

General Operating Guidelines for Wine Pre and Final Filter Cartridges

Introduction

Optimal filtration flow rate, selection of cost saving pre-filtration and installation of membrane filters validated for removal of wine spoilage organisms are all critical factors for successful cold microbiological stabilization of wine prior to bottling. Proper rinsing, sanitization and storage are however just as critical to ensure complete system hygiene.

The purpose of this document is to provide wineries with a set of operating guidelines to ensure the best possible hygiene, the most efficient cartridge performance and a reduced risk of contamination.

Filter Housing Installation and Preparation

The filter housing should be positioned for forward flow as indicated by the arrows on the outside of the assembly. A typical filter set up is shown in Figure 4 with our recommended ancillary equipment. The installed filter should be vertically positioned with the open end of the filter pointing down to ensure complete drainage. The vent valve, V7 is required to purge trapped air from the filter assembly at the start of filtration, for vacuum breaking and for connection to an Integrity Test instrument.

Ensure that the housing tube sheet and seals are clean and free from any surface damage or dirt. If the filter will be installed in a new system where in-line welding work has been conducted, it is recommended that the filter assembly and associated pipe work be thoroughly degreased and washed to remove all debris before the filter cartridge is installed.

Cartridge Installation

New and unused filter cartridges should be stored in a clean, dry environment, preferably in the box as delivered. Do not remove the cartridges from their plastic bag until they are required for use.

Prior to installation, verify that the filter part number corresponds to the production requirement. At the o-ring seal end of the filter, cut open the plastic bag with scissors. Do not use a sharp knife or box cutter and cut along the length of the bag as this can cause damage to the filters (Figure 1).

Do not touch the o-rings and leave the bag on the cartridge to facilitate handling. An effective o-ring seal is essential for optimal filter performance. For ease of installation, lubricate the cartridge o-rings by dipping this end of the filter in clean water immediately prior to fitting them into the tube sheet. To install the cartridge, hold the plastic bag around the cartridge near the o-ring seal end and push the filter into the tube sheet until the o-rings are no longer visible. Code 7 cartridges must be turned clockwise until the locking tabs on the cartridge are firmly locked under the bayonet tabs. Remove the bag after securing the filter (Figure 2).

Caution: Low boiling point (volatile) liquids such as ethyl alcohol should not be used as the o-ring lubricant. If the assembly is steam sterilized or exposed to sanitization temperatures, the vapor pressure of these liquids can result in localized high pressures between the o-ring seals and may dislodge them. As wine is a dilute solution of ethyl alcohol, it should not be used for lubrication. Silicone grease is also not recommended, as it can cause the o-rings to swell at elevated temperatures. Additionally, if grease gets on the filter surface it can result in premature plugging or localized hydrophobic spots.

Figure 1 – Cartridge Handling



Figures 2 – Code 7 Adaptor



Figure 3 - Correct cartridge installation



Sanitization and Sterilization

Filters installed directly upstream of the wine bottling line should be sanitized or sterilized prior to each use. The following recommendations apply to the sanitization and sterilization of the filter assembly only. Pall's recommended final filters are compatible with most common winery sanitization and sterilization methods, however, to confirm compatibility or for advice on installation of downstream equipment, please consult your local Pall representative.

Before sanitization or sterilization, the filters should be properly rinsed and cleaned. The water used to flush the filters should be 1) potable and 2) filtered to remove particles that could foul the filter. The water final filter should be rated at one micron ($\geq 1000 \beta_{1\mu m}$) or tighter. Similarly, steam used for sanitization should be filtered to remove particles like rust and scale. The steam used for sterilization must be dry and saturated. Super heated steam must not be used.

Filter life will be improved if the assembly is regularly washed with cold followed by warm water at 50 ± 5 °C (113-131 °F) after each production cycle. This procedure removes the soluble polysaccharides, sugars and proteins trapped by the filter. This is an effective regeneration procedure and will help to increase total product throughput. If the filters are not cleaned to remove organic residues, high temperature exposure during sanitization or sterilization can cause the organic material to irreversibly adhere to the membrane surface and prematurely plug the filter.

Pressure gauges (P1 and P2) should be installed as shown in Figure 4 to monitor pressure during both process and sanitizing operations. Monitor the temperature at T2 during cleaning and sanitization or sterilization to ensure that the proper temperature is maintained.

Procedure I: Rinsing and Cleaning

Start with all valves closed. Refer to Figure 4.

1. Drain the filter assembly by opening V7, V6 and V8
2. Close all valves except vent valve, V7, which should be fully open and V6 which should be partially open.
3. Open water inlet valve V2 and rinse the filter assembly with ambient temperature water. When the filter housing is full and free of air, (steady stream of water, no bubbles from V7), open V8, V9 and V10 and partially close V7 to continue flushing the top of the filter module. If a steady stream of water is not maintained through V7, increase the water supply flow rate or partially close V6, V8, V9 and V10.
4. Continue in this mode until the effluent appears free of product.
5. Adjust the inlet water temperature to $50 \pm 5 \text{ }^\circ\text{C}$ ($113 - 131 \text{ }^\circ\text{F}$) and continue to forward flush the filter until the effluent is clear.

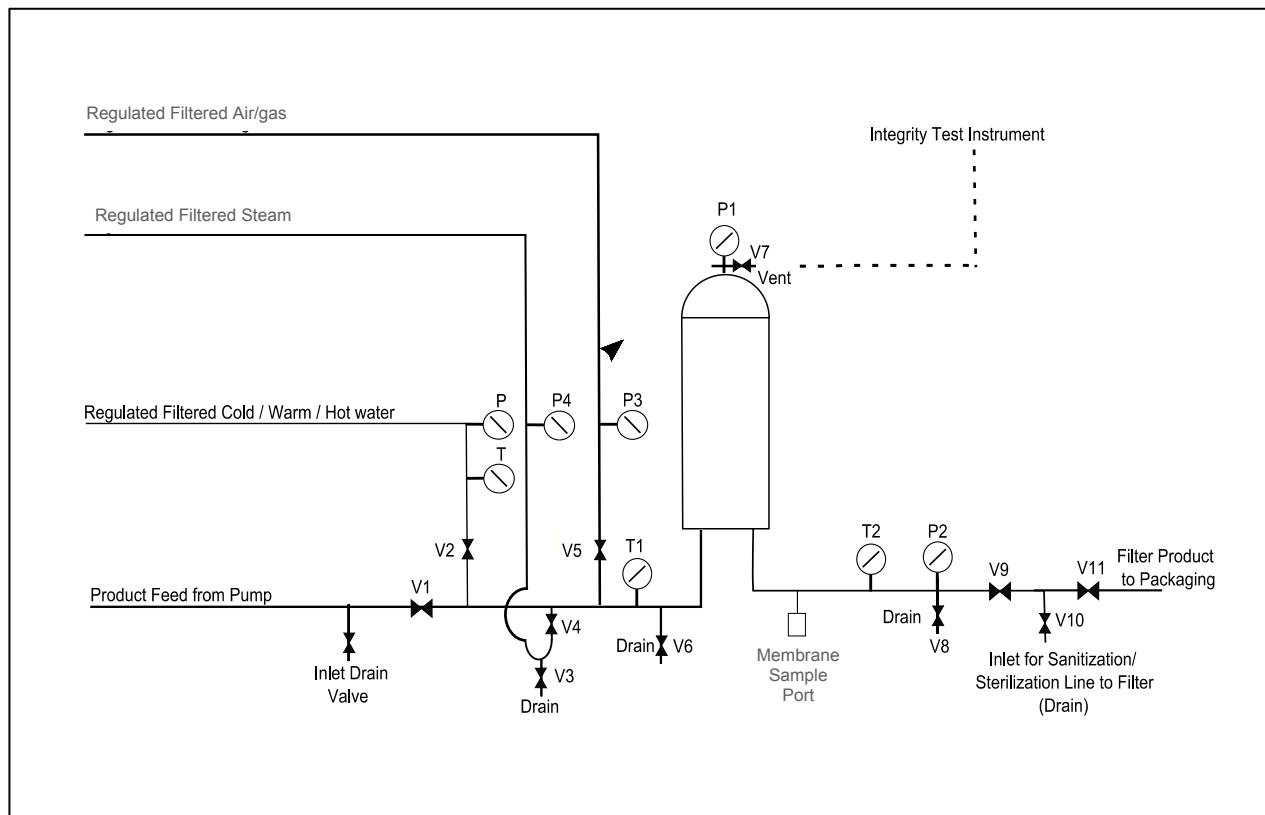


Figure 4 - Schematic Layout for Steam Sterilization or Hot Water Sanitization of a Single Stage Filter System

Note:

1. The rinse water flow rate should be greater than or equal to the process flow rate. Low flow velocity during cleaning may result in insufficient or partial filter cleaning.
2. The housing vent connection needs to be large enough to ensure adequate flow reaches the entire length of the filter cartridge. This is especially important when using 3 or 4 high cartridges.
3. When switching from ambient to warm water, this should be done slowly to avoid large and abrupt temperature changes ($\max \Delta T = 40 \text{ }^\circ\text{C}$ or $72 \text{ }^\circ\text{F}$). The same is true for the reverse, cooling.
4. Backwashing of the final filter cartridges is not recommended. Generally this procedure provides no additional benefit and can be detrimental to cartridge integrity.

5. Pall does offer specific pre-filter cartridges that were designed for regeneration using reverse flow cleaning procedures. For specific information and recommendations for use with these pre-filters, please contact your Pall representative.

In the case of a two or three stage filter system, the recommendations are similar to the aforementioned single stage recommendations; however, the valve arrangement should be configured to include a valve in between the filter assemblies since each assembly must be flushed independently. This is critical as pre-filter washings must not be forwarded onto the finer downstream filter membranes.

Procedure II: Hot Water Sanitization

After rinsing with cold and warm water, the filter assembly may be sanitized by increasing the water temperature to 80 °C (176 °F) at T2, for the validated sanitization temperature and time. See Note 3 above. The flow rate is less critical here; time and temperature are the limiting factors. Maintain a steady flow from the vent valve V7 (to ensure similar temperature as T2), drain valves V6, V8, V9 and V10 but with the valve to the filler, V11 closed. The product outlet line downstream of V9 should be sanitized separately, through valves V10 and V11.

After hot water sanitization, the filter assembly must be cooled prior to integrity testing and wine filtration. If wine filtration is scheduled immediately thereafter, cool the assembly by slowly decreasing the rinse water temperature to ambient remembering that large and abrupt changes in temperature can have a negative impact on the filter life. See Note 3 above. When the system is at room temperature close all the valves, in sequence starting from V10 to V2.

If wine filtration is not scheduled until the start of the following day, the filter assembly should be allowed to cool overnight so that integrity testing and filtration can be performed at the start of the next day. At the end of the sanitization cycle close all the valves, in sequence starting from V10 to V2 to keep the system under positive pressure. For added safety, introduce air or nitrogen into the system by opening V7.

Procedure III: *In-Situ* Steam Sterilization

As an alternative to hot water sanitization, steam can be used to sterilize the filter assembly.

During steaming, the differential pressure (ΔP), (P1 - P2) across the filter assembly should not exceed 0.3 bar g (4.3 psi g), the downstream pressure (P2) should not be less than 1 bar g (14.5 psi g) and the temperature not less than 110 °C (230 °F) to achieve sterilization. Rapid changes in ΔP may damage the filter.

Steam sterilization is recommended after the installation of new equipment, new cartridges, after a long shutdown and preferably within two hours of the filtration. Pall filters are not supplied pre-sterilized.

Steam sterilization procedures must be strictly adhered to. Use only dry saturated steam regulated (P4) to 1.3 bar g (19 psi g) maximum.

Start with all Valves Closed. Refer to Figure 4.

1. Turn on the steam and blow down condensate from the steam line through condensate drain valve V3. When all condensate has been drained partially close V3.
2. Fully open V7 and open V6 halfway.
3. Slowly open steam valve V4. When steam is flowing continuously from vent valve V7, crack open drain valve V8, valves V9 and V10. Leave V11 closed.
4. When steam flows continuously from valves V8 and V10, partially close vent valve V7. Balance V7 against V8 and V9 to achieve 1 barg (14.5 psi g) at P2 with no more than 0.3 bar d (4.3 psi d) ΔP across the filter.
5. Steam in this mode for the validated sterilization temperature and time.
6. Preset the pressure (P3) of regulated air or nitrogen at 200 mbar (2.9 psi) above the steam pressure (P4).

7. Close drain valves V10, V8, V6 and V3 and vent valve V7.
8. Immediately introduce pre-regulated air or nitrogen through valve V5.
9. To assist cooling, steam may be flushed from the assembly by carefully opening vent valve V7 and drain valve V6.
10. Allow the assembly to cool to ambient temperature.
11. Close valve V7 and V6 after flushing
12. Close the air or nitrogen valve V5, unless wine filtration is not scheduled until the start of the following day

In the case of a two or three stage filter system, the recommendations are similar to the aforementioned single stage recommendations.

Procedure IV: Chemical Sanitization

If hot water and steam are unavailable or limited in supply, wineries may use chemical methods for sanitization. Additionally, wineries may supplement their standard sanitization protocols by adding a chemical step once a week. Please consult your Pall representative regarding compatibility of the filter materials, gaskets and O-rings, prior to using any chemical agent. Following chemical sanitization, the filters must be thoroughly flushed with cold filtered water, and integrity tested in situ prior to wine filtration.

Note: As filters become fouled, the concentration of the chemical sanitizer may need to be increased as the sanitizer may be exhausted by contaminants on the filter. If chemical sanitization is used, there should be a measurement device installed downstream of the filter assembly to ensure effective sanitization.

Procedure V: Filter Storage

Prior to filter storage, the cartridges should be thoroughly rinsed, cleaned and sanitized or sterilized. If the filters are not properly cleaned, the efficacy of the storage chemicals at the suggested concentrations may be at risk. See note above. Filter cartridges out of service for a few days can be stored in a solution of 50 - 65 ppm peracetic acid (PAA)*. If the filter cartridges are to be stored for weeks or months, they can also be stored in a solution of 0.5% citric acid with 500 to 1000 ppm of potassium metabisulfite. In either case, prior to reuse, rinse the cartridges with water to remove all traces of storage solution, sanitize or sterilize and integrity test before use.

Alternatively, for long term storage the filters can be stored in the housing assembly under pressure (1 bar, 14.5 psi) with sterile filtered nitrogen. Prior to applying the nitrogen, the assembly should be cleaned, sanitized or sterilized, drained and sealed.

*The values listed refer to the concentration of PAA only, not to the total peroxides concentration.

Procedure VI: Integrity Testing

To ensure integrity of the installed filter, it is essential that the filter assembly is integrity tested before wine filtration. This will also confirm that:

- There is no mechanical damage to the filters and the membrane is intact.
- There are no leaks from the assembly.
- The correct filter cartridges have been installed.

The typical integrity test methods used in wineries are based on the Forward Flow Test, using the Pressure Decay Test technique. While the Pressure Decay Test can be performed manually, to ensure consistent results and avoid operator variability, automated units like the Palltronic® Compact Star are recommended. The Compact Star allows for storage of up to 1000 test records and provides a hardcopy result for batch documentation or troubleshooting in the unlikely event of wine spoilage.

To run an automated integrity test, the Compact Star should be connected to the housing vent valve, V7 as shown in Figure 4. For further specific information or instruction on the Compact Star or other manual test methods, please consult your local Pall representative. Some general considerations when performing an integrity test include:

1. Integrity testing must be performed on cartridges thoroughly wet with water.
2. Do not use wine as the wetting fluid as wine varies by batch.
3. Air and nitrogen are acceptable test gases. Carbon dioxide and oxygen should not be used.
4. The temperature during an integrity test should not vary by more than 1 °C (1.8 °F). IT values are typically provided at 20 +/- 5 °C. If your test will be performed at temperature outside the above values, please consult Pall for the corrected values.

Conclusion

Membrane filters have been used successfully for many years to remove spoilage organisms and particulate from wine. While it is critical to select the proper cartridge filter for the given application, it is just as important to use standard documented operating procedures to ensure optimal filtration performance, maintain system hygiene and reduce the risk for contamination. This document provides guidelines which can be used as the basis for winery standard operating protocols. For additional information or winery specific recommendations, Pall wine specialists are available for consultation.

Disclaimer

These general filter operating guidelines can provide only generic recommendations. They do not substitute due diligence or replace instructions for use for PALL or other manufacturer's equipment. They do not consider or supersede all relevant legal requirements or safety instructions. Whether they are applicable in the individual case or if extra qualification work is to be done, this stays within the responsibility of the user.

References

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