump continues pumping, backing the high vacuum pump. The most common types of high vacuum pumps include diffusion pumps, turbo-molecular pumps, ion sputtering pumps, and cryogenic (cryo) pumps.



*Figure 3:* A sample variety of high vacuum pumps available on idealvac.com. From left to right: Agilent Varian VHS-250 diffusion pump (*P102159*), Leybold 350i turbo pump (*P109074*), Edwards Brooks CTI Cryo-Torr 8 cryo pump (*P109173*), Agilent Varian Vaclon Plus 40 ion pump (*P105784*).

Rough, medium, and high vacuum systems require seals at each joint or connection between any two components or pieces of equipment. The most commonly used seals for these levels of vacuum are rubber O-ring seals, although rubber seals limit performance at the lower range of high vacuum due to their inherent porosity at a microscopic level. For better performance, all-metal face seals can be used.



**Figure 4:** A sample variety of available KF and ISO hardware. From left to right; top: ISO 63 EVAC aluminum metal knife edge seal for high/ultra-high vacuum use (**P103404**), KF-16 centering ring with Viton O-ring (**P101242**), ISO 63 centering ring with Viton O-ring and aluminum retaining ring (**P101766**), ISO 63 red silicone replacement O-ring for high temperature applications (**P103656**); middle: KF-50 centering ring with Viton O-ring (**P101245**), IVP KF-25 hinge clamp (**P101199**), ISO-K 160 to 250 double claw clamp (**P104067**), KF-16 EVAC aluminum outer knife edge seal for high and ultra-high vacuum use (**P103400**); bottom: KF-25 flexible steel bellows hose (**P103719**), ISO 63 mitered 90 degree elbow (**P104017**), KF25 to KF-40 straight reducer (**P103458**), ISO 160 to ISO 63 reducing cross (**P104054**).

Vacuum system cleanliness is necessary for high vacuum operation. Chemical, wet, and dirty processes make high vacuum more difficult to achieve. Using dry or high purity gas to vent the chamber can result in much faster pump down times to high vacuum because it avoids water adsorption onto the chamber walls from normal air, which always contains some amount of water vapor. A single fingerprint generates an estimated gas load of  $1.3 \times 10^{-5}$  mbar l<sup>-1</sup> s<sup>-1</sup>, which would require a pumping speed of approximately 240 CFM to maintain a pressure of  $1 \times 10^{-7}$  mbar (7.5 × 10<sup>-8</sup> Torr).<sup>2</sup> Water, oil, or chemical contamination can often be overcome by regular chamber cleaning, chamber bake out procedures, higher pumping speeds, or extreme patience.





*Figure 5:* Oily fingerprint residue on a machined stainless steel ISO-K DN 160 blanking plate (*P101684*). Wear clean gloves when handling vacuum equipment. It saves time and prevents problems.

## **Ultra-High Vacuum:**

#### Below 10<sup>-7</sup> mbar (<7.5 × 10<sup>-8</sup> Torr)

Ultra-high vacuum is any vacuum with pressures lower than high vacuum. Ultra-high vacuum is used for deep-space simulation, physics experiments, and extreme purity processes that exceed even high vacuum requirements.

Ultra-high vacuum requires the use of a turbomolecular, ion sputtering, or cryogenic pump, and often uses more than one of these types. Ultra-high vacuum also requires the use of all-metal seals or differentially pumped seals.

A vacuum system that uses rubber seals cannot obtain ultra-high vacuum because the seals are too permeable to gas. Differentially pumped rubber seals can be used to obtain ultra-high vacuum after an outgassing period for the inner seal. Most chemical processes and all wet or dirty processes prevent ultra-high vacuum from ever being achieved.



**Figure 6:** A sample variety of high and ultra-high vacuum compatible CF and Swagelok<sup>®</sup> VCR hardware available on idealvac.com. Left to right; top: CF 8 inch silver plated copper gasket (**P103986**), CF 6 inch copper gasket (**P102281**), CF 2.125 inch side ported to 1/4 inch female Swagelok VCR (**P1010534**), Swagelok VCR 4 male plug (**P109923**), CF 6 inch 45 degree elbow (**P104847**); bottom: CF 4.5 inch to CF 2.75 inch reducer tee (**P103788**) with blank (**P102248**) and bolt set (**P104377**), CF 2.125 inch 6-way cross (**P104717**), Swagelok VCR 4 butt weld gland (**P109966**).

# Considerations for Different Types of Processes

# **Chemical Processes**

A chemical process involves or produces **corrosive**, **organic**, **toxic**, or **flammable** vapors. A chemical process is not necessarily wet or dirty, but many chemical processes are. Chemical processes require special considerations when designing a vacuum chamber and when choosing system components. If the process produces corrosive vapors, the chamber and all chamber seals, plumbing, and vacuum pumps must be rated to withstand those vapors. Due to the great variety and differences between chemical structures and properties, each chemical process must be individually examined for safety and chemical compatibility with the entire vacuum system.



# **General Safety**

For all chemical processes, all chamber vents and exhausts must be plumbed safely outside of the working area. Filtration or neutralization "scrubber" systems may be required for especially caustic or toxic compounds.



# Corrosives

Corrosive chemicals typically fall under four categories: acids, bases, oxidizers, and reducers, each of which has a unique corrosion mechanism. Corrosive chemicals may fall under more than one category, such as nitric acid, which is both an acid and an oxidizer. If a chemical is corrosive, it will typically be listed in the chemical's safety data sheet (SDS). Corrosive chemicals require the use of corrosion resistant materials, such as stainless steel, aluminum, noble and exotic metals, and fluorinated plastics and rubbers.

**Table 2,** on the following page, shows many of the more common materials used in vacuum system components and their relative ability to withstand a variety of chemicals that might be used in vacuum processes.

Chemical or corrosive rated pumps are made of more chemically resistant materials than their standard counterparts. To increase chemical resistance, 304 stainless steel parts are usually replaced with 316 stainless steel, and Viton<sup>®</sup> O-rings are replaced with Kalrez<sup>®</sup>. Despite being advertised as "chemicalresistant", chemical compatibility charts for chemical rated pumps are typically not readily available.

If a vacuum pump cannot handle the chemicals produced in the process, a trap or filter may be necessary, placed between the pump and the chamber, to reduce the chemical load on the pump.



Figure 7: Left to right: An Edwards corrosive-rated nXDS20iC dry scroll pump (**P105129**), and a standard nXDS20i dry scroll pump (**P105132**). Both have identical pumping performance for non-corrosive gases. The corrosive-rated nXDS20iC can handle more corrosive chemicals because it is equipped with Chemraz<sup>®</sup> seals and stainless steel springs. A standard XDS20i can be converted into a nXDS20iC with a conversion kit (**P109572**).

Filament vacuum gauges, such as thermocouple, Convectron®, Pirani, hot cathode, and cold cathode gauges degrade quickly under corrosive conditions. Thermocouple and cold cathode gauges have thicker filaments than Convectron, Pirani, and hot cathode gauges, and will last marginally longer. Chemically rated diaphragm style gauges, such as piezo, capacitance manometer, diaphragm manometer, and Bourdon gauges will last much longer and produce more reliable results. This limits the use of high vacuum gauges in corrosive chemical processes. If high vacuum measurement is necessary, a high vacuum gauge can be protected from the process by a valve which opens only when the rough vacuum gauge reaches a sufficiently low pressure. Fortunately, this is easily automated since many gauge controllers have programmable setpoints.



**Figure 8:** Left to right; IVP convection-enhanced Pirani gauge (**P1010258**), Teledyne-Hastings DV-6M thermocouple gauge (**P102046**), IVP hot cathode ion gauge (**P102041**), MKS Granville-Phillips<sup>®</sup> 355 hot cathode Micro-Ion<sup>®</sup> gauge (**P1012041**), Leybold Penningvac<sup>®</sup> PTR 90 N Pirani + cold cathode combination gauge (**P109872**), Inficon PSG500 Pirani gauge module (**P1010832**).

The left three gauge types above in **Figure 8** are inexpensive and easily replaced but require an external controller. The right three gauge modules and combination gauges are more expensive and more difficult to service, but do not require an external controller.



#### Chemical Compatibility of Some Common Vacuum Chamber Materials with Corrosive Chemicals

Chemical Category	Chemical	304 SS <sup>3-5</sup>	316 SS <sup>46</sup>	6061-T6 <sup>4,7</sup>	Copper <sup>4</sup>	Nickel <sup>5</sup>	Chrome <sup>8</sup>	Inconel <sup>5</sup>	Glass <sup>4</sup>	PTFE ⁴	Buna-N 70A ⁴	Viton 75A <sup>4</sup>	Kalrez 4079 <sup>9</sup>
	Hydrochloric Acid (HCl)	D	D	D	D	В	D	В	А	А	С	А	А
	Phosphoric Acid (H <sub>3</sub> PO <sub>4</sub> )	D	D	C-	D	A-	B-	A-	В	А	D	Α	А
ACIDS	Nitric Acid (HNO <sub>3</sub> )	А	А	D	D	D-	Α	D	С	А	D	А	А
	Sulfuric Acid $(H_2SO_4)$	C-	С	D	D-	D	B-	В	A	А	B-	A	А
	Acetic Acid (HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> )	С	B-	B-	В	A-	A	A-	А	А	B-	В	А
BASES	Sodium Hydroxide (NaOH)	В	A-	D	B-	A	А	А	В	А	В	В	А
BAGES	Ammonia (NH <sub>3</sub> )	В	А	A-	D	С	A	А	А	А	В	D	А
	Water (H <sub>2</sub> O)	А	А	А	В	А	A	А	А	А	А	А	А
	Ozone (O <sub>3</sub> )	В	А	А	А				А	А	D	А	А
OXIDIZERS	Hydrogen Peroxide (H <sub>2</sub> O <sub>2</sub> )	В	A-	А	D		A-	А	А	А	D	А	А
	Nitrous Oxide (NO <sub>2</sub> )	В	В	В	В	A			A	А	A	A	А
	Chlorine (Cl <sub>2</sub> )	D	В	D	D	С	B-	С	А	А	D	A-	A-
REDUCERS	Hydrogen (H <sub>2</sub> )	А	А	А	А				А	А	А	А	А
	Sulfer Dioxide (SO <sub>2</sub> )	D	D	В	В		А	С	А	А	D	А	А
	Hydrazine (N <sub>2</sub> H <sub>4</sub> )	А	А	С	А				А	А	В	А	А
	Carbon Monoxide (CO)	А	А	А	А		А		А	А	А	А	А

Table 2: Graded rating of the compatibility of some commonly used corrosive chemicals with vacuum chamber materials, where:

A = Excellent: Can be used continuously and handle elevated temperatures.

**B = Good:** Can be used continuously at room temperature at moderate concentrations. Avoid high temperatures and concentrations.

**C** = Fair: Can be used intermittently at room temperature at low concentrations.

Avoid high temperatures, high concentrations, or prolonged exposure.

D = Poor: Cannot be used.

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NOTICE
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The information in this table has been supplied to Ideal Vacuum by other (reputable) sources and is to be used ONLY AS A GUIDE in selecting equipment for appropriate chemical compatibility. Before permanent installation, test the equipment with the chemicals and under the specific conditions of the application.



Corrosive compounds typically become more strongly corrosive at higher temperatures and less corrosive at lower temperatures. If a chemical process occurs at a high temperature, this effect needs to be considered. By the same principle, sensitive equipment can be provided some level of protection if kept cool, but not so cold that the corrosive chemical condenses on the equipment.

#### **Corrosives: Acids**

Vacuum chambers and fittings made of 304 stainless steel can withstand many acidic vapors up to 3% mole ratio concentrations whereas 316 stainless steel can withstand many acidic vapors up to 20% mole ratio concentrations.<sup>10</sup> Aluminum chambers typically corrode in strongly acidic vapor environments (such as with HCl vapors) but can withstand many weak acid vapors (such as with acetic acid). For greater acid resistance, a plastic or glass chamber can be used, or the chamber can be plated with an acid-resistant coating such as nickel, chrome, gold, PTFE, or quartz.

#### **Corrosives: Bases**

Vacuum chambers and fittings made of 304 stainless steel can withstand most basic vapors and 316 stainless steel is even more resistant. Aluminum chambers are highly resistant to most bases since aluminum spontaneously forms a thin, protective aluminum oxide surface coating. For greater base resistance, a plastic or glass chamber can be used, or the chamber can be plated with a base-resistant coating such as nickel, chrome, gold, PTFE, or quartz.



## **Corrosives: Oxidizers**

# Of all corrosive compounds, oxidizers pose the greatest threat to vacuum pumps.

Vacuum chambers and fittings made of 304 stainless steel are oxidation resistant but cannot withstand strong oxidizers. 316 stainless steel is more resistant, but still cannot stand up to strong oxidizers such as chlorine, bromine gas, thionyl chloride, and

ozone. Aluminum is often more resistant to oxidation than steel due to its spontaneously forming protective aluminum oxide coating. However, oxidizers that are also acids and some strong oxidizers can eradicate or bypass the aluminum oxide coating. Monel<sup>®</sup>, an alloy of copper and nickel, is an exotic material that is highly oxidation resistant.<sup>5</sup> For greater oxidation resistance, a glass chamber can be used, or the chamber can be plated with an oxidation-resistant coating such as nickel, chrome, gold, PTFE, or quartz.

Viton, and other fluorocarbon O-rings (FKM) are highly chemically resistant and are commonly used as vacuum seals in rough and high vacuum systems. However, they can degrade in highly oxidizing environments. Kalrez O-rings are a more chemically resistant option in these cases. If Kalrez is not available or is prohibitively expensive, Simriz<sup>®</sup>, Chemraz<sup>®</sup>, Markez<sup>®</sup>, and Kyflon<sup>®</sup> are alternative perfluorocarbon (FFKM) O-ring materials.



**Figure 9:** Standard KF and ISO Viton O-rings can be replaced with Kalrez for greater chemical and thermal stability. From left to right; top: KF-16 centering ring with Viton O-ring (**P101242**) and with Kalrez O-ring (**P106147**); bottom: ISO 100 centering ring with Viton O-ring (**P101768**) and with Kalrez O-ring (**P108590**).

Metal seals are helpful for high vacuum and required for ultra-high vacuum applications. They may be aluminum, copper, silver-plated copper, or gold-plated copper depending on the required level of chemical resistance. Copper gaskets are commonly used as vacuum seals in high and ultra-high vacuum systems. However, copper degrades under oxidizing conditions. Silver-plated, gold-plated, solid aluminum, and solid gold gaskets are more oxidation-resistant gasket options.





**Figure 10:** Metal seals for high vacuum and ultra-high vacuum applications. From left to right; Inficon KF-16 aluminum ultra sealing ring (**P1011615**), KF-16 EVAC aluminum outer knife edge seal (**P103400**), ISO 63 EVAC aluminum knife edge seal, CF 8 inch copper gasket (**P102282**), CF 10 inch silver plated copper gasket (**P104179**).

#### **Corrosives: Reducers**

Most metals, including stainless steel, aluminum, and copper are highly resistant to reducing compounds. Reducers are a greater threat to rubber and plastic vacuum components. Check chemical compatibility charts for rubber and plastic components with the reducers present in the chemical process. If the specific chemical is not listed, check for chemicals of the same family.

#### **Organics**

Organic vapors do not generally pose a direct threat to most of the metal components in a vacuum system. They are more dangerous to rubber or plastic parts which can absorb the vapors, causing swelling, softening, and possible degradation, leading to increased wear and tear on dynamic seals in pumps, and reduced performance of static seals in chambers. Seal swelling can also prevent a vacuum chamber from resealing after venting. Absorption of organic vapors will lead to significantly longer pump down times in medium and high vacuum systems. **Table 3**, below, lists the maximum swelling that occurs when common vacuum compatible O-ring materials come into contact with commonly used cleaning solvents.

Organic vapors decompose into a solid black carbon residue if they encounter a hot surface in vacuum. One area where residue commonly builds up is on electrical connections and wires. This can eventually lead to electrical arcing or failure. Organic residue degrades vacuum gauge performance. Thin filament gauges such as Convectron, Pirani, and hot cathode are most affected. Thermocouple and cold cathode gauges are less affected. Diaphragm style gauges, such as piezo, capacitance manometer, and Bourdon gauges are least affected, but will still exhibit reduced performance over time.



**Figure 11:** A combination power + thermocouple vacuum chamber feedthrough (**P108066**) used with a process that produced a sticky organic residue. The copper wires were carrying ~20 A of current at 208 VAC and got very hot, carbonizing the organic residue. Eventually the electricity arced to the grounded steel chamber, resulting in a blown fuse, a ruined experiment, and a lengthy cleanup (see Dirty Processes - Scum, page 14).

	Hexanes	Isopropanol	Methanol	Acetone	Methyl Ethyl Ketone	Acetic Acid (30% Solution)	Potassium Hydroxide (30% Solution)
Buna-N 70A 11	<10%	10 - 30%	<10%	Failure	Failure	10 - 30%	10 - 30%
Viton 75A <sup>12</sup>	4%	<10%	90%	Failure	>200%	10 - 30%	Failure
Kalrez <sup>13</sup>	<10%	<10%	<10%	<10%	<10%	<10%	<10%

#### Maximum Percent Volume Increase (Swelling) of O-Ring Materials with Common Solvents

**Table 3**: Maximum swelling of O-rings (in percent relative to original volume) when subjected to common cleaning solvents. Volume increase is relative to the original O-ring volume. "Failure" means that the O-ring swells so much that the rubber surface splits and can no longer maintain a vacuum seal or that the rubber material dissolves in the solvent.



In cases where organic residues are an issue, gauges should be purchased with a separate, external controller to minimize gauge replacement cost. Preferably, gauges are mounted vertically with the inlet facing downwards so that deposition of organic residue is reduced. If high vacuum measurement is needed, a high vacuum gauge can be protected from the process by a valve which opens only when the rough vacuum gauge reaches the high vacuum crossover pressure threshold.



#### **Toxics**

Toxic materials do not pose a direct threat to any vacuum system component unless they also have corrosive or organic properties. However, toxic materials do pose a direct threat to the system's operator and other personnel.



The vacuum system's operator is responsible to understand the unique dangers and safety protocols necessary for their specific toxic process. All interior surfaces of the vacuum chamber system must be considered contaminated after a toxic chemical process takes place. Toxic materials can become absorbed into any liquid, rubber, or plastic present in the system, including vacuum grease, pump oil, and all rubber seals. These components must be considered deeply contaminated until proven otherwise and must be handled with proper care during use, replacement, and disposal. Chambers that contain toxic materials should be rated for positive pressure as well as vacuum to ensure that toxic materials do not escape in case of a pressurization event. To remove residual toxic vapors from the chamber it is often advisable to flow a purge gas through the chamber for an extended period or perform multiple purge-pump cycles before opening the chamber. Utilizing an airlock can be a good solution for introducing or removing items from a toxic-contaminated chamber.



#### **Flammables**

Flammable materials and vapors must be kept away from ignition sources even in vacuum environments. Due to reduced thermal conduction and convection in vacuum environments, electrical components and wires can become very hot. The utmost care must be exercised when electrical devices are used in flammable vacuum processes. Filament vacuum gauges, such as thermocouple, Convectron, Pirani, hot cathode, and cold cathode gauges contain elements that reach glowing hot conditions and may not be suitable for use in flammable chemical processes. Diaphragm style gauges, such as piezo, capacitance manometer, and Bourdon gauges do not have this issue. This limits the use of some types of high vacuum gauges in processes that produce flammable vapors. If high vacuum measurement is necessary, a high vacuum gauge may be energized only when a rough vacuum gauge measures below the combustion pressure of the flammable compounds.



**Figure 12:** From left to right: MKS 355 hot cathode Micro-Ion<sup>®</sup> gauge (**P1011205**), IVP XGC-321 Convection-Enhanced Pirani Gauge (**P1010260**); bottom: MKS Granville-Phillips 358 hot cathode ion and convection-enhanced Pirani gauge controller (**P1011910**). The gauge controller automatically energizes the hot cathode ion gauge once the pressure measured by the convection-enhanced Pirani gauge drops below a set point.



Many flammable materials are also organic. All precautions for organic materials should also be used for flammable materials.

# Wet Processes

A wet process involves or produces **liquid**(s) or **condensable vapors**. A wet process does not have to include water. All liquids (with the exception of vacuum grease) are considered wet.

A wet process is not necessarily chemical or dirty, but many wet processes are. Many vacuum pumps cannot tolerate the presence of liquid and will break if used to pump on a process that generates liquid or condensable vapors. It is important to select the correct type of pump and pump operation mode for the amount of liquid introduced by the wet process.

Caution should be used when performing a wet process when electrified components are in the vacuum chamber. If vapor condenses on the energized electrical components, failure may occur.

Wet processes are typically performed under rough or medium vacuum levels. High vacuum can be achieved with sufficiently high pumping speeds. Ultra-high vacuum is almost impossible to achieve once liquid is introduced into the system.

## Liquids

Liquid is a state of matter where molecules flow freely to conform to the shape of their container but do not expand to fill the container (they have a constant volume). Most liquids have a significant vapor pressure, which means that some molecules leave the liquid phase and form a layer of gas above and around the liquid. This gas layer has a pressure, called the vapor pressure or partial pressure of the liquid, which contributes to the total gas pressure of the system. Under closed conditions, the vapor pressure has a defined value which depends on the system temperature. As a vacuum pump removes some of the vapor, more liquid evaporates to replace it until the liquid has completely evaporated. A few liquids, such as vacuum grease, do not have a significant vapor pressure and will not evaporate under vacuum conditions. These vacuum greases have well defined vacuum limits.

**Table 4**, below, lists the vapor pressures of severalcommon materials used in wet vacuum processes andtheir vapor pressures at normal room temperature.

#### Vapor Pressures at 20 - 25°C of Some Common Materials Used in Wet Vacuum Processes

Substance	mbar	Torr
Water 14	24	18
Methanol 14	169	127
Ethanol <sup>14</sup>	124	93
Isopropanol 14	44	33
Hexane <sup>14</sup>	176	132
DMSO 20	0.8	0.6
Methylene Chloride 14	580	435
Toluene 14	39	29
Ethylene Glycol 14	0.07	0.05
Ethyl Acetate 14	140	105
Acetic Acid 14	21	16
Bromine <sup>14</sup>	280	210
lodine 21	0.3	0.2
Methyl Methacrylate 23	39	29
Bisphenol-A <sup>15</sup>	0.05	0.04
DGEBA (diglycidyl ether of bisphenol-A) <sup>16</sup>	1.5 x 10 <sup>-7</sup>	1.1 × 10 <sup>-7</sup>
Terephthalic acid <sup>17</sup>	8.0 x 10 <sup>-11</sup>	6 × 10 <sup>-11</sup>
1,6-diaminohexane <sup>18</sup>	0.25	0.19
Ethylenediamine 19	14	10.4
Dodecane (wax) <sup>22</sup>	0.19	0.14
Lithium Stearate (lithium grease)	1.2 x 10⁻⁵	9 × 10⁻ <sup>6</sup>

**Table 4:** Vapor Pressures at 20-25°C of some common materials used in wet vacuum processes.

Vapor pressure increases with increasing temperature. The vapor pressure of a substance at 20°C is less than than the vapor pressure of the same substance at 25°C. **Table 4** values were measured at a single temperature within the listed range and may vary slightly from independently measured values.

# Solids

Most solid materials do not have a significant vapor pressure and will not sublimate (turn directly from a solid into a gas) under vacuum conditions. However, some molecular solids, such as ice, light waxes, dry ice, and iodine do have a significant vapor pressure and will sublimate under vacuum. When in doubt, assume that solids do not sublimate.

Some solids contain liquid absorbed inside them. One example is an incompletely cured plastic, resin, or epoxy, which appears solid but contains some residual monomer. Another example is a rubber O-ring which has absorbed some methanol, ethanol, or isopropanol used to clean it. The liquids inside these solids still have a partial pressure and will desorb and evaporate under vacuum. Once the absorbed liquid has completely evaporated the solid will remain.

Any process that involves a liquid, a solid, or an absorbed liquid that creates a vapor pressure should be considered a wet process.

# **Condensable Vapors**

A condensable vapor is a gas that turns into a liquid or solid when compressed or cooled to room temperature. Any vapor that is produced by evaporation of a liquid or sublimation of a solid is a condensable vapor. Many chemical reactions, such as combustion and decomposition also generate condensable vapors. The most common condensable gas is water vapor.

Condensable vapors are damaging to many types of vacuum pumps. Vacuum pumps work by compressing gas at their inlet and releasing it in its compressed form at their outlet. Condensable vapors will condense and form a liquid or solid during the compression process. Because liquids and solids are incompressible, if there is enough condensate built up inside the pump, it will not be able to compress and the pump will seize. Solids and liquids may also gum up the pump mechanism or degrade pump oil and components.

# **Pump Selection for Condensable Vapors**

The full process for selecting the correct pump for condensable vapors is too lengthy to be fully described

here. However, there are a few basic selection considerations. The most important is the amount of water that the pump can tolerate. This value is often published in the pump's data sheet or manual as "water pumping capacity" and expressed typically in g/hour. This specification can be used as an estimate for other condensed liquids. Dry scroll pumps typically can handle the least amount of condensable vapor, followed by dry multi-roots, and rotary vane pumps. Dry screw and roots blower pumps can handle the largest amounts of condensable vapors.

Other selection criteria include peak pumping speed, ultimate vacuum, and compression ratio. These values are generally reported for pumping with no gas ballast, even though most pumps are equipped with one. The gas ballast is a bypass to part of the final compression stage of the pump that allows it to exhaust condensed liquid and prevents further condensation. When pumping condensable vapors, it is best to open or turn on the gas ballast to allow the condensed material to escape. However, when the gas ballast is open to achieve maximum water capacity, peak pumping speed, ultimate vacuum, and compression ratio are all reduced, and the amount of heat and noise the pump generates is increased. Moreover, many pumps can only operate with the gas ballast open for a limited time, usually less than two hours, before overheating.

Wet pumps require more frequent oil changes when pumping condensable vapors as the condensed vapors accumulate in the oil, causing reduced performance. If the chemical vapor is corrosive or otherwise reactive with the pump's components or its oil, damage to the pump can occur if the oil is not changed regularly.

In some cases, the amount of condensable vapors present may exceed any available vacuum pump with the desired pumping speed. In those situations, it may be necessary to add a cooled trap or condenser to remove the condensable vapor before it reaches the pump.

**Table 4**, on the following page, lists some of the more important pump specifications for several of Ideal Vacuum's top selling pumps. Specifications for most pumps can be downloaded at idealvac.com.



## Peak Pumping Specifications and Water Pumping Capacity of Top Selling Vacuum Pumps

Туре	Mfr.	Model	Peak Speed (CFM)	Ultimate Vacuum (Torr)	Compression Ratio	Water Vapor Pressure Max. (Torr)	Water Capac- ity, no gas bal- last (g/hour)	Water Capacity, gas ballast (g/hour)
Dry Scroll	Edwards	nXDS20i	13	0.022	69091	15		
Dry Scroll	Leybold	Scrollvac 18 Plus	11.8	0.0225	67556		24	220
Dry Scroll	Agilent- Varian	IDP-15	7.5	0.01	109600		20.7	235
Dry Multi-roots	Edwards	nXR30i	18	0.022	41455			300
Rotary Vane	Leybold	Trivac D16B	11.7	0.000075	not rated	18.8		305
Rotary Vane	Agilent- Varian	DS402	12.3	0.0015	760000	22		350
Rotary Vane	Edwards	E2M18	12.1	0.015	76000	50		650
Dry Screw	Leybold	VD 65 HD/O2	38.3	0.0075	105067	30		1900

 Table 5: Manufacturer supplied pumping specifications including water capacity of commonly used vacuum pumps.



**Figure 13:** Pfeiffer Alcatel Adixen 2015SD rotary vane pump (**P102310**) with clean oil (left) and mildly dirty oil (right). On most rotary vane pumps, the oil condition can be determined by viewing it through a watch glass. As the oil degrades, its color darkens from clear, to yellow, to brown, to black. Its clarity decreases and its viscosity increases. Solid particles may be present. If other liquids mix with the oil an opaque emulsion may form with a matte appearance.



**Figure 14:** A sample variety of traps for protecting pumps from condensable vapors. From left to right; top: inline molecular sieve trap (**P103869**), zeolite pellets for hydrocarbon and oil removal (**P105408**), Ideal Vacuum inline liquid nitrogen cooled trap (**P103849**); bottom: right-angle water-cooled trap (**P103874**), Posi-Trap right-angle media trap with interchangeable media elements (**P1011050**).



# **Dirty Processes**

A dirty process involves or produces **dust** or one or more substances that belong in the family of gels, slimes, greases, and similar (hereafter called "**scum**") in the normal temperature range of the process. A dirty process is not necessarily chemical or wet, but many dirty processes are. Many vacuum pumps cannot tolerate the presence of dust or scum and will break if used to pump directly on a process that generates these materials. The performance of most gauges will also degrade over time in dirty environments. It is important to select the correct type of pumps and gauges and to provide proper protections for them in order to handle the dirty process.

Caution should be used when performing a dirty process in a vacuum chamber that also has electrical components. If dust or scum builds up on electrical components, electrical failure or arcing may occur (see Figure 11, page 8).

Dirty processes are typically performed under rough vacuum. High vacuum is difficult to achieve, but can be reached with sufficiently high pumping speeds and regular and very thorough chamber and system component cleaning. Ultra-high vacuum is impossible to achieve once too much dust or scum has been introduced into the system.

## Dust

Dust is fine-to-coarse solid particulate matter. It may be made of soft material, such as cloth fibers, or hard material, such as silica sand or metal particles. Most vacuum pumps cannot tolerate dust and it causes a decrease in the performance of most vacuum gauges.

If a dirty process produces dust, it is important to protect the pump(s) by using filters, strainers, screens, and/or mechanical traps. Mechanical traps catch coarse dust particles under rough vacuum conditions and will even catch fine particles once the pressure has dropped below a certain level. Mechanical traps must be removed and cleaned periodically, or dust will accumulate until it either blocks the flow of gas or begins to escape into the pump. Mechanical traps slightly reduce pumping speeds relative to a straight plumbed line. Mechanical traps can be equipped with filter media such as steel wool or mesh, activated carbon, sodium bicarbonate, copper wool, or molecular sieves to increase their effectiveness at removing contaminants, albeit at the expense of reduced pumping speed.



**Figure 15:** A sample variety of filters and traps for protecting pumps from dust and scum. From left to right; top: VisiTrap media trap (**P102738**) with activated charcoal filter element (**P102753**), Inline trap (**P108368**) with steel wool trapping media (**P109302**); middle: Edwards ISO-100 splinter guard centering ring with fine screen (**P106349**), Agilent-Varian ISO-160 splinter guard centering ring with coarse screen (**P108918**); bottom: right-angle trap with interchangeable filter media (**P107579**), activated alumina filter media (**P101869**), polyester filter media (**P101867**).

**Table 5**, on the following page, lists some common filter media types and their best applications as well as some of their limitations that could lead to incorrect application.

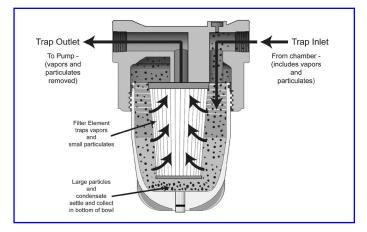
Filters remove particles down to the size listed on the filter and can completely protect vacuum pumps from dust particles if sized correctly. Filters greatly reduce pumping speed and finer filters cause a greater reduction than coarse ones. Soiled filters reduce pumping speeds more than new or clean filters. It can be beneficial to use a combination of a mechanical trap and a filter or multiple filters of increasing fineness to prevent excessive dust buildup on a single filter.



# Media Types and Applications

Media Material	Compounds Removed	Compounds Not Removed	Target Application	Incorrect Application	
Polyester Cloth Filter	Particulate matter with particle size greater than the cloth mesh rating, oil mist	Most chemical vapors	Removing dry dust from a dirty process	Removing large amounts of oil mist or scum from a dirty process. These filters clog easily	
Stainless Steel or Copper Mesh	Coarse particulate matter and debris, oil mist	Fine particulate matter, most chemical vapors	Preventing oil mist back streaming from a wet pump while protecting the pump from coarse particulates	Removing fine particulates or handling harsh chemicals. These filters have a very coarse mesh and can corrode.	
Sodium Bicarbonate or Soda Lime	Acidic and basic vapors	All other compounds, particulates	Protecting a pump from acidic or basic vapors	Protecting a pump from a strong oxidizer that is not acidic or basic, such as ozone.	
Activated Carbon or Charcoal	Organic vapors	rganic vapors All other compounds, Deodorizing pun particulates		Removing large quantities of organic vapors. Activated carbon has a limited adsorption capacity. For larger quantities, use a cold trap.	
Molecular Sieves	Water vapor, oil mist, some organic vapors	Most organic and corrosive vapors, particulates	Preventing oil mist back streaming from a wet pump while protecting pump oil from water contamination.	Removing large amounts of water. Molecular sieves have a limited adsorption capacity. For larger quantities, use a cold trap.	

 Table 6: Some common filter media types, their target applications, and incorrect applications.



**Figure 16:** Diagram of media trap principal of operation as used in the VisiTrap media trap (**P102738**). Right-angle and coaxial inline traps operate under a similar principal. Cold traps often lack filter media but instead have geometry that forces a minimum number of surface strikes before a gas molecule can escape the trap.

Vacuum gauges are less susceptible to fouling by dust than vacuum pumps because there is not a rush of dust-carrying air towards the gauges during initial pump down. However, in very dusty processes it may be beneficial to also protect a gauge with a P-trap or a filter. Because gauges do not require a high flow rate, it is more tolerable for a gauge filter to become dirty than a pump filter. Some gauges can be cleaned. Cleaning instructions are unique to each gauge and are available in the gauge's manual if applicable.

#### Scum

Scum is a diverse and difficult family of compounds to classify. Scum is usually a viscous liquid, gel, or flexible solid that evaporates, splatters, or deposits onto the interior surfaces of the vacuum system. Most types of scum generate a vapor pressure, so the presence of scum makes it very difficult to reach high



vacuum and impossible to reach ultra-high vacuum. Scum is generally bad for vacuum pumps and will gum up their mechanisms leading to reduced performance and ultimate failure. Scum is harmful to all types of vacuum gauges but is lethal to thin, filament style gauges such as Convectron, Pirani, and hot cathode. Thicker filament gauges such as thermocouple and cold cathode gauges are more resistant to scum. Diaphragm style gauges such as piezo gauges, capacitance manometers, and Bourdon gauges are most resistant but performance will still degrade over time. Filters are not recommended for scum protection as they clog quickly.

Scum can decompose into a solid black carbon residue upon contact with a hot surface in vacuum. A common build up area is on electrical connections and wires. This can eventually lead to electrical arcing or shorting (see Figure 11, page 8).

There are three primary sources of scum: splattering, evaporation of a high boiling point compound from a hot process, and chemical change of evaporated compounds.

Splattering occurs when a compound rapidly evaporates or boils under vacuum. If the compound is part of a mixture with less volatile components, droplets of the splattered compound may leave behind a scummy residue. A simple P-trap or rightangle bend is usually sufficient to protect a pump or gauge from splattered droplets.

Some compounds, such as heavy greases, have high boiling points that prevent them from significant evaporation at normal temperatures, even under vacuum. However, a compound's vapor pressure increases non-linearly with temperature according to the Clausius-Clapeyron relation.

When normally non-volatile compounds are heated under vacuum, they can evaporate. If the resulting hot vapors strike a cool surface, the compound will condense, leaving a scummy residue. For many processes, a simple mechanical trap at room temperature will force enough vapor collisions to occur for full deposition within the trap, especially if it is equipped with copper or steel wool. If a room temperature trap proves insufficient, adding a cold trap provides even more protection. Some chemical vapors may polymerize or react with the chamber walls, creating a scummy residue. This is especially common in processes that involve monomers, liquid resins, or hot plastics such as injection molding, polymerization, resin degassing, and plastic annealing. The chemical change that generates scum is often random and chemical vapors may collide with many chamber surfaces before finally depositing as scum. Here, a mechanical trap is inadequate and one or more cold traps below the freezing point of the chemical vapor are needed to protect the vacuum pump(s). Like filters and mechanical traps, cold traps have a finite collection capacity and must be periodically cleaned or thermally regenerated.

# **Pump Selection for Dirty Processes**

Not all vacuum pumps are equally tolerant of dirty processes. It is good practice to select a pump that can handle at least some amount of expected contaminants even when proper equipment is installed to protect the pump from the process. Subjecting any pump to a dirty process will require more frequent oil changes, seal replacements, and service than listed in the pump manual.

Wet pumps, such as rotary vane, rotary piston, and diffusion pumps are more tolerant of dust and scum than dry pumps. Dust and chemical impurities become suspended in the wet pump's oil and do not make frequent contact with sealing surfaces. Consequently, a wet pump's longevity and performance is not as diminished as with a dry pump. However, as contaminants accumulate in the oil, it loses its ability to protect the pump's mechanical components. It is important to change the oil regularly. Adding an external oil reservoir or an oil pump and filter system can increase oil lifespan and service intervals.

Wet pumps often generate a backstream of oil mist into the vacuum system. To prevent systemic oil contamination, a trap or baffle can be installed at the pump inlet which collects the oil mist and allows it to drip back into the pump. A similar style trap can be installed at the pump outlet to prevent oil mist from streaming into the room or exhaust lines. If the recycled oil remains uncontaminated, this can lengthen the time between oil changes.



Dry pumps are less tolerant of dust and scum than wet pumps. Even though some dry pumps have models that are listed as "chemical" or "corrosive" tolerant, these models are not designed for use with dust or scum. For rough and medium vacuum, dry roots pumps are somewhat more tolerant of dust and scum than scroll pumps due to their noncontact pumping action. Scroll pumps will see a rapid decrease in performance when exposed to scum but will be able to continue operating for some time. Scroll pumps cannot tolerate any dust or grit as it will quickly destroy their tip seals.

For high vacuum, turbo pumps can tolerate some scum but will experience a reduction in performance. Turbo pumps, which spin at tens of thousands of rpm, cannot tolerate any particulate matter. It will destroy the turbo blades. Turbo pumps are often oriented with their inlets facing down and fitted with inlet screens (splinter guards) to avoid having dust or larger particles fall onto their blades. Some turbo pumps cannot be oriented upside down, so it is even more important with these pumps to protect them from ingesting process dust and debris.



**Figure 17:** Edwards nEXT930D turbo pump (**P1012398**) fitted with an ISO-K 200 centering ring with fine inlet screen (**P1012402**). This is an example of a turbo pump which cannot be oriented with the inlet facing down.

Cryogenic pumps have no external moving parts and can tolerate a significant amount of dust before their performance is degraded. But, they cannot handle scum because it binds to their adsorbers, permanently reducing their gas absorption capacity. To protect cryopumps from scum, a refrigerated, chevroned baffle can be placed at the pump inlet which will block and freeze any scum molecules before they reach the cryopump adsorber.

lon pumps cannot handle scum. While dust will not directly harm an ion pump because it has no moving parts, ion pumps should only be used in a dusty process after careful consideration.



*Figure 18:* From left to right; Edwards Brooks CTI Cryo-Torr 10 cryopump (*P109174*), and an Agilent VacIon 75 StarCell ion pump (*P105778*).



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