Regeneration and Sanitisation of Brewery Filtration Systems

Technical Guidelines

Revision 3 (August 2019)

Contents

Introduction

Clean-in-place (CIP)

Regeneration: removal/reduction of blockage components to prolong a filter's service life

Sanitisation: removal/reduction of retained microorganisms to prevent microbial build-up

This document provides guidelines that are intended for incorporation into standard operating procedures. The likelihood of premature blockage, integrity test failure and process stoppages will be reduced by following the guidelines in this brochure. Recommendations should be considered flexible and continuous monitoring carried out to improve the process as ongoing experience is gained.

The procedures described in this document are suitable for use with polypropylene media pre-filters (e.g. PREPOR PP/NG) and polyethersulphone membrane final filters (e.g. BEVPOR). It is not recommended to back-flush filters unless they have been specially designed for this (e.g. PEPLYN HA/HD, PREPOR NG).

A two-stage filtration system (Figure 1) is used as an example throughout the document.

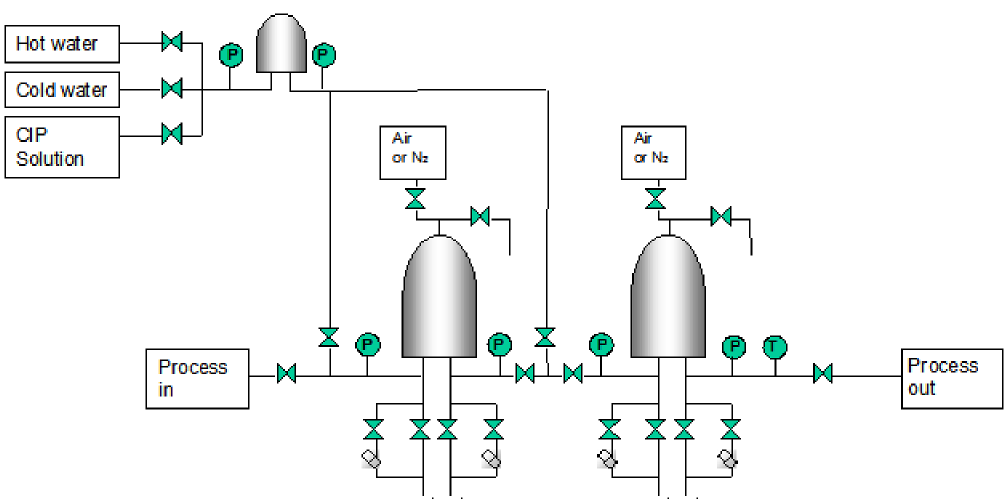

Figure 1: System set-up

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The following 4 guidelines are key to a good CIP practice:

Effective cleaning can greatly increase filter lifetime.

'Little and often' is better than an occasional strong clean.

Sanitisation is important to maintain product quality.

Inappropriate cleaning regimes can damage filters.

CIP should be performed when the differential pressure across the filter rises to ~100-200 mbar above the starting differential pressure (Figure 2a), rather than once the filter has fully blocked (Figure 2B).

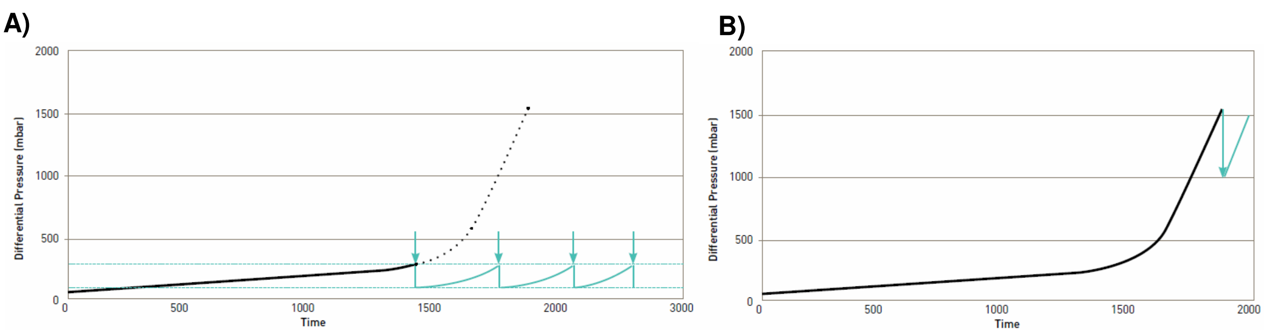

Figure 2: Regular cleaning (A) compared to occasional strong cleaning (B).

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Compatibility

Before using any chemicals, check the compatibility of the filter prior to use. If in doubt, contact Parker TSG (Technical Support Group). The guidelines listed below offer general advice on compatibility. The testing was conducted in a laboratory environment so may not fully simulate damage caused to the filter by process operating conditions.

Table 1: Compatibility Guidelines

Active agent	Condition*	Parker domnick hunter Product	Guideline cumulative contact time
Hot water	85 °C	BEVPOR	1000 hours[1]
		PEPLYN	245 hours[1]
		GF, GP	100 hours[2]
Steam	121 °C	BEVPOR	25 hours[2]
		PEPLYN	100 hours[2]
		GF, GP	10 hours[2]
Caustic [NaOH] in a buffered sanitizer	0.352% NaOH [and <0.6% EDTA] @ 60°C	BEVPOR	336 hours[3]
	0.352% NaOH [and <0.6% EDTA]	BEVPOR, PEPLYN	>1000 hours[3]
		GF, GP	18 hours[2]
Peracetic Acid [PAA]	0.54% PAA [and <0.54% H_2O_2, <0.54% AA]	BEVPOR	>1000 hours[3]
	0.15% PAA [and <0.15% H_2O_2, 0.15% AA] @ 50°C	BEVPOR	240 hours[4]
	0.05% [500 ppm] PAA	GF, GP	168 hours[2]
Hydrogen Peroxide [H_2O_2]	0.15% H_2O_2 @ 50 °C	BEVPOR	600 hours[4]
	0.4% H_2O_2 [and <0.13% AA, <0.1% PAA]	BEVPOR	600 hours[4]
	0.2% H_2O_2 [and <0.065% AA, <0.1% PAA] @ 50 °C	BEVPOR	240 hours[4]
Phospheric [H_3PO_4] / Nitric [HNO_3] Acid	1% HNO_3 and 1% H_3PO_4 @ 55 °C	BEVPOR	256 hours[5]
Sulphur Dioxide [SO_2]	2% SO_2	BEVPOR	168 hours[2]
	0.5% SO_2	BEVPOR, PEPLYN, GF, GP	756 hours[2]
Chlorine Dioxide [ClO_2] Sodium Hypochlorite	10ppm free Cl	BEVPOR, PEPLYN	336 hours[9]
	1700ppm total Cl	BEVPOR	>1000 hours[10]
Chlorinated Alkaline	<500ppm Chlorine [<0.15% KOH] @ 50°C	BEVPOR	72 hours [at least][8]

*All conditions at ambient [25 °C] unless otherwise stated.

References: 1 = T6562, 2 = Beverage Cleaning Guidelines [2003], 3 = T9122, 4 = T9123, 5 = T8716, 7 = T9146, 8 = T8983, 9 = T7439, 10 = VSG9256/ TR13341.

Notes:
The guidelines are based on tests with chemicals from commonly used industry suppliers and the concentrations are based upon the information in the TDS / MSDS.

Caustic Compatibility Case Study

Background

A brewery was using Parker PEPLYN PLUS and BEVPOR filters to clarify and stabilise beer prior to bottling. The filters were part of a bottling line which was cleaned using alternating acid and caustic CIP cleans. The filters were removed from the bottling line whilst it was being cleaned to avoid chemical damage due to incompatibility. The filters were then sterilised by autoclave.

Problem

Removing and then re-installing the filters when cleaning the bottling line resulted in increased down time for the brewery.

Resolution

Working closely with the brewery, Parker Biosciences TSG planned and conducted a CIP compatibility trial. During the trial, filters were exposed to a CIP cycle using a commercially available acidic detergent (containing a mixture of nitric and phosphoric acid) at 1% concentration and a temperature of 60 °C, alternated with a CIP cycle using a caustic detergent at 1 % concentration and a temperature of 60 °C (equivalent NaOH concentration of 0.4 %).

These cleaning conditions allowed the filters to be cleaned in situ, eliminating the time previously used to remove, autoclave and re-install them. The process parameters and integrity test values were recorded by the brewery team and monitored by TSG. After a period of ~6 months and 49 CIP cycles the filters were still integral, this proved that the filters were compatible with the in-situ cleaning regimen, saving time and cost for the brewery.

Rogues Gallery

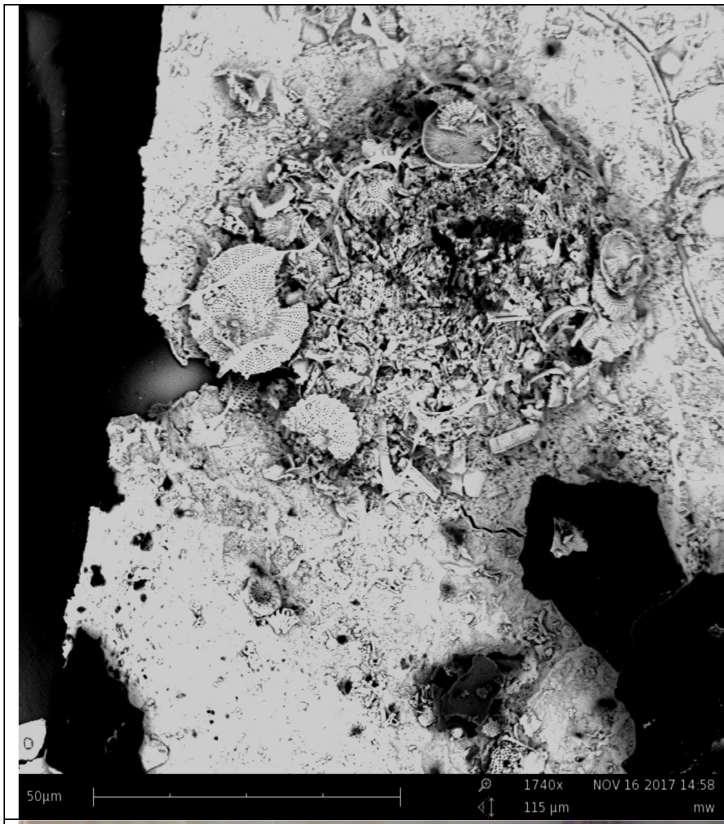

Figure 3: Diatomaceous Earth (D.E)/Kieselguhr.

D.E bleed through from upstream processing of the beer can build up on the filter surface causing blockage.

This SEM image shows DE forming part of a filter cake on the surface of a membrane.

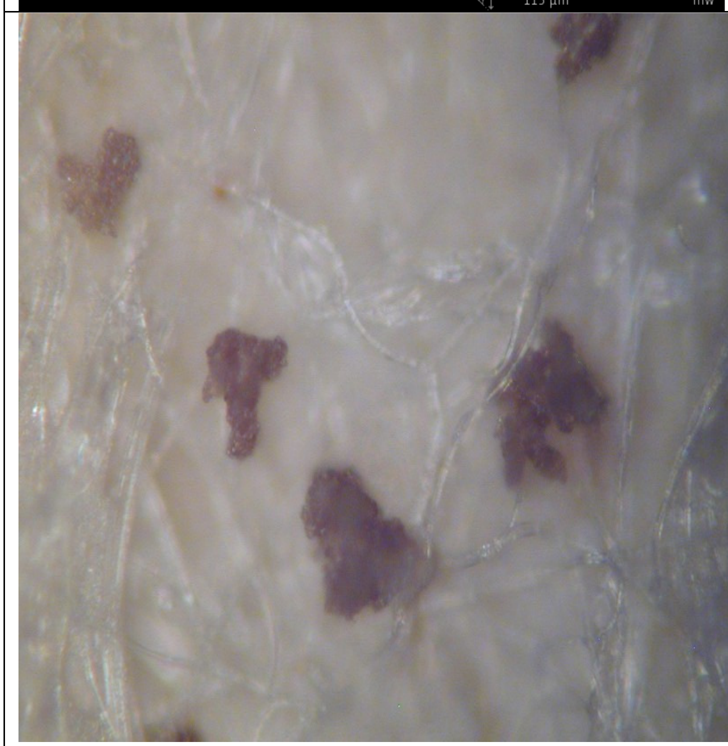

Figure 4: Polyvinylpolypyrrolidine (PVPP)

PVPP is used as an absorbent to remove certain undesirable polyphenols from beer.

During normal beer processing it should be removed prior to coming into contact with the cartridge filters, however in situations where upstream processing of the beer (trap filtration etc.) do not function correctly PVPP can be deposited onto cartridge filters causing blockage.

This image from a light microscope shows PVPP deposited on pre-filter media.

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Figure 5: Beer Stone

build-up of calcium oxalate and rotein normally within tanks and menters. Prevention is better than atment, alternating cleaning using stic and acidic CIP cycles can help prevent heavy build up.

is SEM images shows beer stone l up on the surface of a media-pre-filter.

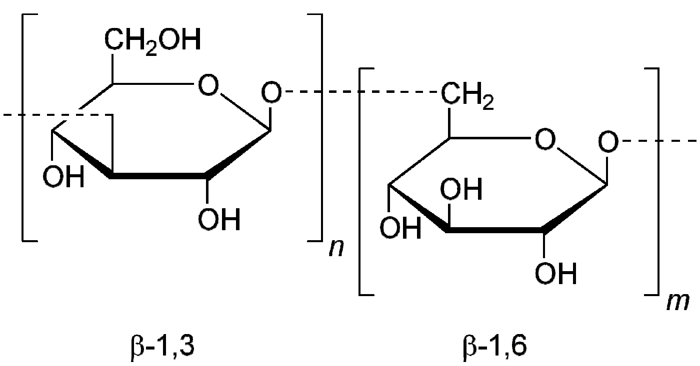


Figure 6: Structural Formula of a repeat unit of β Glucan Polysaccharide

Beta glucans are polysaccharide molecules released from the cell walls of malt and barley. Beta glucans make beer more viscous and also contribute to filter fouling. The enzyme beta glucanase catalyses the breakdown of beta glucans and as such adding it to beer can increase its filterability. Beta glucanase can also be used as a cleaning agent for filters fouled with high beta glucan containing beer.

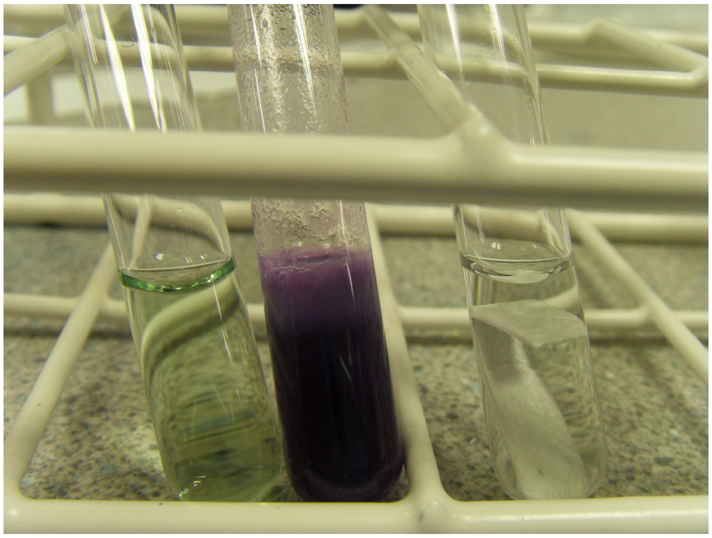


Figure 7: BCA Test for Protein

Various proteins are commonly found in beer. Proteins can interact with other components of the beer such as polyphenols to cause haze. Proteins can also interact with carbohydrates present in the beer to form complexes which can foul filter membranes.

This figure shows a BCA test carried out on a filter media sample from a used beer filter. From left to right; Unused BCA reagent, BCA reagent in the presence of filter media containing protein, unused clean filter media.

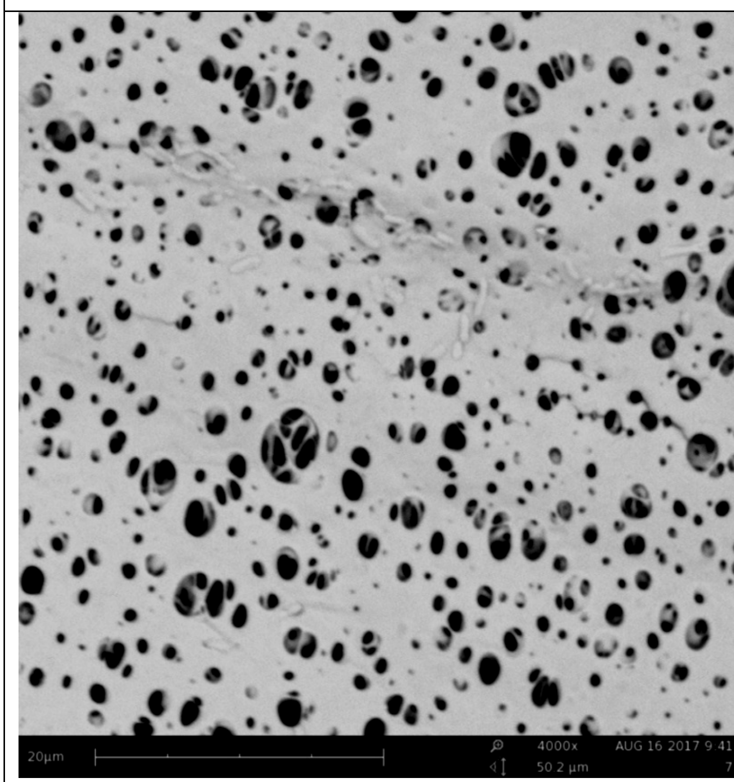


Figure 8: Bacteria

Microfiltration is effective in the removal of spoilage bacteria from beer. In certain cases, if the filters are not regularly and effectively cleaned/sterilised bacteria can build up on the surface and in the pores of membrane filters, this causes the filters to foul. In some cases, bacteria can from biofilms, in these cases groups of bacteria produce a polymeric extra cellular matrix with a consistency similar to slime, this can result in filter fouling.

This SEM images shows bacteria building up on the surface of a filter membrane.

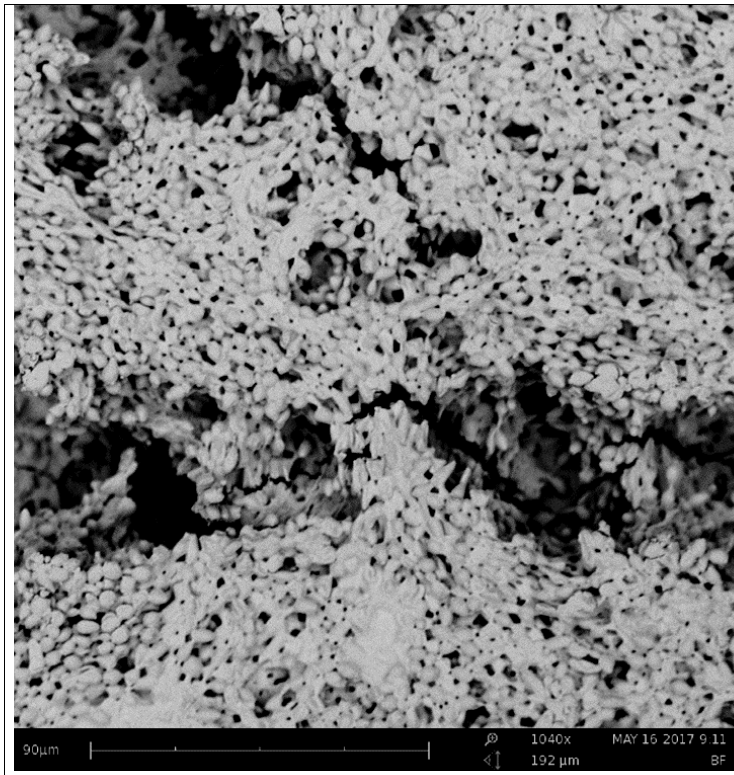


Figure 9: Yeast

Microfiltration removes yeast from the beer, however in cases where downstream processing of the beer has not been effective and it has a very high yeast content, it can build up on the surface of the membrane and cause fouling. Yeast can also propagate on the surface of filters if they are not fully sterilised.

This SEM image shows a large amount of yeast built up on a filter surface causing blockage.

Choosing a CIP solution

The chosen CIP strategy must be related to the contaminants that require removal (Figure 10).

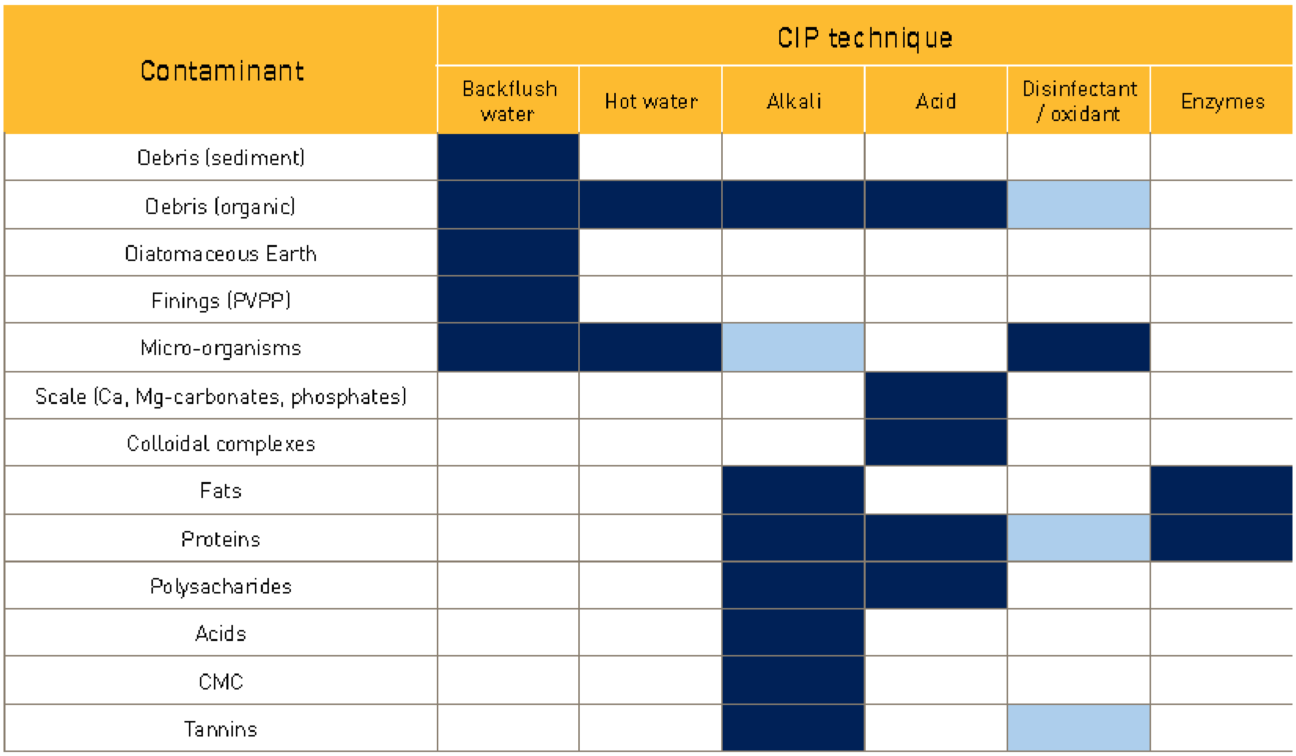


Figure 10: Matrix showing suitable CIP solutions for particular contaminants.

Stages of a CIP regime

There are various CIP options to consider in the brewing industry (Table 2). In addition, it is recommended that the filter lines, lines and tanks are cleaned at least weekly.

Table 2: CIP options for beer filters.

Filters	Cleaning type	When	How	Recommended conditions			
				Concentration (%)	Temperature (°C)	Duration (mins)	Minimum flow (% of process flow rate)
Back-flush-able pre-filters (e.g. PEPLYN HA/ HD) used as 'trap' filters	Flush / Chase	Change between beers or pre-CIP	Water flush		Ambient	5 (or until clear)	20
	Regeneration	Daily / weekly or at signs of blockage	Hot water rinse		60-80	10	20
			Water back-flush		Ambient	5 (or until Δp is stable)	150- 200 x process flow*
			NaOH clean	0.3-1.0	Ambient/warm (max 60 °C)	20 (soak after 5 min)	20
Bottling line filtration train (e.g. PREPOR PP/NG, BEVPOR)	Flush / Chase	Change between beers or pre-CIP	Water flush		Ambient	5 (or until clear)	20
	Regeneration	Weekly or at signs of blockage	Hot water rinse		60-80	10	20
			NaOH clean	0.4%	Ambient/warm (max 60 °C)	20 (soak after 5 min)	20
	Sanitisation	Daily	SIP	See SIP guidelines			
			Hot water rinse		80-85	20	20
			Peracetic acid	0.05	Ambient	20 (soak after 5 min	20

* Flow rate should be as high as possible without exceeding the recommended maximum differential pressure of the filter (2 times the process flow rate is recommended, especially in trap filter applications).

Example regime – CIP of general line filters (non-back-flushable)

<table>
<tr><th colspan="3">Step 1: Water flush</th></tr>
<tr><td>Temperature: Ambient</td><td>Duration: 5 minutes (or until clear)</td><td>Flow rate: 10 L/min/10”</td></tr>
<tr><td colspan="3">Purpose:
• To remove residual beer from the system
• To remove soluble beer components – sugars, water-soluble proteins, polysaccharides</td></tr>
<tr><td colspan="3">Procedure:
• Isolate housings
• Drain or chase process liquid with cold water into the first housing
• Flush to drain until clear
• Open inlet to next housing and close drain
• Repeat for all housings in series
• Flush for the specified duration</td></tr>
</table>

<table>
<tr><th colspan="3">Step 2: Warm / hot water rinse</th></tr>
<tr><td>Temperature: 60-80 ºC</td><td>Duration: 10 minutes</td><td>Flow rate: 5-10 L/min/10”</td></tr>
<tr><td colspan="3">Purpose:
• To clear filters of product and water-soluble blocking materials which may not be removed by cold water
• Gradually increases the temperature in preparation for the NaOH regeneration</td></tr>
<tr><td colspan="3">Procedure:
• Isolate housings
• Gradually introduce warm/hot water into the first housing
• Flush to drain until clear
• Open inlet to next housing and close drain
• Repeat for all housings in series
• Flush for the specified duration</td></tr>
</table>

Step 3: NaOH (0.4%) regeneration		
Temperature: 60 ºC max	**Duration:** 20 minutes	**Flow rate:** 2-3 L/min/10"

Purpose:

- To provide more aggressive cleaning to remove water-insoluble beer components – trapped proteins, sugars, biofilm

Procedure:

- Isolate housings
- Introduce NaOH solution into the first housing
- Flush to drain until clear
- Open inlet to next housing and close drain
- Repeat for all housings in series
- Circulate or close valves and allow to soak (Figure 11) for the specified duration

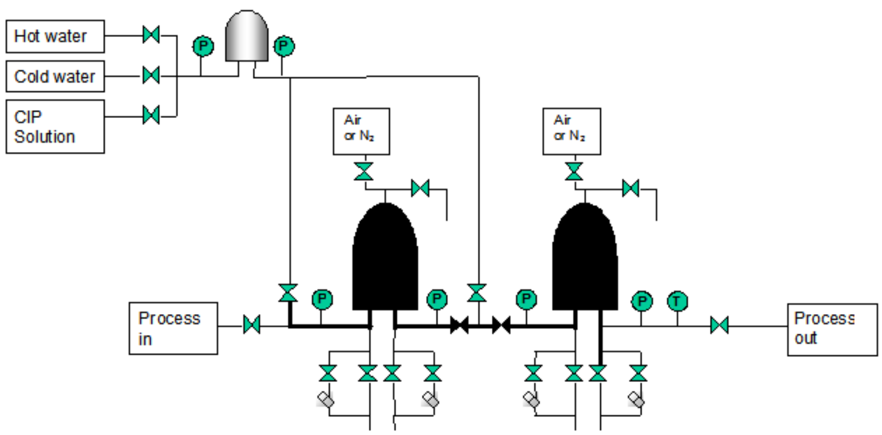


Figure 11: CIP-solution soak.

Step 4: Water flush		
Temperature: Ambient	**Duration:** 5 minutes	**Flow rate:** 10 L/min/10"

Purpose:

- To remove NaOH from the system prior to sanitisation; residual NaOH can cause filter damage.

Procedure:

- Isolate housings
- Drain or gradually chase NaOH solution with cold water into the first housing
- Flush to drain until clear
- Open inlet to next housing and close drain
- Repeat for all housings in series
- Flush for the specified duration

Step 5: Peracetic acid (0.05 %) sanitisation		
Temperature: Ambient	**Duration:** 20 minutes	**Flow rate:** 2-3 L/min/10”

Purpose:

- To sanitise the system by killing microorganisms
- Peracetic acid is an ideal anti-microbial agent due to its high oxidising potential

Procedure:

- Prepare fresh peracetic acid, as it dissociates over time so should not be reused
- Isolate housings
- Introduce peracetic acid solution into the first housing
- Flush to drain until clear
- Open inlet to next housing and close drain
- Repeat for all housings in series
- Flush for the specified duration

Step 6: Water flush		
Temperature: Ambient	**Duration:** 5 minutes	**Flow rate:** 10 L/min/10”

Purpose:

- To remove peracetic acid from the system
- To ensure filters are fully wetted prior to integrity testing

Procedure:

- Isolate housings
- Drain or chase peracetic acid with cold water into the first housing
- Flush to drain until clear
- Open inlet to next housing and close drain
- Repeat for all housings in series
- Flush for the specified duration

Additional considerations

Preparation and pre-filtration of CIP solutions

- Fresh CIP solutions should be prepared each time – recovered CIP solutions (including water) should not be re-used.
- Pre-filter all solutions (including water) using a filter with one grade of retention higher than the filter being cleaned – i.e. a 0.6 µm CIP pre-filter for cleaning a 0.45 µm filter, or a 3 µm CIP pre-filter for cleaning a 1 µm filter.
- For filters suitable for back-flushing, CIP pre-filters should be the same grade of retention as the filter being cleaned.
- Conductivity of CIP solutions should be monitored and recorded before and after cleaning to ensure maintenance of the correct concentration; chemical suppliers should advise the relationship between conductivity and concentration.

Running the CIP regime

- CIP should be performed in the forward direction unless stated otherwise.
- Temperature changes should be gradual – sudden shocks can cause filter damage.
- The initial rinse solution from each individual housing must always be flushed to drain, not directly onto the next filter (applies to all cleaning stages). When the rinse solution runs clear, the inlet to the next filter can be opened and the drain closed so the rinse solution flows to the next filter (Figure 12).

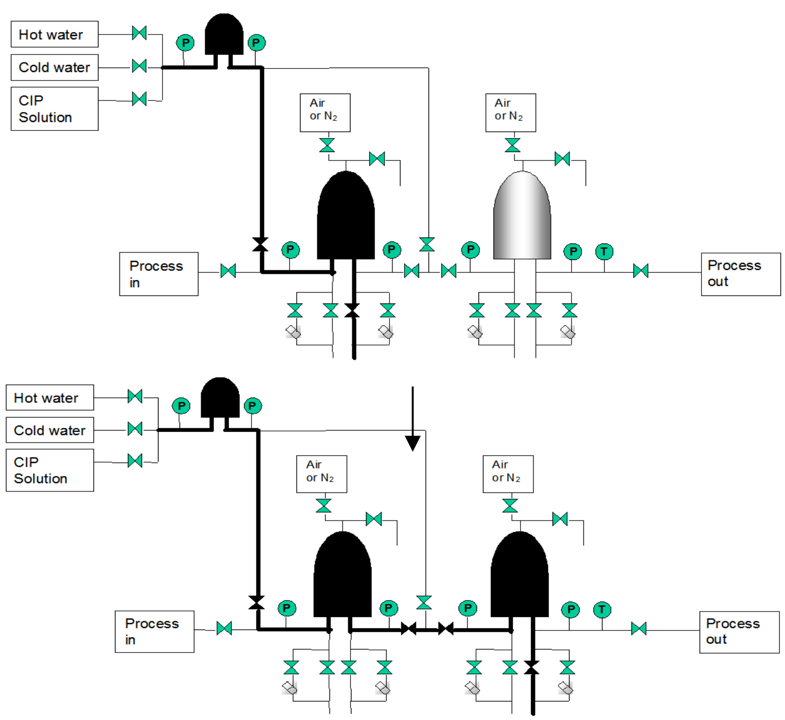


Figure 12: Rinsing of two filter stages in series.

- Alternatively, filter stages can be rinsed simultaneously, in parallel (Figure 13). This will reduce the overall cleaning time. Note: solution pre-filtration must be sufficient to protect the final filter.

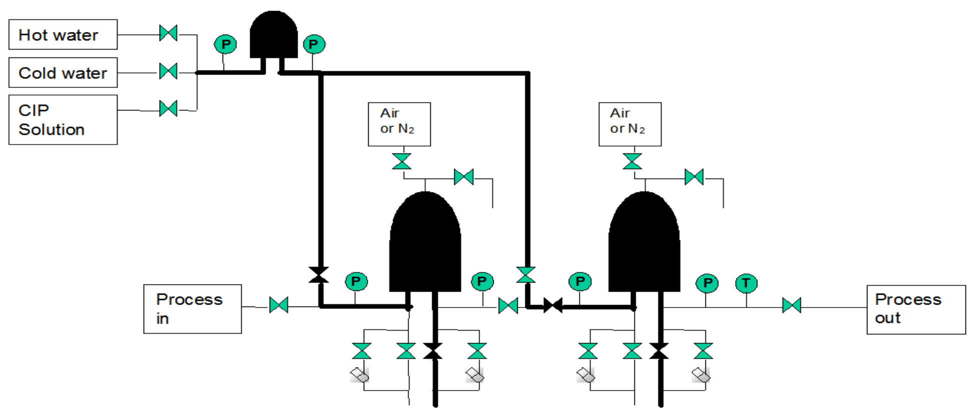


Figure 13: Rinsing of two filter stages in parallel.

- The temperature downstream of the filters should be monitored and recorded to ensure that the required CIP temperature is achieved.
- Integrity testing of the final filters should always be performed following CIP.

Back-washing

- Most efficient method for removing particulate material (Kieselguhr, PVPP, etc.) from trap filter systems.
- Can be performed using water alone or with the addition of cleaning chemicals if necessary.
- Only effective for pre-filtration stages; should not be performed on membrane filters
- Flow rate should be as high as possible without exceeding the recommended maximum differential pressure of the filter (2 times the process flow rate is recommended).
- Duration can be extended if the differential pressure has not decreased after back-washing.

Relationship between ΔP and blockage

The differential pressure at which a filter should be changed is dependent upon the system and the filter specification. Temperature, available pump capacity, filter type, minimum process flow, etc. all must be taken into account. Because of this, Parker do not have any formal document outlining specific circumstances.

However, we can recommend changing the filters when either:

- Flow is 5 – 10 x lower than clean flow
- ΔP is 5 – 10 x higher than clean ΔP
- Starvation to downstream processes occurs
- **ΔP has reached 2 bar**

Once 2 bar ΔP has been reached, very little throughput will be achieved if filter use is continued, as blockage is usually exponential, see figure 14 below.

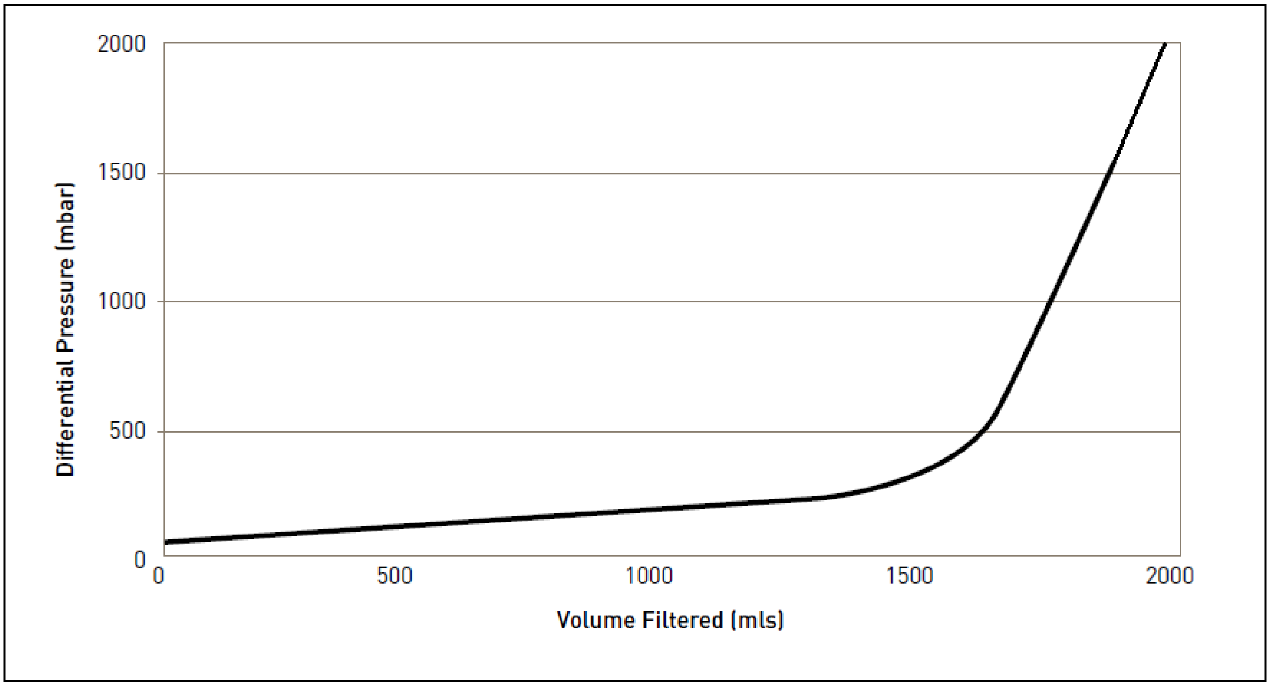


Figure 14: Typical blockage curve.

Storage

Short-term (< 1 month)	Long-term (> 1 month)
1. Sanitise the filters as described previously 2. Store the filters in an appropriate solution (Table 3) in the housing	1. Perform chemical regeneration, followed by sanitisation, as described previously 2. Remove the filters from their housing 3. Autoclave or oven-dry at 50-60ºC 4. Store in original packaging

For long term storage, if drying cannot be achieved, store in one of the following solutions:

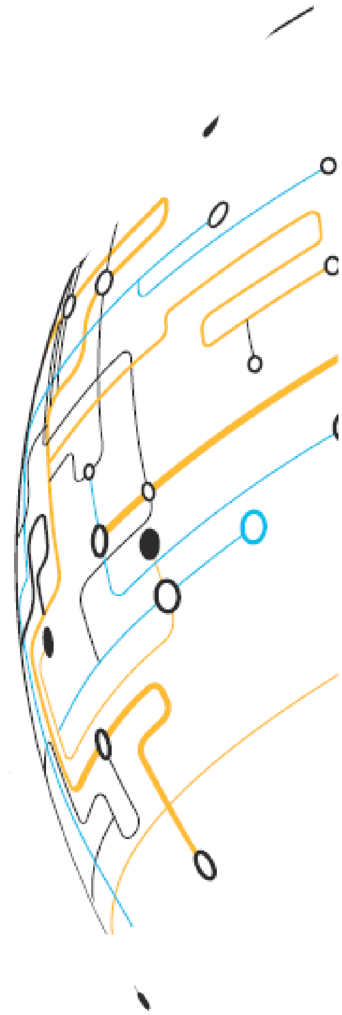
- 70% ethanol.
- 0.1 % citric acid / K2S2O5, refresh the solution every 20 days.
- 50 ppm hypochlorite, refresh solution weekly or when below 40 ppm (test kits available from cleaning solution manufacturer).
- Specified storage solutions by the cleaning solution manufacturer. The chemical solution should be changed regularly according to the manufacturer's instructions. Please contact Parker for compatibility information.

Table 3: Storage solutions.

Solution	Concentration	Comments
Peroxyacetic acid	100 ppm	Up to 24 hours
Peroxyacetic acid	500 - 2000 ppm	1-4 days
Hydrogen peroxide	500 - 2000 ppm	4 days - 1 month Refresh solutions every 7 days
Citric acid / $K_2S_2O_5$	1000 - 2000 ppm	Up to 30 days
Nitrogen blanket	Pure - pressure 1 - 1.5 barg	Up to 30 days Check pressure regularly

TSG Capabilities

The Technical Support Group strive to work together with you to provide an excellent customer experience. Our proactive approach aims to optimise your manufacturing process, by providing innovative filtration solutions, whilst guaranteeing product quality



Training

- Technical, instrumentation & application specific training packages
- Tailor made to customer needs
- Online courses available

New System Design / Optimization

- Application specific filtration selection and sizing
- Small scale trials
- Performance monitoring

Return Product / Sample Investigation

- Troubleshooting customer issues
- Microbial analysis

Compatibility Testing

- Customer specific
- Process simulation

Process Audit

- Troubleshooting
- System optimization

Integrity Testing

- Valairdata 3
- Porecheck IV
- Bevcheck/Bevcheck Plus

Instrument Servicing

- IQ/OQ
- Service and calibration
- Repairs

Technical Queries

- FAQS
- Phone/E-mail TSG Inbox

All the support you need in-house or on site