

HANDBOOK

Cleaning in place

A guide to cleaning technology in the food processing industry

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Introduction

Who is this booklet for?

This booklet is for production managers, technical managers, project managers, quality managers and others who help operate food processing plants. It's also useful for R&D staff who are developing new products or planning investments in new plant equipment or new processing lines.

As a leader in cleaning technology, we at Tetra Pak would like to share what we know about efficient and intelligent cleaning – and keep you informed about the latest developments in the field.

Since this booklet does not cover the hygienic design of processing equipment in detail, line designers and system designers may want to pursue this topic in depth in other ways. We would be happy to discuss this further with you. Just contact your Tetra Pak representative.

What is "cleaning in place"?

Cleaning in place, or CIP, refers to all those mechanical and chemical systems that are necessary to prepare equipment for food processing, either after a processing run that has produced normal fouling or when switching a processing line from one recipe to another. Cleaning in place means that cleaning takes place without dismantling the system.

Why is CIP important?

CIP is an important component in guaranteeing food safety in food processing plants. Successful cleaning between production runs avoids potential contamination and products that don't meet quality standards. Carrying out CIP correctly – from design to validation – ensures secure barriers between food flows and cleaning chemical flows.

It is also important that CIP is carried out effectively and efficiently, and contributes to an overall low total cost of ownership (TCO). From the point of view of food processing, any cleaning time is downtime – the equipment is not productive. Cleaning must also be carried out safely, because very strong chemicals are involved that can be harmful to people and to equipment. Finally, it should be carried out with the least impact on the environment by using minimal amounts of water and detergents and by maximizing the re-use of resources.

What do I need to know?

You need to know your dirt. As well as your chemistry and physics. And you need to know what clean water is, and how water can be re-used in dairy processes.

The food processing industry – whether involving milk, cheese, yoghurt drinks or Béarnaise sauce – benefits immensely from advanced technology that can control processing and protect food quality, from raw materials coming in to packages going out.

How to clean a plant that has been processing food depends on the type of food that has been produced, and under which conditions. Processing temperature and running time affect how the equipment will be soiled. Efficient CIP will depend on your knowledge of how mechanical, thermal and chemical processes work on different types of soiling. It will also depend on your knowledge of how acids and detergents affect different types of soiling and how you can optimize their interaction.

Food soiling

After production using processing equipment, the plant is more or less soiled with the food products that have been inside the plant. As an example, after a tank filled with cream or yoghurt has been emptied it may look like the two first photos on the left.







These two products are rather easy to clean out. A bigger challenge is shown in the third photo, which shows a tubular heat exchanger in a UHT plant that has been heating white milk at about 120 °C.

Generally, the higher the temperature the more soil or fouling is adsorbed on the surfaces of heat exchangers and the more burnt and hard the fouling gets. Production time is also a factor of importance regarding fouling amount, where long production time gives more fouling amount and more burnt-on fouling. The food application and its constituents are of course of importance as well, since it is the food constituents that form the fouling. Juice, milk, starch-rich food, soy milk, etc. all produce different soils with different characteristics and with their own optimum cleaning procedures.

The soil deposited on the walls of the plant comes from the food product that is processed.

The soil is a matrix of the constituents in the food. Soils can initially be divided into two basic types: those that are water-soluble and those that are insoluble in water. Water-soluble soils such as sugars and some minerals are easily removed and are seldom associated with cleaning problems. The water-insoluble soils, however, are harder to remove. These can be divided into **organic soils** and **inorganic soils**.

Organic soils include fats, oils, grease, protein, starch and other carbohydrates. If these components have been heated during processing, the heat may have induced reactions in the soil matrix that make them more difficult to remove. Proteins may, for example, denature and induce further cross-linking reactions with other protein

molecules (see photo above on UHT milk) or may also react with carbohydrates and cause Maillard reactions (caramelization) to take place. Organic soil is most often dissolved by alkaline detergents.

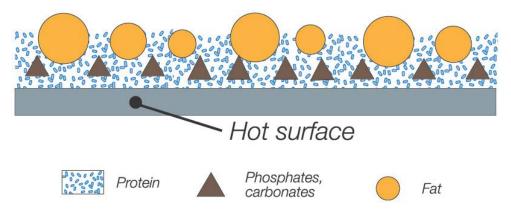
Inorganic soils include mineral and salt deposits. The most common inorganic soil is limescale formed due to high water hardness. Milkstone is also a common inorganic soil. Inorganic soils are most often dissolved by acid.

Alkaline detergents remove organic soil, such as protein and fats.

Acid detergents remove inorganic soil, such as mineral deposits.

Dairy soiling

In a dairy plant there is a clear distinction between soil created on surfaces that are cold, i.e. below 60 °C, and soil created on hot surfaces that are over 60 °C. Examples of cold surfaces are tanks, pumps and pipes. Heated surfaces are all surfaces that have been exposed to temperatures higher than 60 °C, for example, pasteurizers and UHT equipment. On a heated surface, reactions take place between milk components such as protein, fat and minerals. Protein denaturation and aggregation take place, and minerals (in particular calcium phosphate) precipitate. A number of other reactions may also take place. A complex matrix is formed from the milk constituents, which are often difficult to remove during cleaning.



Milk deposits on a heated surface

The temperature on the hot surfaces influences how the milk constituents will form the soil matrix when processing milk products and different reactions occur at different temperatures. In the temperature range from 75 °C to 115 °C the fouling or soiling from a heat exchanger that is heating plain white milk will consist on average of 50-60% protein, 30-35% minerals and 4-8% fat. It is soft and spongy in its texture and is often referred to as protein fouling, or sometimes Type A fouling.

In the temperature range from about 115 $^{\circ}$ C and upwards the fouling or soiling becomes harder and more brittle. The protein content decreases to only 15-20%, fat is more or less maintained at 4-8% and the mineral part increase to 70-80%. This is often referred to as mineral fouling, or sometimes Type B fouling.





Protein fouling

Mineral fouling

There is no distinct temperature where fouling shifts from one type to the other. There is a gradual change towards more and more mineral fouling as the temperature rises and finally at the highest temperature, only mineral fouling is present. This type of fouling is seen in indirect heating of milk. When it comes to direct heating of milk with steam injection or infusion the fouling becomes different, with more protein content in the highest temperature ranges and less minerals compared to indirect heating.

Cleaning in place

Cleaning cooking vessels at home is performed by hand. In the food industry this is called "cleaning out of place", or COP. All equipment is dismantled and cleaned manually.

Today this has been replaced with CIP, cleaning in place, in most parts of the food industry where food is pumped and undergoes continuous processes. Some equipment still needs to be dismantled and manually cleaned, but wherever possible, CIP is the preferred choice. In CIP the equipment is not dismantled, but is cleaned in the same set-up as it was used during production. Cleaning liquid is then circulated through the equipment in a cleaning circuit.

There are two ways of performing CIP. Either the cleaning detergents are put to drain immediately after they have been used. This is called **single-use cleaning** and is often used when the object is very dirty, such as a UHT plant.

The other alternative is when less dirty objects are cleaned, such as tanks or pipes that have cold surfaces. The cleaning solution is not that dirty after one cleaning cycle and it can be reused. This is usually referred to as **recovery CIP**.

Both methods have advantages and disadvantages. In single-use the cleaning solution is always fresh when cleaning is started and the equipment needed to perform single-use CIP is rather inexpensive. On the other hand, this way of running CIP has a high running cost and a high environmental load, as the cleaning solutions are always drained and disposed.

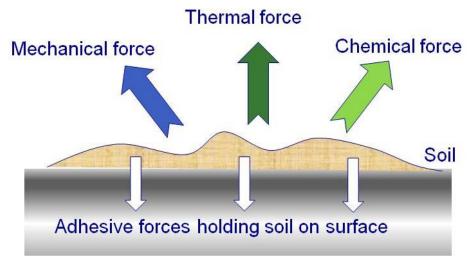
By recovering the cleaning solutions, less cleaning detergent will be consumed, as well as less water and energy. The equipment needed to recover the cleaning solutions is, however, more expensive than the equipment needed for single-use cleaning.

Cleaning parameters

Soil is held on the surfaces by adhesive forces. To get the soil to leave a surface the forces that hold the impurity on the surface have to be overcome. How can we do that?

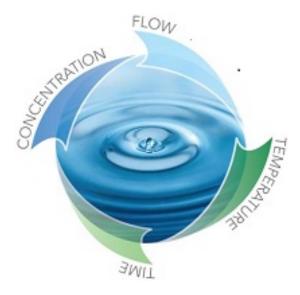
There are four parameters that make up cleaning:

Mechanical force, thermal force (heat), chemical force and the time the forces act.



Forces acting on soil during cleaning

Energy is required in a cleaning process in order to remove the soil and once dissolved, keep it in solution and carry it away. The energy required is kinetic, chemical and thermal energy. These three factors, together with the contact time determine the effectiveness of the cleaning. These four parameters are interconnected and depend on each other, which means that if any of the parameters is changed, the other three might need to be adapted so as to give the same end result as before. They are usually grouped in a diagram called **Sinner's circle** and include flow, temperature, concentration and time. The circle diagram was originally constructed in 1959 by Dr Herbert Sinner, a chemist who worked for Henkel, a German detergent supplier.



Sinner's circle

Mechanical force

The **mechanical force** in cleaning in place is the shear forces created by the flow. Compare cleaning a car with a nozzle on the water hose or without a nozzle. With a nozzle the area through which the water is passing is restricted which increases the velocity of the water and the water jet gets "harder".

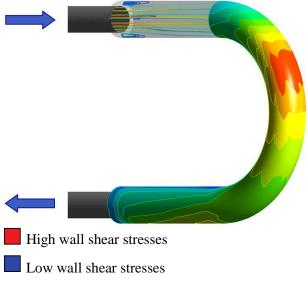
In a plant the flow velocity of the cleaning liquids can be increased by pumping it faster. As a general CIP rule it is said that the flow must be turbulent and that the flow velocity should be at least 1.5 m/s to have an adequate mechanical force. Table 1 below shows the volume flows needed in different pipe diameters to achieve 1.5 m/s.

Table 1 Volume flows needed to achieve 1.5 m/s in different pipe diameters

Pipe Diameter		Flow (I/h)	Volume (litres/100m pipe)
25.0 mm	(1")	~ 2 070	~ 40
38.0 mm	(1.5")	~ 5 100	~ 99
51.0 mm	(2")	~ 9 600	~ 184
63.5 mm	(25")	~ 15 400	~ 287
76.0 mm	(3")	~ 22 500	~ 408
101.6 mm	(4")	~ 40 200	~ 748

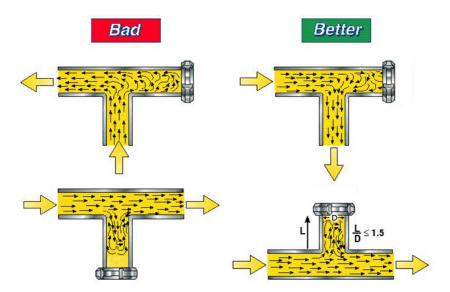
The CIP flow has several purposes – transport the CIP liquid to the soiled surface, react with the soil and finally remove the dissolved soil and transfer it out of the equipment being cleaned.

Naturally, hygienic design of the plant is a prerequisite for the mechanical forces in CIP to have full effect. Perfect cleaning parameters and excellent CIP execution will not give good results if the equipment has design faults, such as dead ends that cannot be flushed through.



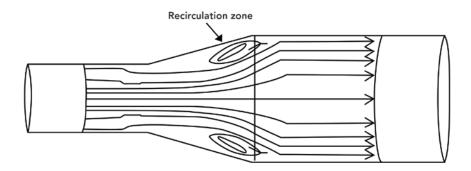
Simulation of wall shear stresses in a bend

Not only average flow velocity is important. Even if average velocity is alright, the shear stresses at the wall can be different in a plant. For example, the pipe bend shown above simulates wall shear stresses. Red means high stresses and blue means low stresses. Then we see there are areas with very high shear stresses and areas with lower shear stresses. The probability of cleaning problems in red areas is low, but problems might develop in the blues areas if average velocity is not high enough.



Flow versus design in a T-piece

From the flow point of view there are some good and bad designs. Dead ends are never desirable, but if you have one, it is better to have flow directed into the dead end than the opposite, to avoid the risk of a stagnant zone. If the dead end is the "leg" of a T-piece, it is better to have the leg turned upwards rather than downwards. The length(L)/diameter(D) ratio should be less than 1.5.



Recirculation zones created by expansion points

Flow at expansion points also merit attention. When the pipe is expanding the flow velocity goes down. Then a recirculation zone is created in this area where shear forces or velocity will be lower than average. This could be a critical point from the cleaning point of view. Too low flow is a common root cause of cleaning problems.

Chemical force

The second force to use to get soil to leave a surface is **chemical force**. To get equipment clean chemicals have to be used in combination with the mechanical

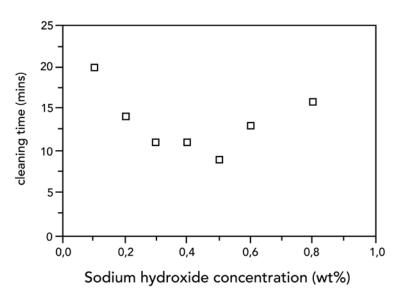
force, the flow. Most often alkaline detergents are used first. They dissolve protein, fat and sugars (i.e. mostly organic soil). The detergent can be pure sodium hydroxide (NaOH) or it can be a formulated detergent usually based on NaOH from a detergent company. In formulated detergents different cleaning aiding components are also added, which might take care of hard water, suspend the dissolved dirt better than pure NaOH, wet the surfaces more efficiently, etc. Sodium hydroxide is typically used at 0.5-2wt% for most applications, but higher levels can be used for some food applications. Too high levels of sodium hydroxide may induce crosslinking of proteins in some food systems, making them even harder to remove.

After an alkaline cleaning step an acid step usually takes place. Acids dissolve minerals, i.e. inorganic soil. It has some effect on fat, sugar and protein as well. Acids commonly used are nitric acid or phosphoric acid. Also for the acid detergents there are formulated detergents available from detergent companies with additional functions than the basic functions of the acid. Nitric acid is typically used in the range 0.5-1.5wt%. The use of nitric acid at higher concentrations needs to be considered with care, since it may then attack polymer material as well as stainless steel.

CIP in dairy applications

How is protein fouling dissolved in a dairy application? Dairy protein fouling consists to a large degree of whey proteins that have denatured and aggregated through various crosslinking reactions. When NaOH comes into contact with the protein fouling, the alkali cuts down the crosslinks holding the protein fouling together. But if the concentration of NaOH is too high it can induce even more crosslinking, which make the fouling even harder to remove. Due to this behaviour of milk protein fouling, there is an optimum NaOH concentration for dissolving the fouling.

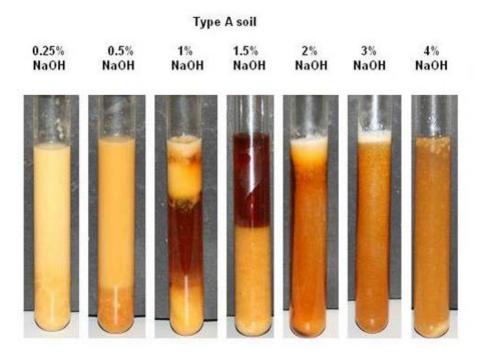
Many studies have been performed to show this optimum value for removing milk protein deposits. Here are the results from one of them. Optimum concentration when cleaning was fastest at about 0.5wt% NaOH.



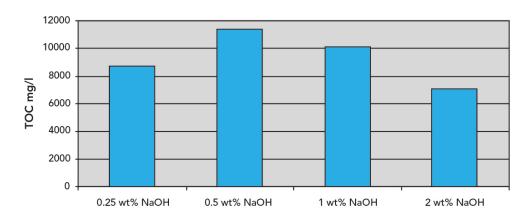
Effect of NaOH concentration on cleaning time of a whole milk deposit at 50 °C Source:

M.R. Bird and M. Bartlett Trans IChemE vol.73 part C June, pp 63-70, 1995

At Tetra Pak, we have also done some lab trials of our own in this area of optimum detergent concentration. Our researchers removed protein fouling from a surface in a plant, put it into test tubes and added hot NaOH of varying concentrations. For protein fouling we observed an optimum dissolving concentration at roughly 0.5% NaOH, in conformity with other results in the literature. From 1.5wt% NaOH and above, the solution gelled into a solid structure. The undissolved material in the test tubes was centrifuged away and the resulting liquid was sent for analysis of total organic carbon, which is a measure of how much organic material was dissolved into the CIP liquid. Here again, the optimum was found to be at 0.5wt% NaOH. Very often, however, the cleaning of dairy equipment uses a dosage from 0.5 up to 1.5% NaOH to avoid using too low of a NaOH concentration and the risk of losing cleaning efficiency.



Milk protein fouling in varying concentration of NaOH at 70 °C



Content of total organic carbon (TOC) in cleaning liquids from the photo above after removal of undissolved matter

For mineral fouling there is no known optimal NaOH concentration as there is for protein fouling; the higher the concentration, the more effective it is, at least up to 2.5wt% NaOH.

Thermal force

The third force to use is **thermal force**, heat. Molecules move faster at an elevated temperature and therefore the effectiveness of a detergent is increased with increased temperature. As a general rule a plant should be cleaned at the same

temperature as it has been processing the food. If a higher cleaning temperature is used, then reactions in the soil layers, such as denaturation and crosslinking may be induced, making the soil harder to remove. Table 2 shows cleaning temperature ranges for some dairy cleaning objects.

Table 2 Cleaning temperature ranges

Type of detergent	Temperature range (°C)	Cleaning objects
NaOH	60-80 °C	Milk collection tankers, tanks and pipes
	70-90 °C	Milk pasteurizers
	90-140 °C	UHT plants
HNO ₃	60-65 °C	Tanks, pipes, milk pasteurizers
	80-85 °C	UHT plants

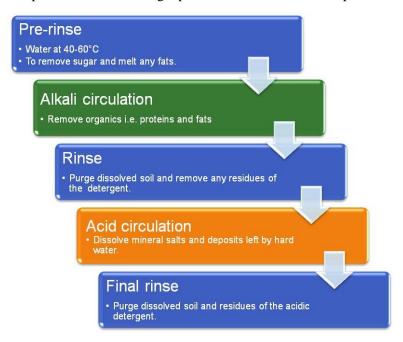
Time

The fourth and last parameter is **time**: how much time the other three forces are in action. Eventually most surfaces will be clean but it will just take longer if the optimal temperature is not used or the correct concentration of detergent or a non-sufficient flow is used.

Cleaning procedures

As part of a normal production cycle, for example, between product runs, it is standard procedure to finalize the production cycle by pushing out the food product with water before the cleaning procedure starts.

The procedure for cleaning a plant often follows these steps.



- 1. The plant is first pre-rinsed with water at 40-60 °C, to remove sugar and melt any fats. The temperature should not exceed 60 °C in order to avoid denaturing any native proteins, which would then become much more difficult to clean.
- 2. Alkaline detergent is then circulated in the system to remove organic soil such as proteins and fats. Alkali is added to the concentration set-point and the temperature is raised to the temperature set-point. The flow is kept at a level giving satisfactorily flow velocity. The alkali step lasts for a pre-set time period.
- 3. Water is then used to purge out the alkaline detergent plus the dissolved soil.
- 4. Acidic detergent is then most commonly circulated through the plant to dissolve mineral deposits and limescale deposits caused by hard water. The frequency of applying an acidic step depends on whether the surfaces are hot or cold, the type of food and the water quality. For example, it is common to only apply an acidic step once a week on cold dairy surfaces. During the acid step concentration, flow and temperature are kept at their set-points for the pre-set time.

5. Water is then used to purge the acidic detergent and rinse out dissolved soil. The final water rinse must also ensure that any detergent residues are removed and only water is left in the plant. Now the plant should be visibly clean.

Disinfection or sterilization is then applied before production starts, in order to kill microorganisms to a certain level. (See more details in Sterilization and disinfection of food processing lines)

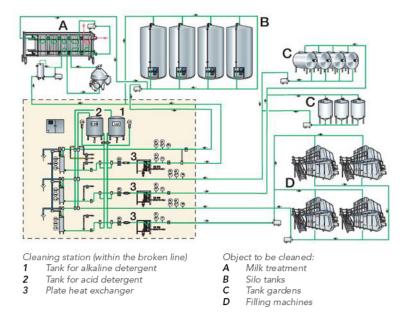
In addition, the results of the cleaning procedure should be verified (see Cleaning verification and validation).

Design of CIP systems

The CIP system

An entire CIP system consists of a CIP station + CIP distribution lines + the objects to be cleaned.

There are in principle two types of CIP system – centralized or decentralized.



Typical components of a centralized CIP system

Centralized CIP systems are most efficient in small plants where the distances are short between the CIP station and the cleaning objects. Centralized systems are also common in relatively large plants where all CIP activities are handled from a centralized cleaning room with one or several CIP stations. Cleaning liquids and water are then pumped from the central CIP stations to the various cleaning objects.

Decentralized CIP systems are more common in large plants where the distances from a centrally located CIP station to the cleaning objects can be extremely long. Instead of using one central CIP room, the decentralized CIP system utilizes several distributed CIP stations positioned close to the cleaning objects. In a decentralized CIP system, it is still common to handle the detergent concentrates centrally. They are then individually distributed to the CIP stations.

In a food plant there are many cleaning objects that should be grouped into larger clusters based on what types of cleaning they demand – for example, cold and hot surfaces – since several cleaning stations are often needed. Cleaning of equipment

handling non-heat-treated food – raw products, for example – should preferably be separated from the cleaning of equipment handling heat-treated food. This is to avoid contaminating surfaces on the processed side with potential surviving bacteria and spores from the raw side, by using the same cleaning liquids on both sides.

CIP system safety

A successful and safe cleaning result is based on controlling the four key parameters of Sinner's circle: the concentration of detergent, the flow rate, the temperature and the time. With safety there is, of course, no room for compromise. You have to measure and control the temperature, and heat detergents to different temperatures, depending on what is to be cleaned. You also have to keep a tight control on the time, even if an alarm or shutdown occurs. Complete control of the flow has to be maintained, adapting it to what is being cleaned. You must also control the concentration of detergent, for example by measuring conductivity.

To assure that no detergent solution contacts the food products, it is important to use a valve solution with "block and bleed" leakage protection at connection points between pipes distributing cleaning liquids and the food processing equipment being cleaned. When the food is being processed there is then security to prevent any CIP liquid present in the CIP distribution pipes from entering the food product flow.

Safety is also crucial in the work environment since cleaning stations use dangerous chemicals. For example, it is advisable to position the dosing pump and the handling of the concentrated chemicals far away from the operator's normal position. Soft metals, such as aluminium, are totally forbidden in a cleaning station since hydrogen gas can develop and form explosive oxy-hydrogen gas when in contact with acid and bases, especially in concentrated form.

The CIP station

A cleaning station contains all the necessary equipment to prepare and store cleaning liquids, distribute the cleaning liquids to the cleaning objects at the correct flow and temperature, and finally monitor the cleaning procedure.

The design of a CIP station is determined by many factors and many questions need to be answered, such as:

- ▶ How many individual CIP circuits will the station serve?
- What are the requirements of the cleaning objects (e.g. hot or cold surfaces)?
- ▶ Will the rinse water and detergent solutions be recovered for re-use?
- Are the product rinses to be collected and processed?
- Which disinfection method will be used: chemical, steam or hot water?
- ▶ What level of automation and control should the system provide:
 - Automatic cleaning recipes and programs?
 - Communication with objects?
 - Local control or central supervision system?

For equipment that is heavily soiled, for instance dairy UHT equipment, the cleaning solutions are often prepared directly in the object to be cleaned and are drained after cleaning since the cleaning solutions are most often heavily soiled and unsuitable for recovery. The equipment is filled with water and a pre-calculated volume of detergent concentrate is added to reach the desired concentration set point. The concentrate is dosed into the object during the time it takes for the water to circulate through the object once. The concentrate will then be equally distributed into the water.

Cleaning verification and validation

Cleaning verification

As there is presently no technique available for measuring cleanliness continuously in line, a plant has to be opened after cleaning at predetermined critical control points in order to assess cleanliness in one or more ways:

- It has to be visually clean without any product residues. This can be checked with a clean white cloth.
- ► Test cleanliness microbiologically, by wiping swabs in certain patterns over specific areas. But incubation and analysis of the samples does take some time.
- A quicker method is to test for the presence of Adenosine triphosphate (ATP) using an enzyme from the firefly (luciferase) that emits light when in contact with ATP. The level of luminescence can then be measured easily, proving the presence of a living organism, or of a substance produced by a living organism.

Cleaning validation

It's one thing to verify the cleaning effectiveness of a particular cleaning cycle. But how can you know if you are systematically following good cleaning regimens that consistently produce an acceptable result that minimizes the risks of spoiled products?

A structured method to validate cleaning must accomplish two things:

- ▶ It verifies the effectiveness of the cleaning procedure for removal of product residues.
- ► It documents evidence that the cleaning process removes residues to predetermined acceptable levels repeatedly and reliably.

A complete validation process might include all these elements:

- 1. Design qualification
 - Hygienic design
 - Hygienic risk assessments
- 2. Installation qualification Checklist of critical areas
- 3. Operational qualification Checklist of critical areas
- 4. Performance qualification
 - Monitor/record critical CIP parameters
 - The mechanical force during cleaning is measured by a flow transmitter.
 - The thermal force is measured by temperature transmitters.

- Time is kept track of.
- Chemical force (detergent concentration) is measured indirectly by electrical conductivity.
- Demonstrate effectiveness and reproducibility of the cleaning process
- Verify that the equipment is cleaned according to predetermined acceptable levels

Water quality

Food processing plants require lots of good quality water – at least drinking water quality – but there are also other requirements that are important for maintaining stainless steel equipment in good operating condition.

Water is used for preparing products in mixing stations, cleaning, starting up lines, as cooling and heating media, flushing out products and rinsing.

Water hardness is of great importance. This is a measure of how much calcium and magnesium there is in the water. There are several units for measuring hardness of water. In Europe use of the German degrees (°dH) are common. But they all can be expressed as mg CaCO₃/L, to make them comparable.

Table 3 Classification of water hardness

Classification of water hardness		
Soft	0 – 6 °dH	
Medium hard	6 – 12 °dH	
Hard	12 – 18 °dH	
Very hard	> 18 °dH	

1 °dH German degree = 17.9 mg CaCO₃/L

Water with a hardness above 7 °dH needs to be softened to a hardness between 4 – 7 °dH (See Table 4). When hard water is heated, calcium carbonate is precipitated, and carbon dioxide and water is formed as well. If equipment is sterilized with hard water, calcium carbonate will precipitate throughout the plant. Scaling of CaCO₃ is, however, easily removed with acid cleaning. Calcium salts may also interfere with detergents and make them less efficient.



Bactofuge disks after cleaning sequence ended with rinse with hard water



Bactofuge disk after cleaning sequence has been aborted during last acid step

Corrosion risks

If chloride (Cl⁻) and chlorine (Cl₂) levels in water are too high, this will cause corrosion of stainless steel. Tetra Pak has recommendations on water quality in order to avoid this corrosion. The most common type is crevice corrosion, some examples of which are shown below.



Crevice corrosion on a flange. Crevice is formed between rubber gasket and metal.



Crevice corrosion at a metal-to-metal contact point on a plate in a plate heat exchanger



Crevice corrosion due to surface scaling. Crevices are formed between scaling and steel.

Tetra Pak criteria for water

In order to ensure proper functioning of Tetra Pak equipment, water that meets the requirements for drinking water is required (European Directive 98/83/EC or WHO Guidelines for Drinking Water Quality). However, intensified demands are required for some parameters for specific types of equipment. The following table shows Tetra Pak recommendations for some water quality parameters that are important from a cleaning perspective.

Table 4 Summary of some Tetra Pak criteria for water

Parameter	Unit	Tetra Pak general design criteria. Max. value
Total hardness	° dH	4-7
Alkalinity (HCO ₃ -)	mg/l	30-120
Chloride (Cl ⁻)	mg/l	30.00 (depending on temperature)
Chlorine (Cl ₂)	mg/l	0.20
Sulphate (SO ₄ ²⁻)	mg/l	100.00
Aluminium (Al)	mg/l	0.10
Iron (Fe)	mg/l	0.10
Manganese (Mn)	mg/l	0.05
рН		7-8.5

Detergents

It is important to understand how detergents are used in cleaning procedures in order to achieve optimal cleaning results – and without wasting money on unnecessary chemicals that further burden the environment.

Detergents can range from pure chemicals such as sodium hydroxide (lye), nitric acid or phosphoric acid to more complex formulated detergents supplied by detergent companies. A third alternative is adding additives to a pure chemical, such as sodium hydroxide, at the food manufacturer. This is a very flexible alternative where you might use only pure chemical for some cleaning objects and create a formulated detergent for others.

It is important to follow the dosage recommendations for the detergents and correctly calculate and dilute the concentrates with water.

Detergent alternatives:

Pure chemicals – sodium hydroxide, nitric acid, phosphoric acid

Formulated detergents

Pure chemicals + additive

Formulated detergents have certain agents added to increase cleaning effectiveness. The main component of all formulated detergents is always an **alkali** or an **acid**. Additional components can include:

- **Surfactants**, or wetting agents, that lower surface tension, enabling them to wet a surface more effectively and make cleaning more efficient.
- **Sequestering agents** can bind calcium and magnesium ions in order to soften water.
- Complex-forming agents can only bind one metal ion per molecule in contrast to sequestering agents, which can bind to a number of metal ions.
- Oxidation Agents can boost cleaning effects. Examples are sodium hypochlorite and hydrogen peroxide.

Detergent concentrations

The detergent solution should be used at a certain concentration, so it is important to know the starting concentration and how to calculate the volume of water needed to achieve desired concentration. The unit for concentration used in industry is **percent** (%), but it is important to distinguish between **weight**% and **volume**%.

Pure chemical concentrates are specified in weight% if nothing else is mentioned. Pure chemicals that are used for cleaning are usually in the following concentration ranges:

Table 5 Concentration ranges of pure chemicals

NaOH (sodium hydroxide)	25-45 wt%
HNO ₃ (nitric acid)	52-68 wt%
H ₃ PO ₄ (phosphoric acid)	75-85 wt%

Recommendations for concentrations used in cleaning programs are almost always in weight%.

Formulated detergent concentrates are always 100% detergent. The composition of the individual components in a formulated detergent is usually not revealed by the manufacturer, only that it is 100% detergent.

Dosing recommendations for formulated detergents are often higher than dosing recommendations for pure chemicals, since they also include water. The detergent concentration levels from detergent companies are usually in weight% if nothing else is written.

Recommendations from suppliers of formulated detergents should always be strictly followed. Guidelines from Tetra Pak Processing Solutions most often refer to pure chemicals, with lower doses than the guidelines for formulated detergents.

Preparation of cleaning liquids

Detergent concentrates have to be diluted with water before they can be used. The final concentration is important, since too low or too high a concentration may result in inadequate cleaning. And concentrations that are too high also waste money and can damage the equipment.

There are in principle two ways of preparing detergent solutions in Tetra Pak processing equipment:

Single-use case (most often for heating units such as sterilizers and pasteurizers) – the detergent concentrate is dosed directly into a water-filled processing unit.

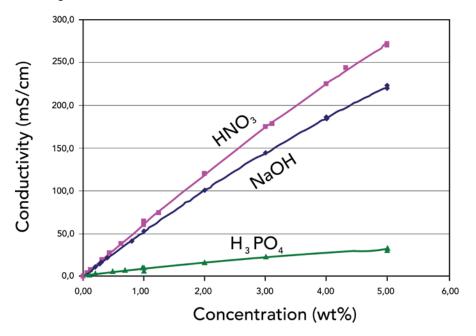
Recovery case – the detergent solutions are prepared in the correct dilutions in a special cleaning station. The detergent solutions are prepared in detergent tanks in which the detergent is recovered after cleaning. When preparing detergent solutions you start with a fixed volume of water into which a certain volume of concentrate should be dosed. These liquids are then pumped through the processing equipment while cleaning.

Inline measurement of concentration

After preparation of the detergent solutions it is important to also measure that the correct concentration has been achieved. This can be done **continuously inline** or using an **offline laboratory method**.

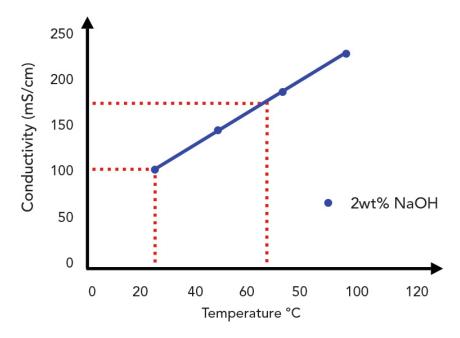
Inline measurement of concentration can be performed by using electrical conductivity: applying an alternating electrical current to two electrodes immersed in a solution and measuring the resulting voltage. How well a solution conducts electricity depends on a number of factors such as chemical composition, concentration, mobility of ions, valence of ions and temperature.

To determine concentration the conductivity must be related to the concentration in a calibration curve. Below you see conductivity for the three most common cleaning chemicals as a function of concentration.



Conductivity versus concentration at 25 °C

Electrical conductivity is strongly dependent on temperature and increases when temperature increases due to increased molecular motion. The conductivity at 25 °C for 2wt% NaOH is about 100 mS/cm. If we measure the same solution at 70 °C the conductivity will be 170 mS/cm. It is thus impossible to compare conductivity at different temperatures!



Temperature dependence of NaOH conductivity

To manage the temperature dependency, conductivity values are recalculated to a value of what it should have been at a reference temperature of either 20 or 25 $^{\circ}$ C. To perform this recalculation, temperature compensation coefficients are used. Conductivity is measured at different temperatures and at the reference temperature. The temperature compensation coefficient (α) can then be calculated.

$$\alpha_{25^{\circ}C} = \frac{(\kappa_{\theta} - \kappa_{25}) \cdot 100}{\kappa_{25} \cdot (\theta - 25)}$$

$$\kappa_{\theta} = \text{conductivity at } \theta^{\circ}C$$

$$\kappa_{25} = \text{conductivity at } 25^{\circ}C$$

From actual measured temperature and conductivity, together with a temperature compensation coefficient (α), the conductivity at the reference temperature can be calculated. This calculation can be done automatically in a conductivity meter when the temperature compensation coefficient has been entered in the meter. This is called automatic temperature compensation.

Temperature
$$\theta$$
 °C Conductivity κ at θ °C α -value $\kappa_{25} = \frac{\kappa_{\theta}}{1 + \frac{\alpha}{100} \cdot (\theta - 25)}$ Conductivity @ 25 °C

Temperature compensation coefficients for the pure chemicals normally used for cleaning is listed below in Table 6.

Table 6 Temperature compensation coefficients

Detergent type	Temperature compensation coefficient (α)
Sodium hydroxide - NaOH	1.7%/°C
Nitric acid – HNO ₃	1.3%/°C
Phosphoric acid – H ₃ PO ₄	-0.01*Temperature + 1.17%/°C

Regarding formulated detergents, information on temperature compensation coefficients should be provided from the detergent companies as well as information on the concentration/conductivity relationship in distilled water. If no information is available on the temperature compensation coefficient, the values for the pure chemicals in Table 6 can be used as an approximation if the detergent is based on sodium hydroxide, nitric acid or phosphoric acid.

Laboratory method

Pure chemicals such as sodium hydroxide and nitric acid

Control of detergent strengths is performed by titration of the cleaning solutions. In titration the neutralization reaction between an acid and a base is utilized. A certain volume of sample (e.g. nitric acid) is metered into a beaker and a titrant (e.g. sodium hydroxide of a known concentration) is added to the acid until it becomes neutral: all hydrogen H⁺ ions have been consumed by the added base OH⁻ and the pH is neutral since only water is left. The sample concentration can be calculated based on the titrant volume used, titrant concentration and sample volume.

Concentration of formulated detergents

To determine concentrations of formulated detergent solutions, follow the instructions on the manufacturer's data sheet. Here is one such example:

Reagents:

- 0.1 N Hydrochloric or sulphuric acid
- Phenolphthalein indicator

Procedure:

Add 2-3 drops of the indicator solution to 10 millilitres of the test solution. Titrate with the acid to a colourless end point.

Calculation:

- \sim % weight/weight detergent = titre (ml) x 0.17
- % volume/volume detergent = titre (ml) x 0.13

The resulting concentration here refers to wt% detergent in contrast to the usual titration procedure above where the result refers to wt% nitric acid or sodium hydroxide etc.

Sterilization and disinfection of food processing lines

Cleaning, sterilization and **disinfection** of processing lines are performed before production re-starts. Sterilization is performed in aseptic lines and lines for extended shelf life (ESL) products. Disinfection is used in non-aseptic production lines (except ESL lines).

After sterilization all microorganisms are inactivated or removed from the surface. Disinfection inactivates all pathogenic microorganisms and reduces the total amount of microorganisms on the surface. Both sterilization and disinfection require cleaning first to be successful.

Cleaning

Before sterilization or disinfection is done a **proper cleaning of the surfaces** has to be performed. Cleaning removes the organic soil that can protect the microorganisms from sterilization and disinfection. Cleaning can also reduce the number of microorganisms on the surface and thus make it easier to sterilize or disinfect. Chemical disinfection is usually more sensitive to how well cleaning is performed than disinfection with heat.

Sterilization

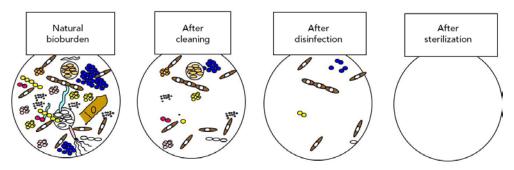
Sterilization is the complete destruction or elimination of all living microorganisms, viable spores, viruses, and viroids. Sterilization can be accomplished by physical (dry or moist heat) and/or chemical methods.

Sterilization of food processing lines with circulation systems are usually done with moist heat. Sterilization can be performed with steam under pressure that gives a temperature of 125 °C for 30 minutes.

Disinfection

Disinfection is a process by which microorganisms are reduced to a level that does not compromise food safety or suitability and is done with chemical and/or physical methods. The major aim of disinfection is to inactivate microorganisms that are harmful to humans. (The term **sanitization** is more commonly used in the food industry in the USA.)

Disinfection of food processing lines with circulation systems can be done with moist heat (hot water at 90-95 °C for 15-20 min or steam <1 bar) or at room temperature using chemicals, to save energy. Disinfection with chemicals requires rinsing with water after disinfection.



Several factors need to be considered when selecting a disinfection method for application in food processing. These include efficacy against vegetative bacteria, yeast, moulds, spores and viruses, so that the desired level of disinfection can be met. For use in food processing equipment the disinfectant must be non-toxic to humans and it must not give any offensive odour to the food. The disinfectant must not be corrosive to the equipment where it should be used. The disinfectant must be stable during the time it should be active and the sensitivity towards presence of organic matter must be known. But it is not desirable to have a disinfectant that is so stable that it will be an environmental problem after use. The disinfectant must be compatible with the water quality of the food production plant.

For use in circulation systems for liquid food processing, thermal or chemical disinfection is applicable, as shown in Table 7.

Table 7 Positive and negative properties of thermal and chemical disinfection methods.

Properties	Thermal disinfection	Chemical disinfection
Bactericidal	Good	Good
Sporicidal	None	Certain effect dependant on temperature, contact time and concentration
Fungicidal	Good except for extremely heat resistant mould spores	Variable
Leaving chemical residues	No	Yes
Rinsing with water (pasteurized or filtered) after disinfection	No	Yes
Cooling of equipment	Yes	No
Penetration ability	Good	Poor/None
Corrosive	No	Yes/No
Energy consumption	High	Low

Novel disinfectants

A lot of work is going on to find new disinfection technologies that can be used in recirculation applications (CIP). The aim is to reduce the environmental impact by replacing traditional chemical disinfectants and reducing the energy consumption used in hot water disinfection. Here we take up two of them: electrolyzed water and ozonated water.

Electrolyzed water

Electrolyzed water – also called activated water, electrolyzed oxidizing (EO) or electro-chemically activated water solution – has recently received attention as an emerging disinfectant technology. It has been applied to water disinfection, disinfection of foods – for example, chicken and cut vegetables – as well as disinfection of food processing equipment. It generally has the same properties as other chlorine-based disinfectants.

Production of electrolyzed water requires only normal salt (NaCl) dissolved in water which is passed through an electrolytical cell within which the anode and cathode are separated by a membrane. Acidic electrolyzed water with antimicrobial properties is produced at the anode containing hypochlorous acid (HOCl), hypochlorite (ClO⁻) and other substances. At the cathode an alkaline electrolyzed water is produced containing mainly hydrogen gas and hydroxide ions, and can be used as a detergent.

Electrolyzed water is very efficient against microorganisms but the effect on spores need more research. Due to the relatively high chloride levels there is a risk of corrosion, especially at temperatures above 25 °C. A minimum pH of 4.5 is required to avoid formation of chlorine gas. Monitoring and control of both chloride levels and pH is thus critical. Any presence of organic material will reduce the antimicrobial efficiency, which requires proper rinsing and cleaning before disinfection with electrolyzed water take place.

Tetra Pak's conclusion is that electrolyzed water is a good chlorine-based disinfectant with a broad spectrum of activity. However, since the presence of chlorine compounds is always a potential threat due to the potential corrosiveness to stainless steel surfaces, further research is needed before this disinfectant can be recommended for use in Tetra Pak processing equipment.

Ozonated water

Ozone (O_3) can be created from oxygen and electricity in an electrolytical cell. The ozone is then dissolved into water, forming ozonated water.

It acts as a strong oxidizer and is efficient as disinfectant. It is also effective on spores, but with a limited active time (\sim 20 min half time). As with electrolyzed water, the temperature must be < 25 °C and the work environment can be a problem.

This technology has mainly been used thus far in waste water treatment plants, swimming pools and aquariums. It is currently not commercially available for CIP systems.

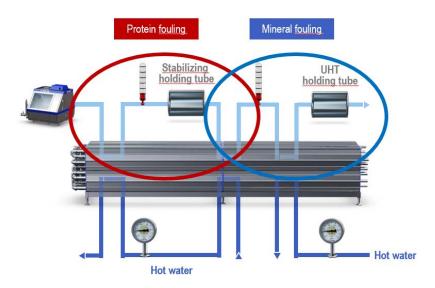
Pushing the boundaries of CIP

Improving the performance of CIP systems is all about manipulating the four basic cleaning parameters – mechanical flow, chemical concentration, thermal energy, and cleaning time – to optimize the overall balance.

Moreover, cleaning is not an isolated event, but integrated into the whole production cycle. The effects we all want to see are increased production time, efficient removal of soil and minimized use of utilities. All of these are closely connected to environmental load and a lower total cost of ownership.

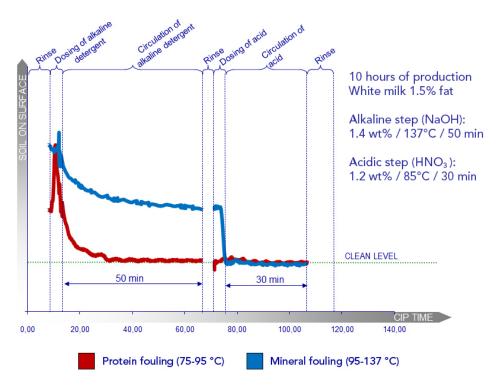
One innovation that Tetra Pak has developed and introduced on indirect UHT is **IntelliCIP**, which uses patent pending sensors to help us "see" what is happening inside the plant when cleaning. This feature is based on the premise that cleaning can be performed faster if it is tailored to a specific product or application, and it can thus affect the whole production cycle and contribute to production uptime.

IntelliCIP monitors the level of soiling present on the internal surfaces of the equipment being cleaned. This allows you to adjust the cleaning parameters (time, temperature, concentration) to what is required for the amount of soiling present, no more, no less.



Typical distribution of fouling in an indirect UHT plant for white milk

In a UHT dairy plant we have two types of soil in general. Protein fouling consisting mostly of protein, while mineral fouling is mainly minerals, brought about by higher temperatures. Since the composition of the two fouling types is very different, they are not dissolved or removed in the same way during cleaning.



Typical monitoring graph from IntelliCIP showing removal of milk fouling in an indirect UHT plant

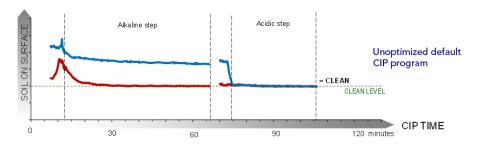
IntelliCIP allows you to monitor how soil leaves the surfaces in the different areas inside the UHT plant. In the example shown in the figure, the low temperature area (75-95 $^{\circ}$ C) is drawn in red and the high temperature area (95-137 $^{\circ}$ C) is drawn in blue.

During the first alkaline step you can see the soil – especially the protein fouling – swell up when it comes into contact with the alkaline detergent. While circulating the alkali, the fouling in the low temperature section is gradually removed until it reaches a clean level. The high temperature areas represented by blue are only somewhat cleaned after circulating alkali for 50 minutes.

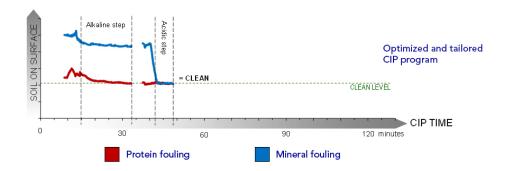
When acid is introduced the mineral fouling is dissolved very quickly and most of the mineral soil is removed after circulating acid only once through the plant.

With this method it is possible to follow how soil is leaving the surface and have an estimate of when the plant is clean. Other cleaning parameters (e.g. concentration and temperature) may also be optimized in a much easier way with continuous monitoring of soil removal.

This method has been validated in full scale. In a dairy plant producing white milk with 1.5% fat after 10 hours of operation, IntelliCIP was shown to shorten the total cleaning time from more than 105 minutes to only 50 minutes, while still being verified to be clean.



Default CIP programme



Optimized CIP programme

IntelliCIP is now available from Tetra Pak. It enables operators to maximize processing uptime and safeguard the CIP result.

Tetra Pak - your CIP partner

Tetra Pak's development engineers, process engineers, designers and field service engineers are very knowledgeable in the field of CIP and cleaning technology. If you would like to gain more insights into hygienic design, operation and cleaning – particularly if you need advice when integrating processing equipment into a line – feel free to contact your Tetra Pak representative or send an e-mail to FCDairy.ProcessingSolutions@tetrapak.com